EFFECT OF ELECTROMAGNETIC STIRRING
ON GRAIN REFINEMENT OF Al-4.5%Cu ALLOY

by

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ABSTRACT

This study investigated linear electromagnetic stirring (EMS) effects on grain refinement, microstructure, and segregation of cast Al-4.5%Cu alloy. Solidification was carried out in a bottom chilled mold and EM stirring was performed using a four turn induction coil surrounding an adiabatic ceramic matrix crucible using a specially designed bottom chilled plate to control the solidification rate in the mold in order to obtain an equiaxed dendritic structure. In this study, stirring was carried out using a coil current of 200A, a frequency of 4900Hz, and the superheat of the alloy at the beginning of solidification was 26ºC. Solidification was carried out for two stirring conditions as well as an unstirred melt for comparison. Modeling was used to determine the characteristic velocity of the EM stirred melts and was predicted to be in the range of 6.8mm/s to 23.1mm/s.

Metallographic examination conducted on the cast samples showed that the average grain size for the unstirred melt was 2.6mm and around 500μm for the stirred melt. For the velocity range used in this study, the effect of stirring on grain refinement was small. Microscopic examination showed that the EM stirred melt exhibited finer secondary dendrite arm spacing (SDAS) than that for the unstirred melt; with an average spacing of 180μm and 230μm, respectively. Solutal fragmentation was also observed microscopically in all cast ingots. The extent of fragmentation in the stirred melt was much higher than the unstirred melt. The stirred samples also showed a very miniscule amount of mechanical shearing.
Copper segregation of the cast alloy was analyzed at three positions. The unstimred sample had average copper concentrations with little variation along the height of the ingot with an average value of 3.6wt% copper. Stirring was found to have a strong influence on macrosegregation and the variation of copper concentration along the ingot increased with the velocity which is consistent with the increased convective transport of copper into the bulk liquid. For the low velocity case the copper concentration varied from 3.26 to 4.44wt% and 2.83 to 4.51wt% for the high velocity condition.
LIST OF ABBREVIATIONS AND SYMBOLS

B  Magnetic flux density
B_0  Magnetic field strength
CET  Columnar to equiaxed transition
ΔT  Change in temperature
Δt  Change in time
EMS  Electromagnetic stirring
EPMA  Electron probe micro analysis
\(g_l\)  Liquid volume fraction
J  Induced electrical current
κ  Permeability of mushy zone
k  Partitioning coefficient
\(\lambda_2\)  Secondary dendrite arm spacing, distance between tips of arms
mT  Millitesla, magnetic flux density
Ø  Diameter
SDAS  Secondary dendrite arm spacing
SLI  Solid-liquid interface
T  Tesla, magnetic flux density
\(\mu\)  Viscosity
\(\mu_0\)  Vacuum permeability
\(V_c\)  Casting speed, solidification front velocity
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INTRODUCTION

There is considerable interest in stirring molten metal during solidification as a means to refine the grain structure and to modify the microstructure of the cast alloy. The purpose of stirring is the introduction of a convective flow across the solid-liquid interface (SLI) and into the mushy zone during solidification of a metal. This convective flow induces changes in the thermal and solutal profiles in front of the SLI [5,6]. These changes affect both the morphology of the solidified metal as well as the composition variation throughout the metal when solidified. The morphology of the metal is altered due to solute rich liquid being brought into contact with the dendrites forming and growing across the SLI. This solute rich liquid penetrates into the interdendritic network and causes some branches of the dendrites to constitutionally remelt at their roots and be transported away in the bulk flow of the liquid [5,6,13]. If there is sufficient undercooling within the bulk liquid, these arm fragments can survive without remelting serving as the nuclei for new grains. This leads to a higher nucleation rate producing a much finer, refined grain structure within the casting [5,6,9]. This process is known as fragmentation. Refining grains increases the tensile strength of metals due to the increase in grain boundaries that can hinder dislocation motion. This strengthening mechanism is validated by the Hall-Petch Relationship [19,21]. Figure 1.1 depicts the mechanism of fragmentation with stirring.
Figure 1.1: Secondary Dendrite Arm Fragmentation Process

The surviving fragments and newly formed grains transported within the bulk flow can impact dendrites protruding from the SLI and mechanically shear dendrite arms [6]. Solutal fragmentation, although, appears to be the dominant mechanism causing fragmentation with convective flows. Remelting and fragmentation of the dendrite arms caused by convection also leads to morphological changes in the dendrite arm spacing [7,14,18] and the formation of secondary arms depending on the orientation of the dendrite to the convective flow when compared to solidification with no convection [13].

With the introduction of a convective flow during solidification the solute will be redistributed throughout the casting [10,13]. The redistribution of the solute within the bulk liquid can lead to changes in the segregation of the alloying elements [10,16,17]. The magnitude of the velocity of the convective flow across the SLI appears to be a key factor in the amount of macrosegregation that occurs within a casting [16,17]. Lower velocity convective flows appear to have less influence on the amount of macrosegregation within a casting [20], whereas high velocity flows actually “wash” the solute from the interdendritic network within the mushy zone.
increasing macrosegregation of the solute [6,7] and affecting eutectic formation during solidification [21].

There are two main approaches to inducing a convective flow within the molten metal during solidification. The first method utilizes mechanical impellers within the melt to induce a flow within the bulk liquid. The second technique, which has the advantage of contactless stirring of the melt, is electromagnetic stirring (EMS). There are numerous approaches in utilizing EMS. Rotational stirring uses either permanent magnets on a wheel [13] or multiple magnetic poles surrounding a cylindrical crucible [10,20-22] to induce a rotational flow within the bulk liquid. Another EMS variation utilizes either DC [14,15] or AC [18,19] magnetic pulse fields in order to introduce forces into the melt that cause fragmentation [18,19] or dislodge heterogeneously formed nuclei from mold walls to serve as seeds for additional grains [15]. A good review of recent advances in electromagnetic stirring during solidification is available in the 2009 and 2012 Electromagnetic Stirring Symposium.

The most common form of EMS is linear stirring which involves an AC induction coil surrounding a crucible with current flowing at a specified frequency [1-5,9]. Linear stirring creates a torroidal flow within a cylindrical melt forcing bulk liquid parallel along the cylinder walls, turning to run parallel across the SLI, and then turning again along the center axis of the cylinder. The purpose of this study is to investigate the effect of the velocity in linear EM stirring on grain refinement of Al-4.5wt%Cu, with emphasis on the grain size, secondary arm spacing, and segregation.
2. LITERATURE REVIEW

The primary effect of stirring molten metal during solidification is to generate a refined grain structure. The primary factor in creating a fine macrostructure within castings is fragmentation. Fragments can be created through solutal remelting of dendrite arm roots and to a lesser extent through mechanical shearing. Fragmentation of the dendrite arms will affect the dendrite arm spacing. Fragmentation is predominantly a solutal driven phenomenon. In the case of forced convection where solutal transport is affected, the overall fragmentation and segregation within a casting can be altered through various EMS processing techniques, but is highly dependent on the characteristic velocity of the flow across the solidification front [17].

2.1 Grain Fragmentation

Fragmentation is critical in creating a fine grained macrostructure within a casting [4-6,9,11-13]. The process of fragmentation is not dependent on having a convective flow [11,12], but is further enhanced by introducing a forced flow across the solidification front [5,6,13]. Fragmentation will alter the dendrite arm spacing, both primary and secondary. In directional solidification situations with no flow conditions the dendrite arm spacing generally increases away from the chill, whereas with the introduction of a convective flow the dendrite arm spacing will decrease further away from the chill. Due to fragmentation being a solutal driven process, the redistribution of solute or segregation during solidification is also related to the amount of fragmentation observed within a system.
2.1.1 Fragmentation Mechanism without Forced Convection

The occurrence of fragmentation during solidification is dependent on a number of variables. Thermal gradients, localized solutal concentration gradients, solidification front velocity, and cooling rate are all variables that affect the amount of fragmentation during solidification [11,12].

The work by Yasuda et al. [11] examined the directional solidification of both Sn-Bi and Al-Cu alloys in-situ using X-ray imaging. Results show that the Sn-Bi alloy had an increased growth rate when the planar solidification front collapsed after perturbations formed, but then a deceleration of the growth rate occurred due to solute and thermal build up in front of the liquidus. This deceleration of growth rate contributed to the fragmentation of dendrite arms. The results pertaining to the Al-15%Cu system were analog to the Sn-Bi system where after the nucleation of grains, and dendrites were formed, a deceleration of the growth rate was observed due to latent heat and solute rejection ahead of the interface. Fully developed secondary arm fragmentation was caused by localized solute transport with a domain of less than 100μm. The authors also stated that with forced convection the domain of solutal transport will be greatly increased promoting fragmentation. They also noted that the main factors influencing fragmentation were reduction of both solidification and cooling rates.

In another study by Li et al. [12] the in-situ solidification of Sn-Bi alloy using controlled cooling rates was conducted. The results showed that using very low cooling rates (8.3x10^{-3}K/s) into the freeze range with a negligible temperature gradient caused remelting of dendrite arms. When the sample was held isothermally at 484K, the dendrite arms coarsened with solidification occurring at the roots of the dendrite arms toward the tips. They also found that a higher cooling rate (3.3x10^{-2}K/s) with a thermal gradient of 1.3K/mm generated fragmentation. The study
concluded that fragmentation occurs in the presence of a finite thermal gradient and that the concentration gradients with diffusive solute transport are key variables.

2.1.2 Fragmentation Mechanism with Forced Convection

Forced convective flow is suspected in influencing the process of fragmentation during solidification. Studies performed using in-situ observations show that forced convection may play a role in fragmentation pertaining to certain alloys [6,13].

Results by Paradies et al. [6] in experiments using a succinonitrile-acetone mixture in a closed loop flow system revealed a strong correlation between the rate of fragmentation and the velocity of the liquid directly adjacent to the mushy zone. The cooling rates (0.1-0.6K/min), flow rates (1.8-11.9cm/s), and thermal gradients (8-18K) were all varied in the course of the experiments. The authors suggested that increasing the velocity of the flow also increased the fragmentation rate within the mushy region and that a deeper mushy zone promoted more fragmentation. The report surmised that bulk fluid velocity was the main factor affecting fragmentation regardless of thermal gradient or cooling rate.

Boden et al. [13] investigated grain fragmentation of a Ga-25wt%In alloy in both the presence and absence of a bulk flow and the results for the two cases were compared. In the no flow scenario, the evidence showed that dendritic growth was dependent on solutal convection. In the forced flow scenario, the solutal boundary layer and redistribution of solute affected the fragmentation and growth of dendrites. Fragmentation was not observed in the upper regions of the mushy zone, but was present in the deeper regions of the mushy zone. The fragments settled to the bottom of the mushy zone and were not transported into the bulk flow across the SLI. Dendrites and their arms preferred to grow in the direction of incoming solute poor liquid
(upstream) and secondary arms were inhibited from growing in the solute rich regions (downstream).

