Advanced Casting Research Center (ACRC) Consortium Meeting

May 15, 2001

Report 01-#1

Metal Processing Institute
WPI, Worcester, MA 01609 USA
www.wpi.edu/+mpi
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A. Microstructural Evolution in Semi-Solid Alloys (MIT)
PROJECT STATEMENT

Objectives

- Obtain fundamental rheological data on microstructural evolution
- Design experiments to obtain information of direct applicability to both the modeling studies at WPI and to the planned experiments for industrial type forming applications
- Determine the effects of semi-solid “slurry” structure on flow at the high shear rates representative of actual forming processes.
- Develop new methods of forming structures for SSF

Strategy

- Current descriptions of the flow behavior of semi-solid materials are for highly idealized conditions, which are not encountered in industrial forming processes. This project examines the high shear rate, transient flow behavior of both rheocast and thixocast alloys.
• Two complementary approaches are being employed to examine the flow behavior of semi-solid metals under experimental conditions that are closer to those found in industrial practice. In the **first approach**, a “drop forge” viscometer is being employed to examine the flow behavior under very rapid compression rates of A357, A356 diluted with pure Al, and Al-4.5Cu alloys. The A357 alloys under investigation are of commercial origin (MHD and SIMA processes) and the rheocast modified A356 and Al-4.5Cu alloys are produced by a process developed at MIT. In the **second approach**, a modified parallel-plate rheometer was used to measure viscosities of commercial A357 slurries. The actual slurries were produced by rapid reheating of MHD material in the rheometer. The combined approaches allow transient rheological measurements to be carried out over a broad range of solid content and under conditions closer to those of actual forming processes.

• In an added activity, the mechanism of formation of structures suitable for semi-solid forming is being investigated, with the aim of determining improved methods of producing these structures.

**ACHIEVEMENTS THIS QUARTER**

**Drop Forge Viscometer**
Viscosity calculations of rheocast, modified A356 was conducted up to volume fraction solid of 0.67. A357 MHD material was examined for comparison with SIMA material. Slow compression studies were carried out with the DFV on A357 SIMA, and segregation was observed in the compressed samples.

**Parallel-Plate Rheometer**
Work completed in January 2001

**Formation of SSM Structures**
Non-dendritic A356 Aluminum alloy was produced by a new approach (New MIT Process): processing with rapid cooling and vigorous agitation during only the first few degrees of solidification. Experiments were conducted to test how much solid must form during processing to create the non-dendritic structure. Other experiments tested the required intensity of liquid flow to achieve these structures.

**CHANGES IN PROJECT STATEMENTS**
None
WORK PLANNED FOR NEXT QUARTER

Drop Forge Viscometer
The segregation phenomenon will be examined, along with the continuation of modeling work to verify existing flow models.

Structure Formation (New MIT Process)
Design and build highly instrumented equipment to further characterize the solidification conditions that lead to non-dendritic structure. Begin heat and fluid flow modeling to aide experimental work in determining required solidification conditions.

OPERATIONAL SCHEDULE

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<td>DROP FORGE VISCOMETER</td>
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<td>Experimentally examine segregation phenomenon</td>
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<td>FORMATION OF SSM STRUCTURES</td>
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<td>Design and build highly instrumented equipment for further tests</td>
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<td>Model heat and fluid flow to understand required conditions</td>
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MICROSTRUCTURAL EVOLUTION IN SEMI-SOLID ALLOYS

M. C. Flemings, J. Yurko, and R. Martinez
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Summary

James Yurko is completing his doctoral thesis this month, on rapid transient measurement of rheology of semi-solid alloys, using his newly developed Drop Forge Viscometer (“DFV”). Raoul Martinez is completing his master’s degree on his newly developed approach to producing semi-solid alloys of the desired non-dendritic structure (the “New MIT Process”). The following summary report of MIT activities comprises the Abstract and Conclusions of those two theses.

Rapid Transient Measurement of Rheological Behavior of Semi Solid Aluminum Alloys (J. Yurko)

Abstract

The rheological behavior and microstructure of semi-solid aluminum alloys were studied using a novel apparatus, the Drop Forge Viscometer (DFV). The viscometer determines force from the curvature of displacement data allowing calculations of viscosities at shear rates in excess of \(10^3\) s\(^{-1}\). Alternatively, the DFV can be operated like a conventional parallel-plate compression viscometer, attaining shear rates as low as \(10^5\) s\(^{-1}\). Durations of an experiment range between approximately 5 ms and 24 hours.

Most rapid compression tests had periods of first rapidly increasing shear rate followed by rapidly decreasing shear rate. Viscosity during the increasing shear rate period decreased by 1-2 orders of magnitude. The viscosity during the decreasing shear rate was an order of magnitude larger than another that had achieved a 75% greater maximum shear rate.

The DFV was used to calculate viscosity as a function of shear rate for Al-Si and Al-Cu alloys that were rheocast with the commercial SIMA and MHD processes, as well as the recently developed MIT method. Experiments were conducted between fractions solid of 0.44 and 0.67. Viscosity of A357 produced by the three processing routes all had similar viscosities, ranging from 300 Pa.s at 120 s\(^{-1}\) to 2.2 Pa.s at 1500 s\(^{-1}\). The final height of compressed Al-Cu was always greater than Al-Si for a given set of experimental conditions.
Segregation was not observed in rapid compression experiments of less than 10 ms, either visually or with EDS characterization. At low compression velocities, segregation was observed and increased with the amount of strain.

Conclusions

1. A novel apparatus, the Drop Forge Viscometer (DFV) was designed and constructed based on the parallel-plate compression viscometer. The viscometer permits calculating force from the second derivative of the displacement data allowing calculations of viscosities at shear rates in excess of $10^{-2}$ s$^{-1}$. Total duration of a test can be less than 10 ms. Alternatively, the DFV can be operated as a conventional parallel-plate viscometer, attaining shear rates as low as $10^{-5}$ s$^{-1}$.

2. A unique feature of the DFV is that a typical experiment yields instantaneous, volume-averaged viscosity first under rapidly increasing shear rate and then under rapidly decreasing shear rate.

3. The apparatus was used to determine the viscosity as a function of shear rate and fraction solid for alloys of non-dendritic structure produced by three means, known as the SIMA, MHD, and MIT processes. The viscosity of the MIT material was similar to alloys produced by the commercial SIMA and MHD processes.

4. Most compression tests in this work (all of the rapid compression tests) were completed within about 6 ms. During the increased shear rate portion of a typical test (lasting about 4 ms), the viscosity dropped by 1-2 orders of magnitude. Viscosity during the decreasing shear rate period decreased with increasing maximum increasing shear rate. For example, in two SIMA alloys of about 0.48 volume fraction solid ($g_s$), the viscosity calculated at 100 s$^{-1}$ was an order of magnitude greater than a sample that achieved a 75% higher shear rate.

5. Viscosity versus increasing shear rate of the Al-Si alloys at 0.48 $g_s$ produced by the three different process routes all showed roughly the same viscosity as a function of shear rate, ranging from 300 Pa.s at 120 s$^{-1}$ to 2.2 Pas at 1500 s$^{-1}$.

6. Viscosity as a function of increasing shear rate of the Al-4.5wt%Cu alloy was larger than an Al-Si alloy for a fraction solid of about 0.48, but the viscosity was about the same as an Al-Si alloy at 0.56 $g_s$. The final compressed height of the Al-Cu was always greater than an Al-Si alloy for similar experimental conditions.

7. Visual examination of cross sections did not show separation of liquid and solid phases in any of the rapidly compressed samples in the shear rate range of 10 to 1500 s$^{-1}$. Segregation was not detected by either quantitative
metallography or chemical composition variations in an A357 SIMA sample that had been compressed at a fraction solid of 0.48 and at shear rates ranging between 15 and 750 s$^{-1}$. However, when the DFV was used as a conventional parallel-plate viscometer to achieve very low shear rates, there was a fraction solid difference from center to edge of 0.20 (calculated from variation in chemical composition) after compression for 15 minutes in the shear rate range of $10^{-2}$ to $10^{-4}$ s$^{-1}$. A sample that was compressed for four hours at shear rates of $10^{-2}$ to $10^{-6}$ s$^{-1}$ had a fraction solid of approximately zero at the edge and 0.70 in the center. Segregation appears to increase with increasing strain in the low shear rate experiments.

8. The maximum fraction solid that was compressed with the DFV at high and low compression velocity was 0.67 and 0.70, respectively.

9. The DFV is a useful process control tool for comparing the relative compression behavior of semi-solid alloys in the two-phase temperature region. Differences between samples are reflected in the final shape and thickness of the compressed samples, and also in the displacement data.

The DFV provides unique transient rheological data of particular relevance to the modeling of semi-solid processing. Validation of rheological models was achieved by comparing experimental results with model predictions.

SSM Structure Formation (Raoul Martinez)

Abstract

A crucial aspect for the commercial use of semisolid forming technology is the economical production of alloys with non-dendritic microstructure. There are currently available a variety of processing routes to obtain these special structures, but each has disadvantages. Attempts are being made to find other simple and effective methods to create non-dendritic material to be used in semisolid forming.

It is now known that the combination of cooling with vigorous agitation during the solidification of an alloy serves to spheroidize the primary solid particles. Several processes apply this combination to create non-dendritic microstructure. What has been unknown up to this point is how much cooling and agitation is required. It is also largely unknown during what portion of the solidification range does processing induce structural changes. The answer to these questions may be critical to the recognition of new processing methods. In this work it is suggested that processing during the very beginning of solidification is most significant for creating non-dendritic structures. A new approach was developed to test this hypothesis.
A rotating copper rod was immersed in a molten aluminum alloy, held just above the liquidus temperature. The rod quickly lowered the temperature of the melt just below the liquidus temperature, and simultaneously stirred the alloy. By removing the rod when the melt temperature had dropped just a few degrees below the liquidus, the combination of stirring and rapid cooling was applied when only small amounts of solid had formed. Results show that good non-dendritic structures can be created by processing in this manner when as little as 1 vol% solid had solidified. No significant microstructural differences were observed when the alloy was stirred by the rod beyond this point in the solidification range. It was determined that all of the rotational speeds used in the experiments induced turbulent liquid flow. The degree of turbulence was not found to significantly affect the microstructures produced.

The following is a brief summary of the conclusions that have been drawn from this work. It has been well documented that providing rapid cooling and vigorous agitation during the initial stages of alloy solidification can create non-dendritic structures. This processing route is a powerful new way to efficiently create semisolid material free of entrapped eutectic. Processing during the solidification of the first 1 vol% solid is most critical in the development of the non-dendritic structure. When the fluid flow during processing is turbulent, the solidified alloy has a non-dendritic structure. Over the range investigated, the rotational speed of the rod was not found to have a significant effect on the sphericity of the primary particles.

