Characterization of Positive Macrosegregation at the Subsurface of Direct-Chill Cast 7050 Aluminum Forging Billets

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Approval Page

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CAL POLY STATE UNIVERSITY

Materials Engineering Department

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Abstract

Segregation of alloying elements during casting of forging billets may persist to the final forged component leading to unacceptable surface appearance. Determination of the elements or compounds that segregate is the essential first step to solving this problem. Differential Scanning Calorimetry (DSC) was used to determine a composition profile of the macrosegregation occurring at the subsurface of an as-cast 7050 aluminum alloy billet. Data collection was carried out by separating 10 to 20 mg samples from the outer centimeter along the radius of the billet, as well as samples from the bulk interior of the billet for comparison with the nominal alloy composition. DSC analysis was performed using platinum capsules on the Exstar DSC6000, from 20°C to 600°C at a rate of 5°C/min over the temperature range of interest. DSC scans showed heat flow peaks for incipient melting and crystallization transformations for Al_2CuMg (S-phase). Size comparison of DSC peaks provided data about the relative masses at each distance from the surface. The partial heat of fusion of samples of several depth profiles was used to determine relative mass fractions as compared to a bulk sample. A sharp increase in S-phase concentration was observed near the surface and a drastic decrease at approximately 3 mm from the surface, followed by a return to bulk alloy values after 5 mm due to shrinkage-induced flow in the casting process.

Keywords: Materials Engineering, Aluminum, 7050, Macrosegregation, Liquation, Forging, Differential Scanning Calorimetry, DSC Shrinkage, Thermo-Solutal Convection, Fusion, Casting

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Introduction

Problem Statement

The goal of this project was to characterize the chemical and microstructural composition of the stock aluminum 7050 ingots used by Weber Metals (Paramount, CA) for their forged components. Many of these ingots contain a defect in which the subsurface layer of the ingot exhibits an undesirably high concentration of alloying elements and constituent particles due to macrosegregation. This phenomenon is commonly seen in direct-chill cast ingots, and is generally corrected for by machining processes such as turning to remove the outer segregated material layer of material. However, machining processes do not always successfully remove all of the segregated material due to imprecise alignment during the turning process or due to shallow cuts. Furthermore, the high-solute segregated region cannot be seen until the surface has been etched, which cannot practically be done until the forging process is complete. Consequently, an ingot containing this defect can undergo the entire forging process and the problem is only revealed during the final etching step. This completed part will then show bands or stripes of light and dark (low-solute and high-solute) material on its surface due to the shaping process **(Figure 1)**. Because the mechanical properties of this defect are unknown, finished parts that exhibit this banding cannot be confidently sold to clients, and must be scrapped.

Figure 1 - Finished Weber forging showing bands of high- and low-solute regions, an artifact from macrosegregation at the surface of the ingot.

Weber Metals

Weber Metals specializes primarily in aluminum and titanium forgings in the aerospace, semiconductor, and automotive industries. They are acknowledged as industry leaders in aluminum hand (open die) forgings. The forge offers four open-die presses, capable of applying forces from 1200 to 5000 tons, with a capacity for up to 8600 pound parts. Additionally, there are five closed-die presses that range in maximum force from 1500 to 33000 tons, accommodating titanium forgings up to 2500 square inches.

Realistic Constraints[1]

The goal this project hopes to achieve is to create a starting point for research into whether or not this segregated material has detrimental effects on mechanical properties of forgings. If the effect is below what is considered acceptable for the forged part applications, then the parts may not need to be scrapped. The effects of the macrosegregation in forged parts are apparent to Weber Metals, but its exact effect is not. Understanding what these effects are may allow them to create more lax quality control for this type of defect, or prove that the tight controls they currently use are necessary. Either way, the realistic constraints discussed in the next section are dependent on this knowledge.

Economic

The presence of economic constraints is straightforward. As parts are forged and completed, some display visible signs of macrosegregation. Because the effect on mechanical properties of these high-solute regions has not been studied, any finished forgings exhibiting this problem must be scrapped. All of these parts represent unnecessary loss for the company. Ensuring that these problems are not seen in finished parts, or proving that the segregation is a purely cosmetic issue allows for less waste and a more profitable business.

Health and Safety

The other present constraint of health and safety is related to the economic constraint. In short, the effects of the presence of macrosegregation in finished forgings on mechanical properties are not fully understood, and as such, cannot be confidently sold to customers. These parts could fail in situations that unaffected parts would not, and because Weber forgings are primarily used in the automotive and aerospace industries, this presents a potential risk to the safety of anyone operating a vehicle containing Weber's parts. For this reason, any forging that exhibits this problem is scrapped and recycled to ensure safety.

7050 aluminum is a heat-treatable alloy typically used in aircraft structural components, and similar applications in which high-strength, formability and low-density are required.

Table I - Table of Composition and Properties of Wrought 7050 Aluminum[2].

