Improving element uniformity and mechanical properties of Al–Mg–Si alloy fabricated by twin roll casting with superheat melt treatment

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Abstract
The effect of superheat melt treatment(SMT) on microstructure and properties of Al–Mg–Si alloy fabricated by twin roll casting(TRC) was analyzed using optical microscope, scanning electron microscopy, emission electron probe micro analyzer and transmission electron microscopy. SMT increased the subcooling degree of melt during TRC. The solidification microstructure with high dendrite density and small dendrite spacing was obtained. The second phase was evenly distributed between dendrites and the solute concentration gradient was decreased. Intergranular solute aggregation caused by Reynolds’ dilatancy in TRC slab was effectively suppressed. The homogeneous solute distribution of TRC slab with SMT can be realized by short-time homogenization heat treatment. The size of insoluble particles was greatly reduced. The complete decomposition of non-equilibrium eutectic phase increased the solute concentration in α-Al, which promoted the precipitation of precipitates during aging heat treatment. The tensile strength and yield strength of T6 slab were improved, while the uniform elongation are almost not decreased. The strength and uniformity of slab in T4P state were both improved. The obtained results can help further shorten the production cycle of TRC slabs and improve mechanical properties.

1. Introduction

As a short process, twin roll casting(TRC) has a great advantage of cost compared with the direct chilling casting(DC), and is widely used in the production of aluminum alloy slab [1]. Metal melt is injected into the gap between two rotating water-cooled roller, solidifies and is rolled at the same time. The process is very suitable for the manufacture of thin specifications of products, such as foil.

TRC is a sub-rapid solidification technology. Temperature, melt flow and pressure affect the solidification process simultaneously resulting in special element distribution in the TRC slab [2, 3]. The uneven distribution of elements also limits the the types of products produced by TRC. Only 1, 3 and 8 series aluminum alloys can be produced in industry at present. Element segregation has become a research hotspot of casting and rolling process in recent decades.

In previous studies, grain refinement is the most common method to improve element uniformity of casting slabs or ingots. The eutectic phase is more dispersed by increasing grain boundary density. Grain refinement has been achieved by different scholars through various methods. Adding grain refiners is one of the most important methods [4, 5]. Commonly used refiners are Al–5Ti–B, Al–Ti–C and so on. These refiners can provide a large number of nucleating particles for the solidification process and achieve grain refinement. In addition to this way, Barekar et al applied an intensive shearing to used for TRC, which resulted in a refined, uniform microstructure and the elimination of centre-line segregation [6]. Yang et al prepared magnesium alloy slabs by TRC with fine grains in a similar way [7]. He et al added the static magnetic field and pulsed field during TRC.
### Table 1. Chemical compositions of S1 and S2.

|       | Al     | Mg     | Si     | Fe     | Mn     | Zn     | Cu     |
|-------|--------|--------|--------|--------|--------|--------|--------|
| S1    | 97.692 | 0.543  | 1.158  | 0.093  | 0.092  | 0.234  | 0.088  |
| S2    | 97.597 | 0.539  | 1.142  | 0.124  | 0.111  | 0.258  | 0.109  |

The high frequency periodic Lorentz force was used to break the dendrite arm and make it break away from the solidification interface to reduce the grain size [8].

The refined grains generally have lower dendrite density. Because the grain growth time is short and dendrites cannot grow adequately. The high solute liquid is almost located at the grain boundary. Previous studies have found that this feature may lead to solute aggregation at grain boundary in TRC slabs. While the melt solidifies, it is also affected by the shear force caused by deformation during TRC. The Reynolds’ dilatancy will lead to the increase of some grain gaps, and the surrounding liquid will be sucked in under the action of negative pressure, eventually forming large eutectic phase [9–11]. The large size eutectic phase is difficult to completely eliminate during subsequent heat treatment, which has adverse effects on product performance.

