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1. EXECUTIVE SUMMARY

Semi-solid metal (SSM) processing is a relatively new process developed at MIT in the early 70's, which incorporates elements of both casting and forging. It capitalizes on thixotropy of semi-solid metals, and offers means to manufacture complex shapes that have substantially higher quality than die castings, but are lower in cost than those produced by alternative methods such as forging. As a result, SSM has emerged as a novel manufacturing scheme for the manufacture of complex net-shaped components of aluminum and magnesium alloys.

The advantages of semi-solid processing include low cycle time, significantly increased die life, reduced porosity, and improved casting qualities and mechanical properties. Therefore it has a significant impact on energy savings, as well as the reduction of process cost. In fact, analyses on process cost (SSM vs. conventional die casting) show that because of the reduced part weight, increased die life, and less machining cost, the cost saving for SSM castings usually falls in between 20-50%, depending on the size and material of the targeted cast part. Moreover, due to a much higher energy efficiency of the process, SSM methods can save up to 20% energy, which is enormous for the die casting industry. Based on the data provided by NADCA, the total tonnage of Al die castings was 1,173,962 in 2002, and the energy expenditure per ton was about 8.3×10^6 BTU. With the adoption of SSM, an estimated yearly energy saving would be 1.95×10^{12} BTU.

The SSM Consortium (now ACRC) at WPI has been carrying out fundamental, pre-competitive research in SSM for several years. Current and past research (at WPI) has generated many results of fundamental and applied nature, which are available to the SSM community. These include materials characterization, yield stress effects, alloy development, rheological properties, process modeling/simulation, semi-solid slurry formation, etc. Alternative methods to produce SSM slurries at lower processing costs and with reduced energy consumption are a critical need. The production of low cost SSM feedstock will certainly lead to a dramatic increase in the tonnage of castings produced by SSM, and will provide end users such as the transportation industry, with lighter, cheaper and high performance materials.

The program of research that was pursued consisted of three major tasks:

1. *Design and construct an apparatus to investigate liquid/liquid melt processing for the selected alloys; Investigate microstructural refinement by liquid/liquid melt thermal treatment and establish process controls for high quality SSM feedstock production.*
2. *Investigate microstructural refinement by addition of Si-B alloys, and establish process control for high-quality SSM feedstock production, and*
3. *Develop computational and modeling studies of SSM slurries.*

A summary of accomplishments and achievements in each of the above Tasks is given below.

1. Design and construct an apparatus to investigate liquid/liquid melt processing for the selected alloys; Investigate microstructural refinement by liquid/liquid melt thermal treatment and establish process controls for high quality SSM feedstock production (the CRP process).

The continuous rheoconversion process (CRP) is a novel, energy efficient, and low cost rheocasting process. The process involves the flow of two melts through a specially designed “reactor”. The two melts can be the same alloy composition, or can be two different alloy compositions to form a new alloy after they flow through and mix in the reactor. By using the CRP at a location before the entry point into the die cavity, one is able to produce SSM slurry as needed directly from the original melts (rather than purchasing SSM ingot stock, reheating it into the two-phase zone and then emplacing the reheated SSM ingot section into the cavity). The CRP is a major innovation allowing the caster to have the ability to produce SSM slurry on demand.

High nucleation rate combined with forced convection leads to (a) copious nucleation of the primary phase, (b) dispersal of these nuclei throughout the bulk liquid, and (c) survival of these nuclei via homogeneous temperature fields. These are the operative mechanisms for the CRP; the net result is total suppression of dendritic growth of the primary aluminum phase. The high heat extraction capability of the reactor combined with forced convection (via mixing) ensures the formation of thixotropic structures even with appreciable superheats in the precursor melts. The high level of grain refinement observed in the as-solidified samples is explained by highly potent nucleation events in the CRP, while the uniformity of the microstructure indicates that these nuclei were dispersed effectively by the fluid flow in the reactor.

The CRP has been shown to consistently generate near-ideal semi-solid structures for a variety of commercial alloys for both thixocasting and rheocasting applications. The results with various commercial aluminum and magnesium alloys indicate that the CRP is highly effective for the manufacture of high quality semi-solid feedstock.

In a follow-up DOE program - “Innovative Semi-Solid Metal Processing”, scale-up work is ongoing. Most recently, we have scaled up the CRP process for commercial applications. Several ACRC Consortium Members (Hayes-Lemmerz, THT Presses, Buhler, Magna etc.) have worked with us in beta trials for evaluation of the CRP. Due to the high energy efficiency and low cost of the CRP process, more and more companies will incorporate the CRP to their die casting facilities for the manufacture of high integrity die cast parts.

2. Investigate microstructural refinement by addition of Si-B alloys, and establish process control for high-quality SSM feedstock production.

SiBloy® SSM circumvents some critical problems encountered in traditional grain refined SSM feedstock such as the lack of grain size uniformity, the fading of nucleating agents, and the agglomeration and settling of insoluble nucleating particles, etc. The WPI team developed

SiBloy® SSM in collaboration with ACRC consortium members and in particular Elkem Aluminum; SiBloy® SSM is an optimized feedstock material for SSM processing. During thixocasting as well as rheocasting Beta trials, SiBloy® SSM proved to be an excellent candidate material for the manufacture of high integrity cast components. Moreover, due to the exceptional high ductility of SiBloy® SSM castings under T5 condition, the high temperature solution heat treatment can be eliminated. Thus enormous energy savings can be achieved. Elkem Aluminum is commercializing SiBloy® SSM material for the North American market.

3. Develop Computational and Modeling Studies of SSM Slurries

Due to their unique rheology, semi-solid metals fill the die cavity in a distinct way that affects the resultant mechanical properties. In this work, we have confirmed the flow patterns and instabilities observed during commercial forming operations by developing appropriate fluid flow models that take into account the yield stress effect. Our modeling results point out that the finite yield stress plays a critical role in determining filling behavior of semi-solid slurries. Flow instabilities that appear as unpredictable events during processing are related to the existence of the finite yield stress, the interplay between inertia, gravity and plastic/viscous resistance to flow, and structure evolution. The stability map developed by the research team is a valuable tool for the optimization of semi-solid processing. These results become the “input” parameters to predictive models thus ensuring that the resultant output is based on sound assumptions and that the physics of the system is adequately and properly expressed.

2. INTRODUCTION

As worldwide competitiveness becomes an increasingly urgent and consistent theme in manufacturing, new net shape forming processes, which offer opportunities to reduce total manufacturing costs, are more and more in demand. For high volume production of cast components, high-pressure die-casting is the process of choice; however, porosity and blistering due to subsequent heat treating and entrained oxides due to the turbulent nature of metal flow during die casting are some of the detractors. Squeeze casting offers certain advantages in that the melt flow rate is significantly low (in the order of 0.5 m/s), and subsequent to filling the cavity, the liquid is solidified under high pressure. Semi-solid metal (SSM) processing is a relatively new process developed at MIT in the early 70's, which incorporates elements of both casting and forging. It capitalizes on thixotropy of semi-solid metals, and offers means to manufacture complex shapes that have substantially higher quality than die castings, but are lower in cost than those produced alternative methods such as forging. As a result, SSM has emerged as a preferred manufacturing scheme for the manufacture of complex net-shaped components of aluminum and magnesium alloys.

Currently, there are two primary semi-solid processing routes: (1) thixocasting, and (2) rheocasting. In the thixocasting route, one starts from a solid precursor material that is specially prepared via magneto-hydrodynamic (MHD) stirring, strain induced melt activation (SIMA) or grain refinement methods. Upon reheating the material into the mushy zone, a thixotropic slurry is formed, which becomes the feed for the casting operation. Whereas, in the rheocasting route one starts from the liquid state, wherein a thixotropic slurry is formed directly from the melt via special thermal treatments, and subsequently is fed into the die cavity [1-4].

The advantages of semi-solid processing include low cycle time, significantly increased die life, reduced porosity, and improved casting qualities and mechanical properties. Therefore it has a significant impact on energy savings, as well as the reduction of process cost. In fact, analyses on process cost (SSM vs. conventional die casting) show [5-7] that because of the reduced part weight, increased die life, and less machining cost, the cost saving for SSM castings usually falls in between 20-50%, depending on the size and material of the targeted cast part. Moreover, due to a much higher energy efficiency of the process, SSM methods can save up to 20% energy, which is enormous for the die casting industry. Based on the data provided by NADCA, the total tonnage of Al die castings was 1,173,962 in 2002, and the energy expenditure per ton was about 8.3×10^6 BTU. With the adoption of SSM, an estimated yearly energy saving would be 1.95×10^{12} BTU.

3. BACKGROUND

The SSM Consortium (now ACRC) at WPI has been carrying out fundamental, pre-competitive research in SSM for the benefit of the industry. Current and previous researches at WPI in semi-solid metal processing have produced a large quantity of results of fundamental and applied nature, which are available to the SSM community. These include materials characterization, yield stress effects, alloy development, rheological properties, process modeling/simulation, semi-solid slurry formation, etc. With the accumulated body of knowledge, we need to investigate alternatives to produce SSM slurries at lower processing costs and with reduced energy consumption. The production of cheaper SSM feedstock will certainly lead to a dramatic increase in the tonnage of castings produced by SSM, and will provide end users such as the transportation industry, with lighter, cheaper and better materials.

The principal goal of the project was to develop and apply efficient grain refining methods to produce SSM billets with consistent characteristics at significantly lower cost premiums relative to methods currently in use (MHD stirring or slurry-on-demand). The second goal of this research was to carry out computational and modeling studies of the fluid flow behavior of SSM slurries. Though much progress has been made at ACRC on this topic, issues related to control of instabilities still remain and case studies have to be developed. A third goal of this research was to establish methods and procedures to demonstrate and accurately quantify the energy and total cost savings realized with the adoption of simpler methods for SSM billet production compared to current methods.

The broad objectives of the project were to: **1)** develop apparatus and processing techniques for liquid/liquid melt treatment (Rheocasting), evaluate the current status of alloys refined by Si-B additions and develop processing techniques for SSM billet manufacturing. Technical solutions must be simple and should be easily implemented in existing industrial facilities; **2)** evaluate the degree of microstructural modification and refinement obtained with the proposed methods. Control variables will be identified and the structure of SSM slurries produced by re-heating grain-refined billets will be evaluated for quality, homogeneity; and reproducibility; **3)** evaluate grain-refined SSM feedstock using industrial forming equipment and carry out a systematic investigation/measurement of cast properties (Thixocasting); **4)** employ computational and modeling studies to investigate slurry constants and slurry structure breakdown, which are sorely needed for predictive models; **5)** use constitutive models to identify, understand, and describe flow instabilities that occur during actual processing of slurries, and **6)** provide mechanisms to control process instabilities, an important step to ensure commercialization of SSM technology.

The approaches were based on two recently developed grain refining processes: the liquid/liquid melt thermal treatment process, and the SiBloy® permanent grain refinement technology developed at NTNU and commercialized by Elkem Aluminum ANS of Norway. These processes have been proven very effective for grain refinement of aluminum alloys. Moreover, they are applicable not only to existing commercial alloys, but also to future generations of alloys tailored more specifically to SSM forming.

The program of research that was pursued consisted of the following three major tasks:

1. *Design and construct an apparatus to investigate liquid/liquid melt processing for the selected alloys; Investigate microstructural refinement by liquid/liquid melt thermal treatment and establish process controls for high quality SSM feedstock production.*
2. *Investigate microstructural refinement by addition of Si-B alloys, and establish process control for high-quality SSM feedstock production, and*
3. *Develop computational and modeling studies of SSM slurries.*

In the following sections, we detail the research results and achievements emanating from the above cited Tasks.

