THIXOCASTING OF ALUMINIUM ALLOYS : FROM MICROSTRUCTURE IN THE SEMI-SOLID STATE TO MECHANICAL PROPERTIES

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ABSTRACT

This paper is concerned with the description of the various production routes of semi-solid aluminium alloys suitable for thixocasting, the characterisation of their microstructures and the correlations between these microstructures and both their rheological behaviour in the semi-solid state and their mechanical properties after thixocasting.

Keywords : *thixocasting, semi-solid, microstructure, rheology, mechanical properties*

1. INTRODUCTION

Thixocasting refers to forming by injection in the semi-solid state of partially remelted billets. For aluminium alloys, it is now becoming an important manufacturing technology for the fabrication of parts mainly for the automotive industry. This development is due to the particular properties of alloys in the semi-solid state when the microstructure of the solid phase is constituted of globular particles suspended in the liquid. The alloy behaves as a solid when at rest whereas it becomes very fluid when it is subjected to high shear rates as during injection. The main advantage of semi-solid over conventional liquid injection is laminar filling of the mould which prevents air entrapment and thus leads to sound parts. Heat treatments are then possible so as structural parts can be produced. In addition, semi-solid casting leads to near net shape parts which thus require less machining and as a consequence are produced at lower cost.

Semi-solid processing, however, has several drawbacks. The microstructure of the semi-solid material must consist of solid globules suspended in the liquid. Such a microstructure is generally obtained by controlled heating of a billet which has been previously solidified with electromagnetic stirring although other treatments are possible. The temperature in the semi-solid state must be also carefully controlled and homogeneous to produce the required amount of liquid. Furthermore, casting conditions are very important to avoid oxide entrapment in the parts and extensive liquid segregation.

The microstructure of semi-solid alloys is thus an extremely important factor for thixocasting both for the rheological properties of the slurry and for the subsequent mechanical properties after forming.

The aim of this paper is therefore to detail the main characteristics of the microstructure of semi-solid aluminium alloys and to correlate these characteristics to their mechanical behaviour both in the semi-solid state and after thixocasting.

In the first part of the paper, the various processes to produce the material will be recalled. The microstructure of partially remelted aluminium alloys will then be described before detailing the main characteristics of the rheological behaviour of such microstructures. Finally, the mechanical properties of thixocast materials will be compared with those of conventionally cast alloys.

2. PRODUCTION OF RAW MATERIAL FOR THIXOCASTING

The microstructure of alloys suitable for thixocasting must consist of globules of the solid phase suspended in the liquid. This microstructure is very different from ordinary dendritic ones in which the solid forms a fully interconnected skeleton. To produce such a microstructure, various processes are possible.

- Mechanical stirring during solidification has been used for the first time to produce globular structures. It can be carried out either with an impeller rotating in the solidifying liquid or through a Couette type viscometer in which the alloy is sheared between two cylinders while cooling of the liquid occurs. The advantage of the viscometer is the possible simultaneous measurement of the rheological properties of the slurry as a function of the various parameters. The shearing forces produced by stirring are thought to break the forming dendrites or at least to bend them. A grain boundary is then expected to form by recovery or recrystallisation and to melt if $\gamma_{gb} > 2\gamma_{sl}$, where γ_{gb} and γ_{sl} are the grain boundary and solid-liquid energies respectively [1,2]. A variant of mechanical stirring is passive stirring in which the solidifying alloy is forced to flow through a tortuous static mixer device by gas pressure or an electromagnetic pump [3]. Another variant, called shearing-cooling-rolling (SCR), was proposed by Kiuchi in which the semi-solid state globularises by the shearing action during the deformation process [4].
- Electromagnetic stirring (ES) is now used industrially in combination with continuous or DC-casting. The advantage is essentially the absence of contact with the liquid metal in comparison with mechanical stirring and the possible preparation of large quantities of raw material. Despite the lower shear rate, the process allows globular particles or degenerated dendrites to be formed. A process using electromagnetic stirring for single slug production has been proposed recently [5]. In this case stirring seems much more efficient so that a fully globular structure is formed prior to the reheating stage.
- Spray deposition [6] together with powder compaction [7] can produce materials with a globular structure. However, they are not viable for mass production.
- Plastic deformation prior to partial melting is very efficient to produce globular microstructures. Indeed, deformation beyond a critical strain will induce recrystallisation during subsequent heating with mostly high angle grain boundaries. Such boundaries will then melt leading to globular solid particles. Various variants have been proposed including the SIMA (Strain induced melt activated) [8] and the RAP (recrystallization and partial melting) [9] processes.
- Partial melting only is sometimes sufficient to generate globular microstructures. This is in particular the case of initially fine dendritic or grain refined microstructures which rapidly transform into globular ones when kept for a short time in the semi-solid state.