The expansion of intergranular gap under shear force is almost unavoidable during TRC. In addition to increasing grain boundary density, increasing dendrite density also improves solute uniformity. This study tries to find suitable technology to achieve this purpose. Superheat melt treatment (SMT) technology has been paid more and more attention in recent decades. The short-range order structure in the melt can be eliminated by increasing the superheat temperature and holding for a period of time, and the subcooling degree required for spontaneous nucleation is increased to achieve a large number of uniform nucleation and grain refinement. Luo et al prepared Al-20Cu alloy with fine grains by mold casting with SMT [12]. In addition to the effect on the morphology of α-Al, the technique can also refine the second phase. Qin et al refined the size of primary Mg2Si in Mg-Si/Al–Si–Cu composite by increasing the superheat temperature of the melt [13].

In the previous studies, the mold casting process was adopted. The cooling speed was low and the cooling process was stable. TRC is a sub-rapid solidification process, and the melt flow rate in the molten pool is very high. The probability of dendrite arm breakage and oxide involvement is much higher than that of static mold casting. These particles can become heterogeneous nucleation points. Therefore, it is difficult for the melt after SMT to realize large-scale uniform nucleation in the process of TRC. However, the high subcooling degree of melt can reduce the dendrite spacing.

Based on this idea, Al–Mg–Si alloy with high density dendrite was prepared by combining SMT with sub-rapid TRC. The second phase is evenly distributed between high density dendrites, which improves the uniformity of solute distribution. After short time homogenizing heat treatment, the slab prepared by this process has more uniform solute distribution than the conventional TRC slab. The mechanical properties of the slab in T4P and T6 states are improved.

### 2. Experimental procedure

The Al–Mg–Si alloy used in the experiment consists of 0.55 wt% Mg, 1.15 wt% Si, 0.1 wt% Mn, 0.1 wt% Cu and 0.25 wt% Zn. Pure aluminum, pure magnesium, pure zinc, Al–20Si, Al–10Mn and Al–50Cu were melted in a medium frequency induction furnace. Two groups of melts were prepared for TRC. The preparation method of the first group of melt accorded with the conventional TRC. Melt was heated to 710 °C and held for 20 min, and 0.4 wt% Al–5Ti–B grain refiner was added before casting. The prepared slab was named S1. In the second group, melt was heated to 1200 °C and held for 20 min to eliminate short-range order structures in the melt. The slab prepared by TRC with SMT was named S2. The experiment was carried out on the vertical TRC machine developed by our laboratory. The casting speed is 2.5 m·min⁻¹, the roll width is 100 mm, and the rolling force is 180kN. Both two groups of slabs were subjected to short-time homogenization heat treatment at 550 °C for 5 h. Then the slabs were cold rolled to 1 mm thickness. Cold rolled slabs were subjected to T4P and T6 treatment for the testing of mechanical properties. T4P treatment is the process of 565 °C × 10 min solid solution heat treatment, water quenching and 150 °C × 8 min pre-aging heat treatment. T6 treatment is the process of 565 °C × 10 min solid solution heat treatment, water quenching and 175 °C × 8 h aging heat treatment. The actual compositions of S1 and S2 listed in table 1 were obtained by x-ray fluorescence (XRF, ZSXPrimus II, Rigaku, Japan).

An optical microscope (OM, BX53, OLYMPUS, Japan) and a scanning electron microscope (SEM, ULTRA PLUS, Zeiss, Germany) were used to observe the microstructure of the longitudinal section of TRC slabs. The samples for observing the second phase distribution by OM were etched with Keller reagent. The samples for
observing grain shape with OM were anodized by a mixed solution of phosphoric acid and sulfuric acid at 20 V for 90 s. The elemental composition of non-equilibrium eutectic phase was qualitatively analyzed by the energy dispersive spectrometer (EDS) equipped in SEM. The element distribution of homogenized samples were analyzed by the emission electron probe micro analyzer (EPMA, JEOL JXA-8530F, Japan). In order to ensure the accuracy of elemental analysis, all samples used for elemental analysis are unetched. Differential scanning calorimetry (DSC) was used to analyze the melting enthalpy of non-equilibrium eutectic phase of TRC samples before and after homogenization heat treatment. The sample heating rate was 10 °C·min⁻¹, and the standard sample was Al₂O₃. The precipitate density of samples in T6 state were analyzed by transmission electron microscopy (TEM, Tecnai, G2F20, Netherlands). TEM thin foils were prepared at −25 °C by twinjet electropolishing with a solution consisting of 70 vol% methanol and 10 vol% nitric acid at a voltage of 15 V. The tensile test was carried out on a CMT5105 tensile test machine at 20 °C and the initial strain rate was 1 × 10⁻³ s⁻¹. The mechanical properties obtained are the average values of the three groups of samples. The length and width of the parallel section of tensile specimen are 60 mm and 12.5 mm respectively, and the gauge length is 50 mm.

