공학석사 학위논문

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ABSTRACT

Effect of Casting Speed on Internal Macro-segregation Behavior of Aluminum AA 6016 Alloys and Effect Lanthanum on High Mg Al-Mg Strips Fabricated by Twin-roll Casting

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Aluminum alloys have unique characteristics, such as high specific strength and light weight that make them attractive for structural applications in automotive, aerospace and number of other general engineering fields. Aluminum alloys wrought products are highly useful due to their specific properties such as high Ultimate Tensile Strength (UTS) and Yield Strength (YS).

Twin-roll casting (TRC) is a proven technology for the economical production of thin aluminum sheets manufactured directly from the melt. The advantages of this casting technique are numerous: - reduced capital costs, energy consumption, operating costs and scrap rate compared with a conventional Direct Chill (DC) casting route. Basically, molten metal is delivered via a refractory feeder tip directly into the gap between two internally water-cooled rolls, where it solidifies and undergoes hot deformation, before emerging as a solid strip or sheet.

TRC is a complex process containing solidification followed by hot deformation, which makes it necessary to control numerous parameters, such as the initial melt temperature, melt feeding rate, casting speed, nozzle shape, roll gap, amount of coolant and so on, in a narrow solidification region from the nozzle tip to the roll nip (or kissing point). It is also important to understand the mutual interaction between these parameters.

The quality of the final strips is closely related to the interactions of numerous working parameters, and the casting speed is one of the most important factors that reflect the interplay of those parameters. Few papers, however, have discussed stress or mechanical responses of the strips during TRC. When the solidification of melt is incomplete during contact with the cooled rolls, the casting speed is high. This increases the possibility of failure of the TRC process by spilling the melt from the strips. The quality of the strips degrades due to insufficient solidification.

On the other hand, if the solidification is complete far enough away from the roll nip, the casting speed is too low. This causes the rolls to get stuck in the nozzle, and the possibility of cracks in the formation of the strip increases. The exit of the nozzle is also stopped and operation is affected. Overall, control of the casting speed is important in successful fabrication of strips.

Keywords: Al-Mg-La alloys, AA6016 Alloys, Twin-roll Casting, Micro-structure, Internal Macro-segregation

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1. Introduction

1.1 Wrought aluminum alloys

Aluminum alloys have attracted much attention in the automotive industry due to their significant advantages such as weight reduction related to fuel efficiency. However, it is difficult to produce high strength aluminum strips by conventional processes based on direct-chill casting because of its numerous post-processes. As a result, the high strength aluminum alloy sheets from conventional processes are more expensive than steel. For this reason, producing aluminum alloy sheets has prevented their widespread applications in the automotive industry [1].

One of the techniques for solving this problem is the twin roll casting process. Twin roll casting is regarded as the most prospective technique to fabricate near-net-shape casting strips. It is also well known as an economic process to make aluminum alloy strips because it involves a one-step process from melt to wrought strip by combining solidification during plastic deformation.

Wrought aluminum alloys belonging to the 6xxx series possessing a high strength to density ratio and good corrosion resistance are considered a replacement for steel in the production of automotive body panels. Although the 6xxx series alloys are habitually named Al-Si-Mg alloys, they contain other components. While some of them (e.g., Cu, Mn, and Cr) are deliberately added, Fe is a component that is neither wanted nor avoidable. The presence of iron worsens the formability of the aluminum alloys, thus hindering their usage in the auto industry. The poor formability caused by the formation of various brittle iron-containing intermetallic compounds induces damage and premature failure during forming and bending operations. Since some of the intermetallic are less detrimental than others, different approaches and techniques were tried to modify the Fe-bearing intermetallic, i.e., to alter their type, shape, size, and distribution in wrought aluminum alloys. In spite of improved homogeneity achieved by high solidification rates, TRC aluminum strips reveal macro as well as micro segregation. The latter is inherited from dendritic solidification and scales with dendrite arm spacing which is in the order of several microns. Micro segregation is not a serious threat to the quality of the sheet products processed from TRC aluminum strips and is thus tolerated unless customer applications involve demanding forming operations. Besides, high temperature annealing treatments employed at the start of downstream processing help to homogenize dendritic segregation. Macro segregation in TRC aluminum strips, on the other hand, is most pronounced near the center plane of the strip and cannot be taken care of in a homogenization cycle. This type of segregation could range from a mild and gradual coarsening of the Al–Fe based intermetallic particles to solute-rich channels running more or less parallel to the casting direction. The latter is referred to as centerline segregation and is analogous to the channel segregation often encountered in ingots and slabs. The severity of centerline segregation is claimed to be linked with the solidification behavior which in turn is dictated by the alloy composition [2].

Segregation problems can be eliminated by controlling the solidification process carefully. Understanding the solidification curves of aluminum alloys, i.e. solid fractions versus temperatures during solidification, under conditions which approximate twin-roll casting process, are highly important for the control of solidification structures [2].

In order to produce thin strip traditional aluminum sheet production need many additional processing steps, such as the fabrication of a large slab by direct-chill casting, homogenization, surface scalping, and hot/cold rolling processes with inter-annealing. TRC can fabricate a 2–10 mm thin strip directly from the molten metal and has been gaining attention because of production cost reduction. TRC enables fabrication of near-net shape products with few additional processes such as, rolling and heat treatment which may reduce the production cost. On the other hand, despite the history of TRC, most mass-produced aluminum products are limited to low alloy systems, such as the 1xxx, 3xxx, and 8xxx series [3].

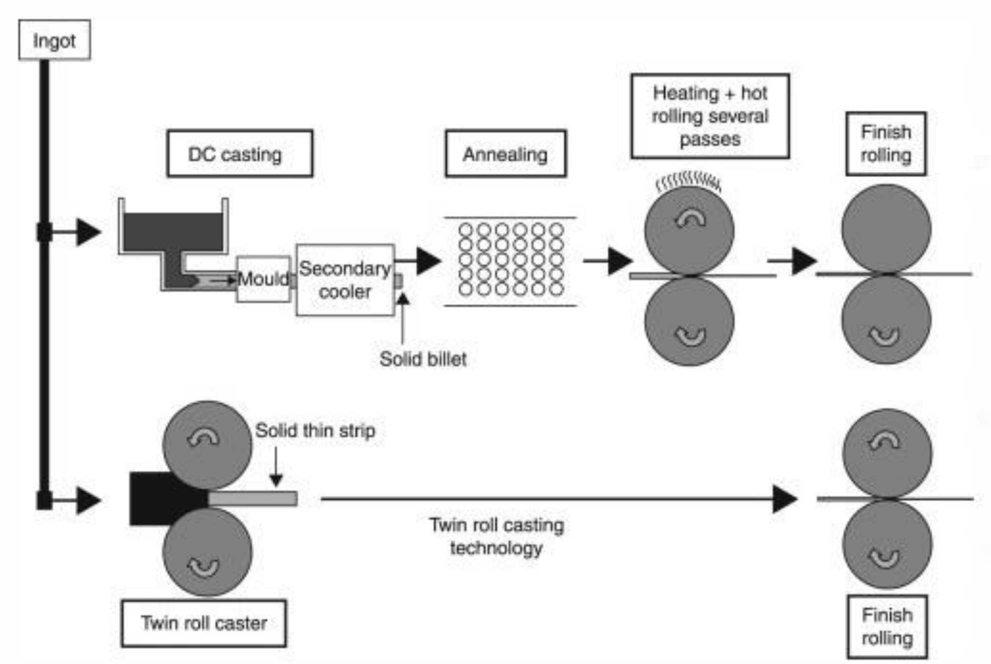


Figure 1.1: Process Scheme of Direct Chill Casting and Twin Roll Casting [4]

Many researchers have focused on characterizing center segregation; However, most treated alloys had a low amount of the alloying element or without rare earth elements. Only few works have focused on defining center segregation structure and the formation mechanism with high alloying elements and rare earth elements. In order to understand formation mechanism of center segregation with high range of solidification range, more researches should be done.

In the present study focused on characterizing center segregation in commercial AA 6016 alloy strips and Al alloys with high Mg content and adding small amount of La content.

In order to observe changes in the center segregation type with casting speed, the effect of different amount of elements were investigated.

1.2 Macro-segregation in Twin-roll Cast Aluminum Strips

Severe segregation and well-developed columnar dendrites always occur in a TRC sheet as a result of directional solidification as well as high content elements and wide solidification temperature range in the alloy. These segregated phases often form a network-like structure and provide an easy path for crack growth during hot plastic deformation at low temperatures. They also cause partial melting due to eutectic reaction at high temperatures, which narrows the temperature and strain rate ranges for successful hot working [5].

During TRC of aluminum, various parameters; such as alloying elements and casting speed may affect the ability of the sheet to be produced smoothly, as well as the microstructure of the sheet. Some research pointed out that high casting speed tended to form macro-segregation in the center of a TRC sheet. Thus the metallurgical defects in the TRC sheet may be abated by adjusting the casting speed.

Centerline macro-segregation is a big challenge area for TRC production. This defect strongly influences the microstructure and mechanical properties, reduces the fatigue strength, and cracks. Hence, control of centerline macro-segregation during the TRC process is an aspect of significance. The rolling parameters to eliminate macro-segregation in aluminum alloys strips have been studied for decades. But the subject still unclear and new researches are required to understand the mechanism of centerline macro-segregation [6].

In conventional direct chill (DC) casting, macro segregation is also a problem. To overcome the defect, external physical fields are applied to the DC casting processes, such as low frequency electromagnetic field, alternating and direct electromagnetic field, electric current pulse and ultrasonic field [6]. The experimental results indicated that these approaches are more or less effective to reduce or eliminate macro-segregation in DC casting [6]. Based on this, it is of potential interest to apply physical fields in the TRC process, which may effectively help eliminate macro segregation. However, the experiments have not been reported [6].

There are two types in twin-roll casting in the aspect of roll arrangement: horizontal twin-roll casting and vertical twin-roll casting. One roll is on top of the other so that the melt flows horizontally in the former, while two rolls are aligned side-by-side so that the melt flows vertically in the latte. This study only carried out on horizontal twin-roll casting.

All over the world many researchers have been investigating the effects of thermomechanical processes on mechanical properties of Al–Mg–Si alloys. However, these alloys were produced by conventional casting processes followed by a rolling process. Although it is well known that the metallurgical and micromechanical aspects of the factors controlling microstructure, unsoundness, strength and ductility, and other properties are complex, the solidification processing variables are a high order of importance in order to determine the resulting microstructure and its corresponding properties [7].

In the present investigation, a study on Al AA6016-0.3wt.%Cu ,Al-5.0wt.%Mg and Al-5.0wt.%Mg-0.5wt.%La alloys were processed by TRC by varying rolling speed and different amounts of alloying elements in order to clarify the effect of rolling speed and alloying elements on microstructure behavior and mechanical properties.

1.3 Current Research Objectives

Continuous casting in the aluminum industry is an effective and powerful manufacturing process for mass production of aluminum sheets. Twin roll casting (TRC) is a kind of continuous casting and it is applied to many different kinds of metals. However, due to center-line segregation along the sheet, its application is limited. Few works have focused on clearly defining center segregation structure and the formation mechanism.

The present study focused on characterizing center segregation in TRC AA6016 alloys with 0.3wt.%Cu addition, Al–Mg alloy strips with a high Mg content and Al-5.0wt.%Mg-0.5wt.%La. Changes in the center segregation type with casting speed were investigated, and the formation mechanism was analyzed.

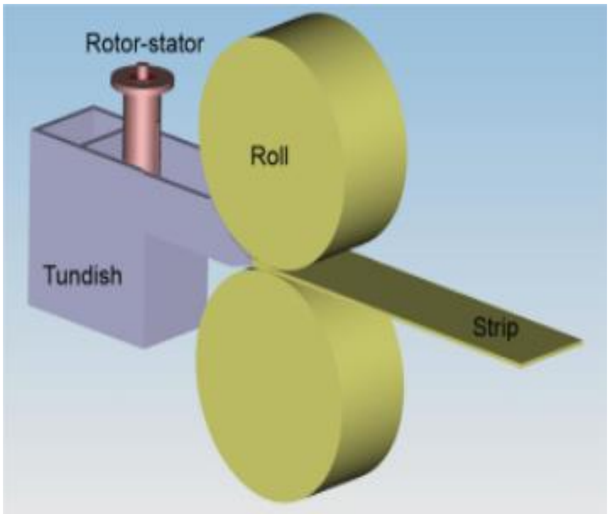


Figure 1.2: Schematic Illustration of the MC-TRC process [8]

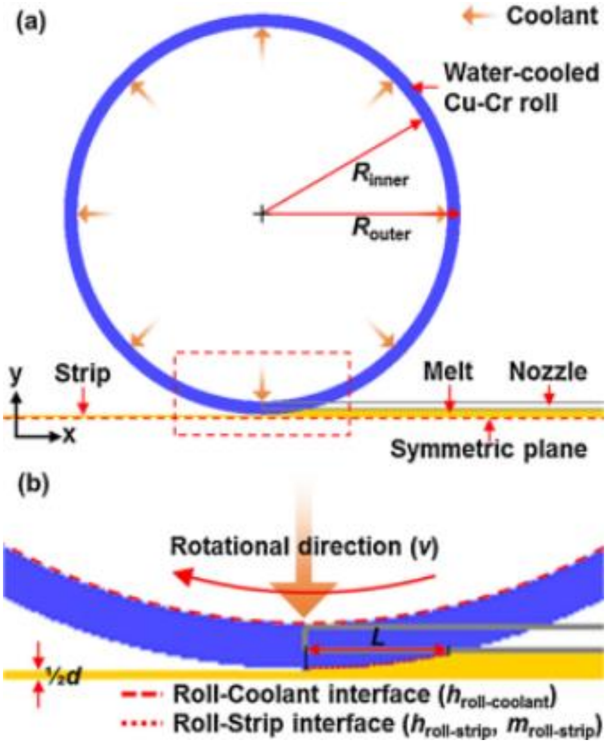


Figure 1.3: Schematic Representation of Horizontal TRC Process [9]

2. Experimental method

2.1 Twin-roll Casting Process in Current Research

Aluminum alloys were melted in an electric furnace. Strips were fabricated by using a horizontal-type twin-roll caster consisting of a pair of Cu–Be rolls. The strip thickness was changed from 4.8 mm to 3.8 mm as the casting speed increased. The diameter of the roll was 140mm. Available casting speeds range up to 8mpm, while strips are producible with casting speeds from 2.5 to 5.3mpm. Coolant flow rates of the equipped running water tunnels can be adjusted from up to 4 gallon/min. A nozzle made from rubiel bulk which resists 1400^{o}C, was employed during TRC casting to offer stable melt flow and feeding. For each single TRC lab-scale casting, up to 3.2 Kg aluminum ingots can be melted inside the hot chamber. During the TRC process, a motor-driven pendulum will move down at a constant speed pushing the melted aluminum into the runner and finally transferring the melt into the rap between two rotating rolls.

Roll			
Material	Cu-Be Alloy	Diameter	140mm
Speed Range	0~8 mpm	Coolant Flow Rates	0~4 gallon/min
Nozzle			
Material	Rubiel Bulk	Thickness	3.5mm
Hot-chamber			
Capacity	3.2 kg Al	Max. Temperature	900^{o}C
Pendulum			
Material	Rubiel Bulk	Volume	90*90*180 (mm)

Table 2.1 Parameters of the Twin-roll Caster

2.2 Microstructure Analysis

A longitudinal cross-section was taken from the middle part of as-cast strips as shown in the figure 2.1 and mounted with a resin holder to avoid rounding during the mechanical polishing. After the cross-sectioned samples were mechanically polished and etched, the microstructure was observed by optical electron microscopy (OEM). The samples were mechanically polished and etched with Weck's reagent (100 ml water + 4 g KMnO4 + 1 g NaOH), followed byy hundreds of consecutive images being obtained by optical microscopy. In addition, microstructures of the as-cast alloy samples were investigated by electron backscattered diffraction (EBSD) systems.