AA7050, Wrought Aluminum Alloy				
Composition	**Zinc**	**Copper**	**Magnesium**	**Other (< 0.2% each)**
	5.6 – 6.7%	2.0 – 2.6%	1.9 – 2.6%	Fe, Si, Mn, Zr, Ti, Cr, other
Mechanical Properties	**Yield Strength**	**Tensile Strength**	**Elongation**	**Young's Modulus**
	446 MPa	524 MPa	15%	70.3 GPa

Table I details the composition and general mechanical properties of 7050 Aluminum, but some discussion of its intermediate phases and strengthening mechanisms is necessary for an understanding of the importance of the macrosegregation problem. 7050 Aluminum is an age-hardening aluminum alloy, meaning that it is strengthened by aging heat treatments in order to precipitate intermediate intermetallic phases. In 7XXX-series aluminum alloys, $MgZn_2$ (η-phase) is the principal strengthening precipitate. During casting, Zinc and Aluminum are largely soluble, and a different intermetallic phase dominates the microstructure, Al_2CuMg (S-phase)[3] **(Figure 2)**. During the aging process, 7050 Aluminum is solutionized at 477° C at approximately the S-phase melting point, causing dissolution. It is then age hardened to precipitate out fine η-phase precipitates, resulting in greater strength due to precipitation strengthening.

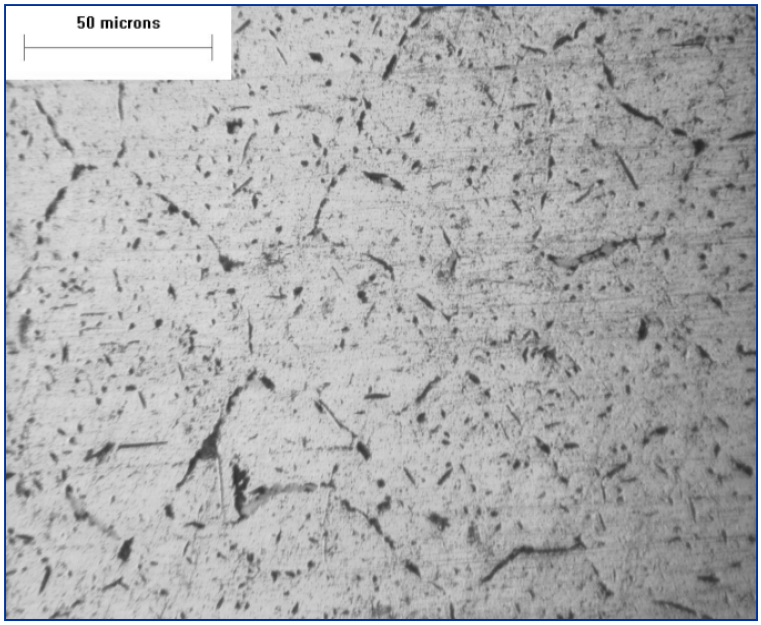


Figure 2 - As-cast microstructure showing occurrence of S-phase precipitates (black).

Semicontinuous Direct-Chill Casting

The most common process for the fabrication of aluminum ingots is the semicontinous direct-chill (DC) casting method (**Figure 3**). In this process, molten alloy is poured through a distributor which is generally a flat plate with holes to distribute the liquid. The liquid drains through the distributor into a short water-cooled mold of the desired cross-section. The process starts with a dummy block closing off the bottom of the mold to prevent molten alloy flowing out. Water streams contact the mold surface to draw heat from the ingot and through the mold; this is called primary cooling. Once the outer edges of the ingot have begun to solidify, the dummy block is drawn downward, and the solidified metal encapsulates further liquid entering the mold, resulting in a hollow shell containing the melt. As the ingot is pulled down, water jets are fired at the bottom of the ingot through holes in the bottom of the mold; this is called secondary cooling. Finally, as it continues being drawn down, it is lowered into a pool of standing water to complete the cooling process. Casting is complete when the billet reaches its bottom position[4].

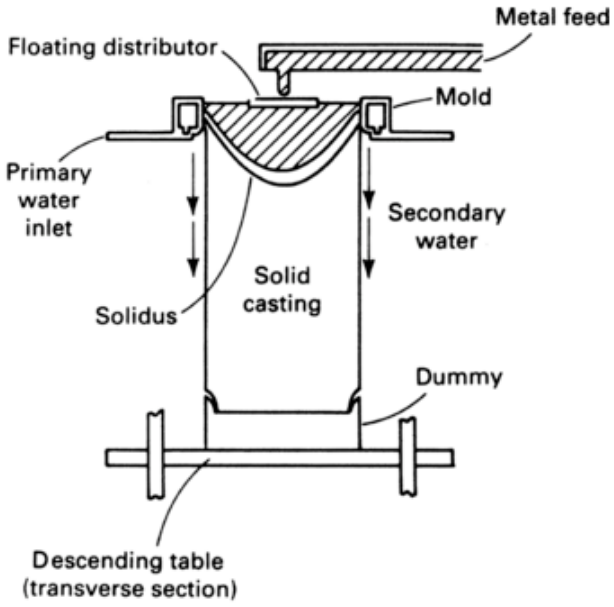


Figure 3 - Schematic of the semicontinuous direct-chill method of casting. This shows how the liquid is fed into the mold, encapsulated by the solidified shell, water-cooled, and the solid casting is then drawn out of the mold[4].

