Optimization of the Heat Treatment of Semi Solid Processed A356 Aluminum Alloy

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OPTIMIZATION OF THE HEAT TREATMENT OF SEMI SOLID PROCESSED

A356 ALUMINUM ALLOY

By

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ABSTRACT
This research investigated the relationship between T5 heat treatment and elongation in thixocast and rheocast SSM components as a means to reduce the energy, time, and cost associated with T6 treatments while still producing improved properties over the as-cast condition. Temperature and time were varied as a part of work to optimize aging conditions for SSM materials. Both conventional furnace and fluidized bed heat treatments were employed. Tensile bars were fabricated from the heat treated A356 components and were pulled. Extensive SEM and stereo microscopy were performed to examine the factors which produced favorable results in the T5 condition. Data generated for T6 and as-cast components were also collected for purposes of comparison. Quality index calculations were employed to help evaluate the results. Optimized procedures and aging parameters have been presented.
ACKNOWLEDGEMENTS

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1.0 INTRODUCTION

Since the bronze age, casting techniques have involved liquid melt processing in one fashion or another. Very recently, however, a new technique has been introduced. Due to the differences in thermal history and rheological character between semi solid material and liquid metal, the necessary processing steps are not the same as they have been for thousands of years of foundry experience. The intuition of foundrymen, developed as the trade itself did over the millennia, is not necessarily correct when it comes to semi solid metallurgy.

Although knowledge of the mechanisms behind heat treatment of metals is not nearly as old as metal casting itself, those mechanisms have been around for almost 100 years. Through forging, heat treatments have been around in one fashion or another for much longer. As with all new processes, after the mechanisms for SSM heat treatment were commercialized, the first heat treatments applied to it were essentially those already in use for dendritic materials. These treatment regimes are not necessarily the optimal ones, as the differing microstructure and solidification history of SSM components changes a number of factors.

There are two main routes for producing semisolid aluminum parts. These are thixocasting and rheocasting. Thixocasting involves a specially-prepared billet of solid material that is subsequently heated into the semisolid range. Due to the
preparation of the billet, the final microstructure displays a series of spheroidal or rosette α-aluminum globules surrounded by the eutectic [1]. The other route, rheocasting, involves taking liquid and cooling it into the semisolid range. Measures are taken to provide copious nucleation and to retard grain growth. Once the part is cooled, the microstructure exhibits similar spheroidal structures to those found with thixocast materials but lacks entrapped eutectic. A356 aluminum has been selected as the alloy system to study in both thixocast and rheocast conditions. Two chief benefits of SSM processing are the presence of a stable flow front which helps prevent the entrapment of air in parts, and a reduced processing temperature which helps to extend die life significantly. In this work, rheocast components were produced by THT Presses (Dayton OH, USA) using the sub liquidus casting (SLC) process and thixocast components were produced by Salzburger Aluminium AG (Salzburg, Austria).
In general, due to the increased costs associated with recycling thixocast scrap as well as the necessity for an outside manufacturer for billet production, the trend in industry has been towards a new generation of rheocast routes to generate SSM components. It is expected that this trend will continue, though properties of both thixocast and rheocast material has been investigated in this thesis. Thixocasting data was included for the sake of completeness, to assist those still using thixocast material, and to connect this research to earlier research in this area.

In the silver anniversary lecture at the 108th AFS casting congress in Chicago, Mr. John Jorstad pointed out the promise of SSM in the T5 heat treated condition to meet or exceed design specifications while reducing energy costs [2]. He explained how as time passed, it was discovered that certain heat treatments in SSM produced desirable combinations of material properties. In particular, he discussed how quenched and T5 aged SSM components retained their high as-cast ductility while improving yield strength and ultimate tensile strength to values just below the same material in the T6 condition. However, the mechanisms of this retention of ductility are still poorly understood and there are questions regarding the generality of this behavior.

The heat treatment principles for conventionally cast aluminum alloys are well understood, but the different microstructure and solidification history of SSM
components indicate that heat treatment conditions which were optimized for conventionally cast materials do not apply to SSM components. Consequently, a combination of heat treatment and tensile testing with detailed microstructural analysis of samples before and after heat treatment has been conducted.

An examination of case studies of SSM parts from the 8\textsuperscript{th} Biannual S2P conference shows a majority of the castings in the 356/357 family of Al Si Mg alloys are heat treated to the T5 temper [3]. The chief reasons for selecting SSM processing for these parts were the high toughness, ductility, strength, and low porosity of SSM components.

Fluidized bed technology, through the fluidization of hot sand via blown air, is another modern technique to improve the heat treatment of castings. The high heat transfer coefficients associated with the process, as well as uniform bed temperature, allow for heat treatments which take place in dramatically less time as compared to conventional furnace heat treatments with lower heat transfer coefficients. As with most new processes, there is still some discussion of the mechanistic details of this process. This project is concerned chiefly with to what extent fluid bed techniques improve the properties of castings as compared to conventional furnace techniques.
As a consequence of these factors, the Department of Energy (DoE) and Worcester Polytechnic Institute Advanced Casting Research Center (WPI ACRC), along with the ACRC’s industrial partners, have sponsored this work as part of the Energy Smarrt Program- “Innovative Semi-Solid Metal (SSM) Processing. The goal is to optimize the heat treatment process of SSM components to reduce energy consumption, and consequently, manufacturing cost.

The unique microstructure of SSM processed components offers a shorter diffusion path, and the thermal history of the components promotes enhanced solutionizing before heat treatment begins. These factors combine to suggest that T6 heat treatments may be unnecessary to provide high quality aluminum components. Reduced cycle time and the outright elimination of T6 heat treatments would be highly advantageous in an era of increasing energy costs and decreasing energy reserves. This is what made research in this area so important.
2.0 LITERATURE REVIEW

An understanding of the historical context of this research assists in understanding the more pragmatic procedural decisions discussed later. This will begin with a discussion of the history of SSM processing, a relatively young field, including a discussion of the ‘Rheocasting Revolution,’ which has motivated much of the processing development research in the past decade. A basic discussion of the processing mechanisms is followed by a discussion of the aspects of heat treatment relevant to this research. The section concludes with a discussion of the fluidized bed method of heat treatment and the quality index, as well as a discussion of some specific work that was key in motivating this research.

2.1 History of Semi Solid Metal Forming (SSM)

The intent of this work was to evaluate the potential for heat treatment of this material. Consequently, the primary focus of this literature review is on heat treatment rather than the semi solid metal casting processes and this treatment of SSM processing will be brief.

A detailed history of the development of SSM metallurgy is described by Flemings and Kirkwood in their respective review papers, but a summary will be attempted [4, 5]. SSM processing was first discovered by Spencer while he worked on hot tearing in the lead-tin system [6]. It was found that liquid metal alloys which had been vigorously stirred through partial solidification exhibited novel
properties. In particular, material processed in this way would retain very high viscosities while still partially liquid. When a sheer stress was applied which exceeded a critical yield value, the material flowed as a viscous liquid. Billets could stand up under their own weight until sheered, as with a knife in the classic photo.

Multiple means were investigated in the beginning of SSM processing to prepare SSM material [1, 17-19]. The first billets which could be reheated into the semi solid range from room temperature were produced by the magnetohydrodynamic (MHD) process for producing thixotropic continuously cast billets.

In the MHD process, magnetohydrodynamic stirring is employed to fracture dendrites as they form.

In the past 10 years, a number of in-house mechanisms for producing castable SSM slurry have become available. Unlike the vigorous active mechanical stirring of earlier processing (whether rheocast or thixocast), the new rheocasting processes do not employ such vigorous means of agitation. Most of the new processes work via controlling the nucleation and growth process so that spheroidal rosettes are formed instead of larger dendritic networks. These developments have coincided with an increased interest by industry in SSM components, as part weight and cost become more of an issue and additional
material data became available. A variety of processes have been developed. The first of these new processes was the “New Rheocasting Process,” developed by Ube [1, 20-21]. The process used to produce the rheocast materials used in this work was the Sub Liquidus Casting (SLC) process, from THT presses [1, 22-25]. It makes use of a wide shot sleeve diecasting machine to produce the slurry in the shot sleeve, then injects that slurry into a die. B.J. Bernard details other processes extensively in his thesis [26-56].

2.2 Heat Treatment

2.2.1 Introduction to Heat Treatment

This work concerns itself with heat treatments intended to improve strength, but it should be noted that there are treatments which improve ductility at the expense of some strength (e.g. T4 treatments, annealing). Many wrought and cast aluminum alloys were specifically designed with heat treatment strengthening in mind. When strength does not occur from cold working or solid solution strengthening, natural or artificial aging by way of precipitation hardening is the only alternative. To be strengthened by heat treatments, the alloys must contain suitable elements. These substances must be soluble at one temperature but insoluble at a lower temperature. Examples for aluminum systems include magnesium, copper, and lithium. While some of these hardening elements remain in solution following casting, complete solution can be ensured by performing a solutionizing heat treatment. Whether or not the alloy is solutionized before artificial aging is the main difference between T5 and T6 heat
treatments. It should be noted that Mg$_2$Si is the hardening phase in A356, not Mg by itself [57-67].

2.2.2 The F Temper

Material which is cast and whose properties are not subsequently modified by either cold work or heat treatment is said to be in the “F” temper. In SSM, the F temper presents a high ductility with intermediate strength. The low strength of F temper aluminum alloy components generally relegates them to nonstructural applications. An exception is Silafont 37, which naturally ages in a short period of time to a relatively strong condition [68]. (Silafont was not designed with SSM casting in mind.)

2.2.3 The T5 Temper

During casting, at least some of the hardening atoms remain in solution. If the part is elevated in temperature, the rate of nucleation and growth of these precipitates can be accelerated. This is artificial aging (T5) treatment, and improves strength through precipitate hardening. In dendritic materials, the improvements in strength so obtained are at the expense of ductility. Some T5 treatments are performed for purposes of stabilization rather than improving strength. These treatments generally take place at a higher temperature and take less time as compared with strengthening treatments. The process of natural aging can lead to distortion and the gradual changes (improvements) in strength over time are undesirable in some applications.

If a part is quenched from the diecasting machine soon after it has solidified completely, a greater percentage of the solute will remain in solution. This modified treatment is sometimes referred to as a “QT5.” Benefits of a QT5 can be lost if it is not possible to quench the part shortly after casting.
2.2.4 The T6 Temper

The most common heat treatment for critical components composed of aluminum-silicon casting alloys containing a precipitate hardening constituent (the 3xx series alloys) is the T6 treatment. When alloys are modified with small amounts of strontium, the aluminum silicon eutectic which forms takes on a different morphology (A coral, rather than needle morphology). The coral structure which forms easily spherodizes during solution treatment. This change in eutectic morphology is responsible for the improved ductility of both dendritic and SSM components in the T6 even as strength is improved.

