Microstructure and Hot Workability of Twin-roll Cast Dispersoid-strengthened Al- Mg-Si-Mn alloys

Xiaoming Qian

Yanshan University

Yu Zhang

Northeastern University

Zhaodong Wang (✉ zhaodongwang@263.net)

Northeastern University

X.-Grant Chen

University of Quebec at Chicoutimi

Yong Li

Northeastern University

Guangming Xu

Northeastern University

Research Article

Keywords: Aluminum alloy, twin-roll casting (TRC), α -Al(Fe, Mn)Si dispersoids, hot workability

Posted Date: December 29th, 2021

DOI: <https://doi.org/10.21203/rs.3.rs-1134249/v1>

License: © ⓘ This work is licensed under a Creative Commons Attribution 4.0 International License.

[Read Full License](#)

Abstract

Dispersoid-strengthened Al-Mg-Si-Mn aluminum alloys were produced by twin-roll casting (TRC) and conventional mold casting (MC). An extra-low temperature homogenization was performed at temperature of 430 °C for 6 h, which was followed by uniaxial hot compression tests. The results showed that the as-cast TRC samples had a lower eutectic fraction with a smaller size and a higher solid solution concentration compared to the as-cast MC samples. During the extra-low temperature homogenization, a large number of α -Al(Fe, Mn)Si dispersoids precipitated, and the dispersoids in the TRC sample had a greater number density than those in the MC sample. Precipitation-free zone (PFZ) formed near the eutectic regions, TRC sample had a lower PFZ fraction than that of MC sample. The TRC samples yielded higher flow stresses of hot deformation than MC sample owing to the stronger dispersoid strengthening effect. Severe edge cracking occurred in the deformed MC samples due to the high fraction of coarse AlFeMnSi intermetallic particles, no edge crack formed in the TRC samples owing to its lower fraction and fine intermetallics which improved the hot workability of TRC sample.

1 Introduction

One of the common strengthening mechanisms for aluminium alloys is via introducing numerous dispersoids in the aluminum matrix, such as Al_3Sc , Al_3Zr , Al_3Y , and Al_3Er , which stabilize the microstructure and generate a particle hardening effect in the alloys [1–3].

In early studies, α -Al(Fe, Mn)Si dispersoids were found to have potential in improving the mechanical properties of aluminum alloys. It has been discussed whether the unit cell type of α -Al(Fe, Mn)Si dispersoids is simple cubic or body-centered cubic. A well-accepted conclusion is that the unit cell of α -Al(Fe, Mn)Si dispersoids is simple cubic in alloys with a low Fe/Mn ratio and shifts into the body-centered cubic when the Fe/Mn ratio increases [4, 5]. α -Al(Fe, Mn)Si dispersoids usually present in the microstructures of 6060 (Al-0.37Mg-0.51Si-0.19Fe-0.1Mn) alloys after homogenization [6]. However, in conventional alloys with traditional processing regimes, the number of α -Al(Fe, Mn)Si dispersoids is quite limited that they are not capable of strengthening the alloys.

Recently, α -Al(Fe, Mn)Si dispersoid-strengthened 6082 (Al-0.8Mg-1Si-0.24Fe-1Mn) alloys were prepared by adding 0.5-1% Mn to 6082 alloys and performing a specific heat treatment [7]. In the 6082 alloy with 0.5% Mn addition, α -Al(Fe, Mn)Si dispersoids started to precipitate at 350°C and reached the highest number density at 400–450°C. Correspondingly, the microhardness of the alloy increased significantly. When the heat treatment temperatures were greater than 500°C, the dispersoids coarsened, and the number density sharply decreased over time. Accordingly, the microhardness dropped severely, indicating the loss of the dispersoid strengthening effect. In previous research by the author of this study [7], the activation energy of deformation in α -Al(Fe, Mn)Si dispersoid-strengthened alloys was quantitatively studied. The activation energy of deformation increased from 191 kJ/mol for the dispersoid-free alloy to 315 kJ/mol for the alloy with 1% Mn addition due to the considerable dispersoid strengthening effect.

To induce large numbers of α -Al(Fe, Mn)Si dispersoids, supersaturated solid solutions of Fe, Mn and Si should be created to provide an adequate driving force and sufficient solution element storage during an appropriate heat treatment. However, the solubilities of dispersoid formers in the Al matrix, such as Fe and Mn, are generally very limited [8], which makes the formation of high concentrations of supersaturated solid solutions difficult under conventional conditions. Liu et al. [9] reported that the high-temperature strength and creep resistance of 3004 (Al-1.24Mn-1.18Mg-0.58Fe-0.25Si) alloy deteriorate rapidly when the addition of Fe was greater than 0.3% due to its consumption of Mn to form $\text{Al}_6(\text{Mn, Fe})$ eutectics during solidification; this phenomenon resulted in a considerable insufficiency of available Mn for dispersoid formation, so fewer dispersoids precipitated during heat treatment.

