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Producing thixotropic semi-solid A356 alloy: microstructure formation x forming behaviour

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ABSTRACT

Purpose: The work discuss the phenomena involved in the formation of the microstructure of semi-solid thixotropic alloy A356 produced by different techniques and the relation between microstructure and forming behaviour of the material.

Design/methodology/approach: Thixotropic slurries of A356 alloy were produced from liquid and from solid conditions; in the first case either by stimulating nucleation rate or crystal multiplication during solid growth; in the second case by recrystallization and partial melting of deformed structures. Flow behaviour was analysed by viscosity measurements or by flow ability in forging operations.

Findings: Results show that different production techniques activate different mechanisms, leading to distinct structure features in the semi-solid slurry, and, as consequence, in its forming characteristics. Techniques which promote formation of isolate globules of primary phase, like those involving nucleation stimulation and recrystallization, result in a semi-solid with better flowing behaviour. On the other hand, techniques based on crystal multiplication during growth lead to more interconnected globules in the slurry and poorer forming behaviour.

Research limitations/implications: Semi-solid processing is suitable only for alloys with appropriate liquid fraction x temperature relation.

Practical implications: the knowledge of the phenomena involved in the formation of thixotropic metallic slurries produced by different techniques, and their consequences in the material structure and flow behaviour, allow the decision of the adequate slurry production method for a specific application, in order to take the best advantage of the semi-solid technology.

Originality/value: The analysis of the relation between production process x flow behaviour of semi-solid thixotropic A356 is original.

Keywords: Casting; Semi-solid alloys; Al alloys; Thixoforming

1. Introduction

Semi-solid metallic alloys (SSM) have been increasingly used in the so called thixoforming technology, as raw material for casting processing, although already proved suitable for other forming processes like forging, extrusion and even drawing [1-5]. Once liquid fraction can be controlled in the thixotropic metallic slurry, the same raw material can be used in either forming process ranging from casting to drawing. However, the production of the appropriate semi-solid can involve quite different techniques, which have been under development in the last 30 years [6-11].

Such techniques can be gathered in two groups depending on the state of the starting material: liquid or solid. In the first group are those techniques which produce semi-solid from liquid by interfering in its solidification, either by increasing nucleation rate or by stimulating crystal multiplication of growing solid; while in the second group are the techniques which produce globular semi-solid directly from solid material by heat treatments at T>T*solidus*. In this case melting of secondary phases and spheroidization of the primary solid phase in liquid environment lead to the thixotropic material.

Each different production route is based on the activation of certain metallurgical phenomena that command the structure formation, resulting in specific and probably distinct characteristics in the slurry, such as size and morphology of globules of the solid phase, as well as the degree of interaction among them. The flow behaviour of the thixotropic slurry, its ability to fill dies and the quality of the final thixoformed product are dependent on the structural features of the raw material. Therefore, it is fundamental to understand the metallurgical principles involved in each technique used to produce the semi-solid raw material, to properly manipulate the processing parameters to obtain the desirable structure.

Therefore, the proper control of the mechanisms involved in the formation of the slurry in a specific production technique makes it possible to obtain the desirable characteristics in the slurry for a specific forming process.

This work seeks to contribute to SSM technology by analysing the mechanisms involved in the formation of thixotropic slurries structures by different production routes, and the consequent flow behaviour in the semi-solid state.

2. Experimental procedures

In the experiments was used the alloy A356, with basic composition Al/6.9%Si/0.2Mg/0.2Cu/0.2Fe/0.2Mn (wt%). DTA tests showed a solidification range between T*solidus* = 578 ± 2ºC and T*liquidus* = 618 ± 2ºC.

Different routes were utilized to produce semi-solid materials: (a) interfering in the number of solid nuclei in the liquid; (b) interfering in the solid growth after nucleation; (c) modifying deformed dendritic structures by partial melting.