Campanella et al. [5] developed a criterion for the fragmentation of dendrite arms based on their experiments using a Bridgman type directional solidification system and solidification rate of 4mm/min. Two different copper alloys (C97 1%Ni-1%Pb-0.2%P and BZ4 4%Zn-4%Sn-4%Pb), two inductive power levels, and two distances between the inductor and the solidification front were trialed. A computer model was also developed in parallel to compare results from experimental findings. Permeability and velocity of the fluid in the mushy zone influenced fragmentation. Fragmentation was found only in the deeper regions of the mushy zone at a distance of approximately $8\lambda_2$ (SDAS) or greater. In summary fragmentation was promoted by a smaller distance between the inductor and the liquidus (higher velocity), higher strength of the magnetic field, low solidification rate, and high permeability of the alloy. Campanella et al. [5] concluded that the fluid velocity along the thermal gradient must exceed the solidification rate for fragmentation to occur. The fragmentation criterion is:

$$C_R \approx \frac{1}{V_c} \frac{\kappa}{\mu} \frac{B_0^2}{\mu_0 d_{ind}} > 1$$

Where $V_c$ is the solidification rate, $\mu$ is the dynamic viscosity, $\kappa$ is the permeability of the mushy zone, $g_l$ is the volume fraction of liquid, $B_0$ is the magnetic field strength, $\mu_0$ is vacuum permeability, and $d_{ind}$ is the distance between the liquidus front and the inductor.

2.1.3 Effect of Stirring on Dendrite Arm Spacing

Generally in the directional solidification process without stirring the dendrite spacing, particularly the secondary dendrite arm spacing (SDAS), increases as a function of the distance from the chill plate [7,12]. With the introduction of convective flow into a directional
solidification system, the dendrite arm spacing usually decreases with distance from the chill [7,14].

The research by Turchin et al.[7] compared dendrite arm spacing of an Al-4.5%Cu alloy during directional solidification under no flow and constant flow situations. The no flow condition generated dendritic arm spacing between 15μm at 2mm from the chill and increasing to approximately 25μm at 10mm from the chill surface. Dendrite arm spacing with convective flow was reported for numerous bulk flow velocities. Lower velocity (0.05m/s and 0.10m/s) experiments showed increasing dendrite arm spacing, 48μm and 65μm respectively, at 2mm from the chill decreasing to 25μm and 41μm at a position 10mm from the chill. Higher velocity (0.30m/s and 0.35m/s) experiments actually formed finer initial dendrite spacing as a function of the velocity starting at 40μm and 35μm 2mm from the chill and decreasing to 20μm and 18μm respectively, 10mm from the chill. In all cases with forced flow the dendrite arm spacing was larger at the start of the solidification, when compared to the no flow condition, and decreased further away from the chill. Higher velocity flows reduced the coarsening effect near the chill.

Zhao et al. [14] conducted an experiment applying a DC pulse magnetic field to directionally solidified Al-7Si alloy. Primary and secondary arm spacing were affected as a function of the pulsed magnetic field intensities and were directly proportional to the fluid flow velocity at the dendrite tips. Three magnetic intensities were used 0.158, 0.192, and 0.226Tesla as well as a no flow condition. Both primary and secondary dendrite arm spacing were reduced as a function of the velocity. With no flow conditions the primary arm spacing was around 330μm and dropped to approximately 260μm at the highest magnetic intensity. Secondary dendrite arm spacing started at 53μm with no flow conditions and decreased to 34μm at the highest magnetic pulse intensity (highest velocity).
2.1.4 Segregation of Alloying Elements

The introduction of a forced convective flow across the solidification front affects the distribution of solute by interfering with the normal solutal boundary layers that form around dendrites during solidification with no flow conditions [13]. The redistribution of solute in the bulk fluid due to convection during solidification can cause variation of the solute concentration in the solid state [10,16,17,20,21,23]. In certain cases the studies revealed that introduction of convection increased the amount of segregation [10,16,21,23], and in other cases the forced flow reduced segregation [16,20].

In a study by Roósz et al.[10] rotating EM fields were applied to a unidirectionally solidified eutectic Al-7.5wt%Ni alloy and compared to a sample solidified with no stirring. Solidification rate (0.1mm/s) and thermal gradient (8K/mm) were kept constant. The sample without stirring had very little segregation. The samples processed with stirring exhibited strong macrosegregation of nickel in both the radial and axial directions. Jardy et al.[16] created a model to estimate segregation in arc remelted Ti and Zr alloy ingots. The model is based on linear stirring with reversal of the current for differing time periods. The model predicted that titanium processed with high magnetic field strengths and long reversal times created the deepest melt pool depth, highest fluid velocity, and turbulence leading to strong segregation. Continuous stirring of the zirconium alloy also demonstrated strong segregation along the centerline, whereas alternating stirring actually generated a uniform distribution of solute.

A comparison between pulsed and continuous rotating magnetic fields was performed by Råbiger et al.[21] using an Al-7wt%Si alloy in a unidirectional solidification process. Three levels of magnetic field strength (1.6mT, 3.2mT, 9.2mT) were utilized as well as an experiment using no EMS. The pulsed field used only the highest field strength of 9.2mT. The results
showed that the amount of segregation was directly proportional to the magnetic field strength in the case of continuous stirring. Area fractions of the eutectic were used to determine the segregation. The sample with no stirring had an eutectic area fraction range of 39-44\% compared with 34-48\% for the 9.2mT continuously stirred sample. The sample exposed to the pulsed rotating field had a 37.5-47\% range indicating that pulsed fields have less influence on segregation when compared to continuous fields.

A model for the purification of industrial grade aluminum in a continuous cast process was presented by Fashu et al. [23]. This model is based upon inserting a SiC mechanical stirrer directly in front of the solid-liquid interface to promote convection. Casting speed was held constant at 2mm/min. The model utilized empirical data regarding the SiC stirring speeds defining 10rpm to be optimal. The results predicted an ability to increase the purity of the aluminum from 99.77\% to 99.925\% through efficient segregation of the impurities due to convection.

Jiang et al. [20] presented a study where rotational magnetic stirring of Incoloy 800H actually reduced the segregation of Fe, Cr, and Ni within an ingot. A frequency of 8Hz and 250A current was used for the stirring parameter. The results concluded that the fragmentation caused by the stirring convection and the transport of the fragments toward the center of the ingot reduced the overall segregation.

A model to relate velocity, turbulence, wall friction, and solidification rate to predicted segregation is presented by Delannoy et al.[17]. The authors introduce a convecto-diffusive parameter based on the mass fraction at the wall with a value between 0 and 1. The results indicated that a minimum required flow velocity is required to promote segregation and below
this minimum velocity the segregation is negligible due to solute transport being diffusion driven rather than convection driven.

2.2 Grain Refinement using EMS Systems

Various methods of inducing a convective flow within molten metal using electromagnetic fields are currently being used. The most common form of EMS is linear stirring utilizing an AC coil surrounding the melt and inducing a current through the coil. Another popular form of EMS uses rotating magnetic fields created by surrounding the melt with magnetic poles then applying current through the poles to generate a spinning magnetic field, generally perpendicular to the solidification direction. The last technique that is widely used for EMS is the pulsed magnetic field. Pulsed magnetic fields may be either AC or DC based and are used often in rheocasting [31] or thixoforming [32] operations.

2.2.1 Linear Stirring

EM linear stirring is generated by coil surrounding a crucible creating a time varying EM field. A general configuration for linear EMS is presented in Figure 2.1 below.

![Figure 2.1: Flow circulation pattern generated by linear electromagnetic stirring.](image)
In directional solidification linear stirring will create an axial recirculating flow with almost constant velocity parallel to the SLI. The application of the electromagnetic field not only induces a convective flow, but also generates Joule heating in the sample [5].

The recirculating flow pattern and the intensity of the flow depend on the inductor position with respect to the liquidus, current applied to the coil, and the frequency. The flow intensity along with the alloy solutal concentration can change the macro and microstructure of the alloy being solidified [5,9].

Campanella et al. [5] found that low concentration copper alloys (C97 < 3wt% alloying elements) were more difficult to grain refine when compared to the higher concentration alloys (BZ4 12wt% alloying elements). This was due to the higher permeability of the mushy zone in the higher concentration alloy allowing solute rich bulk fluid to penetrate further into the interdendritic network promoting fragmentation. They discovered that both no flow and flow generated by a 2kW power supply produced columnar grain structures. With the coil at higher power (6kW), but maintaining a 23mm distance from the liquidus, the higher concentration alloy formed an equiaxed dendritic structure while the lower solute alloy retained a columnar structure. Reducing the inductor to liquidus distance to 13mm and maintaining high power stirring (overall higher bulk fluid velocity) produced equiaxed structures in both the high and low concentration alloys.

Paes et al.[9] used linear stirring to modify Al-4.5%Cu microstructure for rheocasting applications. Rheocasting operations are conducted inside the freeze range of an alloy below the liquidus temperature (no superheat). A comparison of samples with and without stirring was presented. The samples undergoing stirring were subjected to a current reversion time of 2 seconds. The as cast samples showed typical dendritic structure, but stirred samples evolved a
globular structure preferable for rheocasting. The study found that EMS was more effective in producing the globular structure than inoculants.

2.2.2 Rotational EMS

Rotational EMS uses a multi-pole AC induction coil that generates a spinning magnetic field when current is applied. This spinning magnetic field will induce an opposite spinning flow on a molten metal with the highest tangential velocity at the furthest radius reducing to zero tangential velocity at the center. A schematic of a rotating EMS system is shown in Figure 2.2.

![Figure 2.2: Flow pattern generated by rotational EMS](image)

The effects of rotational stirring has an effect on both grain refinement and segregation according to various studies [10,20,21,22]. Roósz et al. [10] reported that significant changes to the macrostructure of eutectic Al-7.5wt%Ni were observed in stirred samples with cellular boundaries becoming curved when compared to solidification with no stirring. Strong segregation was observed in both radial and axial directions in all stirred samples using magnetic field strengths of 10 and 50mT under constant growth rate (0.1mm/s) and thermal gradient (8K/mm) conditions.

Jiang et al.[20] found that applying a rotating magnetic field using 250A current and 8Hz frequency to Incoloy 800H during solidification reduced the amount of segregation of Fe, Ni,
and Cr in the solidified ingot. Fragmented dendrites were observed travelling to the center of the ingot aiding in the redistribution of solute thereby reducing segregation.

Räbiger et al.[21] indicated that applying both continuous and pulsed magnetic fields to Al-7wt%Si alloy refined grain structure, increased segregation range, and improved tensile strength. The study also stated that solute was depleted at the outer walls and solute enrichment occurred along the center axis, supporting Jiang et al.[20] findings that transport occurs from the outer radius toward the center in rotating systems.

Qu et al.[22] demonstrated that when a rotating magnetic field was applied to Cu-2wt%Fe and Cu-8wt%Fe samples at different field strengths of 23, 45, and 65mT at varying frequencies of 4, 10, and 16Hz that grain refinement was more readily achieved with the low Fe content alloy compared to the high Fe content alloy. Another benefit was that large Fe-rich particles that form in solidification with no stirring, due to a liquid miscibility gap, were greatly reduced with stirring relative to the intensity. The authors also concluded that inductor current had more influence on refinement than the frequency.

2.2.3 Pulsed Magnetic Field

Current research in the area of applied pulsed magnetic fields is also being conducted for the purposes of grain refinement and segregation control [14,15,18,19]. In the case of DC pulse stirring, the Lorentz forces create a large pressure gradient in the melt initiating a flow in the bulk liquid [14]. The Lorentz forces act perpendicular to the axial direction initiating flow in the radial direction. The magnitude of the Lorentz force is defined as $F=J \times B$ where $F$ is the Lorentz force, $B$ is the magnetic flux density and $J$ is the induced current. A sketch illustrating a configuration for DC pulse stirring is shown in Figure 2.3.
The application of pulsed DC stirring for grain refining pure aluminum in an omnidirectional solidification situation was discussed by Fu et al.[15]. Under these conditions the application of a pulsed DC magnetic field caused the dislodgment of formed nuclei from the mold walls. These nuclei having higher density fell to the bottom of the mold and served as seeds for grains. Increasing grain refinement was observed with increasing levels of current (25, 100, 250A) applied to the coils at the bottom of the mold. The area toward the top of the mold was grain refined compared to samples without a pulsed magnetic field applied, but average grain size at the top of the mold remained constant for all three magnetic field intensities.