In terms of solidification, fast cooling is a practical approach to achieve high solid solution concentrations of alloying elements. Twin-roll casting (TRC) is a technology featuring a high solidification cooling rate. TRC is a process in which the molten metal is directly poured on water-cooled rolls, solidification takes place immediately on the surface of the rolls, deformation zone is displayed next to the liquid zone and mushy zone (Fig. 1), along with rolling, cast strips are produced through the combined processes of casting and rolling. It has been reported that the cooling rate in TRC could be up to 2-3 orders of magnitude higher than that in conventional casting. For instance, the cooling rates of DC casting, mold casting was 5.7°C/s [10]. Li reported the cooling rate of TRC process is one order of magnitude higher than that of traditional casting process [11]. Bareker reported the cooling rate of TRC process 10^3 K/s [12].

Regarding wrought alloys, hot deformation is generally applied to achieve desirable dimensions. The hot workability of aluminum alloys is attributed to many factors, such as the alloying element types and contents and the heat treatment history [7, 13, 14]. Qian et al. [6] reported that the hot workability of 6060 alloys was improved by incremental 0.1% Mn addition through the accelerated intermetallic transformation of β -AlFeSi into α -AlFeSi during homogenization. Prasad et al. [2] implied that the hot workability of AZ31 magnesium alloy was controlled by the dissolution of primary eutectics during homogenization by decreasing the risk of intercrystalline cracks during hot deformation.

For decades, dispersoid-strengthened Al alloys have been thoroughly studied, especially alloys related to Al_3M phases with $\text{L}1_2$ structures [15–18]. Recently, α -Al(Fe, Mn)Si dispersoids and their strengthening effect in Al alloys have also started to draw the attention of researchers [19, 20]. However, to date, no systemic study of α -Al(Fe, Mn)Si dispersoid-strengthened alloys produced via TRC can be found in the literature. One of the main reasons may be related to the difficulty of preparing these types of alloys. TRC is more appropriate for producing 1xxx, 3xxx and 8xxx alloys that have a narrow solidification temperature range (less than 30°C). Regarding Al-Mg-Si-Mn alloys, owing to the requirement of a dispersoid former element, a large number of alloying elements are usually added. This addition can cause the solidification temperature range to become as wide as $70\text{--}80^\circ\text{C}$, which makes the preparation of cast strips of Al-Mg-Si-Mn alloy with TRC challenging.

In this study, an α -Al(Fe, Mn)Si dispersoid-strengthened alloy is produced via TRC, and the microstructure and mechanical properties of the alloys are investigated. To comparably study the effect of the superior cooling rate of TRC, a strip made from conventional casting was also prepared. The extra-low temperature homogenization was applied to promote the maximum precipitation of α -Al(Fe, Mn)Si dispersoids. The as-cast and as-homogenized microstructures were observed and quantitatively analyzed. A hot compression test was performed to investigate the hot workability of the experimental alloy. The emphasis was placed on the unique features created by TRC and its corresponding effects on the hot workability.

2. Experimental Methods

Experiments were carried out on an Al-0.8Mg-0.75Si-0.75Fe-1Mn (in wt.%) alloy. TRC was performed on a self-developed horizontal TRC system with water-cooled steel rollers, the dimension the rollers were 500 mm in diameter and were also 500 mm in length. For preparing the alloy, industrial pure Al (99.7%), pure Mg (99.8%), Al-20Si, Al-50Fe, and Al-10Mn master alloys were utilized. During melting operation, pure aluminum ingots were first placed in a TiO₂-coated stainless steel crucible and heated to 750°C, followed by the addition of the master alloys of Al-20Si, Al-50Fe, and Al-10Mn. The melt was then kept at 720°C for 1 h to ensure the full dissolution of the master alloys, and then pure Mg ingots were carefully pressed into the melt, followed by degassing and slag removal. Then the temperature of the melt was smoothly reduced until 690°C when the casting began. The melt was poured into a chute and nozzle made of MgO heat resistant material, and flowed into the gap of the rollers. The rolling speed was 1.2 m/min and the nozzle width was 200 mm, respectively. Strips with a 7 mm thickness were produced. The cooling rate in the biphasic zone during TRC was measured by placing thermocouples in the synchronized nozzle that moved along with the casting strip, and the cooling rate was determined to be 170°C/s. The total length of the solidification-deformation zone was 75mm for the strip with 7mm thickness when using rollers with 500 mm diameter.

In addition to TRC, a sand mold casting (MC) was also performed with the same alloying elements, the smelting and casting temperatures were also the same as those of TRC (720 and 690°C, respectively), a rectangular plate measuring 200 mm × 100 mm × 7 mm was cast with a cooling rate of 1.4°C/s, which is in the range of cooling rates for 7 mm-thick industrial DC casting ingots. Cast strips and plates were subjected to a specific heat treatment at 430°C for 6 h with a heating rate of 100°C/h. This regime was fixed based on the result of our previous investigations, under which the precipitation of α -Al(Fe, Mn)Si dispersoids could be highly promoted [7, 21].