4. RESULTS AND DISCUSSION

4.1 Liquid Mixing: The Continuous Rheoconversion Process (CRP) (Task 1)

In the early days of SSM development, it was thought that one had to cool the liquid down into the two-phase region, form dendrites, and then shear off and break the dendrites (i.e. melt agitation via mechanical or, later on, magnetohydrodynamic [MHD] stirring) in order to produce a slurry. However, during the last few years, work sponsored at ACRC – MPI by the Department of Energy [8], as well as work by the research team at MIT [9] led to the discovery that one did not need to break off dendrites to produce the semi-solid structure of globular primary alpha phase. Instead, if the temperature of the melt was such that one could produce many nuclei (“copious nucleation”), and if the nuclei did not grow past a certain point (i.e. suppression of dendritic growth), nor melt back into the bulk liquid, then one could produce a slurry with the ideal semi-solid structure directly from the melt. Armed with the new understanding, the research team has developed a novel, low cost rheocasting process termed the Continuous Rheoconversion Process (CRP). The following sections give an overview of the CRP, and detail the salient experimental results of applying the process to the manufacture of high-quality SSM feedstock for commercial alloys.

4.1.1 CRP: Concept & Apparatus

The CRP is a simple process in which two melts (either of the same alloy, or two different alloys), held at a particular level of superheat, are passively mixed within a reactor. The reactor provides heat extraction and forced convection during the initial stage of solidification, leading to the formation of thixotropic structures [10-11]. Figure 1 illustrates the concept of the CRP process. The advantages of the CRP include process simplicity, flexibility, tight control over SSM structure evolution, fast adjustment of solid fraction, and incorporation of scrap metal for recycling. The term “flexibility” refers to the ability of the process to be viable for both thixocasting and rheocasting applications.

Figure 2 is a photograph of the CRP laboratory apparatus. The major characteristics of the CRP apparatus include independent temperature control of each precursor alloy melt, a heated channel system to transport the two melts, and a reactor that enables two phenomena to occur: (a) copious nucleation within the melt, and (b) forced convection of the two streams as they flow through the reactor. The reactor can be preheated to vary the level of heat extraction. Important parameters include the superheat and chemical composition of the melts, the heat extracting rate of the reactor, and the temperature of the receiving reservoir.

4.1.2 CRP: SSM Structures

The CRP is a flexible process in that it can be used for both the thixocasting and rheocasting routes. As shown in Figure 3, in the thixocasting route, the slurry is solidified in air within a clay-graphite crucible, after which small “slugs” from the solidified sample are reheated into the two-phase range and quenched for microstructure analysis. In the rheocasting or slurry-on-demand

route, the slurry is collected and quenched into water at various temperatures within the two-phase range of the alloy.

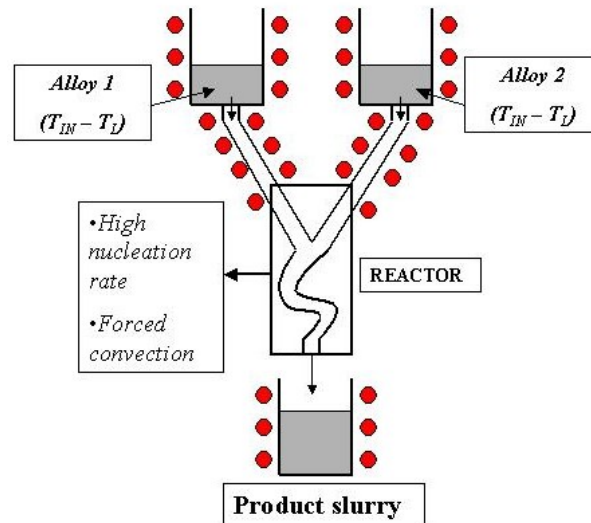


Figure 1: Conceptual schematic of the Continuous Rheoconversion Process (CRP).



Figure 2: Photograph of the CRP laboratory apparatus.

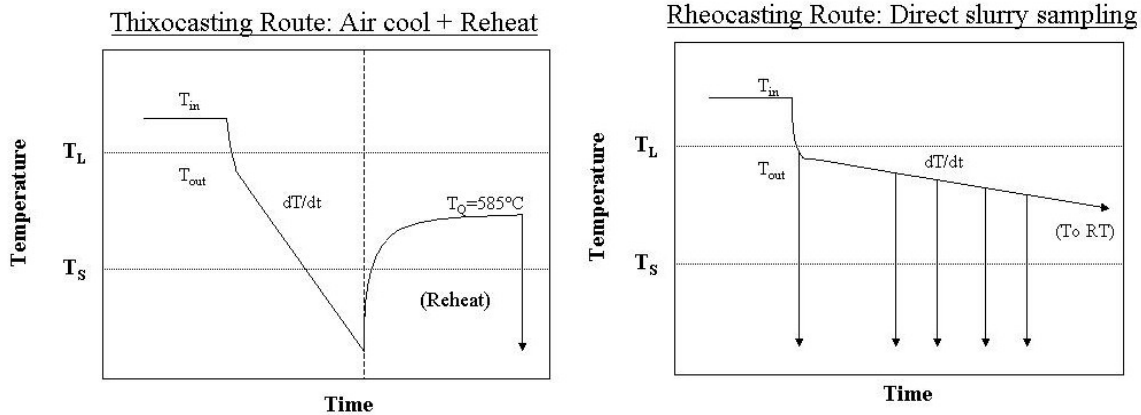


Figure 3: Schematic showing the solidification paths of the CRP's thixocasting and rheocasting routes. Arrows indicate water quenching of slurry.

Figure 4 exhibits typical semi-solid structures of an A356 alloy (without grain refinement) obtained by mixing two low-superheat melts with the same initial temperature (superheat: 9°C). Whereas Figure 5 shows typical semi-solid structures of the same alloy obtained by mixing two high-superheat melts (superheat: 44°C). From Figures 4 and 5, one can see that under both conditions, a globular, near-ideal semi-solid structure can be obtained. This indicates that the reactor is able to extract a very large amount of heat in a small amount of time. Thus the CRP has a large processing window, and therefore excellent process control (in terms of thermal management).

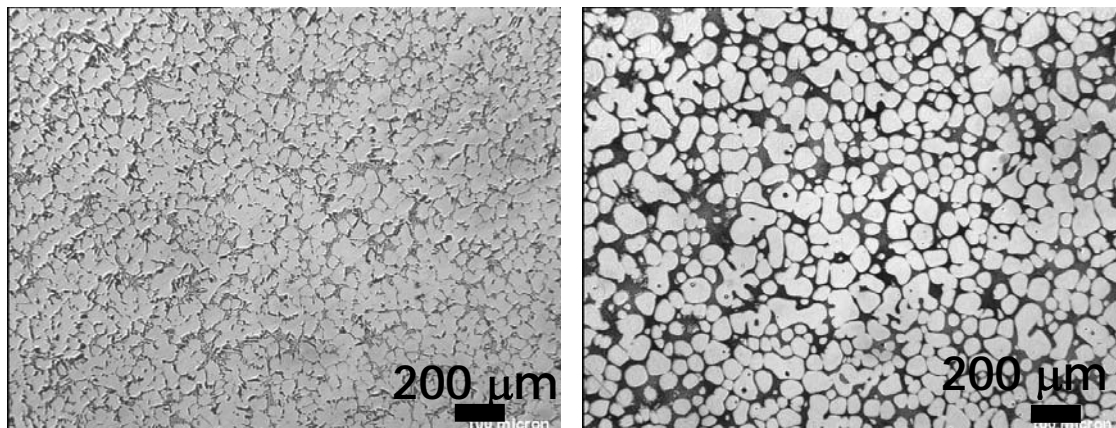


Figure 4: Microstructures of an A356 alloy obtained by mixing two low-superheat melts (superheat: 9°C): (a) As-solidified (left); (b) Reheated to 585°C and water quenched (right).

Figures 6 and 7 illustrate typical semi-solid structures of TiB_2 grain-refined A356 alloys obtained under different rheocasting conditions. Figure 6 compares semi-solid microstructures of A356

alloy slurries sampled at two different temperatures (605°C vs. 585°C). The successful use of this sampling method was an important indicator that the CRP can indeed generate slurries with near-ideal semi-solid microstructures directly from the molten state; i.e., the rheocasting route can be effectively followed with the CRP. The Figures document the gradual change in morphology of a slurry batch that is cooled very slowly through the two-phase temperature range. Figure 7 compares semi-solid structures of A356 alloy slurries with and without grain refinement. From Figure 7, one can see that grain refinement does not significantly change the resultant semi-solid structures. The presence of grain refiners in an alloy only improves the resultant structures to a small degree, suggesting that the level of nucleation provided by the reactor itself is sufficient for the formation of high quality semi-solid structures.

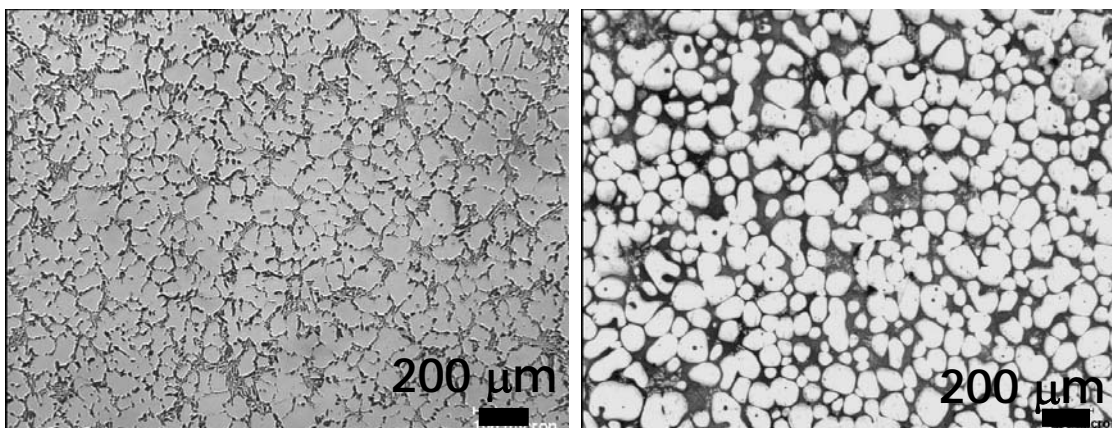


Figure 5: Microstructures of an A356 alloy obtained by mixing two high-superheat melts (superheat: 44°C): (a) As-solidified (left); (b) Reheated to 585°C and water quenched (right).

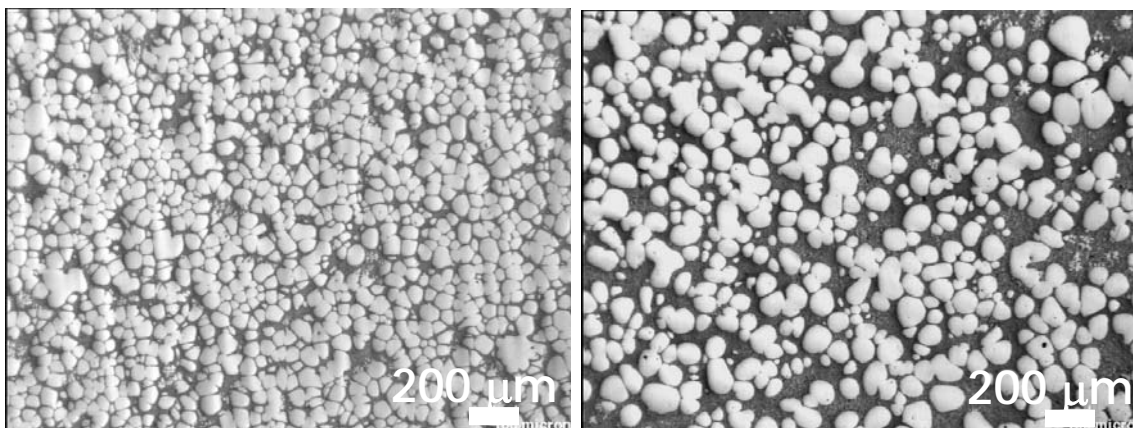


Figure 6: Microstructures of grain-refined A356 slurries sampled at two different temperatures in the two-phase region, (a) 605°C (left), and (b) 585°C (right).