Conclusions

A. Process Development
1. A new processing approach for the formation of non-dendritic structures has been developed. The technique involves immersing a rotating copper rod, initially at room temperature, into an A356 melt held just above its liquidus temperature. The rod rapidly cools the alloy below its liquidus to initiate solidification while vigorously stirring the melt. The rod remains in the melt for short periods, just long enough to cool the melt a few degrees below the liquidus temperature.

1. The processing technique used can create semisolid slurries directly from the liquid alloy in a simple and efficient manner.

1. The non-dendritic structures created by this technique have demonstrated a semisolid flow behavior comparable to that of commercially available SIMA material.

1. The material produced by this method is essentially free of entrapped eutectic. This may be a significant advantage when using the material in semisolid forming operations.
2. Although most of the material produced by the new processing technique is non-dendritic, some regions of structural inhomogeneity are present. The formation of these regions is likely caused by stirring with the rod geometry.

B. Required Degree of Solidification During Processing

1. During the onset of solidification, or shortly thereafter, processing can lead to the formation of the non-dendritic structures. These structures have been created when the cooling and agitation induced by the spinning copper rod was applied for a very small solidification interval, such that as little as 1 vol% solid was formed before the rod was removed. No major microstructural differences were observed when the rod was removed after more solid fraction had formed, indicating that processing during the solidification of the first 1 vol% solid is the most critical for creating non-dendritic structure.

2. The previous conclusion was verified by calculating the average shape factor for reheated material which had been stirred for 2, 5, 11, and 20 seconds. These stirring times cooled the melt different amount below the liquidus temperature, corresponding to varying degrees of solidification ranging from 1 to 7 vol% solid. For each stirring time, the particles have a constant shape factor value near 0.75. Since this value is independent of stirring time and the associated degree of solidification, the results support the previous conclusion that microstructural changes must occur by processing during the solidification of the first 1 vol% solid.

C. Required Amount of Liquid Flow During Processing

1. Using the new processing technique, "low" rotational speeds are effective in creating the non-dendritic microstructure. When the copper rod was rotated at only 60 RPM and immersed for 20 seconds, non-dendritic material was produced.

2. After reheating, the average particle shape factor was 0.71 for material stirred with 60 RPM; only slightly less than the shape factor for material processed with higher rotational speeds of 508 and 1001 RPM which was 0.73 and 0.78, respectively. Reheated dendritic material had an average shape factor of 0.51. The transition between non-dendritic and dendritic morphology must occur when rotational speeds less than 60 RPM are used.

3. By calculating the Taylor number, it was shown that the liquid flow induced by the stirring rod is always in the turbulent flow regime, even when the lowest stirring speed of 60 RPM was used.
B. Development of Alternate Semi-Solid Aluminum Alloys (ORNL)
DEVELOPMENT OF ALTERNATE SEMI-SOLID ALUMINUM ALLOYS

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PROJECT STATEMENT

Objectives

- Model thermodynamic phase equilibria for alternate alloy systems, such as aluminum-silicon-magnesium, aluminum-magnesium, and aluminum-copper.

- Model solidification behavior and determine key characteristics.

- Use the information on solidification behavior to tailor and develop alloys that are better suited for SSM processing.

Strategy

- Use Thermocalc and Dictra thermodynamic modeling packages together with an aluminum alloy database to determine phase equilibria for SSM alloys.

- Identify key characteristics of the alloy that can be determined from model.

- Identify desired characteristics of an ideal SSM alloy.
• Use thermodynamic model to develop alloy with ideal characteristics.
• Carry out tests on new alloys to provide feedback and validate results.

ACHIEVEMENTS THIS QUARTER

• Solid fraction and solid fraction variation as a function of processing temperature has been simulated for 500 series of alloys over the composition range of 1~10wt% magnesium and 0.05~3wt% of silicon.

• A region in the composition range where there is a peak of solid fraction variation has been identified. The peak value increases with increasing silicon concentration and decreasing magnesium concentration.

• Samples have been prepared for measurements of composition distribution using electron microprobe analysis in order to validate the thermodynamic simulations.

CHANGES IN PROJECT STATEMENTS
None

WORK PLANNED FOR NEXT QUARTER

• Carry out industrial and/or experimental validation of new SSM alloys.

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Development of Alternate SSM Alloys

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Review Meeting
Worcester, May 15, 2001

Work Since The Last Review Meeting
Addresses Two Issues

- Validation of Thermodynamic Predictions in SSM 357 Alloy
  - Composition distribution in the primary phase and in the eutectic regions.
- Simulations of Al-Mg (500 series) alloys.
  - Alloys are Al - (1~10)Mg - (1~3)Si - 0.5Fe - 0.15Cu - 0.35Mn - 0.15Zn - 0.25Ti
  - Solid fractions and solid fraction variations as a function of processing temperatures.
Thermodynamic Predictions of Solute Distribution in 357 Alloy Have Been Validated

- JEOL 773 Electron microprobe was used to measure solute distribution across grains in SSM Billet.
- The distance across a grain was converted into solid fraction, a non-dimensional parameter. The solid fraction at the edge of the grain was assumed to be 0.5, since eutectic occurs at 0.5 solid fraction.
- The measurements were compared with calculations made assuming Scheil behavior.

Prediction of Silicon Distribution Agrees Well with Measurements

- Measurements of silicon concentration are higher than predictions.
  - It is unlikely that grains were sectioned at the center.
- The minimum values of the measurement fit the predictions.
- Back diffusion is not included in the model.
- The exact ingot composition is not known.

Alloy composition (wt%):
Si 7.0, Mg 0.55, Fe 0.1, Ti 0.06
Cu 0.008, Mn 0.0005, Zn 0.001
Prediction of Magnesium Distribution Agrees Well with Measurements

- The minimum value of the measured Mg concentration matches the prediction.
- Magnesium concentration in the grain is only 1/3 of the bulk concentration.

![Graph showing magnesium concentration vs. solid fraction]

Alloy composition (wt%): Si 7.0, Mg 0.55, Fe 0.1, Ti 0.06, Cu 0.008, Mn 0.0005, Zn 0.001

Simulation of Al-Mg (500 Series) Alloys Has Been Completed

- Solid fraction versus temperature curves for alloys in the composition range of 1-10wt% Mg and 1-3wt% Si have been determined.
- Solid fraction variation for Al-Mg (500 series) alloys as a function of processing temperature has been calculated.
- Al-Mg alloys appear to have good potential for semi-solid processing.
- It appears that Al-Mg alloys can be processed at solid fractions greater than 0.5.
Both the Solid Fraction and the Solid Fraction Variation with Temperature Affect SSM Processing

Note
A small solid fraction variation indicates a large processing window at that temperature.

The Solid Fraction Curve of Al-Mg Alloys Is Very Different Compared to Al-Si Alloy
Al-4% Mg-(0.5~2)% Si Alloys Appear to Have Potential for SSM Processing

- When the silicon concentration is low, the solid fraction variation decreases with decreasing processing temperatures.
- When the silicon concentration is higher than 1 wt%, there is a sharp increase in solid fraction variation when the processing temperature is below 600°C.
- The alloys may be processed at a solid fraction greater than 0.5.

Al-6% Mg-(0.5~2)% Si Alloys Have Potential at High Solid Fraction or Low Si

- A peak in solid fraction variation occurs at a processing temperature of 590°C. The peak value increases with increasing silicon concentration.
- The solid fraction corresponding to the peak value is about 0.6.
- The smallest solid fraction variation is obtained at high solid fraction (>0.75).
Al-8%Mg-(0.5-3)%Si Alloys Can Be Processed at $f_s \geq 0.6$

- The peak in solid fraction variation, which occurs at 590°C, increases with silicon concentration.
- When the solid fraction is higher than 0.5, the solid fraction variation decreases with decreasing processing temperatures.

Al-10%Mg-(0.5-3)%Si Alloys Can Be Processed at $f_s \geq 0.5$

- For Si%<1, the solid fraction variation decreases with processing temperature.
- For Si%>2, a peak in solid fraction variation occurs at a solid fraction less than 0.5.
- The alloys may be processed at high solid fraction with low solid fraction variation.
The Solid Fraction Variation Decreases with Decreasing Processing Temperature When Si% = 0.05

- Solid fraction variation decreases with decreasing processing temperatures, indicating that the alloys should be processed at higher solid fraction.
- The peak shifts to lower processing temperatures with increasing magnesium contents.

Increased Peak Values of Solid Fraction Variation Occur When Si = 1wt%

- With increasing magnesium concentration, the peak is shifted to lower processing temperatures.
- To avoid high solid fraction variation in 500 series alloys, the magnesium concentration should be either lower or higher than the region of the peak, when the silicon concentration is about 1 wt%.
High Peak Values Occur around 590°C When Si=2 wt%  

- The peak in solid fraction variation is as high as 0.3-0.4.  
- Alloys of low Mg (<4%) or high Mg content (10%) can still be processed at low solid fraction variation.  
- When Mg is 6-8%, the alloy must be processed at low solid fraction (<0.4) to avoid high solid fraction variation.

The Peak in Solid Fraction Variation Increases With Increasing Silicon Concentration
Al-Mg Alloys Can be Processed at Low Si or High Solid Fraction

- Solid fraction and solid fraction variation as a function of processing temperature over the whole composition range of Al-Mg alloys have been computed.
- The alloys can be processed at high solid fractions. Alloys with Si < 0.5% can be processed at a wide range of solid fractions.
- A peak in solid fraction variation has been identified for each composition when Si > 0.5%. The peak occurs around 560-590°C depending on the alloy composition.
- The processing temperature of 500 series alloys should be determined such that the peak in solid fraction variation is avoided.

Future Work

- Validation of low silicon 357 alloys.
  - Test of low silicon (5.5–6.5%) 357 alloys for less liquid drip and improved process consistency.
- Dilatometry tests of 357 SSM alloys and squeeze cast alloys.
  - Compare dynamic behavior of these two alloys during T4 and T5.
  - Thermodynamic and kinetic modeling if necessary.
- Suggestions for SSM 500 series alloys.
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A. Time Dependent Rheology of Semi-Solid Alloys (MIT)
TIME DEPENDENT RHEOLOGY OF SEMI-SOLID ALLOYS

Report No. PR–01#2

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PROJECT STATEMENT

Objectives

- Obtain fundamental data on rheology of semi-solid metals
- Design experiments to obtain information of direct applicability to modelling studies at WPI
- Investigate the rapid transient measurement of rheological behavior of semi-solid aluminum alloys

Strategy

A drop forge viscometer was employed to investigate the flow behavior under very rapid compression rates of A357, A356 diluted with pure aluminum and Al-4.5%Cu alloys. The A357 alloys were of commercial origin (MHD and SIMA) and the rheocast, modified A356 and Al-4.5Cu alloys were produced by a process developed at the solidification laboratory of MIT.
ACHIEVEMENTS TO DATE

- A “drop-forged viscometer” (DFV) was designed and constructed based on parallel-plate viscometry. This viscometer allows viscosity measurements in experiments lasting less than 10 ms and at shear rates exceeding 1000 s⁻¹.