The primary reason for conducting the solutionizing treatment, however, is to insure that the solute (in the case of A356, magnesium and silicon) is at its maximum solubility in the aluminum matrix. This treatment also helps eliminate any coring which may have taken place.

Following the solution treatment, components are removed from the furnace and quenched. Care must be taken with this quenching treatment to avoid creating residual stresses or distortion by cooling too quickly, while still cooling quickly enough to insure incoherent precipitates do not form. Another important consequence of quenching is the retention of vacancies from the elevated solutionizing temperature which can promote higher rates of diffusion later on. Quenching liquids and procedures are altered to optimize this process.
Parts are held at room temperature prior to artificial aging. At this point, it is believed clusters of aging atoms begin to form GP zones. This time is controlled to insure process uniformity, with eight hours being standard.

Parts are subsequently artificially aged to accelerate the nucleation and growth of the precipitate hardening phases. Artificial aging of T6 components is essentially identical to that for T5 components, although there may be slight differences in temperature and time to insure optimal results. These temperature and time differences are a consequence of the varied levels of solute in the matrices.

### 2.2.5 Precipitation Hardening

All of the improvement of strength and reduction of ductility as a consequence of artificial aging treatments is due to precipitate hardening. Small phases of coherent or semi-coherent precipitates induce strain on the lattice of the phase with which they are at least partially coherent. This induced strain impedes dislocation motion, which in turn improves strength and reduces ductility. Due to the small size of the precipitates involved, detailed investigation of them is most often performed with a Transmission Electron Microscope (TEM) [69-71].

### 2.3 SSM Tensile and Heat Treatment Research

Even though a great deal of work has been carried out by many researchers over the years investigating the mechanical properties of SSM processed aluminum
based components, the literature is void of any systematic work which addresses T6 versus T5 tempers of SSM cast parts [1, 11, 50, 72-83].

2.4 Fluidized Bed Heat Treatment

2.4.1 Introduction to the Fluidized Bed process

Fluid bed technology has been around for some time, and is used in a wide variety of industries. There were a number of problems with the fluid bed systems first available to the aluminum casting industry, but the most recent designs have overcome them. Many of those early problems were related to the heating mechanism being the air itself, which meant that it was not possible to separate the inlet flow speed from the energy applied to the sand. Newer designs have corrected this problem.

In a fluid bed, sand is heated and air is forced up through it, causing the sand to flow. When the air flow is optimized, the heat transfer coefficients available are much higher than those of conventional furnaces. The high heat capacity of sand insures that the bed does not drop in temperature as parts are added. Many environmental problems associated with heat treatment are eliminated, but fluid beds have their own hazards, chief among which is that of fine particle inhalation. Because of the rapid kinetics of the process, parts can be heat treated in a fraction of the time. This eliminates a bottleneck in the supply chain, which is desirable to many manufacturers.
Quenching in water can produce residual stresses and distortion due to the severity of the quench. There are alternate quenching media with less severe quenching properties, but many have undesirable medical or environmental properties. Fluid bed quenching uses cool sand to quench the parts. The slow quench insures there will be no distortion. While this quench may be too slow for some applications, manufacturers especially interested in reducing the distortion of their parts are particularly attracted to fluid bed technology [84].

2.4.2 Mechanisms in Fluid Bed heat treatment

The primary way fluid bed heat treatment differs from conventional heat treatment is the higher heat transfer coefficient afforded by the fluidized sand. This leads to different mechanisms in the heat treatment process and allows for a marked reduction in the time of treatments [82, 85].

While in all heat treatments there is some propensity for grain growth, the mechanism whereby this occurs in strontium modified Al-Si-Mg alloys is Ostwald ripening, instead of grain coalescence. The precipitation kinetics of Mg$_2$Si particles are faster in fluidized bed heat treatment, and show an increased propensity for spherical shape [86-99].
Another factor allowing fluid bed treatments to be conducted in less time is the high degree of control the furnaces have with respect to bed temperature. The temperature of the bed can be controlled to within a few degrees centigrade, which means that higher solution temperatures can be used without risking individual components reaching yet higher temperatures. This also means that batches do not need to be heated longer so that the parts in the coldest part of the furnace reach their minimum properties, since all parts are heated equally \[84\].

### 2.5 High ductility SSM T5 treatments

It has been suggested by John Jorstad and others that it is possible to retain the high as-cast ductility in an SSM casting while improving strength through artificial aging. This was the subject of his silver-anniversary lecture at the AFS NADCA casting congress [2] as well as of a paper he co-authored with the author of this thesis for the following year’s congress [100]. That paper is included as an appendix to this thesis. Jorstad, Pan, and Apelian further explore the microstructural changes involved [101].

If T5 or QT5 treatments can be conducted so as to retain all or most of the high as-cast ductility while still producing strength values which rival those of the T6 condition, there are a number of benefits. The elimination of the solutionizing step will reduce energy consumed in the process, and the reduction in time and
energy expended will help to reduce costs. It remains unclear how the ductility can be retained while strength improves, but the most likely explanation seems to be that the unique thermal history and microstructure of SSM components allows for unusual, desirable microstructural changes. There is still some debate as to the generality of this behavior.

2.6 The Quality Index

The Quality Index was developed by Drouzy et al. as a measurement of the properties of aluminum casting alloys [102]. Drouzy’s initial formulation was based on the observation of the trends in empirical data. That work was expanded on and applied in the optimization of heat treatments by Shivkumar et al. [103]. C.H. Caceres et al. have done further work to show the fundamental basis of the quality index [104-108]. It is Drouzy’s definition of quality index which is used in this work, although there are many other indices called “the” Quality Index.
3.0 OBJECTIVES

- The primary objective of this research was to examine the potential for low cost/time/energy heat treatments for aluminum A356 SSM components.
  - Establishing whether or not the high as-cast (F) ductility could be retained, as suggested, in artificial aging (T5) heat treatments was an important aspect of this objective.
  - If the retention of as-cast ductility proved to be a real phenomenon, it would be important to establish the mechanism by which it operates.
  - Establishing the tensile properties of as-cast, T5, and T6 components was also related to the primary objective.
  - Fluid bed heat treatments also have the potential for improved aging efficiency, and were investigated as a part of this work towards those ends.
- Since thixocasting components are going out of fashion with many casters and rheocasting methods are continuing to develop, one objective of this work was to establish whether thixocast and rheocast components heat treat in the same manner.
- Temperature and time requirements for optimal tensile properties as well as operational windows were established.
• Although not an objective when this work began, it quickly became apparent that establishing the sensitivity of the experiment to variations of various experimental parameters was an important objective.
4.0 PROCEDURE

This section contains the procedures used in collecting the data presented in Section 5.0, Results and Discussion. Discussion begins with the materials used in this work including chemical analyses of each sample type. This is followed by details of the heat treatment procedures used for the T5 and T6 treatments in both fluid bed and conventional furnace heating situations. Tensile testing procedures, including the machining of tensile bars and fracture analysis subsequent to failure, detail how the critical physical property data were obtained. Finally, details of microstructural sample preparation as well as a brief discussion of the SEM, optical, and stereographic optical techniques used are presented.

4.1 Materials

Rheocast samples were supplied by THT Presses (THT) from their SLC process in two geometries, and Salzburger Aluminium AG (SAG) provided thixocast material. Pictures of the sample types follow (Figure 4.1).
While the 1” THT components used in this study were water quenched from the die, they were not necessarily quenched before reaching the critical temperature region. Consequently, the 1 inch THT bars in the T5 condition presented here are not necessarily “QT5.” Portions of the ¾” THT material were quenched from the die above this critical temperature by making use of an improved die design incorporating an ejection mechanism while other components were deliberately not quenched so that the effect of this step in the operation could be investigated.

4.1.2 Alloy Chemistry

Since samples were produced at different locations by different companies at different times, compositional differences between the batches of samples are
expected. To investigate the compositional differences, a Spectro spark analyzer\(^1\) was used. Parts analyzed are first ground with silicon carbide paper to produce a uniform, flat surface. After the sample has been placed on the tester and the arm which insures electrical contact is attached to the top of the sample, the software automatically purges the area of interest with argon gas, tests the pre-spark, and finally sparks the sample and analyzes the plasma produced. The sparked surface is visually inspected to insure that the test ran properly. Multiple good sparks (7 or more) are averaged to produce the results presented later in the report.

### 4.1.3 Thermodynamic Software Analysis

Using the compositions determined by spark testing, the relative fractions of phases stable at various aging temperatures were calculated using the Pandat software package. While these calculations do not represent the exact fractions of phases expected to be present after heat treatment, since no commercial heat treatment ever goes to equilibrium, they do indicate whether there is a significant difference between the various aging temperatures in terms of the phases which are nucleating at each temperature. The maximum theoretical safe temperature for solutionizing was computed. While the maximum temperature found was 562 °C, furnaces were set no higher than 555 °C, since no furnace is infinitely precise or accurate.

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\(^1\) Spectromax model LMXM3. 160 Authority Dr, Fitchburg MA 01420. (978)-342-3400.
4.2 Heat Treatment Conditions

Since an off-site manufacturer provided samples, there were several months of natural aging time between casting and heat treatment for the first batch of samples tested (thixocast and 1” rheocast). Resistance-heated box furnaces were used for conventional furnace heating.

There were two groupings of heat treatments performed. The initial group of trials was conducted on the thixocast and 1” rheocast components. The second group of trials was performed on the 3/4” rheocast components to address questions raised by the first set of trials. The second set of experiments included procedural improvements when the possible effect of the previous procedures was not under investigation.

All temperatures reported are the ones to which the furnace was set. This was done to promote repeatability and precision. It is known that there was a 10 °C thermal gradient between the top and bottom of the resistance-heated furnaces when the furnaces were below 250 °C. There were no large thermal gradients observed at solution temperatures (540 °C, 555 °C). Gradients at all temperatures in the fluid bed were less than 2 °C.
4.2.1 T5 Conventional Furnace

The conventional furnaces (resistance heated) were brought to temperature before parts were loaded into the furnace. It is known from past experience that with the furnace loads used in the T5 conventional furnace treatments, parts reach the temperatures of interest in an hour. Times listed begin after this one hour of heat up. All parts treated at a given temperature were held in the furnace at the same time.

With the first group of samples (1” rheocast and thixocast), the parts (10 per condition) were removed and quenched into room temperature water. Quenching is not a standard part of T5 treatments, but was done to ensure consistent cooling among different experimental runs and bars within the same batch. For the first trial, both thixocast and rheocast parts were heat treated in the same furnaces at a given temperature, as seen in Table 4.1 below.

With the second group of samples, this post aging quenching was only done for trials intended to test the effect of such quenching. There were six bars per condition tested in the second set of trials.