In a case using unidirectional solidification Zhao et al.[14] found that the depth of the mushy zone was increased from 4.4mm without pulse stirring to 8.4-8.8mm with DC pulse fields applied. Cooling rate with DC pulse stirring was reduced from 1.44K/s to 0.73K/s and temperature gradient ahead of the SLI was reduced from 7.8K/mm without pulse stirring to 4.2-4.4K/mm with pulse stirring. Calculated flow velocity at the dendrite tips increased with 0.158T and 0.192T strength magnetic fields to 36.965μm/s and 88.011μm/s respectively, but decreased to 75.471μm/s at the highest field strength of 0.226T.
The study conducted by Cao et al.[18] on AC linear pulse stirring Al-10wt%Cu alloy using 30V and 1000Hz pulsed field shows that Joule heating rather than solute convection is the primary cause of remelting dendrite arms. Thermal gradients were held constant at 1.5K/mm and two different cooling rates were used for the experiments. Considerable deceleration of the growth rate (solidification rate) was seen when magnetic pulse stirring was applied until the effects of continuous cooling overcame the Joule heating and the growth rate accelerated once again until impingement of grains occurred. No fragmentation or grain refinement was observed in the solidified samples.

In a study by Fu et al.[19] a low voltage pulsed magnetic field was applied to AZ80 magnesium alloy. A potential of 100V and current frequency of 5Hz was used for the course of the experiments. Results indicated that the pulsed magnetic field both shortened the length and increased the radius of dendrite arms due to Joule heating at the dendrite tips. Thin pointed dendrites that formed in solidification without magnetic pulse stirring were transformed into a rosette morphology approaching a globular structure. Strong grain refinement with uniform distribution of grains across the ingot was attributed to the pulse stirring. The only drawback was the formation of continuous β-phase precipitates around the primary α-phase magnesium grains, that led to a slight reduction in elongation. The grain refinement and morphology modification increased both yield strength (78MPa to 109MPa) and ultimate tensile strength (155MPa to 172MPa) when compared to samples tested with no pulse stirring.

2.3 Flow Intensity in Stirred Solidification Systems

Quantifying the bulk fluid velocity within a system solidifying under forced convection still presents challenges for researchers. Placing instrumentation within flows can lead to physical interference with the flows, thereby precluding the accuracy of the data being obtained.
Due to the constraints of physically measuring velocity and turbulence within a melt, many researchers are developing advanced numerical computer simulations to model the velocity fields within systems having forced convection applied during solidification. [5,16,17,24].

2.3.1 Experimental Measurement of the Velocity

Determining the velocity within the bulk fluid during solidification with forced flow is critical to understanding the convective effects on fragmentation, growth rates, thermal gradients, and segregation. A closed loop system with constant volumetric flow and known cross-sectional areas using acetone-succinonitrile (SCN) being unidirectionally solidified in a transparent test chamber was used by Paradies et al.[6] to study the effect of velocity on fragmentation. Velocities were varied from 1.8 to 11.9 mm/s, cooling rates ranged from 0.1 to 0.6 K/min, and the temperature difference between the melt and chill was adjusted between 8 and 18 K. The solidification in the transparent test chamber was recorded by a high resolution video camera. Fragment velocity in the bulk fluid was determined from the video evidence. Results indicate that as the velocity of the bulk flow near the mushy zone was increased, the rate of fragmentation also increased. Also the particle or fragment velocity within the bulk fluid was substantially less than the bulk fluid velocity. The study also noted that no fragmentation occurred with low concentration alloys (0.3 wt% acetone).

In two studies by Turchin et al.[7,8], an open system using a magnetic pump was used to flow Al-4.5%Cu through a tube of known dimensions across a chill plate. Again, velocity calculations were possible due to the known mass flow rate out of the system and the cross-sectional area of the tube. In the first study [7], four different velocities 0.05, 0.10, 0.30, and 0.35 m/s were used in conjunction with three different cooling rates, and two superheat conditions (700°C and 740°C). Dendrite arm spacing coarsened during initial solidification for
the two low velocity regimes, and became finer at the two higher velocity regimes. Low superheat (700°C) at high velocity (0.30m/s) with maximum cooling rate generated a coarse equiaxed to columnar structure. As cooling rate was decreased, the structure changed to a fine equiaxed structure near the chill and a coarse columnar structure away from the chill. At low velocity (0.10m/s) and low superheat (700°C) the structure was primarily equiaxed, but at high superheat (740°C) the structure became columnar (or feathery per the authors’ description). The second study [8] investigated the formation of feathery grains using the same apparatus as [7], but with a wider velocity range between 0.03 to 0.5m/s. Thermocouples placed in the solidification chamber provided temperature data for determining solidification rate and thermal gradients. The results indicated that low velocity (0.03m/s) and high superheat led to feathery grain formation with thermal gradients between 8 and 12K/mm and solidification rates between 0.6 and 1.4mm/s. The authors’ concluded that lower thermal gradients create equiaxed structures and higher thermal gradients lead to columnar structures.

The experimental determination of velocity was enabled in experimental set-ups by using constant flow rate systems (either open or closed) with known cross-sectional areas.

2.3.2 Theoretical Prediction of the Flow

The limitations of physically measuring velocity directly within a melt undergoing electromagnetic stirring are many. With the advent of ever more powerful computers, numerical simulations based on 3D transport (continuity, momentum, energy) equations that can account for numerous variables (solid mass fraction, turbulence, viscosity, wall friction) are currently being developed. Two general approaches are utilized in the development of solidification models with convective flow. The first method is the finite element analysis method that utilizes a grid system with nodes where the average values for the interface flux of temperature or mass
between the nodes are calculated using Euler or Taylor series methods and carried into the next iteration as the new value for the node of interest on the basis of time stepping. The second method is a control volume approach that solves discretized thermal, mass, momentum, species equations for a defined finite control volume while maintaining complete volumetric conservation. One of the first control volume approaches for solidification was developed by Patankar [30].

A 2D finite element method (FEM) model using CALCOSOFT-2D software was used by Campanella et al. [5] to determine characteristic velocity in the bulk fluid, interdendritic liquid velocity in the mushy zone, and secondary dendrite arm spacing ($\lambda_2$). The maximum bulk fluid velocity of 27mm/s was calculated by the model. Plots comparing the fragmentation criterion, depth of the mushy zone, interdendritic fluid velocity, and SDAS showing boundaries between “no remelting”, “remelting”, and “fragmentation” were generated by the model. The model did not account for the effect of turbulence.

A model based on the finite volume method was developed by Jardy et al. [16] to ascertain the amount of macrosegregation to be expected with vacuum arc remelted (VAR) Ti and Zr alloys subjected to rotational EMS. The macroscopic portion of the model accounted for solutal and thermal transport in the convective flow generated by thermosolutal buoyancy and 3D forced convection. Permeability and turbulence factors were utilized. Phase change was determined at the microscopic level by the lever rule or grain growth with limited diffusion. The model predicted that thermal solutal buoyancy only affects segregation at the center of the ingot and convective forces dominate the segregation at the outer radius of the ingot.

Delannoy and Zaidat [17] created a general model using FLUENT software to account for wall friction within turbulent domains that determined the minimum stirring intensity
required for “efficient” segregation. The model was constructed in three stages starting with no forced convection where diffusive mass transport was dominant. The next stage included laminar convective flow and the third stage accounted for turbulent flow. The turbulence model was modified with wall damping functions. The model produced plots with defined boundaries between diffusive and convective mass transport regimes and allowed comparison of friction velocity and solidification rate with respect to bulk velocity as well as the ratio of stirring speed to the solidification rate and Reynolds number. This model presented boundary conditions indicating the minimum stirring velocity (intensity) required to promote solutal segregation.

A model was developed by Poole and El-Kaddah [24] to calculate velocities in the liquid region and mushy zone for a unidirectional solidification situation. The mushy region was divided into two domains. The suspended particle region at the top of the mushy zone was the first domain, and deep into the mushy zone where particles were fixed was the second domain. Turbulence was accounted for within the liquid and mushy regions. In the suspended particle region, terms for damping of the velocity with respect to solid fraction and packing factors were used. In the fixed particle domain, the strong damping eliminated turbulence and created a laminar flow situation. Using Al-4.5wt%Cu alloy, a three turn coil, and 200A current at an applied frequency of 1kHz as parameters, the model predicted a characteristic velocity of 20mm/s in the liquid. When the ingot was completely mushy, turbulent flow was no longer predicted. Characteristic velocities were shown to be much higher in the suspended particle region compared to the fixed particle region. The characteristic velocity continued to decrease as solidification proceeded.
3. EXPERIMENTAL TECHNIQUE

3.1 Experimental Setup and Apparatus

Figure 3.1 shows a sketch of the experimental apparatus. It is comprised of a bottom chilled crucible with a specially designed air and water cooling system for melting and solidification of the alloy and an induction coil connected to a high frequency power supply for melting and stirring the alloy during solidification. The crucible was fitted with a battery of thermocouples to measure the temperature of the charge at various locations along the centerline axis of the crucible during melting and solidification. Temperature data were recorded using a data acquisition system. A detailed description of each component is given below.

![Figure 3.1: Schematic of processing system configuration](image-url)
3.1.1 Bottom Chilled Crucible

The bottom chilled crucible is in essence a refractory tube placed upon a stainless steel chill plate.

The refractory tube used in this study was reinforced silica matrix composite RSLE-501 manufactured by ZIRCAR Refractory Composites, Inc. This material has low thermal expansion, high resistance to thermal shock, and low thermal conductivity. The nominal inner diameter (ID) of the crucible was 76.2mm (3”) with a nominal outer diameter (OD) of 88.9mm (3.5”). The refractory tube was placed onto an embossment on the chill plate for support.

The lid for the crucible was constructed from 25.4mm thick high temperature fiber board cut to an outer diameter of 88.9mm. The bottom of the lid was tapered to fit into the inner diameter of the crucible. The tapered surface protruded 15.875mm (0.625”) into the tube. The remaining 9.525mm (0.375”) of the height rested on the tube’s upper edge. The lid was split into two halves each with a relief for the thermocouple pack. This allowed charging the crucible with the thermocouple pack in the containment volume.

The chill plate was designed utilizing a flow through concept that directed coolant to the chill. The chill plate was machined from a non-magnetic AISI type 304 austenitic stainless steel to avoid any Joule heating of the chill plate by the induction coil. The chill plate used two concentric cylinders placed on top of one another with the smaller cylinder on top. The 73.66mm (2.900”) OD of the small cylinder fit inside the 76.2mm (3.000”) nominal ID of the crucible tube and allowed for diametrical deviations. Heat transfer from the charge occurred through the top surface of the small upper cylinder.

The insides of both cylinders were machined hollow and a specially designed baffle was installed. This baffle effectively directed the coolant from the lower cylinder and channeled it
directly beneath the top of the small cylinder through a 12.70mm (0.500") gap to promote even, consistent heat transfer. 308L welding rods were used to attach the baffle and cylinders to one another. The lower cylinder had two holes bored and tapped at locations 180° apart for installation of the coolant intake and exhaust lines.

