The Microstructural Characterization of Semi-Solid Slurries

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Due to recent interest in semi-solid metal (SSM) processing, there is a need for fundamental knowledge on the formation and solidification of primary particles. The primary particle size, distribution, morphology, and percentage are the main concerns because particle quantity and size affect not only the mechanical properties of as-cast SSM parts but also the flow characteristics of SSM slurries during die filling. Microstructural characterization is a basic tool for measuring the critical parameters that influence the resulting properties. This article describes the tools and techniques available for structural analysis of SSM slurries. It also attempts to elucidate the ambiguities and interpretations of special structural features.

INTRODUCTION

Conventional solidification of foundry alloys including Al-Si takes place with the dendritic formation of the primary α-Al phase in the eutectic matrix. The alloy composition, temperature gradient within the melt, convection, and rate of heat extraction and the resulting constitutional supercooling are the most effective parameters on the morphology of the primary α-Al phase. Variation of any of these factors during solidification should alter the as-cast structure. For instance, the introduction of agitation (forced convection) into the solidifying melt changes the distribution of chemical composition. This could remove constitutional supercooling and promote dendrite-to-equiaxed transformation (the breakdown and globularization of the α-Al phase). The degeneration of the α-Al phase creates some opportunities that are of commercial interest.

The advantages of semi-solid metal (SSM) processing and available technologies have been discussed in the past by other researchers, including the authors.[1] There is no doubt of the great potential of SSM. However, the lack of industrial interest in the 1980s and 1990s stemmed mainly from the high cost of billet preparation and the issue of recycling the returned and scraped parts. Those problems were solved with the application of new materials and alloying systems as well as the introduction of more cost-effective rheocasting techniques. The techniques involve the preparation of SSM slurry from the liquid phase and directly transferring it into a die or mold for component shaping.

In order to generate a semi-solid structure, the alloy system plays a key role where the co-existence of liquid and solid within a temperature range is the prerequisite for the slurry preparation. The mechanics and mechanisms of the primary particles' evolution (dendrite-to-equiaxed transformation) is the next concern since the formation of globule morphology is expected to enhance die filling and improve mechanical properties of as-cast parts. The ideal microstructure for an SSM slurry is individual fine spherical solid particles uniformly distributed within a liquid matrix. The solid fraction should be considered carefully, since a low-fraction solid may lead to handling and mold-filling problems due to insufficient viscosity and turbulence. High-fraction solids, however, may have die-filling troubles or increase the cost of machinery.

According to the described requirements, characterization of semi-solid material is necessary to confirm, modify, and obtain an optimum structure for the SSM component-shaping process. This knowledge not only gives an idea about the material, but also could lead to better understanding of rheological behavior

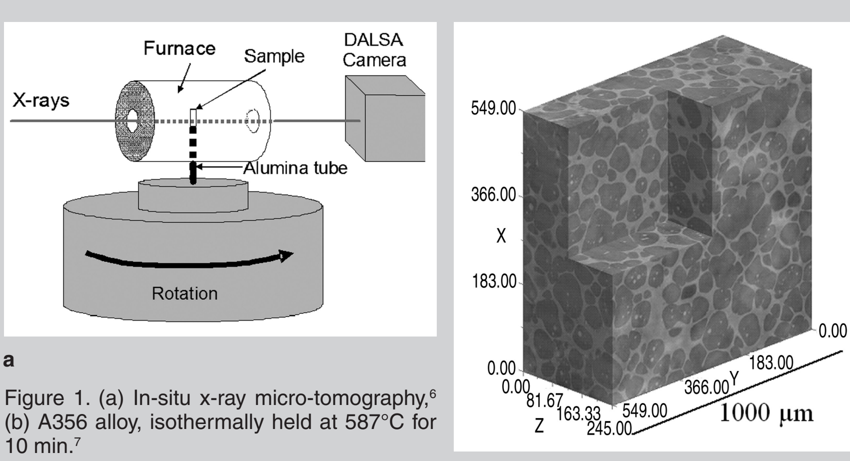

Figure 1. (a) In-situ x-ray micro-tomography,[6] (b) A356 alloy, isothermally held at 587°C for 10 min.[7]

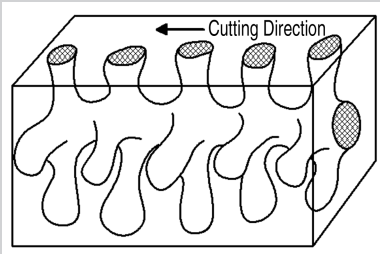

Figure 2. A 3-D view of dendrite-polished surface intersection.

