Effect of Pre-deformation on Precipitation Behavior of Automotive Al-Mg-Si-Cu Alloy (Postprint)

Authors: Cui Li, Guo Mingxing, Peng Xiangyang, Zhang Yan, Zhang Jishan, Zhuang Linzhong

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Abstract

The effects of pre-deformation on the precipitation behavior of Al-Mg-Si-Cu alloy were investigated using mechanical property testing, DSC, and TEM. The results show that during slow heating rate processes, the dissolution rate of GP zones in pre-aged alloys decreases with increasing pre-deformation amount. Using the Avrami-Johnson-Mehl method, the activation energies for GP zone dissolution in alloys subjected to 0, 5%, and 15% pre-deformation were determined to be 137.1, 189.5, and 141.3 kJ/mol, respectively. If the alloy is directly subjected to paint baking hardening at 185 ℃ for 20 min after different pre-deformations, pre-deformation can effectively promote precipitate phase formation and increase the paint baking hardening increment, reaching up to 160 MPa. However, when the pre-deformation amount exceeds 10%, the increase in paint baking hardening of the alloy slows down. Furthermore, the GP zone dissolution rate in paint-baked alloys after pre-deformation treatment is relatively low at certain temperatures, but above a certain temperature, the corresponding GP zone dissolution rate becomes higher than that without pre-deformation treatment, and the resulting ln[(dY/dT)f/f(Y)]-1/T curve even exhibits a transformation phenomenon from high activation energy to low activation energy. However, with increasing pre-deformation amount, the activation energy for β phase precipitation continuously decreases, and the precipitation rate continuously increases.

Full Text

Preamble

Influence of Pre-Deformation on the Precipitation Behaviors of Al-Mg-Si-Cu Alloy for Automotive Applications

CUI Li, GUO Mingxing, PENG Xiangyang, ZHANG Yan, ZHANG Jishan, ZHUANG Linzhong
State Key Laboratory for Advanced Metals and Materials, University of Science and Technology Beijing, Beijing 100083

Correspondent: GUO Mingxing, associate professor, Tel: (010)82375844, E-mail: mingxingguo@skl.ustb.edu.cn

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Abstract

To reduce the weight of car body, Al-Mg-Si-Cu alloys have been widely used to produce outer body panels of automobiles due to their favorable high strength-to-weight ratio, corrosion resistance, weldability and good formability. Al-Mg-Si-Cu alloys belong to age-hardenable aluminium alloys, whose strength derives mainly from the matrix precipitation during aging treatments. However, their bake hardening response still need to be further improved to enhance their dent resistance. A novel thermo-mechanical treatment consisting of conventional pre-aging, pre-deformation and re-aging was developed to enhance the tensile properties and bake hardening increment of Al-Mg-Si-Cu alloys. In this work, the effect of pre-deformation on the precipitation behaviors of Al-Mg-Si-Cu alloy was studied by DSC, mechanical property measurement and TEM. The results show that, the GP zone dissolution rate decreases with increasing pre-deformation during the slow heating up process for the pre-aged alloy, the corresponding activation energies of 0, 5% and 15% pre-deformed alloy calculated by Avrami-Johnson-Mehl method are 137.1, 189.5 and 141.3 kJ/mol, respectively. If the pre-deformed alloys are directly bake hardened at 185 ℃ for 20 min, precipitation and bake hardening increment can be greatly improved by pre-deformation (the highest bake hardening increment is 160 MPa), but the bake hardening increment rate gradually decreases if the pre-deformation is above 10%. In addition, the GP zone dissolution rates of pre-deformed alloys after bake hardening treatment are much lower when the heat treatment temperatures are below one certain value, but if the treatment temperatures above it, the corresponding GP zone dissolution rates are higher than that of alloy without pre-deformation, finally, the activation energy changes from high value to low value even can be observed in the ln[(dY/dT)f/f(Y)]-1/T curve. For the β precipitation in the alloys, with increasing pre-deformation, its activation energy gradually decreases, corresponding gradually increase of precipitation rate.