Samples were aged at different temperatures and for various intervals of time. The heat treatment schedule is shown below in Table 4.1. Bars were inspected for blistering subsequent to heat treatment, but as expected no blistering was
observed in the T5 treated samples. Some discoloration was evident, presumably because the parts were not cleaned prior to heat treatment and oils on the metal surface burned. Lubrication during the subsequent machining of these samples into tensile bars removed this discoloration. Below is a chart showing the temperatures and times of T5 experiments. All bars from the first THT samples and from selected portions of the second THT samples were ejected from the die and quenched to retain as much super saturated solute in solution as possible. SAG bars were somewhat thinner, and are believed to have solidified more quickly than unquenched THT material as a result, but they were not quenched.
Table 4.1: Heat treatment schedule for T5 temper in conventional furnaces

<table>
<thead>
<tr>
<th>Temper</th>
<th>Material</th>
<th>Dim. [cm]</th>
<th># bars</th>
<th>Quench</th>
<th>Age T. [°C]</th>
<th>Age time [hr]</th>
<th>Post quench</th>
</tr>
</thead>
<tbody>
<tr>
<td>F</td>
<td>rheocast</td>
<td>2.5x2.9</td>
<td>10</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>F</td>
<td>thixocast</td>
<td>3.2x0.3</td>
<td>10</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>F</td>
<td>rheocast</td>
<td>1.7x1.5</td>
<td>6</td>
<td>---</td>
<td>---</td>
<td>---</td>
<td>---</td>
</tr>
<tr>
<td>F</td>
<td>rheocast</td>
<td>1.7x1.5</td>
<td>6</td>
<td>---</td>
<td>---</td>
<td>---</td>
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</tr>
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<td>2.5x2.9</td>
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<td>180</td>
<td>4</td>
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<td>10</td>
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<td>180</td>
<td>8</td>
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<td>10</td>
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<td>10</td>
<td>---</td>
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<td>yes</td>
</tr>
<tr>
<td>QT5</td>
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<td>no</td>
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<td>from cast</td>
<td>170</td>
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<td>6</td>
<td>from cast</td>
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<tr>
<td>QT5</td>
<td>rheocast</td>
<td>1.7x1.5</td>
<td>6</td>
<td>from cast</td>
<td>180</td>
<td>4</td>
<td>no</td>
</tr>
<tr>
<td>QT5</td>
<td>rheocast</td>
<td>1.7x1.5</td>
<td>6</td>
<td>from cast</td>
<td>180</td>
<td>6</td>
<td>no</td>
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<tr>
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<td>6</td>
<td>---</td>
<td>170</td>
<td>6</td>
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<tr>
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<td>rheocast</td>
<td>1.7x1.5</td>
<td>6</td>
<td>---</td>
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<td>6</td>
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<tr>
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<td>rheocast</td>
<td>1.7x1.5</td>
<td>6</td>
<td>---</td>
<td>170</td>
<td>6</td>
<td>no</td>
</tr>
</tbody>
</table>

In the above chart, most entries are self-explanatory, however, some abbreviations for column headings have been necessary. “Temper” refers to the heat treatment employed. It serves primarily to distinguish as-cast (F) samples from common T5 samples, and quenched T5 samples (QT5) from unquenched T5 samples (UT5) when both are present for the same geometry. “Material”
refers to the process which produced the sample. “Dim” refers to the cross sectional dimensions of the sample. “# bars” indicates how many tensile bars were fabricated and tested. Some bars may have been eliminated due to material defects. “Quench,” in this chart, refers to whether or not the bars were quenched from the cast condition (“from cast”). “Age T” indicates the artificial aging temperature as measured by the furnace. “Age time” indicates the time at temperature for artificial aging. “Post Quench” indicates whether or not the bars were quenched in room temperature water subsequent to aging.

4.2.2 T6 Conventional Furnace

Thixocast and rheocast parts were heat treated to a T6 temper. T6 heat treatment involves a solutionizing step followed by quenching, a period of natural aging, then artificial aging at a temperature above room temperature but well below the solutionizing temperature. Calculations with the Pandat software indicated specific alloy chemistries would experience incipient melting at around 562 °C, so samples were not heated above this temperature. Times measured are times at temperature, (as was the case for the T5 treatments).

For the first set of trials, solutionizing time and temperature were varied, but aging temperature was not. Solutionizing was conducted at 540 °C and 555 °C, for between two and eight hours. After aging for five hours at 160 °C, bars were

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2 Pandat Version 4-0-M. Computherm, LLC. 437 S. Yellowstone Dr. Suite 217, Madison WI 53719 (608)-274-1414. www.computherm.com
again quenched into water to cease aging. This final quenching will be eliminated in future tests, as it may have created residual stresses that lowered the ductility of the bars, as discussed in Section 4.2.1. The effect of this post-age quenching was investigated in the second set of trials. Natural aging time between quenching and artificial aging was between 15 and 24 hours. Since natural aging proceeds logarithmically with time, significant variation in properties over this time range is not anticipated. This variation in natural aging time between quench and artificial aging was necessary because the bars were removed from the hot furnace after they had been in long enough rather than adding them such that all bars could be removed at the same time. Bars were quenched in room temperature water. These factors (e.g. natural aging time between quench and artificial aging, quench temperature) were investigated in the second set of trials.

In the second set of trials, bars were added to the furnace at appropriate times such that all bars are pulled and quenched at the same time. This allowed for more uniform natural aging times. The time used was the 8 hours specified by industrial standards for dendritic materials. Quenching water was 80 °C. Scrupulous attention was paid to the orientation of the bars as they entered the quench. They were suspended vertically and lowered into the bath so that there was separation between the bars. This was facilitated by wrapping wire around
one end and lowering them in like a string of firecrackers or socks on a laundry line.

Table 4.2: Heat treatment schedule for T6 temper in conventional furnaces

<table>
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<tr>
<th></th>
<th></th>
<th></th>
<th></th>
<th></th>
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<td>room T.</td>
<td>16.5</td>
<td>160</td>
<td>5</td>
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<tr>
<td>thix.</td>
<td>3.2x0.3</td>
<td>10</td>
<td>555</td>
<td>2</td>
<td>room T.</td>
<td>20.7</td>
<td>160</td>
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<tr>
<td>thix.</td>
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<td>555</td>
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<td>room T.</td>
<td>18.7</td>
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<td>555</td>
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<td>room T.</td>
<td>16.7</td>
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<td>3.2x0.3</td>
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<td>540</td>
<td>2</td>
<td>room T.</td>
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<tr>
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<td>3.2x0.3</td>
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<td>540</td>
<td>6</td>
<td>room T.</td>
<td>16.6</td>
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<td>rheo.</td>
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<td>540</td>
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<td>80 C</td>
<td>8</td>
<td>160</td>
<td>8</td>
<td>no</td>
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<tr>
<td>rheo.</td>
<td>1.7x1.5</td>
<td>6</td>
<td>540</td>
<td>4</td>
<td>80 C</td>
<td>8</td>
<td>160</td>
<td>8</td>
<td>no</td>
</tr>
<tr>
<td>rheo.</td>
<td>1.7x1.5</td>
<td>6</td>
<td>540</td>
<td>4</td>
<td>80 C</td>
<td>8</td>
<td>160</td>
<td>5</td>
<td>no</td>
</tr>
<tr>
<td>rheo.</td>
<td>1.7x1.5</td>
<td>6</td>
<td>540</td>
<td>4</td>
<td>80 C</td>
<td>8</td>
<td>170</td>
<td>6</td>
<td>no</td>
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<tr>
<td>rheo.</td>
<td>1.7x1.5</td>
<td>6</td>
<td>540</td>
<td>4</td>
<td>80 C</td>
<td>8</td>
<td>180</td>
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<td>160</td>
<td>5</td>
<td>no</td>
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</table>

In Table 4.2, most entries are self-explanatory, however some abbreviations for columns are necessary. “Mat,” short for material, refers to the process which produced the sample. “Dim” refers to the cross sectional dimensions of the sample. “# bars” indicates how many tensile bars were fabricated and tested. Some bars may have been eliminated due to material defects. “Sol’n T” refers to the solution temperature. “S. time” refers to the time at temperature for solution heat treatment. “Quench,” in this chart, refers to the temperature of the quenching water used following solution heat treatment, with “room T” referring
to room temperature water. “Nat’l age time” refers to the time between quenching and insertion into the furnace for artificial aging. “Age T” indicates the artificial aging temperature as measured by the furnace. “Age time” indicates the time at temperature for artificial aging. “Post Quench” indicates whether or not the bars were quenched in room temperature water subsequent to aging.

**4.2.3 T5 & T6 Fluidized Bed**

Heat treatment using the fluidized bed reactor is very similar to that conducted in the resistance heated furnaces, but times are greatly reduced by the increased heat transfer coefficient, and temperature variations within the furnace are much smaller. The time for parts to reach their treatment temperatures is also greatly reduced, but the times measured are times-at-temperature, as with the conventional furnace treatments.

Before inserting samples, the fluidization of the bed was visually examined to ensure that the flow rate was not too small, in which case proper fluidization and circulation of sand would not be achieved, or too great, in which case bubbles of gas would form which would reduce the heat transfer coefficient. Work to insure proper fluidization of the bed and to establish the heat transfer coefficients in the reactor have already been conducted [86-100].
Since it takes approximately two days for the reactor to cool from solutionizing to aging temperature, this is the length of the natural aging time experienced by the bars in T6 treatments between quenching and artificial aging. Times of natural aging cannot be reduced below this cooling rate limit while using a single fluidized bed unit. Variation in natural aging time was the result of different strategies and schedules used while cooling the bed. The most effective technique involved leaving the blower and venting on while the heat was off combined with close monitoring of temperature after 20 hours.

In the first set of trials, bars which were T5 heated were heated at 204 °C for 30 minutes. T6 bars were solutionized at 538 °C, quenched in room temperature water, and then aged at 204 °C for 30 minutes, just as the T5 samples were. Subsequent to the final aging in both cases, bars were quenched in water.