3. Results and discussion

3.1. Solute distribution and second phase

Figures 1(a) and (c) respectively show the grain morphology and second phase distribution of S1. The most prominent feature of S1 is the large size eutectic phase with folded lines distributed between grain boundaries. The Angle between the distribution direction and RD direction is close to 45°, which is a typical characteristic of semi-solid melt solidifies under shear forces [9]. The extended intergranular gap is Reynolds’ dilatancy phenomenon, and the high solute liquid phase flows into the extended intergranular gap under negative pressure, forming the large-size eutectic phase. The thickness of TRC is generally thin, and in the deformation process, most areas are significantly affected by the friction force of roll, forming shear type structure. Rolling type structure exists only in the center of slab, but local solute aggregation caused by Reynolds’ dilatancy is still unavoidable. This effect of pressure on intergranular liquid flow has been described in many previous studies on central segregation [14, 15]. The short-range order structure in the melt can be eliminated by SMT at 1200 °C. Compared with conventional mold casting, the uniform melt structure can be retained more effectively during TRC with high cooling rate. Figures 1(b) and (d) respectively show the grain morphology and second phase distribution of S2. As expected, grain refinement was not observed as in previous mold casting studies [12]. There is a complex flow field in the molten pool, which belongs to a highly disturbed state. A small amount of
oxides and broken dendrites can be used as heterogeneous nucleation points to induce early nucleation. Due to the rapid solidification rate, the heterogeneous nucleated grains grow rapidly before the spontaneous nucleation points are generated in large numbers. Compared with S1 in which grains are refined by a large number of heterogeneous nucleating particles, the heterogeneous nucleation points in S2 samples are greatly reduced due to the extremely high molten pool temperature. Therefore, the grain size of S2 is obviously larger. However, the dendrite structure with high density is obtained. The second phase distributes more uniform in the way of distributing between dendrites. As the tortuous dendrite gap provides greater resistance to liquid flow, more liquid with high concentration solute is trapped in the grain, resulting in the reduction of the size of the intergranular eutectic phase. According to the Kurz-Fisher model \[16\], the secondary dendrite spacing, cooling rate and non-equilibrium nucleation temperature have the following relations:

\[
\lambda_2 = 5.5 \left( M \frac{\Delta T}{T_c} \right)^{1/3}
\]

where \( \lambda_2 \) is secondary dendrite spacing, \( M \) can be regarded as a constant due to its extremely small variation range, \( \Delta T \) is the temperature range of non-equilibrium solidification, \( T_c \) is cooling rate. The superheated melt has a lower initial solidification temperature, resulting in a decrease in \( \Delta T \). There is almost no difference in \( T_c \), because the same TRC process was used to prepare S1 and S2. Therefore, the secondary dendrite spacing can be reduced by SMT during TRC.