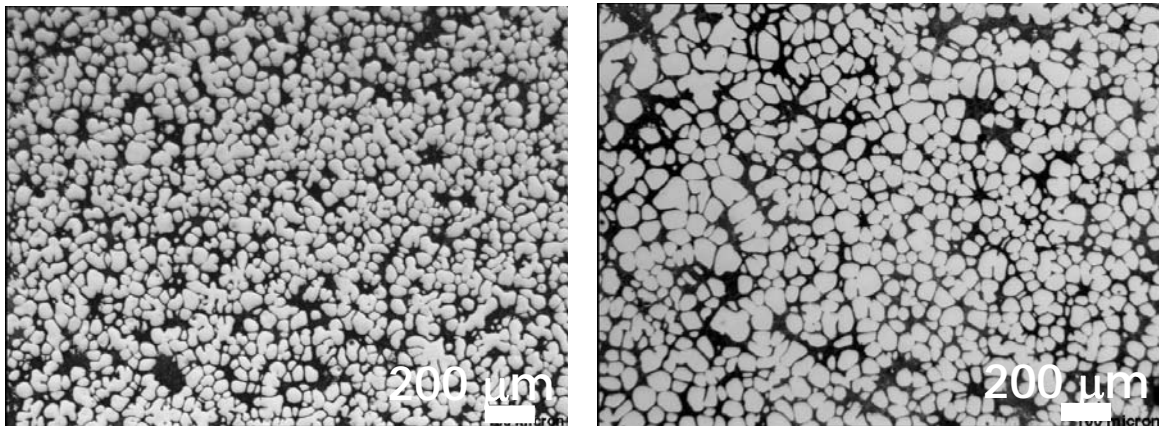


Figure 7: A comparison of semi-solid structures of TiB₂ grain refined A356 slurry (left); and A356 slurry without grain refinement (right). Samples quenched at 590°C.

In addition to Al-Si foundry alloys, we have processed a variety of other alloy systems using the CRP, including hypereutectic alloy 390, wrought alloys, Al-Cu alloys etc. It has been found that the CRP can consistently produce high quality semi-solid structures even for those alloys that are not “castable,” or which are known for their poor castability. Figure 8 shows such a structure from an A206 alloy (Al-4.5wt%Cu). Experimental results pointed out that the CRP can improve the castability of the alloy significantly. The reason for this is that the CRP changes the morphology of the primary phase from coarse dendrites to fine globules, which eliminates the feeding problem during solidification that is associated with traditional casting methods. Therefore, this alloy is actually “castable” using the CRP, and this holds true for various wrought alloys as well.

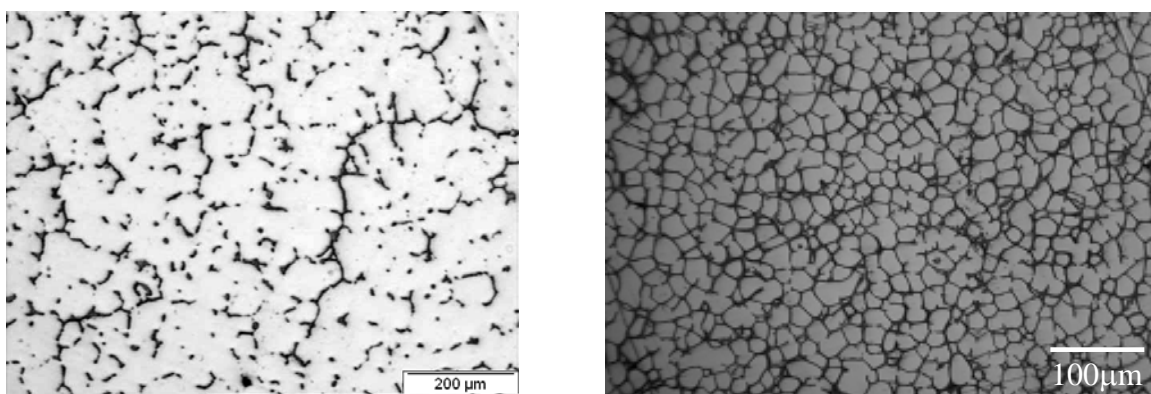


Figure 8: Microstructures of an A206 alloy: (a) Air-cooled from the liquid state (left), (b) As-solidified structure obtained with the CRP (right).

4.1.3 CRP: Operative Mechanisms

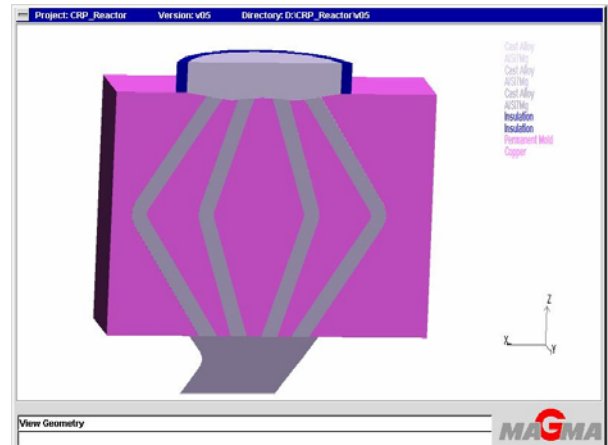
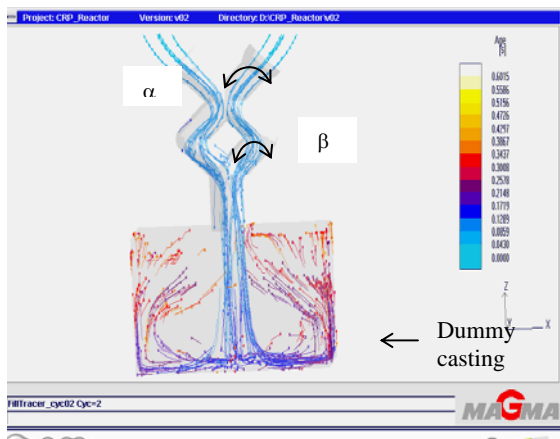
High nucleation rate combined with forced convection leads to (a) copious nucleation of the primary phase, (b) dispersal of these nuclei throughout the bulk liquid, and (c) survival of these nuclei via homogeneous temperature fields. These are the operative mechanisms for the CRP; the net result is total suppression of dendritic growth of the primary aluminum phase. The high heat extraction capability of the reactor combined with forced convection (via mixing) ensures the formation of thixotropic structures even with appreciable superheats in the precursor melts. The high level of grain refinement observed in the as-solidified samples can be explained by highly potent nucleation events within this reactor, while the uniformity of these structures throughout the samples indicates that these nuclei were dispersed effectively by the fluid flow in the reactor.

4.1.4 CRP: Reactor Optimization

As mentioned earlier, the reactor provides rapid heat extraction, copious nucleation, and forced convection during the initial stage of solidification, leading to the formation of thixotropic structures. Obviously, the reactor plays a crucial role in structure formation in the CRP. To optimize the reactor design for the ultimate purpose of scaling up the CRP to an industrial level, the reactor was modeled using commercial simulation and computational tools. In addition, fluid flow experiments in plexiglass models were conducted to verify these concepts.

Figure 9 shows the flow pattern of two liquids as they flow through a reactor. Modeling results show that the angles α and β are important. To ensure a certain amount of mixing/convection in the reactor, the value of α and β has to be less than 90° . By analyzing the temperature profile and fluid flows in the reactor, we found that the high heat extraction capability of the reactor ensures the formation of a plurality of nuclei at the inner walls of the reactor, and subsequently, the nuclei are swept away and dispersed uniformly into the slurry due to the mixing action. Figure 10 shows a reactor with a different geometry. Simulation results suggest that by increasing the number of the paths/channels in the reactor, the nucleation rate as well as level of mixing/convection can be maximized. Fluid flow experiments in plexiglass models have verified the flow patterns in these reactors.

In summary, the CRP is a novel, energy efficient, and low cost rheocasting process. It has been shown to consistently generate near-ideal semi-solid structures for a variety of commercial alloys for both thixocasting and rheocasting applications. The results with various commercial aluminum and magnesium alloys indicate that the CRP is highly effective for the manufacture of high quality semi-solid feedstock. It is envisioned that due to the high energy efficiency, and low cost of the CRP process, the process can be easily scaled up and adopted by die casting industry to make castings with high integrity in the near future.



4. 2. Microstructural Refinement: SiBloy® SSM (Task 2)

Compared to MHD techniques, the chemical grain refinement approach is more flexible and cost-effective. Therefore, grain refined SSM feedstock has been employed for semi-solid processing by many die casters. However, there are some drawbacks inherent to the conventional Al-Ti/Al-Ti-B/Al-Ti-C grain refined materials such as lack of grain size uniformity, the fading of nucleating agents, and the agglomeration and settling of insoluble nucleating particles in the melt. These can negatively affect the quality and productivity of SSM castings.

Early on in the Program, we evaluated the feasibility of applying an emerging permanent grain refinement technique-SiBloy® for SSM applications. SiBloy® technology was developed for shaped casting (**NOT** for SSM), and patented by Elkem Aluminum. Unlike traditional grain refining techniques, the grain refining effect of SiBloy® is achieved by adding Si-B master alloy into the melt. During cooling, fresh AlB_2 particles (instead of insoluble TiB_2 , TiAl_3 etc.) precipitate out from the melt just above the liquidus temperature, which, in turn, serve as potent nucleating agents, and thus grain refining the melt. Our evaluation results of Al-Si cast alloys pointed out [12-13] that the Si-1B additive gives rise to the finest grain structure with a small boron addition level ($\sim 0.015\text{wt}\%\text{B}$) in contrast to traditional grain refiners such as Al-6Ti, Al-5Ti-1B, Al-5Ti-0.2B, Al-4B etc. Moreover, the grain refining effect was found to be: 1) independent of holding time (no fading); 2) unaffected by remelting treatments (permanent effect), and 3) independent of cooling rate in the range between 0.5 and 15°C/s . More importantly, SiBloy® technique can circumvent some critical problems encountered in traditional grain refining methods such as the lack of grain size uniformity, the fading of nucleating agents, and the agglomeration and settling of insoluble nucleating particles, etc. This new technique shows a great potential for the manufacture of high-quality SSM feedstock.

Subsequent to the evaluation work, we conducted extensive experiments to tailor/optimize SiBloy® for semi-solid processing. An optimal processing window for SSM applications was established, and provided to our industrial partner-Elkem Aluminum. A brand new SiBloy® alloy (SSM Version) was made using Elkem's production unit (we termed the alloy--*SiBloy® SSM*). Thixoforming Beta trials of the alloy were carried out at Madison-Kipp Corp. WI, in November 2003, and Rheocasting Beta trials were conducted at THT Presses in May, 2004. Both Beta trials were very successful. We were able to produce sound SiBloy® SSM castings with high integrity and at a significantly reduced process cost (15%-20% lower than conventional die casting method). Given below is a summary of our optimization work and some important results from the Beta trials.

As illustrated in Figure 11, we systematically investigated the effect of various processing parameters on billet as-cast microstructure, as well as semi-solid structure of AlB_2 grain refined SiBloy® alloys. These parameters include casting temperature, cooling rate, B level, convection level (casting process); as well as reheating rate, reheating temperature and isothermal holding time (thixoforming process).

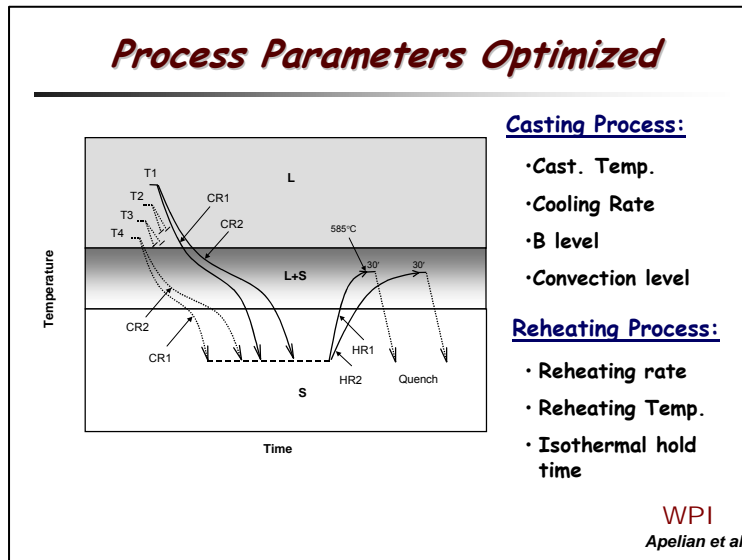


Figure 11: Schematic diagram of the processing parameters optimized.