3. MICROSTRUCTURES OF SEMI-SOLID Al-ALLOYS DURING PARTIAL REMELTING

The driving force for the evolution of the microstructure in the semi-solid state is the reduction of the area of interface between the solid and the liquid phase per unit volume S_v. This parameter is accessible with image analysis techniques on a polished cross section of a specimen using the formula :

$$S_v = (4/\pi).L_{sl}/A$$

where L_{sl} represents the total solid-liquid interface length measured on an area of section A of the specimen. However, the measurements must obviously be carried out after solidification of the alloy. There is in principle no problem with these measurements if the liquid present in the semi-solid state transforms into a solid which is easily distinguishable from that already existing. This occurs when the remelting temperature is just above the eutectic temperature of the alloy. Indeed, the liquid transforms into a two-phase mixture so that the solid-liquid interface is usually well defined except when the eutectic is divorced for low solidification rates or small local liquid fractions. Under these conditions, the volume fraction of solid is also easily accessible.

The situation is much more complex when the remelting temperature is far above the eutectic temperature. In this case, the liquid solidifies first into primary solid which is hardly discernible from the already existing one and then into the two-phase eutectic mixture. Etching might sometimes reveal the primary solid formed after isothermal holding in the semi-solid state owing to composition variations but the boundary between the two solids is very fuzzy so that no precise measurement can be carried out.

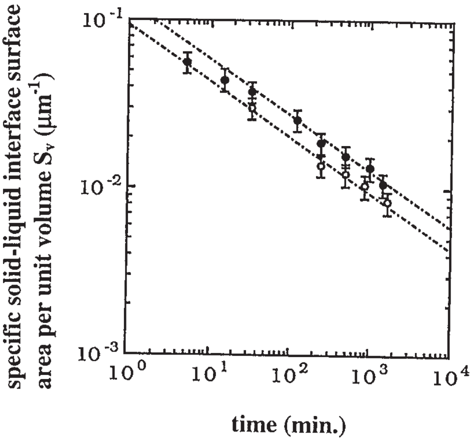

Fig. 1 : Evolution of the solid-liquid interface surface area per unit volume S_v as a function of isothermal holding time during the partial remelting at 580°C of conventionally (o) and electromagnetically stirred (•) DC-cast Al-Si7-Mg0.6.

For the Al-Si7-Mg alloys, which are the main alloys used for thixocasting, remelting is normally carried out at just above the eutectic temperature so that S_v can be measured without any difficulty. Figure 1 shows the variation of S_v with isothermal holding time in the semi-solid state for a conventionally solidified alloy (dendritic microstructure) and an ES one (globular microstructure) [10]. The figure shows that S_v obviously decreases with increasing time but the variations for the two microstructures are very close, despite the important difference in morphology of the solid phase (Figure 2). This inability of S_v to correctly describe the microstructural evolution during partial remelting led Loué to introduce a dimensionless grain-specific shape factor of the globules F_g by considering the square of the average solid-liquid interface area per grain [10]:

$$F_g = S_v^2/6\pi f_s N_A$$

where f_s is the volume fraction of the solid phase and N_A represents the number of grains per unit area of section of the specimen. F_g is equal to 1 for perfectly spherical grains. It is to be noted that a simple object count by image analysis will certainly give a too high value of N_A since two objects can belong to the same grain. For Al-Si7-Mg alloys, N_A was determined by using polarised light after anodising the sample in order to distinguish the different solid phase grains by their crystallographic orientation [10]. A more accurate determination would be to examine the specimen by 3D X-Ray tomography. Indeed, this technique is now accurate enough and can be used with sufficiently big samples thanks to Synchrotron sources. Experiments are planned in April 1998 at the ESRF in Grenoble for this purpose.

