

Semi-Solid Processing of Metallic Materials

Prof Helen Atkinson FEng

Department of Engineering, University of Leicester, University Rd., Leicester, LE1 7RH,
UK., hva2@le.ac.uk

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Department of Engineering, University of Leicester, University Rd., Leicester, LE1 7RH,
UK., hva2@le.ac.uk

Abstract. Semi-solid processing involves forming metallic alloys between the solidus and the liquidus. For the process to operate, the microstructure must consist of solid spheroids in the liquid matrix, rather than dendrites. The material then flows when it is sheared but thickens again when allowed to stand, i.e. it behaves thixotropically. This type of behaviour was first discovered by Flemings and co-workers in the 1970s and is utilized in a family of processes, some now applied commercially. Here the current status of semi-solid processing, both technologically and from a research point of view, will be reviewed.

Keywords: semi-solid processing; thixoforming; rheoforming; thixocasting; thixoforging.

1 Introduction

In the early 1970s, a PhD student at MIT in the US was studying hot tearing of casting alloys by using a rheometer to measure the viscosity as he cooled into the semi-solid state. He found that if he was stirring the material continuously during cooling it showed less resistance to shear than if the material was cooled into the semi-solid state and then stirred.¹ The student and his supervisor identified that the microstructure of the continuously stirred materials was spheroidal (i.e. consisted of spheroids of solid in the liquid matrix), whereas the material which was cooled into the semi-solid state without stirring was dendritic (Fig. 1). The material with the spheroidal microstructure in the semi-solid state was *thixotropic* i.e. when it is sheared it thins and flows, when it is allowed to stand it thickens again.² This property can be exploited in semi-solid processing where the material is forced to fill a die. In comparison with die casting, where the flow is turbulent, here the flow is smooth and laminar and hence the resulting mechanical properties are enhanced. Consequently, parts can be made with thinner sections and hence lighter weight. As the material does not have to cool all the way from the liquid state there is less solidification shrinkage and therefore parts can be made closer to net shape. The lower temperatures than die casting mean less die attrition. In addition, parts can be heat treated and the surface finish is improved. In comparison with forging, complex shapes can be made in one shot, including undercuts, and less force is needed, hence the machinery can be on a smaller scale.

The discovery of the thixotropic behaviour of semi-solid metallic alloys has led to the development of a family of processing routes. Initially, a spheroidal microstructure

has to be obtained. Sometimes this is a prior step (as in *thixoforming*, *thixoforging* and *thixocasting*) and sometimes it is integral to the forming process itself (as in *rheocasting*, *thixomolding* and *rheodiecasting*).

In Fig. 2, one route to the spheroidal microstructure is shown as stirring. There are, however, a range of potential feedstock production routes and these will be discussed further in section 2. After stirring, the material can be solidified and cut into billet. It is then reheated. If the reheated billet is placed into the shot chamber of a die casting machine and forced into the die, this is termed *thixocasting* and usually the fraction of liquid is around 60% or more. If the semi-solid billet is placed between closed dies then it is *thixoforging* and the fraction solid will be quite high (around 60% or more). *Thixoforming*, as a term, is sometimes used to cover both *thixocasting* and *thixoforging*, or to cover a hybrid process where the billet is placed on a ram (rather than in a shot sleeve) and forced into a die. In the diagram, 'deformed billet' means that the route to a spheroidal microstructure involves deformation in the solid state. On reheating into the semi-solid state, the material recrystallises and liquid is formed around the new grains, which are fine and equiaxed. In *rheocasting*, (and in the related routes *rheodiecasting* and *thixomolding*®), the feedstock is not solidified after stirring but rather taken straight from the semi-solid state into the die casting machine.

In section 2, feedstock production routes will be discussed in more detail and in section 3 the semi-solid processing routes. Section 4 will cover industrial applications, section 5 rheology of semi-solid material, section 6 modelling and section 7 the current status with respect to which alloys can be semi-solid processed. The field has been reviewed⁴⁻⁹ and two books have been published recently.^{10,11}

2 Routes to Spheroidal Feedstock

2.1 Mechanisms of Spheroid Formation

From the liquid state, spheroids can form by dendrite fragmentation with the arms breaking off at their roots due to shear forces, or the dendrite arms melting at their roots, or dendrite arm bending causing dislocation generation followed by the formation of grain boundaries and grain boundary wetting.⁵ Alternatively, the conditions for dendrite formation may not be met. For example, constitutive supercooling will not occur if liquid is stirred to homogenise solute concentrations or if the material is held isothermally. The solid state routes to spheroidal feedstock are essentially based around the fact that deformed material will recrystallise as it is heated and the liquid will tend to form at the recrystallised boundaries.⁶