Figures 12 and 13 show the effect of billet casting temperature on the morphology of the as-cast microstructure, as well as the semi-solid structure of an AlB_2 grain refined A356 alloy. By lowering the casting temperature, one changes the morphology of the billet's as-cast microstructure from a highly dendritic structure (Figure 12a) to a rosette-like structure (Figure 12c). As can be seen in Figure 13, the semi-solid structure of billets cast at a relatively low pouring temperature is characterized by small, round alpha particles with much less entrapped liquid in comparison to those cast at a high pouring temperature.

Extensive optimization experiments pointed out that billet casting temperature, cooling rate, as well as B level play an important role in the formation of high quality semi-solid structures. Based on experimental data, an optimal processing window for SiBloy billets (thixocasting applications) was established as follows:

- Billet casting temperature: 640-660 °C
- Billet cooling rate: $\geq 40^\circ\text{C/s}$ (as high as possible)
- B level: 250-300ppm
- Reheated temp.: 585 °C
- Isothermal holding time: 2-5 min.
- Reheating rate: flexible

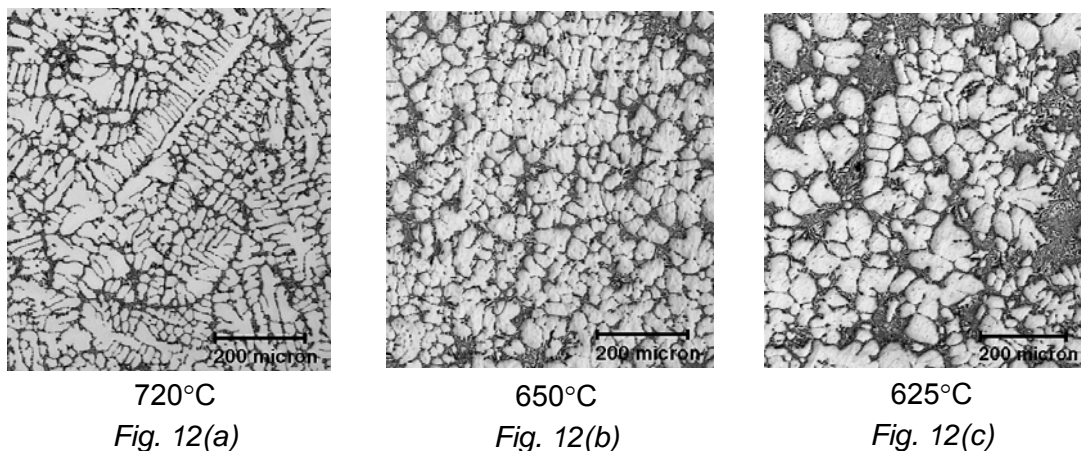


Figure 12: Evolution of as-cast microstructure of an AlB_2 grain refined A356 as a function of billet casting temperature.

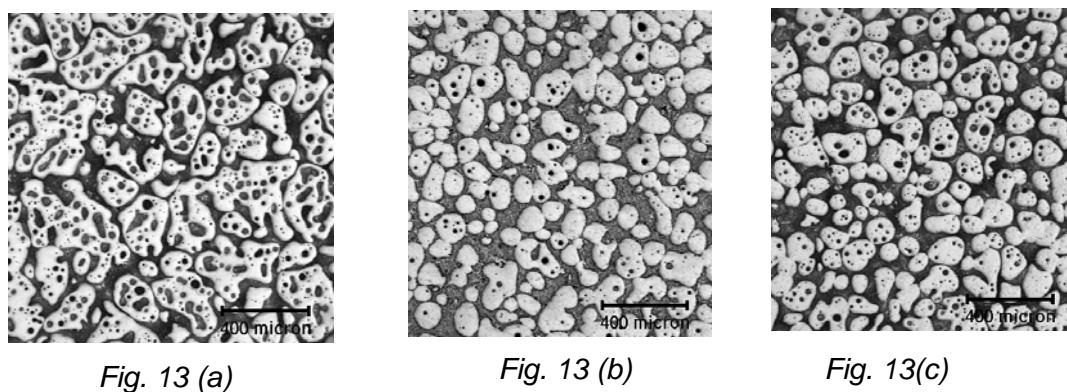
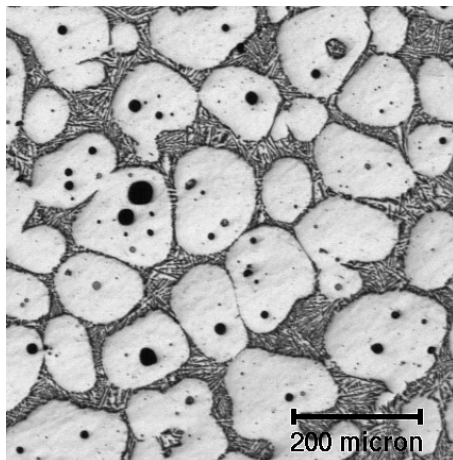


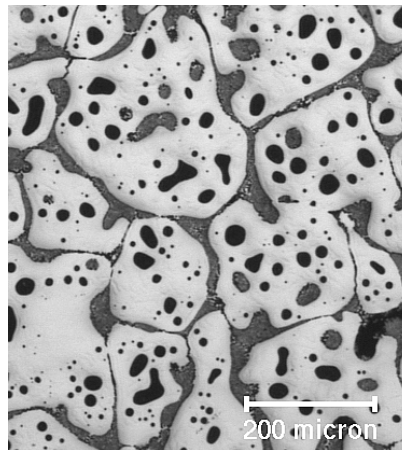
Figure 13: Evolution of semi-solid structure of an AlB_2 grain refined A356 as a function of billet casting temperature: (a) 720°C, (b) 650°C, and (c) 625°C. The billets were all partially remelted at 585°C.

The semi-solid structure of optimized AlB_2 grain refined Al-Si alloys is characterized by small, non-dendritic alpha particles with a small amount of entrapped liquid. Figure 14 compares such an optimized structure with a commercial Ti-B grain refined SSM alloy. Through image analysis work we determined that the semi-solid structure of SiBloy® SSM billets has about 4 times less entrapped liquid content, a smaller alpha particle size (90 μm versus 128 μm), and a better morphology of the alpha phase (shape factor: 1.35 versus 1.40) as compared to commercial TiB_2 grain refined semi-solid A356 billets.



SiBloy® SSM

Fig. 14(a)



GR A356

Fig. 14(b)

Figure 14: A comparison of semi-solid structures of SiBloy® SSM billets (Fig. 14a) with those of commercial TiB_2 grain refined A356 semi-solid billets (Fig. 14b). (Reheated Temperature: 585°C).

Based on the optimal processing window, we manufactured a brand new AlB_2 grain refined A356 alloy (the material is termed SiBloy® SSM) using Elkem's production unit. Thixoforming Beta trials of SiBloy® SSM billets were conducted using the thixoforming cell of Madison-Kipp Corporation. The cell consists of a multi-station 350kW induction reheating unit with 18 induction coils and an 930 ton Buhler die casting machine. It was found that the response of SiBloy® SSM to induction heating is slightly different from commercial MHD materials. To ensure sufficient flow properties for die casting operations, a 4-5°C higher processing temperature is needed for SiBloy® SSM billets (in comparison to MHD ones). Through optimizing both billet reheating and die casting processes, sound semi-solid SiBloy® castings were consistently made, as illustrated in Figure 15. The casting (named "gear shift lever bracket") is an integral part in the steering column for GMT-800 series trucks, which requires good internal integrity and a relatively high strength.

In parallel, rheocasting Beta trials of SiBloy® SSM slurry were carried out in March, 2004 at THT Presses using a 200T SLC casting cell. For comparison, the die used for thixocasting at Madison-Kipp Corp. was also used in this study. More than 40 castings were produced from the rheocasting Beta trials, and later on examined together with those obtained from the Thixocasting trials. Figure 16 illustrates the casting cell and SiBloy® SSM castings produced.

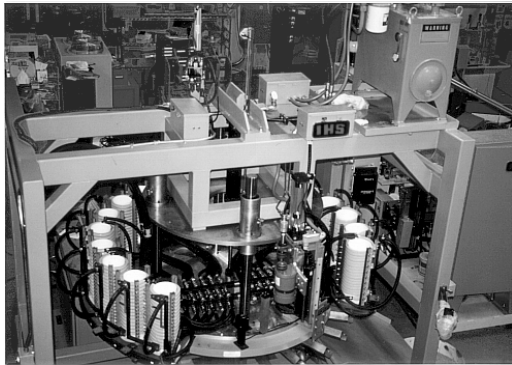


Figure 15: Thixoforming cell at Madison-Kipp Corp. and SiBloy® SSM castings produced.

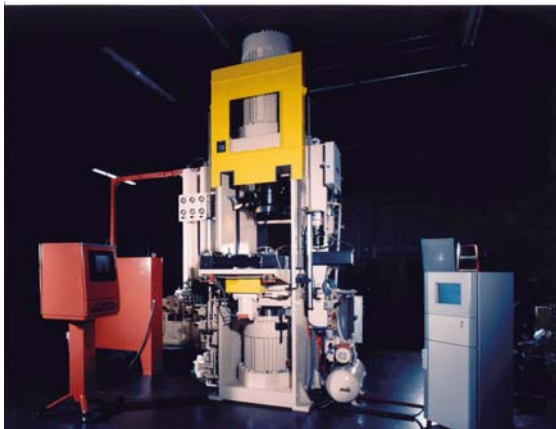


Figure 16: A 200T THT SLC casting cell and SiBloy® SSM castings produced.

Subsequent to the Beta trials, all SiBloy® SSM castings were heat treated under T5 and T6 conditions, and then four tensile samples were cut from each heat treated casting for tensile testing (see Figure 17). Table 1 gives tensile testing results of SiBloy® SSM castings under as-cast, T5 and T6 conditions, and Table 2 gives typical mechanical properties of 357 alloy that has a similar chemical composition as SiBloy® SSM. From Table 1 and Table 2, one can see that SiBloy® SSM castings have excellent mechanical properties. Processing route (thixo vs. Rheo) does not show a significant influence on the mechanical properties of castings. Another important finding is that SiBloy® SSM castings show an exceptional high ductility under T5 condition (for traditional sand/permanent mold castings, there is about 50% ductility loss after

T5 heat treatment). Therefore T6 heat treatment is not needed for most commercial applications, and this is a huge saving for energy, as well as process cost.

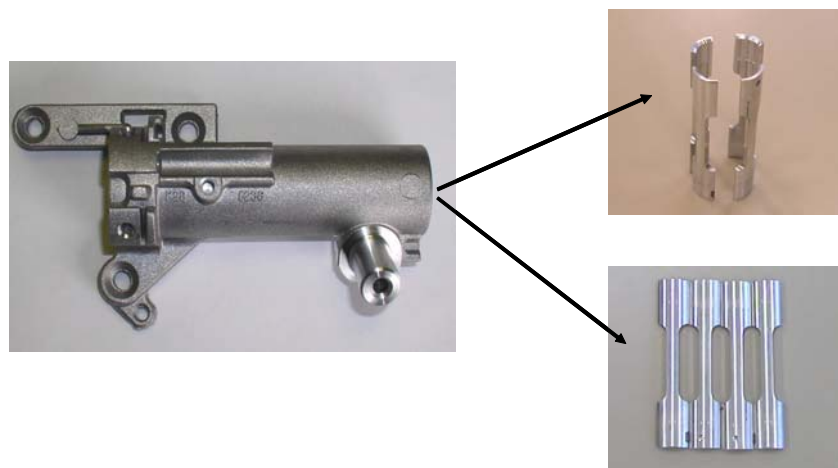


Figure 17: Showing tensile test samples cut from SiBloy® SSM castings.