Specimens for microstructure observation and for compression were taken as indicated in Fig. 2, as-cast and heat-treated samples were prepared and polished. An optical microscope (Leica DMI5000M), a scanning electron microscope (Shimadzu, SSX-550) equipped with an energy-dispersive X-ray spectrometer and a transmission electron microscope (JEM-2100) were used. To reveal the dispersoids in the homogenized samples, the polished specimens were etched with 0.5% HF solution for 30 s. An SDT-Q600 differential scanning calorimeter unit was utilized to determine the phase transformation, in which

the as-cast specimens were heated from 25°C to 700°C with a 10°C/min heating rate. Emission electron probe microanalysis (EPMA) was used to measure the alloying element distributions in the as-cast samples. Hot compression tests were performed using an MMS-300 thermal simulator. Specimens were taken from the homogenized strip and machined into a cylinder with a 15 mm diameter and a 7 mm height. The specimens were hot compressed to a total true strain of from 0.3 to 0.5 at the temperatures of 300-500°C and a strain rate of 1 s^{-1} . Electrical conductivity data were obtained using a Sigmascope eddy-current device at room temperature (25°C), and 10 measurements were taken for each specimen to derive an average value. Electron back-scatter diffraction (EBSD) analysis was used to check the deformed microstructures. During the analysis of the EBSD results, misorientation angles of 2-15° and greater than 15° were defined as low- and high-angle boundaries, respectively. Misorientation angles under 2° were removed to avoid noise resulting from the sample surface and experimental polishing. The EBSD operation scanning step size was set as 3.5 μm .

OM, SEM observation surface Rolling direction of TRC Compression sample 7 mm (b)(c)(a)

3. Results

3.1 As-cast microstructure

The as-cast microstructures of the experimental alloy are shown in Fig. 3. The microstructure is consistent with the α -Al dendrites and eutectics that distributed along the dendrite boundaries. The eutectic structure in the TRC sample (Fig. 3b) are much finer than those in the MC sample (Fig. 3a). The scanning electron microscopy (SEM) images in Fig. 3c and d showed that the eutectics of the MC sample had plate-like or Chinese script-like morphologies (Fig. 3c), whereas in the TRC sample they became mainly plate-like particles with much smaller sizes and more fragmented morphologies (Fig. 3d).

The SEM-EDS results in Fig. 3e (point 1 in Fig. 3c), and f (point 2 in Fig. 3c) reveals that the intermetallics are Mg-, Si-bearing and Fe-, Mn-, Si-bearing. According to the previous studies [7], these intermetallics were AlFeMnSi intermetallic (gray particles in Fig. 3c) and primary Mg_2Si , these phases were also mentioned in the references [21, 22]. Quantitative analysis of the eutectics was performed to measure the area fraction and equivalent length of the eutectic particles, and the results are shown in Fig. 4. The area fractions in the MC and TRC samples are 3.1% and 2.3%, respectively. The equivalent length of the eutectic particles decreased from 18.3 μm in the MC sample to 3.4 μm in the TRC sample.

Differential scanning calorimetry (DSC) analysis was performed on the as-cast specimens, and the results are shown in Fig. 5. Both the endothermic peaks of Mg_2Si and AlFeMnSi of the TRC sample were considerably smaller than those of the MC sample. This result implied the lower volume fraction of eutectics in the TRC sample than that in MC sample, which was consistent with the result of eutectic area fraction in Fig. 4.

In general, the formation of eutectic and its size and fraction was closely related to the solidification conditions [11]. A fast cooling led the solution atoms in the melt arrested at a high rate via motion at the

solid/liquid interface [23], which resulted in more alloying elements to be retained in the Al matrix than in the eutectics. Accordingly, a smaller fraction and smaller size of eutectics are generated.

During solidification, the cooling rate of the casting was directly depended on the heat transfer at the interface between the melt and mold. For MC condition, the heat transfer was considerably low owing to not only the sand mold itself but the gap between the mold and solidified material caused by the mid-wall shrinkage. For the TRC process, solidification took place at the roll surface as the melt encountered the rolls. A higher initial cooling rate was imposed by the water-cooled steel roll compared to the sand mold in MC. Besides, a rolling force performed on the solidified material throughout the whole casting duration, and therefore, the volume shrinkage could be compensated by the rolling process and no gap at the interface between strip and rolls could be generated. Consequently, a smaller fraction and smaller size of eutectics were generated in TRC sample than in MC sample (Fig. 4). Another factor influencing the eutectics in TRC was believed to be related to the deformation occurred during TRC. During TRC, when the eutectics formed and was transferred into the deformation zone from mushy zone (Fig. 1), fracture of the brittle eutectics might occur under the rolling force and high temperatures. The fracture of eutectics accordingly resulted in the fragmented eutectic particles in the TRC sample (Fig. 3b, d), which was attributed to the refined eutectics of the TRC sample comparing with the MC sample in as-cast microstructures.