2.2 Liquid Metal Routes

Liquid metal routes can be divided into those which involve agitation and those which do not. Semi-solid material can be vigorously stirred with a mechanical stirrer but this tends to lead to unacceptable erosion of the stirrer and contamination. In the shear cooling roll technique,¹² liquid metal is poured between a rotating roll and a stationary cooling shoe. Passive stirring involves the liquid being forced through a static mixing device (such as a column containing glass spheres) where the geometry

ensures high mixing without turbulence.¹³⁻¹⁶ Sprayforming is the atomisation of melt into a spray followed by collection of droplets on a substrate. This gives a very fine spheroidal microstructure when remelted and allows alloys which cannot be made by other routes to be fabricated.¹⁷ However, it is relatively expensive. The 'new MIT process' (Fig. 3) is a hybrid of stirring and near liquidus casting.¹⁸ The melt is held just above the liquidus and a cooling finger, which also acts as a stirrer, is inserted. The melt is cooled to just below the liquidus and the finger withdrawn. This generates a cloud of nuclei. This process is very interesting from a commercial point of view. The most important agitation route to date industrially has been magnetohydrodynamic stirring (MHD).⁴ High local shear is generated by rotating, linear or helicoidal electromagnetic fields (thereby avoiding contamination, gas entrapment and stirrer erosion), with stirring deep in the sump of the liquid. The alloy is filtered and degassed before treatment and a fine spheroidal size of about 30µm results. There is some lack of uniformity and spheroids may retain some 'rosette' character, a factor which influences flow. The majority of material which has been thixoformed commercially in the last twenty years has been produced by the MHD route.

There have been significant new developments more recently, including the SEED (Swirled Enthalpy Equilibration Device) by Alcan. This involves, firstly, the rapid extraction of a controlled quantity of heat from the molten metal charge in order to generate a semi-solid slurry and, then, draining away the excess liquid leaving behind a compact self-supporting slug which can then be handled and formed under pressure.

The 'new rheocasting (NRC) process' patented by UBE,^{19,20} is an example of a route which does not involve agitation. Molten metal at near-liquidus temperature is poured into a tilted crucible. Grain nucleation occurs on the side of the crucible and the grain size is fine because the temperature is near-liquidus. To date this has been the most important of the rheocasting-type routes from a commercial point of view.

Other non-agitation routes include the Direct Thermal Method (DTM),²¹ where liquid alloy with a low superheat is poured into a cylindrical metallic mould of very low thermal mass but high thermal conductivity. Heat matching between the alloy and the mould results in a pseudo-isothermal hold within the solidification range. This is a low cost method ideal for the laboratory environment but with a strong limitation on the size of the billet that can be produced.

In the Cooling Slope method,^{e.g. 22} liquid metal is poured down a cooled slope and collects in a mould. Nucleation on the slope ensures the spheroid size is fine. This shows considerable potential for combination with rolling (Fig. 4) but there are issues of gas entrapment and oxide formation on the slope.

A number of manufacturers have aimed to use grain refinement to suppress dendrite growth e.g. titanium diboride in aluminium alloys. It has proved difficult to obtain uniform grain sizes less than 100µm and the volume of liquid entrapped in spheroids tends to be high (entrapped liquid does not contribute to flow).

A new route has recently been developed which is gaining considerable commercial interest.²⁴ It involves bringing two precursor alloys of precisely controlled composition, temperature and quantity into intimate contact and then casting the resultant alloy using a conventional casting process. This yields a component of a predetermined composition with a spheroidal microstructure.

2.3 Solid State Routes

The main solid state routes are Strain induced Melt Activated (SIMA)²⁵ and (RAP),²⁶ both of which have been used commercially. These are compared in Fig. 5. Essentially, as stated earlier, the material is deformed for example by extrusion and then reheated into the semi-solid state. Recrystallisation occurs and the liquid forms around the grains which are fine and equiaxed. The distinction between SIMA and RAP is that the former involves hot working (above the recrystallisation temperature) and the latter warm working. SIMA, therefore, requires an intermediate additional cold work step. RAP is, however, more limited in the billet size because it is difficult to introduce deformation uniformly across the section (and for successful thixoforming a uniform spheroidal microstructure is required). The advantages of these routes are that many alloys are supplied in the extruded (or rolled) state anyway and the spheroids tend to be more rounded, giving better flow than the 'rosette-type' structures sometimes found, for example, with the MHD route.

