Precipitation behavior and tensile properties of A356.2 alloy with different high temperature pre-precipitation temperatures

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Abstract

In order to understand the effect of high temperature pre-precipitation (HTPP) temperature on the precipitation behavior and tensile properties of A356.2 alloy treated by Al-6Sr-7La master alloy, SEM, TEM and tensile tests were applied to investigate the evolution of fracture morphology and precipitates of the alloys under different HTPP temperatures. The results showed that ultimate tensile strength (UTS) and yield strength (YS) of the alloys decrease and elongation (El) increases with HTPP temperature decreasing. When HTPP temperature decreased from 510 °C to 470 °C, coarsen coherent β′ phase appear in α-Al matrix, continuing to decrease HTPP temperature to 450 °C the main precipitate transformed into semi-coherent β′ phase, leading to the change in mechanical properties. In addition, coarsening and transformation of the precipitate were attributed to the reduction of Si concentration which decreases with HTPP temperature decreasing. Moreover, Si nanoparticles precipitated in α-Al matrix, leading to the decrease of UTS and YS to certain extent due to reducing Si concentration during aging process.

1. Introduction

Due to its light weight, high specific strength, high thermal conductivity, high electrical conductivity, corrosion resistance, low thermal expansion coefficient and good fluidity, Al-Si-Mg alloy have been developed in communication field [1–5]. With the development of communication technology, the wall thickness of radiators for communication base station is getting thinner and thinner, which increases the possibility of fracture during transportation and installation [4]. Therefore, higher requirements are needed for Al-Si-Mg alloy mechanical performances. Coarse lamellar or needle-like eutectic silicon and large grain size in unrefined and unmodified Al-Si-Mg alloy have negative effect on its mechanical properties [1, 5–7]. Therefore, in order to improve its comprehensive mechanical properties, refiners and modifiers are usually applied to reduce the grain size of α-Al and improve the morphology of eutectic silicon [8–11]. Even so, solute segregation still existed in as-cast alloy which reduces its mechanical properties [12].

Generally, solution treatment and aging treatment are two main methods to effectively optimize the microstructure and mechanical performances of Al-Si-Mg alloys [13–16]. Recently, researchers have focused on the adjustment of precipitation phase in Al alloys by multi-stage aging, including retrogression and re-aging treatment (RRA), interrupted artificial aging treatment and high temperature pre-precipitation treatment (HTPP). HTPP has a shorter process compared to other multi-stage aging and can be applied to large cross-sectional parts, which is suitable for industrial production of Al-Si-Mg radiators [17–19]. It has been proved that the ultimate tensile strength (UTS) of Al–Zn–Mg–Cu alloy is significantly improved after HTPP treatment [20] and with the increase of HTPP temperature UTS gradually increased. Meanwhile, the UTS reached the maximum when HTPP temperature was close to the solid solution temperature. In addition, there is a larger range of HTPP temperature in Al-5.1Mg-3.0Zn-0.15Cu alloy to reach the maximum of UTS which is 410 °C–450 °C [21].
At present, the alloys using HTPP process are mainly concentrated in 7××× and 5××× aluminum alloys, few researchers focused on Al-Si-Mg alloys. Previously, Al-6Sr-7La composite refinement-modification agent was developed and its synergistic effect on microstructure and tensile properties of A356.2 alloy were investigated [22, 23]. The results showed that Al-6Sr-7La possessed excellent refinement and modification effect which significantly increased the UTS, yield strength (YS) and elongation (El) of A356.2 alloy. Based on this, the effect of HTPP treatment on the precipitation and tensile properties of A356.2 alloy treated by Al-6Sr-7La refinement-modification agent was studied in this work and the precipitation mechanism of the precipitates was analyzed.