EPMA was used to analyze micro-segregation in S1 and S2. An area of 200 \( \mu m \times 200 \mu m \) was selected for matrix dot. A total of 10 \( \times \) 10 evenly spaced points are distributed in the scanning area. Rank sort method \[17\] is used to sort the element concentration at each point, as shown in figures 2(a)–(c). For showing the experimental rules more clearly, the original data points are divided into 10 groups along the horizontal axis, the 10 data points in each group are averaged, and spline curves are used to link shown in figures 2(d)–(f). Compared with S1, the solute concentration gradient from the solidification start point to the solidification end point is smaller in S2. The nucleation temperature of melt decreases after SMT. According to Arrhenius equation, the activation energy of solute atom decreases with the decrease of nucleation temperature, thus improving the diffusion ability of solute atom in melt and suppressing micro-segregation \[18\]. Solutes are trapped in dense dendrites, which also balance solute concentrations in grains and grain boundaries. It can be seen from figure 2 that the concentration of Fe and Si elements in the alloy at the initial solidification stage is significantly reduced, while the Mg element has no such feature after SMT. This may be related to the melting point of the second phase. Fe and Si elements in Al–Mg–Si alloy will form high melting point phases, such as \( \text{Al}_6\text{Fe} \) and \( \beta–\text{Al}_5\text{FeSi} \) phases. These elements have a more obvious short-range order structure in the melt and form rapidly at the initial stage of solidification. SMT can effectively eliminate the short-range order structure and significantly inhibit the formation of the high melting point phase. For low melting point phase with high Mg concentration, such as MgSi, the effect of SMT is obvious at the end of solidification.
The second phase morphology of S1 and S2 were observed by SEM, shown in figure 3. The compositions of the second phase in regions A, B, C and D are shown in table 2. A large amount of rod-like phases and fishbone phases are interlaced to form a large-size eutectic region in S1. The phase types in this region have been analyzed in detail in previous studies, and they mainly include Mg2Si, β-Al5FeSi and Si particles, which are distributed between grain boundaries in the form of binary or ternary eutectic [11, 19, 20]. It can be seen from figure 3(b) that after SMT, the size of the second phase is significantly reduced and the dispersion degree is higher. It is widely accepted that SMT can refine the second phase particles related to uniform melt structure [21]. In addition, the high density dendrite disperses the melt and greatly reduces the local solute concentration. It can be found from the EDS data that there is multiphase symbiosis between the grains of S1, and the contents of Mg, Si and Fe in eutectic region are very high. The concentration of elements in the second phase of S2 decreased, and the decrease of Mg was the most obvious. The end-solidified liquid mainly forms Mg2Si phase with low melting point, and the dendrite has a great influence on its dispersion.

3.2. Eutectic phase decomposition during a short time homogenization

The solidification microstructure with low solute gradient, small second phase can be obtained by TRC with SMT. The time required for complete decomposition of non-equilibrium eutectic phase during

| Mass percentage/ % | Al  | Mg  | Si   | Fe  | Mn  |
|--------------------|-----|-----|------|-----|-----|
| A                  | 71.45 | 8.15 | 14.93 | 4.89 | 0.58 |
| B                  | 73.60 | 7.89 | 13.32 | 4.44 | 0.75 |
| C                  | 79.45 | 0.73 | 9.59  | 8.43 | 1.80 |
| D                  | 79.82 | 3.94 | 11.9  | 3.87 | 0.47 |
homogenization will be greatly reduced. In this study, S1 and S2 were homogenized for 5 h and were denoted as S1-H5 and S2-H5 respectively. DSC was used to analyze S1, S2, S1-H5 and S2-H5, as shown in figure 4.

DSC curves show that an endothermic peak appears at 554 °C, which was caused by the melting of the non-equilibrium eutectic phase. The integral calculation of the peak area gives the change in enthalpy of the sample, which is called the non-equilibrium eutectic phase melting enthalpy ($H_m$). $H_m$ is the integral of DSC with time. (In order to facilitate the observation of temperature cut-off point of endothermic peak, temperature is still selected as the horizontal axis in figure 4). $H_m$ of S1 and S2 are 3.015 J g$^{-1}$ and 5.334 J g$^{-1}$. On the premise of the same eutectic phase in S1 and S2, the melting enthalpy is directly proportional to the mass fraction. Therefore, there are more non-equilibrium eutectic phases in S2. SMT increase the subcooling degree of the melt, which means that the solidification time is shortened. The disequilibrium degree of solidification was further aggravated. The solute concentration of the early formed solid solution decreased. The solute concentration of liquid increased, so that more eutectic phases were formed in S2. However, the eutectic phases in S2 have a larger specific surface area because they are small and dispersed. The second phase have higher decomposition and redissolution rates during homogenization. It can be seen by DSC curve that no endothermic peak appear in S2-H5, while there are still undissolved non-equilibrium eutectic phase in S1-H5.