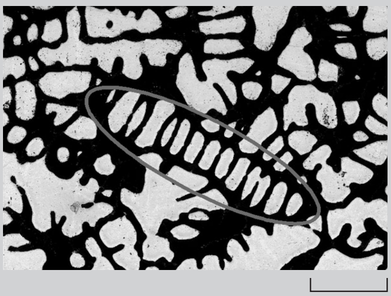

Figure 3. Branches of dendrite, A356 alloy, quenched from 598°C. [*Visit* JOM *on-line and click on the arrow to view the image taken with polarized light microscopy, which reveals that the isolated particles have identical color contrast, and thus should have originated from one single particle.]*

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and eventually improve the mechanical properties of cast pieces.

STRUCTURAL ANALYSIS

In order to be able to accurately characterize SSM microstructure, it is necessary to understand the features and complexity of the resulting solidified structures and be able to differentiate between the observed two-dimensional (2-D) structures and the actual three-dimensional (3-D) morphologies.

Normally after initial visual inspection, the microstructure of the solidified alloy is observed on the plane of polish using optical microscopy. The 2-D analysis may not give a complete picture of the structure and sometimes could lead to invalid conclusions. This is particularly true for cases where, in spite of a well-distributed primary phase in the 2-D state, the inter-connectivity of isolated primary particles from underneath has resulted in misleading conclusions, especially on viscosity.

In the literature, different techniques are described that reveal the true morphological evolution of the primary phase, including reconstruction of 3-D images by serial sectioning, x-ray micro-tomography, and finding the crystallographic orientation relationships. Serial sectioning is based on the successive grinding and polishing of the sample and capturing the consequent images. The major difficulties are calibration of the sectioning distance and also the working frame. These shortcomings are overcome by automatic polishing procedures and drilling guides and controlling holes perpendicular to the polished section. The morphology of primary particles together with their possible interconnectivity are characterized based on the position and shape of each feature along these serial sections. The final 3-D image is constructed with the aid of computer software.[2–5]

Another technique, developed by Suery and his co-researchers, is x-ray micro-tomography (e.g., Reference 6). The system is based on the x-ray beam passing across the sample and consequently capturing the transmitted image by a charge-coupled-device camera. The sample is placed on a high precision rotating table. The final stage is the same as in the serial sectioning method which is retrieving a 3-D image of the sample. In this method, the contrast between the phases is directly related to the atomic number difference between various phases (Figure 1).

The more recent technique is the investigation of the microstructure by electron-backscatter diffraction. This technique is based on the crystallographic analysis of the surface texture, where primary particles with different crystallographic orientations appear in distinct colors. It is a quantitative technique to reveal the grain size, grain boundary, grain orientation and texture, and phase identification.[8]

Structural Complexities in SSM Alloys

Polarized light microscopy and image analysis can be used to render a more reliable characterization of microstructural features in SSM cast parts where certain complexities may generate misleading results. In addition to microscopy-based routes, there are rheological tests as potential production-line quality checks to differentiate between globular and dendritic structures, as explained later in this article.

Dendritic and Non-Dendritic Distinction

Occasionally, solidification conditions may lead to dendritic growth in all or portions of the semi-solid billet. For example, the area most prone to dendritic solidification is near the mold wall. As a result, the final polished microstructure may show dendrites' main trunks and their branches. Also, in some segments, isolated individual globules could be observed which are not real globules. As depicted schematically in Figure 2, the way dendrites intersect the polished surface may generate numbers of pseudo-individual and isolated particles. This is shown in Figure 3 for a metallographically prepared sample.