2. Materials and experimental procedures

2.1. Material and sample preparation
Commercial A356.2 alloy and Al-6Sr-7La master alloy were used as the matrix and composite refinement-modifier respectively in this work. Detailed preparation processes were as follows. Firstly, about 1kg of A356.2 alloy was melted at 750 °C in graphite crucible in a resistance furnace. Secondly, after the alloy was melted, slags were removed and high-purity argon gas with the flow rate of 1.8 l min⁻¹ was used to degas the melt for 2 min. Thirdly, removed the slag and added 0.5 wt% Al-6Sr-7La master alloy into the melt, then lowered the melt temperature to 730 °C and held for 3 min, after that high-purity argon gas with the flow rate of 1.8 l min⁻¹ was used to degas the alloy for 3 min. At last, holding the melt at 730 °C for 3 min, removed the slag and poured the melt into a cast iron mold (with its inner diameter of 45 mm and height of 160 mm) which was preheated to 200 °C to obtain the as-cast samples. The chemical compositions of A356.2 alloy treated by 0.5 wt% Al-6Sr-7La composite refinement-modifier is shown in table 1.

| Alloy | Composition (wt%) |
|-------|-------------------|
|       | Si    | Mg    | Fe    | La    | Sr    | Zn    | Ti    | Mn    | Al    |
| A356.2| 7.1200| 0.3560| 0.0900| 0.0350| 0.0300| 0.0064| 0.0060| 0.0060| Bal.  |

2.2. Heat treatment process
Figure 1 shows the heat treatment process of the alloy. A356.2 alloy treated by 0.5 wt% Al-6Sr-7La composite refinement-modifier was firstly treated at 540 °C for 3h (solid solution treatment) in box type resistance furnace with temperature accuracy of ±1 °C. After that, specimens were quenched into 60 °C water with transfer time from box type resistance furnace to the water less than 3 s. For HTPP treatment, five factors were taken into consideration, that is, HTPP temperature (T1), HTPP time (t2), quenching temperature (T2), aging
temperature (T3) and aging time (t3). The uniform experiment method was used to reduce the amount of experiment and a U10* (10^8) uniform design was selected with detailed parameters shown in Table 2. Data analysis was carried out on Minitab software. The following HTPP temperatures were selected as 400 °C, 410 °C, 420 °C, 430 °C, 440 °C, 450 °C, 460 °C, 470 °C, 480 °C, and 490 °C. As for HTPP time, 30 min, 40 min, 45 min, 50 min, 55 min, 60 min, 70 min, 75 min, 80 min, and 85 min were applied. 0 °C, 20 °C, 40 °C, 60 °C, and 80 °C were used as quenching temperatures. 150 °C, 160 °C, 170 °C, and 180 °C were chosen as aging temperature and the aging times were selected as 6 h, 6.5 h, 7 h, 7.5 h, 8 h, 8.5 h, 9 h, 9.5 h, 10 h, and 10.5 h respectively. Based on the optimal process, HTPP temperatures (540 °C, 490 °C, 470 °C, and 450 °C) were varied to investigate its effect on the precipitation phases and mechanical properties of the alloy.

### 2.3. Mechanical properties

WDW-200 material testing machine with displacement control and stretching speed of 1.5 mm min⁻¹ was used for room temperature tensile testing. Specimens were cut in accordance with GB/T228–2010. At least 3 samples were tested for each parameter to ensure reproducibility and the average data were used in this work.

### 2.4. Microstructure observation

JEOL JSM-6510A with accelerating voltage of 15 kV and working current of 60 μA was used for tensile fractures observation. FEI Tecnai G2 F20 with accelerating voltage of 200 kV was used for TEM observation and specimens for TEM observation were firstly cut into 300 μm slices, then the slices were thinned to 40–50 μm using sandpapers from coarse to fine, followed by punching into Ø3 mm discs. After that the discs were electrolytically double-jet thinned using a double-jet thinner (Struers Tenupol-5, dry ice and ethanol were applied to control the temperature at −30 ± 1 °C, double-jet electrolyte was 30% nitric acid and 70% methanol solution). Then ion thinning (Gatan 691) with voltage, current and incidence angle of ion beam of 5V, 0.5mA and ±3° respectively was used to increase thin zone area. Liquid nitrogen was used for cooling throughout the process.