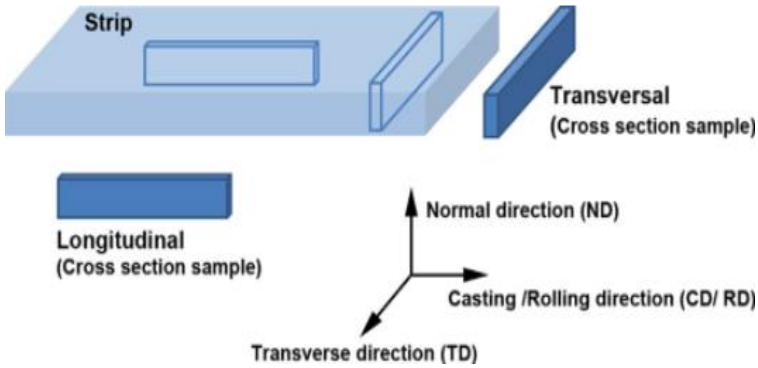


Figure 2.1: Schematic illustration of Sample Selection for Microstructure Analysis [10]

Etching Reagents	Solution Components
Keller's	2ml Hydrofluoric acid in 100ml H_2O
Weck's	1g NaOH and 4g $KMnO_4$ in 100ml H_2O
Barker's	1.8% Fluoboric acid in H_2O

Table 2.2 Components of Etching Reagents.

2.3 Tensile test

Tensile tests were carried out by using Instron 5582 Dynamic fatigue test equipment. The gauge length of the tensile specimen was 20.60 mm and the diameter was 6.40 mm. The whole length of the specimen was 56.32mm, as shown in the Fig. 2.3. This test was carried out at room temperature (25°C).

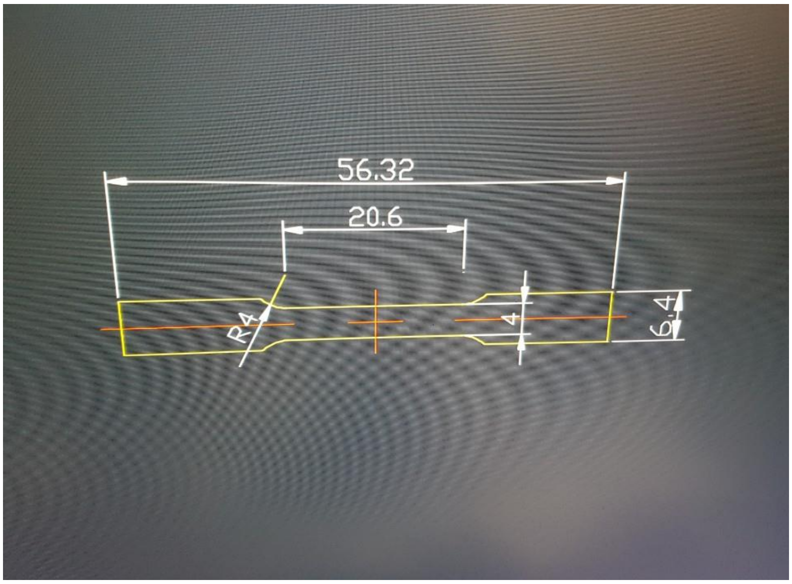


Figure 2.2: Dimension of Specimen for Tensile Test.

3. Analysis of solidification behavior and the effect of casting speed of TRC Aluminum AA 6016 alloy with fixed 0.3wt.% Cu alloys

3.1 Introduction

Currently heat treatable Al−Mg−Si aluminum alloys are being used extensively for auto body sheet applications. Special Al−Mg−Si alloys with improved formability and baked strength are in demand by the automotive industry. The strength and paint bake response of auto body sheet are important and these are not only influenced by the alloy compositions but also by the processing cycles. In Europe, AA6016 sheets are applied particularly successfully in gauges of 1−1.2 mm [11].

For several decades Twin Roll Casting (TRC) or strip casting, has been offered as an alternative to conventional DC-casting and hot rolling. The advantage of TRC is a shorter process route which combines casting and dynamic hot deformation down to a few mm gauge sheet in a single step. However, this shorter route leaves fewer possibilities to control or correct the development of microstructure. Hence careful attention to TRC process parameters is of prime importance, as emphasized by several authors. The cooling rates during solidification of strip cast aluminum are 2 - 3 orders of magnitude higher than those encountered in the DC casting process; as a consequence there are considerable differences in microstructure and mechanical properties between TRC and DC-cast material. The temperature gradients are found to be much steeper near the surface than at the center of the cast strip, hence local solidification times and microstructure will vary across the strip thickness. Due to the high cooling rates, the degree of super saturation of alloying elements will be higher, and the size and volume concentration of primary particles will be smaller than in DC-cast material. The alloying elements retained in solid solution obstruct dislocation movements during cold rolling, a dense dislocation network is therefore formed [12].

The heat treatable 6xxx series [Al–Mg–Si–(Cu)] aluminum alloys are finding increasing use in automotive skin panel applications where relatively high formability and in-service strength for dent resistance are major requirements. All over the world, the alloy of choice for such applications is currently the low Cu-containing alloy AA6016, which typically contains approximately 0.4 wt.% Mg and 1.0 wt.% Si, and which derives its strength from the precipitation hardening phase, Mg_2Si. The volume fraction of Mg_2Si is, in turn, affected primarily through the level of Mg within the alloy, although the Si content is also important [13].

Aluminum alloys are finding increasing application especially in the automotive industry, in view of the need to lower the weight of vehicles. The predominant sheet alloy used at present is AA 6016, an Al-Mg-Si alloy with the addition of Cu. AA 6016 has a good combination of strength and formability. Although the precipitating phases in Al-Mg-Si alloys (with or without the addition of Cu) have been studied for many years, a clear understanding of the nature of the precipitates has not yet been achieved [14]. For this reason precipitation analysis has been carried out in this study.

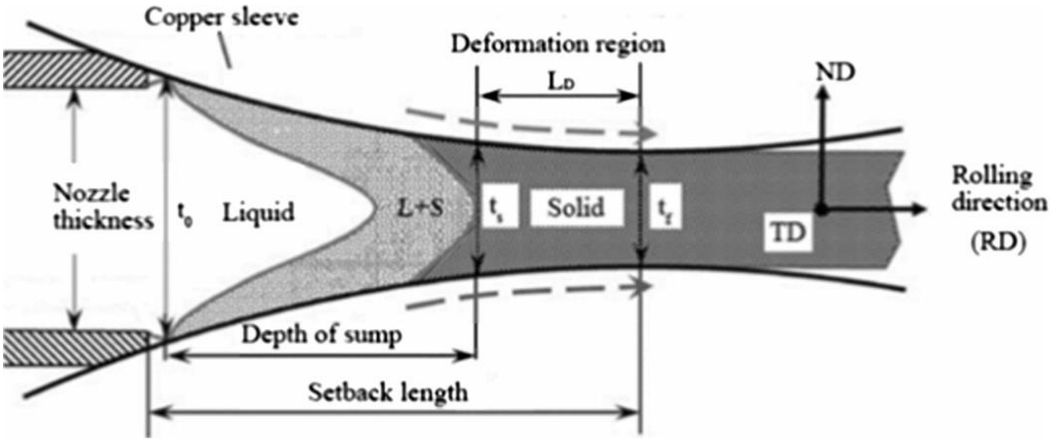


Figure 3.1: Schematic Illustration of Solidification Procedure During TRC [8].

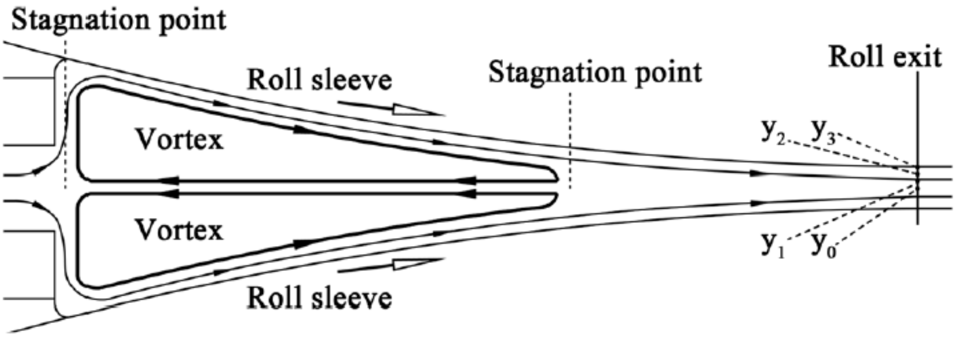


Figure 3.2: Development of Vortex with Stagnation Points.

3.2 Experimental Method

Solidification behavior of TRC Al AA 6016 alloys were investigated by Optical Electron Microscopy (OEM). Solidification range were calculated by JMatPro software Table 3.1. Chemical compositions of investigated alloys were shown in Table 3.2.

In this research Si content has been tried 1.0wt.%Si and 1.5wt.%Si respectively. And Mg content 0.25wt.%Mg and 0.60wt.%Mg has been tried respectively. In addition all of these alloys have been tried with two different casting speeds in order to observe the effect of casting speed on each alloy system. Macro-segregation is the focused point on OEM analysis. Moreover, mechanical properties have been observed such as; tensile and elongation values with changing amount the of elements and the casting speed.

Many different process parameters might affect the solidification behavior of TRC process such as roll separation force, melt temperature, feeding rate, casting speed and so on.

In current study, effect of casting speed has been analyzed on solidification behavior of AA 6016 alloys with fixed 0.3wt.%Cu alloy.

Fig. 3.3 shows the temperature distribution and liquid fraction distribution of AA 6016 alloys during the TRC process under different casting speeds for 3.08mpm and 5. 28mpm by Deform simulation software. According to results of simulation it can be seen that with higher casting speed less deformation region and longer depth of sump were observed which can be explained by the less contacting time between melt and roll [24].

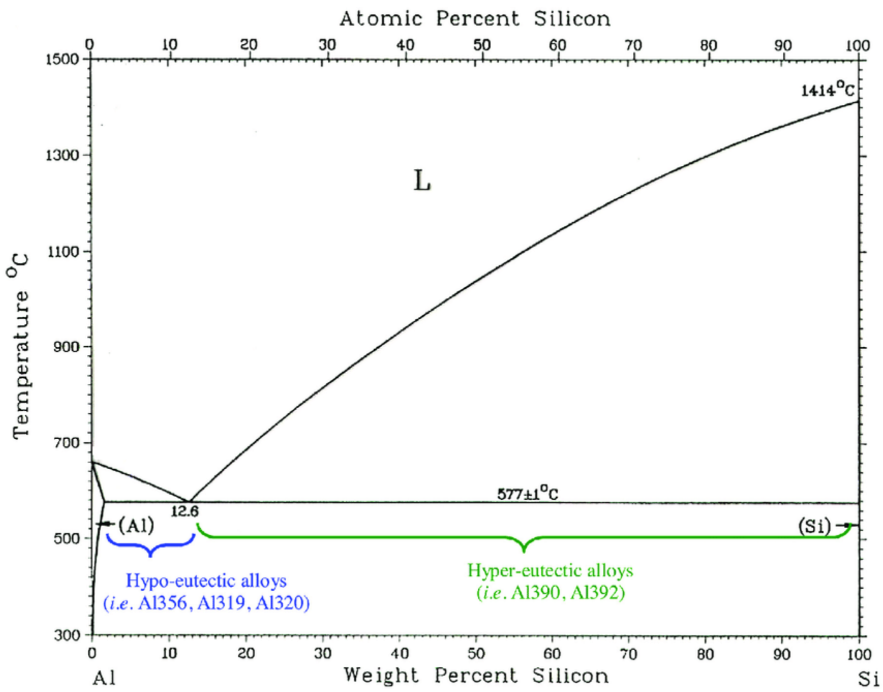


Figure 3.3: Al-Si Phase Diagram

Alloys	Liquidus Temp.	Solidus Temp.	Solidification Range Liquidus ~ Solidus Temp.
	(°C)	(°C)	(°C)
Scheil Condition (JMatPro)			
Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	652.48	530.00	122.48
Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	650.85	530.00	120.85
Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	650.10	530.00	120.10
Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	647.92	530.00	117.92
Al-1.2wt.%Si-0.40wt.%Mg-0.3wt.%Cu	650.61	530.00	120.61
Al-1.2wt.%Si-0.40wt.%Mg	651.41	557.29	94.12

Table 3.1 Chemical Compositions and Solidification Range of Investigated Alloys by using JMatPro Software.

	Alloy	Melt Temperature (˚c)	Casting Speed (mpm)	Thickness (mm)	Initial Separation Force (Kg)
1	Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	690	3.08	3.42	360
2		690	5.28	2.05	360
3	Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	690	3.08	3.18	360
4		690	5.28	2.15	360
5	Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	690	3.08	3.48	360
6		690	5.28	2.11	360
7	Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	690	3.08	3.27	360
8		690	5.28	2.07	360

Table 3.2: Chemical Compositions and Process Conditions of Investigated Alloys.

3.3 Results and discussions

The following Optical Electron Microstructure (OEM) pictures shows the micrographs of 8 different samples of as-cast homogenized at 500^0C for 6 hours. The healing effect of homogenizing on center segregation area has been observed. The relationship between thickness and casting speed has been considered, as exhibited in the OEM pictures. With these results the effect of casting speed on a center segregation area can be observed.

Channel segregation forms when the solidified shells encounter each other, accompanied by the compression of the mid-layer space, the remaining melt flows and is redistributed in the mid-layer region. In addition, when the melt is loaded at a lower level, striped solidification occurs rapidly, owing to the small amount of melt which helps to form less channel segregation.

Microstructures of a longitudinal cross-section of TRC strips were observed and shown. After that, segregation area percentages were observed. High speed casting can increase production efficiency, by making more product per unit of time.

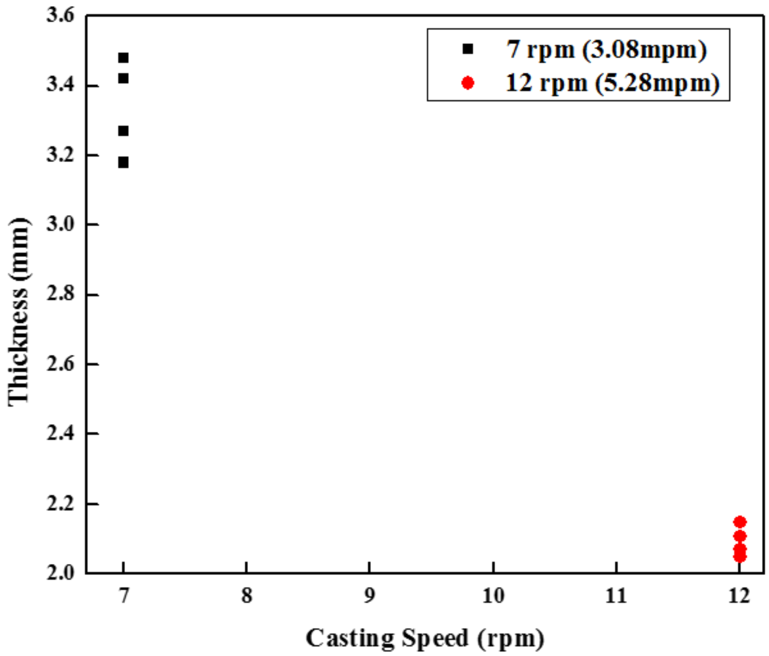


Figure 3.4: Effect of Casting Speed on Thickness.

Specimen	Composition	Casting Speed	Thickness
1	Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	3.42mm
2	Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	3.18mm
3	Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	3.48mm
4	Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	3.27mm
5	Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	2.05mm
6	Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	2.15mm
7	Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	2.11mm
8	Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	2.07mm

Table 3.3: Thickness of As-cast Alloys in Different Casting Speeds.

As has been said, twin-roll strip casting process is near-net-shape casting technology, with the production of thin strips having a thickness of about 2 mm to 5 mm, perhaps even less.

Depending on the strip thickness, the solidification rates occurred in this process. And the solidification rate is one of the important factor for microdefects in TRC strips. Furthermore the feeding rate and casting speed should be controlled carefully.

The most important advantage of twin-roll casting alloys compared to Direct Chill casting is the possibility to create sheets close to the final product. This is due to the reduction in thickness. With thinner specimens, less rolling process will be needed, which can decrease the production costs.

Thickness of as-cast materials always matters for TRC samples. In general, as-cast samples should be made at 3-5mm. After homogenizing and cold rolling, the final strip should be made.

In our research it can be seen that from the figure 3.4 and table 3.5 by increasing the casting speed, the thickness of the as-cast specimen decreases with a fixed feeding rate. Thinner strips need less additional cold rolling process. For this reason production cost can be lowered. That does not mean if we make a thinner specimen it is always better, because cold rolling processes can be minimized for final the product. However, in the case of thicker specimens we have a better chance to minimizing internal defects. For thinner specimens, we can improve the mechanical properties because the possibility of the work hardening is lessened.