In order to directly reflect the solute distribution of S1-H5 and S2-H5, EPMA was used for map scanning analysis of the samples. Figure 5 shows the microstructure morphology and element distribution of S1-H5. It can be seen that the large size of the second phase still exists in the slab. The undissolved second phase not only contains the insoluble $\beta$-Al$_5$FeSi phase, but also the soluble Mg$_2$Si phase. Large size of eutectic phase in S1 greatly increase the time required for dissolution of the soluble phase. From the scanning map of Si element, it can be found that the phenomenon of Si element aggregation has appeared. These Si elements exist in the form of larger Si particles, which are formed by the ostwald ripening of eutectic Si [22]. Even if the homogenization time is long enough, the Si particles and $\beta$-Al$_5$FeSi phase are also difficult to dissolve, which adversely affects the mechanical properties of slabs [23, 24].

Figure 6 shows the microstructure morphology and element distribution of S2-H5. Only ferric phase remains in the slab and its negative effect on mechanical properties would be greatly reduced in such small size. The Mg and Si elements are uniformly distributed in the matrix, except that a small amount of Si elements participate in the formation of $\beta$-Al$_5$FeSi phase. The significance of reducing eutectic phase size by adding SMT process in TRC is not only to shorten the time required by homogenization heat treatment, but also to eliminate the large size insoluble phase which cannot be eliminated by heat treatment.

### 3.3. Precipitation and mechanical properties

The acicular precipitate in Al–Mg–Si alloy has pinning effect on dislocations, thus improving the alloy strength. Formation of precipitates followed the sequence of cluster, GP region, $\beta''$, $\beta'$, $\beta$ precipitates. $\beta''$ precipitates have the most significant effect on mechanical properties [25–27]. S1–H5 and S2–H5 were cold rolled to 1 mm thickness, short-time solution treatment was carried out at 565 °C for 10 min, followed by aging treatment at 175 °C for 8 h, named S1–T6 and S2–T6. TEM was used to observe the samples, as shown in figure 7. TEM bright-field and HRTEM images of precipitates S1–T6 and S2–T6 are presented in figure 7. All images were taken with the electron beam parallel to the [001]Al zone axis. The precipitates in S1 and S2 were identified as $\beta''$.
precipitates by calibrating the fast Fourier transform (FFT) images [28]. The precipitate density of S1 and S2 is different. According to the statistics, the precipitate density of S1-T6 is 2.98 × 10³ N·um⁻², and that of S2-T6 is 3.66 × 10³ N·um⁻², which is 22.8% higher than that of the former. The solute element distribution of S2 sample is more uniform. Within the limited heat treatment time, more solute elements will be redissolved from the grain boundary into the grain, improving the solid solubility and increasing the number of precipitates. Similar results are found in the study of Al–Cu–Li alloy prepared by TRC with electromagnetic oscillation field [29].

Figure 5. Element distribution of S1 after homogenized heat treatment: (a) microstructure; (b) Mg; (c) Si; (d) Fe.

Figure 6. Element distribution of S2 after homogenized heat treatment: (a) microstructure; (b) Mg; (c) Si; (d) Fe.
The mechanical properties of S1-T6 and S2-T6 were evaluated by uniaxial tensile test. Engineering stress-strain curves are shown in figure 8. The yield and tensile strength of S1-T6 are 246.63 MPa and 295.62 MPa. After SMT, the yield and tensile strength of S2-T6 are 266.66 MPa and 314.25 MPa, which are increased by 8.12% and 6.30% respectively. The strength contribution of Al–Mg–Si alloy mainly includes grain boundary strengthening ($\sigma_{gb}$), solid solution strengthening ($\sigma_{ss}$), dislocation strengthening ($\sigma_d$) and precipitate strengthening ($\sigma_p$). Thus, the yield strength ($\sigma$) of the alloy can be expressed as:

$$\sigma = \sigma_{gb} + \sigma_{ss} + \sigma_d + \sigma_p$$

(2)

