

Formation and evolution of second phase particles in 1060 aluminum alloy sheet produced by twin-roll casting and annealing

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Abstract: Twin-roll casting (TRC) is a process capable of producing aluminum sheets at significantly reduced cost. High-quality aluminum sheets as critical raw materials of the battery aluminum foil have become increasingly important in ensuring both performance and reliability. This study investigated the second phases and their formation mechanism in a 1060 aluminum alloy sheet processed by TRC and followed by annealing treatment. The results show that the microstructure of the TRC aluminum alloy is composed of α -Al grains, Fe-containing intermetallic phases, and Al_3Si phase. After annealing treatment, the α -Al grains exhibit a sharply increase in size, while the second phase particles show a reduction in average size, with an increased number density and a more homogeneous distribution. The annealing process partially dissolves the second phases and causes the transformation of some Fe-containing intermetallic compounds (FIMCs), leading to the growth of α -Al grains and the decrease of low angle boundaries. Finally, the annealed samples exhibit a decrease in tensile strength and an increase in elongation compared with the TRC aluminum alloy. It is found that both coarsen FIMCs and their inhomogeneous distribution tend to cause localized strain-concentration regions, thereby, promoting crack initiation.

Keywords: twin-roll casting; 1060 aluminum alloys; microstructure; second-phase particles; annealing

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1 Introduction

Twin-roll casting (TRC) is a typical compact processing for production of thin strips directly from molten metal, and it has been widely applied due to its low cost and high efficiency^[1-3]. Nowadays, TRC processing is widely applied to manufacture aluminum sheets for foil, and the metallurgical quality of the TRC sheet generates great impact on the formability of aluminum sheets and the performance of final foil products^[4-6]. For example, coarsen second phases can lead to pinholes and strip breaks especially for ultra-thin battery

aluminum foils below 10 μm thickness^[7, 8].

Although extensive research has explored the quality control of TRC sheets through adjustments of process parameters, annealing treatments, and the application of external fields^[9, 10], the specific mechanisms governing the microstructure evolution during the production of TRC aluminum alloy sheets remain underexplored. Existing studies addressed grain refinement and microstructure homogenization via TRC parameter optimization and grain refiners^[11], focusing on the melt's temperature, concentration, and convection fields^[12-14]. Subsequent heat treatments have been shown to influence the microstructure of TRC sheets^[15-17], in which high-temperature annealing ($\geq 400^\circ\text{C}$) promoted slight refinement of intermetallic particles in Al-0.36Fe-0.4Si alloys, resulting from partial dissolution or decomposition of silicon-rich phases. Additionally, various external fields, such as electromagnetic^[18-20] and ultrasonic fields^[21-23], have been demonstrated to effectively modify melt flow and solute distribution,

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leading to microstructure refinement and homogenization within the twin-roll casting zone. In conclusion, although process conditions and solidification behavior are known to influence the formation of second phases and microstructure in TRC sheets, a deeper understanding of the mechanisms governing microstructure evolution during subsequent heat treatment remains a crucial research gap.

Commercially pure 1060 aluminum alloy, known for its excellent thermal conductivity, superior corrosion resistance, and good formability, is widely used in packaging, electronics, and construction^[23]. With an aluminum content exceeding 99.6%, the 1060 aluminum alloy only contains minimal amounts of iron and silicon. But the aluminum and iron elements in 1060 aluminum alloys can form iron containing intermetallic compounds (FIMCs) during solidification. These FIMCs disrupt the continuity of the aluminum matrix, reduce the plastic deformation capability, and adversely affect the cold rolling or deep processing performance^[24, 25]. Therefore, it is necessary to control these second phases at the beginning of the TRC process. Understanding the development of FIMCs during TRC can provide valuable insights for optimizing microstructure evolution and properties during subsequent heat treatment.

The TRC process creates a strong cooling, forcing the alloy into a non-equilibrium solidification state^[26, 27]. This rapid solidification usually leads to inevitable centerline segregation and the formation of non-equilibrium phases. It was reported that during the solidification process, the non-equilibrium θ - $\text{Al}_{13}\text{Fe}_4$ phase formed firstly, followed by a transformation to the β - Al_5FeSi phase driven by diffusion of elements such as silicon within the aluminum alloy^[28]. However, the formation of FIMCs is determined more by kinetics factors than thermodynamics, making the control of competitive phase selection among different FIMCs challenging in practical solidification processes^[29]. As a result, equilibrium phase diagrams become unreliable for accurately predicting the microstructure of TRC 1060 aluminum alloy.

Given the challenges of controlling FIMCs formation during TRC, this study utilized 1060 aluminum alloy to investigate the mechanism behind the formation and transformation of these second phases during both the TRC process and subsequent annealing treatment, and to explore the influence of the second phase on the grain structure and overall properties in 1060 aluminum TRC sheets.

2 Experimental materials and methods

For the current research work, commercially pure 1060 aluminum alloy with a minimum aluminum content of 99.6wt.% was chosen as the base material. The chemical compositions, as measured by ARL3460 DES spectrometer, are detailed in Table 1. Additionally, Al-5Ti-B master alloy was introduced as a grain refiner to promote nucleation during solidification.

The inclined twin-roll continuous casting machine was used to

Table 1: Chemical compositions of 1060 aluminum alloy (wt.%)

Si	Fe	Cu	Mn	Mg	Ti	Zn	Al	Others
0.023	0.223	0.048	0.002	0.004	0.022	0.006	99.641	0.031

produce TRC sheet, and the pouring direction of the molten metal was inclined at an angle of 15° with the ground horizontal line. The continuous casting machine consisted of two stainless steel rollers with a diameter of 1,000 mm and a width of 1,800 mm. Cooling water entered the roller core through two inlet holes for circulation, and was then discharged through two return water holes. During the TRC process, the 1060 aluminum ingot was melted in a furnace, degassed and filtered, and the melt temperature was kept at 685°C . Then, the 1060 aluminum alloy melt was cast into sheets using TRC. The resulting TRC sheet had a thickness of 6.6 mm and a width of 1,650 mm. During the TRC process, the temperature on the surface of the roller was controlled at $65\pm 10^\circ\text{C}$, the rolling speed was $960\text{ mm}\cdot\text{min}^{-1}$. In order to compare the effects of cooling rate on microstructure of 1060 aluminum alloy, ingot casting was also performed in this study, and cylindrical ingots with a diameter of 40 mm and a height of 28 mm were cast using the same melt for TRC.