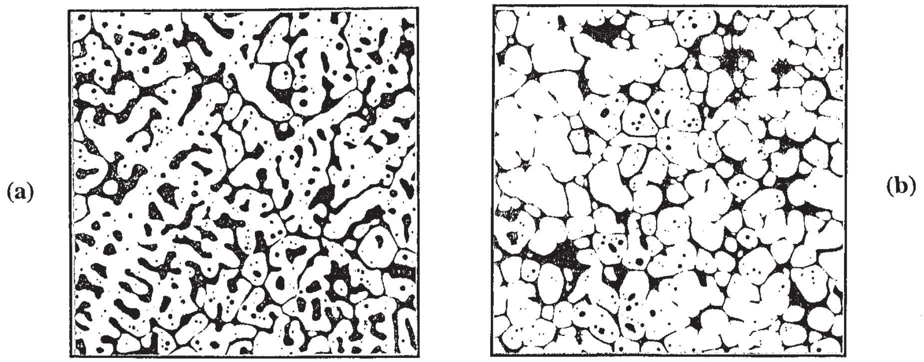

Fig. 2 : Microstructural evolution during partial remelting for 5 min at 580°C of a Al-Si7-Mg0.6 alloy (a) conventionally cast or (b) electromagnetically stirred and cast.

Figure 3 shows the variations of F_g for the two previously defined alloys. One observes that F_g remains far from 1 for the conventionally cast alloy even for long isothermal holding time whereas it rapidly approaches unity for the ES alloy, corresponding to a perfectly globular microstructure. F_g seems thus to correctly reflect the microstructural evolution during partial remelting and subsequent holding, as observed in Figure 2.

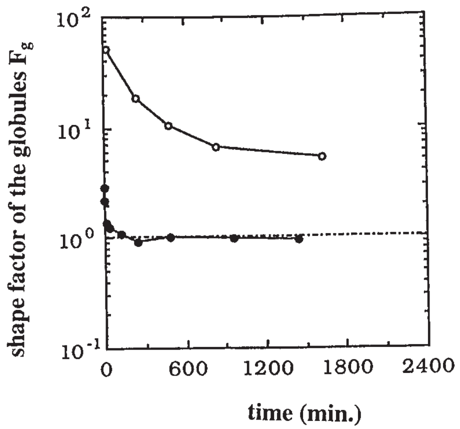

Fig. 3 : Evolution of the shape factor of the globules as a function of the holding time at 580°C for a Al-Si7-Mg0.6 alloy cast (o) without and (•) with electromagnetic stirring.

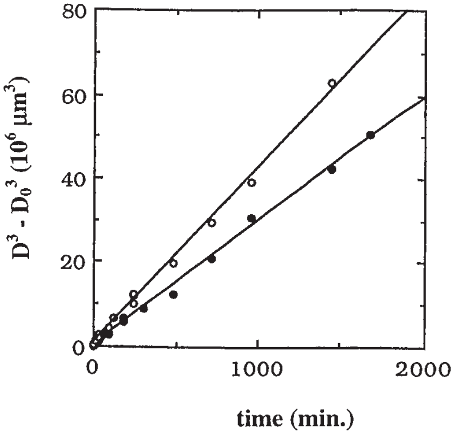

Fig. 4 : Evolution of the globule size with holding time at 580°C for a Al-Si7-Mg0.6 alloy electromagnetically stirred in the (o) as cast conditions or (•) after 25% reduction cold working before remelting