As a result, it is hard to generalize that thinner or thicker specimens create better final products. Without analyzing microstructure it is hard to make a comment. For this reason microstructures have been analyzed first for as-cast samples. Al-(1.0-1.5)wt.%Si-(0.25-0.60)wt.%Mg-0.3wt.%Cu As-cast alloys for 3.08mpm and 5.28mpm respectively.

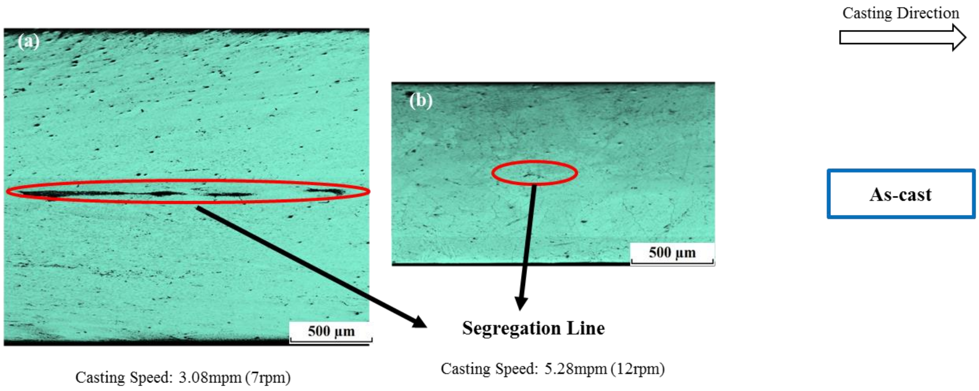


Figure 3.5: Optical Electron Microstructure image of TRC Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu As-cast alloys: (a) 3.08mpm and (b) 5.28mpm.

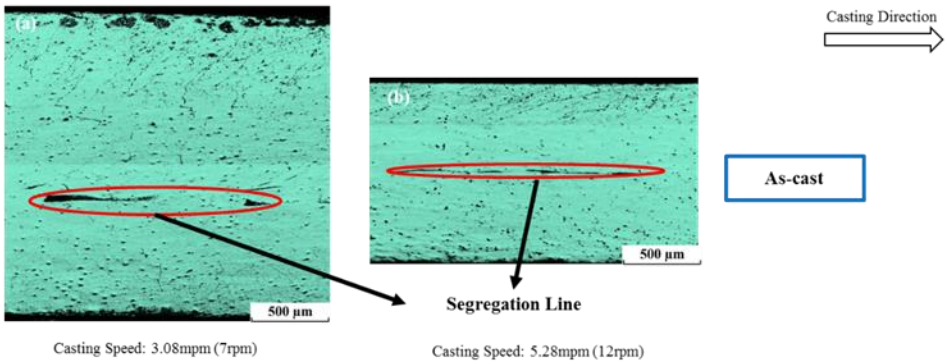


Figure 3.6: Optical Electron Microstructure image of TRC Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu As-cast alloys: (a) 3.08mpm and (b) 5.28mpm.

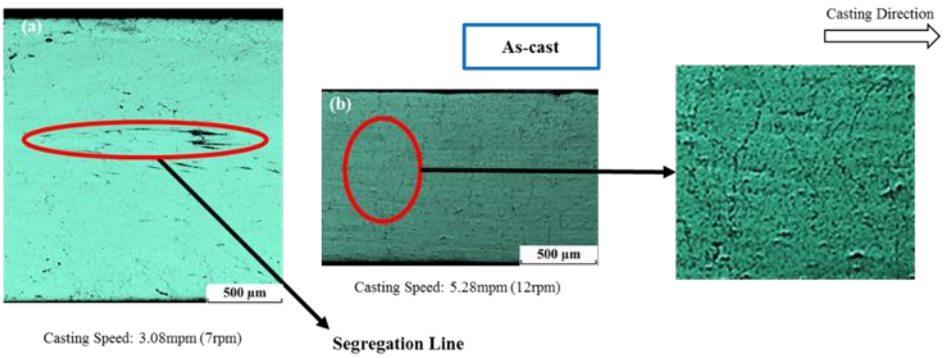


Figure 3.7: Optical Electron Microstructure image of TRC Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu As-cast alloys: (a) 3.08mpm and (b) 5.28mpm.

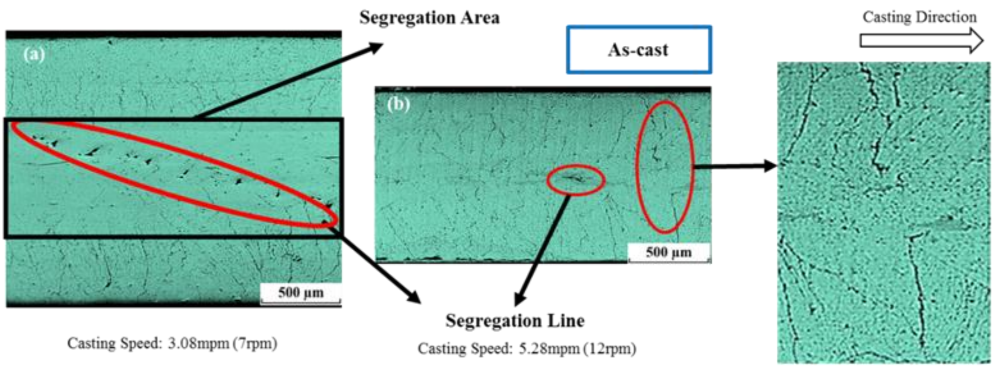


Figure 3.8: Optical Electron Microstructure image of TRC Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu As-cast alloys: (a) 3.08mpm and (b) 5.28mpm.

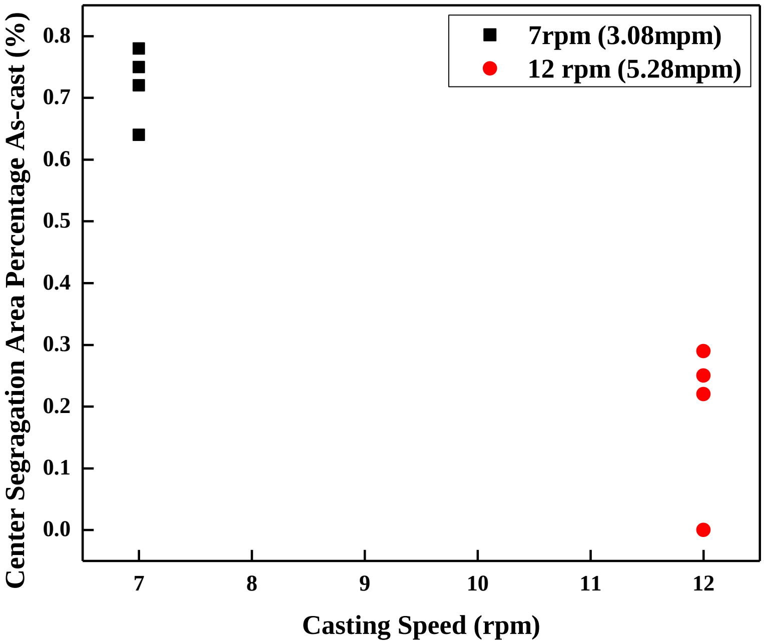


Figure 3.9: Effect of Casting Speed on Center Segregation Area Percentage.

Specimen	Composition	Casting Speed	Center Segregation Area Percentage As-cast (%)
1	Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.72
2	Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.64
3	Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.75
4	Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.78
5	Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	0.22
6	Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	0.29
7	Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	0.00
8	Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	0.25

Table 3.4: Center Segregation Area Percentage for 8 samples.

Roll separation force is an important mechanism of the center segregation area in the TRC strips. In our experiment, roll separation force was fixed at 350kg to clearly effect of casting speed. It should be simply said that in the solidification stage of twin-roll casting process, solid shells begin to grow on the roll surfaces after the molten metal is supplied between rolls [55].

OEM analysis clearly shows us the center segregation area for as-cast samples. The center segregation area percentages have been analyzed and reported.

This segregation analysis shows us there is no significant difference between different alloys. That is because there is no huge difference between the amount of alloying elements in the solidification range.

In addition, the casting speed has an important effect on the center segregation area. High speed created a thinner specimen with a fixed feeding rate and less segregation area percentages, which is in high demand for this research field, but only if we do not observe internal cracks.

Having said that, in a high speed case more internal cracks appeared, which should instigate more analyzes on internal cracks for as-cast samples. For this reason, internal cracks were observed in the following figures, 3.10 and table 3.7.

Moreover, in a high speed case (12rpm) Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu As-cast alloy has no center segregation area. Also in a low speed case Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu As-cast alloy has the lowest center segregation area percentage among low speed alloys.

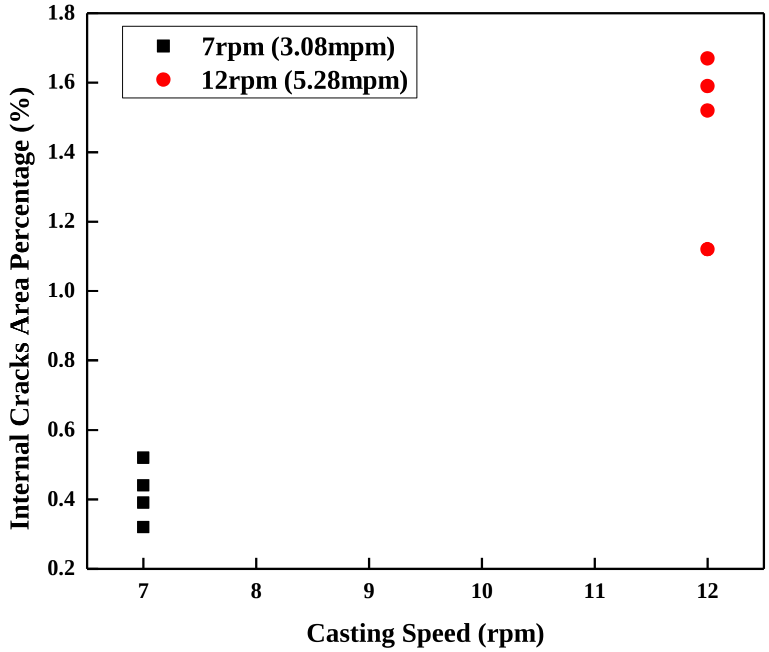


Figure 3.10: Effect of Casting Speed on Internal Cracks Area Percentage.

Specimen	Composition	Casting Speed	Internal Cracks Area Percentage (%)
1	Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.32
2	Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.39
3	Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.52
4	Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.44
5	Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	1.59
6	Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	1.12
7	Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	1.67
8	Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	1.52

Table 3.5: Internal Cracks Area Percentage for 8 samples.

As has been reported, there is a significant difference between two speeds in terms of internal cracks and center segregation area percentages. In a high speed case (12rpm-5.28mpm) many more internal cracks were observed and reported.

Even though high speed specimens tend to have less center segregation area than low speed cases, they have more internal cracks which have an important effect on mechanical properties.

As can be seen by figure 3.1 when casting speed increase depth of sump should be increase which lead to decrease the deformation region. The reason high speed cases have more internal cracks is that by increasing the casting speed the deformation region is low, which leads to less deformation in the TRC process.

If the roll separation force is low, the heat-transfer coefficient between the roll and strip will be small [53]. As long as the deformation rate is lower, enough strip cannot be cooled sufficiently. For this reason, control of all the parameters at once is critical for the TRC process.

After analyzing, homogenizing was carried out at 500^0c for 6 hours for all 8 samples. In order to see the healing effect of homogenizing on the center segregation area and internal cracks,-homogenized OEM pictures have been taken and center segregation area percentages were analyzed again.

Moreover, Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu As-cast alloy has the lowest internal crack area percentage among high-speed alloys. Apart from that, small differences in Magnesium and Silicon content have no significant effect on either center segregation area percentages nor internal crack area percentage.

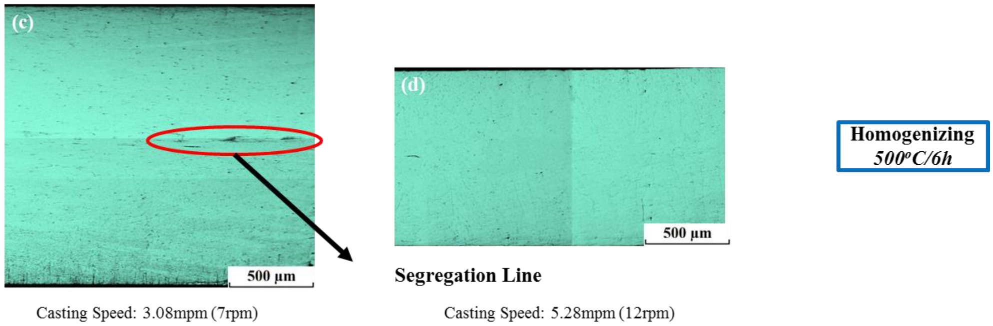


Figure 3.11: Optical Electron Microstructure image of TRC Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu As-homogenized alloys: (a) 3.08mpm and (b) 5.28mpm.

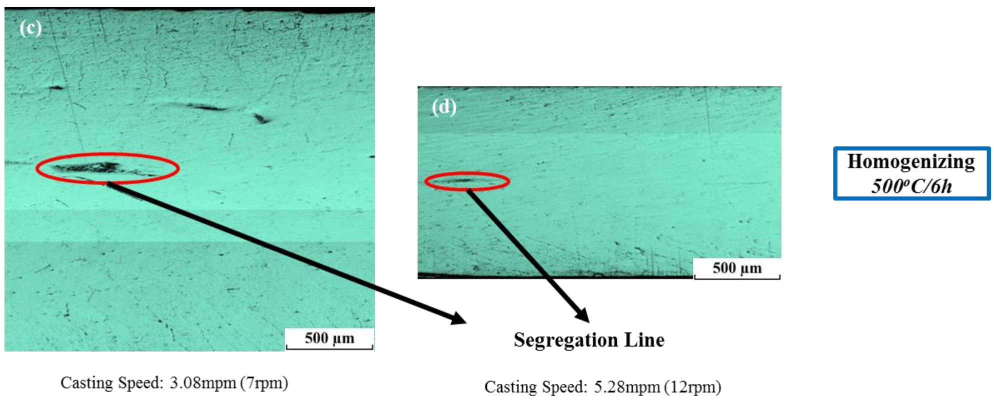


Figure 3.12: Optical Electron Microstructure image of TRC Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu As-homogenized alloys: (a) 3.08mpm and (b) 5.28mpm.

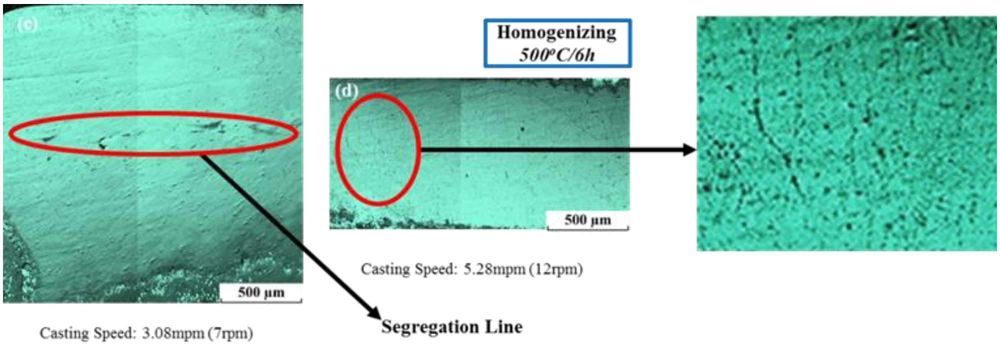


Figure 3.13: Optical Electron Microstructure image of TRC Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu As-homogenized alloys: (a) 3.08mpm and (b) 5.28mpm.

