Dendrite orientation in aluminum magnesium alloys

Erin Hannah Sunseri
Iowa State University

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Dendrite orientation in aluminum magnesium alloys

by

Erin Hannah Sunseri

A thesis submitted to the graduate faculty
in partial fulfillment of the requirements for the degree of

MASTER OF SCIENCE

Major: Materials Science and Engineering

Program of Study Committee:
Rohit Trivedi, Major Professor
Ralph Napolitano
Kai-Ming Ho

Iowa State University
Ames, Iowa
2009

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## DEFINITION OF SYMBOLS

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<th>Definition</th>
<th>Units</th>
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<tr>
<td>$\gamma$</td>
<td>Orientation depended interface energy</td>
<td>$[J(m^2)]$</td>
</tr>
<tr>
<td>$\gamma_0$</td>
<td>Integral mean interfacial energy</td>
<td>$[J(m^2)]$</td>
</tr>
<tr>
<td>$\gamma(\theta)$</td>
<td>Surface energy as a function of $\theta$</td>
<td>$[J(m^2)]$</td>
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<td>$\Delta$</td>
<td>$r_{max}/r_{min}$</td>
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<tr>
<td>$\varepsilon_1$</td>
<td>Anisotropy parameter</td>
<td>---</td>
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<td>$\varepsilon_2$</td>
<td>Anisotropy parameter</td>
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<td>$\varepsilon_4$</td>
<td>Anisotropy parameter</td>
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<tr>
<td>$\theta$</td>
<td>Angle</td>
<td>$[\text{rad}]$</td>
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<td>$\kappa_1$</td>
<td>Curvature at the interface with surface tangent direction 1</td>
<td>$[m^{-1}]$</td>
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<td>$\kappa_2$</td>
<td>Curvature at the interface with surface tangent direction 2</td>
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<td>$\phi$</td>
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<td>$C_0$</td>
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<td>Composition of the solid</td>
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<td>$r_0$</td>
<td>$(r_{max}/r_{min})/2$</td>
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<tr>
<td>$r_{max}$</td>
<td>Circle is the tangent along the $&lt;100&gt;$ direction</td>
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</tr>
<tr>
<td>$r_{min}$</td>
<td>Circle is the tangent along the $&lt;110&gt;$ direction</td>
<td>$[m]$</td>
</tr>
<tr>
<td>$\Delta S_f$</td>
<td>Entropy of fusion</td>
<td>$[J(m^3 \cdot K)]$</td>
</tr>
<tr>
<td>Symbol</td>
<td>Definition</td>
<td>Units</td>
</tr>
<tr>
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<td>------------------------------------</td>
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</tr>
<tr>
<td>S(θ)</td>
<td>Stiffness as a function of θ</td>
<td>[J(m⁻²)]</td>
</tr>
<tr>
<td>T*</td>
<td>Actual temperature</td>
<td>[K]</td>
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<tr>
<td>T_L</td>
<td>Liquidus Temperature</td>
<td>[K]</td>
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<tr>
<td>ΔT_c</td>
<td>Undercooling</td>
<td>[K]</td>
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<tr>
<td>z</td>
<td>Distance from the interface</td>
<td>[µm]</td>
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I would like to thank Dr. Rohit Trivedi for his support and the opportunities he has provided me with while working with him including: research assistantship, travel in the U.S. and abroad, academic discussion, and life lessons. Working with him was a true pleasure.

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ABSTRACT

Aluminum magnesium alloys comprise an important class of alloys often used in the automotive, aerospace, and marine industry because of their low density and good corrosion resistance. These parts are often produced by a casting processing; creating microstructures, most commonly dendrites, that can affect the properties of the material. Critical experiments have been carried out on the Al-Mg system in order to understand how dendrite growth direction varies with composition. Through experimental studies under steady state growth, critical compositions at which the transition in preferred growth direction occurs were examined. The orientations of the resulting dendrites were measured using orientation imaging microscopy (OIM). OIM data showed the primary dendrite trunk was not affect by the change in magnesium composition keeping a (100) orientation throughout. However, there was transition in the secondary dendrite arm orientation from (100) to a (110) that occurred around 15 – 20 wt. % Mg. Though the OIM data suggests only a (100) primary dendrite trunk orientation, at some compositions a 60° angle between the dendrite trunk and side arms was also observed indicating a (110) primary dendrite trunk and (110) secondary arms.
CHAPTER 1: INTRODUCTION

1.1 GENERAL INTRODUCTION

Solidification is a naturally occurring phase transformation that can be observed in nature in something as simple as snowflakes or in manufacturing in casting. Casting make up a large portion of manufactured metallic parts because it is a very economical manufacturing method. Over time, casting has evolved from rudimentary tool making to a highly sophisticated process. It is important to control the casting conditions to optimize the quality of the casting as defects tend to persist though subsequent processes.

Cast aluminum magnesium alloys comprise an important class of alloys called the 3xx.x series. Fundamentally, aluminum has an FCC structure which can be alloyed with HCP magnesium up to 36 wt. % while still maintaining an FCC structure. One of the important properties of these alloys is their low densities making them promising materials in the automotive, railcar, and aerospace industries where weight savings is crucial in an ever demanding need for energy conservation. With the density of these alloys being one third that of steel the energy savings can be significant. Magnesium is alloyed with aluminum to increase the strength without decreasing ductility as long as the magnesium content is kept below ~5 wt. %. These alloys also have good corrosion resistance and weldability.

Casting is commonly used to shape Al-Mg alloys used in applications such as car rims, automotive parts, and marine engine components due to its corrosion resistance. One major issue in solidification is that the solid does not grow isotropically and the growth is enhanced in certain preferred crystallographic direction. For example, in casting, initially equiaxed crystals form with different orientations, but the transition to columnar crystals occurs since the grains with preferred crystallographic direction tend to align with the heat flow direction thereby producing a texture in the material that influences the properties of the cast material. Preferred growth directions are also observed in the growth of a single crystal by the directional solidification technique. In addition, the
equiaxed crystals form dendrites with the dendrite front growing in the preferred crystallographic direction.

The understanding of the preferred growth direction of crystals is important for the technological applications and for the fundamental science that can give an insight into the physics that lead to the preferred growth direction. For example, the single crystal of Terfenol-D (Tb-Dy-Fe) grows in <001> direction, whereas the crystal growing in <111> direction would have superior magnetostriction properties. Significant work has been done to predict the preferred growth direction of dendrites in a solidified material. Dendrites are produced by instabilities in the solid liquid interface and are common in solidified metal alloys. These instabilities form in specific crystallographic direction. Initially, the preferred growth direction was attributed to crystal structure\(^1\), however, dendrites in FCC Al-Zn and Al-Mg alloys have been found to form in different crystallographic directions and the orientation is found to depend on the composition of the alloy. As more appropriate criterion has been examined, and it is proposed that the preferred growth direction is governed by the anisotropy in interfacial properties. It has been predicted that the anisotropy of the interfacial energy is responsible for the dendrite growth directions at low velocities, while, in at very high growth rates the dendrite growth directions are more influenced by the anisotropy in interface kinetics\(^2\).