Ingot Solidification

Ingot solidification in the case of advanced engineering alloys is a complex process involving a variety of different reactions. The two largest factors defining the microstructure of a DC cast billet are those of standard supercooling, in which the liquid remains in the liquid state below its equilibrium freezing temperature, and constitutional supercooling. Constitutional supercooling occurs as material begins to solidify, expelling excess solute into the liquid where it is more soluble, resulting in liquid with a higher relative solute concentration. This, in turn, results in a lower freezing temperature, creating larger and larger supercooling as the process continues[5]. The freezing process begins once the melt is poured into the mold, and makes contact with the room-temperature mold wall. Because the outer wall of the mold is continuously cooled, liquid material nearest this wall cools rapidly, falling well below the equilibrium freezing temperature of the alloy, resulting in considerable supercooling. In the presence of large supercooling, dendritic growth becomes favorable, and the numerous nucleation sites cause the crystals to impinge upon each other quickly, limiting their growth. This region close to the mold wall consisting of small crystals is known as the chill zone (**Figure 4a)**. Moving further from the wall, the cooling rate, and consequently the amount of supercooling, is much lower. Because fusion generates heat at the liquid-solid interface, dendrite arms that form perpendicular to the mold wall will grow the

most rapidly. Similarly, the heat of fusion generated at their surface further inhibits the growth of dendrites in other directions[6]. The result is that dendrite arms perpendicular to the mold wall become heavily favored, and because the nucleation rate decreases due to the decreased supercooling, these arms are able to grow to a large size, forming columnar grains (**Figure 4b**). This region beyond the chill zone is referred to as the columnar zone. Beyond this region, in the center of the ingot, is the point where the decreased cooling rate overcomes the constitutional effect and the liquid is no longer supercooled. At this point, equilibrium cooling takes place, and primarily equiaxed grains form. This interior region is known as the equiaxed zone.

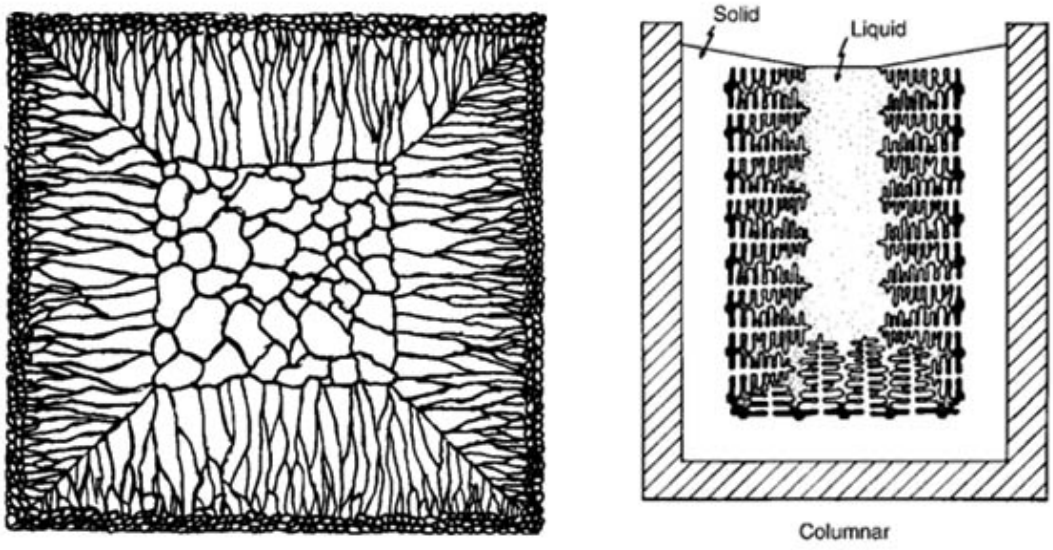


Figure 4 – (a) Sketch of ingot structures showing chill zone, columnar zone, and equiaxed zone (Flemings) and (b) Schematic of dendrite growth in the columnar zone[7].

Macrosegregation

Macrosegregation, at its foundation, occurs only due to the existence of microsegregation, in which solute elements are separated to different concentrations between the solid and liquid phases during solidification, as discussed in the overview of ingot freezing. The extent to which microsegregation, and consequently macrosegregation, occurs can most easily be quantified using the partition coefficients, K, defined as the slope of the solidus line over the slope of the liquidus line for a given binary phase system. The closer the partition coefficient is to unity, the lower the occurrence of microsegregation. In the case of 7050 aluminum, K is 0.17 for copper, 0.43 for magnesium, and 0.45 for zinc, resulting in an enrichment of the liquid with solute as seen in the DC cast billet[8]. The effect of solute enrichment as solidification begins at the mold wall and moves inward, however, seems to imply a segregation of

solute material to the center of the ingot, rather than at the surface as seen in the 7050 aluminum billets. It is only after considering the effects of several different fluid flow mechanisms, coupled with the channels created by the formation of a dendritic structure, that this specific pattern of macrosegregation can be understood (**Figure 5**). There are three primary accepted mechanisms of macrosegregation, and each contributes in a different way. Two forms cause negative centerline segregation, in which the center of the ingot is solute-lean, and the surface subsequently becomes solute-rich. A third causes the opposite effect, positive-centerline segregation. It is the superposition of these effects, with varying magnitude, that results in the general segregation profile seen below.