The conventional bright-field micrograph in Figure 3 confirms the inadequate nature of analysis carried out on 2-D sections. The dendrite secondary branches in this alloy will be treated as individual and isolated particles if processed by an image-analyzing system. This leads to erroneous interpretation of quantitative metallography results and

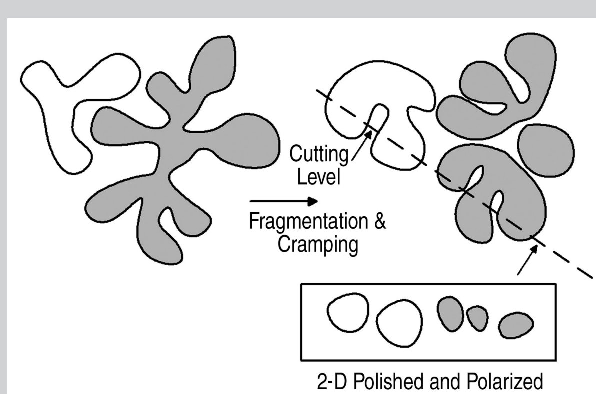


Figure 4. A schematic representation of the transformation of dendrites to cramped ones due to the stirring.[9]

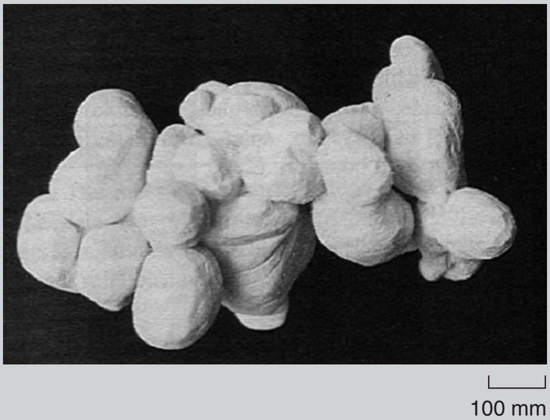


Figure 5. A 3-D reconstruction showing the interconnection of single agglomerate.[5]

compromises parameters attributed to particle size and morphology, such as average circular diameter and sphericity, where a dendritic structure is wrongfully interpreted as globular.

Pseudo-Globule

Primary particles in semi-solid microstructure have complex morphology. The particle that is supposed to be one globule may be interconnected to other globules; the proper nomenclature for such a particle is "pseudo-globule." The pseudo-globule formation is schematically shown in Figure 4. This phenomenon is more prevalent in stir-based processes such as mechanical or electromagnetic stirring routes. By stirring, the resulting forced flow obliges the pre-formed dendrites to break up by mechanical fragmentation[10] or dendrite-root remelting mechanisms.[11] However, if the applied shear force is not sufficient enough to disintegrate the branches, they may plastically bend to form the so-called "cramped" dendrites [*View this article on-line to see an image of the formation of cramped dendrites in A356 alloy quenched at 598°C.*][9] Therefore, it is expected to have different colors for the isolated particles in the former case, which are true isolated particles, while similar color contrast should be expected in the latter case of cramped particles. These particles are not the result of the agglomeration of isolated spherical particles since they have the same color contrast. Therefore, an interpretation based on the bending and intertwining of dendrites could be a valid conclusion.[9] The only justification for such isolated particles is due to the metallographic 2-D sectioning. Pseudo-globules play a significant role in the rheological properties of SSM slurries and lead to a rise in the viscosity value.

Breaking and fragmentation of primary dendrites by mechanical stirring are the original routes for semi-solid processing. However, in such structures, there are large primary agglomerations that have very complex shapes. These groups of primary particles seem to have formed due to the agglomeration of small and individual particles, which is not an accurate assumption. The investigation of agglomerated particles goes back to the work of Ito et al.[5] on particles formed during mechanical stirring of Al-6.5%Si. The obtained slurry was sheared at a rate of 900 s^{-1} for 2 h at 0.2 fraction solid and then quenched. The quenched sample was polished and examined in successive sections 10–20 μm apart. Reconstruction of the 3-D model from such serial sections confirmed that particles that appear to be isolated are actually interconnected to others. Figure 5 is a 3-D reconstruction of this microstructure showing that the bulk of the particles in the region studied are interconnected to form a single agglomerate.