In the second set of trials the improvements discussed above under T6 heat treatment were also made. The experimental matrix of the second set of tests was larger than the initial T5 and T6 trials, and included lower temperature data.
Table 4.3: Heat treatment schedule for T6 temper in fluidized bed

<table>
<thead>
<tr>
<th>Mat.</th>
<th>Dim. [cm]</th>
<th># bars</th>
<th>Sol'n T. [°C]</th>
<th>S. time [hr]</th>
<th>Quench</th>
<th>Nat'l age time [hr]</th>
<th>Age T. [°C]</th>
<th>Age time [hr]</th>
<th>Post quench</th>
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<td>2.5x2.9</td>
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<td>204</td>
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<tr>
<td>rheo.</td>
<td>2.5x2.9</td>
<td>10</td>
<td>538</td>
<td>0.5</td>
<td>room T.</td>
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<td>204</td>
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<td>thix.</td>
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<td>---</td>
<td>---</td>
<td>---</td>
<td>204</td>
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</tr>
<tr>
<td>thix.</td>
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<td>0.5</td>
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<tr>
<td>rheo.</td>
<td>1.7x1.5</td>
<td>6</td>
<td>---</td>
<td>---</td>
<td>from cast</td>
<td>---</td>
<td>160</td>
<td>1</td>
<td>no</td>
</tr>
<tr>
<td>rheo.</td>
<td>1.7x1.5</td>
<td>6</td>
<td>---</td>
<td>---</td>
<td>from cast</td>
<td>---</td>
<td>170</td>
<td>1</td>
<td>no</td>
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<tr>
<td>rheo.</td>
<td>1.7x1.5</td>
<td>6</td>
<td>---</td>
<td>---</td>
<td>from cast</td>
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<td>180</td>
<td>1</td>
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<tr>
<td>rheo.</td>
<td>1.7x1.5</td>
<td>6</td>
<td>538</td>
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<td>rheo.</td>
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<td>80 C</td>
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<td>1</td>
<td>no</td>
</tr>
</tbody>
</table>

In Table 4.3, most entries are self-explanatory, however some abbreviations for columns were necessary. “Mat,” short for material, refers to the process which produced the sample. “Dim” refers to the cross sectional dimensions of the sample. “# bars” indicates how many tensile bars were fabricated and tested. Some bars may have been eliminated due to material defects “Sol’n T” refers to the solution temperature. “S. time” refers to the time at temperature for solution heat treatment. “Quench,” in this chart, refers to the temperature of the quenching water used following solution heat treatment, with “room T” referring to room temperature water and “from cast” referring to whether the bars were quenched from the cast condition if they received no other quenching prior to artificial aging. “Nat’l age time” refers to the time between quenching and insertion into the furnace for artificial aging. “Age T” indicates the artificial aging temperature as measured by the furnace. “Age time” indicates the time at
temperature for artificial aging. “Post Quench” indicates whether or not the bars were quenched in room temperature water subsequent to aging.

4.3 Tensile Testing

4.3.1 Machining

Tensile specimens and testing conformed to ASTM E8-98. Samples were heat treated prior to machining. Given the differences in initial geometry, the thixocast and rheocast samples could not be machined into identical tensile specimens. All were machined into rectangular ASTM ‘dog bone’ type tensile specimens.

Machining was done in the HAAS Technical Education Center at WPI. Part geometry required slight modification to the ASTM specifications, but the critical specifications (minimum radius of fillets and length of neck) were followed strictly. Since the SAG geometry was significantly narrower than the THT geometry, the SAG (thixocast) part follows the ASTM specification for a rectangular tensile bar which is one quarter of an inch in diameter at the neck and had a gauge length of 1”. The 1” THT (rheocast) samples were machined into bars which had neck thicknesses of ½” and had a gauge length of 2”. The narrower THT samples were machined with a 1” gauge length, but with longer grip sections than the SAG part as there was additional material available. The thixocast samples required subsequent band-saw cutting to free the tensile section from the rest of the part. Since this manual machining did not impact the
gauge section, the width of the grip section has a greater degree of variation, but this is not believed to have impacted results significantly.

In the case of both the 1” THT rheocast bar and the SAG thixocast bar, the difference between the ASTM standard and these tensile bars lies in the reduced length of the grip sections. Since the jaws that hold the samples function well, this reduction in length is not believed to have substantively changed the results. The smaller THT bars meet all ASTM specifications, including the length of the grip sections. It was found that significant lubrication and cooling was needed during the machining process to prevent ‘gumming up’ the tooling, but once the correct parameters were obtained, machining went very smoothly. Pictures of finished tensile bars follow.

![Figure 4.2: SSM tensile geometries. From top to bottom, THT 1”, SAG, THT 3/4”](image-url)

Figure 4.2: SSM tensile geometries. From top to bottom, THT 1”, SAG, THT 3/4”
4.3.2 The Tensile Test

The tensile tester used was an Instron Model #5500 that has been upgraded and is electronically controlled using Merlin software to control the test and record the data. The strain rate was 0.1 inches per minute, as per the ASTM standard. An extensometer\(^3\) was used to measure bar extension with increased precision. After fracture, samples were numbered with permanent marker so that subsequent fracture analysis could be correlated with the tensile testing results. Data for ultimate tensile strength (UTS), percent elongation, and the elastic modulus were immediately available, but automatic 0.2% yield stress calculation could not be made because of a software limitation. Yield stress values were calculated in Excel subsequent to testing.

4.3.3 Fracture Analysis

Using a Nikon C-BD115 stereomicroscope, the fracture surfaces were examined and qualitatively evaluated. If the surface indicated that fracture was due to inclusions, porosity, or other defects then the data in question was not included. If inclusions and pores were present but were small in size and did not appear to play a major role in the failure of the bar, data were retained. This was done to decouple the effect of macro-defects such as pores and oxide inclusions on the strength of test bars from the effects of heat treatment on the intended

\(^3\) Instron model 2630-100. Instron Corporation, 100 Roall St. Canton MA 02021, (800)-473-7838 www.instron.com
microstructural phase(s). It was expected that some test bars would fracture prematurely due to the presence of macro-defects.

4.3.4 Quality Index

Also presented are Quality Index (QI) values. QI relates the UTS and ductility into a single term, and allows for comparisons when there are variations (e.g. alloy composition, heat treatment).

The Quality Index (QI) helps to gauge the relative improvement brought about by a heat treatment. Additional information on the history of this parameter is available in Section 2.7. Below is the equation for the Quality Index used in this research [102, 103].

\[ Q \, (\text{MPa}) = \text{UTS (MPa)} + 150 \times \log(\% \, \text{elongation}) \] \hspace{1cm} (4.1)

4.4 Microstructure Sample Preparation and Analysis

4.4.1 Polishing and Etching

Samples for microscopic analysis were cut from the grip sections of tensile bars. These samples were successively ground with finer grits of silicon carbide paper (320/ 600/ 1200/ 4000 grit) on a water-cooled and lubricated wheel rotating at 240 rpm. Following grinding, the sample was carefully cleaned with a wet cotton ball before being polished with 0.3 micron alumina and ultrasonically cleaned in
acetone for at least five minutes. For final polishing, an electropolisher was used to polish and etch the sample simultaneously. The electrolyte contains 60% ethanol, 20% perchloric acid, and 20% ethyl glycol monobutyl ether. Samples were electropolished for 15-30 seconds at two Amps current and subsequently rinsed in running tap water. Care was taken to prevent fire when using the electropolisher. A final acetone ultrasonic cleaning ensured that there is no debris or dust on the sample when it was placed into the scanning electron microscope (SEM).

Samples for optical microscopy analysis were not electropolished. Instead, the final polishing medium was colloidal silica. The colloidal silica was applied to a clean Struers MD Chem pad (200 mm wide, porous neoprene, reserved for use of only colloidal silica) and polished by hand. Either electropolishing fluid or Keller’s Reagent etch were used to etch the samples. Keller’s Reagent was only used for the polished sectioned fracture surfaces.

4.4.2 SEM

SEM analyses were performed using a JEOL JSM-840 scanning electron microscope with an energy dispersive x-ray (EDX) unit attached.

While investigations of a sample included a thorough examination, there were certain things which were of particular interest. In addition to low-magnification
pictures which showed the morphology of the primary $\alpha$-aluminum grains, pictures were taken which showed the eutectic silicon morphology, entrapped eutectic (if any), and any phases which had nucleated within the primary $\alpha$-aluminum. These age hardening species were only visible under the highest magnification, and confirmation of their chemistry proved impossible due to their small size and low volume fraction. Changes due to heat treatment were noted.

Other techniques were line scanning and chemistry x-ray mapping. With line scanning, the EDX software measured the composition of the sample in a series of points along a line. It then could qualitatively indicate the variation in alloy chemistry. Any oxides present were also clearly visible, however the oxides in these samples were few in number and small in size. More oxides were found along fracture surfaces because of their weakening effect on the tensile properties, but when a random plane was polished and etched, it was unlikely one of the few oxides would be found. Similarly, through a rastered scan procedure, two dimensional maps of sample chemistry could be generated, although resolution was limited by the area excited by the electron beam.

**4.4.3 Optical Microscopy**

In this work, the main application of optical microscopy was to evaluate transverse sections of fracture surfaces. While optical samples were prepared to examine the low-magnification features discussed in Section 4.4.2 above, there
were no features visible in optical which were not apparent in the lowest magnification SEM images. In fact, the SEM was able to go to lower magnifications than the optical microscopes used as well as much higher ones.

Fracture samples were sectioned with the slow speed water-cooled saw so as to deform the material as little as possible. Other aspects of polishing are discussed above in Section 4.4.1. Samples were placed on the microscope and digital images were obtained.

4.4.4 Stereo Optical Fracture Analysis

Once it was discovered that some fraction of the samples contained macroscopic defects which were producing dramatic changes in tensile properties, it became crucial to examine fracture surfaces with the stereo microscope. Although almost all defects which were eliminated were ultimately visible to the naked eye, the small cross sectional area of the fracture surfaces made guaranteed detection an issue. Lighting under the stereomicroscope is also more consistent than that present during naked-eye observations.

Fracture surfaces were cut from tensile bars by band saw so that they could be viewed under the microscope. They were temporarily mounted in Silly Putty™ and illuminated with the lighting ring. Investigation started at the lowest magnification and was increased as needed to view specific features. Generally,
the presence or absence of each defect was visible quite rapidly once experience had been obtained with regard to what fracture surfaces should look like. Cutting and quick mounting of the samples took much more time than the actual inspection. Judgment was needed when the size of the defects was small and it was uncertain whether the small defects viewed had played a significant enough role to warrant exclusion from the data set. It was also important to consider the cumulative impact of multiple smaller defects.
5.0 RESULTS & DISCUSSION

Presented below are the results of this thesis accompanied with the discussion relevant to each portion of the research. In general, results are presented in the same order as the preceding discussion of experimental procedures. Initial modeling calculations of a thermodynamic nature were made with the Pandat software, which is followed closely by the chemical data that were used to make the calculations. The bulk of this section is made up of the tensile results for the various samples and heat treatments. The section closes with microstructural results at a variety of size scales.

5.1 Thermodynamic Software Analysis

![Phases of interest at equilibrium](image)

Figure 5.1.1: Relative fractions of phases at equilibrium in THT material

In Figure 5.1.1, certain phases were excluded for purposes of clarity. Obviously, the largest fraction of the sample is composed of aluminum and silicon phases.
Strontium and titanium containing phases were also excluded, as they do not play a major role during aging treatments. Clearly, these data indicate there is little variation in the phases which are nucleating at these temperatures. Instead, differences must arise from heating rate due to non-equilibrium factors, such as diffusion rate at a given temperature. It is also possible there are differences in the non-equilibrium phases that form (the $\beta'' \rightarrow \beta' \rightarrow \beta$ sequence).

As mentioned previously, the Pandat software and spark test data also allowed the determination of 562 °C as the highest theoretically safe temperature for this alloy composition for solutionizing.