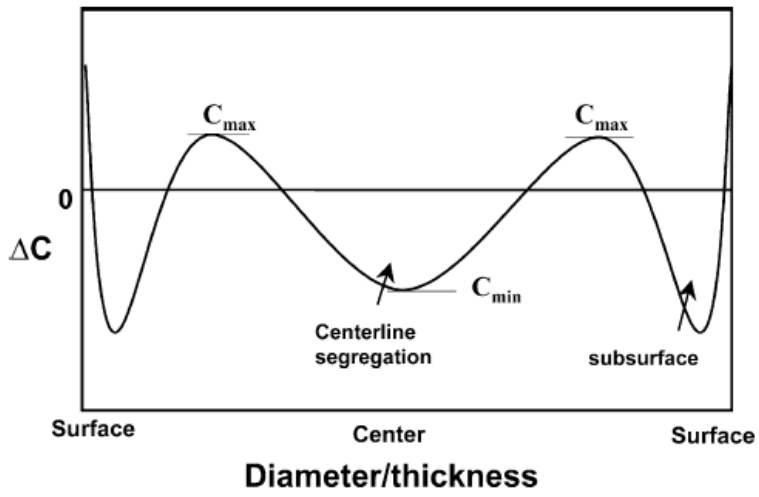


Figure 5 - Diagram of the segregation pattern seen in DC cast 7050 alloys[8].

Shrinkage-Induced Flow

The most influential flow mechanism by which solute-rich liquid is transported to the surface of the ingot is that of shrinkage-induced flow. There are three types of shrinkage that occur as a melt is poured into a mold and then solidified: liquidus, solidification, and solidus shrinkage. Liquidus and solidus shrinkage, as the names imply, are associated with the change in volume that occurs as a material cools. Solidification shrinkage, similarly, is the change in volume that occurs during the solidification of the liquid into a denser solid[9]. It is solidification shrinkage that contributes most to this form of flow, both because it is the largest volumetric change, and because of the existence of both liquid and solid phases during solidification. As the liquid nearest the mold wall begins to solidify and subsequently shrink, as much as 6-8% change by volume, consider the liquid immediately preceding the solidification front, recalling that it is being enriched in solute material. Because of the compression on the liquid due to the shrinkage of the solid, this liquid is drawn through channels created by the

interdendritic regions, which have yet to solidify, and out toward the surface to fill the now-empty space at the mold wall **(Figure 6)**. While the penetration distance of the liquid in this two-phase region, referred to as the mushy zone, is relatively low, the process is continually repeating as more solid is formed and more shrinkage occurs[8]. This allows for solute-rich liquid to migrate large distances in the duration of ingot solidification. The net result of this flow mechanism is a high solute concentration at the surface of the ingot, and a low solute concentration just below the surface.

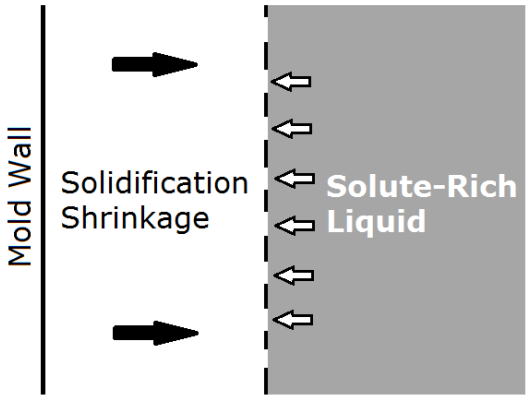


Figure 6 - Schematic representation of the shrinkage-induced flow macrosegregation mechanism.

Thermo-Solutal Convection

As with any fluid system involving a thermal gradient, thermal convection is a large force influencing fluid flow. The flow pattern is as expected, in which cooler, and thus denser liquid sinks and hotter, less dense liquid rises. The cooler liquid at the wall begins to sink downward along the sloped solidification front, creating a momentum that draws the rising low-solute center region back down against the solidification front (**Figure 7**). Because the solidification front consists of the permeable mushy zone, as seen in shrinkage-induced flow, the low-solute liquid that flows toward this front is driven in, where it solidifies, changing the overall concentration in this region.

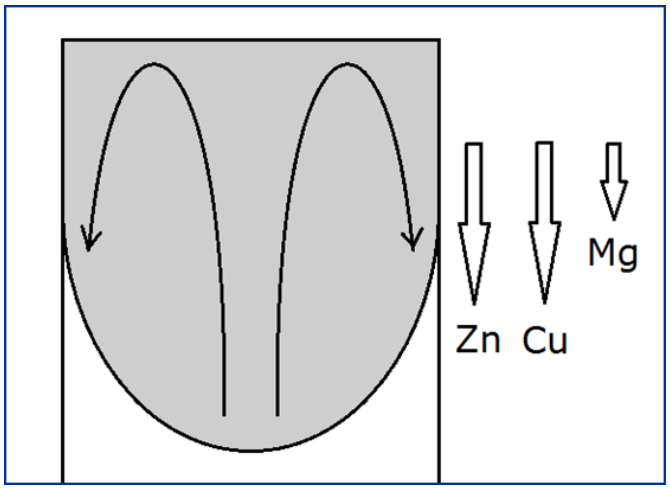


Figure 7 – Schematic view of thermo-solutal convection. Heavy elements such as zinc and copper reinforce the existing convection current and penetrate deeper into the solidification front, while lighter magnesium counteracts it and penetrates less deeply.