A $10\text{ mm}\times 10\text{ mm}\times 10\text{ mm}$ cubic sample was taken from the center of the ingot for microstructure analysis. The TRC sheet was divided into two portions. One portion was cut into samples with sizes of $10\text{ mm}\times 8\text{ mm}\times 6.6\text{ mm}$ for microstructural and thermal property studies. The other portion was subjected to an annealing treatment: heated to 550°C over 2 h, held at 550°C for another 2 h, and then furnace cooled to room temperature. Finally, the second phase and grain morphology in both the TRC sheet and the annealed sheet were observed on the three surfaces aligned with the transversal direction (TD), normal direction (ND), and rolling direction (RD). Tensile specimens with sizes in Fig. 1 were prepared before and after annealing for comparative analysis.

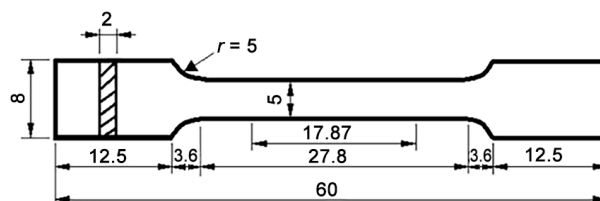


Fig. 1: Dimensions of tensile specimen (unit: mm)

Samples for microstructure analysis were subjected to mechanical grinding, mechanical polishing, electrolytic polishing, and anodizing treatment in sequence. Among them, the metallographic examination samples were prepared using anodic coating technology. The anodic polishing electrolyte consisted of a 1:49 (volume ratio) mixture of fluoroboric acid (HBF_4) and deionized water. Electropolishing was performed at a constant voltage of 20 V for a duration of 150 s. Samples for electron backscatter diffraction (EBSD) analysis were prepared via electropolishing using a solution of perchloric acid (HClO_4) and anhydrous ethanol in a 1:9 volume ratio. Electropolishing was performed at 20 V for 10 s. An optical

microscope (Zeiss Axiovert 40 MAT) was used to observe the microstructure. Electron backscatter (EBSD signal source) images and secondary electron images were obtained using a field emission scanning electron microscope (JEOL JSM7800F-2), and the alloy phase constitution was identified using a multifunctional high-resolution X-ray diffractometer (XRD, Empyrean). Grain orientation analysis was performed using the EBSD technique with a scanning step size of 0.3–0.7 μm . The thermodynamic properties of the alloy were tested using a differential scanning calorimeter (DSC, Netzsch 404 F3 DSC) with a heating/cooling rate of 5 $^{\circ}\text{C}\cdot\text{min}^{-1}$. The room temperature tensile properties were tested using a CMT5105 testing machine at a tensile rate of 2 $\text{mm}\cdot\text{min}^{-1}$. In addition, transmission electron microscopy (TEM, JEM2100F) was used to observe the bright field image morphology of TRC aluminum sheet and perform energy spectrum analysis.

3 Results

Figure 2 shows the grain morphology of the TRC 1060 sheet specimen at different positions through the thickness, as observed in the TD, ND, and RD directions. Elongated grains along deformation direction are found in the TRC sheet, and samples along the TD exhibit a much less deformation degree comparing with those samples along the RD. The top and bottom surface layers along the RD exhibit fine, elongated fibrous grains with indistinct boundaries [Figs. 2(a) and (c)], whereas the central region is characterized by banded grain structures. As shown in Figs. 2(b) and (e), the mean intercept length of $\alpha\text{-Al}$ grains is measured to be 174 μm in RD and 21 μm in ND, with a length-to-diameter ratio of 8.3:1.

Figure 3 shows the EBSD orientation map and corresponding pole diagram along the RD for the TRC 1060 aluminum alloy sheet in both TRC and annealed conditions. The solid black line in these two figures represents high angle grain boundaries (HAGB), while the thin-white line indicates low angle grain boundaries (LAGB). The EBSD orientation mapping of the TRC sheet shows a predominantly blue contrast, indicating the presence of a strong $\{111\}$ texture with the corresponding crystallographic planes preferentially aligned parallel to the sheet normal direction. The TRC sheet exhibits a high fraction of LAGBs at approximately 59.0%, indicating a significant presence of subgrains. Comparatively, the EBSD orientation map of the annealed TRC sheet exhibits a predominantly red contrast, suggesting a dominant $\{100\}$ texture with the corresponding crystallographic planes preferentially aligned parallel to the sheet normal direction. After annealing, the fraction of LAGBs in the TRC sheet becomes negligible, suggesting the complete consumption of subgrains and the transformation into fully recrystallized grains. The pole diagram shown in Fig. 3(c) exhibits a $\{111\}\langle 110\rangle$ texture, in which the $\langle 110\rangle$ crystal direction of the TRC sheet is parallel to the RD, and the $\{111\}$ crystal plane is perpendicular to the normal surface. After annealing, the texture transforms into a dominant $\{001\}\langle 100\rangle$ recrystallization texture, in which the $\langle 100\rangle$ crystal direction on the TRC sheet is parallel to the RD, and the $\{001\}$ crystal plane is normal to the sheet surface.

The XRD patterns for the 1060 aluminum alloy in the ingot, TRC, and annealed conditions are shown in Fig. 4. It can be determined that the primary phases in the ingot samples and TRC samples are $\alpha\text{-Al}$, intermetallic phases of Al_9Si , $\text{Al}_{0.5}\text{Fe}_3\text{Si}_{0.5}$, $\text{Al}_{0.7}\text{Fe}_3\text{Si}_{0.3}$, and $\text{Al}_{13}\text{Fe}_4$. The main phases of the