In the first case (a) two different techniques were used to increase the number of solid nuclei in the liquid at the onset of the solidification: continuous casting using a cooling slope; and intensive nucleation promoted by chilling effect in mould walls. In the cooling slope method, the liquid metal was poured over a cooled, inclined plate before reaching a metallic mould where was quickly solidified. The process is described elsewhere [12]. Figure 1 shows schematically the experimental assembly used in the cooling slope technique. In the chilling technique, the liquid metal was poured with low superheating (620°C) in a metallic mould at room temperature.

In the second case (b), the alloy was submitted to electromagnetic stirring while solidifying in a continuous system. Conditions used were: 1200W/kg, casting speed 1cm/s. In the third case (c), as-cast dendritic structures were submitted to cold deformation and then heated at temperatures within the solidification range. Experimental conditions were: 80% deformation by compression; heating conditions at 580°C/15min. At this temperature it is expected approximately 0.45 solid fraction in the slurry.

Samples produced by cooling slope, chilling and electromagnetic stirring techniques were re-heated to 580°C/15min to promote structure modifications towards the typical globular thixotropic condition.

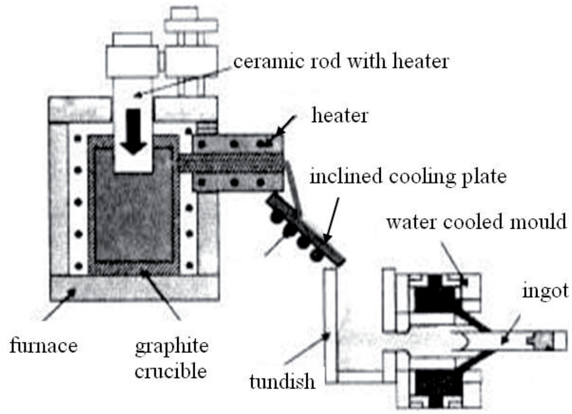

Fig. 1. Schematic illustration of horizontal continuous casting apparatus with inclined cooling plate used in the experiments

From semi-solid state (at 580°C after holding for 15min), part of the samples were cooled in water to "freeze" the structure for microstructure analysis, to make it sure that fabrication and heating conditions were properly chosen to produce similar globules size and liquid fraction in all cases.

Conventional metallography preparation and Keller's etching were utilized.

Another series of samples of semi-solid material were used for evaluation of the flow behaviour. Two different experiments were performed to evaluate the material flow ability: viscosity measurements or thixoforming tests.

Viscosity was measured by compression tests using a MTS equipment, with compression rate of 10mm/s; samples were hold 4min at 580°C before forming. Load x displacement curves were plotted and analysed.

The shear rate (γ_{AV}) is given by [13]:

$$\dot{\gamma}_{AV} = -\left(\sqrt{\frac{V}{\pi}}\right)\left(\frac{\delta H / \delta t}{2H^{2.5}}\right) \quad (1)$$

where V is the sample volume (m^3) and is constant, H is the instantaneous height (m), (δh/δt) is the compression rate (m/s).

The apparent viscosity (μ_{AP}) in Pa.s was calculated using the equation given by [8]:

$$\mu_{AP} = \left(\frac{8\pi F}{3V^2}\right)\left(\frac{1}{H^4} - \frac{1}{H_0^4}\right)^{-1} t \quad (2)$$

Where F is the load (N) in a time t, H_o is the initial height (m).

Thixoforming experiments were carried out to produce parts from cylindrical samples with dimensions ϕ 44.5mm x h 10mm, by forging into a steel die. Forging temperature was 580°C, punch speed was 4mm/s. Applied forces were monitored during thixoforming operation and load x time curves were plotted.

Figure 2 shows schematically the forging assembly used in the experiments.