Key Words: Al-Mg-Si-Cu alloy, automotive sheet, pre-aging, pre-deformation, kinetics

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Introduction

With growing awareness of energy conservation and environmental protection, automotive lightweighting has become a frontier and hot issue in automotive industry technology in the 21st century. One important approach to achieve lightweighting is the use of lightweight aluminum alloy materials, such as Al-Mg and Al-Mg-Si series alloys. The latter is widely used for manufacturing automotive body outer panels due to its excellent stamping formability and bake hardening performance. Currently, widely applied alloy grades in the Al-Mg-Si series include AA6016, AA6111, and AA6022, which have demonstrated good formability and bake hardening capability, attracting significant attention and application from automotive manufacturers both domestically and internationally.

However, with continuous development and in-depth research on aluminum alloys for automotive lightweighting, it is difficult for alloy sheets prepared with a single composition or process to fully meet the different requirements for formability and strength of various automotive body components. For example, some parts undergo large deformation during stamping, imposing strict requirements on sheet formability, while also requiring the highest possible bake hardening increment during the painting process to avoid denting of the final painted alloy sheets during collision. Previous studies have shown that pre-aging treatment of solution-treated Al-Mg-Si alloys is beneficial for improving or stabilizing the stamping formability of alloy sheets and ensuring good bake hardening increments during the painting process. However, after pre-aging, the subsequent stamping deformation varies with component geometry, leading to differences in bake hardening increments during the painting process. Therefore, understanding the influence 规律 of different pre-deformation amounts on subsequent aging precipitation behavior, precipitate size, morphology, distribution, and bake hardening increment is crucial for better designing aluminum alloy materials for different automotive components and optimizing processing parameters.

This work focuses on pre-aged Al-0.6Mg-0.9Si-0.2Cu-0.07Mn (mass fraction, %) alloy sheets. The alloy was subjected to different degrees of pre-deformation, followed by investigation of the corresponding precipitation behavior, bake hardening response, and microstructural evolution, aiming to obtain the influence 规律 of different pre-deformation amounts on bake hardening increment, precipitation behavior, and microstructure, thereby providing guidance for the production and application of this alloy system.

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1 Experimental Methods

The experimental alloy had a composition of Al-0.6Mg-0.9Si-0.2Cu-0.07Mn. Raw materials with mass fractions of 99.7% pure Al, Al-25.73%Mg, Al-12.7%Si, Al-50.29%Cu, and Al-9.56%Mn master alloys were melted in a resistance crucible furnace and cast into a water-cooled steel mold (180 mm length × 110 mm width × 90 mm height). The ingot was then cropped and milled, followed by two-stage homogenization at 485 ℃ for 3 h + 555 ℃ for 16 h. Hot rolling was performed on a two-roll mill with 350 mm diameter at a rolling speed of 5.5 r/min, starting temperature of 550 ℃, and finishing temperature below 300 ℃ to a final thickness of 4 mm. After intermediate annealing at 400 ℃ for 1 h, the sheet was cold-rolled to 1 mm thickness. The cold-rolled samples underwent salt bath solution treatment at 555 ℃ for 2 min + pre-aging at 80 ℃ for 12 h, followed by room temperature storage for 14 days (T4P condition). Subsequently, the T4P samples were pre-deformed to 0, 5%, 10%, and 15% strain, and finally subjected to simulated bake hardening treatment at 185 ℃ for 20 min to investigate variations in bake hardening increment and precipitation behavior.

The precipitation behavior of different alloy states was analyzed using a Q2000 differential scanning calorimeter (DSC). Disc specimens of 3 mm diameter × 1 mm thickness (~15 mg) were prepared, with high-purity Al as reference, heated from 20 ℃ to 400 ℃ at 10 ℃/min. Tensile testing was conducted on an MTS810 electro-hydraulic servo material testing machine at a strain rate of 3 mm/min using specimens sampled along the rolling direction according to GB3076-1982 standard. Microstructural observation was performed using a Tecnai G2 F30 field emission transmission electron microscope (TEM). Samples were prepared by mechanical thinning followed by double-jet electropolishing in an electrolyte of 25%HNO_3 + 75%CH_3OH (volume fraction) at -25 to -10 ℃ and 20-30 V.