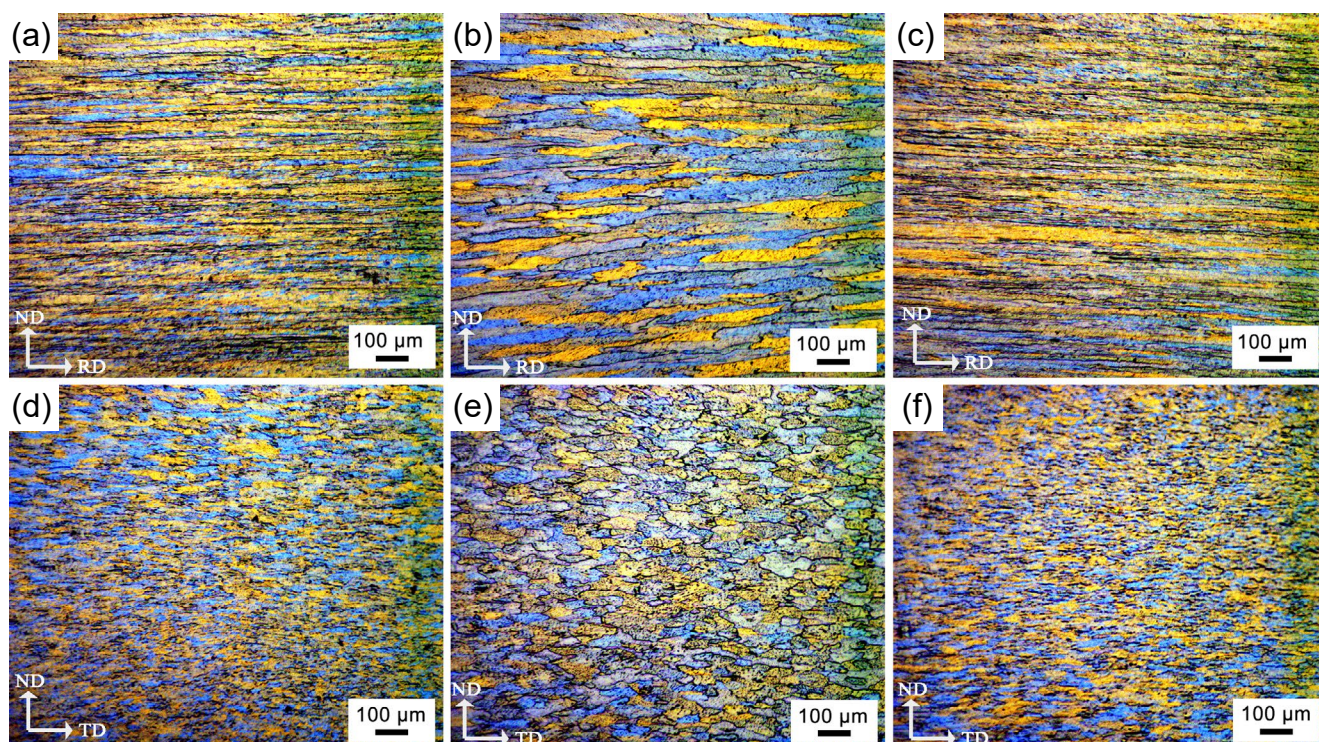


Fig. 2: Grain morphology of TRC 1060 sheet at different positions through the thickness observed in TD, ND, and RD directions: (a, d) top surface; (b, e) center zone; (c, f) bottom surface

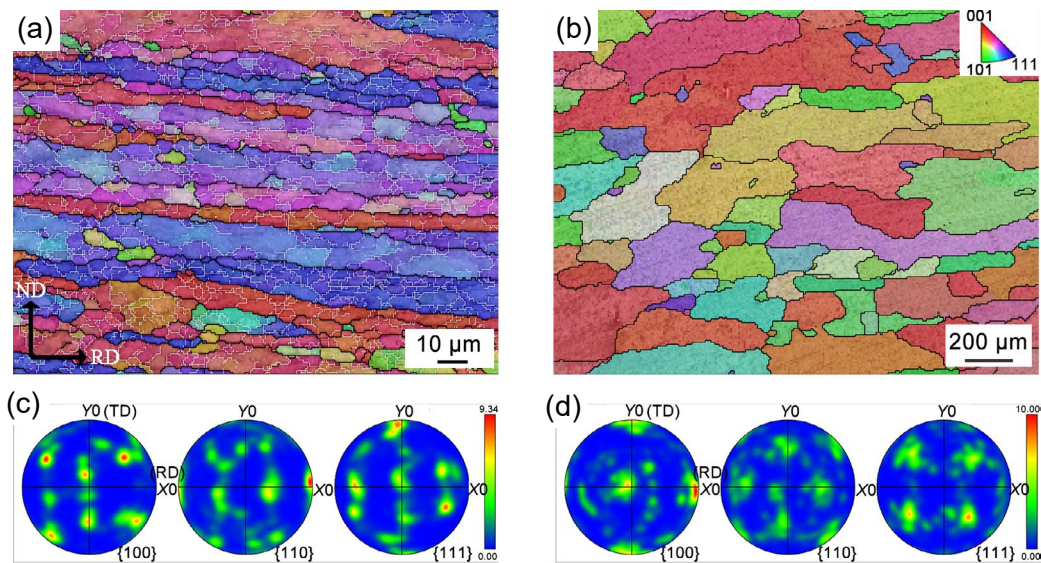


Fig. 3: EBSD orientation map and pole diagram of TRC (a, c) and annealed (b, d) sheets

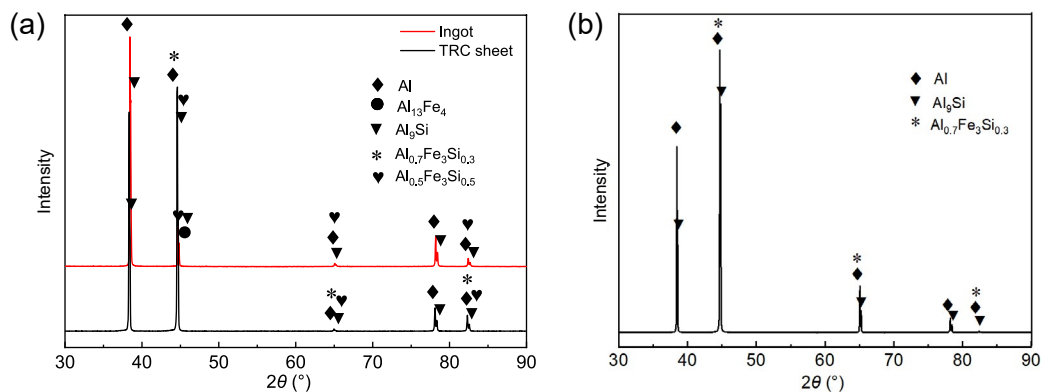


Fig. 4: XRD patterns of 1060 aluminum alloy from ingot and TRC sheet (a) and annealed sheet (b)

annealed sample include α -Al, intermetallic phases of Al_9Si , and $\text{Al}_{0.7}\text{Fe}_3\text{Si}_{0.3}$. XRD analysis reveals that the FIMCs in the as-cast ingot samples correspond to the $\text{Al}_{0.5}\text{Fe}_3\text{Si}_{0.5}$ and $\text{Al}_{0.7}\text{Fe}_3\text{Si}_{0.3}$ phases. The XRD patterns reveal a significant decrease in the intensity of $\text{Al}_{13}\text{Fe}_4$ phase peaks in the annealed samples compared to the TRC samples, suggesting the near-elimination of this second phase during annealing. This observation emphasizes the influence of TRC process and subsequent heat treatment on the evolution of intermetallic phases in 1060 aluminum alloy, highlighting the importance of controlling these processes to optimize microstructure and properties. Interestingly, the presence of the Al_9Si phase is observed in all three samples. Although Al_9Si phase is rarely reported in conventionally processed 1060 aluminum alloy, it has been theoretically and experimentally confirmed in 3D printed aluminum alloys with rapid solidification rates^[30].