In another study, Niroumand et al.,[2] by working on the mechanical stirring of Al-10.25%Cu and using serial sectioning, proved the microstructure consists of pseudo-particles and clusters in a 2-D view. Many researchers interpreted this phenomenon as a process of agglomeration of small globule particles but they believed most of the particles in 2-D sections were interconnected in 3-D and that they were single primary grains. Figure 6 shows a pseudo-cluster and resulting serial sectioning of the sample, indicating the structure's complexity.

Sintering and Coalescence

The sintering and coalescence of primary particles are the other mecha-

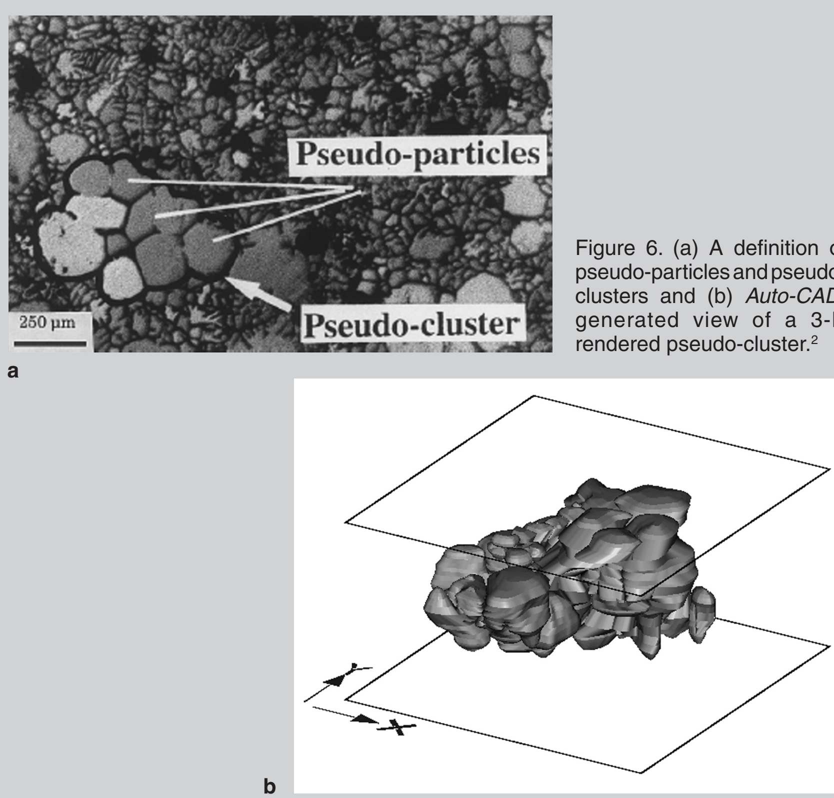


Figure 6. (a) A definition of pseudo-particles and pseudo-clusters and (b) *Auto-CAD*-generated view of a 3-D rendered pseudo-cluster.[2]

100 μm

Figure 7. The sintering of globules in electromagnetic stirred Al7Si alloy and isothermally held at 583°C for 10 min. (the arrows are welded joints due to sintering). [*View this article on-line to see the figure with the application of polarized light microscopy.*]

nisms active during SSM processes. It is envisaged during stirring that the solid particles collide with each other to form welded joints which are eventually strengthened due to high temperature and easy diffusion. An example of sintering in SSM materials is shown in Figure 7. The figure shows that two or more isolated solid particles joined and formed a pseudo-cluster which is associated, for instance, with stabilized contact during stirring. The sintering mechanism has been attributed to grain boundary and solid-liquid phase boundary energies.[12]

This effect was seen in regions with low stirring or stagnant liquid and solid. Different globules present different primary α-Al particles with different orientations. Thus, sintering of individual primary particles could be easily detected by different color contrasts. In semi-solid science, this is called agglomeration,[13] where primary particles, basically consisting of simple, isolated globular or rosette shape, come into contact and sinter together to form agglomeration.