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Table 2. Uniform experimental parameters U10* (10^8).

| Levels | T1(°C) | t1(min) | T2(°C) | T3(°C) | t3(h) |
|--------|--------|---------|--------|--------|-------|
| 1      | 490    | 45      | 20     | 170    | 10    |
| 2      | 480    | 60      | 60     | 190    | 9     |
| 3      | 470    | 75      | 0      | 160    | 8     |
| 4      | 460    | 50      | 40     | 190    | 7     |
| 5      | 450    | 50      | 80     | 160    | 6     |
| 6      | 440    | 65      | 0      | 180    | 10.5  |
| 7      | 430    | 85      | 40     | 150    | 9.5   |
| 8      | 420    | 40      | 80     | 180    | 8.5   |
| 9      | 410    | 55      | 20     | 150    | 7.5   |
| 10     | 400    | 70      | 60     | 170    | 6.5   |

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Figure 2. Engineering stress-strain curves (a) and mechanical performance statistics of the alloy under different conditions (b) in uniform experiment.
3. Results

3.1. HTPP uniform experiment

Tensile stress-strain curves for each parameter of the HTPP uniform experiment is shown in figure 2(a). Taking sample 4 as an example, the curves consist of three stages, that is, elastic deformation stage (I), plastic deformation stage (II) and fracture stage (III). During the elastic deformation stage, stress increases rapidly with the increase of strain until the strain exceeds a certain value which reaches to the plastic deformation stage. Continue to increase the strain, necking fracture occurs. Figure 2(b) shows the evolution of mechanical properties of the alloy under different conditions. With the decrease of HTPP temperature, UTS and YS decrease gradually while El increases. In addition, it is clear Sample 1 possesses the largest UTS (305 MPa) and YS (284 MPa). While, Sample 10 has the smallest UTS (144 MPa) and YS (86 MPa), which reduce by 52.79% and 69.72% respectively compared with Sample 1. But Sample 10 possesses the highest El (24.80%) and the El (10.96%) value of Sample 2 is the lowest.

UTS values shown in figure 2(b) were imported into Minitab software and regression analysis was performed to obtain regression equation on UTS (shown in equation 1). Moreover, influence degree of the factors affecting UTS and fit effect of regression equation are shown in table 3.

\[
Y_{UTS} = 5876 + 0.0328T_1(T_1 - 835.3659) + 0.0159T_2(T_2 - 91.8239) + 0.0041T_3^2
\] 

(1)

The maximum value of Y in the corresponding range of each parameter can be obtained through regression equation. According to the regression equation and actual factory production conditions, the best UTS process parameters is shown in table 4.

| Table 3. Multivariate stepwise regression effect. |
|-----------------------------------------------|
| Project | Influential factor | S    | R-Sq | R-Sq(adjustment) |
|---------|--------------------|------|------|------------------|
| UTS     | T1 > T2 > t3       | 10.9 | 98.49| 96.60            |

| Table 4. Optimal HTPP parameters for UTS. |
|------------------------------------------|
| T1  | t1  | T2  | T3  | t3  |
|-----|-----|-----|-----|-----|
| 490 | 85  | 25  | 190 | 6   |

Figure 3. Engineering stress-strain curves (a) and mechanical performance statistics of the alloys under different HTPP temperatures (b).
3.2. Tensile properties

UTS is an important index to express the tensile properties of alloys and as shown in table 3, HTPP temperature has the greatest influence on UTS. Based on the optimum process parameters about UTS (table 4), four HTPP temperatures were applied to further optimize the tensile properties of the alloy and the tensile properties are shown in figure 3. It can be seen UTS and YS gradually decrease with HTPP temperature decreasing while the change in EI is opposite. UTS for the alloys with HTPP temperatures of 510 °C and 490 °C are almost unanimous.
Moreover, there are cracked Si particles in the dimples, indicating the occurrence of transgranular fracture. With the decrease of HTPP temperature, the dimples gradually become deeper, indicating the increase of plasticity.