- The DFV allows instantaneous, volume-averaged viscosity determinations first under rapidly increasing shear rates and then under rapid decreasing shear rates.

- The apparatus was used to determine viscosity as a function of shear rate and fraction solid for alloys of non-dendritic structure at solid contents near 70 vol%.

- Metallographic examination of sample cross sections did not reveal separation of liquid and solid phases in any of the rapidly compressed samples in the shear rate of 10 to 1500 s⁻¹. Segregation was also not detected by quantitative metallography or chemical analyses and occurred only very slow rates of deformation (<<10 s⁻¹).

- Validation of rheological models was achieved by comparing DFV results with model predictions.

CHANGES IN PROJECT STATEMENT

None

WORK PLANNED FOR THE NEXT QUARTER

The project has been completed

PROJECT SCHEDULE

The project has been completed.

APPENDIX

A detailed description of the process is contained in the appendix (paper to be submitted to Metall. Mater. Trans.)
"Rheology and Microstructure of Semi-Solid Aluminum Alloys Compressed in the Drop Forge Viscometer"

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ABSTRACT

The rheological behavior and microstructure of semi-solid aluminum alloys were studied using a novel apparatus, the Drop Forge Viscometer (DFV). The viscometer determines force from the second derivative of displacement data permitting calculations of viscosities at shear rates in excess of 1000 s^{-1}. Alternatively, the DFV can be operated like a conventional parallel-plate viscometer, attaining shear rates as low as 10^{-5} s^{-1}.

Rapid compression experiments result in periods of first rapidly increasing then decreasing shear rate in periods of less than 10 ms. Viscosity decreases by 1-2 orders of magnitude during the increasing shear rate period. The viscosity during the decreasing shear rate period was an order of magnitude smaller (relative to another experiment) when it achieved a 75% greater maximum shear rate. In addition, the viscosity during the decreasing shear rate period was a function of the maximum achieved and instantaneous shear rate, and not the duration of shear for tests lasting between 0.01 and 1 second.

The dual observed phenomena of (1) a very rapid drop of viscosity with increasing shear rate followed by (2) a relatively slow increase of viscosity with decreasing shear rate thereafter have much potential significance for future machine and process design. For example, it should be possible to form higher fraction solid slurries than is now feasible by applying vigorous shear to semi-solid slurries just before the metal is introduced to the die entrance.

The DFV was used to calculate viscosity as a function of shear rate for samples produced by the commercial SIMA and MHD methods, as well as the recently developed MIT method. Experiments were conducted between fractions solid of 0.44 and 0.67. Viscosity of A357 and A356 produced by the various methods was similar.

Separation of liquid and solid phases was not observed in rapid compression experiments shorter than 10 ms, either visually or with EDS characterization. At low compression velocities segregation was observed and increased with the amount of strain.
I. Introduction

The fluid flow behavior of semi-solid metal alloys possessing non-dendritic microstructure has been studied using a variety of characterization techniques. Semi-solid alloys were originally produced by mechanically stirring molten metal as it cooled below the liquidus temperature; therefore, a natural device for examining the fluid flow behavior was the Couette rheometer, or concentric cylinder rheometer. The Couette rheometer creates globular semi-solid metal in-situ during cooling, and numerous studies have been conducted that characterize the viscosity as a function of shear rate, cooling rate, particle size and morphology, and fraction solid.

Typical industrial semi-solid forming processes occur at transient, high shear rates and use alloys that were prepared by means other than mechanical stirring. Most commercial processes are conducted at shear rates of $10^2 - 10^4$ s$^{-1}$ in less than 0.1 seconds. However, rotational viscometers use in-situ prepared semi-solid material and are of limited use in transient, high-shear rate experiments. Other experimental approaches have also been utilized, including the back-extrusion method, capillary viscometer, indentation and vane techniques, and parallel-plate rotation and compression viscometers. Although these techniques have more complicated fluid flow patterns than a concentric-cylinder device, rheological data can be extracted that sheds light on the behavior of semi-solid alloys at transient, high shear rates.

The parallel-plate compression viscometer was originally used to study Sn-Pb semi-solid alloys by Laxmanan and Flemings in the fraction solid range of 0.15 to 0.6 and for shear rates less than 1 s$^{-1}$. Other researchers used this device for studying semi-solid aluminum alloys and composites for similar fractions solid and low shear rates. Kapranos et al. adapted the parallel-plate viscometer by increasing the compression speed of the plates to achieve shear rates as high as 200 s$^{-1}$. Semi-solid aluminum alloys were studied under a wide range of conditions, and the flow was characterized as a function of processing route, fraction solid, and compression velocity. The instrument has the unique ability to measure force during the initial moments of rapid compression.

Recently, a novel compression-type viscometer that can achieve very high shear rates and strain was built at MIT. The Drop Forge Viscometer (DFV) has a geometry similar to the parallel-plate viscometer, except the upper platen is suspended and permitted to fall under the influence of gravity. As a result, the upper platen impacts the semi-solid metal sample at high velocity. With the aid of a digital high-speed camera, the deceleration of the platen is measured, and the viscosity of the fluid is subsequently determined. During a typical rapid-compression experiment lasting less than 10 ms, the shear rate of the reheated semi-solid sample will initially increase to a maximum before decreasing.

II. Experimental Procedure

A. Materials

Three rheocast aluminum alloys produced by three different process routes were studied in this work. The commercial hypo-eutectic Si alloys A356 and A357 were used, as well as an A356 alloy that was diluted with pure Al to lower the Si content to
approximately 4.5wt%. The A357 used in the experiments was produced by the commercial SIMA (strain induced, melt activated) and MHD (magnetohydrodynamic) methods and acquired from ALUMAX (now a part of ALCOA). The A356 and modified A356 were rheocast at MIT using a new process that forms semi-solid material directly from molten aluminum (referred to as MIT material). The nominal compositions are listed in Table 1.

Cylindrical samples with a diameter of 25 mm and a height of 15 mm were machined from the various materials. The SIMA material was procured in 25 mm and 1.5 m length billets. The MHD and MIT material were also cylindrical, with a 75 mm diameter and a height of approximately 100 mm. The composition of the SIMA material was evaluated at three different locations along the length of the billet using wet chemical analysis (Table 1). The MHD and MIT material were analyzed along the cross-section of the cylinder. Because of the larger compositional variation in the MHD material, samples were machined from the center axis of the slug.

Weight percent fraction solid was calculated using the software package Thermo-Calc® and the SGTE thermodynamic database. Chemically analyzed compositions were used with the Scheil equation to predict fraction solid as a function of temperature. Weight percent fraction solid was converted to volume fraction solid using information on the density of the liquid and solid phases.

B. Drop Forge Viscometer

Figure 1 is a schematic diagram of the Drop Forge Viscometer. The DFV consists of a lower and upper platen, with an attached rod to track platen motion with time. All components are contained inside of an air-recirculating furnace to maintain the apparatus in an isothermal condition (± 1 °C in the vicinity of the bottom platen and sample). The DFV is similar to the traditional parallel-plate compression viscometer except the upper platen falls under the influence of gravity, rather than the conventional approach of resting the upper platen on the fluid.

Both platens are fabricated from steel, and the lower rests on two rectangular steel pieces to suspend it above the floor of the furnace. The five inch diameter upper platen connected to the stainless steel rod is coated with graphite and boron nitride to prevent sticking to the sample. Extra mass may be added to the upper platen to increase the force on the fluid during compression. The rod, which travels through a graphite sleeve in the furnace ceiling to minimize friction, is suspended outside of the furnace by an adjustable solenoid, controlling the z-position of the platen. The maximum height of the upper platen is 30.5 cm.

A high-speed digital camera, the Kodak EktaPro 4540 Motion Analyzer rests on a platform attached to the furnace assembly and images the rod as it falls. A photovoltaic sensor attached to the furnace triggers the camera when the rod achieves a specified position. The rod is backlit with a 300W floodlight, and attached to the rod is a marker, which appears as a sharp dark edge in an image. TIFF images with 264 x 64 pixels of 8-bit resolution are captured at a rate of 18,000 frames per second. During a typical test (5.56 – 11.1 ms), 100 – 200 images are captured, and Figure 2 shows a sample sequence of images. After test completion, the graphic files are downloaded to a personal computer from the camera via a GPIB data acquisition card and a National Instruments Labview® interface.
Using the commercial image analysis software program IPLab®, (registered product of Scanalytics, Inc., Fairfax, VA) the images are analyzed to measure the z-displacement of the sample as a function of time. A macro routine was written that detects the contrasting marker on the rod, measures the height of the sample as a function of the frame number, and exports the data to a text file. The pixel and frame number data is converted to millimeters and seconds, respectively, as shown in Figure 3a. The displacement data is curve fit using a fourth order polynomial equation, and the first and second derivatives of displacement as a function of time are calculated (Figures 3b and c).

The DFV can be operated like a conventional parallel-plate viscometer by resting the platen on the fluid, or the upper platen can be dropped from a known height to compress the sample at velocities greater than 2 m/s. The combination of the two test methods produces rheological data over a large range of shear rate, between $10^{-5}$ and $10^4$ s⁻¹.

During a rapid compression experiment, the furnace is first equilibrated at a desired temperature, and a sample is centered on the lower platen that is inserted into the furnace. A hole was drilled in the center of the initial samples and temperature was monitored using a Type-K thermocouple. The sample was reheated for approximately 90 minutes before compression (Figure 4). After compression, the lower platen with the sample was removed, and the process could be repeated.