3.2 Microstructures after homogenization

The microstructures after homogenization are shown in Fig. 6. In general, no obvious change in eutectics was observed. In Fig. 5, it was indicated that the dissolution temperature of Mg_2Si and $AlFeMnSi$ was about 555 °C and 589 °C respectively. The temperature of homogenization in this study was set as 430 °C (for the purpose of promoting the dispersoids precipitation), which was actually considerably below the dissolution temperature of Mg_2Si or $AlFeMnSi$. In this case, it was not able to achieve the dissolution or transformation of Mg_2Si or $AlFeMnSi$. Therefore, the eutectics remained stable after homogenization. However, a large number of fine dispersoids emerged in the Al matrix (Fig. 6a and b). The precipitation of fine dispersoids occurred mainly inside the dendrite cells, whereas nearly no precipitates formed near the eutectic regions. The field of view could be divided into dispersoid zones and dispersoid-free zones (PFZs) as indicated by yellow dotted lines. The PFZs in the MC sample were obviously larger and more recognizable than those in the TRC sample. A higher-magnification SEM images in Fig. 6c and d shows the dispersoid zone of both samples, in which the number density of fine dispersoids in the TRC sample was overall higher than that in the MC sample. Transmission electron microscopy (TEM) observation revealed that the dispersoids mostly had short plate- and rod-like shapes (Fig. 6e and f), which could be attributed to the projection of the short plate-like morphology. EDS analysis was then conduct on these dispersoids, and the EDS result of the particle indicated in Fig. 6e (red arrow in Fig. 6e) is shown in Fig. 6g. The EDS results (Fig. 6g, test position indicated by the arrow in Fig. 6e) revealed that these particles were Fe, Mn, and Si bearing. In our previous studies [7], these dispersoids have been thoroughly investigated, hereby it is confirmed based on the morphology and EDS results that the dispersoids were to $\alpha-Al(Fe, Mn)Si$ dispersoids, which was also reported in the references [4, 21].

The dispersoids and the PFZ were quantified based on a series of SEM images, and the results are shown in Fig. 7. For dispersoids (Fig. 7a), the equivalent diameters of in the TRC and MC samples were 42 and 73 nm, respectively, the number density increased from $11.6 \mu\text{m}^{-2}$ in the MC sample to $14.7 \mu\text{m}^{-2}$ in the TRC sample. Regarding the PFZ (Fig. 7b), the fraction was 10.6% and the equivalent width was $3.4 \mu\text{m}$ in the MC sample, whereas in the TRC sample, the fraction was 4.6% and the equivalent width was $1.3 \mu\text{m}$.

The precipitation of dispersoids during homogenization could be attributed to the concentration of dispersoid formers and the existence of heterogeneous nucleation sites. As previously described in section 3.1, TRC had a higher cooling during solidification and hence created a higher degree of supersaturated solid solution relative to. During homogenization, the TRC sample was able to provide a stronger driving force for dispersoid precipitation and more sufficient alloying solutes for the decomposition of the solid solution. To qualitatively follow the solid solution concentrations and their changes, electrical conductivities were measured, and the results are shown in Fig.8. The change of electrical conductivity between the as-cast and homogenized sample could give an index of the solute level that precipitated during homogenization. After homogenization, the both electrical conductivities of the TRC and MC samples increased owing to the decomposition of the supersaturated solid solution via dispersoid precipitation. However, the change in electrical conductivity in the TRC sample (17.5% IACS) was greater than that in the MC sample (16.3% IACS), confirming that a larger amount of alloying elements in the TRC sample were precipitated out of solid solution during homogenization, producing a greater number density of dispersoids (Fig.7a).

In the cast strip produced via TRC, dense of dislocations were generated at the deformation region of the cast strips [24]. Fig.9 shows the microstructure evolution during the homogenization. Fig. 9a shows the microstructure when the homogenization began (at 0 h of homogenization, representing the temperature just reached at 430°C with a 100°C/h heating rate), the inset selected area electron diffraction pattern was obtained from the Al matrix while the image was taken, for Fig. 9b, c and d, the images were taken from the same direction of Al matrix as Fig. 9a. In Fig. 9a, a number of $\alpha\text{-Al(Fe, Mn)Si}$ dispersoids were observed along with the dislocation lines that were generated during TRC. In addition, finer dispersoids (as indicated by dotted red rectangle) were also observed in the Al matrix. Fig. 9b shows the enlarged dispersoids in the dotted red rectangle of Fig. 9a, the size of these particles were about 5-25nm and were with the plates and the short rods morphology. In the microstructure after 1 h of homogenization (Fig. 9c), dispersoid growth was observed, wherein all the dispersoids became more recognizable size. As the homogenization time increased to 6 h (Fig.9d), the number density of dispersoids increased and the size of the dispersoids became more uniform, regardless of whether they were located along dislocations or in the dislocation-free zone. TEM observations revealed that the pre-existing dislocations acted as favorable heterogeneous nucleation sites and accelerated the precipitation of $\alpha\text{-Al(Fe,Mn)Si}$ dispersoids, which was another reason for the promoted precipitation of $\alpha\text{-Al(Fe,Mn)Si}$ dispersoids in the TRC sample.