A systematic experimental study on the dendrite growth direction as a function of composition has been carried out only in one alloy system of Al-Zn in which the dendrite growth direction is observed to change from <001> direction to <011> direction as the composition of Zn is increased\(^3,4,5\). Near the transition conditions, different orientations of dendrites have been identified. Since no information on the variation of interface anisotropy as a function of composition is available, these results have been compared with the general model of the effect of anisotropy in interface energy on the preferred growth direction.

The goal of this research was to carry out critical experiments in Al-Mg system to determine if the preferred growth direction is dependant upon composition. The critical
composition at which the transition in preferred growth direction occurs will be examined and the sharpness or the diffuseness of the transition will be determined. The transition zone was determined where dendrites within the sample appeared to be in competition with each other forming secondary arms in multiple directions on the same dendrite trunk. In order to examine these aspects, directional solidification experimental studies were carried out in Al-Mg alloys over the composition range of 5.0 – 30.0 wt. % magnesium. The orientations of the resulting dendrites were measured using orientation imaging microscopy (OIM). It was found that the secondary dendrite arms underwent change in orientation from <001> to <110> with increasing magnesium content; with the transition occurring around 20 wt. % magnesium. The primary dendrite trunk remained in a (100) orientation when characterized by OIM.

1.2 THESIS ORGANIZATION

This thesis is written in a concise, logical manner in which the figures and tables are embedded within the text as referenced. In Chapter 2, the fundamental theory and key objectives of dendrite growth directions are discussed. The experimental objectives were defined and detailed experimental design was described in Chapter 3. Experimental results and discussion were presented in Chapter 4. The overall impact of the research is concluded in Chapter 5, with future work discussed in Chapter 6. Work done with Prof. Michel Rappaz in Lausanne, Switzerland will be discussed in the appendix.

REFERENCES


CHAPTER 2: BACKGROUND

2.1 DENDRITE GROWTH DIRECTIONS

The model of preferred growth direction of dendrites was first proposed by Weinberg and Chalmers\(^1\), who predicted that dendrites would grow in the direction that will form low energy interfaces. Their experiments in Pb, Sn, and Zn all showed dendrite arm growth in the direction of the close packed planes. Thus, in cubic crystals the growth direction will be \(\langle 001 \rangle\) since it will generate low energy (111) planes. In cubic systems the dendrite arms will also form in \(\langle 001 \rangle\) directions, perpendicular to the dendrite trunk. Based on this proposal the following dendrite growth directions are predicted: (100) in BCC and FCC, (110) in BCT and (10\(\overline{1}0\)) in HCP crystals. For dendrites forming in the basal plane the side branches should form at an angle of 60° with the direction of the trunk. Although these directions are generally found in most experiments, the growth directions are based on the crystal structure only. However, in some systems, such as Al-Zn, different growth directions have been observed as a function of composition, while the crystal structure remains the same. Consequently, a more general criterion is needed to predict the dendrite growth direction.

A more general criterion for the selection of the growth direction should be based on the anisotropy in interface properties. This includes the anisotropy in interface energy and in atomic attachment kinetics at the interface. It has been shown that the anisotropy of the interfacial energy is responsible for the dendrite growth directions at low velocities, while the anisotropy in interface kinetics becomes important only under rapid solidification conditions in metals\(^2,3\).

The predicted (100) orientation in cubic systems occurs due to the anisotropy in interfacial energy, which causes increased growth rates along that direction. In directional solidification at a constant velocity, dendrites growing along the (100) directions have a slightly higher undercooling and as a result grow more rapidly and crowd out crystals
with other orientations. The higher undercooling along the (100) direction is due to higher capillary undercooling that corresponds to the direction in which the stiffness is minimum.

Assuming dendrites will select growth directions where capillary forces are the weakest thereby limiting smoothing of the interface, stiffness and curvature can be related to the local equilibrium temperature through the Gibbs-Thomson condition. Herring\textsuperscript{4} relates undercooling due to interfacial energy as:

\[
\Delta T_c = \frac{1}{\Delta S_f} \left[ \left( \gamma + \frac{\delta^2 \gamma}{\partial n^2} \right) \kappa_1 + \left( \gamma + \frac{\delta^2 \gamma}{\partial n^2} \right) \kappa_2 \right] \tag{1}
\]

where $\Delta S_f$ is the entropy of fusion, $\gamma$ is the orientation depended interface energy, and subscripts 1 and 2 indicate the surface tangent directions of the two principle curvatures $\kappa_1$ and $\kappa_2$. The term $\left( \gamma + \frac{\delta^2 \gamma}{\partial n^2} \right)$ represents the stiffness of the interface at a given orientation.

\textbf{2.2 TWO DIMENSIONAL MODEL}

The anisotropy in interface energy was initially considered for two-dimensional growth with interface energy varying with orientation in a plane perpendicular to the (100) direction; exhibiting four-fold symmetry. The interface variation was written as

\[
\gamma(\theta) = \gamma_0 (1 + \varepsilon_4 \cos 4\theta) \tag{2}
\]

where $\gamma_0$ is the mean value of $\gamma$, and the angle $\theta$ is measured from the <001> direction.

The anisotropy in interface energy was measured experimentally by Liu et al.\textsuperscript{5,6} in Al 4.0 wt. % Cu. To ensure ease of analysis, a single crystal with a (001) orientation in the growth direction was used so that the other four <001> directions would lie in the transverse section. Thus, a transverse section made at the middle of the droplet will give variation in interface energy with orientation in the (100) plane. The equilibrium
conditions were established by measuring composition in the solid that was found to be uniform with no concentration gradient.

To determine the anisotropy parameter the equilibrium shape of a liquid droplet in a solid was measured, shown in Fig. 2.1. In order to quantitatively measure the anisotropy of the droplet, two tangential circles were superimposed over the image. The solid circle that is tangent along the <100> direction corresponds to \( r_{\text{max}} \) and the dotted circle tangent along the <110> direction denotes \( r_{\text{min}} \). For small \( \varepsilon_4 \), the measured equilibrium shape on (100) section follows the same four-fold variation as interface energy variation, i.e.,

\[
  r \equiv r_0 (1 + \varepsilon_4 \cos 4\theta)
\]  

(3)

Thus, each cross section was analyzed in the (100) plane to determine, \( \Delta = r_{\text{max}} / r_{\text{min}} \), which gives

\[
  \Delta = \frac{1 + \varepsilon_4}{1 - \varepsilon_4}
\]  

(4)

The anisotropy parameter, \( \varepsilon_4 \), is thus given by:

\[
  \varepsilon_4 = \frac{\Delta - 1}{\Delta + 1}
\]  

(5)

From eq. (2), the stiffness is given by

\[
  S(\theta) = \gamma_0 (1 - 0.1455 \cos 4\theta)
\]  

(6)

Stiffness is at a minimum when \( \theta = 0 \), or in the <001> growth direction, therefore, \( \varepsilon_4 \) the anisotropy parameter, is considered to be positive so the value of \( \gamma \) is at a maximum.