The cooling fluids used in this study were air and water. Air was used during melting and homogenization while water was used for quenching the molten alloy. The coolants entered the chill plate through a 9.525mm (0.375") OD copper intake tube. A tee placed at the other end of the intake tube with a valve placed on each side of the “T” allowed the switching of the coolant fluid. One valve controlled the flow of compressed air and the other valve controlled the flow of water. The exhaust pipe consisted of a 200cm (78.740") length of 9.525mm (0.375") OD copper tubing which terminated inside an EPDM hose with 14.86mm (0.585") OD and 9.525mm (0.375") nominal ID. A compression screw clamp attached the EPDM exhaust hose to the exhaust tube. The EPDM hose allowed the fitting of a type-K thermocouple probe into the exhaust stream to monitor coolant temperature. The exhaust hose exited into a floor drain. The general geometry of the chill plate is shown in Figure 3.2.

3.1.2 Induction Coil and Power Supply

Melting and stirring the charge was carried out using a four turn induction coil. The coil was made of a single 6.3mm (0.250") copper tube with an inner diameter of 4.32mm (0.170").
Figure 3.3 shows the coil dimensions. The coil had a total outer diameter of 136.35mm (5.368”), an inner diameter of 123.65mm (4.868”), and a mean diameter of 130mm (5.125”). The stacking height of the coil was 44.45mm (1.750”) between the bottom of the lowest loop and the top of the highest loop. Centerline to centerline distance between the upper most and the lowest loop was 38.10mm (1.500”) with a 12.7mm (0.500”) centerline to centerline distance between each loop.

![Figure 3.3: Sketch of the induction coil geometry](image)

The coil was connected to an Inductotherm power supply model 35-96 with voltage range between 0-1200 Volts AC, which yields current in the range of 200 to 2400 Amps and a maximum power output of 35 Kilowatts. The frequency range is 5000-7000Hz.

3.1.3 Test Stand

A specially designed test stand was designed to support the coil, to place the crucible inside the coil, and to vary the location of the coil along the crucible during melting and solidification. The stand consisted of an outer frame, horizontal base plate, worm gears, recirculating ball blocks, connecting plate, and a vertical backing plate. The outer frame was built from 25.4mm x 25.4mm (1.000”x1.000”) angle iron with four 60.9cm (24”) legs attached to a square 60.9cm horizontal
frame with a vertical frame measuring 76.2cm (30”) tall and 60.9cm wide, used to mount the vertical backing plate, attached to the rear of the horizontal frame.

The square horizontal base plate made from 12.70mm (0.5”) thick 6061 aluminum plate sat flush on top of the horizontal frame. Two vertical worm gears with 38.1cm separation were attached 20.38mm from the front edge of the base plate. The gears and chain drive for the worm gears were mounted underneath the horizontal base plate. A horizontal 19mm (0.750”) thick top plate connected the top of the worm gears with two additional vertical stabilization tubes connected to the base plate for stability.

A horizontal 12.7mm (0.5”) thick aluminum connecting plate was attached to the recirculating ball blocks mounted on each worm gear. The connecting plate was used to concentrically support the crucible inside the induction coil along the centerline axis. A manual crank on top of one worm gear was used to vary the height of the crucible with respect to the statically fixed induction coil mounted on the vertical backing plate made of 9.525mm (0.375”) thick PMMA acrylic plastic. A picture of the test stand is shown in Figure 3.4.

Figure 3.4: Test stand assembly with chill plate and plumbing installed
3.1.4 Data Acquisition System

Temperature and time were the primary data collected during the experiments. A thermocouple pack consisting of five type-K 20-gauge thermocouples at various heights (10, 20, 40, 60, 75mm) from the chill was utilized to collect temperature data during the experiments. The individual thermocouples were located with respect to one another using a scale and then tied to one another using inert 0.38mm (0.015”) constantan wire. All thermocouples were cut to a length of 228mm (9”) so that equivalent electrical resistance was maintained. A stainless steel clamp was installed near the top of the thermocouple pack to provide additional compression holding the thermocouples together and to locate the height of the thermocouples within the containment volume.

Another type-K thermocouple was placed into the exhaust stream from the chill plate just before exiting into the floor drain. This thermocouple provided in-situ feedback of the exhaust temperature and allowed regulation of the cooling air during the heating and homogenization phases. The exhaust thermocouple also provided a defined point during the change over from the homogenization to solidification processes (switch from air to water for the cooling).

The thermocouples were connected to an Omega Engineering TC-08 8-channel interface synchronized to Omega Data Logging Software. Data collection frequency was set to 1 second intervals. The five thermocouples in the containment volume were connected to the TC-08 interface using equal length 2134mm (84”) Type-K connecting wires to maintain equivalent electrical resistance within the circuits. The exhaust thermocouple was connected using the 915mm (36”) wire that extended from the probe.
3.1.5 System Assembly

The test stand was prepared by lowering the connecting plate with the worm gear crank handle to the lowest position possible. The chilled crucible assembly was inserted through the induction coil and placed into the locator ring on the connecting plate. The crucible assembly was checked to ensure that it was sitting flat on the connecting plate. The crucible was moved upward to a height where the intake and exhaust lines could be connected with the least amount of stress and connected to the chill plate. Water was allowed to flow through the lines to check for leaks at the connections.

The Z-height reference marks on the outside surface of the crucible were used to place the coil’s lower plane 80mm from the chilled surface. A check was made to ensure that the crucible was centered within the induction coil to promote even heating and mixing within the cylinder. The four longest thermocouple wires were bent into an “S” configuration with one of the bends contacting the lid to provide support for the thermocouple pack and keep it aligned with the centerline axis during the experiments. The five thermocouple pack plugs were connected by lead wires to the Omega Engineering TC-08 data acquisition module. The lead wires were kept away from the induction coil to avoid any EM interference. The exhaust thermocouple was also attached to the TC-08.

The Omega Engineering data acquisition software was set-up to collect temperature data from all 6 thermocouples every second for 7200 seconds. The thermocouples were tested by briefly starting the furnace. Water flow through the exhaust was used to check the exhaust thermocouple. Once all thermocouples were validated to be reading accurately, the experiment was ready to start. A picture of the assembled system is shown in Figure 3.5.
3.2 Experimental Procedure

The experiments were conducted in three phases: heating, homogenization, and solidification.

3.2.1 Crucible Preparation

In order to minimize melt contamination from RSLE-501 silica composite crucible, the inner surface was coated using Aerodag G graphite spray supplied by Acheson. The top surface of the chill plate was cleaned with ethanol. Moldable SALI alumina compound was then placed on top surface of the chill plate to serve as an insulating layer. Because of Joule heating of EM stirring, the thickness of this layer was varied to achieve the same cooling rate in both stirred and unstirred melts. The experiments performed without EMS used a 5mm layer, and a 1mm layer was used for the experiments with EMS. The chill plate was then placed in an oven for a
minimum of four hours to ensure all moisture was removed from the moldable SALI alumina. The chill plate with alumina insulating layer is shown in Figure 3.6.

![Figure 3.6: Chill plate with moldable Al₂O₃ insulating layer](image)

With the chill plate removed from the oven, the crucible tube was placed onto the chill plate so the tube rested on lower cylinder embossment. The gap between the inner radius of the crucible and the outer radius of the chill plate was filled with moldable SALI alumina compound to seal the containment volume.

The thermocouple pack was coated with boron nitride spray supplied by ZYP Coatings to allow better wetting and reduce porosity around the thermocouples upon solidification. The thermocouple pack was located by placing one half of the lid on top of the crucible assembly, setting the thermocouple pack into the relief in the lid and allowing the lowest thermocouple tip to contact the alumina chill surface. A 1mm aluminum U-bracket was then placed around the thermocouple pack and the position marked. The thermocouple pack was removed and another mark was placed 10mm lower from the first mark to set the final Z-height of the lowest thermocouple 10mm from the chill surface. The bottom of the stainless steel clamp was aligned
with the lower mark and fastened tight. The thermocouple pack was then ready to be used during charge preparation.

3.2.2 Charge Preparation

The chosen material for the course of this research was a hypoeutectic aluminum-4.5wt% copper alloy with no other alloying elements. Initial attempts to homogenize pure copper pellets or turnings within pure aluminum indicated very long process times. In order to reduce the processing time of the experiments a eutectic 33wt%Cu master alloy was utilized in the experiments due to a low melting temperature (547°C) compared to pure aluminum (660°C). The concept being that the copper contained in the eutectic master alloy would already be in liquid suspension when the pure aluminum started to melt. Once the aluminum melted, the copper would diffuse and mix readily with the aluminum creating a homogenized charge.

The master alloy was produced in a resistance furnace with approximately 4kg of pure aluminum bars (99.9% pure) and 2kg of copper bearings (99.9% pure). The dross was skimmed and the alloy was poured into steel ingot molds. Upon cooling the ingots were removed and sectioned laterally in differing widths. Exposed surfaces were grinded to remove oxides and inclusions. The sections were weighed and cut further to provide 135g of eutectic master alloy for each experimental run.

The crucible assembly was loaded after placing the thermocouple pack into the crucible and resting the lowest thermocouple on the center point of the chilled surface. While maintaining the location of the thermocouple pack along the centerline axis, 865g of aluminum pellets were poured into the crucible to a height between 60 and 70mm. Then 135g worth of master ingot pieces were placed against the wall to maximize the induction heating of the master alloy and promote melting. The rest of the aluminum pellets were then loaded into the crucible.
One half of the lid was installed for use as reference. The thermocouple pack was pulled upwards until the bottom of the stainless steel clamp was even with the top surface of the lid. The other half of the lid was installed. The thermocouple pack was gently pulled upward and the aluminum U-bracket inserted between the lid and the stainless steel clamp. This located the thermocouple pack at the final Z-height position. Figure 3.7 illustrates the chilled crucible assembly prepared for experiments.

Figure 3.7: Diagram of prepared crucible with thermocouple positions shown

3.2.3 Heating and Homogenization Phases

With the crucible loaded on the test stand the furnace was turned on and the data acquisition software initiated to collect temperature data from the thermocouples. The induction
coil current was set to 600A with a frequency range 6650-6750Hz. The air intake valve to the chill plate was opened and flow set to around 4psi on the tank regulator.

The temperatures from the thermocouple pack were monitored along with the exhaust air temperature. The intake valve for the air was adjusted to keep the exhaust temperature between 55-65°C during the heating and homogenization phases. When the temperature reached 660°C (melting point of pure aluminum) at the thermocouple position 20mm from the chill plate surface, the induction coil was lowered to a position 60mm above the chill surface since the charge had fallen lower into the crucible volume. This action took place between 12 and 16 minutes into each run.

Once all the latent heat of fusion was overcome, the entire charge climbed in temperature to the target temperature of 750°C. This temperature was reached between 14 and 18 minutes into each experimental run. Once the target temperature was achieved, the induction coil current was reduced to between 350 and 400A to allow a slow cooling from 750°C to the target solidification start temperature of 675°C. This initiated the homogenization phase of the experiments.