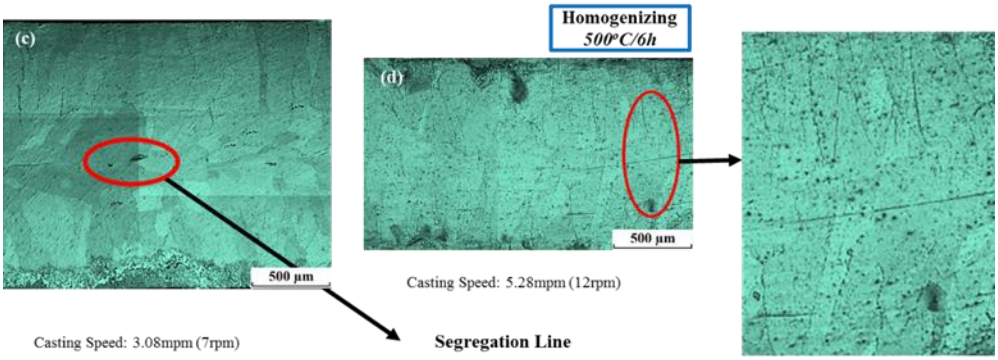


Figure 3.14: Optical Electron Microstructure image of TRC Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu As-homogenized alloys: (a) 3.08mpm and (b) 5.28mpm.

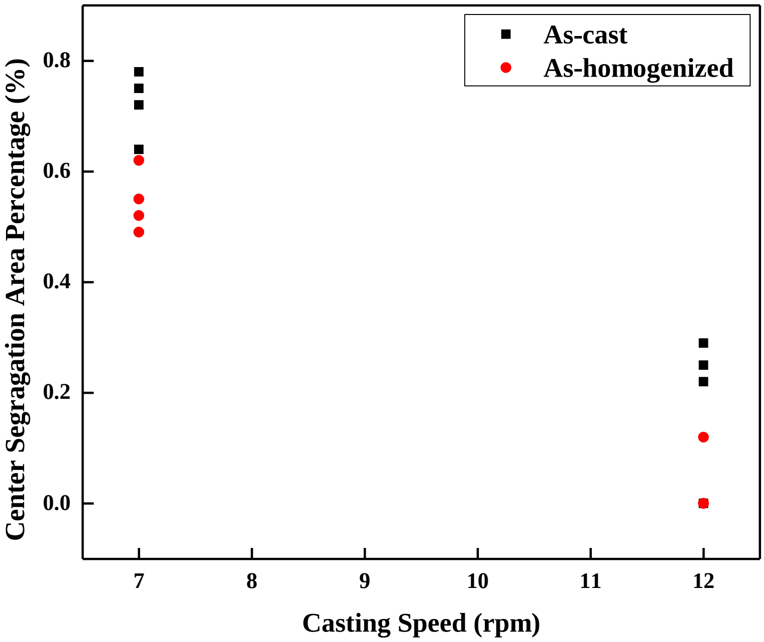


Figure 3.15: Effect of Homogenizing on Center Segregation Area Percentage.

Specimen	Composition	Casting Speed	Center Segregation Area Percentage As-cast (%)	Center Segregation Area Percentage After Homogenizing (%)
1	Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.72	0.62
2	Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.64	0.52
3	Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.75	0.55
4	Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	0.78	0.49
5	Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	0.22	0.00
6	Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	0.29	0.12
7	Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	0.00	0.00
8	Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	0.25	0.00

Table 3.6: Center Segregation Area Percentage for 8 samples before and after Homogenizing.

In this study, Al-(1.0~1.5)wt.%Si-(0.25~0.60)wt.%Mg-0.3wt.%Cu alloys have been investigated. In addition samples were examined after being homogenized at 500^0C for 6 hours to see the healing effect of homogenization on center segregation behavior.

Homogenization has a significant effect on centerline segregation in the twin roll casting process. The homogenization process was so effective in controlling centerline macro-segregation in sheet formation using TRC as compared to as-cast microstructure images [18]. It is important to understand the operational difficulties and microstructural defects that are encountered as the roll speed increased. These changes in operating conditions can have a dramatic effect on the microstructure because they affect the position and shape of the solid-liquid interface. In this paper we describe two microstructural defects such as; internal macro segregation and internal cracks.

It can be seen from the above pictures, that homogenizing can heal segregation but it cannot be totally removed. During homogenization, heat drives the melt with forced convection, which causes partially solidified dendritic re-melting. Then broken dendrites are driven to the two-phase region, new crystals are nucleated and solidification is completed at a higher cooling temperature, which results in a fine grained structure in the center [15].

In addition, in order to understand the characterization of the segregation area, Scanning Electron Microscopy (SEM) has been carried out.

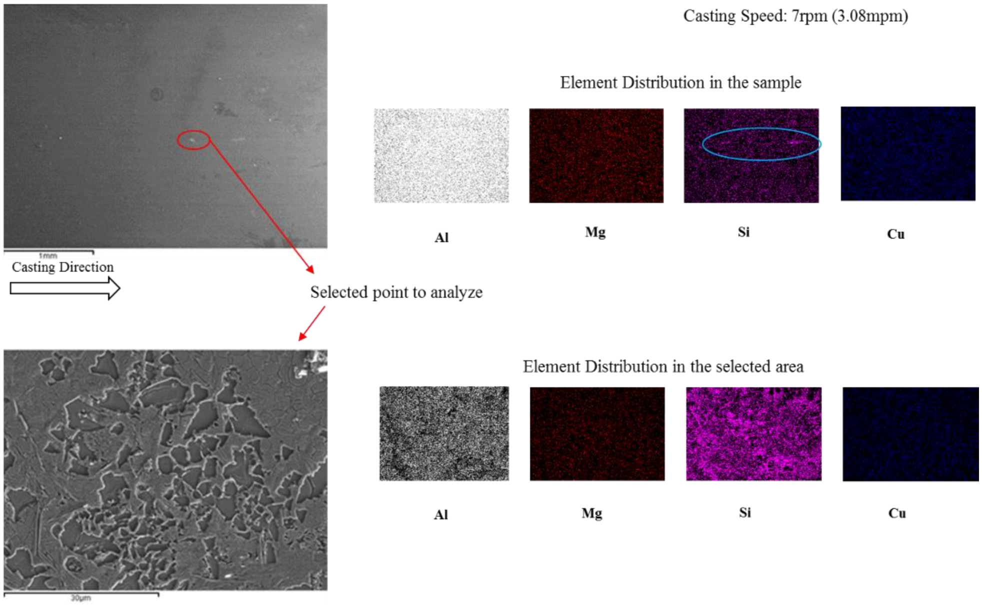


Figure 3.16: Al-1.0wt.%Si- 0.60wt.%Mg-0.3wt.%Cu Alloy As-cast SEM Analysis for Low Casting Speed (7rpm-3.088mpm).

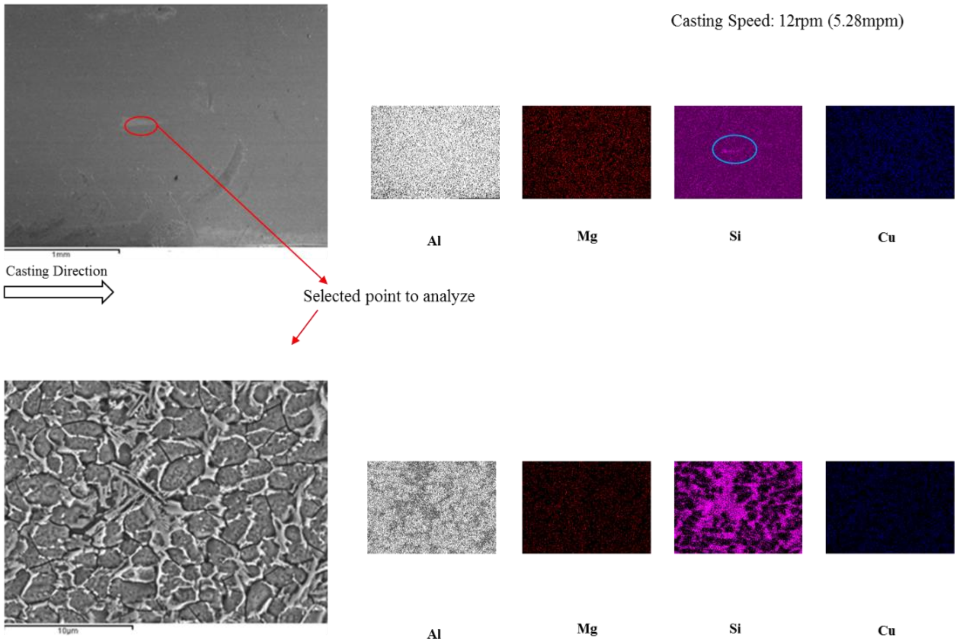


Figure 3.17: Al-1.0wt.%Si- 0.60wt.%Mg-0.3wt.%Cu Alloy As-cast SEM Analysis for High Casting Speed (12rpm-5.28mpm).

In the TRC process solidification starts when molten metal touches rolls which form the surface of a strip. Solidification continues from the top to bottom area and finishes at the center of the strip where center segregation area occurs.

The center segregation area is also highly effected by the solubility of alloying elements. Generally, the center segregation area is solution enriched.

From the SEM analysis it can be seen that center segregation area is Silicon rich. As well-known solubility of Si in Al is quite low. In the Al-Si binary diagram there is very little solubility at room temperature for Si in Al [39]. The maximum solubility of Si in Al occurs at the eutectic temperature and is 1.65wt.%Si. For this reason Si content in Al alloys should be carefully controlled.

It is well known that macro-segregation is directly related to the solidification process. When solidification advances from the surface of two rolls towards the hotter part of the casting, the driving force (the difference in free energy between the solid phase and liquid phase) leads the solute to become enriched in the solidification front, until segregation occurs near the kiss-point. Without any doubt, homogeneous solidification is the key point for script mechanical properties.

In order to characterize the segregation area Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu alloy has been selected and analyzed. Since there is no huge difference among Mg and Si alloys, only one alloy for two different casting speed results have been reported.

Si distribution seems not homogeneous. Mg and Cu distribution is more homogeneous. Mg and Cu might be solved in Al, but we observed Si segregation in Al alloys. Which means to minimize the center segregation percentage of Silicon content in Aluminum alloys they should be carefully controlled.

After analyzing the center area of the samples, to understand more about the effect of casting speed on solidification behavior, dendrite analysis was carried out and dendrite width was calculated.

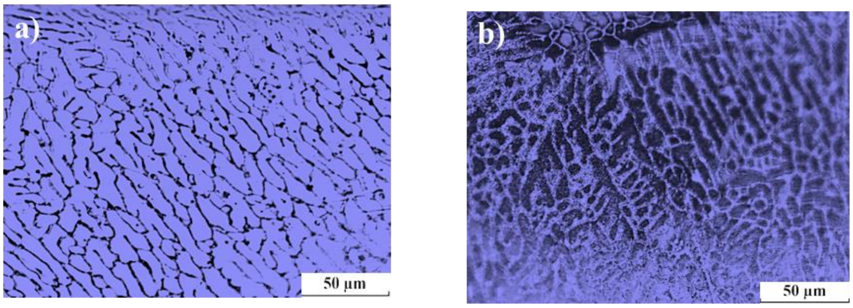


Figure 3.18: Al-1.0wt.%Si- 0.25wt.%Mg-0.3wt.%Cu Alloy As-cast Dendrite Images for (a)Low and (b)High Casting Speed.

Specimen	Composition	Casting Speed	Dendrite Width
a)	Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	10.3µm
b)		12rpm (5.28mpm)	7.2µm

Table 3.7: Al-1.0wt.%Si- 0.25wt.%Mg-0.3wt.%Cu Alloy As-cast Dendrite Width Results for (a)Low and (b)High Casting Speed.

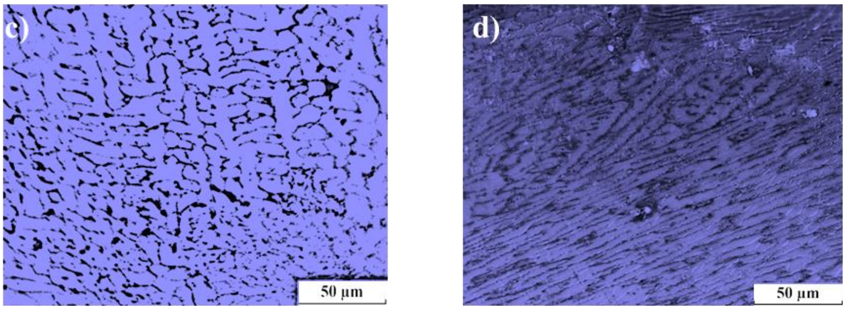


Figure 3.19: Al-1.0wt.%Si- 0.60wt.%Mg-0.3wt.%Cu Alloy As-cast Dendrite Images for (c)Low and (d)High Casting Speed.

Specimen	Composition	Casting Speed	Dendrite Width
c)	Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	11.2µm
d)		12rpm (5.28mpm)	7.8µm

Table 3.8: Al-1.0wt.%Si- 0.60wt.%Mg-0.3wt.%Cu Alloy As-cast Dendrite Width Results for (c)Low and (d)High Casting Speed.

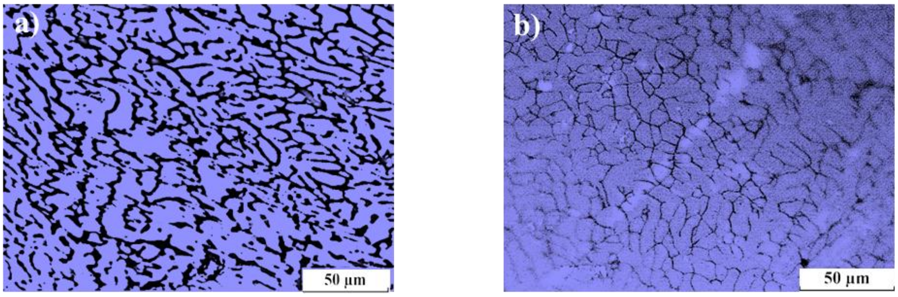


Figure 3.20: Al-1.5wt.%Si- 0.25wt.%Mg-0.3wt.%Cu Alloy As-cast Dendrite Images for (a)Low and (b)High Casting Speed.

Specimen	Composition	Casting Speed	Dendrite Width
a)	Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	9.2μm
b)		12rpm (5.28mpm)	5.8μm

Table 3.9: Al-1.5wt.%Si- 0.25wt.%Mg-0.3wt.%Cu Alloy As-cast Dendrite Width Results for (a)Low and (b)High Casting Speed.

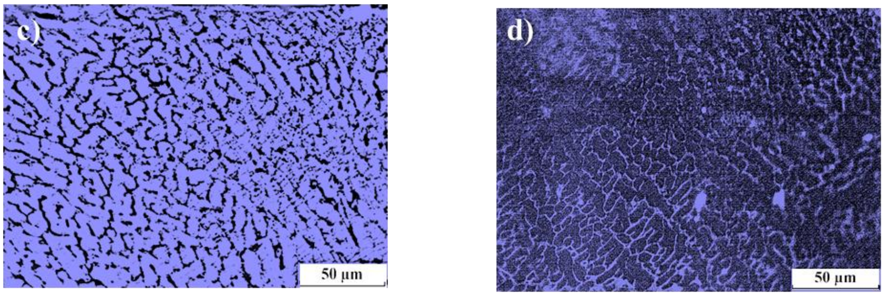


Figure 3.21: Al-1.5wt.%Si- 0.60wt.%Mg-0.3wt.%Cu Alloy As-cast Dendrite Images for (c)Low and (d)High Casting Speed.

Specimen	Composition	Casting Speed	Dendrite Width
c)	Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	9.7µm
d)		12rpm (5.28mpm)	6.1µm

Table 3.10: Al-1.5wt.%Si- 0.60wt.%Mg-0.3wt.%Cu Alloy As-cast Dendrite Width Results for (c)Low and (d)High Casting Speed.

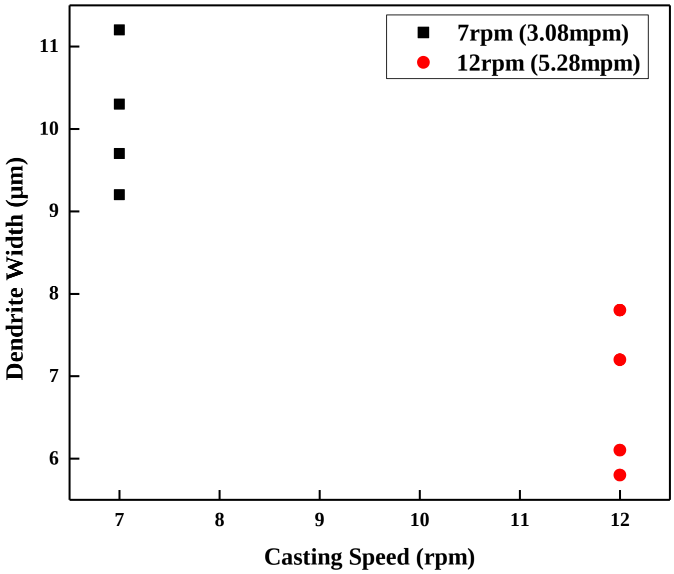


Figure 3.22: Al AA 6016 Alloys As-cast Dendrite Analysis for (c)Low and (d)High Casting Speed.