During isothermal holding in the semi-solid state, the globules of the solid phase are coarsening owing to Ostwald ripening and coalescence. This coarsening is usually well described by the relationship :

$$D^3 - D_0^3 = Kt$$

where D_0 is the initial globule size and D is the globule size after an isothermal holding time t. K is a temperature dependent parameter. Experiments carried out with various alloys have shown that coalescence is an important if not dominating coarsening mechanism [10, 11]. However, the coarsening rate K depends for a given alloy on the manner the material has been processed in order to obtain the globular structure. As an example, figure 4 shows the variation of the globule diameter as a function of time for an ES Al-Si7-Mg0.6 alloy in the as-cast or after 25% reduction cold-working before remelting. The as-cast alloy shows a higher coarsening rate than the cold worked one which suggests that coalescence is more important. This difference in coalescence can have at least two origins : either a less random orientation of the globules in the as-cast alloy (making coalescence easier) or a more random grain orientation in the cold worked alloy (making coalescence more difficult). This explanation is in agreement with very recent local texture measurements (EBSD) carried out on a 6082 alloy which have shown that the ES alloy contains a significantly large proportion of low angle grain boundaries [12]. In contrast the same alloy produced by grain refinement presents a lower proportion. Another explanation could be that in the cold worked alloy, the distribution of the grain sizes is more narrow than in the as-cast ES alloy, thus reducing the rate of Ostwald ripening. Further experiments are needed to clearly elucidate this question.

A parameter which is very important to characterise the microstructure of alloys in the semi-solid state is the amount of liquid entrapped inside the globules. Indeed, this liquid does not participate to the flow behaviour of the semi-solid slurry so that the effective volume fraction is less than that given by the equilibrium diagram. The viscosity of the slurry is therefore higher than expected. It is, however, necessary to distinguish the really entrapped liquid from that which seems to be entrapped on a 2D section of the sample. This distinction is usually not very difficult owing to the particular morphology of the really entrapped liquid. Figure 5 shows a globule in an Al-Cu alloy in which a large amount of entrapped liquid is present. It is clearly observed that the liquid which is really entrapped is spherical and solidified in a divorced manner, thus being constituted of the θ Al_2Cu phase. Conversely, the liquid which is not really entrapped, solidified in a similar way as the interglobular one. The parameters which influence the amount of entrapped liquid in a semi-solid alloy are presently not fully elucidated. Fine dendritic structures are in particular known to generate upon partial remelting a large amount of entrapped liquid.

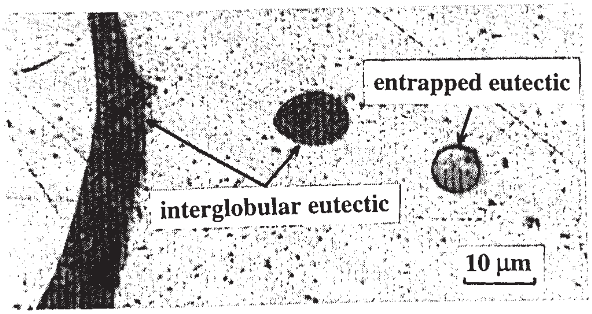

Fig. 5 : Entrapped eutectic in a globule of a Al-10%Cu alloy consisting of Al_2Cu phase in comparison with the normal eutectic which is similar to the interglobular one.

The volume fraction of entrapped liquid f_l^e changes during isothermal holding of the alloy in the semi-solid state. In the case of a Al-Si7-Cu3-Mg alloy [13], the evolution is rather complex especially during short holding times since starting from an absolute value of 5-6%, f_l^e decreases after 1 min to 2.5%, increases up to 5% and then decreases rapidly to reach nearly 3% after 5 min. Such an evolution was explained by the two coarsening mechanisms of the globules, Ostwald ripening and coalescence, which have opposite effects on entrapped liquid [13].

Finally, there is another factor which has a strong influence on the rheological behaviour of a semi-solid alloy, that is the agglomeration parameter of the globules. This parameter was introduced by several authors to account for the thixotropic behaviour of semi-solid alloys. Agglomeration is defined as the reversible bonding together of two or more individual globules. The globules in an agglomerate moves together during deformation , but they remain distinguishable. Deagglomeration is the reverse process, i.e. the breaking down of an agglomerate into its constituent globules. Agglomeration and deagglomeration processes occur continuously when a shear rate is applied to the alloy and they are competitive. Therefore, a dynamic equilibrium is maintained between the two. Also, agglomeration varies when the material is kept at rest, especially when the volume fraction of the solid phase is high.