Figure 5 illustrates the secondary electron images of the second phases observed in the ingot, TRC sheet, and annealed sheet. The elemental compositions of these second phases are determined using EDS, and the results are presented in Table 2. Secondary electron images in Figs. 5(a) to (d) reveal a similar distribution of second phases in both the ingot and TRC sheet. In both conditions, the primarily intermetallic phases include Al-Si phases (Point H), Al-Fe phases (Points A and B),

and Al-Fe-Si phases (Points C, D, and E) dispersed within the aluminum matrix. However, the annealed sheets observed in Fig. 5 primarily consist of the aluminum matrix and Al-Fe-Si phases (Points I, J, and K). Additionally, the Al_9Si phase is observed in the XRD results in Fig. 4(b), but is not detected in the SEM analysis. The size of the FIMCs in the annealed sheet is less than 1 μm . These second phases primarily consist of Al-Fe phases with minor Si, Al-Fe-Si phases rich in Si, and Al-Si phase that appears well-distributed within the aluminum matrix with minimal visible grain. The XRD patterns in Fig. 4 reveal the presence of the Al_9Si phase, which belongs to the displacement type solid solution^[31]. Some studies have reported that the Al_9Si phase can be decorated with the continuously distributed Si particles, even coexisting with Si phase^[32]. In addition, the TRC sheets contain a small amount of undissolved Ti element, visible in Fig. 5 at Points F and G. These second phases, including the Ti-rich inclusions, are all relatively small, typically on the micrometer scale.

In order to further observe the morphology of the second phases in TRC sheets, TEM bright field images were acquired, as shown in Fig. 6. The energy spectrum analysis shown in Table 3 reveals the presence of two second phases. The second phase of Al-Fe-Si (Point L, about 1 μm in diameter, rich in Si) is composed of discontinuous clusters, and these Al-Fe-Si

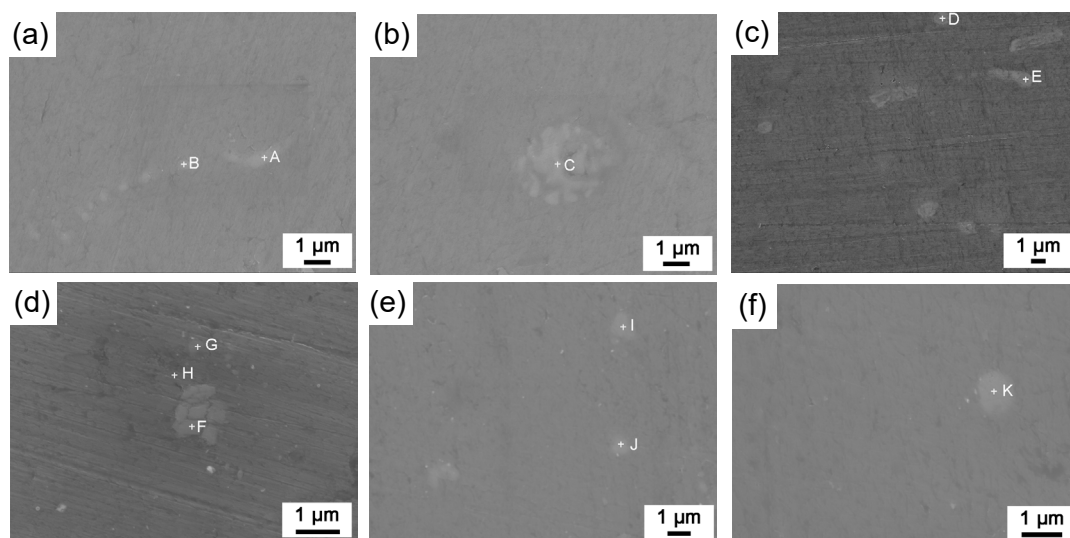


Fig. 5: Second phases in ingot (a, b), TRC sheet (c, d), and annealed sheet (e, f)

Table 2: EDS spectra of second phases in Fig. 5

Point	Mole fraction (%)						Predicted phases
	Al	Si	Fe	Ti	Cu	O	
A	94.5	0	5.5	0	0	0	Al-Fe
B	95.6	0	4.4	0	0	0	Al-Fe
C	85.9	3.0	11.1	0	0	0	Al-Fe-Si
D	92.6	1.6	5.6	0	0.2	0	Al-Fe-Si
E	93.0	1.2	5.5	0	0.2	0	Al-Fe-Si
F	90.6	0.2	0.1	8.5	0	0.7	Al-Ti
G	94.0	0	0.1	3.7	0	2.1	Al-Ti
H	89.6	8.0	0	0.1	0	2.2	Al-Si
I	92.8	1.9	5.3	0	0	0	Al-Fe-Si
J	90.5	2.2	6.9	0	0.4	0	Al-Fe-Si
K	90.5	2.3	6.9	0	0.3	0	Al-Fe-Si

second phases may be $\text{Al}_{0.7}\text{Fe}_3\text{Si}_{0.3}$, $\text{Al}_{0.5}\text{Fe}_3\text{Si}_{0.5}$ according to the XRD results in Fig. 4. Several Al-Fe particles (Point M, less than 1 μm in size) containing trace Si typically forms discontinuous line, and these Al-Fe particles are identified as $\text{Al}_{13}\text{Fe}_4$ through SEM-EDS analysis results in Table 3 and further confirmed by indexing the corresponding XRD peaks in Fig. 4(a). The combined results suggest the coexistence of multiple FIMCs in the alloy.