PFZ were observed in the heat-treated microstructure. In general, the formation of PFZ was accessed with lack of dispersoids former [4, 25, 26, 27, 28]. Scheil calculations for the experimental alloy system was performed using the Thermo-Calc software with TCAl6 database. The element distributions in the Al

matrix are shown in Fig. 10. With the increasing solid fraction, Mg content increased until the end of solidification, the content of Si raised until a peak was reached at 90.1% of solid fraction, after which the content decreased. For Mn and Fe, the content decreased with the increasing solid fraction. For example, the content of Mn at 0.3 solid fraction was as high as 0.760%, then it decreased to 0.216% at 80% solid fraction, when the solidification fraction reached 90%, the content of Mn had decreased to as low as 0.045%. The content of Fe during solidification followed a similar tendency as that of Mn. This result indicated that Mn- and Fe-deficient regions formed when the solidification of Al dendrite was about to complete, that is, when the eutectic began to form. Accordingly, PFZ of α -Al(Fe, Mn)Si dispersoids formed in the Mn- and Fe-deficient regions due to the lack of dispersoids former during the subsequent homogenization. The cooling rate during solidification can effectively reduce the microsegregation of solute elements, thus reducing the fraction of Mn- and Fe-deficient regions near the eutectics. Therefore, the smaller fraction of PFZ in TRC samples than that in MC samples was generated.

3.2 Hot deformation

3.2.1 Flow stress

Uniaxial hot compression tests were performed with a strain rate of 1 s^{-1} . The deformation temperatures of MC sample were from 300 °C to 500°C, and the deformation temperatures of TRC samples were 300 and 400°C, and the stress-strain curves are shown in Fig. 11. For both MC and TRC samples, the flow stress increased quickly at the beginning of the compression until reaching a yield point. After that, the flow stress increased slowly but continually until the end of deformation. The flow stress levels of both MC and TRC samples decreased with the increasing hot deformation temperatures. In general, the flow stresses of TRC sample were higher than those of MC sample at any given test condition. For instance, TRC sample possessed 9.2 and 7.4 MPa higher flow stress at 0.2 strain under 300 and 400°C deformation temperatures, respectively.

Hot deformation stress is dependent on many factors, such as the deformation regime, alloying element types and contents, and heat treatment history. For dispersoid-containing alloys, the hot deformation stress is mainly affected by the size, number density and distribution of the dispersoids. During the hot deformation, dislocations were generated and were retarded at the points where they encountered the dispersoids, and thus the dispersoids acted as stronger barriers to deformation. In this manner, the pinning effect of the dispersoids on dislocation movement yielded significant increases in flow stress and resulted in the increasing of flow stress during hot compression [6, 8, 9]. In a study conducted by one of the authors of this study [6], the hot deformation flow stresses of 6082 alloy were determined by the number density of α -Al(Fe, Mn)Si dispersoids. The flow stress increased from 55.5 MPa for the dispersoid-free alloy to 85.7 MPa for the dispersoid-strengthened alloy when hot deformation was performed at 400 °C/ 1 s^{-1} owing to the dispersoid strengthening effect. The α -Al(Fe, Mn)Si dispersion coarsened after 500°C/2h heat treatment, resulting in a significant decrease in the strength of the alloy.

In the present study, the higher hot deformation stress level of the TRC samples compared to the MC samples could be explained by the high number density of dispersoids and low fraction of PFZ in the experimental samples. The dispersoid strengthening effect was strongly related to the space between particles among other factors. During hot deformation, when moving dislocations encountered the dispersoids, they were forced by the applied stress to bow around and bypass the dispersoids, leaving a dislocation loop around the particle, which is called an Orowan loop.

In the TRC sample after homogenization, the dispersoids had a higher number density, which corresponds to a shorter distance between dispersoids, explaining the higher hot deformation stress relative to MC samples, resulting from the enhanced interaction between dispersoids and mobile dislocations.

3.2.2 Edge crack susceptibility during hot deformation

As shown in 3.2.1, the hot workability of MC samples was moderately lower than that of TRC sample owing to higher flow stress. However, the edge crack susceptibility is another important factor influencing hot workability, particularly for rolling process of TRC strip. After deformation, the cylindrical specimens were compressed into short “cake” shapes, Fig. 12a and b shows sample after deformation under 300 °C and 0.5 strain. A number of edge cracks (visible by naked eyes) emerged on the side surface of the deformed MC sample (Fig. 12a). The cracks were several micrometers in length and approximately 0.5-1 micrometer in width. However, no any edge crack was observed in the deformed TRC sample (Fig. 12b), for all the deformation conditions (at 300 and 400 °C up to the maximum 0.5 deformation strain applied in the experiment). Fig. 13 shows the edge cracks of MC sample, in which numerous edge cracks could be observed at the 450 °C and 0.5 strain (Fig. 13a); with decreasing deformation temperature to 350 °C, more and larger edge cracks appeared (Fig. 13b). Fig. 13 (c) shows the microstructure of the sample deformed at 350 °C with strain of 0.5 near the side surface. A number micro cracks (indicated by the arrows) developed within the coarse AlFeMnSi intermetallic particles and propagated along intermetallic particles into the aluminum matrix, indicating that the intermetallic particles were the source of the initiation and propagation of micro cracks from which the macro edge cracks built and extended to the side surface.