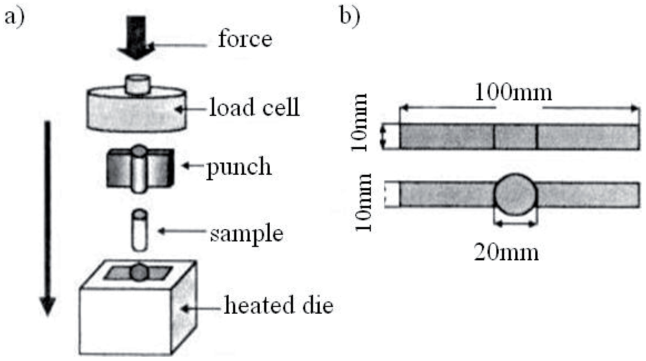

Fig. 2. Schematic illustration of (a) the thixoforging operation; (b) dimensions of final product

3. Results and discussions

3.1. Producing SSM by stimulating nucleation - cooling slope technique

Figure 3 shows typical material obtained by rapid cooling of the alloy A356 in a metallic mould, after pouring over a metallic, cooled, inclined plate.

It can be observed well defined, near rounded crystals of the α primary phase, far different from the usual dendritic structure produced by conventional solidification in practical cooling conditions. This result shows the efficiency in the suppression of dendritic formation when the cooling slope is used. Measurements of the size of the α primary crystals indicate values ranging from 80 to 120μm.

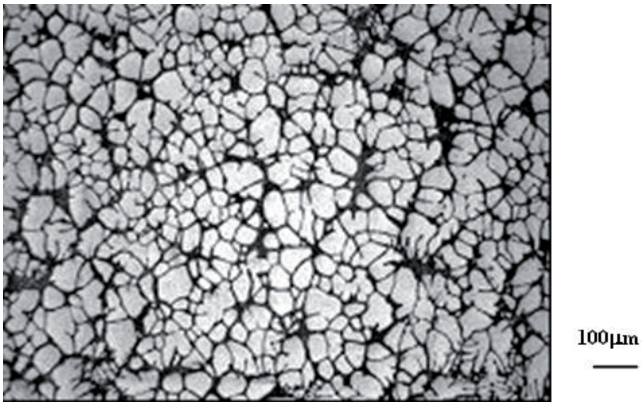

Fig. 3. Microstructure of A356 alloy obtained by continuous casting with cooling plate

In the cooling slope method, when the liquid is poured over the inclined plate, the initial contact with the cold surface of the plate promotes the necessary undercooling for solid nucleation; therefore solid crystals appears in the region. As the pouring operation continues, such initial crystals can be detached from the plate surface and join the liquid downwards the mould. The free surface of the cooling plate can now promote new nucleation and successively crystals are formed and detached from this surface, which can then be considered a "source" of nuclei.

Therefore, when using a cooling plate, the metal filling the mould is no longer simply a liquid, but constituted by liquid and a certain fraction of solid as fine equiaxial crystals. Once in the mould, if rapid cooling is provided, dendritic degeneration is limited or even totally suppressed, resulting in a fine near globular structure, as the one observed.

The detachment of crystals from the surface of the cooling plate or, in general, from cold mould walls, can occurs as a consequence of the solute segregation in the solidification front taking place during solid growth in a metallic alloy. A layer of enriched solute content is formed in the liquid ahead of the solidification front, as solute redistribution in the whole liquid is limited in practical solidification conditions.

The difficulty of the segregated solute atoms to disperse in the liquid is higher in the region of the nucleus in contact with the mould surface, resulting in lower growing rate in this region. As a consequence of this local deceleration of the growing rate a necking is developed; the necked crystal can now be easily detached from the surface where it lies.

Figure 4 (a) shows schematically the mechanism of necking and detachment of crystals from mould walls. This mechanism is known as "separation theory" and was firstly described by Ohno and Motegi [12] when explaining the formation of equiaxial grains in castings.

