Effects of Particles of Different Sizes on Microstructure, Texture and Mechanical Properties of Al-Mg-Si-Cu Alloy System: Postprint

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Abstract

Through tensile testing, OM, SEM, TEM observations, and EBSD analysis, the effects of differently sized particles on the mechanical properties, microstructure, and texture of Al-Mg-Si-Cu alloy sheets were investigated. The results show that with increasing solute element concentration, both the yield strength and tensile strength of the alloys continuously increase, while the elongation slightly decreases, with certain differences observed among the three directions. In addition, the alloy's average plastic strain ratio $\bar{r}$ also increases with increasing solute element concentration. The differently sized particles in the three alloy matrices are mainly Mg2Si, Al15Mn3Si2, and a-Al(Fe, Mn)Si iron-rich phases. Reasonable matching of these particles' sizes and concentrations can not only induce the particle stimulated nucleation (PSN) effect but also effectively inhibit grain growth, ultimately leading to the formation of numerous fine recrystallized grains during solution treatment, with texture components dominated by rotated Cube texture CubeND18, Goss texture {011}<100>, P{011}<122>, and Cu{112}<111>. Additionally, based on the quantitative relationships among alloy composition, thermomechanical processing, and microstructure, a schematic model illustrating the influence of differently sized particles on the recrystallization nucleation and growth process was proposed.

Full Text

Influence of Particles with Different Sizes on Microstructure, Texture and Mechanical Properties of Al-Mg-Si-Cu Series Alloys

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Abstract

To reduce the weight of car body, Al-Mg-Si-Cu alloys have been used to produce outer body panels of automobiles due to their relatively good formability in the solution treated condition and high strength in the age hardened condition. However, their formability is significantly poor compared to that of steels, which are the major drawbacks to wide-scale application of aluminum in the automotive industry. The microstructural characteristics developed during recrystallization, most notably grain size and crystallographic texture, play a dominant role in controlling the mechanical properties and formability of sheet in the T4 condition. In this work, the effect of particles with different sizes on the mechanical properties, microstructure and texture of Al-Mg-Si-Cu alloys was studied through tensile test, OM, SEM, TEM and EBSD measurement. The results reveal that with increase of solute concentration, the average plastic strain ratio r, yield strength and ultimate tensile strength increase, but the elongation decreases and with different extents in the three directions. In addition, the number of observed particles with different sizes in the alloy matrix such as Mg_2Si, $Al_{15}Mn_3Si_2$ and a-Al(Fe, Mn)Si phases also increases. When the size and concentration of these particles are controlled appropriately, lots of finer recrystallized grains can form during solution treatment due to the particle stimulated nucleation (PSN) effect of coarse particles and pinning effect of finer particles. The main texture components include CubeND18, Goss{011}<100>, P{011}<122> and Cu{112}<111> for the alloy with fine-grained structure. At last, according to the relationship among alloy composition, thermomechanical processing and microstructure, the model of nucleation and growth of recrystallized grains affected by the particles with different sizes was also proposed.

KEY WORDS Al-Mg-Si-Cu alloy, particle, recrystallization, texture, PSN effect

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Introduction

With the continuous increase in automobile production, achieving vehicle lightweighting to reduce emissions has become a critical issue for further development in the automotive industry. Aluminum alloys, with their unique advantages, have emerged as key materials for automotive lightweighting. Among the major aluminum alloy series, the 6××× series (Al-Mg-Si alloys) exhibit excellent properties including moderate strength, heat treatability, good corrosion resistance, and easy surface coloring. These alloys are supplied in the solution-treated condition with relatively low yield strength, providing good stamping formability that can be further enhanced during the final paint-baking process. These characteristics make them highly suitable for automotive body outer panels, and several alloys such as AA6016, AA6111, and AA6022 have already been widely applied [1~4].

However, research and application of Al-Mg-Si alloys for automotive body panels have revealed that while these alloys possess good stamping formability (with average plastic strain ratio r generally reaching above 0.55), there remains a performance gap compared to steel [5]. Therefore, improving the stamping formability of these aluminum alloys while maintaining good paint-baking hardenability is crucial for their widespread application. The manufacturing process for automotive outer panel Al-Mg-Si alloys involves: melting and casting → homogenization → hot rolling → intermediate annealing → cold rolling → solution treatment → pre-aging → paint baking [6]. Variations in alloy composition and this thermomechanical processing sequence lead to significant changes in texture components, content, and microstructure, which in turn substantially affect the stamping formability of the alloy sheets [7~9].