From the work done by Liu et al.\(^{5,6}\), the anisotropy parameter was determined to be \( \varepsilon_4 = 0.0097 \). Equation 7 below, describes how surface energy varies with orientation. Equation 8 describes the stiffness, or the anisotropy in surface energy. Equation 9 shows inverse stiffness, which relates orientation with preferred growing directions.
\[ \gamma(\theta) = \gamma_0 \left( 1 + 0.0097 \cos 4\theta + ... \right) \]  \hspace{1cm} (7) \\
\[ S(\theta) = \gamma_0 \left( 1 - 0.1455 \cos 4\theta \right) \]  \hspace{1cm} (8) \\
\[ \frac{1}{S(\theta)} = \frac{1}{\gamma_0} \left( 1 - 0.1455 \cos 4\theta \right) \]  \hspace{1cm} (9)

Stiffness and inverse of stiffness show that $<001>$ direction has the lowest stiffness and thus, will be the preferred dendrite growth direction. To predict a change in preferred growth direction a three dimensional model is necessary. Fig. 2.2 – 2.4 show visually what equations 6-8 predict mathematically. The plot of the inverse of stiffness (Fig. 2.4) shows that the preferred growth direction is along the $<001>$ directions.
Fig. 2.1 Micrograph of equilibrium shape droplet with circles for anisotropy measurements overlaid.
Fig 2.2 Polar plot of $\gamma/\gamma_0$.

Fig 2.3 Polar plot of $S/\gamma_0$; “Stiffness”.

Fig 2.4 Polar plot of $\gamma_0/S$. 
2.3 THREE DIMENSIONAL MODEL

As previously mentioned, the two dimensional model assumes fourfold symmetry in cubic systems. However, many other dendrite growth directions exist creating a need for a three dimensional $\gamma$ plot that can accommodate such systems as Al-Mg and Al-Zn which have been found to change dendrite orientation with composition. In cubic systems the interface energy, $\gamma(n)$, where $n$ is the normal direction to the interface, can be described by the following equation:

$$\gamma(\theta,\phi) = \gamma_0 (1 + \varepsilon_1 K_1(\theta,\phi) + \varepsilon_2 K_2(\theta,\phi))$$  \hspace{1cm} (6)

where $K_1$ and $K_2$ are cubic harmonics, which are combinations of the standard spherical harmonics, $\gamma(\theta,\phi)$ for cubic symmetry. This is a departure from the two-dimensional case where only the first cubic harmonic with positive $\varepsilon_1$ was considered in the prediction of dendrite growth in the <100> direction. Studies have shown that not only the first cubic harmonic but also the second term in the expansion is important for describing the interface energy anisotropy. Karma et al.\textsuperscript{7} has demonstrated that a positive $\varepsilon_1$ favors a (100) orientation, whereas, a negative $\varepsilon_2$ favors a (110) orientation; creating a variety of preferred crystallographic growth directions. These preferred directions are imposed by the stiffness minimum, which, plotted as inverse stiffness in a polar plot, will show protrusion in the preferred growth directions. The inverse stiffness plot shown in Fig. 2.4, has a positive $\varepsilon_1$ with $\varepsilon_2$ equal to zero.

Studies done by Rappaz and co-workers\textsuperscript{8,9} in Al-Zn have shown a change in dendrite growth orientation depending on composition. At low Zn concentrations a (100) orientation was observed and at high concentration of Zn a (110) orientation existed. Phase field calculations have shown that when $\varepsilon_1$ is positive and $\varepsilon_2$ is negative but small, (100) orientations show bumps on the inverse stiffness plot, predicting the (100) growth direction. In contrast, a (110) orientation is predicted when a more negative value of $\varepsilon_2$ is used. This change in orientation with composition is correlated with experimental results in the Al-Zn system. Haxhimali et al.\textsuperscript{10} has developed a more rigorous criterion for
dendrite growth, indicating anisotropy in the interface energy controls dendrite growth at low velocities while anisotropy in interface kinetics controls dendrite growth at very high velocities.

The predicted (100) orientation in cubic systems occurs due to the anisotropy in interfacial energy, which causes increased growth rates along that direction. Phase field calculations have also shown that when $\varepsilon_1$ is positive and $\varepsilon_2$ is negative but small, (100) orientations show bumps on the inverse stiffness plot, predicting the (100) growth direction, whereas, a (110) orientation is predicted when a more negative value of $\varepsilon_2$ is used.

In the two-dimensional model the anisotropy in interface energy was represented by one parameter only, i.e. $\varepsilon_4$. The relationship (2) is still valid for the shape on the (100) plane in the 3D model, but the parameter $\varepsilon_4$ can be shown to be related to the parameters $\varepsilon_1$ and $\varepsilon_2$, as:\textsuperscript{11}

$$\varepsilon_4 = (\varepsilon_1 + 3\varepsilon_2) / 4$$

(10)

Thus, experimental measurements of anisotropy parameters $\varepsilon_1$ and $\varepsilon_2$ require the determination of the 3D equilibrium shape or the equilibrium shape on two different planes.
Fig. 2.5 Orientation selection map from minimum interfacial stiffness. There is a continuous degeneracy of orientation on this line where all directions contained in (100) planes have equal stiffness minima.\(^7\)
Fig. 2.6 Inverse stiffness plots: (a) (100) orientation (b) (110) orientation.
REFERENCES


CHAPTER 3: EXPERIMENTAL STUDIES

3.1 EXPERIMENTAL OBJECTIVES AND OVERVIEW

To examine the effect of composition on dendrite growth direction, experiments were carried out in the Al-Mg alloy using the Bridgman furnace, Fig 3.1 - 2. The compositions studied, between Al - 5 wt. % Mg and 30 wt. % Mg, can be referenced from the phase diagram, Fig. 3.3. The phase diagram shows the formation of $\Delta$-Al dendrites over a composition range 0 - 30 wt. % Mg where the $\Delta$-phase is FCC over this region. Steady-state growth velocity experiments were performed using alumina tubes. The angles between the primary and secondary dendrite arms were analyzed as a function of composition for all compositions studied.