Composition	**Casting Speed**	**Dendrite Width**
Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	10.3μm
Al-1.0wt.%Si-0.25wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	7.2μm
Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	11.2μm
Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	7.8μm
Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	9.2μm
Al-1.5wt.%Si-0.25wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	5.8μm
Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	9.7μm
Al-1.5wt.%Si-0.60wt.%Mg-0.3wt.%Cu	12rpm (5.28mpm)	6.1μm

Table 3.11: Al AA 6016 Alloys As-cast Dendrite Analysis for 8 samples.

Nucleation and growth was affected by the solidification behavior of alloys, which determine the grain size and dendrite structure. In this study, solidification behavior was mostly affected by the casting speed.

During solidification, atoms lose their kinetic energy and the process becomes exothermic. A dendrite growing in an undercooled melt can be approximated as a parabolic needle-like crystal that grows in a shape-preserving manner at constant velocity.

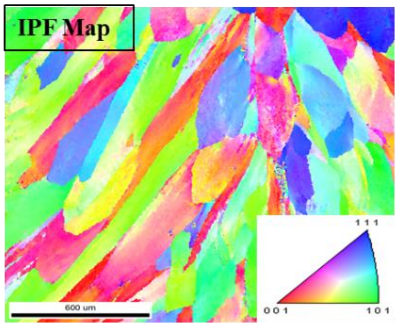
In general, if the melt is cooled slowly, nucleation of new crystals will be less than in large undercooling. The dendritic growth will result in dendrites of a large size. As a consequence of that, a rapid cooling cycle with long undercooling will increase the number of nuclei, and in this way minimize the size of the resulting dendrites.

All the different morphologies are thought to be the results of combined solidification and rolling processes. The different structures can be discussed in terms of changing the alloying elements amount and casting speed at other fixed parameters, such as melt temperature, roll separation, feeding rate, etc.

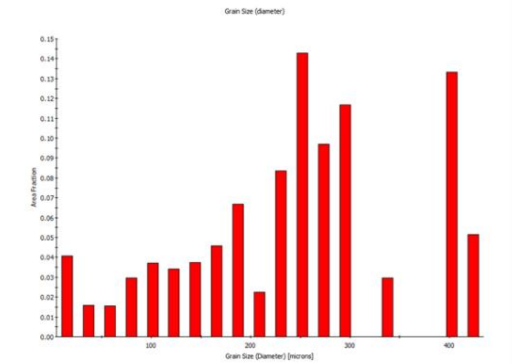
The dendrite structure is significant to microstructures during unidirectional solidification of alloys [25]. The relationship between solidification processing parameters (casting speed) and dendrite width changes have been investigated based on the experimental results. The dendrites of low speed grew larger than high speed cases.

In high casting speed cases, secondary dendrite arms cannot be distinguished. As a result of this, dendrite fragmentation occurs. And also less segregation was observed with relatively high speeds. However, mechanical properties were badly affected due to dendrite fragmentation which will be discussed later.

In the next step X-ray Diffraction (XRD) analyses were carried out to see the precipitation difference when adding small differences in the amount of elements , as well as the effect of homogenizing on precipitations.

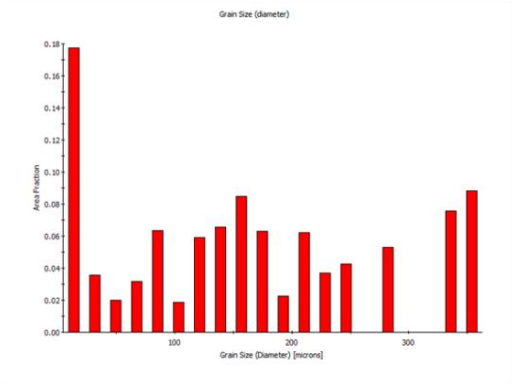


a) IPF Map of low speed (3.08mpm-7rpm)



c) Grain size distribution table of low speed (3.08mpm-7rpm)

b) IPF Map of high speed (5.28mpm-12rpm)



d) Grain size distribution table of high speed (5.28mpm-12rpm)

Figure 3.23: EBSD Analysis of Al-1.0wt.%Si-0.6wt.%Mg-0.3wt.%Cu alloys IPF Map of (a)Low Speed (b)High Speed and Grain Size Distribution Table of (c)Low Speed (d)High Speed

Composition	Casting Speed	Average Grain Size
Al-1.0wt.%Si-0.6wt.%Mg-0.3wt.%Cu	7rpm (3.08mpm)	244µm
	12rpm (5.28mpm)	163µm

Table 3.12: Al-1.0wt.%Si-0.6wt.%Mg-0.3wt.%Cu Alloys Average Grain Size Analysis for two Casting Speeds

Since grain sizes are not visible by OEM, to get the average grain size data, microstructures of the as-cast alloy samples were investigated by electron backscattered diffraction (EBSD) systems.

If the casting speed is high deformation region is low. With a lower deformation region, less deformation occurs. Since the TRC process combines casting and hot rolling together into a single operation, solidification and plastic deformation are processed in the same step.

As can be seen by EBSD results when the casting speed is high, TRC process provide a fine microstructure. That does not mean smaller grains lead to good mechanical properties by following the Hall-Petch Equation all the time. In order to reveal more information about mechanical properties, a Tensile Test is required, which will be discussed later in this study.

As can be seen from the Table 3.14, the average grain sizes are 7 and 12rpm are 244µm and 163µm respectively. This result shows us with higher casting speed, finer (smaller) grains can be obtained. It is also said that at higher cooling rates, finer grains are obtained [42]. It is generally found that grain size reduction increases the metal strength. The well-known Hall-Petch Equation shows that the yield strength is proportional to the reciprocal of the square root of the grain diameter [45].

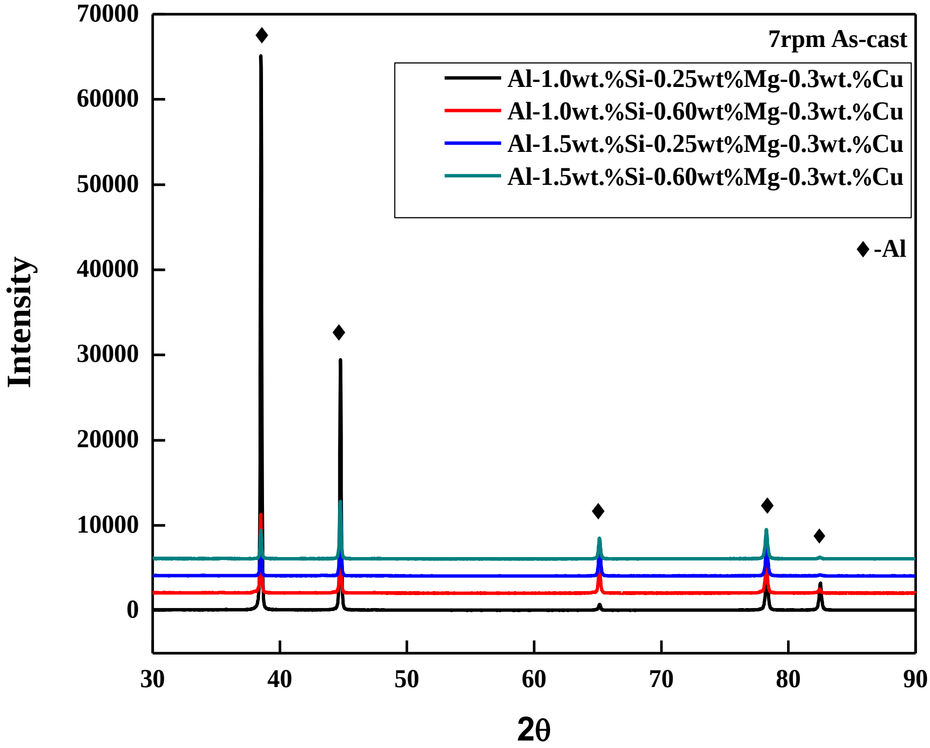


Figure 3.24: Al AA 6016 Alloys As-cast XRD Analysis for Casting Speed of 7rpm (3.08mpm)

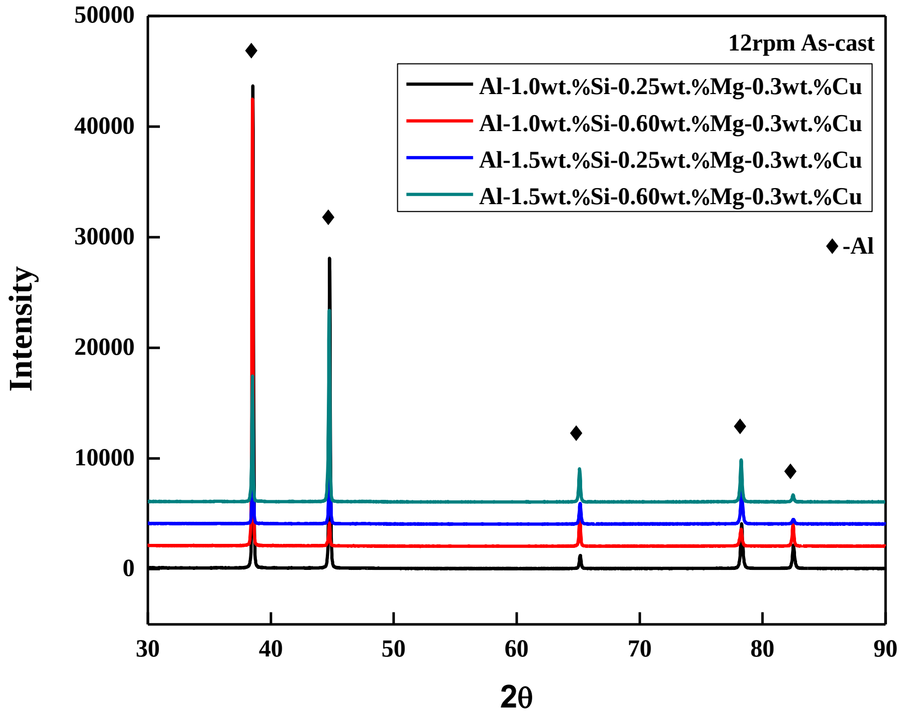


Figure 3.25: Al AA 6016 Alloys As-cast XRD Analysis for Casting Speed of 12rpm (5.28mpm)

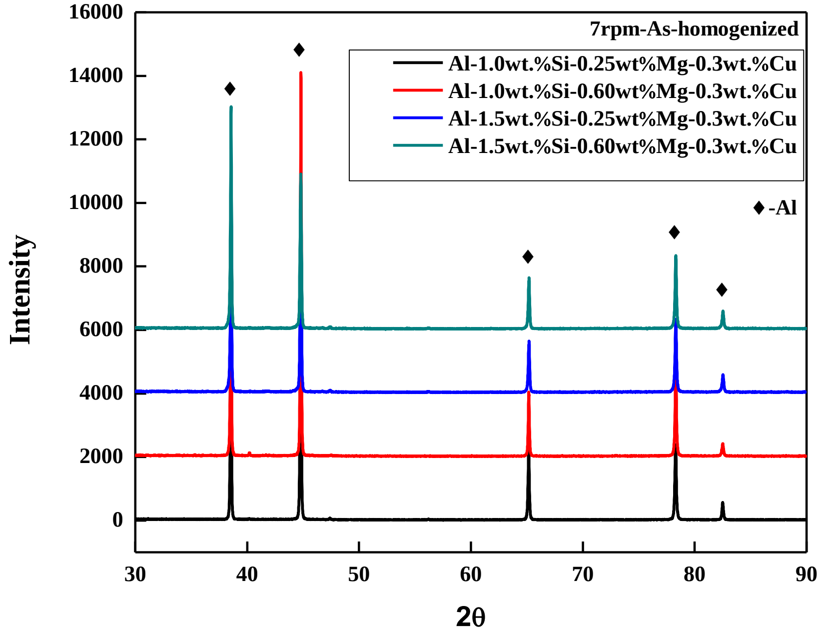


Figure 3.26: Al AA 6016 Alloys As-homogenized XRD Analysis for Casting Speed of 7rpm (3.08mpm)

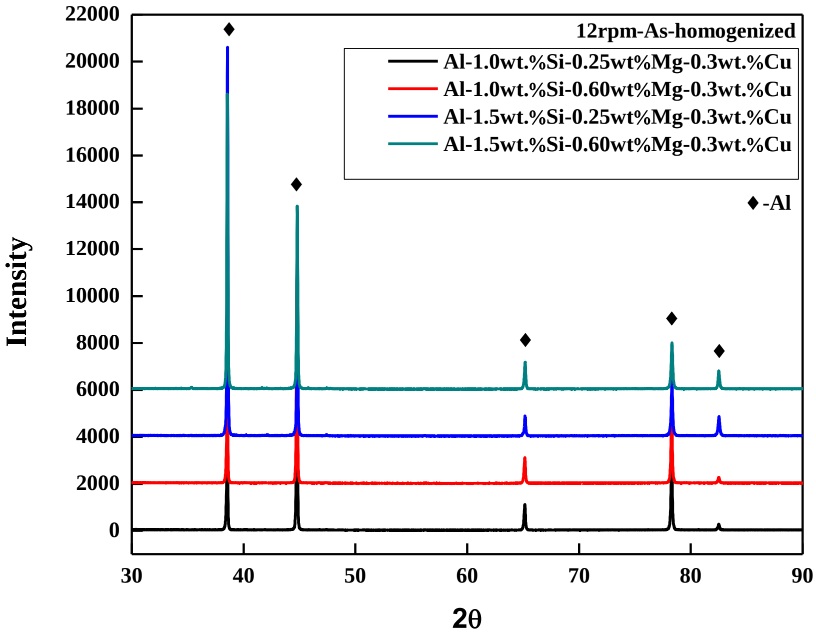


Figure 3.27: Al AA 6016 Alloys As-homogenized XRD Analysis for Casting Speed of 12rpm (5.28mpm)

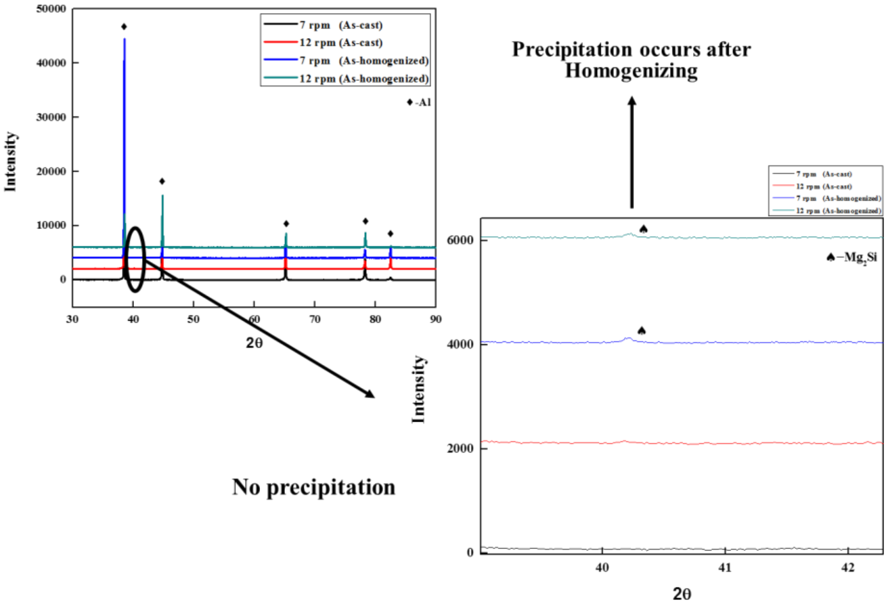


Figure 3.28: Al-1.0wt.%Si-0.60wt%Mg-0.3wt.%Cu Alloy As-cast and Homogenized XRD Analysis for the Casting Speeds of 7 rpm (3.08mpm) and 12rpm (5.28mpm)

Since alloying elements amounts are so close to each other, there is no significant difference between as-cast alloys even in high and low-speed cases in terms of precipitation. However, after the homogenizing process some more precipitations occurred which do not exist in as-cast materials.