### 5.2 Chemical Analysis

As previously discussed, since the samples were produced by three different manufacturers, compositional variation was expected. Magnesium contributes to the strengthening behavior of the alloy. The presence of titanium and strontium indicates that the alloy eutectics have been modified and their primary aluminum grains refined. In A356 alloys it is $\text{Mg}_5\text{Si}$ which contributes to the strengthening via precipitate hardening. While standard deviation is not displayed and varied from element to element and from sample to sample, it was calculated to be around ten percent. Within this range, all alloys were within specifications.
Table 5.1: Chemical composition of SSM A356 for tensile sections

<table>
<thead>
<tr>
<th>Material</th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Ti</th>
<th>Sr</th>
<th>Al</th>
</tr>
</thead>
<tbody>
<tr>
<td>SAG</td>
<td>7.9</td>
<td>0.15</td>
<td>0.01</td>
<td>0.01</td>
<td>0.38</td>
<td>0.11</td>
<td>0.03</td>
<td>91.4</td>
</tr>
<tr>
<td>THT (1&quot;)</td>
<td>7.0</td>
<td>0.09</td>
<td>0.05</td>
<td>0.01</td>
<td>0.41</td>
<td>0.15</td>
<td>0.01</td>
<td>92.2</td>
</tr>
<tr>
<td>THT (3/4&quot;)</td>
<td>8.4</td>
<td>0.12</td>
<td>0.03</td>
<td>0.01</td>
<td>0.42</td>
<td>0.09</td>
<td>0.004</td>
<td>91.0</td>
</tr>
</tbody>
</table>

5.3 Tensile Results

5.3.1 THT (rheocast) 1 inch samples

Below are the tensile testing results (YS, UTS, elongation, and Q) for rheocast 1” THT samples. These were heat treated in the T5 or T6 temper for varied times and temperatures in either the fluidized bed reactor or in conventional resistance-heated furnaces. As-cast (F) results are also presented for comparative purposes. Data are organized according to the varied temperatures and times. Details of the exact procedures are available in Tables 4.1 to 4.3. Discussion follows the graphs. Ten bars were tested for each data point, but bars with macro-defects were excluded through fracture analysis using stereomicroscopy.
Figure 5.3.1: Elongation values of 1” rheocast samples

In Figure 5.3.1, the bars heated at 140 °C had the highest elongation of the bars heated in the T5, but in reviewing the strength properties of these bars, one finds that heating at 140 °C is not advantageous. All bars heated in the T5 showed a reduction in elongation. T6 results showed a significant increase in elongation as a result of the spherodized, strontium modified, eutectic structure. T5 fluidized bed treatment produced elongation results comparable to the conventional furnace elongation results. Since there was not a dramatic decrease in elongation in the T6 results heated at 555 °C, there has not been melting along grain boundaries. The same behavior was seen in the thixocast results. In dendritic castings, the elongation values typically drop markedly after T5 heat treatments. This is in contrast with what was seen for both the rheocast data.
above and thixocast elongation data in Figure 5.3.5, where the drop-off was much more gradual. T6 fluid bed heating reduced elongation while improving strength. Microstructural analysis was consistent with these results.

![THT treatments chart](image)

Figure 5.3.2: Yield strength values of 1” rheocast samples

Figure 5.3.2 shows the yield stress results for the rheocast material. As aging time and temperature are increased, strength values continue to improve. At 180 °C, strength values are reached which rival the T6 samples. T5 treatments show a consistent yield strength improvement as aging time and temperature is increased. T6 samples do not show especially high yield stress values. Since aging time was not a variable, this is perhaps to be expected, but it is interesting
to note that the yield strength with respect to aging temperature and time remains approximately constant within the range of experimental uncertainty. Both results in the T5 and the T6 conditions using the fluidized bed reactor show a large improvement in yield strength at the expense of some ductility.

![THT treatments](image)

**Figure 5.3.3:** Ultimate tensile strength values of 1” rheocast samples

Figure 5.3.3 shows that aging at 180 °C leads to continued improvement in strength. Aging at 160 °C takes twice as long to reach these levels of strength. Similar strength values are seen for the fluidized bed T5 results. Aging time and temperature was not a variable for the T6 samples, and all samples show comparable strength values. T6 components visibly exceed the strength of all T5
bars, although the highest T5 and lowest T6 values are close. The UTS of the fluidized bed T6 components is quite good, at the expense of some ductility. Figure 5.3.7 shows slightly different results for the thixocast components. There is slightly more magnesium in the THT samples, which may account for this behavior.

Figure 5.3.4: Quality Index values of 1” rheocast samples

The Quality Index results, shown in Figure 5.3.4, gauge the net effect on the alloy through heat treatments. When SSM material is successfully heat treated to retain the high as-cast ductility, Quality Index is not conserved, but instead increases above the as-cast value. Here, the T5 results only exceed the as-cast
value at 180 °C for four hours, but do so for both the SAG and THT 1” material. Quality Index results are closer to the as-cast value for the remaining T5 treatments than the thixocast T5 results. All T6 results show a net improvement of the Quality Index, as was expected. In T6 treatments using strontium as a eutectic modifier, the spherodization of silicon allows for an increase in both ductility and strength. Also of interest are the Quality Index results for the fluidized bed heat treatments that show comparable QIs to the conventional treatments, confirming that fluid bed T6’s low elongation was exchanged for improved strength.

5.3.2 SAG (thixocast) samples

Below are the tensile testing results (YS, UTS, elongation, and Q) for thixocast SAG samples. These were heat treated in the T5 or T6 temper for varied times and temperatures in either the fluidized bed reactor or in conventional resistance-heated furnaces. As-cast (F) results are also presented for comparative purposes. Data are organized according to the varied temperatures and times. Details of the exact procedures are available in Tables 4.1 to 4.3. Discussion follows the graphs. Ten bars were tested for each data point, but bars with macro-defects were excluded through fracture analysis using stereomicroscopy.
In the elongation graphs, the SAG results in Figure 5.3.5 show that amongst the T5 results, the bars heated at 140 °C have the highest elongation, but in reviewing the strength properties of these bars, it is seen that heating at 140 °C is not advantageous. T6 results show a significant increase in elongation as a result of the spherodized, strontium modified eutectic structure. T5 fluidized bed treatment produced elongation results comparable to the conventional furnace elongation results. T6 fluid bed samples traded some elongation for higher strength. Since there is not a dramatic decrease in elongation in the T6 results heated at 555 °C, there has not been melting along grain boundaries. The same
behavior is seen in Figure 5.3.1 in the THT elongation T5 results. Microstructural analysis is consistent with these results.

![SAG treatments](image)

**Figure 5.3.6: Yield strength values of thixocast samples**

Figure 5.3.6 shows the yield stress results for the thixocast material. It should be noted that the uncertainty of the yield strength of the T5 thixocast data is higher than other data presented (on the order of 30 Mpa as opposed to 10 Mpa). Still, certain trends are evident. Significant improvement is only seen in the T5 temper at the higher end of the temperature/time range. T6 treatments, which had a low degree of variation, show a consistent yield strength improvement. Since aging time was not a variable, this is to be expected. Both results in the T5 and the T6 conditions using the fluidized bed reactor show a large improvement in
yield strength. These yield stress values, particularly the T6 values, are quite low for this alloy in this temper. The T6 fluid bed results, however, are an exception as they show good results.

![SAG treatments graph](image)

Figure 5.3.7: Ultimate tensile strength values of thixocast samples

Figure 5.3.7 shows that aging at 180 °C quickly reached the highest UTS values. Aging at 160 °C took twice as long to reach these levels of strength. Similar strength values were seen for the T6 results and fluidized bed T5 results. The UTS of the fluidized bed T6 components were quite good, at the expense of some ductility. Figure 5.3.3 showed slightly different results for the rheocast components. The fluidized bed T6 performance was not dramatically better than the conventional T6 bars, and continued improvement in strength was seen at
increased time at 180 °C. There was slightly more magnesium in the THT samples, which may account for this behavior. There was also additional improvement in strength in the T6 conventional furnace results as compared with the T5 conventional furnace results. Since the SAG part was smaller, it is believed to have cooled in the die more quickly than the THT component. As a result, there was variation in the influence of solutionizing providing additional magnesium to harden the THT T6 components.

![SAG treatments](image)

**Figure 5.3.8:** Quality Index values of thixocast samples

The Quality Index results in Figure 5.3.8 show the impact of heat treatments. When SSM material is successfully heat treated to retain the high as-cast ductility, Quality Index is not conserved but instead increases above the as-cast
value. Here, the T5 results only exceed the as-cast value at 180 °C for four hours, but do so for both the SAG and THT 1” material. Quality Index results for the THT heat treatments are quite flat when compared to the rheocast results in the T5. All T6 results show a net improvement of the Quality Index, as is expected, but this improvement is not substantial. In T6 treatments using strontium as a eutectic modifier, the spherodization of silicon allows for an increase in both ductility and strength.

**5.3.3 THT (rheocast) ¾” samples**

Below are the tensile testing results (YS, UTS, elongation, and QI) for rheocast ¾” THT samples. These were heat treated in the T5 or T6 temper for varied times and temperatures in either the fluidized bed reactor or in conventional resistance-heated furnaces. As-cast (F) results are also presented for comparison. Data are organized according to the varied temperatures and times. Details of the exact procedures are available in Tables 4.1 to 4.3. Discussion follows the graphs. Six bars were tested for each data point, but bars with macro-defects were excluded through fracture analysis using stereomicroscopy.

This set of samples was fabricated with a number of specific questions in mind. Following a general discussion of each graph is an analysis of these specific questions raised in Sections 5.3.1 and 5.3.2, as well as the procedures which produced those data. It should be remembered that many procedures in this set
of experiments were designed to learn what to avoid, and so the results for those data are expected to be lower.

![New THT treatments](image)

Figure 5.3.9: Elongation values of 3/4” rheocast samples

The elongation results shown in Figure 5.3.9 allow for a number of immediate conclusions. When comparing the two sets of F temper results (those for bars quenched and unquenched from the die), the quenched bars clearly have superior elongation properties. This result continues through T5 testing as quenched and unquenched samples with identical aging regimes are compared, although the Quality Index is a better tool to examine this facet of the experiments as it considers both the improvement to strength as well as the level of ductility. Another interesting result is the impact of post-aging quenching on
elongation after T5 treatments. Although there was concern that using this step in the earlier set of trials may have negatively impacted properties, post-aging quenching of T5 samples improved the elongation of both post-die quenched and non-post-die quenched samples (aged at 170 °C for six hours). Although some of the sub-optimal T6 treatments have elongation values comparable to the best T5 results, it is clear that the best T6 results have superior ductility to the T5. The very high values of ductility seen in fluid bed components are noteworthy, but it will be seen that these components have not been strengthened sufficiently.