The buoyancy of different elements also has an effect on the overall convection current in much the same way. If the solute or solutes present in the enriched liquid have a density higher than that of the solvent metal, such as zinc and copper in aluminum, the thermal convection current is reinforced, causing deeper penetration. If however, the solutes are lighter than the solvent, the opposite is true, as in the case of magnesium[8]. The combination of thermal convection and solutal convection, in general, result in positive centerline segregation and negative surface segregation, opposite the effect of shrinkage-induced flow.

Characterization Using Differential Scanning Calorimetry (DSC)

As discussed above, the occurrence of macrosegregation is an artifact of the DC casting process. There are process parameters that can reduce the effect, but many of these decrease production and increase cost to a point where it is no longer economically feasible for a manufacturer such as Weber Metals to require them from an ingot supplier. For this reason, characterization and understanding of the effects of macrosegregation are of prime importance, rather than controlling it. One effective way of characterizing a sample of material sample is through analysis using a DSC. A DSC works by using a small sample of material, generally on the order of 10 to 20 milligrams. This sample is set in a pan, and heating at a constant rate next to a reference sample, generally an empty pan of the same type. As the sample of interest is subjected to the same temperature change, its change in heat flow can be

measured in comparison to the reference sample **(Figure 8)**. This heat flow data then shows a number of peaks and deviations which correspond to latent heat released or absorbed during phase changes, recrystallization, and other phenomena[10]. This data can then be interpreted using reference data to determine a number of sample properties, such as specific heats, or in this case, mass fractions of intermediate phases.

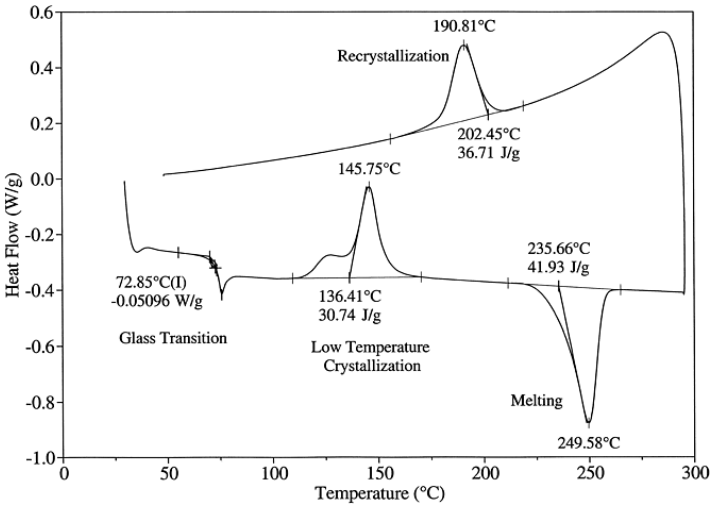


Figure 8 - Representative data from a DSC test run. Peaks indicate changes in heat flow, indicating the presence of phase changes and other phenomena[10].

Experimental Procedure

Microstructural Analysis

Microstructural analysis was carried out using standard metallographic practice. A section of aluminum billet normal to the surface was taken and mounted in mineral-filled diallyl phthalate and polished through sandpaper grits and diamond suspensions down to a 1 µm diamond suspension. This sample was then etched using a dilute Keller's Reagent (95 mL water, 2.5 mL HNO_3, 1.5 mL HCl, 1.0 mL HF), diluted to 80% water. Optical microscopy was then used to take images at 500x magnification at locations of interest on the sample. These microstructures provided corroboration for the analysis of DSC results.

Differential Scanning Calorimetry

Sample preparation for the DSC from semicircular billet sections was carried out as described in **(Figure 9)**, involving three cuts. A thin profile section was taken out of the billet section normal to the outer

diameter (OD) using an abrasive saw. Sample sections were then cut from this profile section at approximately 2 mm intervals, down to 6 mm using a diamond wafering saw. From the sample sections, DSC samples of approximately 15 mg were clipped with bolt cutters, or cut with the diamond saw when necessary. In total, three depth profiles, each containing three 2 mm steps, were taken from across two ingots. Prepared samples were then loaded into open platinum pans in a Seiko Exstar 6000 DSC. The temperature control was set as detailed in **Table II**.

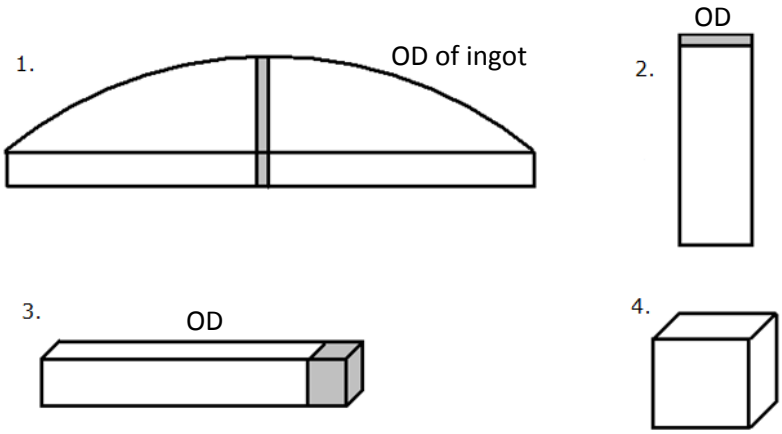


Figure 9 - Sample cutting procedure. Grey sections indicate the section of material taken for cutting in the next step.