3.2 ALLOY PREPERATION

Different compositions of Al-Mg were studied including 5, 10, 15, 20, 21, 23, 25, 27, 29, 30 wt. % Mg which can be referenced from the phase diagram, Fig. 3.3. The aluminum used to prepare these alloys was 99.99% pure and the magnesium was 99.98% pure. Aluminum typically contains impurities such as Si, Fe, and Cu in the range of 1-5 ppm. Magnesium typically has impurities including: Al, Fe, Si, Zn, Ni, Pb, Mn, Cu and Ca; with the highest levels approximately 30 ppm. All alloys were prepared by chill casting one inch ingots then, using electrical discharge machining (EDM), cut into 4x4mm sections longitudinally. Samples were cut using EDM because they could not be swagged due to the fact they were brittle and would crack.

3.3 DIRECTIONAL SOLIDIFICATION EXPERIMENTS

Critical experiments have been performed using the Bridgman apparatus, Fig. 3.1 - 3.2. For solidification to occur heat must be extracted from the melt creating an external heat flux. There are several methods of heat extraction including directional solidification (Bridgman Type). In this process the alloy is pulled upward through a temperature
gradient (G) at a constant velocity (V). A small diameter ampoule is necessary to minimize convention effects. The directional solidification unit, Fig. 3.1, consists of an alumina ampoule centered within a furnace (top) and a liquid metal coolant chamber (bottom) containing In-Ga-Sn ternary eutectic alloy. The thermal gradient created by the external heat flux is shown in Fig. 3.2. The furnace and coolant chamber are secured to a metal structure, known as the carriage. The carriage is attached to a threaded shaft that is controlled by a belt connected to a step motor. Controlled electronically, the carriage can be set in motion at a constant velocity while the ampoule remains fixed in an inert atmosphere. This Bridgman process has been selected to study how dendrite orientation changes with composition since the parameters of alloy composition, temperature gradient, and velocity can be controlled independently.

An alumina ampoule ($\Omega_{\text{ID}} = 5.5\,\text{mm}, \Omega_{\text{OD}} = 7.0\,\text{mm}$) was used for all experiments. An alumina ampoule was chosen versus a quartz ampoule due to the fact that silica reacts with aluminum forming alumina silicates. A temperature control module was used to impose the necessary thermal conditions, heat flux, on the ampoule. A high temperature furnace (<1000 °C) was allowed to heat up to 900 °C while a cooling chamber, consisting of a direct immersion bath of liquid metal (Ga-In-Sn ternary eutectic) was cooled by a continuous flow of water at 15 °C. A custom fabricated lava rock piece created an adiabatic zone below the furnace but above the cooling chamber. The alumina ampoule was evacuated and backfilled with an inert argon atmosphere to approximately 10 psi to prevent oxidation of the alloy.

After the system had reached 900 °C it was allowed to equilibrate for 20 minutes to ensure a consistent melt. A computer-controlled step motor moved the carriage upward at a predetermined velocity while the sample ampoule remained at a constant position. After the carriage was set in motion, direction growth proceeded in the vertical direction. The experiments conducted all used steady-state velocity, meaning the velocity remained constant throughout the duration of the experiment. In steady state experiments, those in which the velocity remains constant, the interface velocity will be equal to the
Fig. 3.1 Schematic of the Bridgman directional solidification unit. The furnace is shown in red, and the coolant chamber is shown in blue\(^1\).

Fig. 3.2 Schematic of the thermal conditions within the Bridgman directional solidification unit\(^1\).
Fig. 3.3 Al-Mg phase diagram.
furnace velocity. The velocity for all experiments was set at 80 [µm·s⁻¹] as this proved to be ideal for producing dendrites.

When solidification was completed the sample was quenched in the liquid metal eutectic, and then broken at the top of the ampoule using compressive force to allow it to be removed from the furnace. The sample was then removed from the ampoule and mounted in epoxy resin. The mounted samples were ground using SiC paper (grit size: 400 – 1200) and then polished using alumina-water slurry (alumina powder size: 0.3 µm) on a velvet polishing cloth. The samples were finished off using colloidal silica to polish to a mirror finish. After final polish, the mounted samples were washed with methanol and air-blown dry. The samples were etched with a 4 grams of NaOH, 200 milliliters water solution for 7 minutes.

The polished samples were observed using optical microscopy. A one centimeter region around the observed interface was then removed by a diamond blade saw and was used for orientation image mapping (OIM) analysis to determine the dendrite arm growth directions in relation to the dendrite trunk.

Before OIM could take place the sample had to be first ion milled for approximately ten minutes with the following conditions: 15° sample tilt, 4.5kV, 0.3 ma, on a rotating stage. Following ion milling, the sample was electropolished using a solution of 300ml methanol, 175 ml butyl alcohol, and 50 ml perchloric acid at a temperature of approximately -25°C at 30V DC for 5 seconds.

3.4 EQUILIBRIUM SHAPE EXPERIMENTS

To conduct equilibrium shape experiments directional solidified sample of Al 15 wt. % Mg and Al 5 wt. % Mg were made in the same manner as described in section 3.2. These compositions were chosen as they bracket the solidus region on the phase diagram. The sample preparation departed from section 3.2 once the samples were extracted from the alumina tube. At this point they were then cut using a diamond blade saw into 1 cm sections. These sections were then placed in a three zone furnace at a temperature that
allowed approximately 5% liquid to form within the sample. Each zone of the furnace was set to slightly different temperature (~3 °C apart) as there is always some discrepancy in the phase diagram. Since this experiment was based on the liquid droplets achieving equilibrium shapes approximately 5 weeks was allowed before the samples were water quenched and removed from the furnace. The samples were then mounted in epoxy resin and polished in the same manner as described in section 3.2. The polished samples there then inspected, using a scanning electron microscope (SEM), for characterizing droplet shapes.

Though both compositions were attempted, only the Al 15 wt. % Mg was successful. As magnesium oxidizes very quickly at high temperatures, it was difficult to keep the furnace under an inert atmosphere for five weeks. Several modifications had to be made to the furnace, most importantly installing an argon tank specifically for the three zone furnace. Upon completion of the experiment using Al 15 wt. % Mg, the SEM showed liquid coalescence; however, due to the fragile nature of these particles, they most likely broke during polishing creating a very sharp edge around each equilibrium shape. The SEM uses an electron beam, so when the beam hit this sharp edge it likely bounced back directly into the detector creating a sort of white halo around the equilibrium shape, obscuring the edge and making precise measurements impossible.

REFERENCES

CHAPTER 4: DENDRITE ORIENTATION STUDIES

4.1 INTRODUCTION

Dendrites are common microstructures existing in cast metal parts. Formed by instabilities in the solid liquid interface, they grow along directions corresponding to the maximum curvature in the equilibrium shape of a system. These maximum curvature regions are consistent with minima in the stiffness plots of the solid-liquid interfacial energy as shown in Fig. 2.3 – 2.4.