Table 1 shows the edge crack susceptibility as a function of deformation conditions the MC samples. In general, edge crack occurred at the low deformation temperature and large strain. For instance, no crack was generated at 350 °C and 0.3 strain, but the cracks appeared when the strain increased to 0.4 and further to 0.5. With the increasing deformation temperature up to 450 °C, the cracks could only observed at the 0.5 strain.

Table.1 The edge crack susceptibility as a function of deformation temperature and strain in MC samples. “Yes” represents the occurrence of edge cracks and “No” indicates no crack

T	300°C	350°C	400°C	450°C	500°C
Strain					
0.3	No	No			
0.4	Yes	Yes	No	No	
0.5	Yes	Yes	Yes	Yes	No

Fig. 14 shows the EBSD images and Kernel average misorientation (KAM) map of deformed MC sample. In Fig. 14a, a large amount of substructures represented by the white lines were observed, indicating the high densities of dislocation cells and subgrains generated during deformation. In addition, non-uniform distribution of substructures was observed, as indicated by the yellow ellipse; dense substructures were located near the grain boundaries. In the KAM map (Fig. 14b), high strain accumulation regions were also recognizable at the near the grain boundaries (as the yellow ellipse indicated), which was at the same position of the dense substructures in Fig. 14a.

The regions of dense substructures and high stress accumulations were actually where the most coarse AlFeMnSi intermetallic particles located. In the duration of plastic deformation, the brittle and coarse intermetallics were not able to adjust the deforming matrix by a corresponding deformation, thereby forming a high stress concentration in the microregion of intermetallics. With the progressive deformation, when the local deformation near the intermetallics exceeds the endurance limit of the aluminum matrix, microcracks form. The coarser and greater number of intermetallics, the easier microcracks form. In the TRC samples, no cracks occurred even at low deformation temperature and high strain owing to the low fraction and fine intermetallics in the as-cast microstructures (Fig. 3). During hot deformation, the accumulation of local stress caused by incompatible deformation of the aluminum matrix was kept at a comparably low level, and the risk of the crack initiation and propagation could be almost eliminated.

The coarse intermetallics in the microstructures of MC samples was the large limiting factor of the hot workability, owing to their high edge crack susceptibility during rolling. Traditionally, a high-temperature homogenization ($\sim 580^{\circ}\text{C}$) was necessary to make the coarse intermetallics partially dissolved or fragmented, to improve the hot workability [6, 15]. However, for the experimental dispersoids-strengthened alloys, the homogenization temperature was set as 430°C , to enhance the dispersoid precipitation; it was not appropriate to conduct a heat treatment over 500°C to avoid the coarsening of dispersoids [7]. Such a low homogenization temperature was not able to achieve the goal of the dissolution or the fragmentation of eutectics. Consequently, the intermetallics remained unchanged in the matrix and negatively influenced the hot workability of MC samples. However, for TRC samples, the intermetallics in the as-cast condition were already refined and uniformly distributed, which brought a significant improvement of hot workability during the subsequent hot deformation process.

4. Conclusions

An Al-Mg-Si-Mn aluminum alloy was produced by twin-roll casting (TRC) and conventional mold casting (MC). Homogenization was performed at a low temperature of 450 °C for 6 h to promote the precipitation of α -Al(Fe, Mn)Si dispersoids, which was followed by uniaxial hot compression tests to verify the hot workability of the alloy. The following conclusions were drawn:

- (1) Compared to the as-cast MC samples, the as-cast TRC samples had a lower eutectic fraction with a smaller size and a higher solid solution concentration.
- (2) During homogenization, a large number of α -Al(Fe, Mn)Si dispersoids precipitated in the aluminum matrix, and the dispersoids in the TRC sample had a greater number density than those in the MC sample. The result was attributed to the high cooling rate and rolling force of TRC process, providing the higher supersaturated solid solution and pre-existing dislocations in the as-cast microstructures of the TRC sample.
- (3) Precipitation-free zone (PFZ) formed near the eutectic regions. The formation of PFZ was related to the Mn- and Fe-deficient regions near the eutectic formed during the solidification.
- (4) The TRC samples yielded higher flow stresses at all hot deformation conditions applied owing to the strong dispersoid strengthening relative to MC samples. During hot deformation, the severe edge cracking occurred in the MC samples at the low deformation temperature and large strain due to the high fraction of coarse AlFeMnSi intermetallic particles. In contrast, no cracks formed in the TRC samples owing to its lower fraction as well as fine and uniformly distributed intermetallics, which greatly improved the hot workability for the subsequent rolling process.