Backscattered electron images (Fig. 7) were employed to examine the distribution of second phases in TRC and annealed sheets. The phases rich in Si are identified as Al-Fe-Si, as confirmed by EDS analysis in Fig. 6, while the phase with possible minor Si content is predominantly Al-Fe. Notably, the phase with minor Si is predominantly located at grain boundaries, whereas the phase rich in Si resides within grains

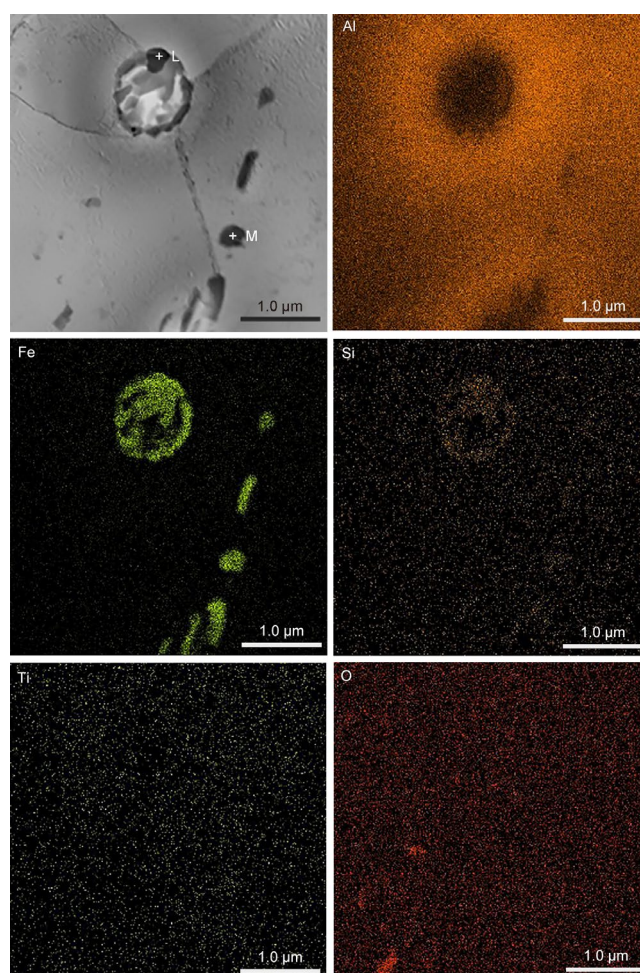


Fig. 6: TEM bright field image and element distribution maps of TRC sheet sample

Table 3: EDS results of second phases indicated in Fig. 6

Point	Mole fraction (%)						Predicted phases
	Al	Si	Fe	Ti	O	C	
L	68.37	2.30	6.18	–	6.34	16.81	$\text{Al}_{0.7}\text{Fe}_3\text{Si}_{0.3}$, $\text{Al}_{0.5}\text{Fe}_3\text{Si}_{0.5}$
M	74.20	0.89	5.19	–	5.73	14.00	$\text{Al}_{13}\text{Fe}_4$

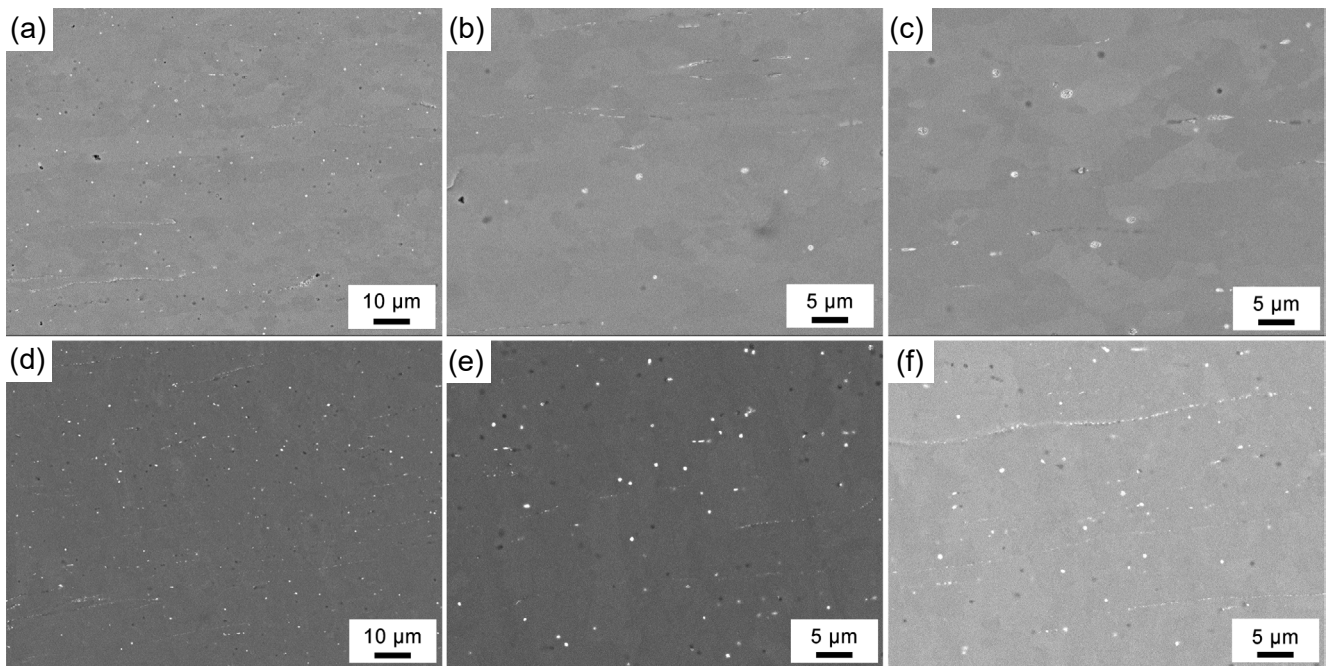


Fig. 7: Backscattered electron images showing second phase distribution of TRC (a, b, c) and annealed (d, e, f) samples

and at triple junctions. Annealed samples exhibit a reduction in size and quantity of the phase with minor Si compared to TRC sheets. Conversely, the phase rich in Si in annealed samples exhibits a decrease in size but an increase in quantity.

The DSC curves for the 1060 aluminum alloy TRC sheets are presented in Fig. 8. Three endothermic peaks can be observed in the heating curve at 650.0 °C, 656.1 °C, and 669.0 °C. Conversely, the cooling curve exhibits only two exothermic peaks at 646.0 °C and 641.2 °C. It is worth mentioning that the absence of a corresponding exothermic peak in the cooling curve for the first endothermic peak at 650.0 °C, which suggests that Al_3Si phase is formed due to the rapid cooling rate in the TRC process. Combined with the XRD results, the solidification of the 1060 alloy initiates with the primary precipitation of $\alpha-Al$ phase from the melt, followed by the formation of FIMCs along grain boundaries. Finally, the remaining liquid solidifies into the metastable Al_3Si phase due to the rapid solidification rate.