In order to completely homogenize the charge the induction coil was lowered to a position 20mm above the chill surface for approximately 8 minutes to bring maximum velocity, mixing, and heating to the area immediately above the chill surface. The coil was lowered between 22 to 24 minutes into each experiment. The coil was then raised back to 70mm above the chill to mix the upper levels of the charge for approximately 8 minutes. The actual time depended on how quickly the charge was cooling and approaching the 675°C target solidification start temperature. Generally the heating and homogenization processes lasted 37.5 to 39.75 minutes.
3.2.4 Solidification Phase

At the target temperature of 675°C, the furnace was turned off for the “no stir” experiment. The low velocity stirring experiment kept the coil height at 70mm above the chill surface. The coil was moved to 40mm above the chill for the high velocity stirring experiment. The coil current was reduced to 200A with a frequency between 4880-4930Hz in the case of the EMS experiments. The air intake valve was closed and the water intake valve opened to initiate the unidirectional solidification of the charge.

The EMS experiments were allowed to cool to a temperature of 400°C well below the eutectic formation temperature of 542°C before the furnace was shut off. The temperature data were recorded to a temperature of 200°C then stopped. The charge was cooled to below 100°C before shutting the water off. The crucible was then removed from the test stand.

3.3 Metallographic Preparation

After the Al-Cu 4.5% ingot was removed from the crucible, the thermocouple pack was cut off at the entry point on top of the ingot.

The first machining operation removed the upper section of the ingot where oxides and porosity were highly prevalent. A Struers Discotom-5 water-cooled 250mm diameter rotary saw with a 10S25 blade was used to remove 10-15mm from the top of the ingot.

The position of the thermocouple pack in relation to the center axis could then be identified. The next machining operation involved cutting the ingot near the center axis without interfering with the thermocouple pack. The half cylinder exposed a large flat surface representing the center axis and the entire radial direction of the ingot. One half was used for macrostructure analysis and the other cylinder half was further machined for the microstructure and compositional analyses.
The ingot halves for microstructure and compositional analyses were further sectioned into two quarter cylinder wedges. Both wedges were marked with radial locating lines at 20mm, 40mm, and 60mm axial heights from the bottom. Further lines were drawn at 5mm intervals above and below the first lines that created specimens with 10mm height. Lines were drawn 20mm parallel from the centerline axis for the width of the specimens. The faces of the specimens were marked with “I” for inner, “O” for outer, “T” for top, and “B” for bottom to maintain an orientation reference during and after mounting. The inner “I” position represented the edge closest to the centerline axis of the ingot. Depths of the specimens were between 8 to 12mm. See Figure 3.8 below.

![Figure 3.8: Sectioning of the ingot](image)

The six specimens, two with 20mm centerline heights, two with 40mm centerline heights, and two with 60mm centerline heights were then mounted in phenolic epoxy using Leco Corporation black epoxy powder and a Struers LaboPress-3 molding machine. After the specimens were mounted, the backside of each puck was engraved with the “I”, “O”, “T”, and “B” markings to maintain reference to the orientation.

All specimens were then ground using 180, 240, 320, 400, and 500 grit SiC sandpapers on a Struers Rotopol-22 turntable using 300rpm and water cooling. Final polishing was
completed using Struers MD-Mol wool polishing discs with DP-Suspension A 9µm, 3µm, and 1µm diamond paste and OP-S suspension compounds.

3.4 Etching Technique

For the macrostructure analyses of the ingot halves Flick’s Reagent with 90mL DI H₂O, 12mL 37% HCl, and 8mL 49% HF was used. A higher concentration version (38mL DI H₂O, 8mL 37% HCl, and 5mL HF) yielded excellent results on the ingot without EMS, but was unusable on ingots that had been stirred.

The ingot halves were immersed in the Flick’s Reagent and then inverted upright allowing the etchant to “roll” around on the surface until the grain boundaries were revealed. Rinsing in water followed by methanol stopped the etching process. This process generally took between 20 and 40 seconds to ensure clearly defined grain boundaries on the samples.

The samples used for microstructure analyses were etched in Keller’s Reagent consisting of 95mL deionized H₂O, 2.5mL 69.1% NHO₃, 1.5mL 37% HCl, and 1mL 49% HF. The samples were immersed in the reagent and then inverted upwards and tilted in a manner that rolled the etchant around the surface. The etching times were between 20 to 40 seconds until grain structure became just visible to the eye. The samples were rinsed in water then methanol and dried using compressed air.

A color tinting etchant, Weck’s Reagent, was used for the qualitative microsegregation analysis. Weck’s Reagent consists of 4g KMnO₄ and 1g NaOH dissolved into 100mL of deionized H₂O. The NaOH pellets were allowed to dissolve into the water for approximately 10 minutes, then the KMnO₄ was added and allowed 5 minutes to go into solution. The polished samples were immersed in Weck’s Reagent for 20 seconds, rinsed with water and ethanol then
dried with compressed air. This etchant created a slightly gold tinted surface with visible black dendritic structures.

3.5 Macrostructure Characterization

3.5.1 Photography

A Polaroid MP-4 photography stand with an adjustable height camera mount was used to take digital photographs of the macroetched ingot surfaces. A Canon Rebel EOS T3 12.2 megapixel SLR digital camera was attached to the photography stand. Maximum optical magnification of 55mm (approximately 3.5x) was selected as the basis and the height of the camera adjusted until the entire ingot could be imaged with each shot.

In order to maintain a correlation to the size of the features within the photograph a metal scale was attached to an edge of the ingot extending 11mm into the ingot surface. This allowed scaling of the photograph in the image analysis software.

Photographs were taken with no flash and with lighting from various angles to provide the most accurate representation of the grains. The photographs with the best resolution were kept for further image analysis.

3.5.2 Image Analysis

The Nikon NIS Elements BR 3.0 software package was used to analyze the average grain size of the specimens. The software package has a specific metallography application for determining the average grain size through contrasting of grains and/or the grain boundaries.

The digital photographic images were further cropped and magnified to bring high resolution in the area toward the bottom of the ingot. These images were then opened with the Nikon NIS Elements software for analysis.
Detection presets were set to aluminum, a single data smoothing routine, and contrasting grains for the stirred samples. The method chosen for the analysis was the Abrams Circle consisting of horizontal / vertical line lengths of 10000µm, diagonal line lengths of 15000µm, small circle diameter of 5000µm, medium circle diameter of 7500µm, and large circle diameter of 10,000µm.

Multiple measurements were made on multiple photographs for each sample in the 11mm to 30mm region from the bottom of the ingot using the ASTM E1382-97 standard. Measurements were collected until reasonable statistical accuracy between 3-4% was achieved. The averaging of the data between the 11mm and 30mm heights gave good correlation to the average grain size at the 20mm height location where thermal data was recorded. The software provided a distribution chart of the various grain sizes along with average grain size, deviation, and statistical accuracy.

The ingot with no stirring required a different approach than that used for the EM stirred ingots. The software was very sensitive to the large grains in the unstirred sample. The software detected small macro-pores and other features within the grains and counted them as separate small grains destroying the accuracy of the results. Manual intercept methods had roughly determined the correct grain size range, but the software overcompensated.

A high quality picture of the “no stir” ingot was printed out. Using the actual ingot as reference, every grain boundary was drawn by hand onto the printed photo of the unstirred ingot. Then another picture was taken of the picture with the hand drawn boundaries.

This picture with hand drawn boundaries was then uploaded and analyzed using presets “contrasting grains with dark boundaries”. This yielded very good results that compared very well with the previous manual intercept measurements that had been done.
3.6 Microstructure Characterization

3.6.1 Optical Microscopy

A Nikon Epiphot 200 light optical microscope with RS-232 stage controller and Q-I
Imaging RGB / Monochrome side mounted camera was used for the imaging of the samples.
The camera output was input into a PC running Nikon NIS Elements BR 3.0 image analysis
software. The total magnification used was either 50x or 400x.

A 50x magnification was utilized to image and measure secondary dendrite arm spacing
(SDAS) in the specimens. A statistical spreadsheet was maintained by the software such that
numerous measurements could be made at various locations on multiple samples and compiled
into a single averaged SDAS measurement. Generally at least 100 measurements were made on
each sample. A total of nine samples were measured: 3 ingots (no stir, low velocity, high
velocity) each at 3 heights (20mm, 40mm, 60mm).

Other photographs were taken of dendrite arm fragmentation, suspected mechanical shear
surfaces, and fragmented dendrites created by the effects of electromagnetic stirring during
solidification.

The composition was analyzed qualitatively at the micro-level using color tint etching
that revealed the concentration of copper in and around the dendrites. Both the rear polarizer
(with λ-wheel) and tinted lens underneath the nosepiece were used to create the polarized light
effect. A 30° polarization offset angle was used to create the highest resolution of the copper
rich regions. When the RGB camera mode was used, the copper was naturally highlighted in the
green spectrum.
Pictures of the unstirred and high velocity samples were taken at 50x total magnification. The photographs were taken at the 20mm height location. Similar features were chosen for the purpose of comparison.

3.6.2 Electron Probe Micro Analysis (EPMA)

A macrosegregation analysis was conducted on samples removed from different heights in the ingots using EMPA. The polished samples were analyzed for average composition in the JEOL JXA-8600 Superprobe electron probe micro analyzer using Bruker Esprit software. The purpose of this analysis was to provide large area compositional maps of the various samples.

In order to provide the most accurate results the spot size of the electron beam was set to 10µm diameter, accelerating voltage was 20kV, probe current was set to 40µA with the Faraday cup engaged, and magnification was set to 50x.

A total of nine samples (unstirred, low and high velocity at 20, 40, and 60mm heights above the chill) were analyzed using areas along the center axis of the ingot and in a radial direction near the center height of each sample. Between five and six measurements in the axial direction and ten to eleven in the radial direction were conducted for each sample for a total of around fifteen measurements per sample. The scan pattern across the sample is shown in Figure 3.9.

![Figure 3.9: EPMA scan pattern across mounted sample](image-url)
The compositional analyses for each sample were stored in a running spreadsheet within the software. These spreadsheets were then compiled into Excel files and averaged to create the compositional data at each of the three positions above the chill for all three solidification scenarios.
4. RESULTS and DISCUSSION

The experimental results and findings concerning the unidirectional solidification of Al-4.5wt%Cu alloy while subjected to electromagnetic stirring are presented in this section. The results include model calculations of the characteristic velocities for the two stirring conditions (low and high velocity), cooling rates, and solidification times. Post processing results present the findings regarding macrostructure, grain refinement, secondary dendrite arm spacing, fragmentation, and segregation of copper within the solidified ingot.

The relevant properties of Al-4.5%Cu are listed in Table 1. The density data is for Al-2024 aluminum alloy that has a chemical composition 3.8-4.9wt%Cu, 1.2-1.8wt%Mg, 0.3-0.9wt%Mn, and trace amounts of Fe, Si, Ti, Cr and Zn. Al-2024 is the closest commercial grade aluminum alloy to the laboratory Al-4.5wt%Cu alloy used in the experiments.

<table>
<thead>
<tr>
<th>Physical Properties of Al-4.5%Cu Alloy</th>
</tr>
</thead>
<tbody>
<tr>
<td>Liquidus Temperature T_L</td>
</tr>
<tr>
<td>Solidus Temperature T_S</td>
</tr>
<tr>
<td>Eutectic Temperature T_E</td>
</tr>
<tr>
<td>Eutectic Concentration C_E</td>
</tr>
<tr>
<td>Maximum Solubility C_{am}</td>
</tr>
<tr>
<td>Liquid Diffusivity D_L</td>
</tr>
<tr>
<td>Solid Diffusivity D_S</td>
</tr>
<tr>
<td>Partitioning Coefficient k</td>
</tr>
<tr>
<td>Solid Density ρ</td>
</tr>
</tbody>
</table>
4.1 Solidification of Al-4.5%Cu Alloy in an EMS Melt

The main premise of this work is the comparison of unidirectionally solidified Al-4.5%Cu alloy with and without stirring and to study the effect of the flow on grain refinement of the alloy. Stirring induces a convective flow on the bulk liquid metal. The magnitude of the bulk convective flow velocity influences solutal and thermal transport as well as thermal gradients near the solid-liquid interface (SLI). A method to predict characteristic velocity near the SLI was needed. Also, cooling rates that affect thermal gradients at the solidification front, SLI velocity, and solidification morphology (CET) were determined from the cooling curves.