It has become clear that some materials are readily thixoformable when reheated from the state which they are usually supplied in e.g. high performance steels.²⁷

3. Semi-Solid Processing Routes

3.1 Rheocasting

In rheocasting, alloy is cooled into the semisolid state and injected into a die without intermediate solidification. The non-dendritic microstructure can be obtained by (see section 2): mechanical stirring; stimulated nucleation of solid particles (as in the NRC process); electromagnetic stirring in the shot sleeve (the Hitachi process²⁸); or by sub-liquidus casting (the THT process).²⁹ The NRC process is shown in figure 6. The inversion of the billet causes the oxide skin to run into the biscuit and runner. With the NRC process, there is no need for specially treated thixoformable feedstock and scrap can be readily recycled within the plant. However, there is a lack of flexibility; the crucible size and heating/cooling arrangements are specific to that volume of metal.

In sub-liquidus casting (Fig. 7),²⁹ no processing equipment other than a die casting machine with a large shot diameter and a short stroke is required. The product emerges free of gates so no trimming is needed. The melt is chemical grain refined.

3.2. Rheomoulding

Rheomoulding is allied to polymer injection moulding. It employs either a single screw^{30,31} or a twin screw.^{32,33} Liquid metal is fed into a barrel where it is cooled while being mechanically stirred by a rotating screw. The semisolid is then injected into a die cavity. This allows the continuous production of large quantities of components and no specially produced feedstock material is needed. Workers at Brunel University are in commercial trials of what they call the Rheodiecasting

(RDC) process³⁴ (Fig. 8) having found a solution to the challenge of what material to use for the screw and the barrel to avoid interaction with aluminium alloys.

3.3 Thixomoulding

Thixomolding[®] is licensed by a firm called Thixomat. Many firms in Japan, the US and the Far East use the process to produce components from magnesium alloys. Magnesium alloy pellets are fed into a continuously rotating screw. The process is therefore similar to rheodiecasting but the starting material is solid pellets rather than liquid. The energy generated by shearing contributes to heating the pellets into the semisolid state (but some additional heating is applied). The screw action produces the spheroidal microstructure and the material is fed into a die. One of the advantages of the process is that it allows forming with a range of solid fractions from 5% to 60% (although typically it tends to be relatively low).

3.4 Thixoprocesses

In thixocasting, the material is solid initially and the alloy has been treated in such a way that when it is reheated into the semisolid state a spheroidal microstructure is obtained. The liquid content is relatively high (>50vol.%). Reheating can be in a radiant furnace or an induction heating furnace. The high liquid fractions mean that the billet tends to collapse and is therefore tipped into the shot sleeve from a container. This process has been used commercially for making fuel rails for cars.

Thixoforging^{35,36} involves heating the feedstock into the semisolid state and placing it between die halves. The parts of the die are then brought together. Material is used efficiently because of the lack of runners, gates and press discard. The liquid volume fraction is generally less than 50 vol.% so that the billet can be handled. The process has not been used commercially, perhaps because of the difficulty in obtaining repeatability.

In thixoforging, ^{e.g.} ¹⁷ the billet is heated as a vertical cylinder (in contrast with thixocasting) and then injected into the die, either horizontally or vertically. The liquid content is between 30 and 50 vol.%. A range of automotive and other components have been produced by this route in industry. Billets are heated on a carousel with induction. Process control is demanding but cycle times are very comparable with those in die casting, and indeed may be faster because full solidification is not required.

4 Industrial Practice and Applications

4.1 Process Issues

For the rheocasting routes heating of the feedstock billet is not required. However, for the thixoprocesses the general method is to use induction heating in a carousel arrangement so that a billet is always ready for processing. The induction heating sequence may be step-wise in order to obtain as uniform a temperature profile across

the billet as possible (given that induction heating works through the skin effect). Process control is demanding because the spheroidal microstructure must be as uniform as possible across the cross-section with a consistent liquid/solid fraction. The viscous slug must retain its shape until the shear force is applied. Experience shows that it is difficult to obtain satisfactory thixoforming unless the equipment has real-time and closed loop control. For example, feedback must occur so that, as the ram makes its stroke, the velocity is constantly monitored and controlled to given values. Otherwise, as the ram forces material into the die and encounters increasing resistance, it will slow down. In practice, successful semi-solid processing is based around optimised profiles for pressure and velocity versus time.