Tensile tests were performed on TRC sheet and annealed sheet to acquire engineering stress-strain curves (Fig. 9). The TRC sheet exhibits a tensile strength of 123.0 MPa and an

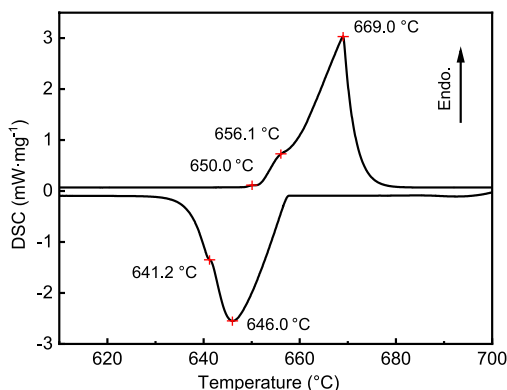


Fig. 8: DSC curves of TRC sheet

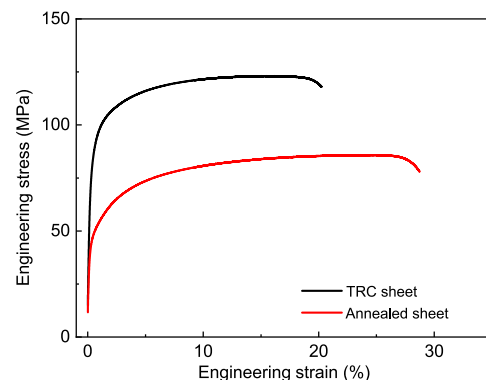


Fig. 9: Tensile curves of TRC sheet and annealed sheet

elongation of less than 20.5%. In contrast, the annealed sheet displays a lower tensile strength of approximately 85.8 MPa but a significantly improved elongation of 28.13%. Compared to the unannealed state, annealing treatment results in a decrease in tensile strength of roughly 30.24% and an increase in elongation of approximately 40.30%.

The fractographies of tensile samples from TRC sheet and annealed sheet are shown in Fig. 10. Both samples underwent substantial plastic deformation and necking under tensile loading. A large number of smaller dimples alongside minor river patterns are found on the fracture surface of the TRC tensile specimens, indicating a mixed-mode fracture. Dimple formation in this context likely originates from the presence of second-phase particles, which act as initiation sites for microvoids during tensile deformation. River patterns may be associated with intergranular fracture along grain boundaries containing inclusions or weak second-phase particles. In contrast, the annealed sheet displays a reduction in dimple density on the fracture surface, which suggests that the fracture mechanism of annealed samples can be attributed to weak second-phase along grain boundaries.

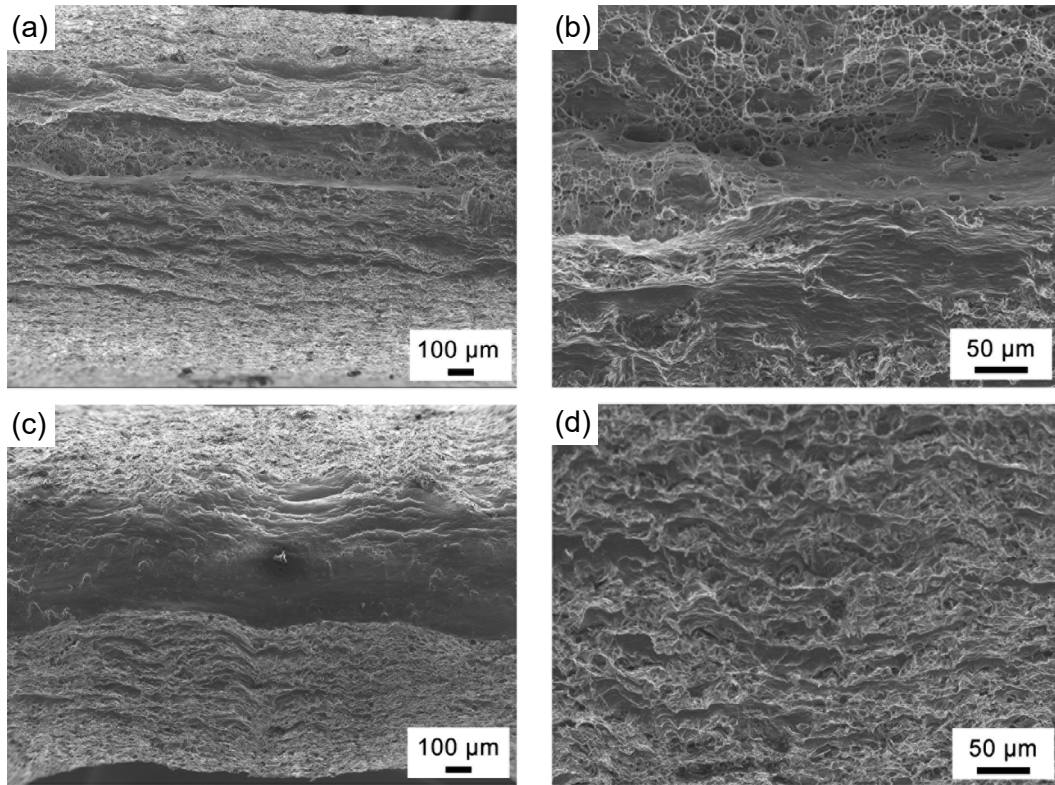


Fig. 10: Tensile fracture morphology of TRC sheet (a, b) and annealed sheet (c, d)