4.1.1 Velocity Field

The characteristic velocity of a convective flow across the solidification front is a crucial factor to determine. In general in-situ measurements of velocity within EMS systems are difficult to conduct. Therefore numerical models are used to determine characteristic velocity. The components of the velocity flowing perpendicular and parallel to the solidification front influence particle transport of fragments. The component of the velocity acting parallel but opposite to the growth direction determines the amount of solute rich liquid that can penetrate into the mushy zone promoting fragmentation.

In this study, the characteristic velocities of the flow at the SLI during solidification were determined from modeling. Calculation of the velocity field in the EM stirred melt was carried out using a model that was developed by Greg Poole and Dr. Nagy El-Kaddah [24]. Table 2 summarizes the operating condition of the stirrer for the two cases considered.

<table>
<thead>
<tr>
<th>Case</th>
<th>Coil Height from Chill</th>
<th>Current</th>
<th>Frequency</th>
<th>Power</th>
</tr>
</thead>
<tbody>
<tr>
<td>Condition 1</td>
<td>70mm</td>
<td>200A</td>
<td>4890Hz</td>
<td>2.9kW</td>
</tr>
<tr>
<td>Condition 2</td>
<td>40mm</td>
<td>200A</td>
<td>4920Hz</td>
<td>2.9kW</td>
</tr>
</tbody>
</table>

Table 2: EM Stirring Parameters
Figure 4.1 illustrates a physical representation of the two cases studied. Figures 4.2a-c show the evolution of the velocity field at different stages of solidification corresponding to the SLI at 20, 40, and 60mm respectively for the first case (low velocity).

**Figure 4.1:** Stirrer configurations

**Figure 4.2a:** Low velocity plot, SLI 20mm above the chill
Figure 4.2b: Low velocity plot, SLI 40mm above the chill

Figure 4.2c: Low velocity plot, SLI 60mm above the chill
The corresponding figures for the second case (high velocity) are shown in Figures 4.3a-c

**Figure 4.3a:** High velocity plot, SLI 20mm above the chill

**Figure 4.3b:** High velocity plot, SLI 40mm above the chill
Table 3 summarizes the predicted velocities at different stages of solidification for the two cases considered.

### Table 3: Velocity Predictions for both EMS Cases

<table>
<thead>
<tr>
<th>Case</th>
<th>SLI Location from Chill (mm)</th>
<th>Mean Characteristic Velocity (mm/s)</th>
<th>Maximum Radial Velocity (mm/s)</th>
<th>Maximum Axial Velocity (mm/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Low Velocity</td>
<td>20</td>
<td>6.8</td>
<td>14.6</td>
<td>-4.1</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>8.6</td>
<td>25.4</td>
<td>-7.0</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>6.2</td>
<td>12.3</td>
<td>-30.8</td>
</tr>
<tr>
<td>High Velocity</td>
<td>20</td>
<td>12.4</td>
<td>20.6</td>
<td>-4.1</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>14.5</td>
<td>25.1</td>
<td>-8.1</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>23.1</td>
<td>34.9</td>
<td>-8.5</td>
</tr>
</tbody>
</table>

The velocity plots from the model displayed a single recirculating flow loop for both the high and low velocity stirring conditions when the SLI had reached the 20mm position above the chill maintaining a unidirectional flow across the SLI. The high velocity case where the...
induction coil was lowered increased the radial mean characteristic radial velocity by 82% and the maximum radial velocity by 41%.

For the low velocity stirring situation, the model indicated two recirculating loops within the velocity field for the low velocity stirring condition along the SLI at the 40 and 60mm positions. These two recirculating loops divided the flow directions across the SLI. At the 40mm SLI position, the divergent flow retained a higher radial velocity than axial velocity. At the 60mm position the indicated flow had very high axial components that drastically lowered the mean radial velocity across the SLI, but increased the penetration velocity carrying solute into the SLI.

The model of the high velocity stirring condition determined that a single recirculating loop of fluid flow was maintained across the SLI at both the 40mm and 60mm positions. The magnitude of the predicted mean and maximum radial velocities increased by 59% and 39% respectively as the SLI moved from the 40mm to the 60mm position.

4.1.2 Cooling Rates

The cooling rate is critical in determining the structure of a solidified alloy. It influences the solidification rate, undercooling, and thermal gradient during solidification, all of which affect solidification morphology and microstructure of the cast alloy.

Experiments were conducted without stirring in order to establish a baseline for determining the effect of stirring on grain refinement and to ascertain the required cooling rate and solidification time to create an equiaxed dendritic macrostructure. All experiments started solidification at 675°C with a superheat of 26.3°C in order to aid the promotion of an equiaxed dendritic structure. Rough calculations were made to determine the amount of reduction of the insulating Al₂O₃ layer on the chill plate necessary to offset the Joule heating effect of the stirring.
on the rate of solidification and to maintain the same cooling rates and solidification times for all samples.

The initial cooling rates from superheat temperature to the liquidus temperature were very linear in nature. A simple calculation consisting of $\Delta T/\Delta t$ determined the initial cooling rates going into the start of solidification. A comparison of the initial cooling curves for all three cases (without stirring, low velocity, high velocity) at position 20mm from the chill is shown in Figure 4.4. Cooling curves for the 40mm and 60mm positions are shown in Figures 4.5 and 4.6 respectively. Table 4 summarizes the cooling rate data for the three cases investigated.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Distance from Chill (mm)</th>
<th>Initial Cool Rate (°C/s)</th>
<th>Solidification Time (sec)</th>
<th>Average Solidification Cooling Rate (°C/s)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unstirred</td>
<td>20</td>
<td>0.4029</td>
<td>1039</td>
<td>0.0820</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>0.3896</td>
<td>1047</td>
<td>0.0829</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>0.3474</td>
<td>1030</td>
<td>0.0829</td>
</tr>
<tr>
<td>Low Velocity</td>
<td>20</td>
<td>0.3956</td>
<td>935</td>
<td>0.0905</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>0.4005</td>
<td>952</td>
<td>0.0888</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>0.4056</td>
<td>969</td>
<td>0.0875</td>
</tr>
<tr>
<td>High Velocity</td>
<td>20</td>
<td>0.4085</td>
<td>959</td>
<td>0.0883</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>0.4088</td>
<td>977</td>
<td>0.0867</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>0.4032</td>
<td>990</td>
<td>0.0855</td>
</tr>
</tbody>
</table>

**Figure 4.4:** Initial cooling curves, $\Delta T/\Delta t$ between $0.3955^\circ$C/s and $0.4033^\circ$C/s
Figure 4.5: Initial cooling curves, $\Delta T/\Delta t$ between 0.3896°C/s and 0.4088°C/s

Figure 4.6: Initial cooling curves, $\Delta T/\Delta t$ between 0.3474°C/s and 0.4056°C/s

At the 20mm position above the chill both stirred samples and the unstirred sample achieved average cooling rates of approximately 0.4°C/s with a range of less than 0.02°C/s. The initial cooling rate in the unstirred sample decreased as distance from the chill plate increased. This decreased cooling rate is typical for unidirectional solidification due to the increasing distance the heat must travel to be extracted by the chill plate. The stirred samples both showed no decrease in the cooling rate regardless of position and maintained a constant cooling rate of 0.4°C/s. This was due to the induced convective currents redistributing the heat throughout the
liquid and allowing extraction of the heat from the liquid metal flowing across the chill surface. Even with the decreasing cooling rate of the unstirred sample, the difference in cooling rate at the 60mm position was less than 0.06°C/s.

Another observation was that both stirred samples reached the liquidus temperature at all five thermocouple positions (10,20,40,60,75mm) within 2 seconds of each other. The sample not subjected to stirring had a 20 second delay between the closest position (10mm) and furthest position (75mm) from the chill. This indicated that the forced convection in the stirred samples distributed the thermal energy within the liquid more evenly than conditions without convection where conduction and thermal diffusion controlled the extraction of heat.

Once solidification started the cooling curves became non-linear due to latent heat rejection and solid fraction evolution. The total solidification times between the liquidus (648.7°C) and the solidus (564°C) temperatures as well as linear average cooling rates were used for comparison. Since the solidification times were very similar, the overall solidification rates were also synonymous. Plots showing the solidification times of the three cases (no stirring, low and high velocity) at 20, 40, and 60mm from the chill are given in Figures 4.7, 4.8, and 4.9 respectively. The cooling rate data during solidification were summarized in Table 4 shown earlier in this section.

![Figure 4.7: Local solidification times for all three cases](image)
The sample solidified without stirring had the longest localized solidification time at all three positions. The two samples solidified with stirring locally solidified quicker than the sample without stirring due to increased heat transfer from the convective flow across the solidification front. At 60mm away from the chill, the solidification time differences between the stirred samples and the unstirred sample were reduced since heat flow by conduction, rather than convection, became the dominant mode of heat transfer to the chill.

Due to solute rejection into the liquid and the low partitioning coefficient of Al-Cu (k=0.14), pools of eutectic composition remained in the interdendritic regions at temperatures below the solidus temperature of 564°C. This eutectic composition liquid finally solidified once
sufficient undercooling was achieved to force the solidification. The experiments conducted here had eutectic formation temperatures in the 541-543°C range due to the non-equilibrium solidification with thermal undercooling effects of between 5-6°C. A relationship was observed regarding the eutectic formation times and the average composition at the three positions above the chill. The eutectic formation times are shown for a stirred sample in Figure 4.10. Further discussion of this finding is made later in this chapter.

![Figure 4.10: Low velocity stirred sample eutectic formation times](image)

**4.2 Characterization of Al-4.5%Cu Alloy**

One of the basic premises for this work was to compare the effects of electromagnetic stirring in a unidirectional solidification system on grain refinement. Al-Cu alloys generally solidify into columnar dendritic or equiaxed dendritic structures [3,7,8,9,11]. Comparisons of columnar grains pose challenges regarding size, width, and length. Equiaxed dendritic structures are somewhat simpler to compare, but care must be taken to analyze the grains at the same position from the chill. In absence of grain refinement (i.e. no stirring) grains will generally
grow larger as a function of the distance from the chill since the cooling rate is also reduced further from the chill. Unidirectional solidification without convection and low cooling rates usually (unless poured into a cold mold) starts by nucleating and growing columnar grains until the undercooling ahead of the SLI is sufficient to nucleate equiaxed grains that impede the continued growth of the columnar grains. Most of the characterization in this report pertains to the position 20mm from the chill where the smallest equiaxed grains were present for comparison. The analysis of macrosegregation was carried out at three positions; 20, 40, and 60mm from the chill surface.

4.2.1 Macrostructure of Solidified Alloy

Figure 4.11 shows a photograph of the grain structure of the cast ingot in an unstirred melt. As seen in this figure the unstirred sample exhibited columnar growth during the initial solidification near the chill surface and underwent a columnar to equiaxed transition (CET) approximately 10mm above the chill. One location at the outer radius grew columnar grains 20mm from the chill. The unstirred sample also displayed centerline shrinkage between grains and the dendrite arms of the larger grains. The shrinkage was most likely caused by insufficient
feeding of liquid into the interdendritic regions during solidification and the inability of absorbed gases to escape.