Table II - Temperatures and Rates for DSC Method

Step	Start Temperature	End Temperature	Rate
Ramp-Up	20°C	400°C	30°C/min
Hold	400°C	400°C	0°C/min
Heating Measurement	400°C	600°C	5°C/min
Cooling Measurement	600°C	400°C	5°C/min
Ramp-Down	400°C	20°C	30°C/min

Results

S-Phase Evolution in As-Cast Billets

While η-phase precipitates are the main strengthening mechanism for 7050 Aluminum, it is the S-phase mass fraction that is of interest in this project. The Al-Zn-Cu-Mg alloy system is complex, and because of this, the microstructure during processing changes drastically, not only in the character of precipitates, but in their nature. In the as-cast state, much of the copper and magnesium precipitate into course S-phase precipitates, and the zinc remains in solution with aluminum. It is only after solutionizing and aging that the strengthening η-phase precipitates are formed. For this reason, the S-phase precipitates are of primary interest. This can be made most clear in the DSC scan of an ingot bulk sample **(Figure 10).**

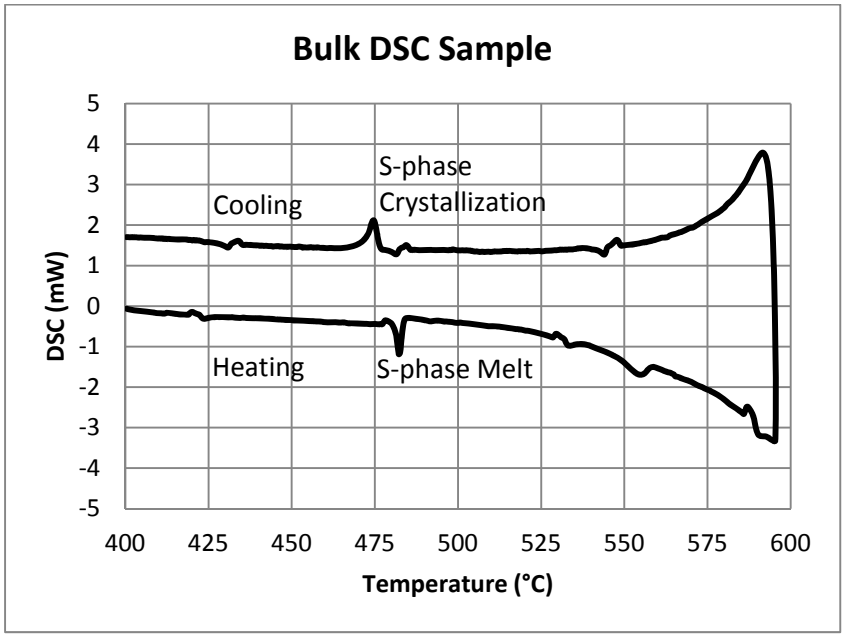


Figure 10 - DSC scan of a bulk sample of an as-cast billet. The large peaks occurring at 478°C denote melting and crystallization of S-phase intermetallics.

Large peaks in heat input occur at 478°C corresponding to the melting point, and thus melting and crystallization, of the S-phase. The other smaller peaks that appear above correspond loosely to other minor constituents, but their results register barely above baseline, and likely lie outside the sensitivity of this experiment. In short, it is clear that S-phase precipitates dominate the microstructure, as expected.

Depth Profiling

As discussed in the macrosegregation section, as the surface of the ingot is profiled, there should appear a large fraction of S-phase occurring near the surface, followed by a sharp drop below the bulk fraction, and up to normal values. Comparing the DSC results of crystallization peaks from the cooling curves at samples of average depth of 1.01 mm, 3.66 mm, and 6.06 mm, this trend is seen as described **(Figure 11)**.