4.2 Examples of Commercial Components

Components made by thixoforming because of the near net shape requirement include: computer heat sinks (see figure 9); industrial bolt connectors; racing bike swing arms; computer disk drive motor bases and mountain bike suspension parts. For other parts, it is the capability to achieve enhanced mechanical properties which is significant eg. motorcycle chassis frame arms and mountain bike forks. In some cases, thixoforming can produce a component which is pressure tight where die casting cannot, for example, intricately shaped gas control valves. One of the problems with die casting is the incorporation of oxide films because of the turbulent flow; this does not occur with semi-solid processing where the flow into the die is smooth and laminar. Other examples of leak-tight, pressurized applications include: master brake cylinders; ABS system valves; missile connector supports and airbag canisters. All these are made from aluminium alloys, usually A356 or A357 in the heat treated condition.

Many components for mobile phones, laptops and automotive applications are made from magnesium alloys by thixomoulding (Figs. 10 and 11).

5 Experimental Determination of the Parameters for Semi-Solid Processing

5.1 Background Rheology

Fig. 12 shows different potential types of fluid behavior. In a Newtonian fluid, the shear stress is proportional to the shear rate, and the constant of proportionality is the viscosity η . Thixotropic fluids are non-Newtonian i.e. the shear stress is not proportional to the shear rate. The viscosity is then termed the apparent viscosity and is dependent on shear rate, pressure, temperature and time. Some non-linear fluids also show viscoelasticity i.e. they store some of the mechanical energy as elastic energy. Thixotropic materials are generally thought not to store energy elastically and show no elastic recovery when the stress is removed. However, Favier and Atkinson³⁷ have recently shown that to accurately model the initial stages of the rapid compression response of thixotropic billets, an assumption of elastic behavior by the interconnected solid skeleton prior to deformation is required.

If a fluid exhibits a yield stress and then gives a linear relationship between shear stress and shear rate, it is termed a Bingham material. The Herschel-Bulkley model is where behaviour is non-linear after yield.

There is dispute over whether thixotropic semi-solid alloys display yield and whether they should be modelled as such. Barnes³⁸ concluded, in a review of thixotropy which did not include semisolid metallic alloy materials as such, that the presence of a yield stress as reported by some workers for thixotropic materials is probably due to the limitations of their experimental apparatus in not being able to measure shear stresses at very low shear rates. Koke and Modigell³⁹ have, however, used a shear stress controlled rheometer to measure yield stress directly on Sn15%Pb. They distinguish between a static yield stress where the fluid is at rest prior to the application of a shear stress, and a dynamic yield stress where the fluid is being continuously sheared. The yield stress increases with rest time prior to deformation because of the increasing degree of agglomeration of the spheroids of solid. In terms of modelling semi-solid alloy die fill, the use of a yield stress may be appropriate because a vertical billet does not collapse under its own weight unless the liquid fraction is too high. In addition, in rapid compression experiments (to be described later) an initial peak in the load versus displacement curve is detected.

The Ostwald de Waele relationship ($\tau = k\dot{\gamma}^n$) is used to describe fluids which do not have a yield point and where there is a power law relationship between the shear stress τ and the shear rate. If the exponent n is 1, this reduces to the expression for a Newtonian fluid with the constant k equal to the viscosity η . A shear thinning material (whose viscosity decreases as the shear rate increases) would have a value of n of less than 1 and a shear thickening material would have n greater than one. Thixotropic materials are essentially shear thinning but also thicken again when allowed to rest (i.e. all thixotropic materials are shear thinning but not all shear thinning fluids are thixotropic).

For a thixotropic material at rest, when a step increase in shear rate is imposed, the shear stress will peak and then gradually decrease until it reaches an equilibrium value for the shear rate over time (Fig. 13). The higher the shear rate after the step, the lower the equilibrium viscosity. The peak viscosity encountered will increase with increasing rest time before it recovers back to the equilibrium viscosity of the shear rate specified.

In semi-solid metallic alloys (as in other thixotropic materials such as clays, mousses, emulsions and flocs), the thixotropic behaviour is associated with agglomeration and disagglomeration. When the material is allowed to stand, or stirred at a low shear rate, spheroids link together to form agglomerates (figure 14). With a step change up in shear rate, the agglomerates are broken down and the viscosity decreases towards a new equilibrium level.