Previous studies [10~15] have shown that when a certain number of coarse particles (diameter > 1 m) exist in the alloy matrix, particle stimulated nucleation (PSN) occurs in the deformed alloy during high-temperature heat treatment, generating numerous recrystallization nuclei around particles. This leads to recrystallization textures dominated by rotated cube texture CubeND and P texture, which are beneficial for improving sheet formability. However, if only coarse particles are present, the distribution of fine recrystallized grains becomes non-uniform, and the fine recrystallized grains produced by PSN can easily grow. Therefore, if additional alloying elements are introduced during composition design or thermomechanical processing is controlled to produce simultaneous presence of micron, submicron, and nanometer-scale particles in the cold-rolled alloy matrix, the high-temperature heat treatment can not only induce abundant recrystallization nucleation but also inhibit rapid growth of these nuclei. This ultimately results in a large number of fine, uniformly distributed recrystallized grains in the alloy matrix [16,17], yielding excellent stamping formability and other mechanical properties. Based on this concept, this work designed novel Al-Mg-Si alloy compositions and conducted systematic research on the influence of different-sized particles on microstructure, texture, and mechanical properties of Al-Mg-Si-Cu alloys. The objective is to establish alloy design principles and thermomechanical processing control strategies to provide important guidance for further development of Al-Mg-Si-Cu alloys for automotive lightweighting applications.

Experimental

The chemical compositions of the experimental Al-Mg-Si-Cu alloys are shown in Table 1 . The raw materials included 99.99% (mass fraction) high-purity Al, industrial pure Mg, industrial pure Zn, and master alloys of Al-10% Mn, Al-20%

Fe, Al-20% Si, Al-50% Cu, and grain refiner Al-5%Ti-1%B (mass fraction). The materials were melted in an SG2-12-10 resistance crucible furnace in a specific order, then poured into a water-cooled steel mold with dimensions of 180 mm × 110 mm × 90 mm. The ingots were subsequently trimmed and subjected to two-stage homogenization treatment (485 °C for 3 h + 555 °C for 16 h) before hot rolling. The hot rolling started at 550 °C and finished below 300 °C, with a final thickness of 4 mm. After intermediate annealing at 400 °C for 1 h, the sheets were cold-rolled to 1 mm. The cold-rolled samples were then subjected to salt bath solution treatment at 555 °C for 2 min and pre-aging at 80 °C for 12 h, followed by room temperature storage for 14 days (T4P condition) before tensile testing.

Tensile tests were conducted on an MTS810 electro-hydraulic servo material testing machine at a crosshead speed of 3 mm/min. Tensile specimens were prepared according to GB3076-82 standard, sampled at angles of 0°, 45°, and 90° to the rolling direction, as shown in Figure 1 [Figure 1: see original paper]. Microstructural observation was performed using an Axio Imager A2m optical microscope (OM). Phase identification was conducted using a SUPRA 55 scanning electron microscope (SEM) equipped with energy-dispersive spectroscopy (EDS). OM and SEM samples were prepared using standard metallographic techniques and etched with Keller's reagent (H_2O:HF:HCl:HNO_3 = 95:1:1.5:2.5 by volume). Texture measurements were carried out using EBSD on an LTRA55 SEM at 20 kV acceleration voltage, working distance of 15-20 mm, and 70° tilt. Data acquisition was computer-controlled, and three-dimensional orientation distribution functions (ODF) were calculated using a two-step method, presented as constant Euler angle ϕ_2 sections ($\Delta\phi_2 = 5°$). EBSD samples were prepared by mechanical polishing followed by electropolishing (20 V, $HClO_4$:CH_3CH_2OH = 5:95 by volume). TEM observations were performed on a G2 F30 S-TWIN field emission transmission electron microscope. TEM samples were prepared by mechanical thinning followed by twin-jet electropolishing (H_2NO_3:CH_3OH = 1:2 by volume).

Results and Discussion

2.1 Mechanical Properties

Figure 2 [Figure 2: see original paper] shows the engineering stress-strain curves of the three alloys after T4P treatment along three directions. Based on these curves, the strength and elongation values at 0°, 45°, and 90° were compared, as shown in Figures 3a-c. With increasing solute element concentration, both yield strength and ultimate tensile strength increase continuously, with essentially consistent values in all three directions. However, elongation varies with alloy composition and tensile direction. Alloy 1 exhibits relatively high overall elongation, particularly reaching approximately 27.5% at 45°, while the other two alloys show slightly decreasing elongation with increasing solute concentration. Notably, Alloy 3 shows the lowest elongation at 45°.