Declarations

Acknowledgements

The authors gratefully acknowledge the support of the National Natural Science Foundation of China (Grant No. 51790485), the Fundamental Research Funds for the Central Universities (No. N2007008), Nanning science and technology major special projects (No. 20191002) and the Key research and development project of Shandong province (the first batch of competitive selection of major scientific and technological innovation projects, No. 2019JZZY010401). The authors would like to also acknowledge the financial support from the Natural Sciences and Engineering Research Council of Canada (NSERC) and Rio Tinto Aluminum, through the NSERC Industry Research Chair in Metallurgy of Aluminum Transformation at the University of Quebec at Chicoutimi.

References

- [1] Knipling K E, Dunand D C, Seidman D N. Nucleation and Precipitation Strengthening in Dilute Al-Ti and Al-Zr Alloys [J]. Metallurgical and Materials Transactions A, 2007, 38: 2552-2563.

- [2] Wen S P, Gao K Y, Li Y, Huang H, Nie Z R. Synergetic effect of Er and Zr on the precipitation hardening of Al-Er-Zr alloy [J]. Scripta Materialia, 2011, 65: 592-595.
- [3] Foley J C, Perepezko J H, Skinner D J. Formation of metastable $\text{Li}_2\text{-Al}_3\text{Y}$ through rapid solidification processing [J]. Materials Science and Engineering: A, 1994, 179-180: 205-209.
- [4] Lodgaard L, Ryum N. Precipitation of dispersoids containing Mn and/or Cr in Al-Mg-Si alloys [J]. Materials Science & Engineering A, 2000, 283: 144-152.
- [5] Mulazimoglu M H, Zaluska A, Gruzleski J E, Paray F. Electron microscope study of Al-Fe-Si eutectics in 6201 aluminum alloy [J]. Metallurgical & Materials Transactions A, 1996, 27: 929-936.
- [6] Qian X M, Parson N, Chen X G. Effect of Homogenization Treatment and Microalloying with Mn on the Microstructure and Hot Workability of AA6060 Aluminum Alloys [J]. Journal of Materials Engineering and Performance, 2019, 28: 4531-4542.
- [7] Qian X M, Parson N, Chen X G. Effects of Mn addition and related Mn-containing dispersoids on the hot deformation behavior of 6082 aluminum alloys [J]. Materials Science and Engineering: A, 2019, 764: 138-253.
- [8] Shakiba M, Parson N, Chen X G. Effect of homogenization treatment and silicon content on the microstructure and hot workability of dilute Al-Fe-Si alloys [J]. Materials Science and Engineering: A, 2014, 619: 180-189.
- [9] Liu K, Chen X G. Evolution of Eutectics, Dispersoids, and Elevated Temperature Properties at Various Fe Contents in Al-Mn-Mg 3004 Alloys [J]. Metallurgical and Materials Transactions B, 2015, 47: 3291-3300.
- [10] Qian X M, Li X L, Li Y, Xu G M, Wang Z D. Microstructure evolution during homogenization and hot workability of 7055 aluminum alloy produced by twin-roll casting [J]. Journal of Materials Research and Technology, 2021, 13: 2536-2550.
- [11] LI S J, WEI B W, YU C, LI Y, XU G M, LI Y. Evolution of microstructure and properties during homogenization of the novel Al-Li alloy fabricated by electromagnetic oscillation twin-roll casting [J]. Journal of Materials Research and Technology, 2020, 9: 3304-3317.
- [12] Barekar N S, Dhindaw B K. Twin-roll casting of aluminum alloys—an overview [J]. Materials and manufacturing processes, 2014, 29: 651-661.
- [13] Wang H B, Zhou L, ZHANG Y W, CAI Y H, ZHANG J S. Effects of twin-roll casting process parameters on the microstructure and sheet metal forming behavior of 7050 aluminum alloy [J]. Journal of Materials Processing Technology, 2016, 233: 186-191.