Aluminum alloys are of great technological interest and have a very low solid-liquid interfacial energy anisotropy. Studies done on Al-Si and Al-Cu alloys show dendrite trunks grow primarily in the <100> directions, but the secondary arm growth directions are biased towards the temperature gradient. Napolitano et al.\textsuperscript{1,2} examined the equilibrium shape of liquid pockets in Al alloys and determined the anisotropy of the interfacial energy of the solid liquid interface to be on the order of 1 percent, leading to the conclusion that the low anisotropy was responsible for the variety of microstructures observed in aluminum alloys\textsuperscript{3,4}.

Herenguel et al.\textsuperscript{5} was the first to study unusual dendrite morphology in feathery grains or twinned dendrites. Many years later, Henry et al.\textsuperscript{3} showed these twinned dendrites were in fact <110> dendrites split by a (111) twin plane with <110> arms extending on both sides. Work was also done on Al – 45 wt. % Zn hot dipped coatings on steel\textsuperscript{4}. Dendrites found in this study were strange in the fact that a (100) plane would not exhibit 4 <100> dendrites but instead showed eight <320> growth directions. These results have been explained by Henry\textsuperscript{3} and Sémoraz\textsuperscript{5} by a change in the weak anisotropy of interfacial energy of aluminum, which may have occurred by an attachment kinetic contribution or induced by additional solute elements.

Based on these findings, we have decided to further investigate the dendrite growth directions of the Al-Mg system. This system was chosen as it bears many similarities to
the Al-Zn system in that Mg is has an HCP crystal structure much like Zn. By increasing
the Mg concentration one can see the effects of composition on FCC Al dendrite
orientation.

4.2 EXPERIMENTAL PROCEDURE

Directional solidification studies were carried out using a Bridgman furnace and Al-Mg
alloys containing varying amounts of Mg. The Bridgman furnace was used to achieve a
stable thermal gradient and the liquid metal bath provided fast quenching. Solidification
microstructures were investigated using both optical and OIM analysis. Dendrite arm
angle in relation to the dendrite trunk were carried out using ImagePro software.

4.3 DENDRITE ORIENTATION RESULTS

Two different orientations of dendrites were observed, one at lower compositions and the
other at higher compositions. In Fig 4.1, notice the shift in the orientation of the dendrite
arms between Al 10 wt. % Mg and Al 30 wt. % Mg, from 90° to 45° with respect to the
dendrite trunk. This shows the outer edges of the compositions studied and we will now
explore what happens in between these two compositions. Detailed studies with different
compositions were carried out to establish the range of compositions over which each of
the above orientation is present, and to investigate the orientations that may be present in
the transition regime.

Fig. 4.3 – 4.12 show the OIM results of the alloys studied. The data was taken over the
entire area shown in the confidence index of each composition. The confidence index
(CI) is a parameter that is calculated when the diffraction patterns are automatically
indexed. The software ranks the diffraction patterns detected with possible orientation
solutions. The CI range goes from 0 to 1. One can notice by looking at the pole figures of
each composition that all of the primary dendrite trunks are aligned along the <100>
direction. The sample set-up inside the OIM was consistent with Fig. 4.2.
Fig 4.1 (a) Micrograph of Al 10 wt. % Mg, notice the dendrite arms are nearly perpendicular to the trunk indicating a (001) orientation. (b) Micrograph of Al 30 wt. % Mg, notice the dendrite arms at 45° from the trunk which is indicative of a (100) dendrite trunk orientation with (110) dendrite arm orientation.
Fig. 4.2 Diagram of OIM sample set up; TD indicating the transverse direction, RD indicating the rolling direction, and ND the normal direction.
Fig. 4.3 Al 5 wt% Mg (a) confidence map with reds and oranges showing areas of higher confidence, blues and greens show lower confidence (b) (001) pole figure showing the primary dendrite trunk in the (001) direction and (011) pole figures further confirms this fact.
Fig. 4.4 Al 10 wt% Mg (a) confidence map with reds and oranges showing areas of higher confidence, blues and greens show lower confidence (b) (001) pole figure showing the primary dendrite trunk in the (001) direction and (011) pole figures further confirms this fact.
Fig. 4.5 Al 15 wt% Mg (a) confidence map with reds and oranges showing areas of higher confidence, blues and greens show lower confidence (b) (001) showing the primary dendrite trunk in the (001) direction along TD and (011) pole figures further confirms this fact.
Fig. 4.6 Al 20 wt% Mg (a) confidence map with reds and oranges showing areas of higher confidence, blues and greens show lower confidence (b) (001) showing the primary dendrite trunk in the (001) direction along TD and (011) pole figures further confirms this fact.
Fig. 4.7 Al 21 wt% Mg (a) confidence map with reds and oranges showing areas of higher confidence, blues and greens show lower confidence (b) (001) showing the primary dendrite trunk in the (001) direction along TD and (011) pole figures further confirms this fact.
Fig. 4.8 Al 23 wt% Mg (a) confidence map with reds and oranges showing areas of higher confidence, blues and greens show lower confidence (b) (001) showing the primary dendrite trunk in the (001) direction along TD and (011) pole figures further confirms this fact.
Fig. 4.9 Al 25 wt% Mg (a) confidence map with reds and oranges showing areas of higher confidence, blues and greens show lower confidence (b) (001) showing the primary dendrite trunk in the (001) direction along TD and (011) pole figures further confirms this fact.
Fig. 4.10 Al 27 wt% Mg (a) confidence map with reds and oranges showing areas of higher confidence, blues and greens show lower confidence (b) (001) showing the primary dendrite trunk in the (001) direction along TD and (011) pole figures further confirms this fact.
Fig. 4.11 Al 29 wt% Mg (a) confidence map with reds and oranges showing areas of higher confidence, blues and greens show lower confidence (b) (001) showing the primary dendrite trunk in the (001) direction along TD and (011) pole figures further confirms this fact. The dendrite trunk is coming slightly out of the plane of the sample, causing the pole figure to shift slightly away from the (100) orientation.
Fig. 4.12 Al 30 wt% Mg (a) confidence map with reds and oranges showing areas of higher confidence, blues and greens show lower confidence (b) (001) showing the primary dendrite trunk in the (001) direction along TD and (011) pole figures further confirms this fact.
To construct Table 4.1 and Fig. 4.13, optical micrographs were used in conjunction with ImagePro software. The software was used to apply an initial line down the center of the primary dendrite trunk, adding additional lines connecting the secondary dendrite arms to the trunk. This angle was then measured and reported in the table below.