Figure 5.3.10: Yield strength values of 3/4” rheocast samples
As one would expect, Figure 5.3.10 shows that yield strength increased more for the QT5 and T6 conditions than the simple T5 case, which in turn is somewhat higher than the as-cast yield strength. QT5 had more magnesium in solution than T5 treatments, but less than T6 treatments. The highest values of yield strength are those for the T6 treatments. These fluidized bed treatments at aging temperatures have not displayed the same improvements in strength that were seen in equal time for the higher aging time investigated in the earlier tests. Since many industrial applications call for a minimum of 207 MPa (30 ksi) yield strength, the treatments which fail to meet this criterion would not be acceptable if identical values were found in high stress regions of industrial components. All of the best QT5 conventional furnace components meet this value, and the T6 components exceed it.
Figure 5.3.11: Ultimate tensile strength values of 3/4” rheocast samples

The ultimate tensile strength values in Figure 5.3.11 follow the same general trends as yield strength, except that there is greater uniformity of ultimate tensile strength values than there was for the yield strength values in Figure 5.3.10. This uniformity suggests that most of the samples were strengthened as much as their respective quantity of magnesium in solution allowed. The results of some of the procedural experiments are evident in these data. When natural aging time was increased to 15 hours, a decrease in ultimate tensile strength became apparent. Although there is some improvement in the ultimate tensile strength of samples that were solution treated for eight hours instead of four or five, there is a corresponding drop in ductility (presumably from grain growth).
Although they experience a decrease in ductility, unquenched bars do not show a marked decrease in UTS except in the as-cast state.

![New THT treatments](image)

Figure 5.3.12: Quality Index values of 3/4” rheocast samples

In Figure 5.3.12, the first result which is apparent is the conservation of quality index in the T5. In both conventional furnace and fluidized bed treatment, the quality index values of the T5 bars do not exceed their F-temper counterparts within the limits of experimental error. The quality index of T6 bars, on the other hand, is not conserved (T6 QI values exceed F and T5 values for QI). Thanks to the spheroidization of silicon, there is a net improvement in QI. It is also clear that the die quenched bars have superior properties as compared to the bars which were air cooled from the die.
Quenching bars between solutionizing and aging shows the same improvements discussed above under elongation. Solutionizing for eight hours instead of four has no impact on Quality Index, but differences in UTS and ductility were noted above. As these above changes are relatively small, it can still be stated that eight hours of solutionizing is probably not necessary for samples of this size. Increasing natural aging from eight hours to 15 hours after the quench before artificial aging decreased the QI slightly, although aging temperature and time are a much larger factor.

T6 aging for eight hours at 160 °C produces results very similar to aging at 180 °C for four hours. T6 aging at 160 °C for five hours results in a lower QI. Fluidized bed T5 results are comparable in QI to the non-fluidized bed T5 results. T6 fluid bed results are not as good as the most promising T6 CF results, suggesting an increased aging temperatures such as those used in the earlier trials would improve QI results. Alternately, an increased aging time could be employed at these lower temperatures.

5.4 Results of Microstructure Analysis

5.4.1 Scanning Electron Microscopy and Analysis

Below are a number of SEM micrographs which were taken using the procedure described in Section 4.4.1. Discussion follows the micrographs.
Figure 5.4.1: Micrographs of SSM cast A356 alloy prepared through (a) rheocast, and (b) thixocast methods. Note the entrapped eutectic in b. Both samples are in the as-cast condition.

Figures 5.1 a, and b show micrographs of SSM cast A356 alloy prepared through the rheocast and thixocast routes. The volume fraction of eutectic phases in thixocast alloy is greater than those in rheocast alloy. This is because the silicon content in the rheocast A356 alloy (7.0%) is lower than the thixocast alloy (7.9%). At this magnification, there is little, if any, difference between the heat treated and as-cast structures. Entrapped eutectic is visible in the thixocast sample, but the dark features inside the primary α-aluminum grains in the rheocast sample are not entrapped eutectic. These zones are almost chemically identical to the neighboring aluminum. They are a result of the electropolishing
procedure used on these samples, and a different procedure eliminated the
discoloration.

Figure 5.4.2: Rheocast sample (F)  Figure 5.4.3: Thixocast sample after
aging for 4 hrs at 180 °C

Figure 5.4.2 shows a primary $\alpha$-aluminum grain and the neighboring eutectic
that allowed the image to be focused. The eutectic silicon particles have acicular
or flaky morphology at the periphery of the eutectic region and fibrous at the
center of the eutectic region (Figure 5.4.4 and 5.4.5). Figure 5.4.3 shows a
portion of primary $\alpha$-aluminum. While the phase present in Figure 5.4.3 is too
small to be conclusively chemically analyzed with EDX, it is morphologically
similar to $\text{Mg}_2\text{Si}$. The absence of such structures from Figure 5.4.2 and similar
images of samples which have received less heat treatment than Figure 5.4.3
suggests that four hours of aging at 180 °C is necessary to get noticeable
precipitation. The phase visible in 5.4.3 is the result of heat treatment.
In Figures 5.4.4 and 5.4.5, no coarsening of eutectic silicon is seen. The larger silicon particle in the center of Figure 5.4.5 and the similar structure at the lower right corner of the untreated sample are flakes formed during solidification. A limited eutectic growth was observed for both thixo cast and rheocast samples, and this is probably the reason that T5 does not significantly reduce the elongation of the alloys [3].
**Figure 5.4.6:** Micrograph of A356 SSM in the T6 condition. Solutionized at 540 °C for eight hours and aged at 160 °C for five hours.

**Figure 5.4.7:** Micrograph of A356 SSM in the as-cast (F) condition.
**Figure 5.4.8:** Micrograph of A356 SSM in the artificial aged (T5) condition. Aged at 160 °C for eight hours.

**Figure 5.4.9:** Micrograph of A356 SSM in the T6 condition. Solutionized at 555 °C for two hours and aged at 160 °C for five hours.
In the SEM images presented in Figures 5.4.6 through 5.4.9, the α-aluminum and silicon are identified. At this magnification, it is possible to see slight differences in Figure 5.4.6 in the eutectic region as compared with Figure 5.4.1, which is in the F rather than T6 condition. (T5 and F eutectics look quite similar at this magnification.) When Mg$_2$Si is present in the eutectic, it appears as a grey phase with a similar morphology to the silicon, however it is not necessarily the case that a particular gray phase is Mg$_2$Si instead of silicon. Micron bars in the figures indicate the respective magnifications. In addition, a iron rich phase was also observed. EDX analysis shows that it is a π phase (AlFeSiMg).

Since the visible change was taking place in the eutectic, while the primary phases are not visibly changed during heat treatment, one low-magnification image is accompanied by a higher magnification picture for each heat treatment condition (F, T5, T6). The as-cast (F) SSM components display a fine, fibrous network of eutectic silicon due to the effects of strontium additions and perhaps also to the unique solidification conditions encountered during semi solid processing. This same microstructure is retained in the T5 condition, as heat treatment temperatures were not high enough to modify the silicon. However, after T6 heat treatments, when much higher solutionizing temperature conditions were applied, the silicon network has transformed into a series of small, discrete spheroids of silicon. These observations are not unique to SSM A356
components. Indeed, all hypoeutectic aluminum silicon alloys which have been modified with strontium show these kinds of structures before and after T6 treatment regardless of whether the casting process produces dendritic or SSM primary aluminum. Normally, the only way to improve yield and ultimate strength and maintain a high ductility via heat treatment is by performing a T6 heat treatment. The spheroidization of the silicon is a mechanism known to improve ductility while precipitation hardening decreases it, at least in dendritic materials. Consequently, one mechanism decreases ductility as another increases it. When heat treatment times are chosen carefully, the net result is a cast part with a high yield stress and ductility.

**5.4.3 Optical Fracture Surface Images**

Figure 5.4.10 a and b: a) medium magnification normal fracture surface b) fracture surface of sample bearing macroscopic defect. 1 mm along bottom.

In Figure 5.4.10 a and b, the relative difference in fraction solid between the ‘good’ (a) and ‘bad’ (b) fracture surfaces can be seen. _The merits of these
fracture surfaces were judged based upon oxide content and tensile properties.) Although microstructures such as these help to reveal the underlying causes of poor properties in defective bars, the chief interest of this work is the improvement of bars which have been produced so as to avoid macroscopic defects. Given this fact, and the time required for the preparation of individual micrographic samples, stereomicrographic analysis was preferred for defect analysis. The need for low magnification analysis of the entire plane of the fracture surface rather than just a slice through it, was also a significant factor.

The stereomicroscopy procedure is detailed in the Procedures section. During stereomicrographic analysis, the goal was to find and exclude the samples which contained macroscopic defects. Consequently, bars were classified as either ‘defect free’, ‘contains negligible defects’, or ‘flawed.’ Bars which were classified as ‘contains negligible defects’ generally had one or more very small defects or a somewhat larger defect which did not appear to have played a role in the failure of the part. Below are some examples of photographs taken with the camera attached to the stereomicroscope. It should be noted that actual depth of field in a photograph is necessarily not as good as binocular vision through the microscope.
Figure 5.4.11: Stereomicrograph of both halves of a fracture surface with ellipsoid defect.

Figure 5.4.11 shows an example of an otherwise normal fracture surface with a hemispherical ball shaped defect which apparently solidified early or in the presence of an oxide film which led to a weak spot in the sample. This bar, and any others with comparable or worse macroscopic defects, were excluded from the data sets. If the sample had had a much smaller defect or if it had been defect free, the rest of the fracture surface was acceptable, and so the data would have been included. The remainder of the area of this fracture surface serves as an example of a defect-free surface.
The defect in Figure 5.4.12 is an example of one of the worst defects encountered in the experimental work. Here, a large oxide formed where two masses of metal should have welded together as a part of one continuous casting. Instead, the oxide skin led to failure at a low load. The fracture surface near the top approaches normalcy.

5.5 Blistering and surface finish

All heat treatments result in slight changes to surface coloration. SSM processed material is no different in this regard. Elevated temperatures can result in increased oxidation, and the changes in the oxide layer give rise to this altered appearance. If die lubricants are present on the bar at the time of heat treatment, they can discolor (burn) during heat treatment. The sand in the fluid bed generally removes this material as heat treatment progresses.
By far the most serious impact of heat treatment is the possibility of blistering. While not necessarily a serious mechanical defect, parts with blisters are aesthetically displeasing and unacceptable to commercial customers. Consequently, parts which develop blisters upon heat treatment are rejected. No bars which were subjected to T5 heat treatment in either the fluidized bed or the conventional furnaces developed blisters.

Under the T6 temperatures, some blistering did occur in all samples. Of the 1” rheocast samples, only those with other macroscopic defects blistered significantly and those blisters were uncommon as well. Blistering in the 3/4” rheocast samples was also uncommon, but was slightly more common than in the thicker stock. The thixocast samples were more prone to blistering than either batch of rheocast samples. These three sample sets had chemical differences and may well have been lubricated differently in their respective dies. It is known that die lubrication plays an important role in the development of blisters, so the relative occurrences of blisters in these processes is not necessarily indicative of the strength or weakness of the processes themselves in this regard. It was clear, however, that the higher solutionizing temperatures and longer treatments were more likely to produce blistering. Although fluidized bed treatment accelerates the rate of solutionizing, it also seems to increase blistering in rough proportion to that same rate increase. Through careful
tailoring of the solution temperature and time for a specific part, it may be possible to reduce or eliminate blistering, although that work is outside the scope of this project. Reducing the temperature with an increased time would be another approach to solve that same problem.
6.0 CONCLUSIONS

Having conducted numerous experiments, there are a series of conclusions which can be drawn with regard to both the heat treatment procedures employed in these experiments as well as indications of what these experiments suggest the ‘optimum’ treatments are.