4. Discussion

4.1. Strengthening phase evolution

Generally, mechanical properties of aluminum alloys are greatly affected by precipitates in Al-Si-Mg alloys [15]. The morphology evolution of strengthening phase in alloy under different HTPP temperatures is shown in figure 5 and it is clear HTPP temperature has great influence on the precipitates, e.g., as the temperature decreases, the size of precipitates increases, and the number of precipitates decreases (figures 5(a-1)–(d-1)). According to the microscopic morphologies (figures 5(a-2)–(d-2)) and Fast Fourier Transform (FFT) results (figures 5(a-3)–(d-3)), nanoscale phase is needle-like \( \beta' \) phase as the HTPP temperature decreases from 510 °C to 470 °C. When HTPP temperature decreases to 450 °C, the precipitates change to rod-like \( \beta' \) phase and a small amount of needle-like \( \beta'' \) phase. Generally, strengthening effect of \( \beta'' \) phase is better than \( \beta' \) phase in Al-Si-Mg alloys [15, 24, 25]. Therefore, as HTPP temperature decreasing, the number of \( \beta'' \) phases decrease with \( \beta' \) phases increasing, resulting in YS decrease.

\( \beta'' \) phase and \( \beta' \) phase are all composed of Mg element and Si element, and their precipitation is influenced by solid solubility of Si and Mg. Mg content in the alloy is much lower than its solid solubility limit, while Si content is much higher than its solid solubility limit in the range of 450 °C–510 °C. Therefore, as the HTPP temperature decreasing from 510 °C to 450 °C, supersaturation of Mg element remains basically unchanged, while supersaturation of Si element decreases, resulting in decrease of Si element participating aging process. Figure 6 shows that some spherical nanoparticles appear in the \( \alpha'\)-Al matrix with the HTPP temperature of 450 °C. According to the HRTEM (figure 6(b)) and FFT results (figure 6(c)), the spherical nanoparticle is determined to be Si nanoparticle. In addition, the Si nanoparticle is not found in the \( \alpha'\)-Al matrix. It shows that Si nanoparticle is easier to precipitate at lower HTPP temperature than the HTPP temperature decreasing from 510 °C to 450 °C. The Si nanoparticle can grow up during the aging process [26], which can reduce the content of Si element participating precipitation of \( \beta'' \) phase and \( \beta' \) phase during aging process. Therefore, due to the decreasing of HTPP temperature and the precipitation of Si nanoparticle, the amount of Si element participating in the precipitation of \( \beta'' \) phase and \( \beta' \) phase during subsequent aging process decreases, leading to \( \beta'' \) phase and \( \beta' \) phase decrease. Generally, in the aging process of Al-Si-Mg alloy, the shorter the peak aging of the same alloy, the larger precipitate under the
same aging time and aging temperature. Some studies have shown that the reduction of Si concentration can shorten peak aging time [27, 28]. Therefore, lower concentration of Si atoms as the HTPP temperature decreasing from 510 °C to 470 °C, shorter peak aging time, resulting in β ′ phase coarsening.

In Al-Si-Mg alloy, the accepted precipitation sequence is: super-saturated solid solution (SSSS) → clusters/GP zones → β ′ → β → β + Si [29–31]. In this study, the precipitation phase is mainly β phase with a small amount of β ′ phases when HTPP temperature is 450 °C. Studies have shown that there is no β ″ phase in Al alloys at temperatures above 400 °C [29, 32, 33]. Therefore, β phase is not precipitated during HTPP process. Meanwhile, it has been shown that β ′ phase will transform into β phase at the temperature of above 200 °C [29, 32, 34, 35]. However, in this work, aging temperature is lower than 200 °C, indicating that precipitation of β ′ phase is not due to high temperature overaging. When supersaturation of Si atom is lower in alloy matrix, β ″ phase is easier to nucleate and grow than β ′ phase during aging stage [36]. Therefore, it is inferred that precipitation of β ′ phase is caused by the change of Si concentration. This can be explained by considering the nucleation probability or rate J of β ′ phase and β phase, expressed by equation (2) [37, 38].

$$J \propto \exp \left( -\frac{\Delta G}{k_BT} \right)$$

(2)

where ΔG is nucleation energy barrier, k_B is Boltzmann constant, T is temperature. In addition, ΔG can be expressed by equation (3) [37, 38].

$$\Delta G = \frac{\Omega}{k_B T^2} \left[ \ln \left( \frac{C_{Si}}{C_{eq}} \right) \right]^{-2}$$

(3)

where C_{Si} is supersaturate of Si atom in matrix before aging, C_{eq} is characteristic concentration value of Si atom, below which precipitate phase cannot precipitate. Moreover, Ω is related with the interfacial energy between nuclei and matrix, which can be assumed as constant for a certain phase to nucleate during aging [39].