Quantitative Metallography

The application of polarized light microscopy enables the investigator to identify SSM structures as fully isolated true globular or pseudo-globular morphologies including dendritic, degenerated dendritic, and rosette structures, depending on the color contrast, as mentioned and illustrated previously. Once the distinction is made, image processing and analysis may be carried out to characterize the structure quantitatively.

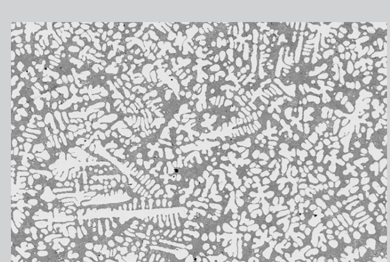
According to the authors' experience, the major task during image analysis is the preparatory work done before processing and analyzing of the image. Superior sample preparation, including polishing and etching, coupled with appropriate microscope selection and even working magnification, provides reliable resulting analyses. For image analysis, the following are of particular importance: image acquisition, image enhancement, thresholding, image processing, and data extraction.

Semi-solid metal processing is based on the formation of globules and, as such, the principal objectives are to quantify the volume fraction, size, and morphology of primary particles. For morphology, the aspect ratio and sphericity numbers are proposed to be the most important factors to characterize particle shape while for size analysis, average circular diameter is suggested.

Fraction Solid

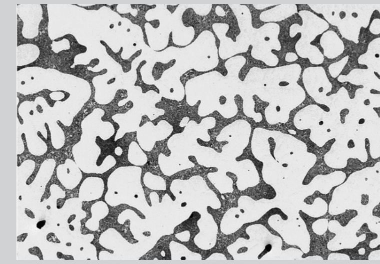
Fraction solid is determined by different methods such as the lever rule (thermodynamic equilibrium condition), Scheil's equation (no diffusion in the solid),[9] thermal analysis,[14] or quantitative microscopy. It is important to comprehend the shortcomings of each route when comparing the results calculated/measured by any of the mentioned techniques. The lever rule, Scheil, and thermal analysis routes may give results greatly different from that of quantitative microscopy.

For example, in the case of the Al7Si alloy, quenching from 593°C leads to a solid fraction of 59±2% (Figure 8), in



800 μm

Figure 8. A quenched Al7Si billet from mushy zone (593°C), conventionally cast, primary solid ~59%.



400 μm

Figure 9. The results from isothermal holding of A356 alloy for 10 min. at 583°C, cast in a graphite mold.

contrast to 26% calculated from thermal analysis, Scheil, and lever rule.[15] The large discrepancy may have its origin in the following sources:

- Inefficient quenching method; the formation of eutectic colonies confirms the ineffectiveness of water quenching in preventing further liquid → solid transformation from the quenching temperature. This could be due to vapor formation around the billet, which prevents effective quenching.
- Growth of the primary particles is another possibility for inaccuracy in the measurement. During the transferring and quenching period, the residual liquid may also precipitate on the pre-quench primary solid phase. Martinez et al.,[16] by working on Al-4.5%Cu and by measuring the thickness of the darkened periphery of the etched grains, have shown that a primary spherical globule with 40 μm diameter grows almost 40 μm in diameter during the quenching period.
- Depressing the eutectic line to lower temperatures due to the quenching.

Fraction of Entrapped Liquid

Entrapped liquid is a distinct feature of the thixocasting route. The thixo-route is basically a three-step process involving preparation of a feedstock material having an equiaxed or globular/non-dendritic structure or, indeed, having the potential to transform into an equiaxed structure on further processing. The second step is reheating of the feedstock material to temperatures between solidus