In summary, SiBloy® SSM circumvents some critical problems encountered in traditional grain refined SSM feedstock such as the lack of grain size uniformity, the fading of nucleating agents, and the agglomeration and settling of insoluble nucleating particles, etc. Both thixocasting and rheocasting Beta trials show that SiBloy® SSM is an excellent candidate material for the manufacture of cast components with high integrity. Moreover, due to the exceptional high ductility of SiBloy® SSM castings under T5 condition, the high temperature solution heat treatment can be eliminated. Thus enormous energy savings can be achieved. Currently, we are assisting our industrial partner-Elkem Aluminum to finalize a commercializing plan to bring the SiBloy® SSM material to the North American market.

Table 1: Tensile test results of SiBloy® SSM castings

	Thixo			Rheo		
	UTS (ksi)	YS (ksi)	EL(%)	UTS (ksi)	YS (ksi)	EL (%)
As-Cast	36.0	26.0	9.0	33.0	24.0	8.0
T5	42.0	32.0	8.0	40.0	30.0	8.0
T6	48.0	43.0	12.0	45.0	40.0	11.0

Note: The alloy composition of SiBloy® SSM is close to 357 alloy.

Table 2: Typical properties of A357 alloy in F, T-5 and T-6 tempers.

Method	% Mg/Cu	Temper	UTS (ksi)	YS (ksi)	EL (%)
A357 Sand	0.6 Mg/0 Cu	As-Cast	24	13	6
		T-5	26	18	3
		T-6	43	33	5
A357, Sqz	0.6 Mg/0 Cu	T-5	29	21	5
		T-6	47	36	8

4. 3. Computational Modeling and Rheology Studies (Task 3)

All existing and future SSM technologies are based on the unique combination of solid-like and liquid-like behavior of semisolid metals whose rheological behavior is not fully understood. Mathematical modeling and simulations remain a critical issue in understanding and optimizing the process. The strategy followed for this Task are to: 1) employ computational and modeling studies to investigate SSM slurry constants and slurry structure breakdown, which are experimentally difficult to do; 2) use constitutive models to identify, understand, and describe flow instabilities that occur during actual processing of slurries, and 3) provide mechanisms to explain and control those instabilities, an important step to promote further commercialization of SSM technology.

To understand the filling behavior of semi-solid metals and the flow instabilities encountered during commercial forming operations, we modified the constitutive model previously developed by the ACRC team [14-15]. Specifically, to deal with the discontinuity in the constitutive relation, a regularized model is developed as follows [16]:

$$\tau = \left[\eta + \tau_o \frac{1 - \exp(-m \dot{\gamma})}{\dot{\gamma}} \right] \dot{\gamma} \quad (1)$$

where τ is the shear stress, η is the effective viscosity, $\dot{\gamma}$ is the shear rate, γ is the second invariant of $\dot{\gamma}$, and the parameter m , which has dimensions of time, controls the exponential rise in the stress at low rates of strain. The material parameters, τ_o and η , are determined from experimental data. The ideal Bingham-plastic behavior can be approximated by relatively large values of m .

Dimensional analysis shows that filling depends on two-dimensionless parameters, the Reynolds number, and the Bingham number, given by:

$$\text{Re} = \frac{\rho U_0 H}{\eta} \quad \text{and} \quad \text{Bi} = \frac{\tau_o H}{\eta U_0} \quad (2)$$

where ρ is the density and U_0 is the average inlet velocity.

A complete map of filling patterns has been developed in a wide range of Reynolds and Bingham numbers experienced in semi-solid processing (see Figure 18). Specifically, five distinct flow patterns have been identified. They are:

Shell: is a filling of the cavity by a Newtonian fluid at a relatively high Reynolds number, where inertia force dominates the flow. The jet emanating from the inlet section reaches the end of the cavity, and splits into two layers along the upper and lower walls of the cavity. This pattern is a typical die filling in liquid metal casting. For SSP process, this type of filling is undesirable as a large volume of gas is entrapped.

Mound: is a filling at a lower Re number (i.e. lower inertia forces), where viscous effect is dominant. The higher viscous stress prevents the flow from splitting into two parts, and filling proceeds with growth and slow spreading of a central column or mound, as shown in Figure 18. For semi-solid processing, this filling pattern is desirable since no gas is entrapped.

Disk: is a filling under large Reynolds and Bingham numbers, where inertia and plastic drag prevail over viscous force. The initial jet splits into two layers, very similar to shell filling. However, these layers are sufficiently thick, and do not propagate backwards along the side-walls; instead, they form a disk front moving from the closed-end to the front of the cavity. In disk filling, the effects of the yield stress balance the inertia, a large part of the material comes to rest, and flow is confined near the advancing front.

Bubble: is a filling under a relatively low Reynolds number, but rather high Bingham number where yield stress is dominant. The filling pattern in this case is different from the previous cases. Once the jet hits the end of the cavity, the main growth of a single "solid-like" core occurs first at the end of the jet, and then moves upstream like a wave, and it eventually reaches the entrance of the cavity. After the wave reaches the entry, the filling proceeds primarily as a growing "bubble" at the open-end of the cavity.

Transition: is an intermediate flow pattern between mound and bubble. Filling in this case starts as disk, where the slurry spreads along the wall at the closed-end of the cavity, but it finally develops into a bubble-like filling pattern. At the moment the bubble starts to develop, the extent of the stagnant region remains constant indicating a local balance of inertia force and yield stress.

The simulations highlight the significant role of the finite yield stress in Bingham type flows. Particularly, the bubble filling cannot be identified if the constitutive relation does not take into account the yield stress of the slurry. More importantly, experimental observations have confirmed the existence of the five flow patterns in semi-solid metals, for example, Paradies and Rappaz [17] observed shell, mound, disk flow patterns in semi-solid A356 alloys while filling a simple cavity under different conditions. Moreover, the filling experiments of Midson *et al* [18] have clearly shown the bubble filling pattern observed in semi-solid 357 alloy. Therefore, it is quite evident that in semi-solid processing where the material exhibits a yield stress, its inclusion in the constitutive flow relations for computational modeling is critical. Modeling efforts that neglect the presence of the material's yield stress may give erroneous results.

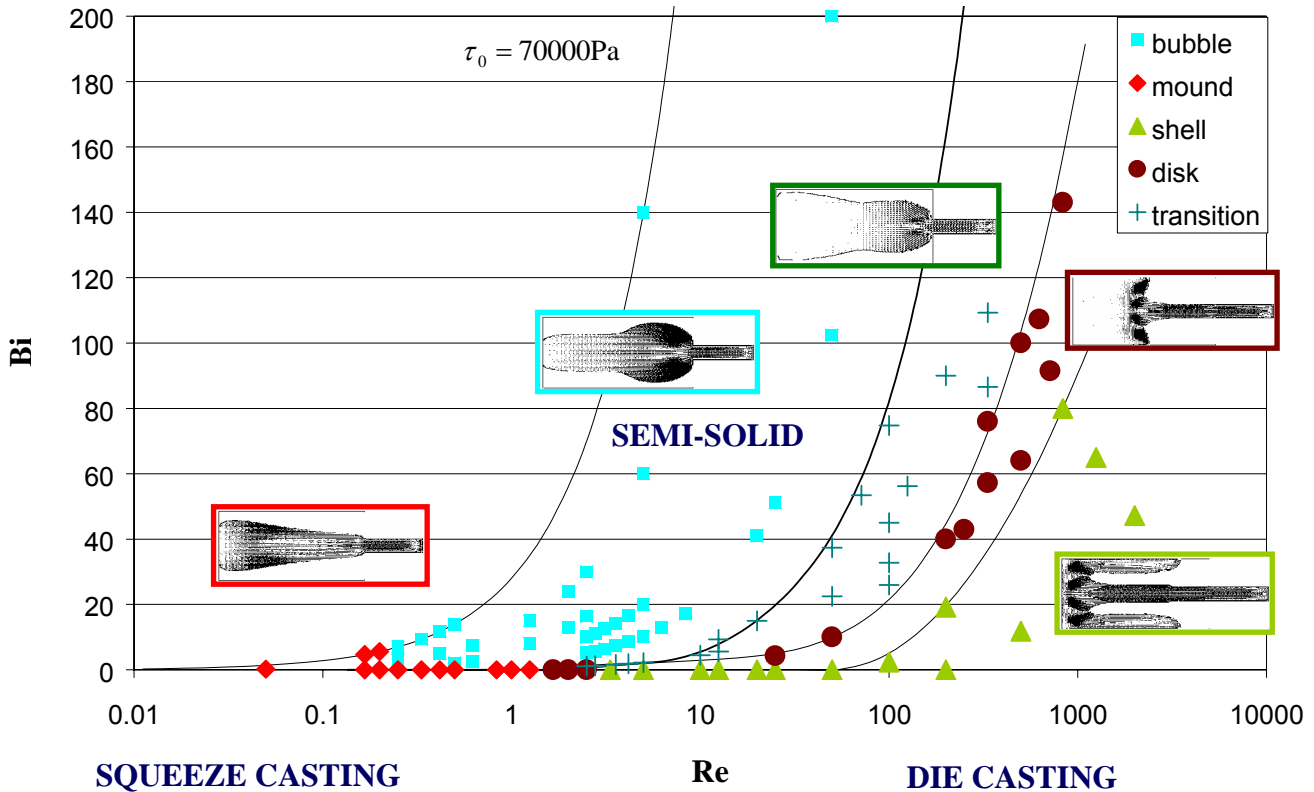


Figure 18: Flow patterns as a function of Bingham number, Bi and Reynolds number, Re . The figure illustrates five distinct filling patterns (bubble, mound, shell, disk and transition) in semi-solid casting.

The challenge in processing semi-solid metal slurries is the scrap rate due to difficulties associated with die filling. Flow instabilities such as the “toothpaste behavior” have been observed in commercial forming operations, as shown in Figure 19. To understand flow instabilities, we analyzed the flow under a 2-D geometry (see Figure 20) using “exact” finite element simulations along with a moving mesh scheme. The simulations are considered “exact” since the mesh of the finite element method follows the motion of the fluid, and the boundary conditions are satisfied exactly. In numerical simulations, the yield stress effect is considered, and a small disturbance is introduced in the flow by imposing an asymmetric velocity profile at the inlet for a short time Δt , beginning at the moment the jet of fluid reaches the vertical wall (defined as $t=0$). For $t > \Delta t$, the inlet velocity is kept constant and symmetric. In both the symmetric and asymmetric cases the volumetric flow rate is kept constant. The flow field and

the jet stability are found to be independent of the magnitude and the duration of the asymmetry.

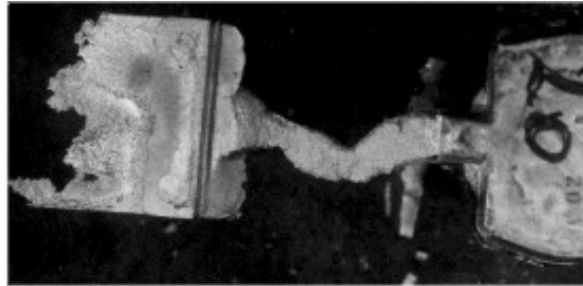


Figure 19: Toothpaste behavior observed in semi-solid casting (courtesy of Aluminum Pechiney).

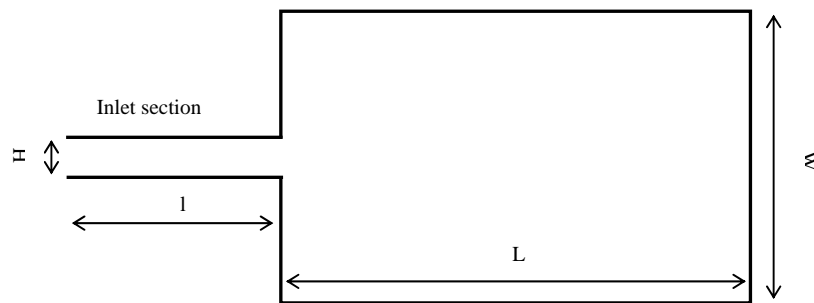


Figure 20: Geometry of the two dimensional cavity: ($H=2$ cm; $l=7.8$ cm; $L=20$ cm and $W=10$ cm).