Ω value of β ′ phase is much higher than that of β ″ phase, C_{eq} of β ′ phase requires smaller Si concentration than that of β ″ phase and C_{eq} is related to temperature [39]. During aging process after different HTPP, the aging temperature and time all are same, so T and C_{eq} is constant value. Therefore, combined with equation (3), the relationship between C_{Si} and ΔG can be expressed qualitatively, as shown in figure 7. In figure 7, ΔG of β ′ phase and β phase decrease with increasing C_{Si}, and there must be a focal point (C_{eq}) between the two curves, which is due to that higher Ω value of β ′ phase resulting in faster descent speed of corresponding ΔG. When C_{Si} < C_{eq}, ΔG of β ′ phase is lower, otherwise that of β ″ phase is higher. It can be seen from equation (2) that the smaller the G value, the larger the J value, and corresponding precipitate is firstly nucleate and grow in supersaturated matrix. Thence, when C_{Si} is lower than C_{eq}, β ′ phase firstly precipitate, rather than transitioning from β ″ phase. Therefore, precipitation of many β ′ phases during aging stage after HTPP process of 450 °C is attributed to very low C_{Si} value before aging stage.

4.2. Corresponding relationship between precipitation phase and YS

In this work, the change of alloy YS is mainly attributed to precipitation hardening of the strengthening phase [32, 40, 41]. According to the Orowan model, YS of the alloy can be expressed by equation (4) [42].

\[ YS = \frac{4\pi R_T - \rho D}{2d} \]

where R_T is tensile strength, ρ is modulus of Si atoms, d is peak aging time, and D is the diameter of precipitated phase. Therefore, the change of YS can be attributed to precipitation hardening of the strengthening phase. When supersaturation of Si atom in matrix before aging, C_{eq} is constant value. Therefore, combined with equation (1) and equation (2), it is characteristic concentration value of Si atom, below which precipitate phase cannot precipitate. Moreover, Ω is related with the interfacial energy between nuclei and matrix, which can be assumed as constant for a certain phase to nucleate during aging [39].

In this study, the precipitation phase is mainly β phase with a small amount of β ′ phases when HTPP temperature is 450 °C. Studies have shown that there is no β ″ phase in Al alloys at temperatures above 400 °C [29, 32, 33]. Therefore, β phase is not precipitated during HTPP process. Meanwhile, it has been shown that β ′ phase will transform into β phase at the temperature of above 200 °C [29, 32, 34, 35]. However, in this work, aging temperature is lower than 200 °C, indicating that precipitation of β ′ phase is not due to high temperature overaging. When supersaturation of Si atom is lower in alloy matrix, β ″ phase is easier to nucleate and grow than β ′ phase during aging stage [36]. Therefore, it is inferred that precipitation of β ′ phase is caused by the change of Si concentration. This can be explained by considering the nucleation probability or rate J of β ′ phase and β phase, expressed by equation (2) [37, 38].

$$J \propto \exp \left( -\frac{\Delta G}{k_BT} \right)$$

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where ΔG is nucleation energy barrier, k_B is Boltzmann constant, T is temperature. In addition, ΔG can be expressed by equation (3) [37, 38].

$$\Delta G = \frac{\Omega}{k_B T^2} \left[ \ln \left( \frac{C_{Si}}{C_{eq}} \right) \right]^{-2}$$

(3)

where C_{Si} is supersaturate of Si atom in matrix before aging, C_{eq} is characteristic concentration value of Si atom, below which precipitate phase cannot precipitate. Moreover, Ω is related with the interfacial energy between nuclei and matrix, which can be assumed as constant for a certain phase to nucleate during aging [39].