The plastic strain ratio r was measured during tensile testing (at 15% elongation), with variations shown in Figure 3d [Figure 3: see original paper]. Both alloy composition and tensile orientation significantly influence r. The average plastic strain ratio $\bar{r}$ for each alloy was calculated from the three directional values using the formula:

$$\bar{r} = (r_0 + 2r_{45} + r_{90})/4$$

where $r_0°$, $r_{45}°$, and $r_{90}°$ are the plastic strain ratios at 0°, 45°, and 90°, respectively. The calculated $\bar{r}$ values are 0.530, 0.566, and 0.578 for Alloys 1, 2, and 3, respectively, indicating that increasing Fe, Mn, and Si content leads to improved $\bar{r}$ values.

2.2 Microstructure

To understand the influence of composition and thermomechanical processing on microstructure, systematic investigation of microstructures at different states is necessary. While composition changes affect as-cast microstructure, the effects are not significant. To illustrate differences in coarse particles before deformation, the homogenized microstructures were analyzed. Figures 4a-c [Figure 4: see original paper] show microstructures after two-stage homogenization (485 °C, 3 h + 555 °C, 16 h) with heating/cooling rates of 30 °C/h. The microstructures are uniform without low-melting-point phase burning. With increasing Fe, Si, and Mn content, a certain number of coarse particles appear in the matrix. Even Alloy 1 contains some coarse particles due to the slow cooling rate (30 °C/h) after homogenization.

These particles are primarily Mg_2Si, $Al_{15}Mn_3Si_2$, and a-Al(Fe, Mn)Si Fe-rich phases, as shown in Figure 5 [Figure 5: see original paper]. The matrix also contains numerous fine submicron or nanometer-scale dispersoids. After hot rolling to 4 mm and intermediate annealing at 400 °C for 1 h, banded structures are evident (Figures 4d-f), with only Alloy 1 showing partial recrystallization. All alloys contain various-sized dispersoids—some fragmented from homogenized particles and others precipitated during hot rolling and annealing (mainly Mg_2Si). Subsequent cold rolling from 4 mm to 1 mm produces typical elongated fibrous structures, with further particle fragmentation (Figures 4g-i).

Since Mg_2Si precipitated during hot rolling and intermediate annealing must be redissolved for good paint-baking response, solution treatment was performed. Figures 4j-l show fully recrystallized microstructures after solution treatment. With increasing solute concentration, recrystallized grain size decreases, particularly in Alloy 3, which exhibits the finest and most uniform grains. This results from the significant influence of uniformly distributed multi-scale particles on recrystallization: coarse particles stimulate nucleation (PSN effect) while fine particles inhibit grain growth. Alloy 3 contains more dispersoids than Alloys 1 and 2 due to its Mn content and, notably, 0.5% Fe, which promotes Al(Fe, Mn)Si phase formation [18,19]. The coexistence of multi-scale particles yields excellent microstructure in Alloy 3 after solution treatment.

2.3 TEM Microstructure

Figure 6 [Figure 6: see original paper] shows TEM images of Alloy 3 after cold rolling from 4 mm to 1 mm. The alloy develops a multi-scale particle distribution including micron, submicron, and nanometer sizes. Nanometer particles strongly pin dislocations, resulting in tangled dislocation lines within grains containing these particles (Figure 6b). These particles significantly influence recrystallization nucleation and growth during subsequent solution heat treatment.

2.4 EBSD Analysis

The presence of different-sized particles leads to distinct mechanical properties and solution-treated microstructures. EBSD analysis was performed to characterize recrystallized grain size, morphology, orientation, and distribution. Since low-temperature pre-aging at 80 °C has minimal effect on recrystallization structure and texture, EBSD samples were taken directly after solution treatment and quenching. Figure 7 [Figure 7: see original paper] shows grain orientation distribution maps and size distributions. With increasing solute concentration, grain size decreases significantly, with numerous fine recrystallized grains typically surrounding coarse particles—most pronounced in Alloy 3. The multi-scale particle distribution enables PSN by coarse particles while fine particles hinder grain growth, resulting in decreasing recrystallized grain size with increasing solute concentration. Some coarse recrystallized grains nucleate and grow within cold-rolled deformation bands where coarse particle density is low, leading to slightly exaggerated grain growth in these regions. Grain orientation variations are evident: red represents rotated cube texture CubeND, yellow represents Goss texture, pink represents Brass texture, green represents Cu texture, and blue represents P texture.

Figure 8 [Figure 8: see original paper] shows ODF maps for the three alloys. The recrystallized grains are primarily randomly oriented, accompanied by some recrystallization texture and minor retained rolling texture. Alloy 1 texture components include CubeND35 (rotated 35° about the normal direction), Goss{011}<100>, and minor Brass{011}<211>. Alloy 2 shows similar components: CubeND25, Goss, and minor Brass, but the rotation angle decreases from 35° to 25°. Alloy 3 exhibits CubeND18, Goss, Cu{112}<111>, and P{011}<122> textures. The volume fractions of these components, obtained from EBSD data, are listed in Table 2 .