After the homogenizing treatment, the developed alloy exhibited a more uniformed size distribution in the secondary phases. The existence of the Mg_2Si constituent phases was implied by the XRD data.

Normally, precipitation hardening can be increased by promoting the nucleation of precipitates, which results in a smaller and denser precipitate distribution, whether the precipitation hardening can be promoted by changing the morphology of precipitates has not been systematically investigated [26].

The age hardening in Al–Si–Mg alloys is caused by the precipitation of the Mg_2Si phase [27]. If the precipitates induced a pinning effect, re-crystallization would be retarded [28]. The strengthening potential of heat treatment Al-Mg-Si alloy is strongly influenced by the precipitates, and because of this, any improved understanding of the crystallography and formation mechanism of precipitates is of importance for improving properties of Aluminum AA 6016 Alloy [29].

Even though homogenizing has been done with the aim of minimizing the center segregation area percentage, it acted like a hardening process due to the high temperature. As a result of the high temperature treatment, Mg_2Si precipitation occurred. Mg_2Si intermetallic particles are formed during homogenization of as-cast samples.

The Mg_2Si phase is a good reinforcement phase with advantages of low density, high melting point, high hardness, and high modulus of elasticity, etc. The high content of the Mg_2Si phase can be directly obtained with a simple melt casting process, which can be easily combined with an external addition into a melt and casting method [30].

It should be noted that Davidkov et al. considered recently that large micron-size Mg_2Si particles formed on grain boundaries are the critical parameter promoting fracture of alloy AA6016 during bending, rather than large AlFeSi intermetallic particles [31].

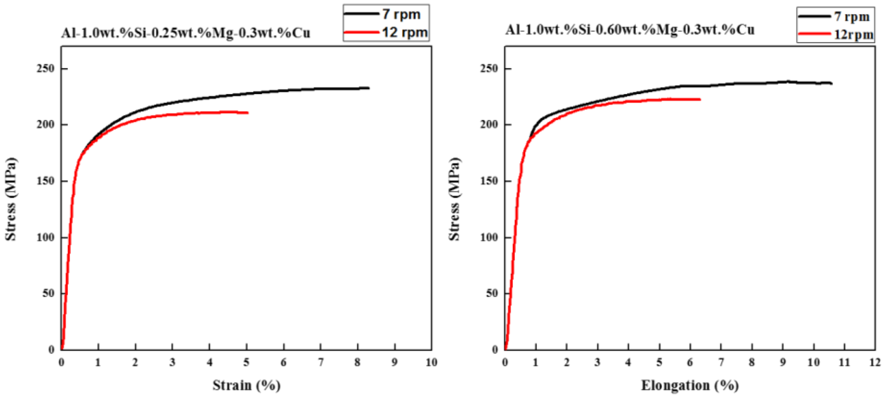


Figure 3.29: Al-1.0wt.%Si-(0.25-0.60)wt.%Mg-0.3wt.%Cu As-cast Tensile Test Results for 7rpm(3.08mpm) and 12 rpm(5.28mpm)

Composition	Casting Speed	Y.S. (MPa)	U.T.S. (MPa)	Elongation (%)
Al-1.0wt.%Si-0.25wt%Mg-0.3wt.%Cu	7 rpm (3.08mpm)	181.33	232.01	9.13
	12 rpm (5.28mpm)	174.53	210.85	5.01
Al-1.0wt.%Si-0.60wt%Mg-0.3wt.%Cu	7 rpm (3.08mpm)	207.91	237.81	10.74
	12 rpm (5.28 mpm)	186.10	222.88	6.29

Table 3.13: Al-1.0wt.%Si-(0.25-0.60)wt.%Mg-0.3wt.%Cu As-cast Tensile Test Results for 7rpm(3.08mpm) and 12 rpm(5.28mpm)

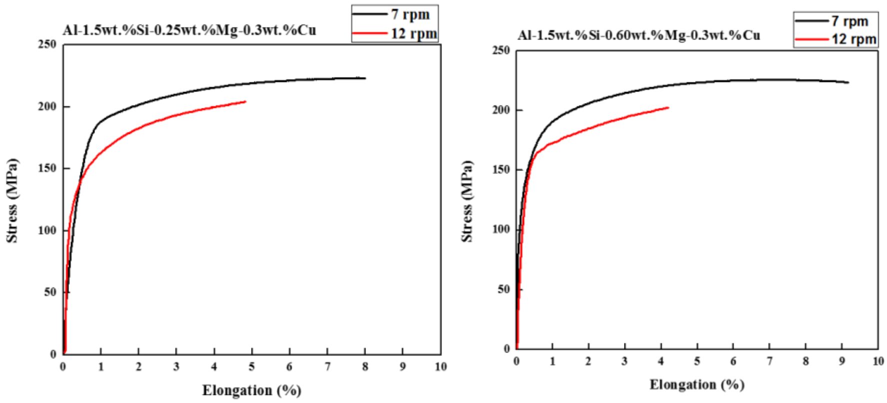


Figure 3.30: Al-1.5wt.%Si-(0.25-0.60)wt.%Mg-0.3wt.%Cu As-cast Tensile Test Results for 7rpm(3.08mpm) and 12 rpm(5.28mpm)

Composition	Casting Speed	Y.S. (MPa)	U.T.S. (MPa)	Elongation (%)
Al-1.5wt.%Si-0.25wt%Mg-0.3wt.%Cu	7 rpm (3.08mpm)	176.77	223.21	8.07
	12 rpm (5.28mpm)	146.82	204.28	4.83
Al-1.5wt.%Si-0.60wt%Mg-0.3wt.%Cu	7 rpm (3.08mpm)	186.20	225.29	9.17
	12 rpm (5.28mpm)	167.01	200.43	4.16

Table 3.14: Al-1.5wt.%Si-(0.25-0.60)wt.%Mg-0.3wt.%Cu As-cast Tensile Test Results for 7rpm(3.08mpm) and 12 rpm(5.28mpm)

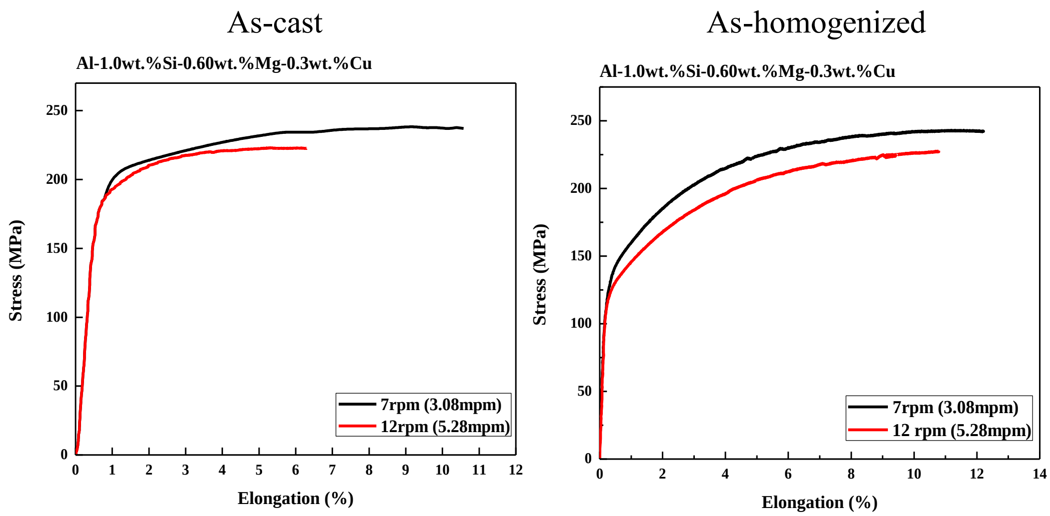


Figure 3.31: Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu As-cast Tensile Test Results for 7rpm(3.08mpm) and 12 rpm(5.28mpm)

Composition	Casting Speed	Y.S. (MPa)	U.T.S. (MPa)	Elongation (%)
Al-1.0wt.%Si-0.60wt%Mg-0.3wt.%Cu	7rpm (3.08mpm) (As-cast)	207.91	237.81	10.74
	7rpm (3.08mpm) (Homogenized)	136.25	243.06	12.22
	12rpm (5.28mpm) (As-cast)	186.10	222.88	6.29
	12rpm (5.28mpm) (Homogenized)	126.80	227.78	10.78

Table 3.15: Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu As-cast and As-homogenized Tensile Test Results for 7rpm(3.08mpm) and 12rpm(5.28mpm)

It is observed that strength and ductility decrease with the increase in rolling speed. That is because major problems like internal cracks and dendrite fragmentation occur.

There have been many studies on the relationship between microstructural and mechanical properties. However, in most cases only a limited range of microstructural parameters have been studied [49]. For cast metals, however, it is not always true that the strength improves with decreasing grain size. Strength will increase with grain size reduction only if the production of small grains does not increase the amount of micro-porosity, the percentage volume of second phase or the dendrite spacing [46][47][48]. A number of studies have pointed out the effect of microstructure, and particularly of dendrite spacing upon mechanical properties [47][49][50]. The dendrite fineness can be even more important in the prediction of mechanical properties than grain size [47].

Less segregation was observed with relatively high speed. However, due to the dendrite fragmentation mechanical properties were badly affected. This affect can be observed in Tensile Test results. As can be seen from the Tensile Test results there is a big difference in tensile strength and strain rate in terms of casting speed.

Moreover, Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu alloy shows the best results in both low and high speed cases. The same alloy system also has the lowest center segregation area rate. In addition Al-1.0wt.%Si-

0.60wt.%Mg-0.3wt.%Cu alloy system has the biggest dendrite width for both high and low speed 11.2µm and 7.8µm respectively. It should be noted that dendrite structures affect mechanical properties considerably. And this structure affected by solidification behavior which in this study is strongly related to casting speed. Since casting speed is the main reason for solidification time and velocity of solidification, it has the main role in mechanical properties.

Ideally, with higher speed good mechanical properties should be obtained. In this study Al-1.0wt.%Si-0.60wt.%Mg-0.3wt.%Cu alloy for high speed shows close strength rate to Al-1.5wt.%Si alloys both with 0.25wt.%Mg and 0.60wt.%Mg alloys.

It should be noted that homogenizing has a great influence on mechanical properties that can be seen in Table 3.16. Recrystallization is a process by which deformed grains are replaced by a new set of defect-free grains that nucleate and grow until the original grains have been entirely consumed.

Recrystallization is usually accompanied by a reduction in the yield strength and hardness of a material and a significant increase in ductility. As a consequence of that effect recrystallization can be used for the softening of metals before cold rolling. Also in the 2nd part of my research, Tensile Tests have been carried out for the final product that cold rolled up to 1mm which will be mentioned later.

Another reason for the improvement of homogenizing on mechanical properties is the effect of internal residual stress relief. Residual stress is the internal stress distribution locked into a material. These stresses remain even after all external loading forces have been removed. They are a result of the material obtaining equilibrium after it has undergone plastic deformation.

Internal residual stress is associated with a loss of dimensional accuracy, and premature failure such as deformation and cracking [40]. Residual stress also has a great effect on mechanical properties and resistance of parts to fatigue, and wear [41]. For this reason great improvement has been observed in mechanical properties for both elongation and strength.

3.4 Conclusion

- Casting speed of 7rpm (3.08mpm) specimens have grater center segregation area percentage as compared to high speed cases 12rpm (5.28mpm).
- High speed (12 rpm-5.28mpm) specimens have lesser center segregation area percentage as compared to low speed. However, high speed cast possess more internal cracks.
- SEM analysis shows that the center segregation area is Si rich.
- Dendrites are thicker in low speed cases than high speed cases.
- After homogenization more precipitation occurred in Al-1.0wt.%Si-0.60wt%Mg-0.3wt.%Cu alloys in both low and high speed cases.
- Grain size found to be finer (smaller) with the increase of the casting speed.
- Mechanical properties of Al-1.0wt.%Si-0.60wt%Mg-0.3wt.%Cu alloy have been found to be enhanced by homogenization.
- Best mechanical property has been achieved in the case of Al-1.0wt.%Si-0.60wt%Mg-0.3wt.%Cu alloys in both speeds.

4. Analysis of Center Segregation Behavior of High Percent Magnesium element in Al-Mg Strip and Analyze the Effect of La Element on Center Segregation Behavior

4.1 Introduction

In order to produce twin roll casting aluminum the sheet process requires lots of steps to make a thin, high quality sheet, such as the fabrication of a twin roll casting, homogenization, and hot/cold rolling processes with inter-annealing. Twin-roll casting can fabricate a 2–10 mm thin strip directly from the molten metal and has been gaining attention because of its economic benefits, especially for the Automobile industry . TRC enables fabrication of near-net shape products with few subsequent rolling and heat treatment processes, which reduces production cost and time. However, despite the decades-long history of TRC, most mass-produced aluminum products are limited to low alloy systems, such as the 1xxx, 3xxx, and 8xxx series. Recently, the increased demand for high-strength and low-cost aluminum sheets has prompted research on the fabrication of high-alloy aluminum products, such as the 5xxx and 7xxx series [3].

When liquid flows from a cold to a hot region in a casting, the liquid changes its composition, and this melts solid. Flow in one region leads to melting, to further flow, and thus to channel formation. Channels are formed in a number of casting situations in which liquid metal flows between the dendrites from a cold to a hot region. Although sheet can be produced, various defects arise which limit the range of operating conditions suitable for commercial exploitation. Usually, in twin-roll casting, the channels are formed in the central plane of the sheet and have almost constant spacing.

Cerium and lanthanum are relatively cheap among rare-earth metals (REM). In the present study, lanthanum was chosen to be added to an experimental high-Mg-Al alloy since, along with cerium they were the cheapest among the 17 members of the REM family. It is worth noting that these two elements together comprise approximately 90% of rare earth metals. The modifying effect of the La element on center segregation was examined by adding 0.5wt.%La.

Many previous studies have focused on characterizing center segregation; however, most treated alloys had a low amount of the alloying element. Just a few works have focused on clearly defining center segregation structure and the formation mechanism. Since the volume fraction of center segregation increases with the content of the alloying element, its formation mechanism needs to be clearly understood in order to control it. [16]. The present study focused on characterizing center segregation in TRC Al–Mg alloy strips with a high Mg content and analyze the effect of Lanthanum element on center segregation line. Changes in the center segregation type with the 2 different casting speeds were investigated, and the formation mechanism was analyzed.