C. Calculation of Viscosity and Shear Rate

When using the parallel-plate viscometer, viscosity is calculated using the Stefan equation. The derivation of the Stefan equation has been performed numerous times; therefore, only the major assumptions will be discussed. Assuming flow is radial, the fluid is incompressible, and wall slip is absent, the equations of motion and continuity reduce to the following equations, where \( P \) is pressure, \( \mu \) is viscosity, and \( v \) is velocity.

\[
\frac{\partial P}{\partial r} = \mu \frac{\partial^2 v_r}{\partial z^2} \tag{1}
\]

\[
\frac{1}{r} \frac{\partial}{\partial r} \left( r \cdot v_r \right) + \frac{\partial v_z}{\partial z} = 0 \tag{2}
\]

Assuming the volume of the sample is constant between the plates, the following expression, the Stefan equation, is a solution to Equation 1, where \( F \) is the applied force on the fluid, \( V \) is the fluid volume, and \( h \) is the instantaneous height of the sample.

\[
F = \frac{-3 \mu V^2}{2nh^2} \left( \frac{dh}{dt} \right) \tag{3}
\]

Covey and Stanmore modified the Stefan equation to include a yield stress term for a Bingham or Herschel-Bulkley fluid of constant radius, where \( \tau_y \) is the yield stress, as shown in Equation 4.  

\[
F = \frac{-3 \mu \pi R^4}{2h^3} \left( \frac{dh}{dt} \right) + \frac{\pi \tau_y R^3}{h} \tag{4}
\]

Equation 4 can be modified for a fluid of constant volume instead of constant radius, resulting in the following expression.
This solution is valid when the major resistance to flow is from viscous effects, as determined by the dimensionless modified plasticity number, $S$, where $R$ is the radius of the fluid.

$$S = -\frac{R\mu}{h^2\tau_o}\frac{dh}{dt}$$

Equation 6 can be modified for a constant volume experiment, as seen in Equation 7.

$$S = -\frac{V\mu^2}{h^2\pi\tau_o^2}\frac{dh}{dt}$$

Covey and Stanmore’s criteria for using Equation 5 is that $S$ is greater than 10, but upon closer examination of their work, it appears that the condition where $S$ greater than 1 justifies the use of the solution. If $S$ is less than 0.05, a numerical solution that assumes the bulk of the resistance to deformation is from the yield stress component is appropriate.

In a conventional parallel-plate viscometer, either the applied force or the compression velocity is constant, and the other variable is experimentally measured. A constant force is applied by placing a known weight on the fluid, but shear rates are normally less than 1 s$^{-1}$. Such experiments are normally long duration and do not produce rheological data that is relevant for commercial forming operations. Conversely, constant compression rate experiments can achieve higher shear rates, but they are limited by the crosshead speed of the platens and the load cell response time. Furthermore, as crosshead speed increases, the likelihood of the platens impacting and damaging the load cells increases; therefore, a safety margin of lower sample strain during compression limits the range of data that can be collected.

In the Drop Forge Viscometer, the compression velocity and applied force vary with time. Both are calculated from the displacement data by using the first and second derivatives with respect to time (Figure 3). The compression rate is simply $\frac{dh}{dt}$, and the applied force is the mass of the platen multiplied by the deceleration, $m_p\frac{d^2h}{dt^2}$. The Stefan equation, which is a balance of the applied and viscous forces on the fluid, is reduced to the following expression.

$$m_p\left(\frac{d^2h}{dt^2} + g\right) = -\frac{3\mu V^2}{2\pi h^5}\left(\frac{dh}{dt}\right)$$

After calculating the derivatives, the variables in Equation 8 are known; therefore viscosity as a function of time can be calculated. This viscosity is an average throughout the volume of the fluid and is an instantaneous value.

A volume-averaged, instantaneous shear rate can be calculated with Equation 9, which is a function of the maximum radial position of the sample, $R$, the height, $h$, and the compression velocity, $\frac{dh}{dt}$.

$$\dot{\gamma} = -\frac{R}{2h^2}\left(\frac{dh}{dt}\right)$$

Volume and instantaneous height are known, thus the radius is determined assuming continuity. In this paper, viscosity and shear rate refer to these instantaneous, volume-averaged quantities.
Although the Stefan equation lacks inertial terms and uses steady-state approximations, it has been determined that inertia is insignificant in the viscosity calculation in this work. The aspect ratio of the sample does appear to be significant because the z-direction of the equation of motion is neglected in the Stefan equation. After examining the data and taking the aspect ratio assumption into consideration, viscosity is calculated after the sample has undergone at least 25% strain to ensure high confidence in the validity of the experimental calculation.

D. Characterization of Samples
In addition to the rheological experiments, select compressed and uncompressed samples were analyzed using quantitative metallography and energy dispersive spectroscopy (EDS). The shape factor, $F_o$, a value that relatively compares the area of a grain, $A$, and its perimeter, $P$, was calculated for each micrograph.

$$F_o = \frac{4\pi A}{P^2} \quad (10)$$

Optical images were acquired with a Spot Diagonostic Instruments digital video camera attached to an Olympus Vanox-T microscope and analyzed using IPLab. Samples were polished to 0.05 μm and observed in the unetched condition.

Energy dispersive spectroscopy was performed on compressed A357 samples to determine Si content. The EDS detector is located inside of a scanning electron microscope (SEM), and magnification was approximately 50X, such that a diffuse area could be analyzed. The accuracy of EDS is limited, but the Si composition data was used in conjunction with the lever rule to determine presence or absence of macrosegregation in the compressed samples.

III. Results and Discussion

A. General Rheological Observations
Experiments were conducted at high ($> 10^2$ s$^{-1}$) and low ($< 1$ s$^{-1}$) shear rates on the rheocast alloys described in the previous section. In addition to alloy type and processing route, three primary variables could be controlled during an experiment: temperature (fraction solid of the alloy), initial platen height, and platen (including rod) mass. The volume fraction solid, $g_s$, was varied between 0.44 and 0.67. For the high shear rate experiments, the platen was set at the maximum height in the furnace, 30.5 cm above the reheated sample, unless extra mass was added to the platen, which lowered its height relative to the furnace ceiling. The mass of the platen system ranged from 0.272 to 1.793 kg. Experiments discussed in this paper are listed in Table 2.

Using the data from Figure 3, Figure 5a is a typical plot of viscosity and shear rate as a function of time for a high-shear rate test. The test is conducted in less than 10 ms and is characterized by rapidly increasing then decreasing shear rate. The viscosity, which first drops rapidly and then recovers during the decreasing shear rate portion of the test, behaves inversely with shear rate. Plotting the viscosity data as a function of shear rate from the same experiment (Figure 5b), it is evident that the fluid behaves in a shear thinning manner, and the absolute change in viscosity is greatest when shear rate is increasing.
Figure 6 is a plot of viscosity versus shear rate for two A357 SIMA alloys at a similar fraction solid and platen mass, one of which achieved a shear rate almost twice the value of the other. In both curves, the viscosity falls rapidly by 1-2 orders of magnitude in less than 5 ms, and it is apparent that there is a large difference in viscosity during the decreasing portion of the test. The experiment that achieved the higher maximum shear rate has viscosity values almost an order of magnitude less during the decreasing shear rate regime.

Comparison of Rheology and Microstructure with other studies

The viscosity results from this work are similar to the transient behavior observed by de Figueredo et al.\textsuperscript{[21]} using a rotational parallel-plate viscometer, as seen in Figure 7. Both studies were conducted on reheated semi-solid material at a fraction solid of approximately 0.48, but the duration of the experiment in this work was less than 10 ms, while their test was completed in approximately 2 seconds. After only 2 ms of shear, the viscosity in this work is similar to the other experiment that achieved the same shear rate in approximately 1 s. Relative to the duration of these two experiments, viscosity during the decreasing shear rate portion of the tests is dependent on the maximum achieved shear rate, not the length of time under shear.

The viscosity results acquired with the Drop Forge Viscometer are similar to other studies that used the back-extrusion and the parallel-plate rotational viscometers at a constant shear rate. Figure 8 is a comparison plot of viscosity as a function of shear rate using data from this work and also that of Loué et al.\textsuperscript{[2]} and de Figueredo et al.\textsuperscript{[10]} The viscosity data was collected over a large range of shear rate using the Drop Forge Viscometer, both under rapid compression and resting the upper platen on the fluid. Loué used back-extrusion and a parallel-plate compression viscometer at high and low shear rates, respectively. The data from de Figueredo was acquired from multiple experiments at various steady-state shear rates using a parallel-plate rotational viscometer. The viscosity values are within an order of magnitude over a large range of shear rate using the different techniques, remarkable considering the different flow geometries and methods for determining shear rate. Viscosity from both transient DFV and steady-state parallel-plate rotational experiments is similar, indicating that the fluid can achieve steady-state values after only milliseconds of shear.

Segregation of liquid and solid phases in semi-solid alloys during processing, and the DFV allows for comparison of samples compressed over a wide range of shear-rate. A357 SIMA samples with 0.48 fraction solid were compressed in rapid compression tests and in low shear rate, long duration tests of 16 minutes and 4 hours. Micrographs were taken of each sample at the center and radial edge, and the shape factor and solid fraction were calculated using quantitative metallography. Because quantitative metallography can overestimate volume fraction solid,\textsuperscript{[22]} the silicon composition of each section in a sample was determined using EDS, and the lever rule was used to determine fraction solid. Results are in Table 3, and micrographs of the center and radial sections of each sample are shown in Figures 9, 10, and 11.

Results agree with previous studies that report separation of liquid and solid phases at low shear rates, and the amount of segregation decreases with increasing shear rate.\textsuperscript{[23]} At high shear rates, there is no apparent segregation, as seen in Figure 9a and b.\textsuperscript{[2]} In the rapid compression experiment, the difference in fraction solid determined by
the two methods is minimal, less than 0.10; however, in the lower shear rate experiments, the difference in fraction solid ranges from 0.19 to 0.67, depending on the characterization technique and duration of the experiment.

A low shear rate experiment was conducted that lasted approximately 24 hours, and the difference in the final dimensionless height of this sample and a sample compressed for 4 hours was approximately 1%. The experiment that was completed after 16 minutes had a dimensionless height almost 20% greater than the longer duration experiments. Apparently compression stopped between 16 minutes and 4 hours.

One possible explanation for this behavior arises from the coherency theory of dendrites and maximum packing fraction. Below a certain temperature, the coherency fraction solid \( f_s^{ch} \) \(^{[24]}\), grains are unable to move great distances in the melt because particles are contacting each other, but they can rearrange over short distances by either stress induced grain boundary motion \(^{[25]}\) or lubricated sliding. \(^{[26]}\) There is a maximum solid fraction, the packing solid fraction \( f_s^{pk} \), where grains can no longer move by these mechanisms, and the samples cease compression because the solid network supports the weight of the upper platen. The exact fraction solid that corresponds to the coherency and packing limit are functions of the grain shape and size and of alloy composition. \(^{[24]}\) The sample compressed for 16 minutes had a fraction solid of 0.53 in the center, which is a value between the coherency and the packing limit. The longer duration experiment has a fraction solid of 0.69, apparently at the packing limit.

**Comparison of SIMA, MHD, and MIT Material**

Most A357 experiments were conducted using samples produced by the SIMA process, but other material produced by the MIT and MHD processes were also tested. Tests were limited to a fraction solid of near 0.50 because the eutectic constituents of the alloy freeze over a narrow temperature range above this fraction solid, thus making viscosity measurements difficult.