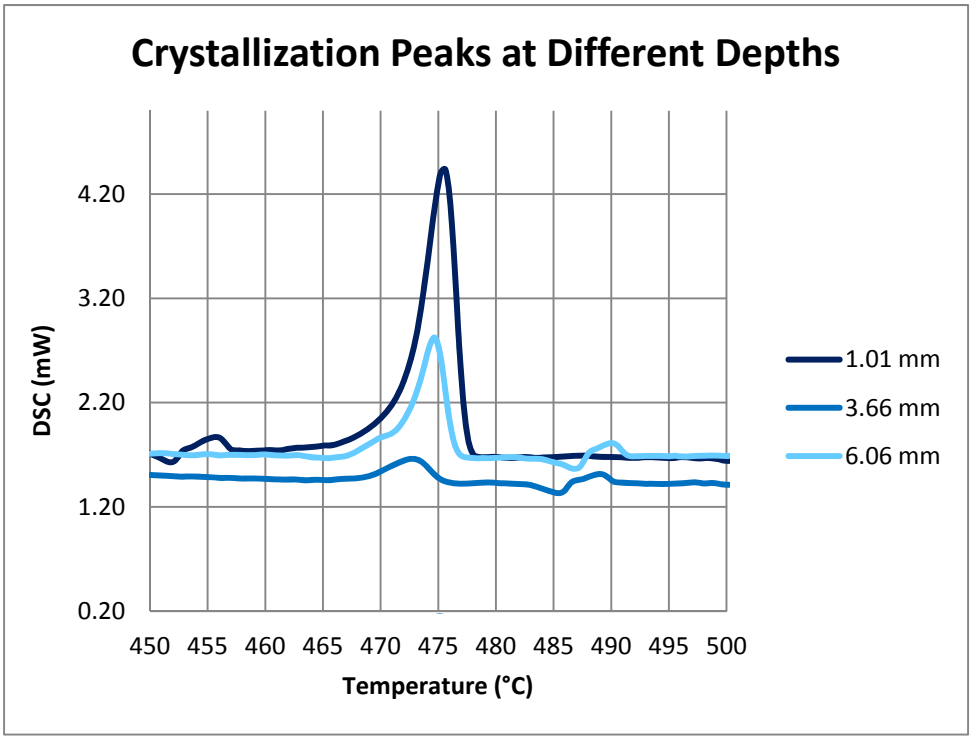
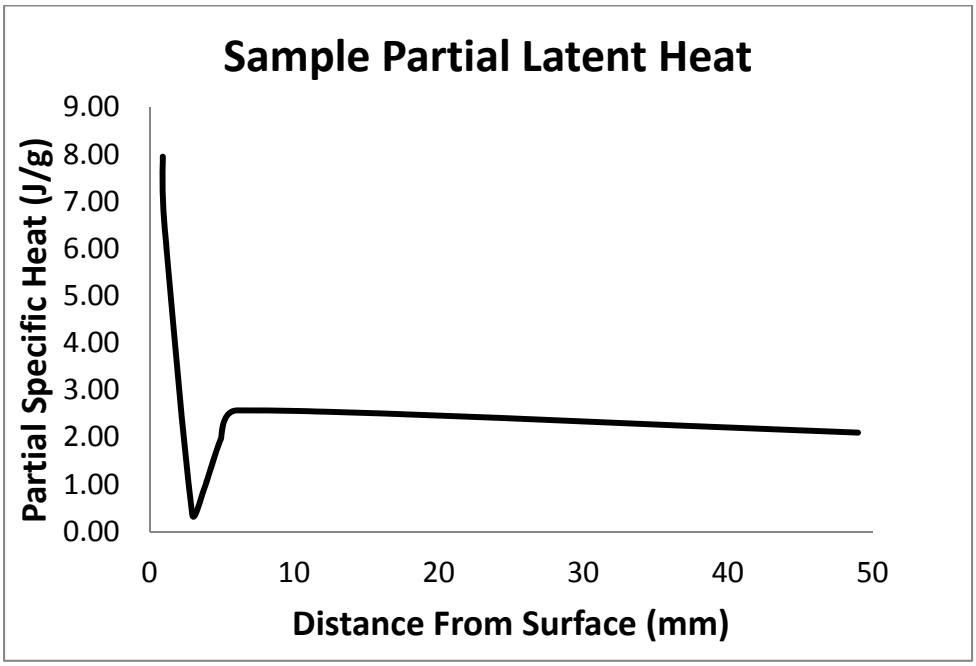


Figure 11 - Crystallization peaks of DSC scans at three different locations of a single profile on one ingot. The high surface fraction, drop, and return to nominal values can be seen.

By integrating the area under the crystallization peaks, the latent heat of fusion of the S-phase solidification in each sample, further referred to as the partial latent heat, can be determined. Note that the partial latent heat refers to the heat evolved due to the S-phase transforming in the 7050 sample, and not the latent heat of fusion of the S-phase transformation in general, which is a fundamental chemical property. In many cases, it is possible to determine the mass fraction of S-phase by using the partial latent heat. However, this process involves an equipment calibration process that was not available in the scope of this project. Instead, relative mass fractions were determined against the bulk

results. By integrating the area under this curve and plotting against the depth of the sample, a clear trend is shown **(Figure 12).**