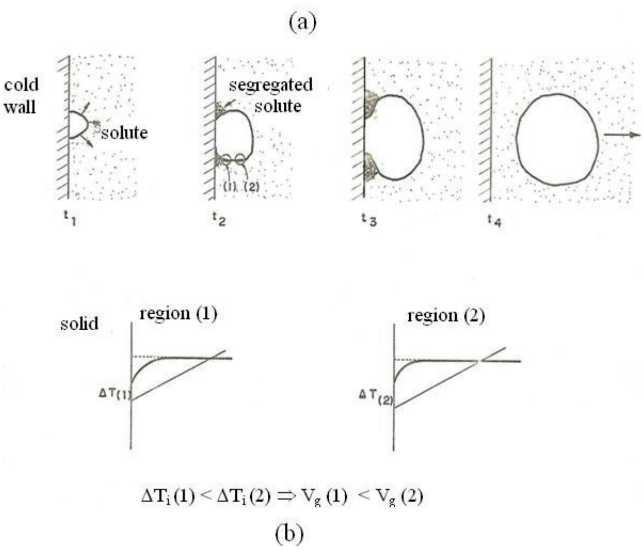

Fig. 4. (a) Schematic illustration of the mechanism of detachment of nuclei from the surface of the cooling slope; (b) temperature profiles in the liquid in the solidification front, for an alloy with partition coefficient K <1

The different grow rate in different regions of the solid surface is due to the temperature profiles in the liquid containing different gradients of solute concentration ahead of the solidification front, as shown in Figure 4 9b). Where gradient is higher (roots of the crystal in contact with the cold surface) higher is the reduction in the transformation temperature, leading to smaller interface undercooling and, as a consequence, smaller growing rate.

It has to be pointed out that the activation of the separation mechanism to promote non-dendritic structures is dependent on the presence of solute segregation; therefore, its influence on the alloy structure is more significant in alloys presenting wide solidification range. The presence of convection in the liquid helps the detachment of the grains from mould walls and also their survival in the liquid, once promotes faster heat exchanges; but can prevent necking if promotes solute redistribution. Another important parameter is the pouring temperature; it must be appropriate to promote the necessary chill effect in the surface of the cold plate. Therefore, the success of the utilization of the cooling slope method to produce non dendritic structures depends on the appropriate choice of the controlling parameters.

The non-dendritic structure obtained in the process can be considered a suitable raw material for thixotropic semi-solid production. By simply re-heating the material to a temperature within *solidus* and *liquidus*, a globular structure can be achieved. Figure 6 shows the result of re-heating at 580°C/15min in the structure shown in Figure 3.

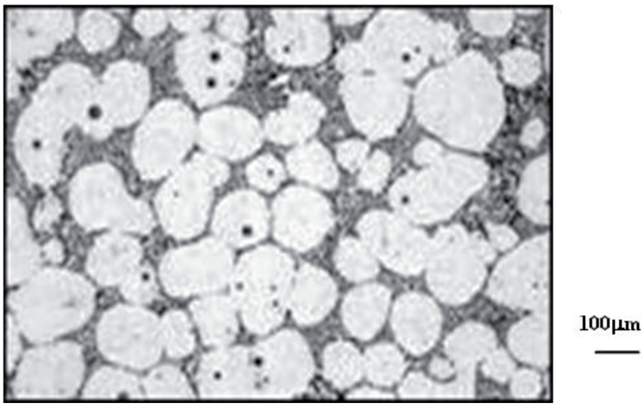

Fig. 5. Thixocast microstructure of A356 alloy produced by continuous casting with cooling plate + re-heating at 580°C/15min

It can be observed typical thixocast structure with the primary solid phase presenting globular morphology. When held within the solidification range, the globularization of the solid phase surrounded by liquid is a natural phenomenon, driven by the need to reduce surface energy. The re-heating and holding at high temperatures promoted also significant increase in the size of the globules of the primary phase. The globularization and growing mechanisms involve solid-solid diffusion (coarsening involving solid/solid interfaces, like coalescence) and solid/liquid/solid diffusion (coarsening involving Ostwald ripening).

Secondary phase (eutectic α + Si) is finely distributed in primary α globules boundaries, as a result of the rapid cooling of the material from the semi-solid state.