Figure 12 is a comparison of the viscosity behavior of the various alloys during the increasing shear rate regime of a rapid-compression experiment. As expected, the viscosity is very similar for all, but the overall maximum shear rate achieved by the MHD material was much lower than the SIMA or MIT material. When comparing the shape factor of each material (Table 4), the MHD clearly has the least spheroidal structure. Experiments conducted by Kapranos et al. have shown that during the initial stages of compression, MHD material produces a larger spike in force relative to other materials. \(^{[27]}\) The larger force value corresponds to a larger decrease in kinetic energy during the initial moments of compression, thus decelerating the upper platen. The velocity of the platen after the initial moments of MHD compression is smaller than for other materials; therefore the maximum achieved shear rate is smaller. After the initial breakdown of the structure, the viscosity of the three different rheocast alloys was very similar at comparable shear rates.

**Modified A356**

The A356 and A357 alloys begin eutectic freezing between 0.50 and 0.55 fraction solid, which limits their use to fractions solid below these levels. A356 was modified with pure Al to increase the amount of primary phase to at least 70%. Composition of the modified alloy is given in Table 1.
The modified A356 was compressed in the DFV at high shear rates in the fraction solid range of 0.44 to 0.67 (Table 2). A photograph of the compressed samples can be seen in Figure 13. Platen mass was increased to achieve high shear rates with the higher fraction solid samples. Figure 14 is a plot of the viscosity as a function of shear rate during the decreasing shear rate portion of a rapid compression experiment. As expected, viscosity increases dramatically with fraction solid, and a sample with a fraction solid as large as 0.67 compressed in a homogenous manner. This result implies that other alloys besides A356 that contain less Si would be suitable for semi-solid casting at higher fraction solid. The highest fraction solid successfully compressed \( (g_s = 0.67) \) was very similar to the peak fraction solid observed in the center of the long duration experiment \( (g_s = 0.69) \).

The viscosity of modified A356 was similar to A357 SIMA for a similar fraction solid and shear rate, as shown in Figure 15. Although the two alloys have different liquid composition and are at different temperatures, the viscosity behavior was the same, thus indicating the strong dependence of viscosity on fraction solid. When comparing the decreasing shear rate portion of the test, the modified A356 also acts like the A357 SIMA, as seen in Figure 16. The viscosity during the decreasing shear rate portion of the experiment is dependent on maximum shear rate, regardless of the processing route.

**Yield Stress Phenomenon**

Some researchers have experimentally determined that semi-solid slurries possess a significant yield stress, even at a fraction solid below 0.50.\[9,14\] Predictions of flow behavior of semi-solid alloys during forming operations would be affected by the inclusion of this term. If the yield stress is indeed significant relative to the viscous shear stress, the Stefan solution (Equation 3), which is used to calculate viscosity in the DFV, would be inappropriate for determining viscosity.

Covey and Stanmore derived a dimensionless number, the modified plasticity number \( S \) (Equation 7), to relatively compare the viscous and yield stresses.\[20\] The modified plasticity number was calculated using experimental data from a compression test of A357 SIMA at a fraction solid of 0.48. A value of 1000 Pa for the yield stress and a conservative steady-state viscosity value of 3 Pa·s was used to calculate the plasticity number as a function of time (Figure 17). The yield stress value is within the same order of magnitude of previously reported values,\[9,14\] and the viscosity was taken from the work of de Figueredo et al. at a fraction solid of 0.48 during steady-state, high shear rate experiments.\[10\]

Because \( S \) is greater than 1 for the majority of an experiment, the modified Stefan equation (Equation 5) may be used to calculate viscosity, assuming the yield stress is known a priori. Comparing the results of the classical Stefan equation (Equation 3) with the modified equation (Equation 5) using the previous values, 1000 Pa and 3 Pa·s, the viscosity changes by less than 1%. Clearly, yield stress does not have significant impact on the high shear rate Drop Forge Viscometer results from this work, and the use of the classical Stefan equation is justified. A yield stress may exist, but for the processing conditions normally encountered during rapid compression, i.e fraction solid of approximately 0.50 and shear rates between 100 and 1000 s\(^{-1}\), it is apparently insignificant.
IV. Conclusions

1. A novel apparatus, the Drop Forge Viscometer, was designed and constructed based on the parallel-plate compression viscometer. The DFV can operate at much higher shear rates ($>10^5 \text{ s}^{-1}$) than a conventional compression viscometer using a new technique for determining the applied force on a fluid that utilizes the second derivative of the sample displacement data. The duration of an experiment can be less than 10 ms, or if operated like a conventional parallel-plate viscometer, as long as 24 hours at shear rates as low as $10^{-5} \text{ s}^{-1}$. Results acquired with the DFV agreed with other rheological studies of semi-solid.

2. The DFV allows for calculation of instantaneous, volume-averaged viscosity under rapidly increasing then decreasing shear rate in periods of less than 10 ms.

3. During the increasing shear rate portion of an experiment, viscosity dropped by 1-2 orders of magnitude. Viscosity during the decreasing shear rate period decreased with increasing maximum achieved shear rate. For example, in two SIMA alloys of 0.48 fraction solid, the viscosity calculated at $100 \text{ s}^{-1}$ was an order of magnitude greater than a sample that had achieved a 75% higher shear rate.

4. Comparing this work with another transient study, it is evident during the decreasing shear rate period of an experiment that viscosity is not time dependent for the duration of approximately 0.01 to 1 s. Viscosity appears to be a function of the maximum achieved and the instantaneous shear rate, but not dependent on the length of time under shear.

5. The very rapid drop of viscosity with increasing shear rate, combined with the relatively slow increase of viscosity with decreasing shear rate, has significant implications for future machine and process design. For example, it should be possible to successfully cast higher fraction solid slurries than what is now feasible by applying vigorous shear prior to die filling, thus achieving low viscosity at the die entrance.

6. Viscosity was determined for non-dendritic aluminum-silicon alloys produced by three techniques, the SIMA, MHD, and MIT processes. The viscosity of each material was similar for a given shear rate.

7. Visual examination did not show separation of liquid and solid phases in any of the rapidly compressed samples in the shear rate range of 10 to 1500 $\text{ s}^{-1}$. Segregation was not detected in select samples using either quantitative metallography or chemical composition variation. However, when the DFV was used at low shear rates, there was a fraction solid difference of 0.20 after 16 minutes, and 0.69 after 4 hours. Segregation appears to increase with increasing strain in the low shear rate experiments.
8. The maximum fraction solid that was compressed by the DFV at high and low compression velocity was 0.67 and 0.69, respectively.

9. Yield stress, if it exists, does not appear to be significant for the process conditions observed in the DFV.

V. Acknowledgments

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VI. References


Table 1: Weight percent composition of alloys determined with wet chemical analysis (all compositions contain balance aluminum).

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<th>Alloy</th>
<th>%Si</th>
<th>%Mg</th>
<th>%Fe</th>
<th>%Mn</th>
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<td>7.0</td>
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Table 2: Master list of experiments discussed in this work.

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<th>Predicted Vol. Fraction Solid</th>
<th>Platen Mass (kg)</th>
<th>Max. Shear Rate (s⁻¹)</th>
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<td>0.48</td>
<td>0.379</td>
<td>0.0043</td>
<td>0.33</td>
</tr>
<tr>
<td>7</td>
<td>A357 MHD</td>
<td>579</td>
<td>0.48</td>
<td>0.379</td>
<td>295</td>
<td>0.28</td>
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<td>8</td>
<td>A357 MHD</td>
<td>579</td>
<td>0.48</td>
<td>0.379</td>
<td>280</td>
<td>0.27</td>
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<tr>
<td>9</td>
<td>A356 MIT</td>
<td>580</td>
<td>0.47</td>
<td>0.272</td>
<td>1270</td>
<td>0.21</td>
</tr>
<tr>
<td>10</td>
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<td>580</td>
<td>0.47</td>
<td>0.272</td>
<td>1500</td>
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<td>11</td>
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<td>0.379</td>
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<td>0.03</td>
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<td>13</td>
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<td>600</td>
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<td>0.56</td>
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<tr>
<td>14</td>
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<td>600</td>
<td>0.55</td>
<td>0.379</td>
<td>115</td>
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<td>15</td>
<td>Mod. A356 MIT</td>
<td>599</td>
<td>0.56</td>
<td>1.793</td>
<td>900</td>
<td>0.25</td>
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<tr>
<td>16</td>
<td>Mod. A356 MIT</td>
<td>594</td>
<td>0.60</td>
<td>1.793</td>
<td>375</td>
<td>0.31</td>
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<tr>
<td>17</td>
<td>Mod. A356 MIT</td>
<td>589</td>
<td>0.62</td>
<td>1.793</td>
<td>N/A</td>
<td>0.44</td>
</tr>
<tr>
<td>18</td>
<td>Mod. A356 MIT</td>
<td>579</td>
<td>0.67</td>
<td>1.793</td>
<td>60</td>
<td>0.59</td>
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</tbody>
</table>
Table 3: Comparison of fraction solid of the three A357 SIMA alloys compressed for different durations and shear rate. Fraction solid was determined optically (area fraction method) and using the lever rule with EDS data. The optical technique overestimates fraction solid, but the relative difference in fraction solid from both methods shows segregation increasing with strain for the low shear rate experiments.

<table>
<thead>
<tr>
<th>Exp. #</th>
<th>Duration of Experiment</th>
<th>Region</th>
<th>Si Content (wt% from EDS)</th>
<th>Vol. Fraction Solid</th>
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<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Optical</td>
</tr>
<tr>
<td>2</td>
<td>10 ms</td>
<td>Radial edge</td>
<td>7.15±0.5</td>
<td>0.71</td>
</tr>
<tr>
<td>2</td>
<td>10 ms</td>
<td>Center</td>
<td>6.26±0.5</td>
<td>0.76</td>
</tr>
<tr>
<td>5</td>
<td>16 min.</td>
<td>Radial edge</td>
<td>8.48±0.5</td>
<td>0.47</td>
</tr>
<tr>
<td>5</td>
<td>16 min.</td>
<td>Center</td>
<td>6.43±0.5</td>
<td>0.72</td>
</tr>
<tr>
<td>6</td>
<td>4 hours</td>
<td>Radial edge</td>
<td>11.95±0.5</td>
<td>0.25</td>
</tr>
<tr>
<td>6</td>
<td>4 hours</td>
<td>Center</td>
<td>4.71±0.5</td>
<td>0.78</td>
</tr>
</tbody>
</table>

Table 4: Quantitative metallography results of the three alloys prior to compression

<table>
<thead>
<tr>
<th>Alloy</th>
<th>Calculated Vol. Fraction Solid (gₐ)</th>
<th>Shape Factor (Fₛ)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A357 SIMA</td>
<td>0.45±0.02</td>
<td>0.81±0.05</td>
</tr>
<tr>
<td>A357 MHD</td>
<td>0.45±0.02</td>
<td>0.71±0.13</td>
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<tr>
<td>Mod. A356 MIT</td>
<td>0.42±0.02</td>
<td>0.78±0.06</td>
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</table>
Figure 1: Schematic of the Drop Forge Viscometer

Figure 2: Digital images from experiment 2 and displayed in 0.5 ms increments (every ninth frame). The thicker portion of the image is a marker on the platen rod that represents the position of the upper platen, and the velocity of the marker is decreasing with each subsequent image.
Figure 3: Instantaneous, dimensionless height (a) versus time for experiment 2, calculated from analyzing a series of images acquired with the DFV. The displacement data was used to calculate the instantaneous compression velocity (b) and acceleration (c) using the first and second derivatives with respect to time.
Figure 4: Temperature as a function of time for a sample heated to 579 °C in the furnace prior to a compression test.