Ω value of β ′ phase is much higher than that of β ″ phase, C_{eq} of β ′ phase requires smaller Si concentration than that of β ″ phase and C_{eq} is related to temperature [39]. During aging process after different HTPP, the aging temperature and time all are same, so T and C_{eq} is constant value. Therefore, combined with equation (3), the relationship between C_{Si} and ΔG can be expressed qualitatively, as shown in figure 7. In figure 7, ΔG of β ′ phase and β phase decrease with increasing C_{Si}, and there must be a focal point (C_{eq}) between the two curves, which is due to that higher Ω value of β ′ phase resulting in faster descent speed of corresponding ΔG. When C_{Si} < C_{eq}, ΔG of β ′ phase is lower, otherwise that of β ″ phase is higher. It can be seen from equation (2) that the smaller the G value, the larger the J value, and corresponding precipitate is firstly nucleate and grow in supersaturated matrix. Thence, when C_{Si} is lower than C_{eq}, β ′ phase firstly precipitate, rather than transitioning from β ″ phase. Therefore, precipitation of many β ′ phases during aging stage after HTPP process of 450 °C is attributed to very low C_{Si} value before aging stage.

**Figure 7.** The relationship between C_{Si} and ΔG in alloy matrix.
where $\sigma_{YS}$ is YS of alloy, $\Delta\sigma_{ppe}$ is precipitation hardening, $G_m$ is shear modulus of the matrix, $b$ is Burgers vector of the matrix, $d_p$ is average particle size and $\lambda$ is the average particle spacing. Also, $\lambda$ is can be calculated by (5).

$$\lambda \approx d_p \left[ \frac{1}{2V_p} \right]^{\frac{1}{3}} - 1$$

where $V_p$ is the average volume fraction of particles.

So YS value of the alloy can be expressed by $V_p$ and $d_p$, as shown in (6).

$$\sigma_{YS} = \frac{0.13G_mb}{d_p} \ln \frac{d_p}{2b}$$

According to equation (6), with $V_p$ value increasing and $d_p$ value decreasing, YS becomes higher ($d_p > e$). In this work, with HTTPP temperature decreasing from 510 °C to 470 °C, $V_p$ value decreases with $d_p$ value increasing, resulting in YS decreasing. Although $V_p$ and $d_p$ are similar at the HTTPP temperature of 450 °C and 470 °C, the strengthening phase contains not only a small amount of $\beta''$ phases but also many $\beta'$ phases at the HTTPP temperature of 450 °C. The semi-coherent $\beta'$ phase reduce YS of the alloy, because low degree of lattice distortion between the $\beta'$ phase and the matrix weakens the pinning effect on dislocations compared with coherent $\beta''$ phase [15, 24, 25].

5. Conclusions

The effect of HTTPP temperature on the precipitation and tensile properties of A356.2 treated by Al-Sr-La composite refinement-modification agent was investigated, following conclusions can be obtained:

(1) HTTPP treatment has a significant effect on the tensile properties of A356.2 alloy, where HTTPP temperature has the most significant effect according to the regression analysis results of the uniform test.

(2) UTS and YS decrease with HTTPP temperature decreasing from 510 °C to 450 °C while the change in EI is opposite. When HTTPP temperature is 510 °C, UTS, YS and EI of alloy can reach to 300 MPa, 271 MPa and 9.3% respectively. During the heat treatment process, the precipitates are mainly $\beta''$ phase and $\beta'$ phase which affect the tensile properties of alloy based on their morphology and quantity.

(3) The solid solubility of Si element decreases during HTTPP process with HTTPP temperature decreasing. Moreover, Si nanoparticles precipitated in $\alpha$-Al matrix reduce the content of Si element participating precipitation of $\beta'$ phase and $\beta''$ phase during aging process. Therefore, the $\beta'$ phase gradually coarsens and the number decreases as the temperature of HTTPP decreases, while the number of $\beta''$ phase increases, leading to the decrease of UTS and YS.

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Data availability statement

Data supporting the findings of this study are available upon reasonable request by the authors.

Conflict of interest

The authors state that there are no conflicts of interest to disclose.
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