The higher the shear rate (faster stirring) then the lower the viscosity. The higher the fraction solid the higher the viscosity. If the shear rate is changed, then over a period of time the viscosity will reach a new equilibrium value (steady state). The viscosity in the steady state depends on the balance between agglomeration and disagglomeration and also on particle morphology; pure spheres give the lowest viscosity. Liquid entrapped within the spheres does not contribute to shear flow.

5.2 Transient Versus Steady State Behaviour

In semi-solid processing, the injection into the die takes less than one second. If the material is initially at rest (as in thixoforming), in that fraction of a second the viscosity is changing by orders of magnitude from 10^6 Pas to 10^3 Pas (equivalent to that of heavy machine oil). (The viscosity change will be less in rheocasting type processes where there has been insufficient time in a rest state for the agglomeration to occur to a great extent). For modelling, we need to use computational fluid dynamics (or for high solid fractions a 'soft forging' type of approach), but the issue is essentially where to derive the material parameters from. Several workers have carried out rheological experiments to obtain data on the steady state,^{2,3,41-44} but there is little information on transients.

After a shear rate increase, there is a characteristic time for the slurry to achieve the steady state. Quaak³ deduced that in fact there are at least two processes occurring (figure 15) and therefore the breakdown should be represented with a double exponential. At the instant after a jump up in shear rate, Quaak argues that the structure is the same ('isostructure'). Very quickly deagglomeration occurs and then over a slower time scale the fragments spheroidise and coarsen by diffusion. It is the very fast process which is most relevant to transient behaviour in thixoforming and hence we must design experiments to investigate that.

Rheometry.

A rheometer can be used to carry out rapid changes in shear rate.^{3,39,40,45-51} The advantage of a rheometer is that the shear rate in the gap between the bob and the cup can essentially be treated as uniform (but note some concerns which are now emerging⁵²). A typical experiment would involve stirring the alloy (usually Sn15%Pb as a model because few rheometers will operate at semisolid aluminium temperatures) while cooling from above the liquidus point to the required semisolid temperature (to within 1°C) to obtain a non-dendritic microstructure. Stirring continues until an apparent steady state is obtained with the specimen protected from oxidation by passing inert gas over the material. Shear rate jumps for several different rest times and initial and final shear rates can be obtained. Typical results are shown in figure 16.⁴⁰ The challenge experimentally is to collect data very rapidly during the shear rate jump (1 Hz in this case) and to separate out the behaviour of the rheometer from the behaviour of the material. It is important to correct for the inertia of the measuring head (by subtracting from the data the results for the same test but in air) and to allow for any electronic switching that occurs during the shear rate change by only using data from after when 90% of the final shear rate is achieved.

Breakdown times can be obtained by fitting an exponential to the data obtained in the second after 90% of the specified final shear rate has been achieved. The longer the rest time prior to the shear rate jump, the lower (ie. slower) the breakdown time due to increased solid particle sizes and degree of agglomeration. These increases would impede the movement of particles when the shear stress is imposed. The peak viscosity increases with rest time because of the greater degree of agglomeration. The

‘first steady state viscosity’ increases with rest time. The ‘first steady state viscosity’ is a pseudo steady-state between the fast disagglomeration process and the slower diffusion process (figure 15). It is not a full steady state. The breakdown time for the fast disagglomeration process is about 0.15s.⁴⁰ The ease with which the particles flow past each other depends on the particle size, the fraction liquid and the degree of agglomeration. The breakdown time is independent of the initial shear rate but controlled by the final specified shear rate. Breakdown is faster for higher final shear rates and faster than recovery.

The challenge now for rheometry is to obtain data for steels, e.g.⁵³ despite the difficulties with operating rheometers at these higher temperatures.

Rapid Compression Experiments.

The difficulty with rheometers is that there are few that can operate above about 50% solid (because the torque that is required is too high). However, thixoforming often takes place with around 60%. One possibility is then to use the thixoformer itself (or a hydraulic press) to carry out rapid compression experiments.⁵⁴⁻⁵⁷ The shear rates and temperatures are then akin to those in the process itself. However, in contrast with a rheometer, the shear rate varies from point to point within the volume of the material. There are two ways of approaching this. One is to assume an average shear rate applies across the material volume. The other is to model the compression process with Computational Fluid Dynamics (CFD), which, in itself, requires the material parameters one is aiming to measure. Those material parameters then have to be obtained by an iterative process, fitting the CFD results to the experiment.