Since we know the primary dendrite trunks analyzed by OIM lie in the <100> direction we see that dendrite arms of Al 5 wt. % Mg and Al 10 wt. % Mg are approximately 90° from the trunk. This is expected, indicating a (100) trunk with (100) dendrite arms. The microstructure becomes less defined at Al 15 wt. % Mg where the angles are around 77°. This is an unexpected angle but when looking at the optical image, Fig. 4.14, one can see why this discrepancy may occur as the Al 15 wt. % Mg the dendrites are not clearly defined. It almost appears as if there is competition between the (100) and (110) orientation, with some arms appearing nearly perpendicular to the dendrite trunk, while others at an obvious angle. This composition is where the transition from (100) secondary arms to (110) secondary arms begins. Moving on to Al 20 wt. % Mg, the primary dendrite trunks can be clearly seen in Fig. 4.15, however, some secondary arms still seem to have a bit of coarsening at the dendrite arm tips indicating a competition in preferred orientation may exist. Al 21 wt. % Mg seems to follow the same trend. Both of these compositions have angles around 64° indicating that the measurements may have been taken from a primary dendrite trunk of (110) orientation with (110) secondary arms or the dendrites may still be undergoing a transition creating this intermediate angle. Though the prediction of (110) dendrite trunk and (110) secondary arms contradicts the pole figures, keep in mind the pole figures are just an analysis of a single dendrite and others may exist that were overlooked in analysis. By the time the composition reaches 23 wt. % Mg, Fig. 4.16, the dendrite arm angle is approximately 45°, which is consistent with a (100) primary trunk and (110) side branches. Al 25 wt. % Mg shows one 45° angle and one 55° angle. The 45° angle would again be similar the 23 wt. % Mg sample while the 55° angle would most likely indicate a (110) primary trunk and (110) secondary arms. The final two compositions of Al 20 wt. % Mg and Al 30 wt. % Mg both show angles of nearly 45° which is to be expected.
There are several sources for error in the angle measurements between the primary dendrite trunk and the secondary arms. Since the angles were measured by hand using ImagePro software the placement of the lines used to calculate the angles is up to interpretation and thus error inherently occur. Errors can also arise when the samples are polished. Since the samples are polished by hand the dendrites may be growing into or out of the polished plane causing foreshortening of the dendrite, or a lower angle measurement than would be expected had the dendrite been perfectly flat in the polished plane. Figure 4.13 below, shows the average angle between the dendrite trunk and arms as well as the standard deviation in the measurement. Notice that the variation seems to peak in the middle compositions around 15-20 wt. % Mg where the transition from (100) to (110) occurs.
<table>
<thead>
<tr>
<th>Composition</th>
<th>Dendrite 1</th>
<th>Dendrite 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Al 5% Mg</td>
<td>85.4</td>
<td>84.8</td>
</tr>
<tr>
<td>Al 10% Mg</td>
<td>83.8</td>
<td>84.4</td>
</tr>
<tr>
<td>Al 15% Mg</td>
<td>77.2</td>
<td>78.1</td>
</tr>
<tr>
<td>Al 20% Mg</td>
<td>63.1</td>
<td>64.2</td>
</tr>
<tr>
<td>Al 21% Mg</td>
<td>64.2</td>
<td>--</td>
</tr>
<tr>
<td>Al 23% Mg</td>
<td>48.3</td>
<td>48.8</td>
</tr>
<tr>
<td>Al 25% Mg</td>
<td>45.8</td>
<td>55.6</td>
</tr>
<tr>
<td>Al 27% Mg</td>
<td>53.9</td>
<td>61.5</td>
</tr>
<tr>
<td>Al 29% Mg</td>
<td>45.8</td>
<td>--</td>
</tr>
<tr>
<td>Al 30% Mg</td>
<td>45.1</td>
<td>46.1</td>
</tr>
</tbody>
</table>

Table 4.1 Summary of dendrite arm angle with respect to the dendrite trunk.
Fig. 4.13 Plot of how dendrite arm angle with respect to the primary dendrite trunk changes with magnesium composition. Error bars indicate the standard deviation. Notice a larger standard deviation in the middle range of compositions where the transition from (100) to (110) occurs.
Fig. 4.14 Al 15 wt. % Mg optical micrograph showing areas where angle measurements were taken. Notice the dendrite arms seem to be in conflict with each other as some appear perpendicular and some appear angled with respect to the dendrite trunk.
Fig. 4.15 Al 20 wt. % Mg optical micrograph showing areas where angle measurements were taken. Notice dendrite arms are much better defined than in Al 15 wt. % Mg but there still appears to be some discrepancy at the dendrite arm trips.
Fig. 4.16 Al 23 wt. % Mg optical micrograph showing areas where angle measurements were taken. Notice the dendrite arms are clearly defined as compared with the previous two images.
4.4 COMPARISON WITH Al-Zn

Studies have been done by Rappaz\textsuperscript{6,7} in Al-Zn to study the effects of Zn composition on dendrite orientation. It was seen that at low Zn compositions a (100) orientation is seen, whereas, at high Zn compositions a shift in orientation to (110) occurs with a transition zone around 20-25 wt. % Zn. Figure 4.17 below, of Al 90 wt. % Zn, looks very similar to dendrites observed in Al 23-30 wt. % Mg samples (Fig. 4.8-12) with their secondary arms at a high angle from the dendrite trunk. The change in angle is linked to Zn composition in Fig. 4.18.

These results are similar to those obtained in this study of Al-Mg with a few key differences. In the work done in Al-Zn a shift is seen in the primary dendrite trunk and secondary arms from (100) to (110) with increasing Zn composition. In Al-Mg a change was also observed, however, the change only occurred in the secondary dendrite arms. As the Mg composition increased the dendrite arms changed from (100) to (110) while maintaining a primary dendrite trunk with a (100) orientation. This difference needs to be investigated further by looking at the anisotropy and surface energy parameters through equilibrium shape studies.
Fig. 4.17 Al 90 wt. % Zn dendrites with 60° angle between dendrite trunk and secondary arms indicating a (110) orientation\(^6\).

Fig. 4.18 Angle between the direction <100> and the growth direction of Al-Zn dendrites as a function of composition\(^6\).
REFERENCES


CHAPTER 5: CONCLUSIONS

Critical experiments have been carried out on dendrite growth orientation in Al-Mg alloys. Careful experiments have shown a transition between (100) secondary dendrite arm orientation to a (110) orientation. Though the OIM data suggests only (100) primary dendrite trunk orientation, at some compositions a 60° angle was observed indicating a (110) primary dendrite trunk and (110) secondary arms. These results are similar to those obtained in Al-Zn studies in the fact that both show a shift in orientation with increasing composition. These results provide critical information required for interpreting the effects of composition on dendrite orientation. They can be used to compare with theoretical models of the role anisotropy and surface energy in dendrite orientation. In turn, given an understanding of how composition influences anisotropy and interface energy composition effects can then be predicted in Al-Mg alloys.
CHAPTER 6: FUTURE WORK

Dendrite growth directions are governed by anisotropy and interfacial energy parameters. Thus, further experimental studies are required to determine parameters $\varepsilon_1$ and $\varepsilon_2$ by characterizing the equilibrium shape as a function of magnesium composition.