3.2. Producing SSM by stimulating nucleation - chilling technique

Figure 6 shows typical microstructure of the alloy A356 produced by low superheating at pouring associated with rapid cooling in the mould. By using these conditions it is expected the stimulation of the chilling effect at mould walls and the provision of conditions for chilled crystals to survive.

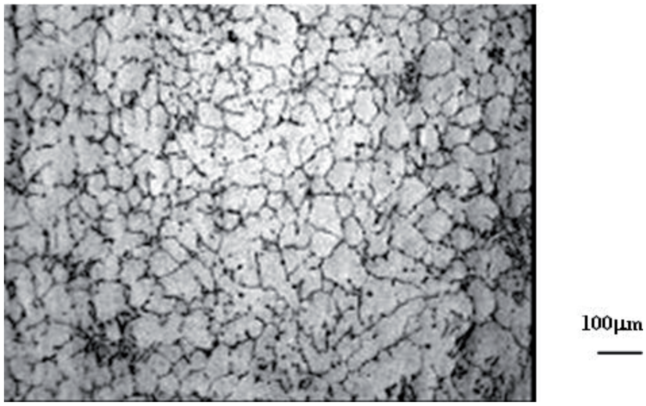

Fig. 6. Microstructure of A356 alloy obtained by pouring with low superheating in a cold metallic mould

It can be observed that the primary α phase is highly refined indicating that restriction of dendritic development could be achieved. Size of primary α phase is in the range of 80 to 120µm, similar to the size of primary phase in the structure produced by the cooling slope method. The structure can be considered ultra-refined.

When pouring a liquid with small superheating in a mould with high thermal conductivity, intensive nucleation can occur in a region close to the mould internal surface, in a "big-bang" effect – a layer with significant thickness of liquid is cooled to the necessary undercooling to promote nucleation. Thermal conditions in the liquid close to mould walls, leading to a chill effect are illustrated in Figure 7.

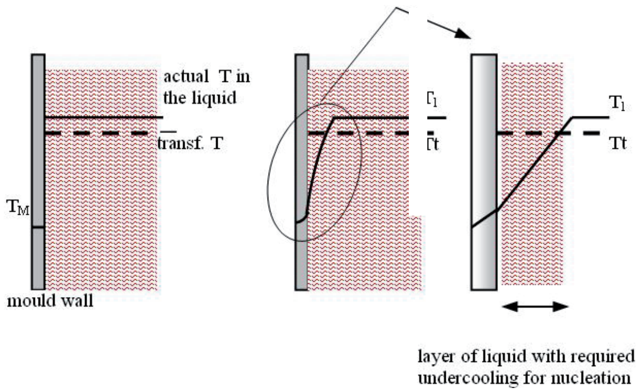

Fig. 7. Schematic illustration of the thermal conditions close to mould walls when a liquid with low superheating is poured in a mould with high thermal conductivity

The rapid heat exchange and the fast growth of the nuclei in contact with the mould surface can provide the necessary conditions for the chill nuclei formed in the undercooled liquid layer to survive. Small thickness of the cast session associated with high initial nucleation rate can promote nucleation throughout the liquid volume, resulting in refining in such a degree that dendritic formation and growth is avoided, as observed in the structure obtained.

As commented earlier, such kind of structure is suitable for thixotropic slurries production.

In Figure 8 is presented the microstructure showed in Figure 6 after re-heating at 580°C and holding 15min at this temperature.

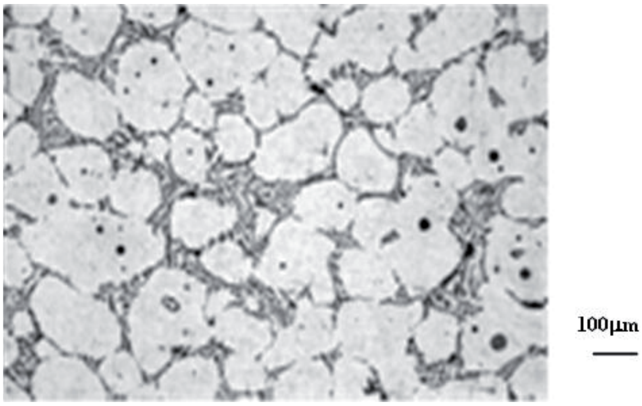

Fig. 8. Thixocast microstructure of A356 alloy produced by stimulating chilling effect + re-heating at 580°C/15min

A typical thixocast structure with globular primary phase can be observed. Dimensions of primary phase particles are higher than the near-globules of the original material, as a consequence of the coarsening mechanisms involved in the structure globularization and growth in the liquid environment.