Figure 17 shows a typical signal response to rapid compression.⁵⁷ The peak load is thought to represent the resistance to flow of the skeletal structure connecting agglomerates. There is rapid breakdown under shear (within 10ms). The peak decreases with increase in temperature (and hence fraction liquid). It increases with increasing soaking time prior to compression as the skeletal structure is built up.

The load versus displacement response can be obtained by using a load cell arrangement.⁵⁷ Recent experiments have shown that the load can also be obtained from data from the ram operation without the need for a load cell.⁵⁸ Typical results obtained using the averaging approach (and assuming Newtonian behaviour in the analysis; a shortcoming) are shown in figure 18. Shear thinning occurs with an increase in shear rate. With increase in temperature, the viscosity decreases. The CFD type of approach has not been fully developed and is discussed further below because it is inherently a modelling approach.

6 Modelling

There have been a variety of approaches to modeling.^{e.g.⁹} Illustrative highlights are given here. CFD approaches include finite difference, both one and two phase, and finite element, again both one and two phase. Micro-macro modelling is also under investigation.⁵⁹ The power of modelling is illustrated in figure 19.

In the interrupted filling with the original die design there are defects where the material is flowing back on itself, with the flow front having been disrupted by the bolt holes and the central boss. The simulation mimics this well. An improved die design widens the ingate, removing the constriction typical of a die casting ingate. The flow front is then relatively smooth. In practice, the improved die design gave excellent results.

Much of the modelling work in the literature is based around the model of Brown and co-workers⁶⁰⁻² where a structure coefficient λ is introduced which varies between 0 for a structure which is fully broken down (disagglomerated) to 1 for a structure which is fully built up (agglomerated). The flow resistance is due to the hydrodynamic flow of agglomerates and the deformation of solid particles within agglomerates. The rate of change of λ is a balance between agglomeration and disagglomeration, with the latter dependent on shear rate because the rupture of particle-particle bonds is shear induced.

6.1 Finite Difference One Phase Modelling

Modigell and Koke⁵⁰ have used a one phase finite difference model (based on FLOW3D from FLOWSCIENCE Inc) to examine flow around an obstacle. Figure 20 illustrates the startling difference in behaviour between a Newtonian fluid and a thixotropic one.

It is important to check that the model can predict not only the flow front but also the rheometer results.⁶³ Results from FLOW3D are shown in figure 21, this time using an ADI solver developed by FLOWSCIENCE to cope with the fact that in thixotforming the viscosity and shear rate change by orders of magnitude over very short distances and times.

A number of workers have used the MAGMAsoft package with the thixotropic module (finite difference, one phase) and obtained reasonable agreement between interrupted filling tests and the simulation.^{e.g. 64,65}

6.2 Finite Element One Phase Modelling

Alexandrou and co-workers^{52, 66-70} have carried out important work with a finite element one phase model, with a continuous Bingham law to avoid the discontinuity at the yield stress. The work has explored the relative importance of the inertial, yield stress and viscous flow effects.

Alexandrou and co-workers have been able to predict the regimes under which various flow patterns occur (figures 22 and 23) and in particular the conditions where defects such as the 'toothpaste instability' are found.

Orgeas et al.⁷¹ use a Power Law Cut-Off model in the PROCAST package (figure 24). Shear thinning only occurs if a cut-off value is exceeded. This can be set at different values in different parts of the component. The behaviour is, therefore, ratchet-type i.e. an increase of the shear rate beyond the largest shear rate experienced so far will lead to decrease in viscosity. Otherwise the viscosity is unchanged. Orgeas et al. also observe that, under certain conditions, flow leads to the concentration of liquid in veins in the structure.

6.3 Finite Element Two Phase Modelling

Two phase models can predict phase separation.⁷²⁻⁸⁰ However, the determination of the rheological parameters required is not straightforward. Modigell and co-workers⁸⁰ have used a finite element two phase model to predict regimes of behaviour (figure 25) and validated the results with Sn15%Pb by filming die filling (figure 26).⁸¹

6.4 Micro-Macro Modelling

Favier et al.⁵⁹ proposed a constitutive equation accounting for the mechanical role of four phases within the material: the solid globules; the solid bonds; the entrapped liquid; and the free liquid. An internal variable which modifies the liquid-solid spatial distribution captures the evolution of the microstructure with the shear rate. The model was originally established in a viscoplastic framework and has been shown to predict the characteristics of the transient response for shear rate jumps for solid fraction lower than 0.5.⁵⁹ It is now being extended to include elastic behavior because the initial sharp rise to a peak in rapid compression tests is not consistent with viscoplastic behavior.³⁷ In the latest results this sharp rise can be modeled (figure 27).