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2 Theoretical Model

Numerous studies have investigated aluminum alloy precipitation behavior using various theoretical models. This work employed non-isothermal DSC analysis combined with the Avrami-Johnson-Mehl model to study precipitation kinetics. The relevant kinetic equation is:

$$Y = 1 - \exp\left[-k_0 \exp\left(-\frac{Q}{RT}\right) t^n\right]$$

where Y is the volume fraction transformed within time t (in min), k and n are constants related to nucleation type and growth mechanism, k_0 is the frequency factor (min^{-1}), Q is the activation energy (kJ/mol), R is the universal gas constant (8.31 J/(mol·K)), and T is the thermodynamic temperature (K).

Taking logarithms of both sides and rearranging yields:

$$\ln\ln\left(\frac{1}{1-Y}\right) = \ln k_0 + n\ln t - \frac{Q}{RT}$$

The value of n can be determined from the slope of $\ln\ln(1/(1-Y))$ versus $\ln t$ plots. Differentiating equation (1) gives the transformation rate during non-isothermal processes:

$$\frac{dY}{dt} = k_0 \exp\left(-\frac{Q}{RT}\right) f(Y)$$

where $f(Y)$ is an implicit function of Y. From equations (1) and (4), we obtain:

$$f(Y) = n(1-Y)[-\ln(1-Y)]^{(n-1)/n}$$

The expression for Y is:

$$Y = \frac{A(T)}{A_f}$$

where $A(T)$ is the area between the baseline and DSC peak from the peak start to temperature T, and A_f is the total peak area.

The expression for dY/dT is:

$$\frac{dY}{dT} = \frac{dY}{dt} \cdot \frac{dt}{dT} = \frac{dY}{dt} \cdot \frac{1}{\phi}$$

where ϕ is the heating rate (10 ℃/min in this experiment). Combining equations (2), (4), and (7) gives:

$$\ln\left[\frac{(dY/dT)\phi}{f(Y)}\right] = \ln k_0 - \frac{Q}{RT}$$

It is evident from equation (8) that $\ln[(dY/dT)\phi/f(Y)]$ versus $1/T$ should yield a straight line, from whose slope $-Q/R$ the activation energy for dissolution or precipitation can be determined, establishing the corresponding kinetic relationship.

3.1 Influence of Pre-Deformation on Precipitation Behavior of Pre-Aged Alloy

[Figure 1: see original paper] shows DSC curves of T4P-treated Al-0.6Mg-0.9Si-0.2Cu-0.07Mn alloy after different pre-deformations. The DSC curves of the pre-aged alloy primarily include dissolution peak a, and precipitation peaks b and c. Pre-aging effectively promotes the formation of nano-sized Mg-Si solute atom clusters that are relatively stable and can directly serve as nucleation sites for precipitation-strengthening phases during subsequent high-temperature bake hardening. Consequently, pre-aged alloys exhibit no other solute atom cluster precipitation peaks in the low-temperature region, and dissolution peak a is smaller compared to alloys without pre-aging. However, without proper pre-aging, solution-treated and water-quenched alloys rapidly form Mg-Mg and Si-Si solute atom clusters within 7 days of natural aging. The Mg-Mg clusters dissolve within 7 days, while Si-Si clusters remain relatively stable at room temperature. With prolonged natural aging, Mg atoms continuously enter Si clusters to form Mg-Si clusters. This precipitation process fails to form a large number of stable Mg-Si clusters or results in clusters that are too small, leading to larger dissolution peaks at lower onset temperatures in DSC analysis. Direct high-temperature bake hardening may cause bake softening, and various other solute atom clusters continuously form during DSC heating. Therefore, recent research on Al-Mg-Si alloys for automotive lightweighting has employed suitable pre-aging treatments to improve both stamping formability and bake hardening performance.