4 Discussion

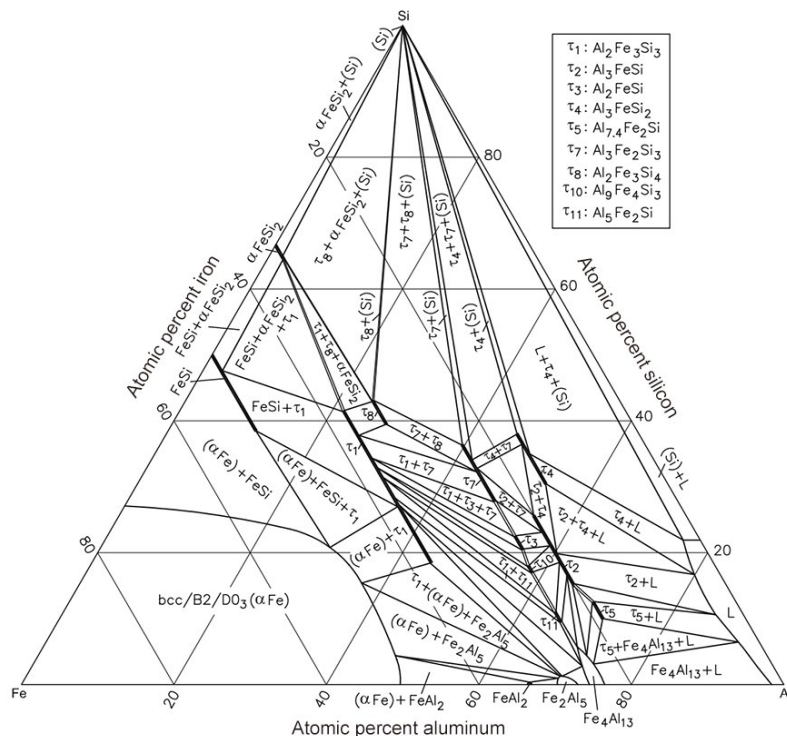
4.1 Microstructure of TRC 1060 alloy

The microstructure of the TRC 1060 alloy reveals elongated grains along RD (shown in Fig. 2), and the surface layer grains of the TRC sheet exhibit a high length-to-diameter ratio due to increased strain. Research suggests that in grain refined TRC alloys, solidification typically begins with the nucleation and growth of equiaxed grains. The development of elongated grains observed in the TRC is the result of deformation occurring after solidification^[33]. FIMCs and Al_9Si phase subsequently form followed by the solidification of $\alpha\text{-Al}$ grains, and these second phases mainly distribute along the grain boundaries. Al_9Si phase is determined as metastable phase controlled by the diffusion of Si element, and increased cooling rate can promote its formation.

Al-Fe-Si alloys contain a wide variety of complex FIMCs, as has been extensively reported in the literature^[29]. As shown in Table 1, the primary secondary-phase-forming elements in the 1060 aluminum alloy, after Al, are Fe, Cu, and Si, in order of decreasing concentration. Within the second phases, Cu exists as a solid solution in aluminum, whereas Fe and Si can form intermetallic compounds with aluminum. Based on Figs. 4 and 5, it can be known that the 1060 TRC aluminum alloy typically contains Al-Si phases, Al-Fe phases, and Al-Fe-Si phases. Due to the near-rapid cooling rates characteristic of the TRC process, kinetic effects tend to dominate over thermodynamic considerations, leading to the formation of non-equilibrium secondary phases. The solidification path of alloys can tend towards Scheil conditions during the TRC process^[9, 14]. Under this

law, the solutes in the remaining liquid become more concentrated in the final stages of solidification compared to the equilibrium condition, resulting in segregation and the formation of a second phase. Based on the equilibrium phase diagram of the Al-Fe-Si system shown in Fig. 11, there are many types of Fe-containing phases in aluminum, including $\text{Al}_{13}\text{Fe}_4$ and $\tau_5\text{-Al}_{7.4}\text{SiFe}_2$. The formation of FIMCs can be attributed to the solute concentration behavior at the solid-liquid front, which is controlled by the cooling rate and solidification sequence of the phase. TRC has been recognized as a near rapid solidification process, representing an intermediate regime between near-equilibrium solidification and rapid solidification^[34], and its cooling rate can reach $10^3 \text{ K}\cdot\text{s}^{-1}$. Under this solidification condition, the concentration of Fe at the solid-liquid front precisely satisfies the formation conditions of $\text{Al}_{13}\text{Fe}_4$ and Al-Fe-Si phases in the equilibrium phase diagram. In addition, it has been reported that due to competition between two different types of FIMCs during non-uniform nucleation, the precipitation of different types of FIMCs during solidification cannot be controlled^[29]. As a result, the 1060 alloy, despite its limited intentional alloying elements, can still form various intermetallic compounds such as Al_9Si , $\text{Al}_{13}\text{Fe}_4$, and Al-Fe-Si phases.

The solidification sequence of the second phases relative to the $\alpha\text{-Al}$ primary grains is crucial for understanding their interaction and impact on microstructure. As shown in Figs. 7(a-c), the second phases within the TRC sheet microstructure exhibit alignment along the rolling direction. Furthermore, the distribution of these second phases is characterized in detail in Fig. 12, and the energy spectrum analysis shown in Table 4 identifies the presence of FIMCs.

Fig. 11: Al-Fe-Si ternary phase diagram^[35]

Notably, these FIMCs tend to aggregate near the grain boundaries. It has been reported that these Fe-containing second phases can contribute to Zener pinning effect, hindering the migration of grain boundaries^[36]. The fine α -Al primary grain observed in Fig. 3(a) strongly suggests that the distribution of second phases within the TRC sheet hinders grain boundary migration through the Zener pinning effect.

As evident from the DSC curve of aluminum 1060 alloy in Fig. 8, the TRC process leads to the primary solidification of the α -Al phase. Due to Scheil-like solidification kinetics, the solute concentration ahead of the solid-liquid interface increases, and this enrichment promotes the formation of FIMCs as the remaining liquid solidifies. Diffusion of iron elements leads to the formation of binary iron-containing phases, while silicon diffusion triggers a phase transformation to form ternary iron-containing phases. Finally, any residual silicon atoms remaining in the liquid solidify into the Al_9Si phase.

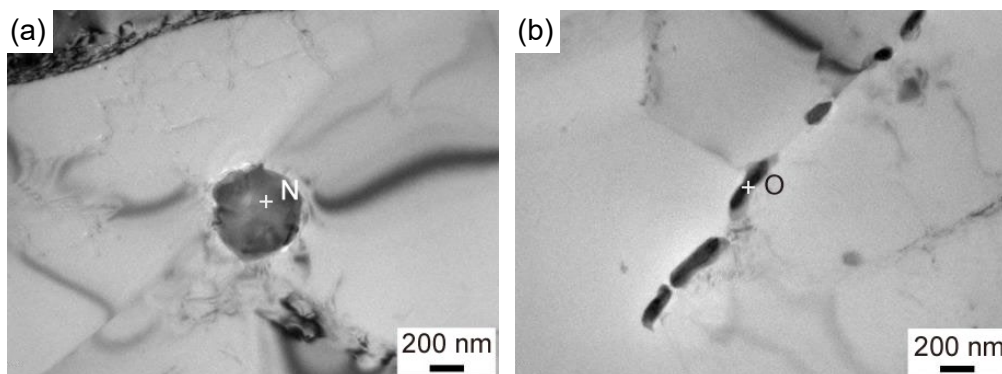
Fig. 12: Second phase distribution at grain boundaries before annealing: (a) $\text{Al}_{13}\text{Fe}_4$; (b) Al-Fe-Si