6.5 Experimental Validation

Apparatus for filming die filling has been reported.^{81,82} One of the challenges which has been overcome is the need for an effective arrangement with the window, which is also inexpensive, for use with aluminium alloys, where the high temperatures are problematic. There are advantages to observing the flow *in situ*. Interrupted filling tests have disadvantages as the material can continue to travel, because of inertia, after the ram is stopped. Results are shown in figures 28. Note that with a broader obstacle the flow fronts meet when the die is more full.

Overall, for modelling, there are few examples of direct comparison between models, few examples of quantitative comparison between flow fronts and predictions, and few examples of *in situ* observation of die filling.

7 Alloys for Semi-Solid Processing

The range of alloys available for semi-solid processing and the research to widen the range of applicability has been reviewed.⁸³ More recently there has been significant work on steel alloys for thixoforming.^{84,85}

Virtually all the existing commercial activity is with aluminium casting alloys, particularly A356 and A357, and with magnesium alloys (in the case of thixomolding). Typical properties for aluminium alloys from Vforge (one of the main suppliers of semi-solid processed aluminium alloys) are 241 MPa yield strength for A356 in the T6 condition (with 10-12% elongation) and 262 MPa for A357, again in the T6 condition, (elongation 8-10%).

There is extensive interest in semi-solid processing higher strength aluminium alloys, which are normally wrought, and higher temperature materials such as copper based alloys, cast irons and steels. In addition, there is increasing interest in composites. Some results for the normally wrought Al alloys are shown in figure 29. The results here show that it is ductility which is the major challenge; the yield strengths are close to target (although the target is exceeded with the high strength casting alloy 201) and, for ductility, with 6082. The difficulty here is separating out the processing defects which are inherent to the composition of the alloy and those which are due to laboratory scale methods of making spheroidal microstructure material. For example, some of these results have been obtained with material from a cooling slope and there are questions as to whether oxide has been incorporated as the material has flowed down the slope. However, wrought composition alloys are inherently vulnerable to hot tearing, and, at least in part, the ductility problems are associated with that. This is because the freezing range tends to be relatively wide with a long 'tail' during the final stages of solidification. There has been extensive discussion in the literature about the design of high strength alloys specifically for semi-solid processing,^{83, 87-95} with consideration of the freezing range and the rate of liquid formation within the semi-solid processing range. Thermodynamic prediction is potentially a useful tool in this. There is no commercial production of high strength alloys yet.

There has been some exploration of copper alloys, particularly in the context of motor squirrel cages (where the alloying element can, in fact, be oxygen, to obtain a freezing range, whilst maintaining a relatively high electrical conductivity. However, currently there is not known to be any commercial application.

Considerable attention is being focussed on steels,^{e.g.84,85} and has included the development of steel compositions specifically designed for thixoforming (figure 30).

The modified alloy gives a relatively low melting temperature and temperature for 50% liquid (a critical consideration for thixoforming) in comparison with the non-modified compositions. The slope at 50% liquid and at the end of solidification are also relatively low. The latter is a key criterion for avoiding hot tearing according to Kazakov.⁸⁹

The highly alloyed steels (such as tool steels) are the most amenable and show good results for short runs. However, to some extent, the lack of availability of commercial equipment suitable for such production (eg. with the facility to avoid oxidation during the high temperature heating) has limited the exploration of long run production issues. Tool materials need development. These issues have been recently reviewed.⁹⁶

Other high temperature materials under investigation include ductile irons,⁹⁷ stellites⁹⁸ and high performance steels.²⁷ Metal Matrix Composites (MMCs) are also of interest.^{e.g.17}

8 Concluding Remarks

The current status of semi-solid processing has been reviewed. The discovery of thixotropic behaviour in semi-solid alloys with spheroidal rather than dendritic microstructures has been described. The terminology has been introduced and the origin of the behaviour identified. Routes to feedstock material, and the continuing fertility of ideas in this area, have been described. The distinctions between process routes have been highlighted and the continuing developments in improving routes so that the economics become increasingly competitive are of note. Some examples of industrial applications have been given. Background rheology has been summarised so that efforts to obtain experimental parameters for modelling can be understood. The variety of models has been discussed and future challenges identified. Finally the range of alloys which are currently semi-solid processed is described. There are challenges in widening the range of applicability but significant progress has been made. The technology is gaining increasing industrial application.