A second battery of tests was conducted to investigate certain procedural steps employed in the first set of trials, and it is necessary to understand the conclusions of that experiment before the overall conclusions can be examined. The procedural conclusions are as follows:

• Post-age quenching did not significantly or negatively effect the results of the first trials of the thixocast and rheocast materials.

• Conventional furnace solutionizing times were sufficient in the first battery of trials, although aging temperatures and times may not have been optimal in the T6.

• Proper quenching procedure is important during T6 treatments, and should not be neglected.

• Increased natural aging time beyond eight hours had a negative effect on tensile properties, although the variation between eight and 15 hours was small.
With these details of procedure understood, optimization conclusions can be reached. They are as follows:

- In the T5 condition in conventional furnaces, aging at an indicated furnace temperature of 180 °C, corresponding to a part temperature of 170 °C, consistently yielded the best results in the least time.

- Given the goals of fluid bed heat treatment, a minimal time in the furnace is key.
  - It is the highest aging temperature treatments which produce the best results in a given amount of time.
  - The highest aging temperature used in these treatments was 204 °C.
  - T6 fluid bed trials using that temperature were also the most successful in that they resulted in the highest quality index in the least amount of time.

- Solutionizing above 540 °C but below 560 °C is recommended if this can be done without blistering or additional distortion in an industrial setting.

- A reduction in solutionizing times is possible in a furnace with uniform temperatures and precise controls.

- Significant differences in the heat treatment requirements of thixocast and rheocast components were not observed.
• Although many T5 components exhibited favorable combinations of strength and ductility, the Quality Index was conserved in these tests for T5 treatments.

• Although the unusual ductility retention has been observed in other systems, it was not observed in these tests.
6.1 Future Work

As is the case with many theses, there are many directions which could be pursued with this work as a starting point. Given the obvious advantages of fluid bed treatments, it would make sense to conduct additional dedicated research in fluid bed semi solid heat treatment. Key factors to investigate in the fluid bed are proposed sand-quenching technologies and a detailed analysis of the effects of fatigue and distortion under various fluid bed treatments on components of interest. In general, quench analysis may be a valuable tool in the study of the heat treatment behavior of this component.

There have been suggestions from industrial partners that the unusual T5 effect is in some way magnesium-linked. Detailed examination of properties with varied magnesium content (0.2-1.0% Mg) would be a way to investigate this phenomenon. There are a variety of different rheocasting methods, and it remains to be proven conclusively that all rheocast material heat treats exactly the same. Given variations in segregation during casting, aging and solutionizing times rather than temperatures would seem to be the key parameter.
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Appendix A

Effect of Artificial Aging on Microstructure and Tensile Properties of Semi Solid Processed A356 Castings

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1.0 ABSTRACT

Commercially produced SSM alloys were T5 and T6 heat treated. Their microstructure and resultant properties were evaluated. T5 offers a potential for significant cost reductions over T6 heat treatment due to lower time and energy requirements of the process. In addition to this, other benefits of T5 temper over T6 temper include prevention of blistering and greater dimensional stability. It has been suggested that SSM castings treated to T5 temper retain ductility vis-à-vis as-cast semi solid casting as opposed to the experience when going from as-cast to T-5 in liquid-cast parts, but opinions differ. Scanning electron microscopy has been used to understand the microstructure-property co-relationship of aged SSM A356 alloy. Results of this study are presented and reviewed. In addition, a comparative study is made between conventional liquid-cast versus SSM cast A356 alloy.
2.0 INTRODUCTION

As with all new processes, after the mechanisms for SSM heat treatment were commercialized, the first heat treatments applied to it were essentially those already in use for dendritic materials. As time passed, it was discovered that certain heat treatments in SSM produced desirable combinations of material properties. In the silver anniversary lecture at the 106th AFS casting congress at Chicago, John Jorstad pointed out the promise of SSM in the T5 heat treated condition to meet or exceed design specifications while reducing energy costs [1]. In particular, he discussed how quenched and then T5 aged SSM components retained their high as-cast ductility while improving yield strength and ultimate tensile strength to values just below the same material in the T6 condition. The mechanisms of this retention of ductility are still poorly understood. There are still questions regarding the generality of this behavior.

An examination of case studies of SSM parts from the 8th Biannual S2P conference shows a majority of the castings in the 356/357 family of Al Si Mg alloys are heat treated to the T5 temper [2]. Chief reasons for selecting SSM processing for these parts was the high toughness/ductility/strength and low porosity of SSM components. A357 is found most frequently for parts requiring the highest strength, as the additional magnesium increases precipitate hardenability.
It is hoped that through an understanding of the fundamental mechanisms, SSM processing can be optimized to provide key material properties with the least expenditures of time, energy, and money. This paper presents intriguing data which underscores the assertion that SSM heat treatment has the potential to save time, energy and money.

There are two main routes for producing semisolid aluminum parts. These are thixocasting, and rheocasting. Thixocasting involves a specially-prepared billet of solid material that is subsequently heated into the semisolid range. Due to the preparation of the billet, the final microstructure displays a series of spheroidal or rosette aluminum phases surrounded by a eutectic [3]. The other route involves taking liquid and cooling it into the semisolid range, the rheocasting route. Measures are taken to provide copious nucleation and to retard grain growth. Once the part is cooled, the microstructure exhibits similar spheroidal structures to those found with thixocast materials. Two chief benefits of SSM processing include the presence of a stable flow front which helps prevent the entrapment of air in parts and a reduced processing temperature which helps to extend die life significantly. In this work, rheocast components were produced by THT Presses (Dayton OH, USA) using the sub liquidus casting (SLC) process.
In this paper, we discuss heat treatment characteristics of SSM 319 and A356 alloys prepared through the rheocasting route. Tensile properties of both T5 and T6 tempered alloy are evaluated, along with a detailed metallographic examination of the as-cast and heat treated microstructures.
3.0 EXPERIMENTAL PROCEDURE

Materials properties and microstructures are only relevant in the context of the chemical makeup, processing history, and bulk geometry. Rheocast samples were supplied by THT from their SLC process. In some tests (indicated by “QT5”) THT material was quenched from the die to retain as much of the elements in solution prior to precipitate hardening; however, the time required for manual part removal from the die, and thus the part temperature at the time of quenching, may have not have been optimized. Data from previous WPI research on the heat treatment of dendritic A356 alloy material will be compared to the data presented here.

Tensile specimens and testing conformed to ASTM specifications. Samples were heat treated prior to machining. An extensometer was used to measure bar extension with increased precision.
**Table 3.1:** Heat treatment region for SSM tensile specimens

<table>
<thead>
<tr>
<th>Region</th>
<th>SHT ºC</th>
<th>Age ºC/hrs</th>
<th>Bar size</th>
</tr>
</thead>
<tbody>
<tr>
<td>P A356 F</td>
<td></td>
<td></td>
<td>0.375&quot; round</td>
</tr>
<tr>
<td>P A356 T-5</td>
<td>x</td>
<td>170/4</td>
<td></td>
</tr>
<tr>
<td>P A356 QT-5</td>
<td>x</td>
<td>170/4</td>
<td></td>
</tr>
<tr>
<td>PS A356 F</td>
<td></td>
<td></td>
<td>0.25&quot; round</td>
</tr>
<tr>
<td>PS A356 T-5</td>
<td>x</td>
<td>172/4</td>
<td></td>
</tr>
<tr>
<td>PS A356 QT-5</td>
<td>x</td>
<td>172/4</td>
<td></td>
</tr>
<tr>
<td>PS A356 T-6</td>
<td>540</td>
<td>172/4</td>
<td></td>
</tr>
<tr>
<td>Squeeze A356 F</td>
<td></td>
<td></td>
<td>0.25&quot; round</td>
</tr>
<tr>
<td>Squeeze A356 T-5</td>
<td>x</td>
<td>155/4</td>
<td></td>
</tr>
<tr>
<td>Squeeze A356 T-6</td>
<td>530</td>
<td>155/4</td>
<td></td>
</tr>
<tr>
<td>P 319 F</td>
<td></td>
<td></td>
<td>0.125&quot; round</td>
</tr>
<tr>
<td>P 319 T-5</td>
<td>x</td>
<td>170/4</td>
<td></td>
</tr>
<tr>
<td>P 319 QT-5</td>
<td>x</td>
<td>170/4</td>
<td></td>
</tr>
<tr>
<td>S B319 F</td>
<td></td>
<td></td>
<td>0.125&quot; round</td>
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<tr>
<td>S B319 T-5</td>
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<td>170/4</td>
<td></td>
</tr>
<tr>
<td>S B319 QT-5</td>
<td>x</td>
<td>170/4</td>
<td></td>
</tr>
<tr>
<td>S B319 T-6</td>
<td>525</td>
<td>185/4</td>
<td></td>
</tr>
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<td>K A356 F</td>
<td></td>
<td></td>
<td>0.25&quot; round</td>
</tr>
<tr>
<td>K A356 T-5</td>
<td>x</td>
<td>170/5</td>
<td></td>
</tr>
<tr>
<td>K A356 T-6</td>
<td>532</td>
<td>170/5</td>
<td></td>
</tr>
</tbody>
</table>

Table 3.1, above, shows the heat treatment conditions of the tensile samples along with their respective bar sizes. “P” 319 refers to primary 319, that is, high purity 319 without Mg. “S” B319 is a secondary alloy which typically contains 0.3% Mg. “K” A356 refers to a primary alloy A356 component which was formed into the same steering knuckle geometry as the dendritic squeeze cast A356 component. Five parts were cut into 3 bars each, for a total of 15 tensile bars tested for these 319 “S” and “P” alloys in their various heat treatment conditions. The “K” data was produced from six bars. Both “P” and “SP” A356 refer to
primary alloys, but their compositions differ slightly. Six bars were produced and tested for each of these A356 heat treatment conditions.

QT-5 refers to a T5 heat treatment of a part which was quenched from the die. The more familiar “F” refers to the as cast condition, T-6 to a solutionizing heat treatment followed by quenching and artificial aging, and T-5 to artificial aging without prior solution treatment or quenching.

Since an off-site manufacturer provided samples, there were several months of natural aging time between casting and heat treatment for the micrographic specimens. Induction heated box furnaces were used for the micrographic samples. Blistering was not observed in any of the samples, tensile or micrographic.