Figure 5: (a) Instantaneous, volume-averaged viscosity and shear rate as a function of time for experiment 2. Error bars indicate a range of values that result when fraction solid and displacement vary ± 0.02 and ± 0.5 mm, respectively. (b) Data from (a) replotted as viscosity as a function of shear rate. The rate of change of viscosity is greater during the increase in shear rate.
Figure 6: Viscosity as a function of shear rate for A357 (experiments 1 and 2). Viscosity during the decreasing shear rate period is dependent on the maximum achieved shear rate.

Figure 7: Comparison of viscosity versus shear rate under a transient shear rate flow condition for A357 at \( g_0 = 0.48 \) from this work and de Figueredo et al. The durations of the two experiments were 6 milliseconds and 2 seconds, respectively. After 4 ms, the calculated viscosity from this work was similar to the other, and the viscosity does not appear to have any time dependence during the decreasing shear rate period relative to the durations of these experiments.
Figure 8: Comparison of A357 SIMA at a fraction solid of 0.48 (experiments 1-6) compressed with the DFV and similar alloys examined by other researchers.

Figure 9: Micrograph of a compressed sample (experiment 2) after fast compression near (a) the center and (b) the radial edge of the specimen.
Figure 10a: Micrograph of a compressed sample (experiment 5) after slow compression for 16 minutes near (a) the center and (b) the radial edge of the specimen.
Figure 11: Micrograph of a compressed sample (experiment 6) after slow compression for 4 hours near (a) the center and (b) the radial edge of the specimen. (c) A sharp transition can be clearly seen after compression.
Figure 12: Comparison of the viscosity behavior of A356 and A357 produced by SIMA (experiments 1 and 2), MHD (experiments 7 and 8) and the MIT process routes (experiments 9 and 10). Viscosities are similar, but the maximum shear rate achieved by the MHD was lower than the other materials.

Figure 13: Photograph of modified A356 MIT material after compression in the DFV for varying fraction solid. The 0.44 and 0.48 and the 0.55, 0.60, 0.62, and 0.67 fraction solid samples were compressed with 0.379 and 1.793 kg masses, respectively (experiments 11, 12, and 15-18).
Figure 14: Calculated viscosity versus increasing shear rate of modified A356 MIT material.

Figure 15: Calculated viscosity as a function of increasing shear rate of modified A356 MIT and A357 SIMA material (experiments 1, 2 and 12). Fraction solid is similar but temperature and liquid composition are different for the two alloys.
Figure 16: Comparison of the modified A356 MIT and A357 SIMA material for the increasing and decreasing shear rate periods of an experiment (experiments 1, 2, and 12). Viscosity behavior of the MIT material is similar to the SIMA alloy.

Figure 17: Plot of the dimensionless modified plasticity number, $S$, as a function of time for experiment 2 with an assumed yield stress of 1000 Pa and a viscosity of 3 Pa-s.
B. Microstructural Evolution in Semi-Solid Alloys (MIT)
MICROSTRUCTURAL EVOLUTION OF SEMI-SOLID ALLOYS

Report No. PR–01#2

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PROJECT STATEMENT

Objectives

Develop new methods of forming structures for semi-solid metal forming.

Strategy

- Investigate mechanisms leading to the formation of structures suitable for semi-solid forming;
- Develop improved methods of producing these structures

ACHIEVEMENTS TO DATE

A new method was developed to produce non-dendritic aluminum-silicon alloys that employs a combination of rapid cooling and vigorous agitation of the melt during the first few degrees of solidification. The process has been used to produce near-ideal semi-solid metal structures in aluminum-silicon casting alloys.

CHANGES IN PROJECT STATEMENT

None
WORK PLANNED FOR THE NEXT QUARTER

The project has been completed.

PROJECT SCHEDULE

The project has been completed.

APPENDIX

A detailed description of the process is contained in the appendix.
Appendix

**Efficient Formation of Structures Suitable for Semi-Solid Forming**

R.A. Martinez¹, A.M. de Figueredo², J.A. Yurko¹, M.C. Flemings¹

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2. Metals Processing Institute, WPI, Worcester, MA 01609 USA

**Abstract**

It is shown in this work that slurries ideal for semi-solid forming can be produced by stirring during the first small fraction of solidification, provided that stirring is combined with rapid heat extraction. Based on this finding, a new approach has been developed in which a rotating copper rod, initially at room temperature, is immersed in an A356 alloy melt held just above its liquidus temperature. Results show that good non-dendritic material, essentially free of entrapped eutectic, can be formed in this manner. Experiments were conducted in which either the stirring time or the rotational speed of the copper rod was varied. The microstructures of the alloy produced were compared to determine the influence of these process variables. The average shape factor of the primary solid was calculated and plotted as a function of both stirring time and stirring speed. The results show that even when the rotating rod was removed after the formation of as little as 1 vol.% solid, a non-dendritic structure is formed. The observation that non-dendritic structures can be achieved by processing during the small, initial interval in the solidification range is of enormous importance. Using the new approach, excellent semi-solid material can be formed from a liquid alloy in a simple and efficient manner, an ability that is becoming increasingly important for making semi-solid technology more economical.

**Introduction**

Semi-solid Metal Forming (SSF) involves forming a part from partially solidified alloy, typically containing 50 vol.% primary solid. The unique non-dendritic morphology of the primary solid particles in SSF alloys allows the solid-liquid slurries to flow in a laminar fashion during forming, which in turn lead to parts with excellent mechanical properties. The available methods for creating non-dendritic slurries directly from a liquid melt, "rheocasting", have been difficult to integrate in a manufacturing setting. This has led to "thixocasting" becoming the dominant form of commercially practiced SSF. Thixocasting uses solidified billet, specially prepared by the Magneto-Hydro-Dynamic (MHD) process. The billet are reheated into the solid-liquid temperature range and then formed. Unfortunately, the thixocasting process has significantly higher costs for part manufacturers compared to conventional casting processes. It is becoming apparent that the rheocasting process route may offer considerable energy and cost savings over thixocasting. Researchers have begun to re-emphasize the search for efficient ways to create the necessary non-dendritic structure directly from fully liquid alloy.

It is clear that liquid flow contributes to the microstructural change from a dendritic to a non-dendritic morphology. The structural evolution in a vigorously agitated, solidifying alloy was reviewed by Flemings [1] and illustrated in Figure 1.
It is thought that as solidification proceeds, shear and agitation change the structure from a dendritic to a "rosette" morphology through ripening and dendrite break-up. Eventually the solid particles will have a spheroidal shape. The amount of microstructural change is known to increase with increasing shear rate and time, and decrease with increasing cooling rate. However, it has been unknown how much liquid flow and cooling are required to form the non-dendritic structure. Another undetermined factor is the solidification interval in which microstructural changes occur.

An experimental method was developed to provide rapid cooling and vigorous agitation to a molten aluminum A356 alloy held just above its liquidus temperature. The rapid cooling and stirring was induced by immersing a rotating copper rod, initially at room temperature, into the melt. The intensity of the liquid flow could be changed by varying the rotational speed of the immersed rod. The design allowed the rod to be quickly removed from the melt after cooling it various amounts below the liquidus temperature. In this way, the combination of cooling and agitation was applied over a narrow interval in the alloy's solidification range when only a few volume percent solid had formed. The basic steps in the experiments conducted are illustrated in Figure 2.

Figure 1. Evolution of the microstructure of an agitated solidifying alloy (a) the dendrite (b) dendritic growth (c) rosette (d) rosette after ripening (e) spheroidal particle. Figure from [1].

Figure 2. Illustration of the three basic steps in the new semi-solid processing approach.
Experimental Procedure

Experimentation was conducted using the modified air-recirculating resistance furnace shown in Figure 3. A large pneumatic piston was mounted on top of the furnace to raise and lower the rotating copper rod into the molten alloy held in the furnace below. An electric motor and controller fixed the rotational speed of the rod for each experiment. The melt temperature was recorded throughout each experiment with a K-type thermocouple and temperature datalogger. The rod could quickly be removed from the melt at specific temperatures by operating the pneumatic switches which controlled the extension of the piston.

Figure 3. Modified air-recirculating furnace used to process the A356 alloy. Cooling and agitation of the melt could be provided by immersing of a rotating copper rod, initially positioned directly above the furnace chamber.

For each experiment, 400 grams of commercial A356 aluminum alloy were melted in a high-density graphite crucible 2 1/2 inches in diameter. The crucible had a wall thickness of 1/4 inch and was spray coated with boron nitride. The copper rod had a diameter of 1/2 inch and was sprayed with a very thin layer of graphite mold release. This coating protected the copper from dissolving in the molten alloy.

The first set of experiments varied the time the copper rod remained in the melt, with all other solidification conditions remaining constant. These experiments were aimed at identifying the solidification interval where processing leads to significant microstructural change. A second set of experiments varied the rotational speed of the copper rod while holding the stirring time and other solidification conditions constant. These experiments were performed to assess the influence of fluid velocity on microstructural change, and identify the amount of liquid flow required to create the non-dendritic microstructure.

The microstructure of the solidified alloy produced in each experiment was observed and photographed to assess the degree of structural modification. A small section of the material produced in each experiment (taken from an identical location) was rapidly reheated to 580°C ($T_r=0.50$), water quenched, and photographed. The average shape factor, $F_0$, of the primary particles in the reheated material was calculated using measurements from image analysis software. Using the expression

$$F_0 = 4\pi \frac{(\text{Area})}{(\text{Perimeter})^2}$$

a perfectly spherical particle would have a shape factor value of unity, and a long needle-like particle would have a shape factor close to zero. Reheating and quenching ensured the eutectic would have good contrast with the primary solid particles, making it easier to measure the area and perimeter of each particle.
Although some spheroidization due to ripening would occur during reheating, it was assumed that its effect was minimal and constant for each sample, all of which were in the semi-solid temperature range for only 3 minutes.