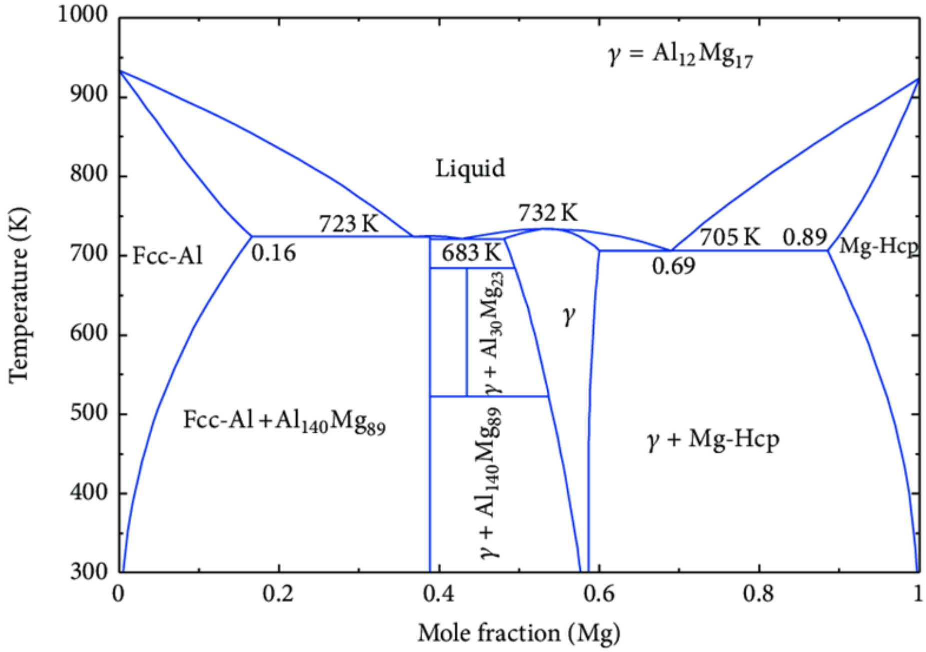


Figure 4.1: Al-Mg Phase Diagram

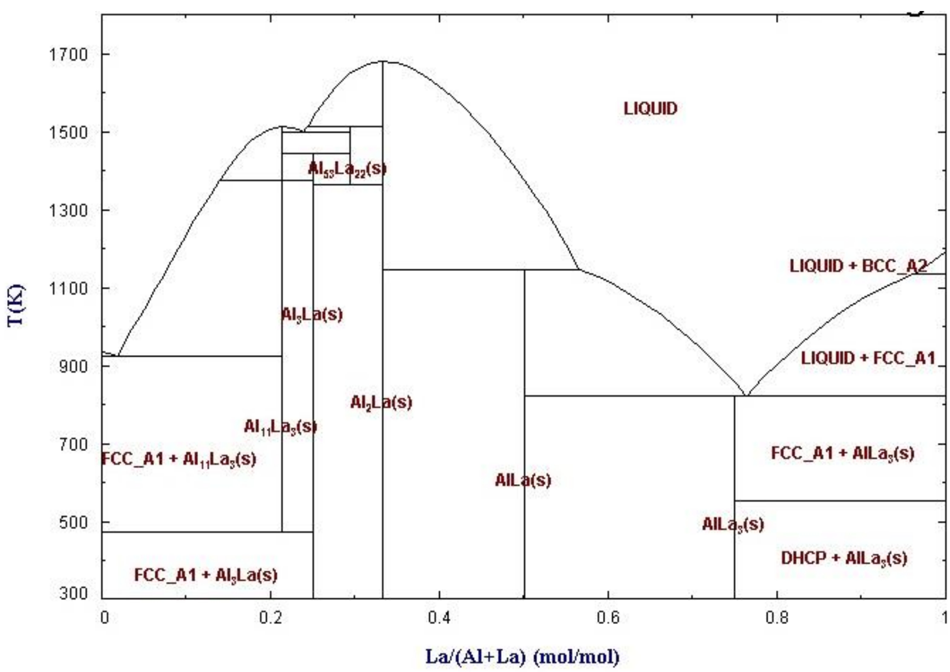


Figure 4.2: Al-La Phase Diagram

4.2 Experimental Method

Al-Mg strips with Mg compositions of 5.0% were fabricated by the twin-roll casting process (TRC) at two different casting speeds. Pure Mg and Al ingots were mixed and melted before twin-roll casting. Also in La case pure Aluminum and Mg-30wt.%La mother alloys have been used as an alloying elements. Each alloy systems has been tested with two different casting speed (6.5rpm and 7.5rpm).

Strips were prepared by a twin-roll caster. The caster consists of a pair of copper rolls 140 mm in diameter, equipped with interior running water tunnels. Springs were positioned between the upper roll and the caster frame to give separation roll force during the casting process, while the lower roll was fixed firmly. 3.2 kg molten metal was prepared in a hot chamber and stirred for 20 minutes to produce a homogenized melt. A pendulum was brought down at a constant speed with a feeding rate of 0.5lt/min. that pushed the melt through a nozzle toward the moving rolls with constant melt loads per unit time.

In the present work, strips were cast at 690°C. The initial roll force was set to 360kg and the casting speed was assigned 2.86mpm and 3.30mpm, see Table 4.1, to produce, strips with a thickness of about 3.14 to 3.50mm

	Alloy	Melt Temperature	Casting Speed	Thickness	Initial Separation Force
1	Al-5.0wt.%Mg	690°c	2.86mpm (6.5rpm)	3.30mm	360Kg
2		690°c	3.30mpm (7.5rpm)	3.14mm	360Kg
3	Al-5.0wt.%Mg-0.5wt.%La	690°c	2.86mpm (6.5rpm)	3.50mm	360Kg
4		690°c	3.30mpm (7.5rpm)	3.46mm	360Kg

Table 4.1: Chemical compositions and process parameters of Al-5.0wt.%Mg and Al-5.0wt.%Mg-0.5wt.%La alloys

Alloys	Liquidus Temp.	Solidus Temp.	Solidification Range (Liquidus ~ Solidus Temperature)
	(°C)	(°C)	(°C)
Scheil Condition (JMatPro)			
Al-5.0wt.%Mg	635.72	450.29	185.43
Al-5.0wt.%Mg-0.5wt.%La	635.00	450.28	184.72

Table 4.2: JMatPro Software Solidification Range Simulation Results of Al-5.0wt.%Mg and Al-5.0wt.%Mg-0.5wt.%La alloys

In order to do a longitudinal analysis of microstructure, a layer was machined and mounted in a resin holder which was followed by continuous polishing. After that, longitudinal cross-sections were etched using a Keller's reagent etchant to reveal the segregation and were anodized in a Barker's reagent under 20V for ~3min to examine the grain structure.

Heat treatment was carried out for all alloys for 8 hours homogenization at 420^0C. The tensile test samples were machined from as-rolled strips. In addition, tensile tests were carried out according to the ASTM standard B557M using an Instron 5582. The tensile test loading direction was parallel to the casting direction.

All the samples were homogenized at 450^0C for 6 hours and cold rolled up to 1mm with 30% reduction.

4.3 Results and discussions

The thicknesses of fabricated strips differ from each other and show the effect of casting speed on thickness at the same feeding rate. Strips cast at a lower casting speed were found to have more thickness.

Moreover, alloys which contain Mg are much less likely to stick, and there is practically no sticking problem with 5.0wt.%Mg alloys. A high-magnesium alloy is usually used to condition newly ground rolls or ones where extreme sticking has occurred [20].

Generally, commercial twin-roll cast aluminum alloys have narrow freezing ranges. However, this research aims to make new alloy designs, so that high freezing range alloys have been chosen to investigate segregation behavior which is so rare in this field.

Twin-roll casting has been used to produce thin sheet (1-4mm thick) in aluminum alloys. The micro and macro-defects found in thin sheet material have been investigated. Defects include surface defects (surface bleeds), internal defects (such as channel segregates and deformation segregates), inhomogeneity of grain and secondary arm spacing (banding), and macroscopic buckling [22]. This research describes the experimental observations and relates their occurrence to the alloying elements and casting speed. Mechanisms are proposed to explain segregation behavior.

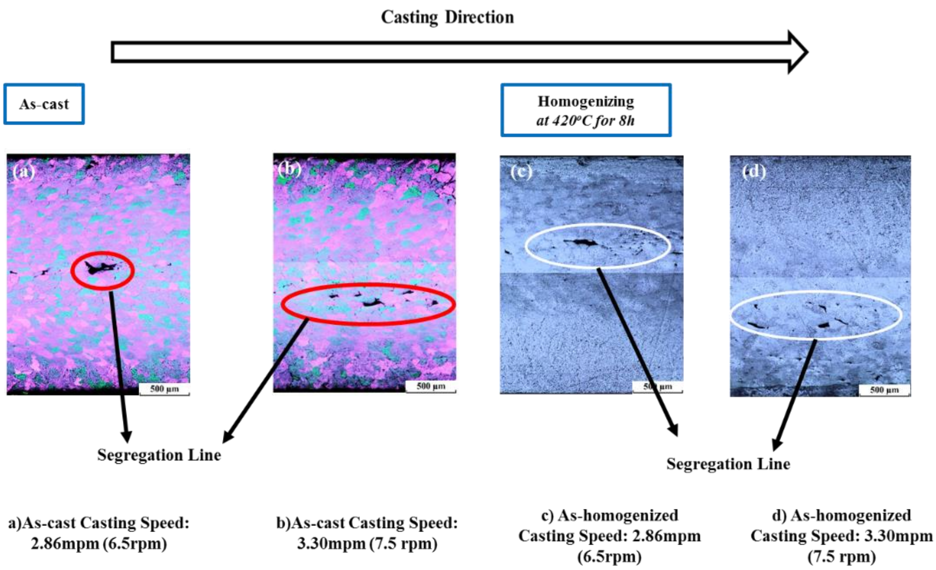


Figure 4.3: Optical Electron Microstructure image of TRC Al-5.0wt.%Mg alloys: (a)As-cast 2.86mpm, (b)As-cast 3.30mpm (c)As-homogenized 2.86mpm, (b)As-homogenized 3.30mpm.

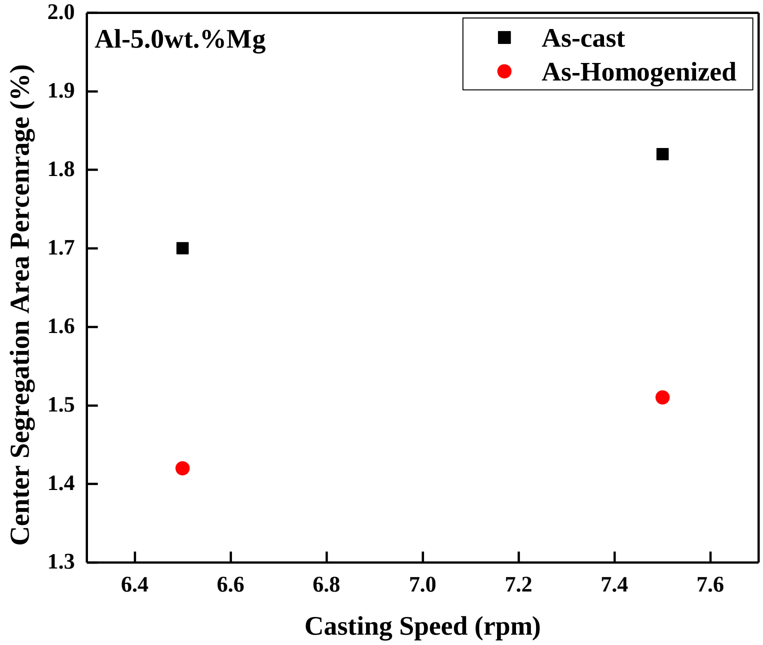


Figure 4.4: Al-5.0wt.%Mg Center Segregation Analysis for As-cast and As-homogenized Samples.

Specimen	Composition	Casting Speed	Center Segragation Area Percentage As-cast (%)	Center Segregation Area Percentage After Homogenizing (%)
1	Al-5.0wt.%Mg	6.5rpm (2.86mpm)	1.70	1.42
2	Al-5.0wt.%Mg	7.5rpm (3.30mpm)	1.82	1.51

Table 4.3: Al-5.0wt.%Mg Center Segregation Analysis for As-cast and As-homogenized Samples.

Hot tearing occurs in the twin-roll casting of thin strip, especially in alloys with long freezing ranges, such as Al-5.0wt.%Mg alloys. Often the tears fill with solute-rich material, in which case they are visible on metallographic sections of the sheet.

Segregation occurs when the deformation process is so rapid that the solid and liquid deforms together rather than the liquid being squeezed out of the solid. During the deformation process, small liquid regions are formed between the solid grains.

Metallographic examination shows that these regions are equiaxed and not elongated in the casting direction.

The center segregation line may cause lattice distortion, which may increase the localized stress and in this way it may cause fracture toughness [52].

As can be seen from the table 4.3, high percent Mg alloys have much higher center segregation area percentages compare to Al AA 6016 alloys which is also shown in simulation showing solidification range differences. Even after homogenizing, center segregation area percentage is considerably high.

To see the composition range of Al-5.0wt.%Mg alloys Scanning Electron Microscope (SEM) analysis have been done that are shown in the following figure.

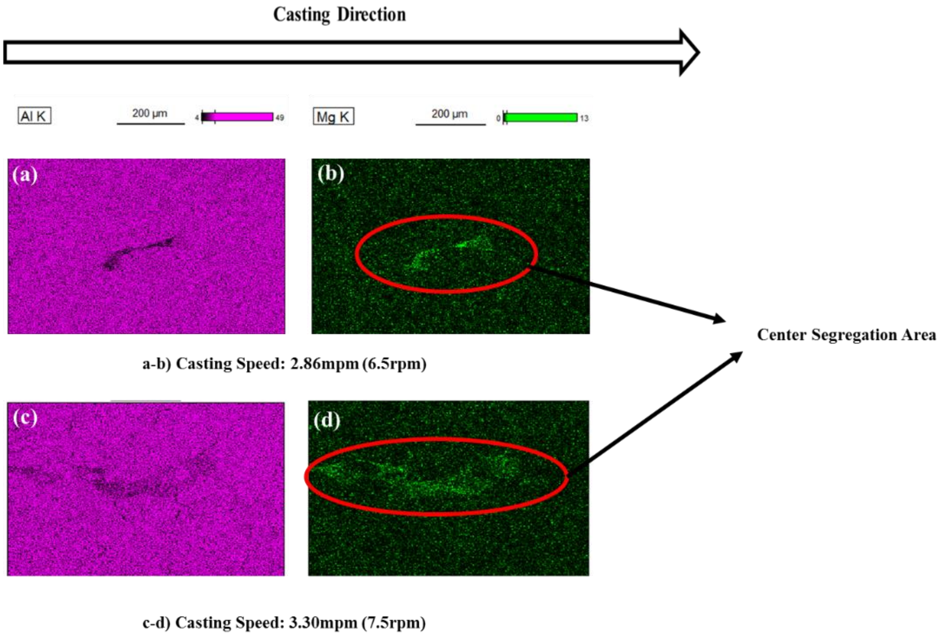


Figure 4.5: SEM Images of As-cast TRC Al-5.0wt.%Mg Alloys with the Casting Speeds of a) and b) 2.86mpm c) and d) 3.30mpm.

The center segregation area which appears in the last solidified zone in the center of the strip, is one of the casting defects and is a typical microstructural feature of a TRC sample. Center segregation area usually remains up to the final product, even after homogenizing and rolling as can be seen from the above OEM images. Therefore, this should be carefully controlled during the casting process.

SEM images show microstructures of the center segregation region which is shown in Fig. 4.6. For central segregation, a content of the Mg rich region was observed. The SEM image clearly shows that the Mg-rich phase was well connected along the casting direction.

For the Al-5.0wt.%Mg alloy samples, the Mg content increased in the middle of the sample. This segregation type occurs because of the high percent of Mg content in the alloy.

The expected effect of the Lanthanum element is to make smaller grain sizes, more finely dispersed second phase particles, and smaller aspect ratios of the particles. In addition, the “healing effect” of REM is also well known in this field. For this reason XRD analysis has been carried out to reveal the precipitations.

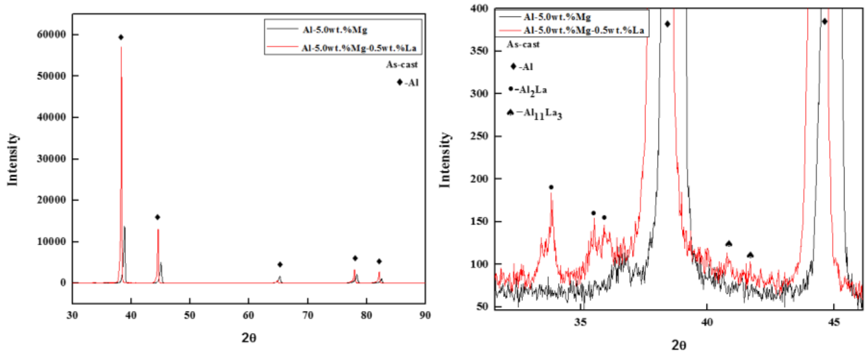


Figure 4.6: XRD Graphs of As-cast TRC Al-5.0wt.%Mg and Al-5.0wt.%Mg-0.5wt.%La Alloys

Intermetallic compounds are usually formed when alloying elements, are added to Al based alloys. Microstructure, mechanical properties and their modifications should be analyzed after adding more alloying elements.

Solid solution strengthening is a type of alloying that can be used to improve of a metal. Solid solution strengthening works by adding an alloying element to another element which makes a solid solution. In our research the La element has been used as an alloying element, forms Al_2La and $Al_{11}La_3$ precipitates as can be seen by figure 4.5

The total amount of intermetallic in as-cast alloys is below 5% volume. Despite little intermetallic in the cast microstructure, the detrimental effects on the ductility and service performance of alloys are huge [32].