One major issue in Al alloy casting is the formation of feathery dendrites in certain compositions. Twins form at the dendrite trip of feathery dendrites and this is completely understood, thus, the role of composition and dendrite orientation that gives rise to the twin interface needs to be examined quantitatively.
APPENDIX: TEXTURE AND MICROSTRUCTURE ANALYSIS OF Al-Mg ALLOYS

A.1 INTRODUCTION

Aluminum, having a weakly anisotropic interfacial energy (-1%), is strongly affected by the addition of a HCP element with high anisotropic interfacial energy such as Mg or Zn. Since interfacial energy anisotropy controls the equilibrium shape of a crystal it can be used to predict and describe dendritic morphology, either columnar or equiaxed.

Recent studies by Gonzales et al.\textsuperscript{1} have shown that in the absence of convection and within a gradient of 30 [K·cm\textsuperscript{-1}] with a solidification speed of 0.05 [mm·s\textsuperscript{-1}], a smooth transition in a (001) plane from <100> dendrite orientation towards <110> dendrite orientation has been observed in the Al-Zn binary system from 20 to 70 wt% Zn, with formation of seaweed structures between 30 and 50 wt Zn. Feathery grains were observed in the range of 10 to 40 wt. % Zn with dendrites growing along <110> directions. Such a structure has been observed in Al-Cu, Al-Ni alloys and industrial - type aluminium alloys\textsuperscript{2}.

A.1.2 Experimental Objectives

The present investigation is aimed to determine dendrite growth directions in Al-Mg alloys directionally solidified under the same thermal conditions imposed to study the Al-Zn system\textsuperscript{1}. A combination of optical microscopy and EBSD measurements were implemented to achieve this objective.

A.2 BACKGROUND

In order to study solidification phenomenon, several setups have been developed to simplify the problem while having well controlled conditions and quantifiable results:
Bridgman and directional solidification. In Bridgman solidification the ampoule is drawn downward through a constant temperature gradient, G, at a uniform velocity, V. Directional solidification (DS) provides for a larger sample than Bridgman but microstructure is not uniform throughout because the growth rate and the temperature gradient decrease as distance from the chill increases. DS can be thought as one-dimensional solidification because heat is only extracted through the bottom of the mold. Experimental work done in this study only includes directional solidification.

In the case of directional solidification, the interface is initially stable and a positive temperature gradient exists. Depending on thermal conditions, a planar front or dendritic patterns can form upon solidification. If the actual temperature is less than $T_L$, there will be no supercooling and thus the planar interface will remain stable. If the actual temperature is greater than $T_L$ near the interface, dendrites will form due to undercooling. As heat is extracted from the solid phase attached to the mold, the liquid near the S-L interfaces is undercooled, which means that the actual temperature of the melt is below the equilibrium freezing temperature.

As seen in Fig. A.2.1, the dendrite tips are at temperature $T^*$ which is below the liquidus temperature $T_L$, thus an undercooling exists.
Fig. A.2.1 Al-Mg phase diagram. The dendrite tips are at temperature $T^*$ which is below the liquidus temperature $T_\text{l}$, thus an undercooling exists.
During alloy solidification there is a change in concentration ahead of the interface. As solidification occurs the rejected solute will pile up ahead of the interface creating a diffusion boundary layer. This change in concentration will affect the local liquidus temperature, $T_L$, of the liquid. The liquidus temperature can be related to composition by Eqn. 1,

$$T_L(C_0) - T_L = m(C_0 - C_L)$$

where $T_L(C_0)$ is the liquidus temperature of the initial alloy composition. Shown in Fig. A.2.2, the liquidus temperature increases with increasing distance, $z$, when the value of $k$, $C_S/C_L$, is less than 1.

As small amounts of liquid solidify the equilibrium freezing temperature can be tracked along the $T_L$ line on the graph. The actual temperature, $T^*$, is due to the temperature gradient within the casting. The difference between these two temperatures is the constitutional undercooling.

When directional solidification occurs, the first to form are randomly oriented nuclei near the mold walls. As these nuclei grow, a positive temperature gradient allows for columnar growth as the latent heat is dissipated through the mold. Those dendrites with a preferred orientation (i.e. parallel and opposite to the heat flow direction) will advance, eventually eliminating non-preferred orientations. This mutual competition forms columnar grains. As the columnar zone advances, dendrite branches will break and these fragments will form an equiaxed zone in the center of the casting. The amount of equiaxed grains depends highly on the amount of convection and nucleation phenomena, the more convection the larger the equiaxed zone. Unlike columnar grains, equiaxed grains grow in the direction of heat flow because the undercooled liquid around them dissipates their heat$^4$. 


Fig. A.2.2 Region of constitutional undercooling shown in crosshatched portion
Completely columnar, or completely equiaxed samples can be obtained. Typically, aluminum alloys’ microstructures are composed of columnar or equiaxed dendrites. Until recently, it was believed, and experience had confirmed, that such dendrites would grow along \(<100>\) directions. Models which took into account the weak interfacial energy anisotropy of aluminum were used to predict dendritic microstructures in a wide variety of alloys and solidification parameters. Sémoroz et al.\(^5\) and Gonzales et al.\(^1\) have found that this anisotropy is highly dependent on concentration and alloying elements and, thus, dendrite growth direction can be different from \(<100>\).

Feathery grains containing columnar twinned dendrites have also been observed in directional solidification. Twinned dendrites seem to have growth advantage over columnar dendrites particularly in high temperature gradients, intermediate growth rates, and alloys containing a critical solute content. Once conditions for twin growth are present, twins can multiply by forming new twin planes from stacking faults occurring on the secondary dendrite arms. This particular microstructure is formed by a lamellar structure of twinned and untwined regions. Henry et al.\(^6\) has shown that the coherent twin plane was of \(\{111\}\) type while dendrite growth directions were debated for several years. Morris et al.\(^7\) suggested that dendrites followed a \(\{110\}\) growth direction but Eady and Hogan et al.\(^8\) defended that they would grow along any direction contained in the plane. The latest work published on the topic showed that twinned dendrites grow along \(\{110\}\) directions in industrial – type alloys\(^9\). Branching mechanisms have been suggested by Henry et al.\(^10\) that explain the alternating orientation of grains and their growth advantage over columnar grains, however, further work is necessary.