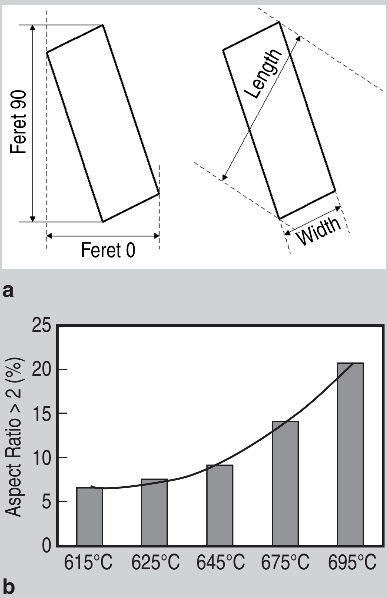


Figure 10. (a) Definitions of feret and aspect ratio and (b) percentage of aspect ratio greater than 2.[18]

and liquidus (mushy zone) to generate a semi-solid structure. The final step is shaping of the mush that has thixotropic characteristics. During reheating stage and as a result of grain coarsening, parts of the liquid become entrapped inside the solid grain (Figure 9). The entrapped liquid adversely affects the deformability of material, which is associated with a reduction in the interconnected liquid phase and therefore influences the rheological behavior of the slurry.

The volume of entrapped liquid depends on various parameters including cooling rate and morphology of as-cast structure, reheating time, and temperature. In fact, contrary to its name, sometimes these pools which seem to be encapsulated in the 2-D surface may be interconnected in the volume.

Particle Size

The ideal microstructure for SSM processing is free from dendrites and has homogenous spherical particles. The globule size plays an important role in the castability and mechanical properties of the as-cast parts. The optimum primary particle size reported for SSM alloys is less than 100 μm.[17]

As shown in Figure 2, it is critically important to comprehend the disability of image analysis in distinguishing between the dendrite branches and individual globules. Therefore, in the case of dendritic structure, calculation will result in a lower value for a circular diameter. Furthermore, since the measurement is based on the area of individual particles being examined and the assumption that they have a shape close to a circle, the more rectangular the particles, the greater would be the error in the calculation.

Sphericity/Shape Factor

The shape of primary particles is a key parameter to characterize the flow and thus the viscosity of the semi-solid slurry. Rounder particles have better flowability in comparison to rectangular ones. Sphericity is given by $4\pi A/P^2$ where A is the total area of primary particles and P represents the perimeter of the liquid-primary particles interface. The sphericity factor varies between 0 for objects having a very elongated morphology and 1 for those having a circular cross section. It is also important to point out the possibility of erroneous results if isolated secondary branches, purely due to metallographic section (Figure 2), are considered true isolated particles. As a result, the sphericity number will increase.

Aspect Ratio

Another complimentary parameter is aspect ratio, which is simply defined as the ratio of the longest over the shortest feret diameters (length per width). Feret diameter is defined as the distance between two parallel tangents on each side of an object (Figure 10a). Values near 1 point to spherical particles while higher values show more needle like particles. Figure 10b provides aspect ratios of 356 Al-Si alloy slurry prepared at various pouring temperatures. To overstress the effect of pouring temperature, the percentage of aspect ratio greater than 2 is presented. As shown, by increasing the pouring temperature, the formation of elongated particles is promoted, which is an indication of dendritic structure.

RHEOLOGICAL ANALYSIS

Since the inception of semi-solid metal processing in the early 1970s,[19] the issue of rheology has been addressed briefly, although there have been specific sessions in the “S2P” biannual international conferences within the last 16 years (1990–2004). Rheology is an integral part of SSM research efforts, even though the concept may not be so clear or the expression familiar for metallurgists.

Rheology deals with the simultaneous deformation and flow of materials. Shear flow is an important type of deformation in rheology and may be visualized as a process in which infinitely thin, parallel planes slide over each other as in a pack of rigid cards. With such a simple definition, the inter-relationship between