The equilibrium and non-equilibrium reactions that occur during the casting of Al alloy account for intermetallic phases. Coarse intermetallic particles are formed in the interdendritic regions during solidification or at a relatively high temperature in the solid state during solution treatment, homogenization, or recrystallization [33].

Rare earth elements can effectively refine the structure in Al–based alloys, such as Lanthanum. Consequently, the structural modification by adding RE is one of the most effective methods for enhancing strength and ductility of the Al alloys [34].

XRD analyses were carried out to identify the modification of precipitations with La additions after adding 0.5wt.%La element to Al-5.0wt.%Mg alloy. As can be observed Al_2La and $Al_{11}La_3$ precipitates have been formed after adding 0.5wt.%La.

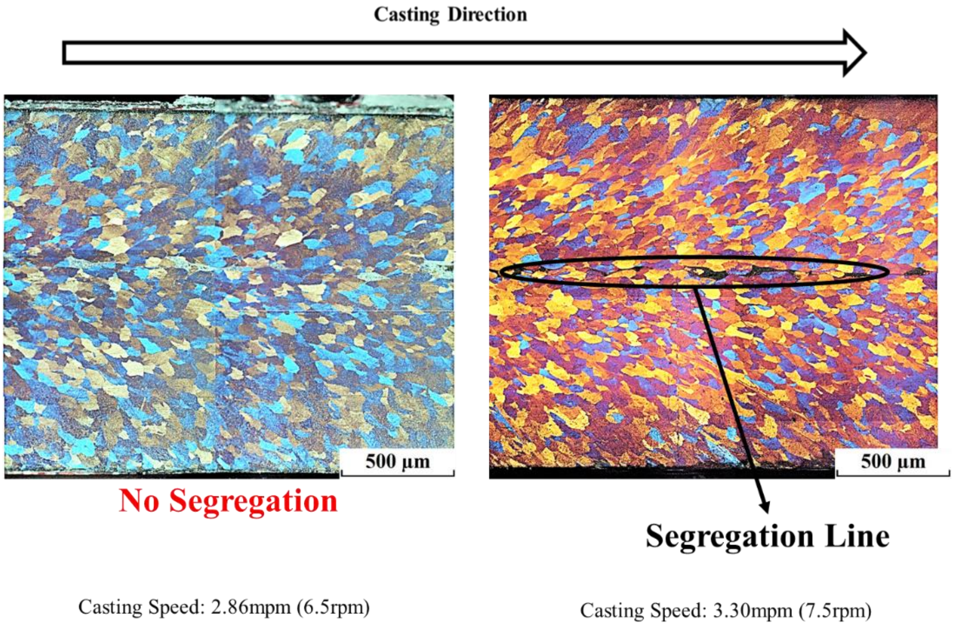


Figure 4.7: Optical Electron Microstructure Image of As-cast TRC Al-5.0wt.%Mg-0.5wt.%La Alloys: (a) 2.86mpm, (b) 3.30mpm

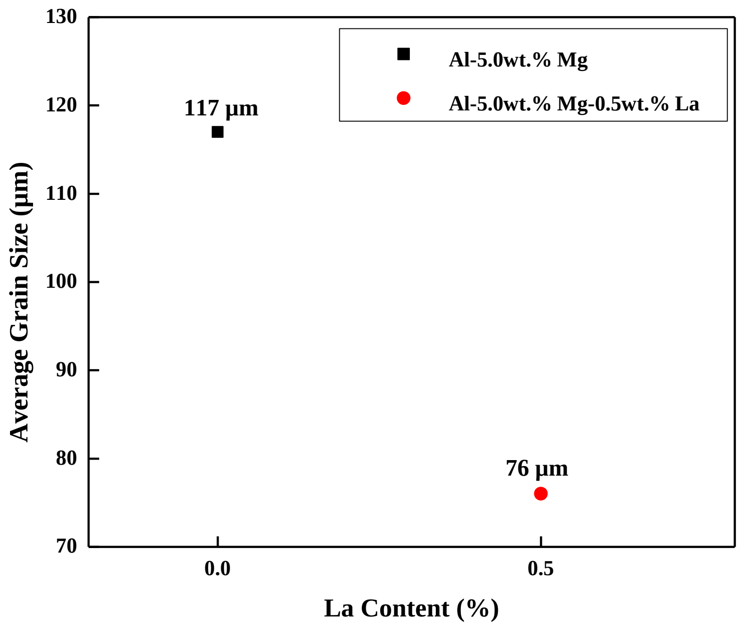


Figure 4.8: Al-5.0wt.%Mg and Al-5.0wt.%Mg-0.5wt.%La alloys Average Grain Size Analysis for As-cast Samples.

Composition	Casting Speed	Average Grain Size
Al-5.0wt.%Mg	6.5rpm (2.86mpm)	117μm
Al-5.0wt.%Mg-0.5wt.%La		76μm

Table 4.4: Al-5.0wt.%Mg and Al-5.0wt.%Mg-0.5wt.%La alloys Average Grain Size Analysis for As-cast Samples.

It is believed that the large grain size of alloys is not preferred in industries; because it reduces the strength of the material. Various techniques have been introduced to reduce the grain size of aluminum alloys such as, adding grain refiners, cold rolling followed by recrystallization.

Grain refinement is a technique used to improve the mechanical properties of the materials by decreasing their grain size which is called as inoculation [43]. With smaller grain, there is smaller area for each dislocation. There is a much greater chance for dislocation to be stopped at a grain boundary with smaller grain. Therefore, the smaller grain is stronger [44].

It was already reported by scientists that low-temperature, permanent deformation of metal comes from the movement of crystalline imperfections, known as dislocations, through the grains in the metal.

With larger grain, a dislocation can move without being stopped by a grain boundary. This type of strengthening is known as Hall-Petch strengthening.

Grain-boundary strengthening (or Hall–Petch strengthening) is a method of strengthening materials by changing their average crystallite (grain) size. It is based on the observation that grain boundaries are insurmountable borders for dislocations and that the number of dislocations within a grain have an effect on how stress builds up in the adjacent grain, which will eventually activate dislocation sources and thus enabling deformation in the neighbouring grain, too[54]. So, by changing grain size one can influence the number of dislocations accumulated at the grain boundary and yield strength. For example, heat treatment after plastic deformation and changing the rate of solidification are ways to alter grain size [54].

$$\sigma_y = \sigma_o + kd^{-\frac{1}{2}}$$

$$or \quad \sigma_y = \sigma_o + \frac{k}{\sqrt{d}}$$

d : diameter of grain,
σ_y : yield strength,
σ_o and k are constant

Figure 4.9: Hall-Petch Equation

The first nucleation (or formation) of solid aluminum will be at the surface of the aluminide particle. The aluminum crystal then grows around the surface of the aluminide. In the process it consumes the dissolved La in the vicinity of the particle, and growth stops. As the metal cools further, dendritic growth begins and continues as solidification proceeds [35].

La element is usually used to produce a finer grain. La has great ability to reduce the tendency of hot tearing and cracking, as well as reduce the segregation and porosity, improve feeding, and improve the surface finish and mechanical properties of both shape castings and direct-chill castings. In recent decades, great efforts have been made to study the grain refinement mechanism and the performance of different grain refiners [21].

La dissolves in Al and that cause resistance to hot cracking. It is so important to produce refined grain for any kind of alloy, to prevent hot cracking during twin-roll casting. After adding La grain sizes get smaller (or finer, which is the basis for the term 'refinement'). Grain refinement changes nucleation and growth behavior of solid Al grains. For this reason, mechanical properties are isotropic and the material is stronger.

A decrease in the size of fine precipitates is a leading indications of grain refinement and improvement of the superplastic properties [51]. That kind of structures uniformly distributes coarse and fine particles in the aluminum matrix [51].

As shown from Figure 4.8 there is no center segregation line in Al-5.0wt.%Mg-0.5wt.%La alloy with the 6.5rpm casting speed. This result is not common in the TRC field. Most of the research is focused primarily on minimizing the segregation line area, not totally removing it. This result proves that removing the segregation line is possible by designing new alloy systems. However with relatively high speed casting (7.5rpm), a segregation line is barely observed. In order to optimize all the casting parameters with a decent alloy system, casting speed is a critical thing. It should also be noted that Solid-state solubility of Lanthanum in Aluminum is very low [23].

In addition, much smaller grains are observed in the alloy with 0.5 wt.% La compared to Al-5.0wt.%Mg alloy, which is shown in figure 4.9 and table 4.4. That observation shows that Lanthanum has the effect of acting as a great grain refiner.

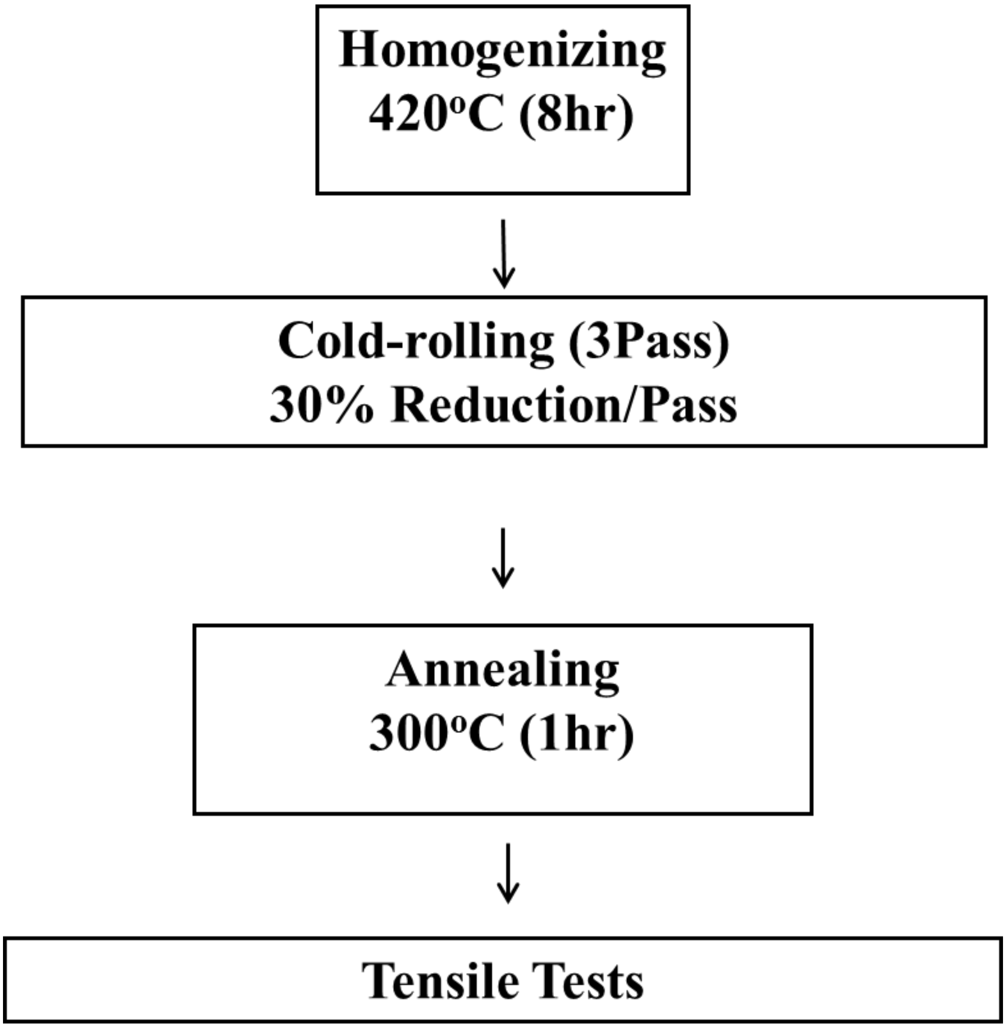


Figure 4.10: Al-5.0wt.%Mg and Al-5.0wt.%Mg-0.5wt.%La Alloys Homogenizing and Cold-rolling Process Chart.

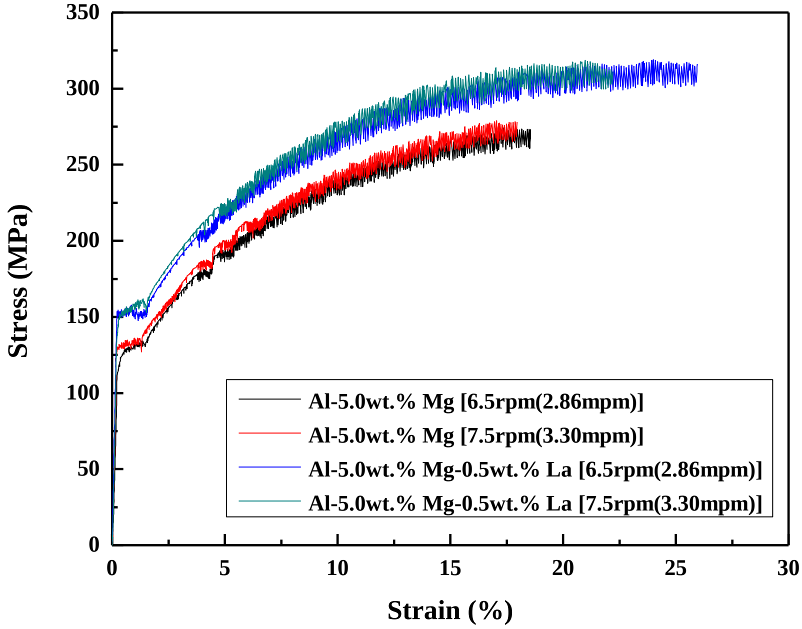


Figure 4.11: Al-5.0wt.%Mg and Al-5.0wt.%Mg-0.5wt.%La alloys Tensile Test Graph

Composition	Casting Speed	Y.S. (MPa)	U.T.S. (MPa)	Elongation (%)
Al-5.0wt%Mg	6.5 rpm (2.86mpm)	111.46	260.49	18.52
	7.5 rpm (3.30mpm)	126.59	278.12	17.96
Al-5.0wt%Mg-0.5wt.%La	6.5 rpm (2.86mpm)	149.01	318.21	25.96
	7.5 rpm (3.30mpm)	153.49	317.39	20.83

Table 4.5: Al-5.0wt.%Mg and Al-5.0wt.%Mg-0.5wt.%La alloys Tensile Test Results

Firstly samples have been homogenized at 420^0c for 8 hours and then cold-rolled up to 1mm thickness then annealed at 300^0c for 1 hours. After those processes, Tensile Tests have been carried out for all samples.

One of the major challenges for the twin-roll casting production route is to improve both physical and mechanical properties of the sheet while achieving high productivity. Thin strip casting (1mm thick) has shown the potential for increases in productivity, as well as for the improvement of materials properties, and therefore has attracted great interest.

After the casting process sheets were homogenized at a high temperature. Highly dense clusters and precipitates are formed that are responsible for the increased strength [36].

Non-uniformity of grain size which means segregation in our research, reduces strength and elongation. Uniformity of properties lead to improved mechanical properties by eliminating the center segregation area which can be achieved by grain refinement.

Lanthanum element has been known as one of the grain refiner in Aluminum alloys, accelerating precipitate phase nucleation or sometimes even changing the type of phase that forms.

As can be seen from figure 4.11, there are some serrations in the graph. These serrations are called the Portevin–Le Chatelier (PLC) effect, named after first reported plastic flow serrations [37].

These serrations occurred due to the pinning and un-pinning of the dislocations he solute atoms. At certain specific strain rates and temperatures, a large number of simultaneous un-pinning of the dislocations leads to the localization of the plastic deformation observed in slip bands which have inclined in the direction of straining. These bands travel along the gauge of the strained samples at lower stresses than required for their formation, leading to characteristic serrations on the stress–strain curves [38].

4.4 Conclusion

- Al-5.0wt.%Mg alloys have more center segregation area percentage than commercial alloys.
- Center segregation area of Al-5.0wt.%Mg alloys has been found to be Mg rich.
- Healing effect of homogenizing has been observed on Al-5.0wt.%Mg alloys.
- Al-5.0wt.%Mg-0.5wt.%La alloy system has found to have no center segregation.
- Grain refinement for the Al-5.0wt.%Mg alloy has been observed with the addition of La.
- Al_2La and $Al_{11}La_3$ precipitates have been observed after adding 0.5wt.%La.
- Al-5.0wt.%Mg alloys have been found in similar mechanical properties with commercial alloys.
- Al-5.0wt.%Mg-0.5wt.%La alloys possess enhanced mechanical properties as compared to commercial alloys.

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