Table 4: EDS spectra of the second phases indicated in Fig. 12

Point	Mole fraction (%)						Predicted phases
	Al	Si	Fe	Ti	O	C	
N	75.33	1.27	6.72	0.03	-	15.64	Al-Fe-Si
O	79.35	0.45	4.89	0.04	-	15.28	$\text{Al}_{13}\text{Fe}_4$

4.2 Evolution of Fe-containing phase in TRC sheet via annealing

The TRC process involves the concurrent solidification and deformation of the alloys within the gap between two rotating rollers, and the partial dissolution promotes the formation of non-equilibrium second phases such as Al_9Si and $\text{Al}_{13}\text{Fe}_4$ along the RD in the resulting sheets. Notably, the presence of $\text{Al}_{13}\text{Fe}_4$ phase (with minor Si) can significantly deteriorate the sheet's workability during rolling due to its anisotropic mechanical properties^[29], therefore annealing treatments

are commonly employed to manipulate the distribution and morphology of these second phases.

The annealing treatment in this study was conducted at 550 °C, which promoted the diffusion of alloying elements, particularly iron and silicon. This diffusion facilitates the transformation of the second phases within the alloy. The Al_9Si intermetallic phase signifies the presence of silicon within the aluminum matrix. However, silicon has a higher diffusivity compared to aluminum. Therefore, rapid cooling rates in the TRC process are crucial to preserve a higher concentration of dissolved Si by suppressing its diffusion out of the Al matrix. Correspondingly, the DSC results suggest that the Al_9Si phase disappears during the slow cooling cycle, which can be attributed to its decomposition and potential reaction with other alloying elements to form new intermetallic products.

Similar to the previously reported substitution of Al atoms by Si in $\theta\text{-Al}_{13}\text{Fe}_4$ ^[39], the XRD patterns shown in Fig. 4 suggest that Si diffusion during annealing likely facilitates the transformation of most $\text{Al}_{13}\text{Fe}_4$ phases into $\text{Al}_{0.5}\text{Fe}_3\text{Si}_{0.5}$ and $\text{Al}_{0.7}\text{Fe}_3\text{Si}_{0.3}$ phases.

It is observed that the Al-Fe phase dissolves earlier than the Al-Fe-Si phase during annealing due to the diffusion of Si^[37,38]. A portion of the Fe-containing intermetallic precipitates form during the TRC process dissolve into the Al matrix^[39], leading to the formation of Al-Fe-Si phases.

Correspondingly, the morphology of the FIMCs largely depends on the subsequent phase transformations. Al-Fe-Si phases generally have a compacted morphology, which is less harmful for the mechanical properties. The diffusion of silicon element into many types of the FIMCs during annealing effectively modifies the second-phase particles, making them smaller and more uniformly dispersed within the matrix (Fig. 7).

Figure 3 illustrates substantial primary grain coarsening during the annealing process. The primary grains in the TRC sheet, initially several micrometers (μm) in size, grow to over 200 μm after annealing. Generally, Fe segregation is well known to intensify stress concentration in its vicinity within the alloy^[6]. Subsequently, stress concentration can induce localized deformation within the alloy, and these high-energy regions can then act as preferential sites for grain growth. The inhomogeneous distribution of FIMCs leads to an inhomogeneous distribution of strain in the 1060 aluminum alloy during the TRC process^[40], and the presence of a grain size difference between the surface and center regions of the TRC 1060 aluminum alloy can be partly attributed to the aforementioned stress concentration effects. After annealing, the second phase becomes even finer. The stress concentration effect near these fine second phases diminishes, reducing the overall FIMC network's resistance to grain boundary migration and promoting grain growth.

The stored distortion energy produced by the TRC process is released by recovery and recrystallization mechanisms during annealing treatment at high temperatures. As shown in Fig. 9, although annealing treatment results in a decrease in tensile strength in TRC sheets, their elongation increases. The TRC process significantly increases the dislocation density within the 1060 aluminum alloy, leading to pronounced work hardening. During annealing, the increased thermal energy facilitates the migration of dislocations and other crystallographic defects, such as vacancies, within the metal matrix. When the annealing temperature reaches the critical temperature, typically around 300–400 °C for aluminum alloys, the externally provided thermal energy is sufficient to overcome the stored energy within the deformed matrix. This triggers dynamic recrystallization in the work-hardened aluminum alloy, leading to the formation of new, strain-free grains. This process reduces the overall dislocation density and stored deformation energy within the sheet, resulting in a decrease in strength and an increase in plasticity.

It is reported that dimple fracture initiates from inclusions or second phase particles in aluminum alloys^[41]. From the fracture surface of TRC sample depicted in Fig. 10, a large portion of second-phase particles are located along grain boundaries. These particles can cause stress concentration and hinder dislocation

movement after deformation, contributing to intergranular fracture and the higher strength observed in TRC sheets^[42]. In contrast, annealing leads to grain growth and a decrease in the number of ductile dimples observed on the fracture surface.

Additionally, the dissolution of some second-phase particles into the matrix leads to a supersaturated solid solution of alloying elements. This reduces the overall stress concentration and weakens the pinning effect of these particles on dislocations, allowing for their easier movement. These combined factors promote ductile fracture in the annealed TRC sheets, further contributing to the decrease in strength and increase in plasticity.

5 Conclusions

(1) The microstructure of the TRC 1060 aluminum alloy primarily consists of α -Al grains, Al_6Si phases, and Al-Fe-Si phases, which can be irregular in shape. During the TRC process, α -Al phase forms at first, followed by the precipitation of $\text{Al}_{13}\text{Fe}_4$ and Al-Fe-Si phases at the solid-liquid interface caused by Scheil-like solidification kinetics. Finally, any remaining silicon in the liquid solidifies into granular Al_6Si phase.

(2) Due to the partial dissolution of FIMCs during annealing, the number of $\text{Al}_{13}\text{Fe}_4$ phases with minor Si significantly decreases. After annealing, FIMCs exhibit an increase in density and a decrease in size. This decrease in the size of FIMCs weakens their resistance to grain boundary migration, facilitating α -Al grain growth.

(3) Annealing the TRC alloy significantly modifies its fracture behavior, as evidenced by the transition from the mixed fracture mode of TRC sheet to the ductile fracture mode of annealed sheet. This change is associated with the dissolution and size reduction of second-phase particles, which promotes grain growth and reduces their ability to impede grain boundary movement. Consequently, the 1060 aluminum alloy exhibits decreased strength but increased plasticity.

Conflict of interest

The authors declare that they have no conflict of interest.

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