**Results**

**Typical structures**

Figure 4(a) shows the typical as-cast microstructures of alloy produced by the experiments. In this example, the alloy was processed by inserting the copper rod for 15 seconds, rotating at 1237 RPM. The rod was removed at a temperature corresponding to the formation of only 0.05 volume fraction solid. The micrograph clearly shows that the primary solid particles are spheroidal in nature, very different from the dendritic structure shown in Figure 4(b), which shows the microstructure that formed when the rod was not inserted.

![Figure 4(a)](image1.png)  ![Figure 4(b)](image2.png)

Figure 4. (a) The typical microstructure produced by immersing the rod for 15 s, rotating at 1237 RPM. (b) The microstructure of un-processed, dendritic alloy.

Figure 5(a) and (b) compare the reheated structure of the "MIT" material (shown in Figure 4(a)) with commercial MHD A357 alloy reheated in an identical manner. The MHD material was taken from a 3 inch diameter billet. The primary particles of the reheated MIT material are on the order of 200 μm, considerably smaller than those in the MHD material. The sphericity of the particles in the MIT material appears better than in the MHD material. This visual comparison indicates: that non-dendritic material can be formed by providing vigorous agitation and rapid cooling during just the first few degrees of solidification, and that the material produced by this technique has a microstructure considerably better than MHD processed alloy.

Another important difference in the reheated structures shown in Figure 5 is the amount of entrapped eutectic. The entrapped eutectic appears as dark spots inside the lighter primary solid. This entrapped eutectic represents "entrapped liquid" in the semisolid temperature range when eutectic compositions are liquid. Almost no entrapped eutectic is evident in the MIT material while much more is present in the MHD material.
Figure 5. (a) The same material as in Figure 4(a) reheated to 585 °C for 10 minutes, and quenched. (b) The structure of 3 inch diameter MHD billet reheated to 585 °C for 10 minutes, and quenched.

Stirring Time Variation

Figure 6(a), (b), (c), and (d) show the microstructure of the samples stirred for 20, 11, 5, and 2 second periods. The micrographs were taken from identical locations: half the distance between the middle and the edge of the ingot and half way up from the bottom. The rod's rotational speed was fixed at 508 RPM. It was inserted when the melt was at 619 °C, only 2 °C above the liquidus temperature. The temperature of the melt when the rod was removed and the corresponding fraction solid are given in Table 1. After removing the rod, cooling of the melt was similar for each experiment.

Figure 6. Microstructures of the A356 alloy when the copper rod stirred for (a) 20 s (b) 11 s (c) 5 s (d) 2 s.
Table I

<table>
<thead>
<tr>
<th>Stirring Time (s)</th>
<th>Temperature at removal (°C)</th>
<th>Fraction Solid at removal</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>617 (just below liquidus)</td>
<td>~0.01</td>
</tr>
<tr>
<td>5</td>
<td>616</td>
<td>0.02</td>
</tr>
<tr>
<td>11</td>
<td>615</td>
<td>0.05</td>
</tr>
<tr>
<td>20</td>
<td>614</td>
<td>0.07</td>
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</table>

By comparing the microstructures in Figures 6(a), (b), (c), and (d), it is apparent that stirring for 2 seconds can produce a non-dendritic structure similar to one produced by stirring for 5, 11, or 20 seconds. The primary particles in each of the micrographs appear non-dendritic and uniform in size. This observation is supported by the average shape factor calculations for these experiments. The number of particles measured, average shape factor value, and standard deviation for reheated material stirred for various times are given in Table II. Figure 7 shows how the average shape factor has a constant value near 0.75 regardless of how long the rod remained in the melt and how much cooling and solidification this induced. The plot supports the observation that similar structures are created by stirring for 2 or 20 seconds.

Table II

<table>
<thead>
<tr>
<th>Stirring Time (s)</th>
<th># of particles measured</th>
<th>Avg. Shape Factor</th>
<th>Std. Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>2</td>
<td>123</td>
<td>0.77</td>
<td>13%</td>
</tr>
<tr>
<td>5</td>
<td>122</td>
<td>0.72</td>
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</tr>
<tr>
<td>11</td>
<td>100</td>
<td>0.75</td>
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</tr>
<tr>
<td>20</td>
<td>123</td>
<td>0.76</td>
<td>15%</td>
</tr>
</tbody>
</table>

Figure 7. The effect of varying the stirring speed on the average shape factor of the primary particles.
Stirring Speed Variation

Figure 8(a), (b), (c) show the microstructure of material processed by spinning the rod at 1001 RPM, 508 RPM, and 60 RPM. As a point of reference, Figure 8(d) was also included to show the dendritic structure of alloy that solidified without inserting the copper rod. The micrographs for each sample were taken from equivalent locations: half the distance between the middle and the edge of the sample, and half way between the top and bottom. For each of these experiments, the rod remained in the melt for 20 seconds, and was inserted when each melt was at 620 °C. After the rod was removed, each melt was cooled similarly.

Figure 8. The effect of varying the rotational speed of the copper rod. (a) 1001 RPM (b) 508 RPM (c) 60 RPM (d) unprocessed dendritic alloy

Table III shows the number of particles measured, average shape factor value, and standard deviation for reheated material originally stirred with various speeds. Figure 9 shows the average shape factor has its highest value near 0.78 when the stirring speed was 1001 RPM and only slightly decreases to a value of 0.73 for 508 RPM, and 0.71 for 60 RPM. The shape factor decreases dramatically to a value near 0.5 for the unstirred dendritic material. It is interesting to notice that the standard deviation in the shape factor calculation increases as stirring speed decreases, as shown in Table III. The normalized standard deviation for the dendritic material is 39%. For the material stirred with 1001 RPM it is only 14%. This data indicates that the shape of the particles became more uniform when the material is processed with higher stirring speeds.
Table III

<table>
<thead>
<tr>
<th>Stirring Speed (RPM)</th>
<th># of particles measured</th>
<th>Avg. Shape Factor</th>
<th>Std. Deviation</th>
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<tr>
<td>&quot;0&quot;</td>
<td>57</td>
<td>0.51</td>
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<td>60</td>
<td>67</td>
<td>0.71</td>
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<td>508</td>
<td>127</td>
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</tr>
<tr>
<td>1001</td>
<td>155</td>
<td>0.78</td>
<td>14%</td>
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</table>

Figure 9. The effect of stirring time on the average shape factor of the primary particles.

Discussion

The microstructures produced by the MIT method have little, if any, eutectic entrapped within the interior of the primary particles. The microstructure of the MHD material shows a greater amount of entrapped eutectic. The absence of entrapped eutectic is significant during forming operations when eutectic compositions are liquid. Problems can arise even if only a few volume percent liquid is entrapped, because a small change in volume fraction liquid can correspond to a large change in the apparent viscosity of the semi-solid alloy. In a thixocasting operation, it is difficult to determine how much entrapped liquid is present in each reheated slug. Therefore, it is difficult to predict the apparent viscosity of each slug, which may contain different amounts of entrapped liquid. When the apparent viscosity of the semi-solid material varies from slug to slug, it is impossible to know the forming pressure required to make each slug flow equivalently. This may lead to a high reject ratio. For this reason, the lack of entrapped eutectic in the material produced by the MIT method is an appealing attribute for its use in manufacturing.

From Figure 8 it is apparent that more spheroidal primary particles are created when higher stirring speeds of 1001 or 508 RPM were used. However, as shown by the shape factor plot in Figure 9, only a moderate rotational speed of 60 RPM can dramatically increase the sphericity of the primary particles.
The most significant observation made from the "stirring time variation" experiments comes from looking at the solid fraction present when the rod was removed from the melt. As shown in Table I, stirring for 2 seconds corresponded to less than a degree of cooling below the liquidus temperature, and the solidification of roughly 1 vol.% solid. The microstructure in Figure 6(d) clearly shows a non-dendritic structure, achieved by processing the alloy with rapid cooling and agitating until the first 1 vol% of solid formed. Stirring for 20 seconds corresponded to 3 degrees of cooling below the liquidus, and 7 vol.% solidified. The average shape factor value of material stirred for 2 s is essentially the same as that for alloy stirred for 20 s, as shown by Figure 7. This evidence is momentous in identifying when during alloy solidification does processing create the non-dendritic structure. The results of these experiments suggest that the formation of the non-dendritic morphology occurred very quickly after the onset of solidification, even when only 1 vol% of the total solid to form was exposed to rapid cooling and agitation. It appears that this brief interval when solidification begins is where most, and maybe all, microstructural change occurs.

Conclusions

A new processing approach for the formation of non-dendritic structures has been developed. The technique involves immersing a rotating copper rod, initially at room temperature, into a molten alloy held just above its liquidus temperature. The rod rapidly cools the melt below its liquidus to initiate solidification, while vigorously stirring the melt. It is only required to remain in the melt for a short period, just long enough to cool it a few degrees below the liquidus temperature. The technique creates semi-solid slurries directly from the liquid alloy in a simple and efficient manner, an ability that is becoming increasingly important for making semi-solid technology more economical.

Reheated and quenched material formed by this process has a microstructure considerably better in particle shape and size compared to reheated MHD billet (3 inch diameter). Using the new MIT process, the non-dendritic material is essentially free of entrapped eutectic.

The solidification interval where microstructural changes occur has been recognized. The experiments show that processing with rapid cooling and agitation during the solidification of the first 1 vol.% solid is most critical for the formation of the non-dendritic structure.

The intensity of the stirring was shown to influence the sphericity of the solidified particles. Stirring the melt with a rotational speed as little as 60 RPM dramatically increased the sphericity of the primary solid (compared to dendritic material). Stirring with much higher rotational speeds of 508 and 1001 RPM only slightly improved the particle sphericity achieved by stirring with 60 RPM.

Acknowledgments

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References

C. Development of Alternate Semi-Solid Aluminum Alloys (ORNL)
Development of Alternate SSM Alloys

Report No. PR-01-#2

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PROJECT STATEMENT

Objectives

- Model thermodynamic phase equilibria for alternate alloy systems, such as aluminum-silicon-magnesium, aluminum-magnesium, and aluminum-copper.

- Model solidification behavior and determine key characteristics.

- Use the information on solidification behavior to tailor and develop alloys that are better suited for SSM processing.

Strategy

- Use Thermocalc and Dictra thermodynamic modeling packages together with an aluminum alloy database to determine phase equilibria for SSM alloys.

- Identify key characteristics of the alloy that can be determined from model.
• Identify desired characteristics of an ideal SSM alloy.

• Use thermodynamic model to develop alloy with ideal characteristics.

• Carry out tests on new alloys to provide feedback and validate results.

ACHIEVEMENTS THIS QUARTER

• Dilatometry test on SSM 357 alloy has been completed.

• 357 alloy has been die cast and dilatometry samples have been prepared.

Appendix A is a detailed report on the development of alternate SSM alloys

CHANGES IN PROJECT STATEMENTS

None

WORK PLANNED FOR NEXT QUARTER

• Dilatometry tests on die cast 357 alloy samples.