Figure 21 illustrates the jet behavior at a low Reynolds number, $Re=1$, and moderate Bingham number $Bi=3$. Under “ideal” conditions (a symmetric velocity profile at the inlet, no disturbance), this flow leads to a bubble pattern. However, if a small disturbance is introduced from the start of the flow ($t=0$) until $\Delta t=0.15$, it triggers an instability that forces the jet to bend, and the fluid starts to flow sideways leading to an unstable jet profile, as shown in Figure 22. This flow behavior is the toothpaste filling observed experimentally.

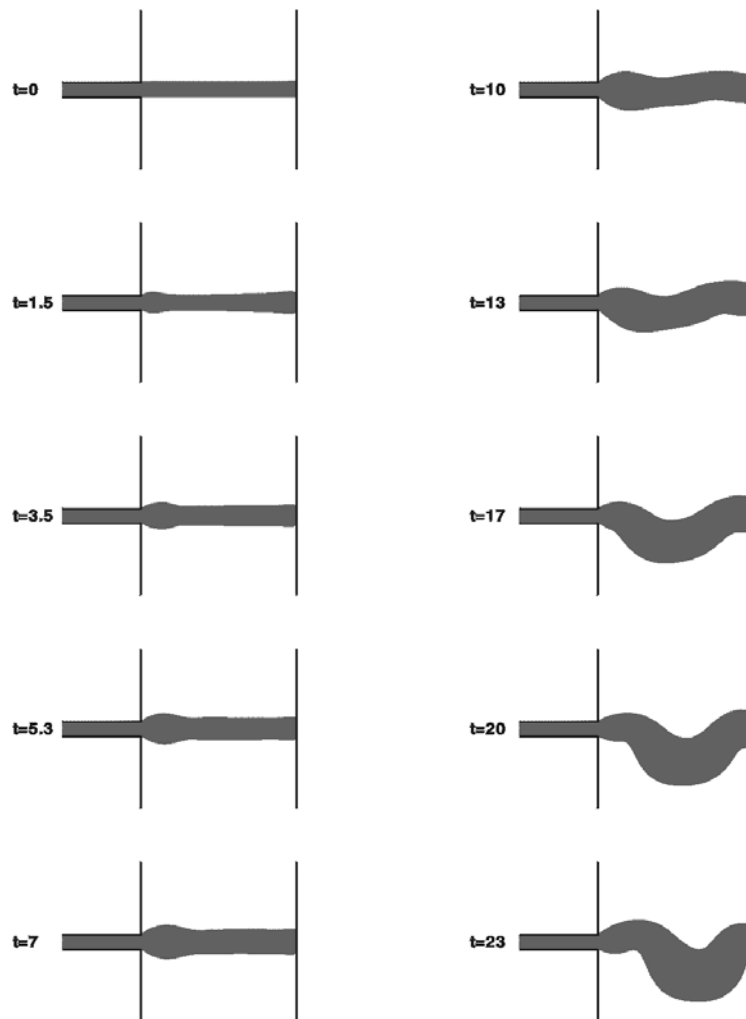


Figure 21: Toothpaste behavior: $Re = 1$, $Bi = 3$ and $L = 10$. The disturbance is imposed from $t=0$ until $t=1.5$.

Figure 22 gives a stability map, which can be used to guide semi-solid processing. Depending on Re and Bi values considered, there are two distinct regions defined as “stable” and “unstable.” It can be seen that a bubble pattern usually leads to unstable jet behavior, whereas shell, disk, mound, and most of transition patterns remain stable. It is quite clear that the instabilities are indeed the results of the finite yield stress and the way yielded and unyielded regions interact with each other. From a processing point of view, the above simulations indicate

that instabilities can be avoided by properly selecting operating conditions from the stability map.

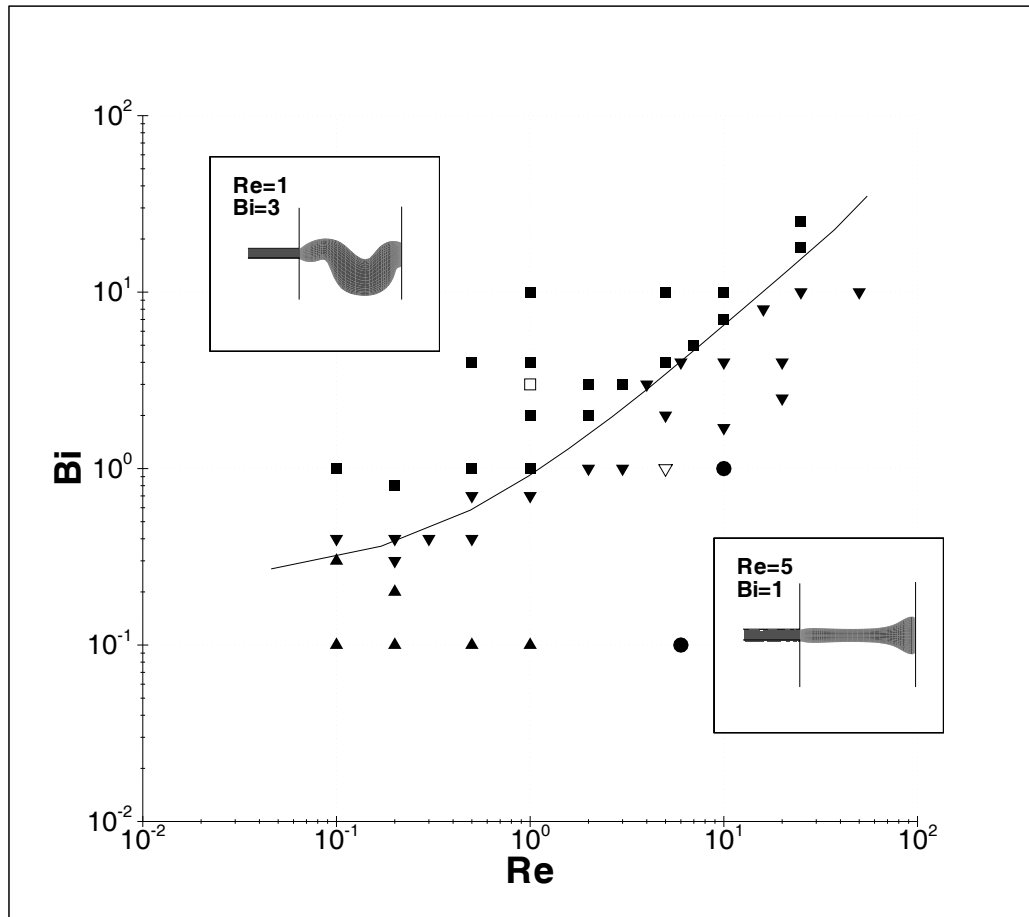


Figure 22: Stability of the jet as a function of Re and Bi . The symbols indicate various flow patterns: ▲ mound pattern; ● disk pattern; ■ bubble pattern; ▼ transition pattern.

A significant challenge encountered in our modeling efforts is the determination of some important rheological constants of semi-solid metals. To address this issue, a “reverse modeling” strategy was used, in which well controlled compression experiments (under both constant shear rate and constant stress conditions) were simulated using “exact” finite-element method. To ensure accurate measurements, a well controlled compression experimental apparatus (see Figure 23) was used. Specifically, a high speed TV camera was utilized to

record the flow behavior of the semi-solid sample during compression through a transparent window. Quantitative data such as shape vs. time, stress vs. time and extension vs. time of semi-solid



Figure 23: Well controlled compression apparatus.

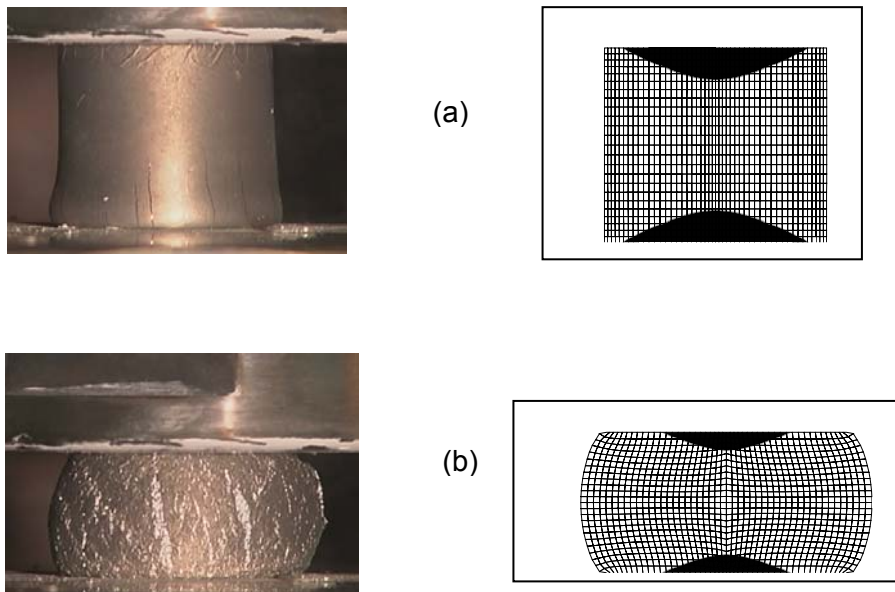


Figure 24: Flow behavior of A356 sample at different shear strains (temperature: 585°C; shear rate: $5.0 \times 10^{-3} \text{ s}^{-1}$) (a) 0, (b) 31.9. The figures on the left are the experimental data and those on the right are the corresponding simulation results.

samples under different compression conditions were determined through an advanced data acquisition system and image analysis. The experimental data and boundary conditions were then used as output and input of the predictive models for reverse modeling. Figure 24 illustrates experimentally observed flow behavior of A356 sample compressed under a constant shear rate ($5.0 \times 10^{-3} \text{s}^{-1}$) in comparison with simulation results. The “reverse modeling” strategy has proven to be reliable to enhance our simulation tools with high degree of accuracy. Some important rheological constants such as the structure breakdown of semi-solid metals were determined.

To support modeling and simulation efforts, we also established a high temperature rheological measurement system, and generated rheological data to input and validate the models developed by the research team. As illustrated in Figure 25, an advanced high temperature rheological measurement system was built here at ACRC. The system consists of a TA rheometer coupled with an optimized vane-cup geometry. Specifically, the optimized vane-cup design circumvents some critical problems encountered in traditional methods such as the wall slip effect etc. Validation experiments with various standard materials indicated that the system can be used to characterize the rheological behavior of semi-solid metals under both steady and transient shear conditions.

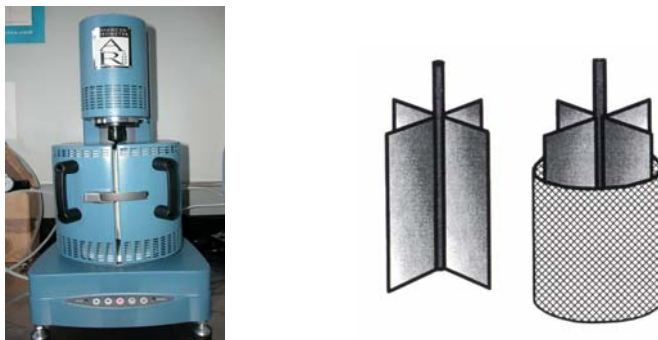


Figure 25: ACRC high temperature rheometer and the optimized vane-in-cup geometry.

Using the new measurement system, we performed systematic rheological measurements with commercial MHD A356 alloys, and generated a vast set of rheological data to enhance our simulation models. Figure 26 shows typical transient steady shear properties of MHD A 356 samples tested in a shear rate range from 4s^{-1} to 357s^{-1} . The experiments were conducted at a constant temperature of 595°C (fraction solid: ~ 0.2) in an argon atmosphere. From the apparent viscosity versus time curve, one can see that the apparent viscosity decreases rapidly

with time within the first 10 seconds of shear, and approaches an equilibrium value after approximately 20 seconds. It is clear that semi-solid metal slurries show time-dependent rheological properties, thus the inclusion of the time effect in constitutive models is critical in modeling semi-solid processing.