The grain size of S1-T6 and S2-T6 are generally small because of the short solution treatment time. The Halle-Petch coefficient of Al is very small [30]. So the difference of $\sigma_{gb}$ between S1-T6 and S2-T6 is very low. The solute content of the alloy in this study is low and the aging time is long enough to fully precipitate solute elements. The $\sigma_{ss}$ caused by solution strengthening can be ignored. S1-T6 and S2-T6 were subjected the same rolling heat treatment process, and no dislocation was found in TEM images, so dislocation strengthening played a negligible role. The effect of $\beta''$ precipitates on the strength of Al–Mg–Si alloy is obvious. According to the model given by Wang et al [31], $\sigma_p$ of $\beta''$ precipitates can be expressed as:

$$\sigma_p = \frac{MF}{bL}$$

(3)

where M is the Taylor factor, $\bar{F}$ is the average obstacle strength of precipitate, b is the Burgers vector, L is the average spacing of precipitates. According to the research results of Zhang et al [32], L of acicular precipitates can be expressed as:
where $\bar{R}$ is the average radius, $f$ is the volume fraction. Figure 4 shows that compared with S1-T6, the number of $\beta''$ precipitates in S2-T6 increases, resulting in an increase in the volume fraction of $\beta''$ precipitates. By calculation, the increase percentage of yield strength caused by the increase of precipitation density reached 10.81% which approximates the percentage increase in actual yield strength. Therefore, the increased density of $\beta''$ precipitates is the main reason for the higher strength of S2-T6. And the good news is that the uniform elongation of S2-T6 were hardly decreased compared to S1-T6. The uniform elongation of S1-T6 and S2-T6 is 10.34% and 10.08%, respectively. Figure 6 shows that the size of insoluble particles in S1-H5 can reach 20 $\mu$m. These particles are composed of easily fractured Si and Fe bearing phase, which form stress concentration areas during deformation and serve as nucleation centers to form micropores. Micropores aggregate and eventually form cracks. The ductility of the alloy cannot be fully developed due to the existence of large insoluble particles. After SMT and homogenization, the size of insoluble particles is greatly reduced, which almost eliminates the negative effect on the ductility of the alloy. Therefore, even though the precipitate density of S2-T6 is significantly higher than that of S1-T6, its ductility remains at the same level as that of S1-T6. Meanwhile, we also tested the mechanical properties of two groups of samples in T4P state, named S1-T4P and S2-T4P, as shown in figure 8. S2-T4P showed better strength and ductility than S1-T4P. In T4P state, precipitation behavior hardly occurs, only clusters has formed [33]. The resistance of dislocation movement of T4P alloy is smaller than that of T6 alloy. During the deformation process, the negative effect of large insoluble particles on the ductility of T4P alloy is further increased. It was found in previous studies that the large particles promoted recrystallization to increase strength of TRC alloy [34]. However, the dense cluster strengthening caused by high solid solubility in S2-T4P seems to contribute more to the strength in this study. Further research on this issue is ongoing.

**4. Conclusion**

In this study, the effect of SMT on microstructure of TRC was analyzed. Compared with the conventional TRC slab, the slab prepared by TRC with SMT is superior in solute uniformity, homogenization treatment efficiency and mechanical properties. The conclusions are as follows:

1. Due to the Reynolds’ dilatancy of the semi-solid melt under shear deformation, large eutectic phase is inevitable in conventional TRC. SMT increases the subcooling degree of melt during TRC, reduces the dendrite spacing. A small number of heteronucleation points promote the preferential growth of some grains. The grains have enough time to growth and generate microstructure with high dendrite density due to the large reduction of spontaneous nucleation points.

2. After SMT, the size of the second phase of TRC slab has decreased. High density dendrites improve the distribution uniformity of second phase. The microstructure has a higher re-dissolution rate of the second phase in the homogenization process compared to conventional TRC microstructure. After 550 $^\circ$C $\times$ 5 h homogenization heat treatment, the solute distribution of the slab with SMT reach an uniform state. However, the large eutectic phase was not completely dissolved in conventional TRC slab.
(3) After SMT, the tensile strength and yield strength of TRC slab in T6 state are improved due to the increase of precipitate density. More uniform solute distribution results in almost no decrease in uniform elongation. The strength and uniform elongation of T4P state are both increased, which may be related to the dense cluster strengthening and uniform deformation.

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Data availability statement

All data that support the findings of this study are included within the article (and any supplementary files).

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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