An important effect of agglomeration is that a certain amount of liquid is immobilised between the agglomerate globules. This liquid fraction, which does not contribute to fluid flow, must thus be added to the entrapped liquid fraction mentioned before. Therefore, the effective fraction of solid drifting in the liquid phase can be much larger than the actual solid fraction.

4. RHEOLOGICAL BEHAVIOUR

The rheological behaviour of semi-solid alloys can be investigated by various techniques. Since partially remelted microstructures are considered in this paper, only the techniques which have been used for these materials will be mentioned.

After partial remelting, the rheology is frequently quantified using parallel plate compression experiments. A cylindrical specimen with sufficiently high solid fraction is deformed between the two plates of a compression press. From the applied strain rate and the measured compression force during steady state deformation, an apparent viscosity may be derived [14]. This test leads, however, to possible liquid segregation at large strain so that it is no longer exploitable. Rather than applying a constant strain rate during the experiment, step changes in strain rate can be performed. Such a procedure allows to determine the constant structure response of the material together with its steady state response.

Back extrusion experiments have also been used for partially remelted alloys [14]. These experiments have the advantage that high shear rates are required to obtain homogeneous deformation without liquid segregation which thus allows the evaluation of the rheological behaviour of the alloy in similar conditions as thixocasting. A piston is pushed at constant velocity into the semi-solid sample present in a container and the force is recorded. Average shear rate and apparent viscosity may be derived from piston velocity and measured force. A variant of this test is the indentation test in which a needle is pushed into the semi-solid alloy [15].

Couette type viscometer experiments can also be used to quantify the rheological behaviour of semi-solid alloys. The alloy must first be solidified in between the two cylinders of the apparatus under various cooling conditions and then partially remelted and tested. The advantage of the test is a stress state of pure shear.

The previous mechanical tests were mainly used to obtain the apparent viscosity as a function of shear rate and solid fraction of partially remelted alloys considered as suspensions of solid globules in the liquid. Other tests have been designed with the objective of obtaining the constitutive equations of semi-solid alloys assumed as porous media saturated with liquid. Drained compression with lateral pressure experiments together with tensile stress state experiments are in this category.

Drained compression allows the evaluation of the densification of the solid phase under various stress states. It can thus be carried out by compression of the specimen placed in a container above a ceramic filter but in this case, the stress state is rather complex and the measured load is altered by the friction of the piston against the container walls. This test allows, however, specimens with various amounts of liquid phase to be obtained without modifying the primary solid phase (which occurs when varying the temperature) [16]. Drained compression has also be carried out by applying a gas pressure in addition to an axial compression stress on a specimen wrapped into a pure aluminium deformable envelope [17]. In this case, no friction occurs and the densification of the solid phase can be accurately measured. Such tests have been carried out also with Sn-Pb alloys wrapped in a silicone rubber envelope and oil pressure as the loading medium [18].

Tensile tests have also been carried out scarcely to evaluate the tensile behaviour of semi-solid alloys. They are, however, important since the tensile behaviour is very different from the compression behaviour due to the cohesion of the solid. This is particular true for globular microstructures for which the cohesion of the alloy is only due to the agglomeration of the globules. In the case of dendritic microstructures, the solid phase forms a continuous network so that the tensile behaviour may not be very different from the compressive behaviour except at low solid fractions.

As previously mentioned, a partially remelted alloy can be considered either as a suspension of solid globules in the liquid or as a porous solid medium saturated with liquid. In the first approach, the apparent viscosity describes the rheological behaviour of the alloy and the solid fraction, the shear rate and the morphology of the solid phase must be considered as parameters. The influence of the solid fraction is usually described by an exponential function of the form:

$$\eta = A\exp Bf_s$$

where B is a coefficient taking values of the order of 15 to 20 when the solid fraction is close to 0.5 [19]. The solid fraction thus sharply influences the viscosity of partially remelted alloys so that its