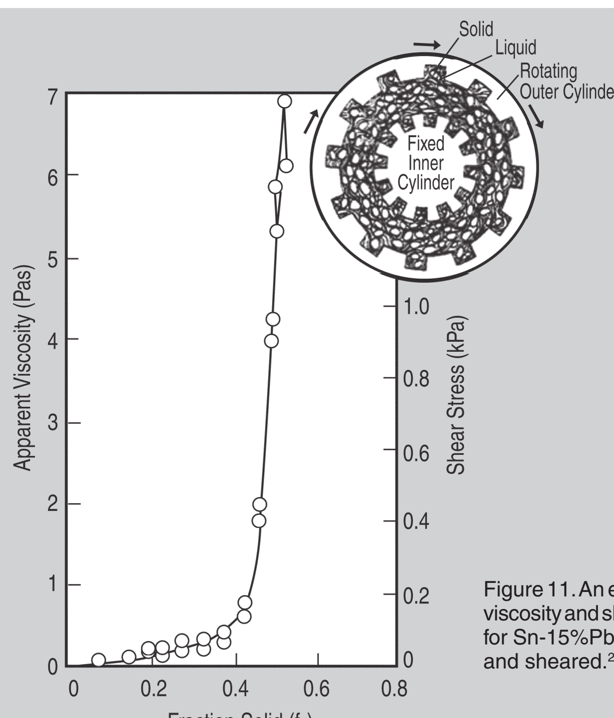


Figure 11. An experimentally determined viscosity and shear stress vs. fraction solid for Sn-15%Pb alloy continuously cooled and sheared.[23]

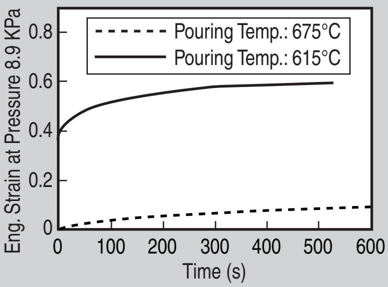


Figure 12. Strain-time graphs for billets with different structures as shown in Figure 13.

Table I. Viscosity Numbers (Log η), Calculated from Strain-Time Distribution Graphs Obtained for Billets Cast at Different Pouring Temperatures[24]

Applied Pressure	Log η (695°C)	Log η (675°C)	Log η (645°C)	Log η (630°C)	Log η (615°C)
Structure	Dendritic	Mainly Dendritic	Rosette	Rosette and Globular	Globular
8.9 kPa	9	8.6	7.3	6.6	6.3

rheology and the mechanical properties of materials is closely tied up with materials' viscosity and deformation behavior within the mushy state. According to several recent review articles, viscometery is identified as an appropriate route for the rheological studies of SSM slurries.[14,20,21]

Based on Newton's first law, viscosity is a constant to show the capability of momentum diffusion through the body of material as expressed mathematically in the following equation:

$$\tau_{yx} = -\eta \frac{dv_x}{dy} = \eta \mathring{\gamma}$$

where dv_x/dy is the velocity gradient, τ is shear stress, η is viscosity, and $\mathring{\gamma}$ is shear rate. The value of η is constant for Newtonian fluids, but interpreted in terms of the power law, relating shear force (τ) to average shear rate, $\tau = m(\mathring{\gamma})^n$, for non-Newtonian fluids. The apparent viscosity is then calculated as the ratio of shear stress to shear rate, $\eta = m(\mathring{\gamma})^{(n-1)}$, where m and n are material constants of consistency and power law index, respectively.[22]

Viscosity is the main parameter to study the rheology of semi-solid metallic alloys. It is an indication of semi-solid metal capability in filling the mold and determines the required force for deformation and flow of materials. During conventional solidification, viscosity rises up steadily with increasing solid fraction until the point where the solid can no longer move freely and the already solidified segment tends to develop strength. At this point, which is defined as the transition from mass feeding to interdendritic feeding during solidification and termed dendrite coherency point (DCP), viscosity increases abruptly.

In SSM processing, the DCP is postponed due to the forced convection or shallow temperature gradient within the melt. The breakdown of dendrites due to stirring coupled with multi-directional growth of fragmented dendrites due to more uniform temperature distribution within the mold (i.e., shallow temperature gradient) resulting from forced convection, encourages the formation of equiaxed grains, thus postponing the rapid rise of viscosity to a higher fraction of solids as reported by Spencer et al.[23] (Figure 11) in their pioneering work on the rheology of Sn-15%Pb alloy during the early 1970s.