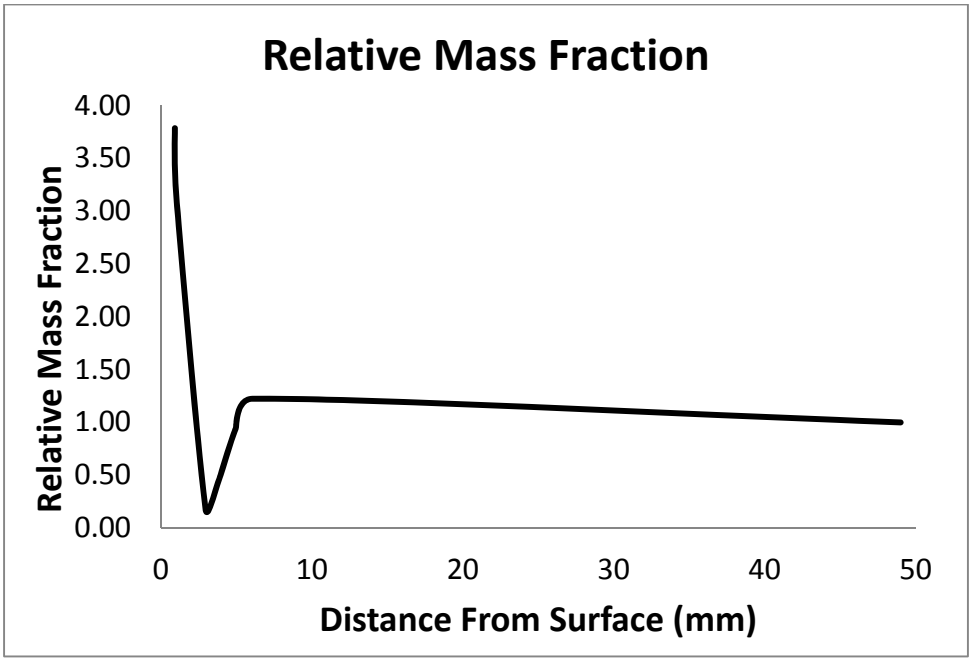


Figure 12 – A) Plot of partial latent heats by distance as compared to the bulk sample, revealing an extremely high surface fraction, followed by a sharp decrease. B) Plot of S-phase mass fractions relative to the bulk sample, directly proportional to that of partial latent heat.

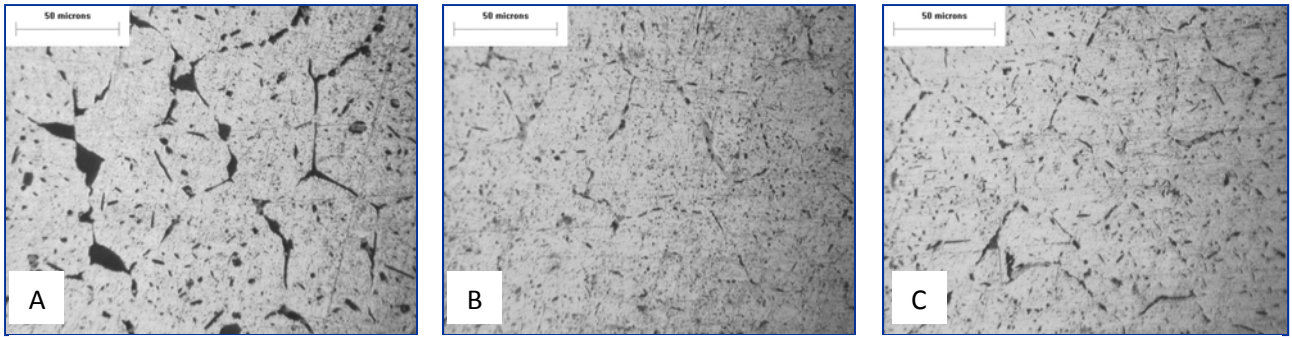


Figure 13 - Microstructures at key depths for comparison to DSC mass fraction results. A) 1 mm depth. B) 3 mm depth and C) 5 mm depth.

In order to verify the results of initial DSC scans, microstructural analysis was conducted at depths corresponding to the major changes in the trend. The microstructures at 1 mm, 3 mm, and 5 mm **(Figure 13)** show visually the changing S-phase mass fraction agreeing closely with the trend plotted above.

Analysis

The data collected using the DSC and metallography conform closely to the S-phase mass fraction trend expected from shrinkage-induced flow. As the ingot solidified, the first material to solidify on the mold wall shrunk inward, causing high solute liquid to be pulled toward the surface, creating the rise in S-phase mass fraction to above 350% of normal bulk values. Consequently, this low-solute solid that shrank inward caused a region just inside the surface, approximately 2-4 mm from the surface in this ingot, with a marked decrease in S-phase, to as low as 25% of bulk values. Further from the melt wall, approximately 5-6 mm deep, S-phase concentrations are at nominal values, and remain reasonably constant into the ingot interior.

The effects of thermo-solutal convection could not be discussed due to testing methodology. Because S-phase is an intermetallic compound with a 1:1 ratio of copper and magnesium, specific concentrations could not be separated with DSC or metallographic results. Similarly, zinc being dissolved in the α-phase solid solution, it was not picked up on the DSC scans. Other characterization techniques would be required to examine these effects. Additionally, floating grain migration was not seen in this experiment, as it is a phenomenon that occurs toward the center of the ingot, rather than at the surface, which was the focus of this project.

Conclusions

1. Nominal alloying element concentrations occur approximately 5 mm from the surface.

2. The S-Phase (Al_2CuMg) mass fraction at the surface appears as high as 3.5 times that of bulk material.

3. S-Phase mass fraction drops drastically to near 0.25 times that of the bulk material at approximately 3 mm from the surface.

4. High S-phase concentrations at the surface suggest an adverse effect on mechanical properties in this region, though the exact effect, because of the complex microstructural evolution through processing is unclear.

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