- [14] QIAN X M, PARSON N, CHEN X G. Effect of post-homogenisation cooling rate and Mn addition on Mg_2Si precipitation and hot workability of AA6060 alloys [J]. Canadian Metallurgical Quarterly, 2020, 59: 189-200.
- [15] Kear B H, Giessen B C, cohen m. Rapidly solidified amorphous and crystalline alloys [C]. Elsevier Science Pub, 1982, 8: 5.
- [16] Wen S p, Xing Z b, Huang H, Li b l, wang w, nie z r. The effect of erbium on the microstructure and mechanical properties of Al-Mg-Mn-Zr alloy [J]. Materials Science and Engineering: A, 2009, 516(1-2): 42-49.
- [17] Knipling K E, Dunand D C, Seidman D N. Precipitation evolution in Al-Zr and Al-Zr-Ti alloys during isothermal aging at 375-425 °C [J]. Acta Materialia, 2008, 56: 114-127.
- [18] Li H y, Gao Z h, Yin H, jiang h f, su x j, bin j. Effects of Er and Zr additions on precipitation and recrystallization of pure aluminum [J]. Scripta Materialia, 2013, 68: 59-62.
- [19] Li Y J, Zhang W Z, Marthinsen K. Precipitation crystallography of plate-shaped $Al_6(Mn,Fe)$ dispersoids in AA5182 alloy [J]. Acta Materialia, 2012, 60: 5963-5974.
- [20] Jiang J Y, Jiang F, Zhang M H, Tang Z Q, Tong M M. $Al_3(Sc, Zr)$ precipitation in deformed Al-Mg-Mn-Sc-Zr alloy: Effect of annealing temperature and dislocation density [J]. Journal of Alloys and Compounds, 2020, 831: 154856.
- [21] Hu R, Ogura T, Tezuka H, Stato T, Liu Q. Dispersoid Formation and Recrystallization Behavior in an Al-Mg-Si-Mn Alloy [J]. Journal of Materials Science & Technology, 2010, 26: 237-243.
- [22] Kumar S, O'Reilly K A Q. Influence of Al grain structure on Fe bearing eutectics during DC casting of an Al-Mg-Si alloy [J]. Materials Characterization, 2016, 120: 311-322.
- [23] Das S, Lim N S, KIM H W, Park C G. Effect of rolling speed on microstructure and age-hardening behaviour of Al-Mg-Si alloy produced by twin roll casting process [J]. Materials & Design, 2011, 32: 4603-4607.
- [24] Liu Z Y, HUANG T T, LIU W J, KANG S B. Dislocation mechanism for dynamic recrystallization in twin-roll casting Mg-5.51Zn-0.49Zr magnesium alloy during hot compression at different strain rates [J]. Transactions of Nonferrous Metals Society of China, 2016, 26: 378-389.
- [25] Starink M J. Reduced fracturing of intermetallic particles during crack propagation in age hardening Al-based alloys due to PFZs [J]. Materials Science and Engineering: A, 2005, 390: 260-264.
- [26] Tolley A, MiTlin D, RADMILOVIC V, DAHMEN U. Transmission electron microscopy analysis of grain boundary precipitate-free-zones (PFZs) in an AlCuSiGe alloy [J]. Materials Science and Engineering: A, 2005, 412: 204-213.

[27] Zindal A, JAIN J, PRASAD R, SINGH S S, SARVESHA R, CIZEK P, BARNETT M R. Effect of heat treatment variables on the formation of precipitate free zones (PFZs) in Mg-8Al-0.5Zn alloy [J]. Materials Characterization, 2018, 136: 175-182.

[28] Zindal A, JAIN J, PRASAD r, singh s s. Effect of pre-strain and grain size on the evolution of precipitate free zones (PFZs) in a Mg-8Al-0.5Zn alloy [J]. Materials Letters, 2017, 201: 207-210.

Figures

Figure 1

Schematic illustration of the TRC process

Figure 2

The manner of taking specimens for (a) microstructure observation and for compression, (b) shape of the specimen after deformation and (c) cross section of the deformed specimen, red rectangle indicates the EBSD analyzed area.

Figure 3

As-cast microstructures of the (a, c) MC and (b, d) TRC samples. (e, f) EDS results of eutectics in the MC sample

Figure 4

Statistical data on the eutectic area fraction and equivalent length of the as-cast samples

Figure 5

DSC curves and the endothermic peaks for the MC and TRC samples

Figure 6

Etched homogenized microstructures of MC sample (a and c) and TRC sample

(b and d); TEM images of MC sample (e) and TRC sample (f); (g) EDX analysis of dispersoid in (e)

Figure 7

(a) Number density and size statistics of the α -Al(Mn, Fe) Si dispersoids and (b) area fraction of the PFZ in the Al-Mg-Si-Mn alloys after homogenization

Figure 8

Electrical conductivity of as-cast and homogenized samples

Figure 9

TEM images of TRC sample after 0 h (a) and (b), 1h (b) and 6h (c) of homogenization, all the images were obtained when the Al matrix were under the direction as the selected area electron diffraction pattern inset in (a)

Figure 10

Scheil calculations of the solidification of the experimental alloy

Figure 11

Typical stress-strain curves of the MC and TRC samples

Figure 12

Appearance of specimens after deformed at 300 °C and 0.5 strain: (a) MC sample and (b) TRC sample

Figure 13

Macro cracks on the MC specimens deformed with 0.5 strain at (a) 450 °C and (b) 350 °C; (c) micro cracks in the specimen deformed with 0.5 strain at 350 °C

Figure 14

All Euler orientation EBSD map (a) and KAM map (b) of the MC sample after hot compression at 400 °C and 0.5 strain. The boundary misorientation angles in EBSD map are marked by white lines for 2-15° and black lines for >15°