The viscosity of SSM slurries is dependent on the metallurgical parameters including the fraction solid, and its morphology such as dendritic or globular, solid particle size and distribution, and pouring temperature.[24] Pouring temperature is one of the most important parameters to influence the evolution of primary particles during semi-solid casting. The importance of melt pouring temperature (superheat) in achieving desirable semi-solid aluminum alloy microstructure has been reported previously (e.g., References 25 and 26).

There are several test procedures to measure/calculate the viscosity of slurries, but the procedures can be divided into two main categories depending on the fraction solid:[24]

- Direct methods of "rotational viscometery," used for low-fraction solids up to 0.4, where the induced torque in the slurries is an indication of viscosity. Couette and Searle-type viscometers are examples of this method where the apparent viscosity is calculated by a set of equations using the measured torque data (e.g. Reference 23).
- Indirect routes, used for high-fraction solids in excess of 0.4–0.5, where the viscosity is calculated from mechanical tests, such as the parallel plate compression test (e.g. Reference 24), direct or indirect extrusion,[27] and indentation and tensile tests.[28]

One way to examine the rheological behavior of paste-like materials is by the parallel plate compression test.[29] In this method, a dead weight is applied on the top surface of an SSM billet and its deformation behavior is studied by analyzing the engineering strain variation with time. The resulting strain-time graph is further treated mathematically to determine the viscosity as an indication of the rheological behavior of the tested alloy. The mathematical treatments vary depending on the assumption of the SSM slurries behaving as Newtonian or non-Newtonian fluids.[24]

The resulting strain-time graphs from the parallel plate test vary with the morphology of the primary phase, as shown in Figure 12. If the graphs in Figure 12 are considered in conjunction with the respective optical micrographs in Figure 13, it is seen that the greatest engineer-

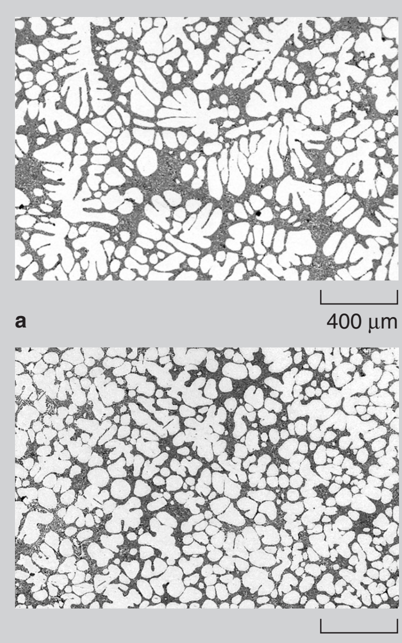


Figure 13. A typical microstructure formed at different pouring temperatures: (a) 675°C-dendritic, (b) 615°C-globular.[24]

ing strain is obtained for the billets with a globular structure (615°C). In contrast to globular structure, alloys with a dendritic primary phase (675°C) exhibit higher resistance to deformation and flow. In other words, the parallel plate test is an effective tool to differentiate between the structures of SSM slurries as being globular, rosette, or dendritic in nature.

This is an important quality issue in slurry-on-demand cast houses where the production of rheocast billets with fully globular structure is the main objective. This is because the globular structure has superior flow and mold-filling capability. The viscosity values calculated from these graphs confirm the lowest value for the globular morphology, as shown in Table I.[24]

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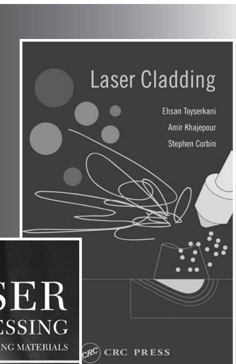

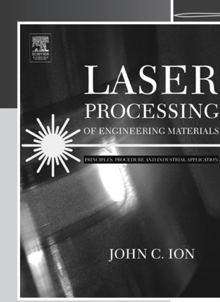

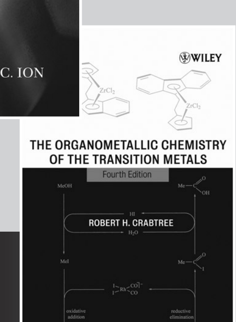

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