3.3. Producing SSM by stimulating crystal multiplication during growth

Figure 9 shows microstructure of A356 obtained by using electromagnetic stirring during solidification. The primary phase morphology is rather globular like than dendritic, with dimensions ranging from 80 to 125µm.

When letting the alloy to solidify in the presence of convection in the liquid in the solidification front, which promotes relative movements of liquid layers, the shearing forces generated can induce modifications in the structure being formed leading to a refined equiaxial grains. The effect is attributed to a phenomenon known as "crystal multiplication" and was firstly studied to understand the effect of stirring of the liquid in the formation of equiaxial grains in castings [14].

When heavily stimulated, the crystal multiplication can inhibit dendritic development, resulting in a fragmented or near-globular structure, which can be suitable for thixotropic semi-solid production by heat treatments at temperatures above *solidus*. The crystal multiplication is based on the detachment of dendrite arms from the main body and their independent growth in the liquid as a new crystal. Therefore, it requires dendritic growth at certain extent and can be present during the whole period of solidification.

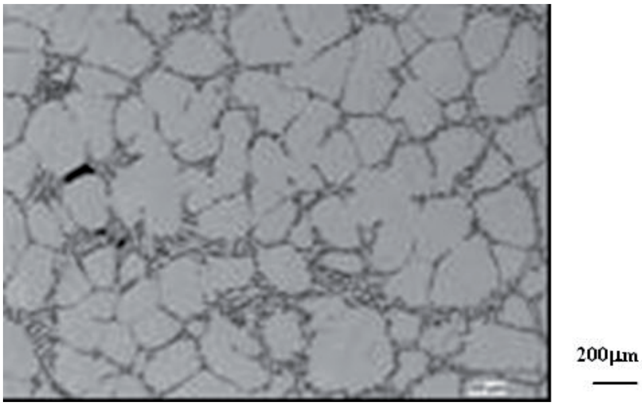

Fig. 9. Microstructure of A356 alloy obtained by electromagnetic stirring of the liquid while solidifying

Different mechanisms can promote crystal multiplication:

- remelting of roots of secondary dendrite arms – as described earlier in this work (section 3.1.), the difficulty of segregated solute from solidifying front to re-distribute in the liquid can promote necking of the crystal in regions close to surfaces where solute can be accumulate. In a dendritic formation, such regions are the roots of secondary arms. Once reducing the joining area between the secondary and primary dendrite arms, the first can easily be detached by remelting of such narrow region. This remelting occurs due the reduction of the local *liquidus* temperature or even due to a sudden flow of liquid at higher temperature in the local;
- detachment of necked secondary dendrite arms by mechanical effect - the shearing effect of liquid movements in front of the growing dendrites can be sufficient to break the dendrite arm which is weakly attached to the primary dendrite body due to the necking effect in its roots;
- detachment of dendrite arms by liquid penetration in high energy boundaries formed due to the bending effect - the liquid movement ahead of the solidification front could only promote bending of secondary dendrite arms instead of their total rupture. In this case, a boundary is formed separating the bent and not bent region. If the condition $\sigma_b > 2\,\sigma_{sl}$ (where σ_b is the energy of the boundary and σ_{sl} is the energy of a solid/liquid interface) is achieved, the requirement of minimum energy leads to liquid penetration in the boundary and detachment of the arm from the original dendrite. Figure 10 illustrates the mechanism.