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Figure Captions

Fig. 1. a) Dendritic cast microstructure and b) spheroidal microstructure. The micron marker applies to both images and the alloy is A356 aluminium alloy.

Fig. 2. Semi-solid processing routes.³

Fig. 3. The 'new MIT process' [courtesy Prof M. C. Flemings]

Fig. 4. A cooling slope, shown here with material from the slope feeding twin rolls.²³

Fig. 5. Comparison of a) SIMA and b) RAP routes (courtesy Z Omar).

Fig. 6. The UBE new rheocasting (NRC) process.¹⁹

Fig. 7. Sub-Liquidus Casting (courtesy THT Presses and Mr John Jorstad)

Fig. 8. Rheodiecasting [courtesy Prof Z Fan at Brunel University]

Fig. 9. Computer heat sink made from A356 in the T5 condition (courtesy Vforge Inc). Net shape, high conductivity.

Fig. 10. Magnesium alloy components made by thixomoulding (courtesy JSW).

Fig. 11. Automotive seat frame (courtesy Thixomat).

Fig. 12. Shear stress versus shear rate and viscosity versus shear rate for a variety of fluid types.

Fig. 13. Behaviour of a thixotropic material with a step-change in shear rate.

Fig. 14. Microstructures of Sn15%Pb alloy (fraction solid 0.36) at various rest times (after stirring at 30 s^{-1})⁴⁰. The figures 0,1,2 on the micrographs indicate 0, 1, 2 hours rest time.

Fig. 15. Fast and slow processes after either a sudden shear rate increase or a sudden decrease.³

Fig. 16. Shear rate jumps from 0 to 100 s^{-1} after different rest times for Sn15%Pb (fraction solid 0.36).⁴⁰

Fig. 17. A typical signal response to rapid compression.⁵⁷

Fig. 18. Effect of temperature on viscosity under rapid compression of Alusuisse A356.⁵⁷

Fig. 19. Thixoforming of motor end plate. a) interrupted filling with original die design; b) simulation with the original die design; c) simulation with the improved die design (work carried out at the University of Sheffield by Ward, Atkinson and co-workers).

Fig. 20. Results from Modigell and Koke using a finite difference, one phase model.⁵⁰

Fig. 21. Results for shear stress versus time during a shear rate jump between 0 and 100 s^{-1} (Sn15%Pb, fraction solid 0.36), comparing model (FLOW3D) with three repeats of the same experiment and with modeled fits using a spreadsheet.⁶³

Fig. 22. Results from PAMCASTSIMULOR showing flow patterns under a variety of flow conditions.⁶⁸

Fig. 23. Graph of Reynolds Number versus the ratio of Bingham Number to Reynolds Number showing the regimes in which the flow patterns in Fig. 22 occur.⁶⁸ Curves (a) and (b) represent simplified analyses a) for a boundary-layer type analysis and b) for a one-dimensional analysis. Further details are given in the reference.⁶⁸

Fig. 24. Results from Orgeas et al.⁷¹ comparing simulation and experiment with a Power Law Cut-Off Model.

Fig. 25. Map of types of flow.⁸⁰ a) Laminar; b) Transient; c) Turbulent. Bi is the Bingham Number, K_c a rheological number, C_1 and C_2 geometric constants and Re the Reynolds Number. K_c , C_1 , C_2 are not specified in the paper.

Fig. 26. Filling by Sn12%Pb of a T-shaped cavity, showing the effect of piston velocity.⁸¹

Fig. 27. Experimental and predicted load-displacement curves from rapid compression tests assuming either elastic-viscoplastic or pure viscoplastic behaviour for semi-solid A356 aluminium alloy.³⁷ The experimental data is from Liu et al..⁵⁷

Fig. 28. Flow visualization of the effect of obstacle size and shape on meeting of split flow fronts.⁸² Sn 15%Pb, 189°C , 0.25 ms^{-1} , splayed die entrance. Left to right: round obstacle 30 mm diameter; round obstacle 20 mm diameter; asymmetric obstacle with a sharp point where the flow first encounters the obstacle; symmetric obstacle again with a sharp point; asymmetric obstacle the same as in the middle image but with higher velocity (1 ms^{-1}).

Fig. 29. Yield strength and elongation for thixoformed alloys (heat treated condition) in comparison with wrought 'target' values.⁸⁶

Fig. 30. Liquid fraction versus temperature for a steel where the composition has been specifically modified ('C38 modif 2') for thixoforming and is produced by Ascometal.⁸⁴