### A.3 EXPERIMENTAL PROCEDURE

#### A.3.1 Directional Solidification

The experimental apparatus (Fig. A.3.1), modified from the setup of Henry et al.\(^11\) was made of a slightly conical stainless steel mold. The mold was coated in a thin layer of
boron nitride to prevent any reactions between the iron mold and the aluminum. A thin stainless steel plate was affixed to the bottom of the mold. The mold was tightly wrapped with an electric heating wire connected to a power source (240V max) and was heated to 700°C. The entire mold was then covered in fiberglass wool for insulation. Three K-type thermocouples were attached to the inside of the mold to measure the thermal gradient as the metal solidified. The thermocouples were placed at 1mm, 5mm, and 25mm from the bottom of the mold. A hole was drilled at approx. 8cm from the base of the mold where argon gas could be introduced through a quartz tube. Another stainless steel plate formed the top of the mold, which had a slot machined in it so the wire containing the magnesium pieces, as well as the rotor for mixing, could be easily introduced. A water jet affixed 1cm beneath the mold provided directional cooling. However, the thermal gradient and the growth velocity could not be separately controlled as both depend on distance from the bottom

A.3.2 Alloy Preparation

Aluminum-magnesium alloys of the following compositions were prepared using the directional solidification technique: Al 10 wt. % Mg and Al 16 wt. % Mg. These compositions can be noted on the phase diagram, Fig. A.3.2. Aluminum was pre-melted before being poured into the mold. Due to the fact that magnesium oxidizes when heated, the Mg was added in pieces of 20 grams each, wrapped in aluminum foil and then attached to a wire, which was coated in boronitride spray. The wire was necessary to hold the magnesium under the surface of the aluminum to prevent large amounts of Mg loss. Argon gas was also introduced into the mold to reduce the amount of oxygen. After all the Mg was added the melt was stirred to promote mixing of the aluminum and magnesium. The rotor was then removed and the melt was allowed to rest to minimize the effects of forced convection from pouring. An experiment was also carried out where two quartz tubes, 5mm and 3mm, were placed into the melt after stirring to see if this would further reduce the effects of convection.
Fig. A.3.1 Photograph of directional solidification (chill cast) experimental setup.
Fig. A.3.2 Al – Mg phase diagram.
The directionally solidified samples were cut longitudinally and then one side was polished with silicon-carbide paper of 220, 500, 1000, and 2000 grit followed by 6 and 1 micron diamond spray. The samples were then etched with a 4g NaOH 200ml water solution for 4.5 minutes. Using the optical microscope the microstructure was observed and photographs were taken. The longitudinal cuts were cut transversely at 1cm and 4.5cm from the bottom. The same process was carried out on the transverse samples. The samples were then repolished using diamond spray to remove the previous etch before electropolishing. Electropolishing was carried out using an A2-Struers solution (72mL ethanol, 20 mL 2-buthoxyethanol, and 8 mL 71% pure perchloric acid) in two steps, 5V for 10s and 25V for 2s. EBSD observations were then performed.

A.3.3 EBSD

Electron backscatter diffraction (EBSD) was used to collect crystallographic information on the samples. In this technique an electron beam strikes a polished sample inclined at 70° where electrons are reflected by the crystal planes in the sample to form electron backscatter diffraction patterns on a fluorescent screen. Because of Bragg diffraction Kikuchi bands are formed, where each band can be labeled with the miller indices that produced it. The crystal orientation can then be calculated using the Hough transform\textsuperscript{13}.

A.4 EXPERIMENTAL RESULTS AND DISCUSSION

A.4.1 Directional Solidification (Chill-Cast) Experiments

Samples that had been solidified were cut at 4.5cm from the bottom yielded only equiaxed and twinned grain. Fig. A.4.1 shows an EBSD generated image of the longitudinal section of an Al 10 wt. % Mg alloy formed by of a combination of these two types of grains. One can see that the equiaxed grains seem to be randomly oriented while the twinned grains show the typically progressive disorientation as well a lamellar structure. From the equiaxed grains, a longitudinal section was analyzed.
The Al 10 wt. % Mg sample, along with twins, contains equiaxed dendrites. One equiaxed dendrite is pictured below in figure 4.2 along with the (110) and (100) pole figures. The direction of the longest trunk of the dendrite corresponds to the point in the 3\textsuperscript{rd} quadrant of the (100) pole figure. The trunk perpendicular to the cut corresponds to the point in the 2\textsuperscript{nd} quadrant of the 100 pole figure and thus it is shown to be deviated from the gradient. A (110) pole figure was constructed and though the trunk direction lies in the center, based on the microstructure the dendrite cannot have a (110) orientation.

A twin grain was analyzed further to obtain the pole figures and optical micrographs of each region, seen below in Figure A.4.3 and A.4.4. Figures A.4.3a and A.4.4a show a longitudinal section that corresponds to the 110 pole figure. Figures A.4.3b and A.4.4b show a transverse section that again is aligned in the <110> direction based on the pole figure. Figures A.4.3c and A.4.4c show a cut parallel to the dendrite growth direction. Normally, dendrite arms do not form perpendicular to the gradient because there is no driving force. However, observing the twins in the transverse section, shows the two arms that are perpendicular to the gradient have formed creating an “X” type microstructure. This transverse section also proves to be well aligned with the (110) pole figure. The parallel section was analyzed to confirm the (110) nature of the dendrites observed in the longitudinal cut. Through the trunks are (110) as seen in the pole figure (A.4.4c), the arms were not, so a parallel cut was necessary.

Experiments were also conducted using Al 16 wt. % Mg. Fig. A.4.5a without convection and A.4.5b with convection both show an equiaxed microstructure corresponding to a (110) orientation.

No twins were found to form in the 16 wt. % Mg sample, therefore, another experiment was made introducing two quartz tubes, of diameter 6mm and 7mm into the melt after mixing to reduce the effects of convection. Seen in Fig. A.4.6, again only equiaxed grains formed, the microstructure inside the tube being finer than that outside. EBSD analysis could not be done on this sample, as the microstructure both inside and outside the tubes was too small to get a usable signal.
Fig. A.4.1 EBSD analysis of transverse cut of Al 10 wt. % Mg. Section shown was cut 45mm from base of DS sample.

Fig. A.4.2 Optical microscope images and pole figures of transverse cut Al 10 wt. % Mg showing equiaxed microstructure.
Figure A.4.3 Al 10 wt. % Mg optical microscope images and pole figures of corresponding twinned grain.
Fig. A.4.4 Al 10 wt. % Mg optical microscope images and pole figures of corresponding twinned grain. The micrograph in part (c) was not necessary because the arms were not aligned with the trunk in the (110) direction.
Fig. A.4.5 Optical micrograph and corresponding pole figures of Al 16 wt. % Mg transverse cut a) without convection b) with convection.
Fig. A.4.6 SEM and optical micrograph of Al 16 wt. % Mg with quartz tube inserted. Notice the microstructure inside the quartz tube is smaller than the microstructure outside.
A.5 CONCLUSION

In directionally solidified samples a transition from columnar/twinned to equiaxed grains was observed in Al-Mg alloys. This transition occurred around 10 wt. % Mg without convection with all higher compositions unable to produce twins both with and without convection. In the Al 10 wt. % Mg samples the equiaxed grains exhibited a (100) orientation while the twinned grains showed at (110) orientation. The Al 16 wt. % Mg sample showed only equiaxed grains with a (110) orientation.

REFERENCES