The effect of re-heating the structure obtained by solidification under stirring promoted by electromagnetic field is shown in Figure 11.

It can be observed the globular morphology of the primary phase typical of thixocast material; also a significant growth of the primary phase particles is promoted by the re-heating imposed. However, the globules are more irregular related to dimensions and shape when compared to those obtained from raw material produced by cooling slope or chill techniques.

The higher irregularity of the solid particles in the semi-solid in this case is a consequence of the globularization phenomenon taking place in a structure formed by detached dendrite arms and remaining dendrite primary arms rather than in isolated near-globular particles of α primary phase.

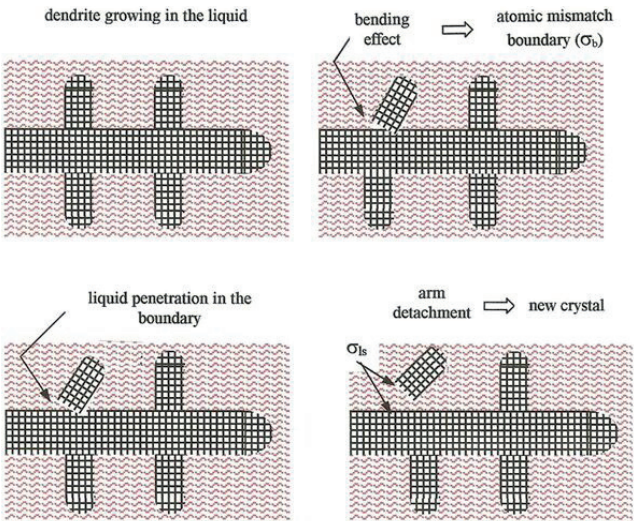

Fig. 10. Schematic illustration of detachment of secondary dendrite arms by liquid penetration in high energy boundary formed by bending due to stirring in the liquid

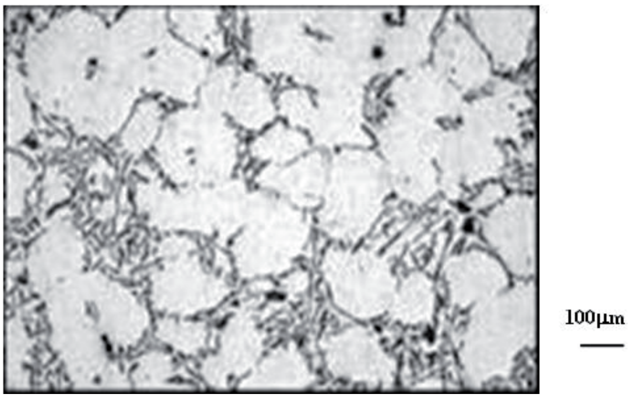

Fig. 11. Thixocast microstructure of A356 alloy produced by electromagnetic stirring + re-heating at 580°C/15min

Therefore, when increasing the initial nucleation rate by chilling effects promoted either in the mould wall or in a device that conduct the liquid to the mould, the structures produced contains fine, isolated grains. This structure is easily spheroidized in liquid presence at high temperatures resulting in slurries with more homogeneous and probably less interconnected particles of solid. On the other hand, semi-solid obtained by partial melting of structures produced by stirring methods, it meaning breaking of the growing solid, can present more interactions among solid particles. These interactions can mean the presence of particles agglomerates that can have strong influence in the flowing behaviour of the semi-solid.

3.4. Producing SS from solid state

If stirring of the liquid during dendritic growth can promote sufficient deformation in some regions of the solid to originate a boundary with high energy, the same condition can be promoted by submitting dendritic material to a certain degree of deformation in the solid state. This is the fundamental idea behind the trials to produce thixotropic semi-solid by simply heating up a deformed structure to temperatures within the solidification range.