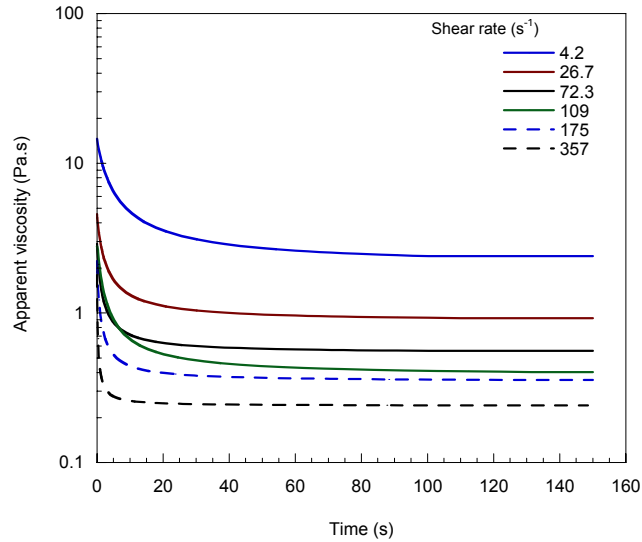


Figure 26: Transient apparent viscosity data of MHD A356 samples (fraction solid: 0.2).

Using a structure kinetic approach, the time dependent properties of the samples tested were determined. The kinetic approach is developed based on an assumption that the change in rheological properties is associated with shear. Through the analysis of experimental data, we found that the rate of structure breakdown of the semi-solid slurry at the solid fraction of 0.2 follows a second order of the structure kinetic model in the shear rate range investigated. Moreover, the rate of structure breakdown increases by two decades when shear rate is increased from 4s^{-1} to 357s^{-1} . Using these valuable experimental data, we have been able to improve the accuracy of our simulation models to a higher level; particularly the capabilities to describe the flow behavior of semi-solid metals during the initial stage of die filling.

In summary, due to their unique rheology, semi-solid metals fill the die cavity in a distinct way that affects the resultant mechanical properties. In this work, we have confirmed the flow patterns and instabilities observed during commercial forming operations by developing appropriate fluid flow models that take into account the yield stress effect. Our modeling results point out that the finite yield stress plays a critical role in determining filling behavior of semi-solid slurries. Flow instabilities that appear as unpredictable events during processing are related to the existence of the finite yield stress, the interplay between inertia, gravity and plastic/viscous resistance to flow, and structure evolution. The stability map developed by the research team is a valuable tool for the optimization of semi-solid processing.

5. ACCOMPLISHMENTS

5.1 Patent:

1. Anacleto M. de Figueredo, Diran Apelian, Matt M. Findon, and Nicholas Saddock, "Alloy Substantially Free of Dendrites and Method of Forming the Same", patent applied for – Docket NO. 1021.2003-000, Sept 22, 2003.

5.2 Publication:

2005

- Q.Y. Pan, L. Wang, D. Apelian and M.M. Makhlouf, "*Optimization of 380 Alloy for Semi-Solid Processing*", NADCA Transactions 2005.
- W. J. Bernard III, Q. Y. Pan, D. Apelian and M.M. Makhlouf, "*The Continuous Rheoconversion Process (CRP): Modeling and Optimization*", NADCA Transactions 2005.
- B. Dewhirst, J.L.Jorstad, D. Apelian, "*Effect of Artificial Aging on Microstructure and Mechanical Properties of Semi-Solid Processed A356 Castings*", NADCA Transactions 2005. (Selected as the Best Paper of the Congress).
- D. Saha, S. Shankar, D. Apelian, M. M. Makhlouf, "*Controlled Diffusion Solidification – Manufacturing Net Shaped Al Based Wrought Alloy Parts*", in *Shape Casting: The John Campbell Symposium*, published by TMS – ISBN # 0-87339-583-2, pp 415-422 (2005).
- D. Saha, S. Shankar, M. Makhlouf and D. Apelian, "*Casting of Aluminum Alloys with a Globular Primary Phase Using Controlled Diffusion Solidification*", submitted to Met and Mat Trans A.
- D. Saha, and D. Apelian, "*On the Dissolution of Al in Al-Si Liquid During the Mixing of Al-25% Si and Al-7% Si Alloys*", submitted to Met and Mat Trans B.
- D. Apelian, D. Saha, "*Novel and Advanced Solidification Processes for the Manufacture of High Integrity Aluminum Cast Components*", in the Proceedings of Second Intl. Light Metals Technology 2005, St. Wolfgang, Austria, published by LKR, pp 203-208.
- J. L. Jorstad, Q. Y. Pan, D. Apelian, "*Solidification Microstructure Affecting Ductility in Semi Solid (SSM) Cast Products*", Mat. Sci & Eng. A (in press).
- J. L. Jorstad, D. Apelian, "*Pressure Assisted Processes for High Integrity Automotive Aluminum Castings – Part 1: Principles and Fundamentals*", submitted to AFS.

- J. L. Jorstad, D. Apelian, *"Pressure Assisted Processes for High Integrity Automotive Castings – Part II: Recent Developments and Innovations"*, submitted to AFS.

2004

- Zachary Brown, Rathindra DasGupta, Dayne Killingsworth, Mark Musser, and Diran Apelian, *"Semi-Solid Metal Casting Practices: Past, Present and Future"*, Proceedings of SAE 2004, Detroit, MI.
- Q.Y. Pan, M. Arsenault, D. Apelian and M.M. Makhlouf, *"SSM Processing of AlB₂ Grain Refined Al-Si Alloys"*, AFS Transactions, Vol. 112, June 2004, pp 04-053.
- M. Findon and D. Apelian, *"The Continuous Rheoconversion Process for Semi-Solid Slurry Production"*, AFS Transactions, Vol. 112, June 2004, pp 04-056.
- D. Saha and D. Apelian, *"Semi Solid Processing of Hypereutectic Alloys"*, AFS Transactions, Vol. 112, June 2004, pp 04-057.
- D. Apelian, Q.Y. Pan, M. Findon *"Low Cost and Energy Efficient Methods for the Manufacture of Semi-Solid (SSM) Feedstock"*, Die Casting Engineer, V. 48, No. 1, January 2004, pp. 22-28.
- Q.Y. Pan, D. Apelian and A.N. Alexandrou, *"Yield Behavior of Commercial Aluminum Alloys In The Semi-Solid State"*, Metallurgical and Materials Transactions (B), Vol. 35B, December 2004, pp 1187-1202.
- Deepak Saha, Sumanth Shankar, Diran Apelian, and Makhlouf M. Makhlouf, *"Casting of Aluminum Based Wrought Alloys using Controlled Diffusion Solidification"*, Metallurgical and Materials Transactions A, Vol. 35A, July 2004, pp. 2174-2180.
- D. Apelian, Q.Y. Pan, M. Findon and M. M. Makhlouf, *"Low Cost and Energy Efficient Methods for the Manufacture of Semi-Solid (SSM) Feedstock"*, in the Proceedings of HTDC (ISBN 88-86259-26-3), Brescia, Italy, published by Edimet, Brescia, Italy 2004, pp. 323-332.
- Deepak Saha, Diran Apelian, and Rathindra Dasgupta, *"Inoculants for the Control of Primary Si Size and Distribution in Hypereutectic Alloys"*, Paper # 8-1 in the Proceedings of the Eighth International Conference on Semi-Solid Processing of Metals and Alloys, Limasol, Cyprus, September 2004; published by NADCA, Wheeling, Illinois.
- Deepak Saha, Diran Apelian, and Rathindra Dasgupta, *"SSM Processing of Hypereutectic Al-Si Alloys - an overview"*, Paper # 22-1 in the Proceedings of the Eighth International Conference on Semi-Solid Processing of Metals and Alloys, Limasol, Cyprus, September 2004; published by NADCA, Wheeling, Illinois.
- Q.Y. Pan, M. Findon and D. Apelian, *"The Continuous Rheoconversion Process (CRP): A Novel SSM Approach"*, Paper # 2-4 in the Proceedings of the Eighth International Conference on Semi-Solid Processing of Metals and Alloys, Limasol, Cyprus, September 2004; published by NADCA, Wheeling, Illinois.

- Q.Y. Pan, D. Apelian and M. M. Makhlof, "*AIB2 Grain Refined Al-Si Alloys: Rheocasting/Thixocasting Applications*", Paper # 13-1 in the Proceedings of the Eighth International Conference on Semi-Solid Processing of Metals and Alloys, Limasol, Cyprus, September 2004; published by NADCA, Wheeling, Illinois.
- A.N. Alexandrou, G.C. Florides, G.C. Georgiou and D. Apelian, "*Rheological Effects of Structure Breakdown in Semisolid Slurries*", Paper # 9-1 in the Proceedings of the Eighth International Conference on Semi-Solid Processing of Metals and Alloys, Limasol, Cyprus, September 2004; published by NADCA, Wheeling, Illinois.
- S. Shankar, D. Saha, D. Apelian, and M.M. Makhlof, "*CDS: Controlled Diffusion Solidification - A Novel Casting Approach*", Paper # 16-2 in the Proceedings of the Eighth International Conference on Semi-Solid Processing of Metals and Alloys, Limasol, Cyprus, September 2004; published by NADCA, Wheeling, Illinois.
- N. Tonmukayakul, Q. Y. Pan, A. N. Alexandrou and D. Apelian, "*Transient Flow Characteristics and Properties of Semi-Solid Aluminum Alloy A356*", Paper # 3-4 in the Proceedings of the Eighth International Conference on Semi-Solid Processing of Metals and Alloys, Limasol, Cyprus, September 2004; published by NADCA, Wheeling, Illinois.
- S. K. Chaudhury, Q. Y. Pan and D. Apelian, "*Response of Fluidized Bed Heat Treatment on Semi-Solid Al Castings on Microstructure and Mechanical Properties*", Paper # 15-4 in the Proceedings of the Eighth International Conference on Semi-Solid Processing of Metals and Alloys, Limasol, Cyprus, September 2004; published by NADCA, Wheeling, Illinois.
- D. Apelian, Q.Y. Pan and M. M. Makhlof, "*Low Cost and Energy Efficient Methods for the Manufacture of Semi-Solid (SSM) Feedstock*", in NADCA Transactions, AFS/NADCA 108th Congress, June 2004, Session 3: Cast Materials, T01-033.

2003

- D. Apelian, A. de Figueredo, M.M. Makhlof "*Energy Efficient Near-net Shape Manufacturing: semi-solid processing routes*", in the Proceedings of The MPMD Fourth Global Innovations Symposium: Energy Efficient Manufacturing Processes, Edited by I. Anderson, T. Marechaux, and C. Cockrill, published by TMS (The Minerals, Metals & Materials Society), 2003, pp 55-62.
- J. L. Jorstad, M. Thieman, R.Kamm M. Lukasson, D. Apelian, R. DasGupta, "*Bringing SSM Casting to the Masses*", Modern Casting, Vol. 93, No. 10, October 2003, pp. 34-36
- Deepak Saha, Rathindra Dasgupta, and Diran Apelian, "*SSM Processing of Al-Si Alloys Utilizing the Concept of Diffusion Solidification*", in the Proceedings of the NADCA Congress, September 15-17, 2003.
- John L. Jorstad, Diran Apelian, and Makhlof M. Makhlof, "*Novel, Slurry-Based, Semi Solid Processing Routes*", In the Proceedings of the Light Metals Technology

Conference 2003, September 18-20, 2003. Brisbane, Australia, Editor: Arne Dahle,
Published by CAST, 2003, pp. 109-114.