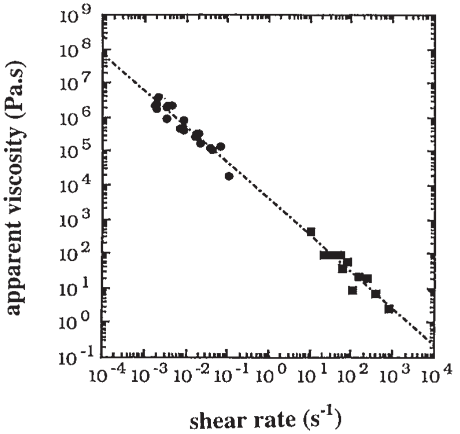

Fig. 6 : Variation of apparent viscosity as a function of shear rate for an electromagnetically stirred Al-Si7-Mg0.6 alloy deformed by compression (low shear rates) and back extrusion (high shear rates) at 580°C after an isothermal holding time of 30 min.

determination must be carefully carried out. In particular, the volume fraction of liquid entrapped in the globules or immobilised between the globules must be considered since it does not participate to the deformation.

The shear rate largely influences the viscosity of semi-solid alloys. Figure 6 shows the variation with shear rate of the viscosity of a Al-Si7-Mg0.6 alloy determined from both compression (at low shear rates) and back extrusion (at high shear rates) experiments [14]. The slope of the straight line is close to -1 indicating a strong shear thinning behaviour. It is to be noted that this corresponds to shear stresses independent of the shear rate. This behaviour is characteristic of steady state deformation of semi-solid alloys which corresponds obviously to varying microstructures with shear rates. However, during thixocasting, the material is deformed initially under transient conditions so that the isostructure behaviour is also very important. In order to study such a behaviour, step changes in shear rate can be carried out. This was particularly done during compression experiments [20, 21]. Figure 7 shows the typical response in terms of compression stress after a rapid step change in strain rate for a partially remelted Al-Si6-Mg0.6 alloy initially produced by electromagnetic stirring. Right after the strain rate change, stress increases rapidly leading to an apparent strain rate sensitivity close to 0.2, and then decreases to reach the value it would have without strain rate change. This result is consistent with a strain rate sensitivity equal to 0 under steady state conditions. Such a behaviour can be explained by deagglomeration of the globules when the shear rate is increased. In a similar way, a decreasing step change in strain rate would lead to an instantaneous decrease in stress followed by a gradual increase to the stress value it would have without strain rate change. In this case agglomeration of the globules would explain the behaviour.

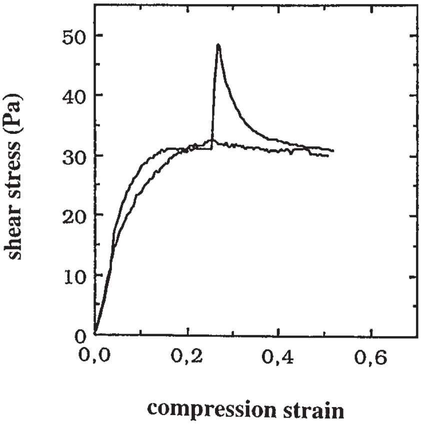

Fig. 7 : Typical stress-strain curve obtained during a step change in strain rate from 5 10^{-3} to 5 10^{-2} s^{-1} in the case of a Al-Si6-Mg0.6 alloy cast with electromagnetic stirring and partially remelted for 30 min. at 580°C before the test. The continuous curve is obtained at a strain rate of 5 10^{-3} s^{-1}.

The morphology of the solid phase is an other parameter which influences greatly the viscosity of semi-solid alloys. As previously mentioned, this morphology has been characterised by a shape factor of the globules F_g. By holding for various times in the semi-solid state different specimens of a Al-Si7-Mg0.6 alloy conventionally solidified with grain refining [22], it was shown that the apparent viscosity increases by 3 orders of magnitude when F_g increases by a factor of 100.