• Comparison of kinetic phase transformation behavior of semi-solid alloy and die casting alloy.

OPERATIONAL SCHEDULE

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<td>Analyze Phase Transformations</td>
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Development of Alternate SSM Alloys

Final Report

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Abstract

Currently, established casting alloys such as aluminum A356 and 357 alloy are also used for semi-solid metal casting (SSM). Process inconsistencies arising from the use of these alloys are partly due to variations in the solid fraction due to temperature variations. Also, the requirement of A356 and 357 alloys to operate at approximately 50% solid fraction, and consequently 50% liquid fraction, results in significant loss of eutectic by dripping during the reheating stage. Consequently, the process would be more robust if the alloys were tailored such that the solid fraction variation with temperature within the SSM process window is minimized. It would also be an advantage to operate at higher solid fractions. For this purpose, thermodynamic simulations have been carried out to determine the phase equilibria and solidification characteristics of 357 alloy. The solid fraction and the solid fraction variation of 357 alloy as a function of processing temperature and alloy composition have been determined. Based on the simulation results, aluminum-silicon alloys have been optimized for the minimum solid fraction variation over the full composition range of 357 alloy. Alternate SSM alloys in the 357 alloy family have been proposed. Similar simulations have also been carried out in aluminum-magnesium-silicon alloys (500 series alloys) containing 4 to 10% magnesium and 0 to 3% silicon. The solid fraction and solid fraction variation as a function of processing temperature, operation window, and alloy composition have been computed. A peak in the variation of solid fraction verses processing temperature has been identified, and it is around 560-590°C depending on alloy composition. It appears that aluminum-magnesium-silicon alloys may be processed at solid fractions greater than 0.5 (i.e., 50% solid). The results suggest that the aluminum-magnesium alloys have a good potential as robust alloys for semi-solid processing if the processing temperature is chosen such that the peak in solid fraction variation is avoided.

I. Objective

- Model thermodynamic phase equilibria for 357 alloy and alternate alloy systems.
- Model solidification behavior and determine key characteristics.
- Use the information on solidification behavior to tailor and develop alloys that are better suited for SSM processing.
II. Highlights

- Phase equilibria for complex alloys have been determined. Phase diagrams of commercial alloys containing seven elements have been calculated.
- Solidification behavior of commercial SSM alloys has been simulated using computational thermodynamics software.
- Key solidification characteristics have been determined.
- Optimum compositions of robust alloy for semi-solid processing have been determined. Alternate SSM alloys have been suggested.

III. Results/Summary

1. Phase Equilibria

Phase diagrams of commercial aluminum alloys have been simulated using the latest aluminum database and computational thermodynamics software. Figure 1 shows a vertical section of an alloy system containing seven elements (Al-Si-Mg-Fe-Cu-Mn-Zn-Ti), in which the contents of magnesium, iron, copper, manganese, zinc, and titanium are fixed at 0.55, 0.1, 0.008, 0.0005, 0.001, and 0.06 wt% respectively. The phase diagram shows phases that will precipitate under equilibrium conditions for alloys containing 0 to 14% silicon. A large number of commercial alloys fall in this silicon composition range.

Figure 1. A vertical section of an alloy system containing seven elements.
2. Solidification Behavior of SSM 357 Alloy

The solidification of aluminum 357 alloy containing 7% silicon has been simulated. Due to elemental partitioning during solidification, solutes are enriched in the liquid as the primary FCC aluminum dendrites grow. The solidification path of the alloy is shown in Figure 2. The liquid composition changes along a straight line (tie-line) until it reaches the binary eutectic valley (L→FCC+Si). The composition then varies along the eutectic valley. Under equilibrium conditions, solidification ends at the eutectic valley. Under the non-equilibrium conditions that exist during SSM processing, Scheil conditions are more appropriate and the simulation shows that more complex eutectics are precipitated.

![Figure 2. Solidification path of 357 SSM alloy containing 7wt%Si.](image_url)

Figure 3 shows the solid fraction versus temperature curves for a 357 alloy (7.0%Si, 0.5%Mg, 0.1%Fe, 0.13%Ti, 0.05%Mn, 0.01%Cu and 0.005%Zn). Aluminum-rich dendrites form below the liquidus temperature of 616 °C. Eutectic silicon forms at the eutectic start temperature, $T_{EU}$ where the solid fraction is about 0.5. Ternary and more complex eutectics precipitate at the end of solidification when the solid fraction is high. In the solid fraction range ($f_s < 0.6$) that is of interest in SSM processing, there is little difference between the curves calculated assuming equilibrium or Scheil conditions. Since the solid fraction versus temperature curve is strongly affected by the composition of the alloy, a large number of solid fraction vs. temperature curves for 357 alloy have been generated for various silicon and magnesium concentrations.

3. Key solidification characteristics of SSM 357 alloy

3.1. Eutectic start temperature, $T_{EU}$

An important feature on the temperature-solid fraction curve is that there is a sharp transition on the $T-f_s$ curve at the eutectic start temperature, $T_{EU}$, shown in Figure 4. When the temperature is higher than $T_{EU}$, a temperature variation doesn't cause a large
solid fraction variation. When the temperature is lower than $T_{EU}$, a small temperature change can result in a large solid fraction variation. The first derivative of the solid fraction with respect to temperature yields the dotted curve in Figure 4. The solid fraction variation decreases with decreasing temperature when $T > T_{EU}$, and reaches a minimum as $T$ approaches $T_{EU}$. At $T = T_{EU}$, a sharp increase in solid fraction variation occurs and the solid fraction variation increases from 0.006 to 0.11, an increase of one and a half orders of magnitude. Consequently, the processing temperature of the alloy should be higher than $T_{EU}$ in order to avoid a large solid fraction variation. However, a high processing temperature results in a lower solid fraction. $T_{EU}$ can be used to determine the ideal temperature for SSM processing. A large number of simulations were carried out to determine the eutectic start temperature as a function of composition.

**Figure 3.** Typical solid fraction verses temperature curves.

**Figure 4.** Relationship between the solid fraction, solid fraction variation, and the processing temperature. A peak in solid fraction variation occurs at the eutectic start temperature.
3.2. Solid fraction variation, $\Delta f_p$

Solid fraction variation is strongly affected by the processing temperature, $T_p$, temperature window, $(T_p-\Delta T_p, T_p+\Delta T_p)$, and the composition of the alloy. The solid fraction variation as a function of processing temperature, processing window and silicon concentration in 357 alloy is plotted in Figure 5 (a) and (b). Figure 5(a) was obtained when the processing window was 10°C ($\Delta T_p=5^\circ C$) and Figure 5(b) was obtained when the processing window was 20°C ($\Delta T_p=10^\circ C$). For comparison, the curves of solid fraction versus processing temperature are plotted in Figure 5(c). The following conclusions may be drawn from the results: 1) The solid fraction variation, $\Delta f_p$, increases with increasing silicon concentration for a given processing temperature and processing window. Thus, reducing silicon concentration in the alloy reduces solid fraction variation during semisolid processing. 2) The processing temperature at which a minimum solid fraction occurs increases with increasing silicon concentration. 3) The solid fraction variation increases when the processing window is increased.
A large number of simulations were also carried out to investigate the effect of magnesium concentration on the solid fraction variation. The results show that the minimum solid fraction variation and the temperature at which the minimum solid fraction occurs decrease with increasing magnesium concentration.

4. Alternate SSM Compositions for 357 Alloy

The above results can be used for developing robust alloys with a large processing window (i.e., low solid fraction variation in the temperature range of operation) and a desirable solid fraction for SSM processing. The solid fraction and solid fraction variation are functions of the processing temperature, temperature window, and chemical composition of the alloy. For a processing window of 10°C and a target solid fraction of 0.5, the composition at which the solid fraction variation is minimized is 6.5%Si and the alloy should be processed at 577°C. If the temperature window is 20°C and the target solid fraction is 0.5, the ideal alloy should contain 6%Si and the alloy should be processed at 582°C.

5. Other Possible SSM alloys

Extensive simulations of phase equilibria, solidification characteristics, and processing window were also carried out for Al-Mg alloys (500 series alloys). Alloys in the range Al-(1~10)Mg-(1~3)Si-0.5Fe-0.15Cu-0.35Mn-0.15Zn-0.25Ti were considered. Solid fraction and solid fraction variation as a function of processing temperature over the whole composition range of Al-Mg alloys were computed. The results indicate that 500 series alloys have good potential for semi-solid processing and that these alloys may be processed at solid fractions greater than 0.5. The main results are as follows:
(1) 500 series alloys with a silicon concentration of about 0.5% have a very good potential for SSM processing. The solid fraction and solid fraction variation as a function of processing temperature and composition are illustrated at Figure 6 (a) and 6(b). The solid fraction variation decreases with decreasing processing temperature when the silicon concentration in the alloy is 0.5 wt%. More importantly, these alloys do not exhibit a sudden increase in the solid fraction variation in the solid fraction range of 0.4-0.6. This indicates that the low silicon alloys have a good potential for SSM processing. The alloys may even be processed at solid fractions greater than 0.5.

![Graph (a)](#)

![Graph (b)](#)

Figure 6. The solid fraction (a) and solid fraction variation (b) as a function of processing temperature and magnesium concentration for alloys containing 0.5% silicon. Figure 6(b) was calculated for processing temperature variation of 10°C.

(2) Al-4%Mg-(0.5-2)Si alloys have potential for SSM processing. Figure 7 shows the solid fraction and solid fraction variation as a function of processing temperature. The
minimum solid fraction variation occurs around 0.6 solid fraction and so the alloy may be processed at solid fractions greater than 0.5.

Figure 7. The solid fraction and solid fraction variation as a function of processing temperature for Al-4%Mg-(0.5~2)Si alloy. The processing temperature variation is assumed to be 10°C.

(3) Al-6%Mg-(0.5~2)Si alloys also have potential for SSM processing. The solid fraction and solid fraction variation curves are shown in Figure 8. These curves are very similar to those of 357 alloys. The minimum solid fraction variation occurs at 0.5 solid fraction.

Figure 8. The solid fraction and solid fraction variation as a function of processing temperature for Al-4%Mg-(0.5~2)Si alloy. The processing temperature variation is assumed to be 10°C.

(4) When the silicon concentration is higher than 1 wt%, a peak in solid fraction variation has been identified for all compositions [see Figure 9(a), 9(b) and 9(c)]. The peak occurs around 560~590°C and the peak value increases with increasing silicon concentration. The processing temperature for these alloys should be determined such that the peak in solid fraction variation is avoided.
Figure 9. The solid fraction variation as a function of processing temperature in alloys containing (a) 1.0% Si, (b) 2.0% Si, and (c) 3.0% Si.
Acknowledgments

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