Since alloy sheets require stamping after pre-aging, the deformation amount varies with component geometry. To better analyze the effect of different deformation amounts after pre-aging on precipitation behavior, T4P-state alloys were pre-deformed to 5%, 10%, and 15%. [Figure 1: see original paper] shows that after 5% pre-deformation, the GP zone dissolution peak is relatively small, while after 15% pre-deformation, the dissolution peak area increases significantly. However, the dissolution end temperatures for both pre-deformed conditions are higher than that of the non-pre-deformed alloy. The higher dissolution end temperature also shifts the β-phase precipitation onset temperature to higher values. This occurs because pre-deformation accelerates solute atom diffusion, causing the solute clusters formed during T4P treatment to grow rapidly or new clusters to precipitate. Since no new solute cluster precipitation peaks are observed in the low-temperature region of DSC curves, pre-deformation does not promote new cluster formation but rather enhances the growth of existing clusters from the T4P state. However, the diffusion rates of Mg and Si solute atoms differ significantly, with Mg being much slower than Si (DMg = 0.06-0.0616 cm^2/s vs. DSi = 2.42-2.45 cm^2/s at 177-325 ℃). This inevitably leads to Si atom segregation into existing Mg-Si clusters during DSC heating, reducing the Mg/Si ratio in the clusters below that of the β'' phase (Mg/Si ratio of 0.9-1.2). Such clusters cannot serve as nucleation sites for β'' phase and must partially dissolve at elevated temperatures. Only when the Mg/Si ratio re-enters the β''

range can they act as nuclei for β’’ growth. With increasing pre-deformation, solute diffusion accelerates further, causing more severe cluster growth during low-temperature DSC heating and greater deviation of Mg/Si ratio from the β’’ range. This explains the observed increase in GP zone dissolution peak area and more pronounced shift of β’’ precipitation peak to higher temperatures with increasing pre-deformation. Therefore, to avoid non-uniform Si diffusion into T4P-formed clusters during stamping, alloy sheets should be heated as rapidly as possible; otherwise, this phenomenon will reduce the bake hardening increment.

To quantitatively analyze the effect of pre-deformation on GP zone dissolution activation energy and kinetics, [Figure 2: see original paper] illustrates the detailed calculation process using the Avrami-Johnson-Mehl model. [Figure 2: see original paper]a shows that after 5% and 15% pre-deformation, both the GP zone dissolution onset temperature and dissolution rate are higher than those of the non-pre-deformed alloy. From the ln[(dY/dT)f/f(Y)]-1/T curves in [Figure 2: see original paper]c, the GP zone dissolution activation energies for pre-aged alloys with 0, 5%, and 15% pre-deformation are determined to be 137.1, 189.5, and 141.3 kJ/mol, respectively. The activation energies of pre-deformed alloys are higher than that of the non-pre-deformed alloy due to the non-uniform Si diffusion into Mg-Si clusters during low-temperature DSC heating. However, the activation energy after 5% pre-deformation is significantly higher than after 15% pre-deformation. This is because the rapid DSC heating process is too short to completely eliminate the high density of dislocations generated by 15% pre-deformation. Residual dislocations promote GP zone dissolution, resulting in lower activation energy compared to the 5% pre-deformed condition. The corresponding kinetic equations are: $Y = 1 - \exp[-2.892 \times 10^{15} \exp(-16534/T)t]$, $Y = 1 - \exp[-1.0642 \times 10^{21} \exp(-22846/T)t]$, and $Y = 1 - \exp[-2.577 \times 10^{15} \exp(-17011/T)t]$. The theoretically predicted GP zone dissolution Y-t kinetic curves at 185 ℃ are shown in [Figure 2: see original paper]d, clearly demonstrating that pre-straining increases dissolution difficulty due to reduced Mg/Si ratio and increased cluster size. The dissolution rates after 5% and 15% pre-strain are lower than without pre-strain, with complete dissolution requiring 20-30 min compared to only 6-7 min for the non-pre-deformed alloy.