The two samples exposed to EMS during solidification exhibited an equiaxed dendritic structure with no formation of columnar structure adjacent to the chill plate as shown in Figure 4.11. Figure 4.12 (a) and (b) shows the grain structure of low and high velocity stirred melts, respectively. This figure also shows that the equiaxed grains of the stirred samples had a more uniform size and even distribution in both the axial and radial directions, especially in the lower halves of the ingots. The upper halves of the ingots contained larger equiaxed grains where cooling rates were reduced. Both stirred samples displayed no signs of centerline shrinkage or porosity due to the convective flow feeding liquid to the interdendritic regions and transporting dissolved gases away from the SLI.

**Figure 4.12:** Low (a) and high (b) velocity stirred ingots unidirectionally solidified

EMS demonstrated a strong influence on grain refinement compared to conditions when no stirring was applied. Measurements made between 11 and 30mm, where the smallest grains were located regardless of convection, indicated that even low velocity flows have the potential to greatly reduce grain size. The high velocity case with an increased mean radial characteristic

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velocity tended to reduce grain size further, but to a lesser extent than observed when no flow conditions were compared to forced flow conditions.

The cast ingot without stirring had an average grain size of 2608.7µm with a minimum of 175.3µm and a maximum of 7387.9 µm. The standard deviation was 1382.1µm with a relative accuracy of 3%. Most grains were in the 1 to 3.5mm size range. The measured grain size distribution of the unstirred ingot is shown in Figure 4.13.

Figure 4.13: Grain size in millimeters of unstirred ingot

Figure 4.14 shows the grain size distribution for the low velocity experiment. As seen in this figure the grain size was much smaller than that for the unstirred melt with much smaller average grain size of 648.6µm with a measurement range between 93.8µm and 2425µm. Standard deviation was 414.4µm with a relative accuracy of 4%. Most grains were between 0.3 and 0.6mm with some larger grains in the 1 to 2mm size range that pushed the average to over 0.6mm.

With the introduction of a forced convective flow across the SLI fragmentation was increased. With the increased amount and frequency of fragmentation, the number of nuclei that survived in the undercooled region increased. Therefore the nucleation rate, in effect, also increased. This increased nucleation rate of grains due to fragmentation appeared to be the primary mechanism of the reduced grain size in the samples subjected to EM stirring.
The grain size for the high velocity stirred melt was slightly smaller than that obtained at lower velocity with an average grain size of 498.7 µm and measured range between 83.8 µm and 2571.9 µm. The standard deviation was 311.3 µm with a relative accuracy of 4%. Most grains sizes were in the 0.2 to 0.5 mm range with very few grains over 1 mm in size. The grain size distribution of the high velocity sample is given in Figure 4.15.

The average grain size of the low and high velocity stirred samples was respectively reduced by 75.2% and 80.9% compared to the unstirred sample. Increasing the characteristic velocity from 6.8 mm/s to 12.4 mm/s (low to high velocity conditions discussed in section 4.1.1) reduced the grain size 23.2% further. This suggests that there is a critical velocity for grain refinement, and the effect of flow is inversely proportional to the magnitude of the velocity to power 1/4. Further statistical data are available for review in Appendix I.
4.2.2 Microstructure

In this section a comparison between unstirred and EMS microstructures using secondary dendrite arm spacing (SDAS) and optical photographs of the general microstructures is given. The microstructure for the cooling rates employed in this study, as given in section 4.1.2, is expected to be dendritic [26].

In most unidirectional solidification systems without stirring the SDAS increases as a function of the distance from the chill. With the introduction of convective flow, studies have indicated a large initial SDAS at the chill with decreasing SDAS further away from the chill [7,14].

The results show there was not a steady trend of increasing or decreasing SDAS as the distance from the chill increased. All three samples displayed parabolic trends with smaller mean SDAS at 20mm from the chill, increasing at the 40mm position, and decreasing again at 60mm from the chill. The mean measured SDAS is shown in Figure 4.16.

![Figure 4.16: Measured mean SDAS in the axial direction](image)

Since cooling rates of both unstirred and stirred experiments were comparable, this study isolated and investigated the effect of convective flow with regards to the secondary dendrite arm spacing. The SDAS of the unstirred sample was larger at all axial locations than the SDAS of the
stirred samples. Micrographs of the secondary arm measurements of the sample without stirring and one with stirring are shown in Figures 4.17 and 4.18 respectively.

**Figure 4.17:** Unstirred sample 40mm from chill, magnification 50x

**Figure 4.18:** High velocity sample 40mm from chill, magnification 50x

The range of measurements was quite large for all three conditions. The smallest measured SDAS on the unstirred sample was 129.3µm and around 100µm for both stirred...
samples. The maximum measured SDAS on the unstirred sample was 382.8µm compared to 298.2µm for the stirred samples. The average range of measurements was around 200µm for the unstirred sample compared to 160µm for low velocity and 143µm for the high velocity specimens. The high velocity sample had the tightest SDAS length distribution followed by the low velocity sample and the unstirred sample had the widest SDAS length distribution.

Due to the large range of measurements the average standard deviation was 44.5µm for the unstirred sample with 32.7µm and 30.5µm for the low and high velocity samples respectively. Stirring reduced the SDAS overall compared to conditions with no convective flow. The velocity difference between the stirred samples had very little effect on SDAS since the mean SDAS values were well within the standard deviation ranges of each other. SDAS distribution charts with accompanying statistical data are given in Appendix I for review.

4.2.3 Grain Refinement Mechanisms

4.2.3.1 Solutal Fragmentation of SDA’s

The process of fragmentation during solidification is a key factor in developing refined grain structures. Fragmentation is not relegated to conditions involving bulk convective flow and can occur during normal solidification if the solutal and thermal conditions are appropriate [11,12].

A micrograph of fragmenting secondary dendrite arms in a no flow situation is shown in Figure 4.19. The equiaxed dendritic structure of the unstirred sample displayed well formed dendrites with secondary arms. Fragmentation occurred where pools of solute rich liquid enclosed the roots of the secondary arms. Without convective flow the detached arms remained near the parent dendrite.
The introduction of convective flow through stirring appeared to have increased the rate of fragmentation. The flow was also found to deform the secondary dendrite arms that were growing during solidification, displayed in figure 4.20, as would be expected [27, 28]. Fragmentation occurred when the arms were surrounded by solute rich liquid as in the case of the unstirred sample. Unlike the unstirred sample some of these fragments were transported to other locations in the bulk convective flow. A large primary dendrite experiencing fragmentation during stirred conditions is shown in Figure 4.20.
The fragments that were carried away from the parent dendrite continued to grow if warranted by the undercooled conditions in the bulk liquid. These fragments and partially formed dendrites were eventually embedded into the forming dendritic structure. The result was a chaotic microstructure observed in both stirred samples. Figures 4.21 and 4.22 show areas of chaotic microstructure due to fragmentation promoted by convective flow.

**Figure 4.21:** Low velocity sample 20mm from chill, magnification 50x

**Figure 4.22:** High velocity sample at 40mm from chill, magnification 50x
The increased fragmentation and bulk transport within the stirred samples produced a more chaotic microstructure made of fragments and partially formed dendrites with deformed arms. The unstirred sample also displayed fragmentation, but the overall microstructure remained much more ordered indicating that transport of fragments elsewhere without forced convection did not occur. A picture of the ordered microstructure found in the unstirred sample is shown in Figure 4.23.

![Unstirred sample 60mm from chill, magnification 50x](image)

**Figure 4.23**: Unstirred sample 60mm from chill, magnification 50x

### 4.2.3.2 Mechanical Shearing of SDA’s

Microscopic examination has shown some fractured grains in both stirred samples as shown in Figures 4.24 and 4.25. The number of identifiable locations with fracture surfaces was miniscule [6] compared to the amount of solutal fragmentation that occurred within the samples, but some fracturing of the dendrite arms was observed.

Secondary dendrite arm fracturing was observed in both the low and high velocity samples at the 20mm location and at 40mm in just the high velocity sample. No fracturing of SDA’s was found in the samples taken at 60mm from the chill. An example of mechanical fracturing is shown in Figure 4.24.
It was noticed that the fractured surface sometimes appeared after the root of the arm started to remelt due to solute rich liquid being adjacent to the root. As the cross-sectional area of the root was being reduced, it became more susceptible to fracture. In situations with solutal fragmentation, the ends of the dendrite arms are smooth and may form a tapered end at the point of separation. Mechanical fracturing leaves a very angular fracture surface where the break occurred. The picture in Figure 4.25 shows a dendrite where some remelting of the root may have occurred before it was fractured.
The fracturing of dendrite arms observed in this study most likely occurred towards the end of solidification. If a dendrite arm was fractured at the beginning of solidification the exposed fracture surface would increase the surface area to volume ratio of the fragmented arm. With the increased surface area due to fracture being exposed, the angular surface would either remelt if exposed to solute rich and high temperature liquid, or would solidify to a curved surface due to the increased heat transfer from the larger surface area created by the fracture. Therefore the exact amount of SDA fracturing during solidification within an EMS system could not be determined.

With the introduction of a forced convective flow across the SLI increased fragmentation can occur and the fragments transported into the bulk liquid. If these fragments survive in a recirculating flow and have sufficient velocity and mass, then it is plausible that the impact of these particle fragments against the dendrites protruding from the solidification front may cause mechanical fracturing of the dendrite arms. Another possible cause of mechanical fracture may be due to hydrodynamic forces (sharp pressure or velocity gradients) at the SLI [29]. Most studies rule out mechanical fracture as a mechanism for grain refinement due to the highly plastic nature of dendrites close to the liquidus temperature. Other studies allude to mechanical fracturing as a possible refinement mechanism, but have discovered no evidence [6].

4.2.4 Segregation of Copper in the Cast Ingot

The aluminum-copper alloy system has a very low partitioning coefficient of 0.14 leading to segregation of the copper from the aluminum α-matrix during solidification. During solidification without stirring the segregation is primarily diffusion driven. With the introduction of stirring, the convective flow can redistribute the copper in the bulk liquid causing compositional differences at both the micro-scale and the macro-scale. At the micro-scale the
amount of solute remaining around a dendrite can cause changes in the secondary arm spacing and the amount of eutectic containing $\theta$-phase CuAl$_2$ that forms around the dendrites during final solidification.

### 4.2.4.1 Microsegregation

Figure 4.26 shows the concentration of copper (represented by the intensity of green) and eutectic formations in the unstirred melt. This sample displayed almost continuous eutectic formations in between secondary dendrite arms as well as at the grain boundaries. Copper concentrations even without the formation of eutectic between the dendrite arms remained quite high.

![Figure 4.26: Unstirred sample 20mm from chill, magnification 50x](image)

With the introduction of stirring the concentration of copper around the dendrites was modified. Figure 4.27 shows dendrites with reduced copper concentrations and discontinuous eutectic between the arms of a stirred sample. This figure shows that continuous eutectic formations were generally only observed at grain boundaries. Copper concentration in between...
arms was reduced due to the convective flow “washing” the solute from the interdendritic network and transporting it elsewhere in the bulk fluid.

![Figure 4.27: High velocity sample 20mm from chill, magnification 50x](image)

In the sample without stirring, the copper formed in between the dendrite arms and at the grain boundaries strictly due to diffusion. The forced convective flow in the high velocity sample with a mean characteristic velocity of 20.6mm/s, as given in section 4.1.1, broke down the diffusive solutal boundary layer and transported the solute into the bulk liquid leading to reduced secondary dendrite arm spacing as discussed in section 4.2.2 and caused the lack of eutectic formation in around the dendrite as shown.