SEM samples for microscopic analysis were cut from the grip sections of tensile bars. These samples were successively ground with finer grits of paper (240/320/600/4000 grit) on a water cooled wheel rotating at 240 rpm. Following grinding, the sample was carefully cleaned with wet cotton before being polished with 0.3 micron alumina and ultrasonically cleaned in acetone for at least five minutes. For final polishing, an electropolisher was used to polish and etch the sample simultaneously. The electrolyte contains 60% ethanol, 20% perchloric acid, and 20% ethyl glycol monobutyl ether. Samples are electropolished for 15-
30 seconds and then subsequently rinsed in running tap water. A final acetone ultrasonic cleaning insures that there is no debris or dust on the sample when it is placed into the SEM. SEM analyses were performed using a JEOL JSM-840 scanning electron microscope with an EDX unit attached.
**4.0 RESULTS**

The presence of Ti and Sr indicates that all alloys were modified and grain refined. In the 319 alloys, CuAl₂ (or Al₂CuMg in B319) plays the main role in age hardening whereas in 356 alloys it is Mg₂Si which contributes to the strengthening. The “Micro A356” is the composition of the samples which were heat treated prior to being sectioned and polished for micrographic examination in the scanning electron microscope.

<table>
<thead>
<tr>
<th></th>
<th>Si</th>
<th>Fe</th>
<th>Cu</th>
<th>Mn</th>
<th>Mg</th>
<th>Zn</th>
<th>Ti</th>
<th>Sr</th>
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Table 4.2: Tensile properties of SSM and dendritic permanent mold samples

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<th>UTS Mpa</th>
<th>YS Mpa</th>
<th>%El</th>
<th>BHN</th>
<th>QI Mpa</th>
<th>SSM</th>
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<td>160</td>
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</tr>
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</tr>
<tr>
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<td>150</td>
<td>10</td>
<td>65</td>
<td>385</td>
<td>yes</td>
</tr>
<tr>
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<td>139-165</td>
<td>7-13</td>
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<td></td>
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</tr>
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<td>205</td>
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<td>100</td>
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<td>7-9</td>
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<td></td>
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<td></td>
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<tr>
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<td>116</td>
<td>10</td>
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<tr>
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<tr>
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<td>Perm mold A356 T6</td>
<td>258</td>
<td>152</td>
<td>10</td>
<td></td>
<td>408</td>
<td>no</td>
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</table>
The most striking part of these data is the high elongation values through the different alloy systems. In all cases, while the range may vary slightly, the mean elongation retains its high as-cast value throughout heat treatment. In dendritic castings, the elongation values typically drop markedly after T5 heat treatments. The mechanism which allows yield stress to improve while retaining the as-cast ductility is not completely established. In the T6 condition, high elongation is known to result from the spherodization of the eutectic silicon during the solutionizing step, but both the low temperatures of T5 treatment and micrographic investigation indicate that this process is not taking place in either the simple T5 or in the quenched-from-die then T5 (QT5) case. It is worth noting that the temperatures of T5 heat treatment employed herein are higher than those typically used with the dendritic castings.

As one would expect, yield strength increases more for the QT5 and T6 conditions than the simple T5 case, which in turn is somewhat higher than the as cast yield strength. This trend follows the increasing presence of dissolved age hardening constituents in the alloys. QT5 traps more in solution than T5 but less than T6. While T6 treatments of SSM parts do produce the highest yield strength values, the improvement between a QT5 and a T6 heat treatment is small compared to the increased energy and time costs, as noted in the
 introduction. Ultimate tensile strength and hardness increase in the same way and for the same reasons.

The Quality Index (QI) helps us to gauge the relative improvement brought about by a heat treatment. This parameter was developed by Drouzy et al. and applied in the optimization of heat treatments by Shivkumar et al. [4,5]. C.H. Caceres et al. have done further work to show the fundamental basis of the quality index [6].

\[ Q \text{ (MPa)} = \text{UTS (MPa)} + 150 \times \log(\% \text{ elongation}) \] (1)

Given what has already been noted regarding the elongation and strength of these alloys under heat treatment, it is not surprising that Quality Indices improves as strength improves. Since elongation is constant to well within the range of experimental variation, this is to be expected. It is the constancy of elongation itself which is unusual. The relatively small difference between QT5 and T6 heat treatments is again underscored by the small difference in their quality indices. The high as-cast elongation results in a high quality index for the as-cast material itself.

The entries listed as “Perm mold A356” F and T6 are dendritic permanent mold samples which have been included for the purposes of comparison. Quickly
comparing the ultimate tensile strength, yield strength, and elongation of the dendritic T6 to the QT5 SSM results, we see that the SSM QT5 parts have much better properties with much less time and energy spent in heat treatment. By examining the Quality Indexes of the A356 dendritic and SSM heat treatments, it is immediately obvious that the SSM components have superior properties. It is also quite clear that the as-cast SSM properties start off much better than those of the dendritic permanent mold material.

The “Squeeze” A356 entries are similarly dendritic. An important fact to note when considering these data is that the aging temperature is lower than that for the SSM A356 components. Even so, the elongation in the T5 condition has decreased by half as compared to the as cast and T6 conditions. The high ductility in the T6 is attributable to the spherodization of silicon, as previously discussed. While squeeze casting is another high integrity casting technique, it doesn’t show the same high T5 ductility that has been seen in the T5 and QT5 treated SSM components. This may or may not present a clue as to the mechanism whereby the elongation of SSM T5 components is higher, as it clearly cannot be due to something which also occurs in squeeze casting. Again, similar trends are seen in Quality Index values. The decrease in quality index between F and T5 suggests that the heat treatment regime applied isn’t necessarily optimal. In fact, dendritic A356 is normally aged to a T-5 temper at 225 C for purposes of growth stability, not at lower temperatures for strength improvement.
Below are a number of SEM micrographs which were taken using the procedure described earlier. Discussion follows each set of micrographs.

**Figure 4.1:** Micrograph of A356 SSM in the T6 condition. Solutionized at 540°C for 8 hours and aged at 160°C for 5 hours.
Figure 4.2: Micrograph of A356 SSM in the as-cast (F) condition.

Figure 4.3: Micrograph of A356 SSM in the artificial aged (T5) condition. Aged at 160C for 8 hours.
Figure 4.4: Micrograph of A356 SSM in the T5 condition. Solutionized at 555C for 2 hours and aged at 160C for 5 hours.

In the SEM images given in Figures 4.1 through 4.4, the α-aluminum and silicon are identified. When Mg$_2$Si is present in the eutectic, it appears as a grey phase with a similar morphology to the silicon, however it isn’t necessarily the case that a particular grey phase is Mg$_2$Si instead of silicon. Micron bars in the figures indicate the respective magnifications. In addition, some Fe rich phase was also observed. EDX analysis shows that it’s a π phase (AlFeSiMg).

Since the visible changes are taking place in the eutectic whereas the primary phases are not visibly changed during heat treatment, one low magnification image is accompanied by a higher magnification picture for each heat treatment condition (F, T5, T6).
The as-cast (F) SSM components display a fine, fibrous network of eutectic silicon due to the effects of Sr additions and perhaps also to the unique solidification conditions encountered during semi solid processing. This same microstructure is retained in the T5 condition, as heat treatment temperatures were not high enough to modify the silicon. However, after T6 heat treatments, when much higher solutionizing temperature conditions were applied, the silicon network has transformed into a series of small discrete spheroids of silicon. These observations are not unique to SSM A356 components. Indeed, all hypoeutectic aluminum silicon alloys which have been modified with strontium show these kinds of structures before and after T6 treatment regardless of whether the casting process produces dendritic or SSM primary aluminum.

What is different here, however, is the retention of the high as-cast ductility in the T5 condition. Normally, the only way to improve yield and ultimate strength and maintain a high ductility via heat treatment is by performing a T6 heat treatment. The spheroidization of the Si is a mechanism known to improve ductility while precipitation hardening decreases it (at least in dendritic materials). Consequently, one mechanism decreases ductility as another increases it. When heat treatment times are chosen carefully, the net result is a cast part with a high yield stress and ductility without the cost of a lengthy solutionization treatment. Semi solid components, on the other hand, do not
show a decrease in their ductility as yield stress is increased. In other words, quality index is not conserved in SSM components which have been T5 heat treated.
5.0 DISCUSSION

As detailed in the introduction, it has been suggested that SSM components subjected to T5 conditions retain their high as-cast ductility [1]. Many commercial manufacturers who produce SSM components have found that they can obtain the desired material properties for their parts with only a T5 heat treatment [2]. Here, data are presented which support these claims with tensile and hardness results.

As can be seen from the tensile data, these phenomena are present for multiple alloy systems (A356 and 319). It occurs in both primary and secondary 319 alloy, with and without the presence of Mg and Zn. T5 properties are improved by quenching components from the die prior to T5 treatment to insure the maximum amount of hardening constituents are in solution at the time of hardening. SEM micrographic analysis shows that the mechanism whereby dendritic T6 elongation is retained or increased, silicon spherodization, is not taking place in T5 components. It is not seen in permanent mold or squeeze cast samples prepared with A356 alloys.

This work clearly shows that there is a definite phenomenon taking place during the T5 treatment of semi solid formed components, the cause of this retention of the as-cast ductility is still being pursued to establish the operative mechanism. A series of experiments has already been planned to explore the boundaries and
causes of these phenomena. In addition to tensile and hardness measurements of thixocast and rheocast A356 components produced by SAG (Salzburger Aluminium AG) and THT Presses (Dayton OH, USA), TEM examination of samples will allow examination of the actual hardening phases present. It may be that the artificial aging kinetics differ in SSM components in some manner which can explain the unusual retained ductility, and these TEM investigations will help us explore this factor.

**Table 5.1:** Proposed experimental matrix

<table>
<thead>
<tr>
<th>MATERIAL</th>
<th>TEMPER</th>
<th>SOL’N TEMP [C]</th>
<th>SOL’N Time (hr)</th>
<th>AGING TEMP [C]</th>
<th>AGING Time (hr)</th>
</tr>
</thead>
<tbody>
<tr>
<td>THT (rheo.)</td>
<td>F</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>SAG (thixo.)</td>
<td>F</td>
<td>--</td>
<td>--</td>
<td>--</td>
<td>--</td>
</tr>
<tr>
<td>THT/SAG</td>
<td>T5</td>
<td>--</td>
<td>--</td>
<td>140</td>
<td>4 / 6 / 8</td>
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<tr>
<td>THT/SAG</td>
<td>T5</td>
<td>--</td>
<td>--</td>
<td>160</td>
<td>4 / 6 / 8</td>
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<tr>
<td>THT/SAG</td>
<td>T5</td>
<td>--</td>
<td>--</td>
<td>180</td>
<td>4 / 6 / 8</td>
</tr>
<tr>
<td>THT/SAG</td>
<td>T6</td>
<td>540</td>
<td>6</td>
<td>160</td>
<td>5</td>
</tr>
</tbody>
</table>

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7.0 REFERENCES