3.2 Influence of Pre-Deformation on Bake Hardening Properties

[Figure 3: see original paper] shows stress-strain curves of T4P-state alloys after 0, 5%, 10%, and 15% pre-deformation followed by simulated bake hardening at 185 ℃ for 20 min. With increasing pre-deformation, the elongation decreases while yield strength increases, with a maximum increase of approximately 160 MPa compared to the T4P state. However, the strength increment for 15% pre-deformation is only slightly higher than that for 10% pre-deformation. The bake hardening increments after 185 ℃, 20 min simulated bake aging are 45.7, 132.4, 158.8, and 167.1 MPa for 0, 5%, 10%, and 15% pre-deformation, respectively, confirming that pre-deformation promotes bake hardening increment.

Pre-deformation introduces dislocations into the alloy matrix, which effectively promote cluster formation and growth during high-temperature bake hardening, resulting in numerous fine precipitates and increased strength. If the pre-deformation is too small, this promotion effect is insignificant; if too large, the effect also diminishes (as seen with 15% pre-deformation). This occurs because although dislocations provide effective diffusion channels for solute atoms, high dislocation density leads to dislocation tangles in local regions, causing non-uniform diffusion and non-uniform precipitate size and distribution, thereby limiting strength improvement. Particularly, the diffusion coefficient difference between Mg and Si, combined with high Si concentration, causes rapid Si diffusion into clusters when strain energy is too high, limiting the formation of the most effective strengthening β'' phase. As predicted by DSC results, only rapid heating to high temperature (e.g., direct 185 ℃ bake treatment) can accelerate β'' precipitation in pre-deformed alloy sheets.

3.3 Influence of Pre-Deformation on Subsequent Precipitation Behavior of Bake-Hardened Alloy

Since Al-Mg-Si alloys remain significantly under-aged after 185 ℃, 20 min bake hardening, with precipitates difficult to characterize, further DSC analysis was performed on bake-hardened alloys to better understand the differences in clusters, GP zones, and β'' phase, as shown in [Figure 4: see original paper]. No solute cluster precipitation or dissolution occurs in the low-temperature region for alloys pre-deformed to 5% or 15%, while some cluster dissolution appears in the non-pre-deformed alloy. This indicates that 185 ℃, 20 min aging transforms existing clusters into numerous fine GP zones or β'' phase while also forming new small clusters or GP zones. Due to fewer diffusion channels, the new clusters/GP zones in pre-deformed alloys are smaller (some below the critical dissolution size r_c), resulting in slight re-dissolution during subsequent DSC heating. At higher temperatures, all alloys exhibit larger dissolution peaks, primarily corresponding to GP zone dissolution, confirming that β'' phase is not extensively formed after 185 ℃, 20 min aging, with GP zones being dominant. Comparison of GP zone dissolution and β'' precipitation peaks reveals that increasing pre-deformation reduces GP zone dissolution peak area, advances β'' precipitation onset temperature (especially for 15% pre-deformation), and significantly increases β'' precipitation peak area compared to peak C in [Figure 1: see original paper]. This further demonstrates that appropriate pre-deformation effectively promotes transformation from clusters to GP zones and β'' phase, significantly enhancing precipitation kinetics during bake hardening, though the promotion effect slows beyond certain pre-deformation levels.