The other approach is to consider the semi-solid alloy as a two-phase porous medium saturated with liquid. In this case, the solid and the liquid are assumed to be continuous. To model the behaviour of the porous solid phase, two functions of the solid volume fraction have been introduced in the expression of the equivalent stress [23] : a function A which measures the shear behaviour of the solid and a function B which determines its densification behaviour. These functions obviously depend on the morphology of the solid phase but again no attempt has been made to introduce a morphology parameter in these functions. This model assumes that the behaviour of the semi-solid alloy is similar in tension and compression which is not true particularly for globular structures. The different behaviour in tension and compression was demonstrated in the case of a Sn-Pb alloy for both a dendritic and a globular morphology. A cohesion parameter of the solid phase was introduced for this purpose [24, 25]. A similar approach would be useful for aluminium alloys but this requires tensile tests to be carried out for various solid fractions.

5. MECHANICAL PROPERTIES AFTER THIXOCASTING

Thixocasting is carried out by injecting the correctly reheated slug into a die cavity using a high pressure die casting machine. The injection speed is usually much slower than during classical high pressure die casting and the final pressure is generally about 100 MPa. Because of the low injection velocity and the much higher viscosity of the material when compared with liquid injection, no air is entrapped in the produced parts. Therefore, mechanical properties comparable to the best levels of mechanical properties that can be obtained in permanent mould or squeeze casting are attainable. As an example, Table 1 gives the yield strength (YS), the ultimate tensile strength (UTS) and the elongation to fracture (A) measured on various thixocast parts in the as-cast and T6 conditions [22].

Part	Alloy	Temper	YS, (MPa)	UTS, (MPa)	A, (%)
fuel rail	A356.0	F	105	220	9-11
bicycle crank	A356.0	T6	260	325	11-14
test part	357.0	T6	290	345	9-11

Table 1: Mechanical properties measured on thixocast parts in the as-cast (F) and T6 conditions
T6 = 10 h at 540°C + water quench + 6 h at 160°C for A356.0 or 6 h at 170°C for 357.0

However, the mechanical properties and particularly the elongation to fracture of thixocast parts are strongly sensitive to the casting defects. These defects are essentially oxide skins, eutectic segregation, coalesced Si crystals and shrinkage porosity. Details about their origin are given in [22].

In order to determine the mechanisms of deformation and fracture of thixocast materials, some tensile tests have been carried out in the scanning electron microscope on specimens taken from test bars [26]. For the as-cast A356 alloy which exhibits fine eutectic, extensive plastic deformation of the globules is observed with also some decohesion around the undeformed pockets of entrapped eutectic (figure 8). Fracture is therefore not confined in the eutectic but occurs also in the globules. The T6 heat treated material shows a different behaviour owing to the presence of much coarser Si particles and harder globules compared to the as-cast material. Fracture of the Si particles occurs then extensively at the beginning of plastic deformation (figure 9) and rupture of the specimen is concentrated in the eutectic mixture.

6. CONCLUSION

In this paper, a review of the main characteristics of the microstructure of partially remelted aluminium alloys has been made including in particular the morphology, the size and the degree of agglomeration of the globules and the amount of liquid entrapped in these globules. Correlations between these microstructural features and the rheological behaviour of semi-solid alloys have been detailed by considering these alloys either as concentrated suspensions of solid globules in the liquid or porous media saturated with liquid. Finally, mechanical properties of thixocast parts have been examined together with fracture mechanisms deduced from tensile tests carried out in the scanning electron microscope.

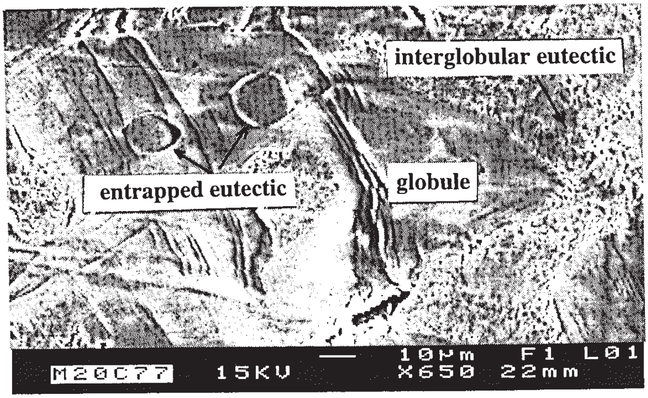

Fig. 8 : Decohesion around entrapped eutectic in a thixocast A356 alloy deformed in the as-cast condition in the scanning electron microscope

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