For fcc metals, rolling texture typically includes Cu{112}<111>, S{123}<634>, and Brass{011}<211>[20~22]. During high-temperature heat treatment, recrystallized grains nucleate and grow in deformation cube bands formed during rolling, and rolling texture transforms to recrystallization cube texture through preferred nucleation and growth mechanisms. However, no cube recrystallization texture appears in these alloys; instead, rotated cube textures dominate. With increasing multi-scale particle concentration, the rotation angle of the cube texture gradually decreases from 35° to 25° and finally to 18°, attributed to significant hindrance of grain rotation by the particles. Additionally, Alloy 3, with the highest particle concentration, exhibits PSN during high-temperature treatment, resulting in P texture that benefits stamping performance. Not all particles induce PSN—only those exceeding a critical size l_c. According to literature [23~25], the critical size can be expressed as:

l_c = (l · γ{gb})/(2γ{sb}) - Z

where l is the subgrain size in the deformed matrix; γ{sb} and γ{gb} are low-angle and high-angle grain boundary energies, respectively; and Z is the pinning force on dislocations due to second-phase particles, expressed as Z = $\alpha\gamma$f/m, where α, γ, f, and m are the order constant, grain boundary energy, particle volume fraction, and average particle radius, respectively. For aluminum alloys, γ = 0.3 N/m and α = 1.3. Calculation yields the critical diameter l_c for second-phase particles at nucleation sites. When coarse Al(Fe, Mn)Si particles exceed this critical size, they induce PSN due to high stored strain energy. The resulting recrystallized grain size D_{rec} satisfies:

D_{rec} = 1/√N

where N is the number density of nucleation sites with particle size d. Consequently, Alloy 3 forms numerous fine recrystallization nuclei through PSN that are difficult to grow, yielding significantly reduced recrystallized grain size (Figure 7c) and higher volume fractions of rotated cube and P textures, consistent with its high r̄ value.

2.5 Recrystallization Model

To illustrate how increasing solute concentration (or multi-scale particle concentration) affects recrystallization nucleation and growth, schematic models for low- and high-concentration alloys are presented in Figure 9 [Figure 9: see original paper]. For low-concentration Alloy 1, the matrix contains mainly nanometer or submicron Mg_2Si and $Al_{15}Mn_3Si_2$ particles. These cannot induce PSN to produce numerous recrystallization nuclei, nor can they effectively hinder grain growth, allowing initial recrystallized grains to grow rapidly (Figures 7a and 9a-c). In contrast, high-concentration Alloy 3 contains abundant multi-scale particles including coarse Al(Fe, Mn)Si and fine Mg_2Si, Al_6Mn, or $Al_{15}Mn_3Si_2$ phases. After cold rolling, high stored energy around coarse particles induces abundant recrystallization nuclei during high-temperature treatment (Figure 9e), while fine dispersoids inhibit grain growth, resulting in fine, uniform recrystallized grains. This pinning effect also increases resistance to grain rotation, so Alloy 3 exhibits rotated cube texture but with only 18° rotation.

Conclusions

1. With increasing solute element concentration in Al-Mg-Si-Cu alloys, the average plastic strain ratio r̄, yield strength, and ultimate tensile strength increase continuously. Strength values are consistent in the 0°, 45°, and 90° directions, while elongation decreases slightly with directional variations.
2. After homogenization, all three alloys contain various-sized particles including Mg_2Si, $Al_{15}Mn_3Si_2$, and a-Al(Fe, Mn)Si Fe-rich phases. Increas-

ing solute concentration rapidly increases the number of these multi-scale particles, whose size, morphology, and distribution change significantly during thermomechanical processing. High-concentration alloys with optimized particle size and concentration effectively control recrystallization nucleation and growth, producing fine, uniformly distributed recrystallized structures.

3. After solution heat treatment, the alloys exhibit primarily rotated cube texture CubeND and Goss{011}<100>, with retained rolling texture Brass{011}<211> or Cu{112}<111>. The rotation angle of the cube texture about the normal direction decreases from 35° to 25° and finally to 18° with increasing multi-scale particle concentration, accompanied by the emergence of P{011}<122> texture.

4. The comprehensive properties, microstructure, and texture components are closely related to the concentration, distribution, and interaction of multi-scale particles. Models illustrating the influence of low- and high-concentration multi-scale particles on recrystallization are proposed.

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Note: Figure translations are in progress. See original paper for figures.