### 4.2.4.2 Macrosegregation

In this investigation it was found that forced convective flow enhanced the solute transfer across the solutal boundary layer into the remaining bulk liquid. Without convective flow solute is redistributed via diffusion during regular unidirectional solidification with concentrations increasing from the chill to the top of the ingot roughly following the Scheil equation.
The unstirred sample displayed classic macrosegregation behavior with slight increases of the copper concentration at the 20, 40, and 60mm positions, indicative of transport by solute diffusion. The copper concentration increased from around 3.5wt% to 3.75wt% between the 20 and 60mm positions. The sample subjected to low velocity stirring had very consistent almost constant copper concentrations at 20mm (3.26wt%) and 40mm (3.27wt%) away from the chill, but increased to 4.44wt% at 60mm. The high velocity stirred sample experienced heavy segregation along the ingot height. The copper concentrations varied from 2.83wt% at 20mm to 4.51wt% at 60mm. A chart summarizing the average measured copper concentrations for all three experimental conditions along the ingot height is presented in Figure 4.28.

![Figure 4.28: Macrosegregation of copper due to experimental conditions](image)

The effect of stirring on macrosegregation appeared to be velocity dependent according to the collected data. The low velocity stirring condition removed solute in a constant manner from the solidification front until the build-up of excess solute caused the average composition to increase toward the top of the ingot. This was mirrored by the results of the velocity calculations given in section 4.1.1 where the mean radial characteristic velocities at 20 and 40mm were very similar (6.8 and 8.6mm/s respectively). At 60mm, the high axial components of the velocity (-
30.8mm/s) and low mean radial velocity (6.2mm/s) forced the solute rich bulk liquid into the SLI and caused the average concentration to dramatically increase. This led to the very non-linear behavior seen in Figure 4.28.

The sample solidified under high velocity stirring conditions experienced “solute washing” from the solidification front rapidly increasing the copper concentration in the liquid leading to strong macrosegregation within the ingot. This was validated through the velocity predictions from section 4.1.1 where the mean radial characteristic velocity increased from 12.4mm/s at 20mm to 23.1mm/s at 60mm as solidification proceeded. The maximum axial velocities were very comparable at the 40 and 60mm locations which indicated that solute transport was dominated by radial flow in the high velocity ingot.

A rough correlation between the eutectic formation time and composition at each location was also noted in this research. Generally a longer eutectic formation time was proportional to increased composition at a fixed location within the ingot. The results of each condition (unstirred, low and high velocity) could not be directly compared with one another, but each individual experiment demonstrated a trend. The times of eutectic formation and compositions at three locations (20, 40, 60mm) for all cases investigated are given in Table 5.

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Eutectic Formation Time @ 20mm</th>
<th>Average Measured Composition @ 20mm</th>
<th>Eutectic Formation Time @ 40mm</th>
<th>Average Measured Composition @ 40mm</th>
<th>Eutectic Formation Time @ 60mm</th>
<th>Average Measured Composition @ 60mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unstirred</td>
<td>50 s</td>
<td>3.495%</td>
<td>67 s</td>
<td>3.627%</td>
<td>72 s</td>
<td>3.749%</td>
</tr>
<tr>
<td>Low Velocity</td>
<td>22 s</td>
<td>3.263%</td>
<td>22 s</td>
<td>3.272%</td>
<td>40 s</td>
<td>4.443%</td>
</tr>
<tr>
<td>High Velocity</td>
<td>15 s</td>
<td>2.831%</td>
<td>18 s</td>
<td>3.731%</td>
<td>49 s</td>
<td>4.509%</td>
</tr>
</tbody>
</table>

The noticed correlation between eutectic solidification times and composition was strong for the unstirred and low velocity samples. The correlation was much weaker for the high velocity sample.
5. CONCLUSIONS AND RECOMMENDATIONS

5.1 Conclusions

Studies were performed to determine the effect of flow on the grain refinement of Al-4.5wt%Cu alloy. Two sets of experiments were conducted; one without stirring to establish a baseline for comparison with two using linear EMS. The experiments were carried out with cooling rates that produced equiaxed solidification morphology. The stirring of the melt produced a 75-81% reduction of the average grain size compared to the unstirred ingot that demonstrated any introduction of convective bulk flow across the SLI can enhance fragmentation leading to a much finer grain structure. The two predicted mean radial velocity ranges employed in this study (6.2-8.6mm/s for low velocity and 12.4-23.1mm/s for high velocity) comparatively reduced the average grain size 23% from 648\(\mu\)m to 498\(\mu\)m. This suggests that the grain size \((d)\) reduction is proportional to the magnitude of the velocity \((V)\) by approximately \(V^{1/4}\).

The EMS decreased the average SDAS compared to the unstirred melt by approximately 22%. Negligible difference in SDAS was noted for the two EMS cases. Solutal fragmentation and transport of fragments was enhanced by EMS along with minute amounts of mechanical shearing that created a chaotic microstructure that may benefit some mechanical properties. The low velocity EMS maintained an almost constant copper concentration (3.26-3.27wt%) at the 20 and 40mm locations from the chill, but increased drastically when solidification reached 60mm likely due to predicted divergent flow with two recirculating loops with high axial components forcing solute into the SLI. The high velocity condition indicated solute washing from the SLI with heavy axial macrosegregation along the ingot height.
5.2 Recommendations

The EMS system employed during the course of this study operated at a high frequency EM field which produced excessive Joule heating and made it difficult to control the cooling rate, moreover it limited this investigation to low velocity stirring regimes. In order to reduce the amount of Joule heating while increasing the characteristic velocity, a low frequency coil should be employed. This would allow further exploration of increased grain refinement as a function of increasing velocity. The current system could easily accommodate running experiments from a lower superheat to aid in creating an equiaxed dendritic structure with higher stirring velocities.

Also, to better understand the thermal gradients near the SLI and solidification rate, additional thermocouples should be employed along the centerline axis to produce a finer resolution of temperature data as a function of position.

This study only investigated segregation of copper at three axial locations 0 to 20mm from the centerline axis. An expansion of the analyzed points both in the axial and radial directions would greatly aid the further development of a control volume model to predict the effect of linear EMS and the various flow patterns on macrosegregation. To further simplify the analysis, experiments should be performed for predicted stirring conditions where only one recirculating loop of bulk fluid sweeps across the radius to establish a well defined flow pattern for investigating segregation.
REFERENCES


APPENDIX I

Statistical Data: Grain Size Distribution

The statistical data for the measured grain size for all three cases are given in Tables A1, A2, and A3.

Table A1: Grain Size Statistical Data, Unstirred Sample

<table>
<thead>
<tr>
<th>Statistics</th>
<th>Aluminum</th>
<th>Units</th>
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</thead>
<tbody>
<tr>
<td>Grain Size G:</td>
<td>00+</td>
<td></td>
</tr>
<tr>
<td>Method:</td>
<td>Abrams</td>
<td></td>
</tr>
<tr>
<td>Standard:</td>
<td>ASTM E1382-97</td>
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<td>Num. of fields:</td>
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<td>Num. of intersections:</td>
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<tr>
<td>Anisotropy:</td>
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<tr>
<td>Mean:</td>
<td>2608.7 µm</td>
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</tr>
<tr>
<td>Minimum:</td>
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<tr>
<td>Maximum:</td>
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</tr>
<tr>
<td>Std. deviation:</td>
<td>1382.1 µm</td>
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</tr>
<tr>
<td>95% Conf. inter.:</td>
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<td></td>
</tr>
<tr>
<td>Rel. accuracy:</td>
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Table A2: Grain Size Statistical Data, Low Velocity Stirred Sample

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<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>Grain Size G:</td>
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<td></td>
</tr>
<tr>
<td>Method:</td>
<td>Abrams</td>
<td></td>
</tr>
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<tr>
<td>Mean:</td>
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<tr>
<td>Minimum:</td>
<td>93.8 µm</td>
<td></td>
</tr>
<tr>
<td>Maximum:</td>
<td>2425.2 µm</td>
<td></td>
</tr>
<tr>
<td>Std. deviation:</td>
<td>414.4 µm</td>
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</tr>
<tr>
<td>95% Conf. inter.:</td>
<td>28.2 µm</td>
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</tr>
<tr>
<td>Rel. accuracy:</td>
<td>4 %</td>
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</tr>
</tbody>
</table>
Table A3: Grain Size Statistical Data, High Velocity Stirred Sample

<table>
<thead>
<tr>
<th>Statistics</th>
<th>Aluminum</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
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<td></td>
</tr>
<tr>
<td>Method:</td>
<td>Abrams</td>
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<td>Mean:</td>
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<tr>
<td>Minimum:</td>
<td>83.8 µm</td>
<td></td>
</tr>
<tr>
<td>Maximum:</td>
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<td></td>
</tr>
<tr>
<td>Std. deviation:</td>
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</tr>
<tr>
<td>95% Conf. inter.:</td>
<td>20.7 µm</td>
<td></td>
</tr>
<tr>
<td>Rel. accuracy:</td>
<td>4%</td>
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Distribution and Statistical Data: SDAS Measurements

The distribution charts for SDAS given in micrometer (µm) units are shown in Figures A1 through A9 for all three cases considered at three locations from the chill. The relevant statistical data are given in Table A4.

**Figure A1:** SDAS distribution unstirred sample 20mm from chill

**Figure A2:** SDAS distribution unstirred sample 40mm from chill
Figure A3: SDAS distribution unstirred sample 60mm from chill

Figure A4: SDAS distribution low velocity sample 20mm from chill

Figure A5: SDAS distribution low velocity sample 40mm from chill

Figure A6: SDAS distribution low velocity sample 60mm from chill
Figure A7: SDAS distribution high velocity sample 20mm from chill

Figure A8: SDAS distribution high velocity sample 40mm from chill

Figure A9: SDAS distribution high velocity sample 60mm from chill

Table A4: Statistical Data for SDAS Measurements

<table>
<thead>
<tr>
<th>Case</th>
<th>Distance from Chill (mm)</th>
<th>Mean SDAS (µm)</th>
<th>Minimum SDAS (µm)</th>
<th>Maximum SDAS (µm)</th>
<th>Standard Deviation (µm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Unstirred</td>
<td>20</td>
<td>241.1</td>
<td>162.5</td>
<td>338.7</td>
<td>41.7</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>257.3</td>
<td>161.3</td>
<td>382.8</td>
<td>50.6</td>
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<tr>
<td></td>
<td>60</td>
<td>222.9</td>
<td>129.3</td>
<td>326.7</td>
<td>41.2</td>
</tr>
<tr>
<td>Low Velocity</td>
<td>20</td>
<td>181.8</td>
<td>104.4</td>
<td>263.5</td>
<td>34.0</td>
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<tr>
<td></td>
<td>40</td>
<td>206.6</td>
<td>130.6</td>
<td>298.2</td>
<td>31.5</td>
</tr>
<tr>
<td></td>
<td>60</td>
<td>176.2</td>
<td>99.4</td>
<td>254.3</td>
<td>32.5</td>
</tr>
<tr>
<td>High Velocity</td>
<td>20</td>
<td>188.5</td>
<td>103</td>
<td>254.8</td>
<td>29.3</td>
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<td></td>
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<td>194.5</td>
<td>129.3</td>
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<td>175.8</td>
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