[Figure 5: see original paper] and [Figure 6: see original paper] show the calculation processes for GP zone dissolution and β'' precipitation activation energies after bake hardening. The intermediate Y-T and dY/dT-T curves for GP zone dissolution ([Figure 5: see original paper]) reveal that at certain temperatures, the dissolution rates of 5% and 15% pre-deformed alloys are lower than the non-pre-deformed alloy, but become higher above a critical temperature. Notably, the 15% pre-deformed alloy exhibits a two-stage ln[(dY/dT)f/f(Y)]-1/T curve: the first stage with high activation energy and the second stage with low activation energy (only 97.7 kJ/mol), even lower than the non-pre-deformed alloy. In contrast, the 5% pre-deformed alloy consistently shows higher activation energy than the non-pre-deformed condition. This confirms that increased pre-deformation not only promotes growth of T4P-formed clusters but also forms new clusters during 185 ℃, 20 min aging. Due to limited time, these new clusters are small and dissolve preferentially during subsequent heating, requiring lower activation energy and resulting in the two-stage phenomenon. For β' ' precipitation ([Figure 6: see original paper]), increasing pre-deformation continuously increases precipitation rate and decreases activation energy. This is because dislocations generated by pre-deformation serve as short-circuit diffusion paths, accelerating atom migration. The new clusters/GP zones formed during 185 ℃, 20 min aging can also act as nuclei for subsequent β' ' growth, gradually reducing activation energy, consistent with results from Hirata and Matsue.

Based on the Q and k_0 values calculated from [Figure 5: see original paper] and [Figure 6: see original paper], the kinetic equations for GP zone dissolution and β' ' precipitation are summarized in . Using these equations, the evolution of GP zone dissolution and β' ' precipitation during further aging at 185 ℃ can be plotted, as shown in [Figure 7: see original paper]. [Figure 7: see original paper]a demonstrates that with increasing pre-deformation, GP zone dissolution rates eventually exceed that of the non-pre-deformed alloy after prolonged aging. [Figure 7: see original paper]b shows that β'' precipitation rates continuously increase with pre-deformation, with the 15% pre-deformed alloy essentially completing precipitation in approximately 2 h.

3.4 TEM Microstructural Analysis

To compare the effect of pre-deformation on precipitate size and distribution, alloys with different pre-deformation amounts were bake-hardened at 185 ℃ for 20 min, then heated from 20 ℃ to 250 ℃ at 10 ℃/min (identical to the DSC process in [Figure 4: see original paper]), water-quenched, and examined by TEM, as shown in [Figure 8: see original paper]. Statistical analysis software calculated area fractions of precipitates in the TEM images as 40.1%, 47.2%, and 49.7% for 0, 5%, and 15% pre-deformation, respectively. With increasing pre-deformation, precipitate size decreases while number density increases significantly compared to the non-pre-deformed alloy. After 15% pre-deformation, the alloy contains numerous fine dot-shaped or short needle-shaped *β*' ' precipitates with higher number density than the other two conditions, though precipitate distribution uniformity begins to decrease due to increased dislocation density causing localized precipitation non-uniformity. This explains why the bake hardening increment for 15% pre-deformation is only slightly higher than for 10% pre-deformation. The Y-T curves in [Figure 6: see original paper]b also show that the transformed fraction Y increases significantly with pre-deformation during heating to 250 ℃, consistent with TEM observations.

Conclusions

(1) Pre-deformation of pre-aged Al-0.6Mg-0.9Si-0.2Cu-0.07Mn alloy during slow heating reduces the Mg/Si ratio of pre-existing clusters due to faster Si diffusion, which not only fails to promote *β*' ' precipitation at high temperatures but also increases the GP zone dissolution area. Using the Avrami-Johnson-Mehl method, the GP zone dissolution activation energies for 0, 5%, and 15% pre-deformation were determined to be 137.1, 189.5, and 141.3 kJ/mol, respectively, and corresponding GP zone dissolution kinetic equations were established.

(2) Direct bake hardening at 185 ℃ for 20 min after various pre-deformations effectively promotes precipitate formation, with bake hardening increment increasing with pre-deformation up to approximately 160 MPa, though the increment rate slows when pre-deformation exceeds 10%.

(3) Avrami-Johnson-Mehl analysis of bake-hardened alloys reveals that GP zone dissolution rates of 5% or 15% pre-deformed alloys are lower than non-pre-deformed alloys below certain temperatures, but become higher above critical temperatures, resulting in high-to-low activation energy transitions in ln[(dY/dT)f/f(Y)]-1/T curves. With increasing pre-deformation, *β*' ' precipitation activation energy continuously decreases while precipitation rate increases.

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