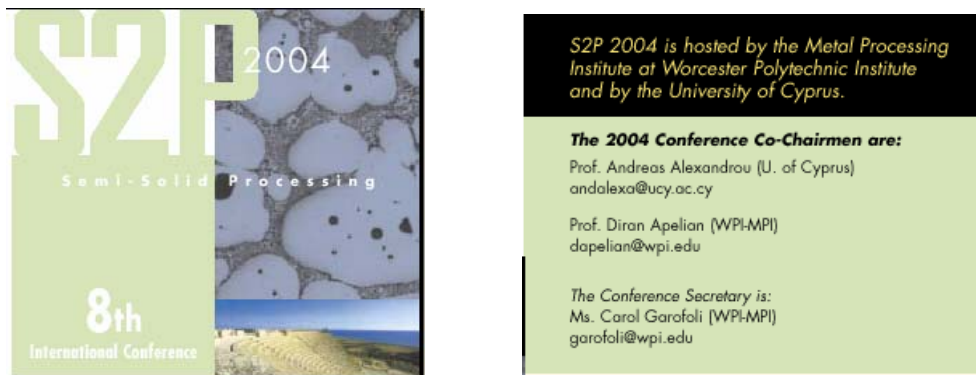
2002

- M. Lukasson, D. Apelian, and R. DasGupta, "*Alloy Characterization for the UBE NRC Process*", in *AFS Transactions* 2002, 02-032, pp.1-14.
- D. Apelian, "*Semi-Solid Processing Routes and Microstructure Evolution*", in the Proceedings of the Seventh International Conference titled *Advanced Semi-Solid Processing of Alloys and Composites*, Tsukuba, Japan, September 24-28, 2002. Published by the Natl. Inst. Of Advanced Industrial Science and Technology, Japan, 2002, pp. 25-30.
- A.N. Alexandrou, P. LeMenn, D. Apelian, G. Georgiou, "*On The Reliability of the Semisolid Metal Process: Effects on the Yield Stress*", in the Proceedings of the Seventh International Conference titled *Advanced Semi-Solid Processing of Alloys and Composites*, Tsukuba, Japan, September 24-28, 2002. Published by the Natl. Inst. Of Advanced Industrial Science and Technology, Japan, 2002, pp. 503-508.
- A.N. Alexandrou, Q. Pan, D. Apelian, G. Georgiou, "*Semisolid Material Characterization Using Computational Rheology*", in the Proceedings of the Seventh International Conference titled *Advanced Semi-Solid Processing of Alloys and Composites*, Tsukuba, Japan, and September 24-28, 2002. Published by the Natl. Inst. Of Advanced Industrial Science and Technology, Japan, 2002, pp. 417-422.
- D. Saha, D. Apelian and R. DasGupta, "*SSM Processing of Hypereutectic Al-Si Alloy Via Diffusion Solidification*" in the Proceedings of the Seventh International Conference titled *Advanced Semi-Solid Processing of Alloys and Composites*, Tsukuba, Japan, and September 24-28, 2002. Published by the Natl. Inst. Of Advanced Industrial Science and Technology, Japan, 2002, pp. 323-328.
- Q. Pan and D. Apelian "*Quantitative Microstructure Characterization of Commercial Semi-Solid Aluminum Alloys*", in the Proceedings of the Seventh International Conference titled *Advanced Semi-Solid Processing of Alloys and Composites*, Tsukuba, Japan, and September 24-28, 2002. Published by the Natl. Inst. Of Advanced Industrial Science and Technology, Japan, 2002, pp. 563-568.
- Q. Pan, D. Apelian and R. DasGupta "*Yield Stress of Commercial Semi-solid Billets: processing effects and the role of microstructure*", in the Proceedings of the Seventh International Conference titled *Advanced Semi-Solid Processing of Alloys and Composites*, Tsukuba, Japan, September 24-28, 2002. Published by the Natl. Inst. Of Advanced Industrial Science and Technology, Japan, 2002, pp. 737-742.
- A.M. de Figueredo, M. Findon, D. Apelian, and M.M. Makhoulf, "*Melt Mixing Approaches for the Formation of Thixotropic Semi-Solid Metal Structures*", in the Proceedings of the

Seventh International Conference titled *Advanced Semi-Solid Processing of Alloys and Composites*, Tsukuba, Japan, and September 24-28, 2002. Published by the Natl. Inst. Of Advanced Industrial Science and Technology, Japan, 2002, pp. 557-562.

5.4 Conference

The 8th International Conference on Semi-Solid Processing of Alloys and Composites was co-hosted successfully by MPI/WPI and the University of Cyprus at Limassol, Cyprus, in September, 2004.



5.5 M.S./Ph.D Thesis

1. Deepak Saha, "Novel Processing Methods and Mechanisms to Control the Cast Microstructure in Al Based Alloys - 390 and Wrought Alloys", Ph.D. Thesis, Worcester Polytechnic Institute, May, 2005.
2. William J. Bernard, III, "The Continuous Rheoconversion Process: Scale-up and Optimization", M.S. Thesis, Worcester Polytechnic Institute, May, 2005.
3. Brian Albert Dewhirst, "Optimization of the Heat Treatment of Semi-Solid Processed A356 Aluminum Alloy", M.S. Thesis, Worcester Polytechnic Institute, July, 2005.
4. Matthew Findon, "Semi-Solid Slurry Formation via Liquid Metal Mixing", M.S. Thesis, Worcester Polytechnic Institute, July, 2003.
5. Nasim Alem, "Novel Methods of Aluminum Grain Refinement", M.S. Thesis, Worcester Polytechnic Institute, February, 2002.

5.6 Book

“Science and Technology of Semi-Solid Metal Processing” Edited by Anacleto de Figueredo, Worcester Polytechnic Institute, published by NADCA, Rosemont, IL 60018-4733.



6. CONCLUSIONS

- The continuous rheoconversion process (CRP) is a novel, energy efficient, and low cost rheocasting process. The process involves the flow of molten metal through a specially designed “reactor” that provides forced convection and copious nucleation, leading to the formation of the desired thixotropic structure. The results with various commercial aluminum and magnesium alloys indicate that the CRP is highly effective for the manufacture of high quality semi-solid feedstock.
- It is envisioned that due to the high energy efficiency, and low cost nature of the CRP process, the process can be easily scaled up and adopted by the die casting industry to make high integrity die castings in the near future.
- SiBloy® SSM is high-quality SSM feedstock that circumvents critical problems encountered in traditional grain refined SSM feedstock, such as the lack of grain size uniformity, the fading of nucleating agents, and the agglomeration and settling of insoluble nucleating particles, etc. Both thixocasting and rheocasting Beta trials show that SiBloy® SSM is an excellent candidate material for the manufacture of cast components with high integrity. Most importantly, due to the exceptional high ductility of SiBloy® SSM castings under T5 condition, the high temperature solution heat treatment can be eliminated, which gives rise to enormous energy savings.
- By developing appropriate fluid flow models that take into account the yield stress effect, we have been able to identify, understand, and describe the flow patterns and instabilities observed during commercial forming operations. Our modeling results point out that the finite yield stress plays a critical role in determining filling behavior of semi-solid slurries. Flow instabilities encountered during commercial operations are related to the existence of the finite yield stress, the interplay between inertia, gravity and plastic/viscous resistance to flow, and structure evolution. Specifically, the stability map developed by the research team provides a valuable tool for the industrial sector to optimize semi-solid process.

7. RECOMMENDATIONS

The research team has completed all planned work in this program. Based on the results of the Tasks pursued, the following recommendations are made:

- 1) Apply the CRP to die cast copper alloys and steels. Although the CRP has been developed solely for Al/Mg alloys, we envision a great potential and a great future to apply the process to other material systems such as copper alloys and steels. Particularly, with the increasing use of Cu and steel, and the needs to make lighter weight, lower cost, and more reliable cast components, the simplicity, and high quality, low cost nature of the CRP will boost and expand the applications of Cu alloys and steels greatly. Accordingly, we suggest that a new research initiative be considered focusing on ferrous and copper alloys.
- 2) Much groundbreaking and pioneering work has been accomplished in developing constitutive models to describe and understand semi-solid processing. Particularly, using rheological data generated from the well designed experiments, we have been able to improve the accuracy of our simulation tools. However, some important issues still remain to be addressed, such as: (1) the effect of wall slip on the filling dynamics; (2) the local time-dependent microstructural changes during rapid shearing; and (3) the origin of the entrapped liquid, and how to quantify its effect on the rheological behavior of semi-solid slurries, etc. In fact, these efforts are being continued in order to incorporate them into our simulation models (in the new program "Innovative Semi-Solid Metal Processing" supported by DOE).

8. REFERENCES

1. D. H. Kirkwood, *Int. Mater. Rev.*, 1994, vol. 39 (5), pp. 173-189.
2. M. C .Flemings, *Metall. Trans.*, 1991, vol. 22B (2), pp. 269-293.
3. Z. Fan, *Int. Mater. Rev.*, 2002, vol. 47 (2), pp. 49-85.
4. D. Apelian, J. Jorstad and A. M. de Figueredo: *Science and Technology of Semi-solid Metal Processing*, NADCA, Rosemont, IL, 2001, pp. 7-1--7-20.
5. J. Jorstad, M. Thieman, and R. Kamm, "SLC[®], the Newest and Most Economical Approach to Semi-Solid Metal (SSM) Casting," *Proceedings of the 7th International Conference on Semi-Solid Processing of Alloys and Composites*, Tsukuba, Japan, pp.701-706 (2002).
6. J. Jorstad, M. Thieman, R. Kamm, "Fundamental Requirements for Slurry Generation in the Sub Liquidus Casting Process & The Economics of SLC[™] Processing", Paper # 07-01, NADCA Transactions, *The 8th S2P International Conference on Semi Solid Processing of Alloys and Composites – Limassol, Cyprus*, (2004)
7. J. Cowie, D. Peters, K. Young, E. Brush, Jr., "Reducing the Cost of Copper Alloy Parts by Semi-Solid Metal Forming", *Die Casting Engineer*, 2004.
8. DOE Report number DE-FC07-98ID13618
9. M.C. Flemings, "Semi-Solid Processing – The Rheocasting Story", Test Tube to Factory Floor: Implementing Technical Innovations, from the Proceedings of the Spring Symposium May 22, 2002 published by Metal Processing Institute, Worcester Polytechnic Institute, Worcester MA 01609 (2003).
10. M. Findon, "Semi-Solid Slurry Formation via Liquid Metal Mixing", M.Sc. Thesis, Worcester Polytechnic Institute, 2003.
11. Q.Y. Pan, M. Findon and D. Apelian, "The Continuous Rheoconversion Process (CRP): A Novel SSM Approach", Paper # 2-4, NADCA Transactions, *The 8th S2P International Conference on Semi Solid Processing of Alloys and Composites – Limassol, Cyprus*, (2004).
12. Q.Y. Pan, M. Arsenault, D. Apelian and M.M. Makhlof, "SSM Processing of AIB₂ Grain Refined Al-Si Alloys", in AFS Transactions, Vol. 112, June 2004, pp. 04-053.
13. Q.Y. Pan, D. Apelian and M. M. Makhlof, "AIB₂ Grain Refined Al-Si Alloys: Rheocasting/Thixocasting Applications", Paper # 13-1 in the Proceedings of the 8th International Conference on Semi-Solid Processing of Metals and Alloys, Limasol, Cyprus, September 2004; published by NADCA, Wheeling, Illinois.
14. A.N. Alexandrou, E. Duc, and V. Entov, *J. non-Newtonian Fluid Mech.*, 2001, vol. 96, pp. 383-403.

15. A.N. Alexandrou, and F. Bardinet, *J. Materials Processing Technology*, 1999, vol. 96, pp. 59-72.
16. Q.Y. Pan, D. Apelian and A.N. Alexandrou, "Yield Behavior of Commercial Aluminum Alloys In The Semi-Solid State", *Metallurgical and Materials Transactions (B)*, Vol. 35B, December 2004, pp 1187-1202.
17. C.J. Paradies and M. Rappaz, Proceedings of the 8th International Conference on Modeling of Casting and Welding Processes, San Diego, June 7-12, 1998, pp. 933-940.
18. S.P. Midson, R.B. Minkler and H.G. Brucher, Proceedings of the 6th International Conference on Semisolid Processing of Alloys and Composites, Turin, Italy, 2000, pp. 67-71.