During heating recrystallization can occur, leading to the formation of new grain boundaries with an associated energy. As the temperature reaches T*solidus*, secondary phases in original grain boundaries melt and the recrystallised structure is then surrounded by liquid. As already explained in section 3.3. and shown in Figure 11, if the condition $\sigma_b > 2\ \sigma_{sl}$ is achieved, liquid can penetrate in the newly formed grains and promote their detachment to the liquid where they can take globular geometry. If excessive growing is avoided, a fine, globular structure can be obtained; globules are probably more isolated than those formed by dendritic coarsening.

Figure 12 shows the structure evolution when heating the alloy A356 at 580°C, from a cold deformed condition – 80% of true deformation by compression was imposed.

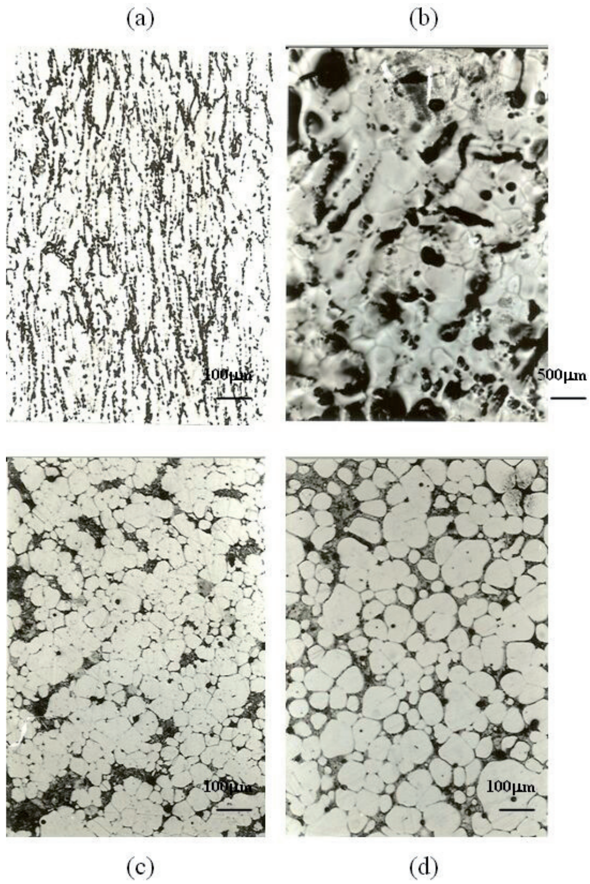

Fig. 12. Microstructures of A356 alloy in different conditions: (a) cold deformed (80% true def.); (b) heated at 580°C/2 min; (c) heated at 580°C/5 min; (d) heated at 580°C/15 min

The microstructures show clearly the re crystallization taking place in the deformed structure and the separation of re crystallised grains to form the globules of the solid phase in the semi-solid material. It can be observed the high kinetics of the mechanisms involved: recrystallization + liquid penetration + grain detachment + grain spheroidization in the liquid take place in about 5 min at the high temperature. Increase in holding time leads to growth of the solid particles, as observed. This growth can occurs by coalescence of particles in contact or Ostwald ripening and can jeopardize the flow behaviour of the slurry.

Unlikely the mechanical stirring process proposed in the early years of the semi-solid technology development [4], or the electromagnetic stirring commercially used in the production of rheocast Al alloys billets, or even in the cooling slope technique, when using partial melting of deformed structures to produce thixotropic slurries no special equipments are required. Thixotropic material can be easily produced by simply holding the alloy within the solidification range for times as short as 5 min, as observed. In addition, the globules obtained present high degree of spheroidization, small size and probably small degree of interactions among them.

Moreover, the process is more easily controlled, being the degree of initial deformation and the heating conditions the controlling parameters.

3.5. Flow behaviour – viscosity tests

Figure 13 shows results on viscosity measurements of semi-solid A356 alloy produced by re-heating of ultra-refined and electromagnetic stirred structures. It can be observed in both cases low values of apparent viscosity, indicating the forming ability of the alloy in the semi-solid state. The viscosity behaviour is clearly dependent on the shear rate, as expected for a non-newtonian fluid as the thixotropic slurry considered here.

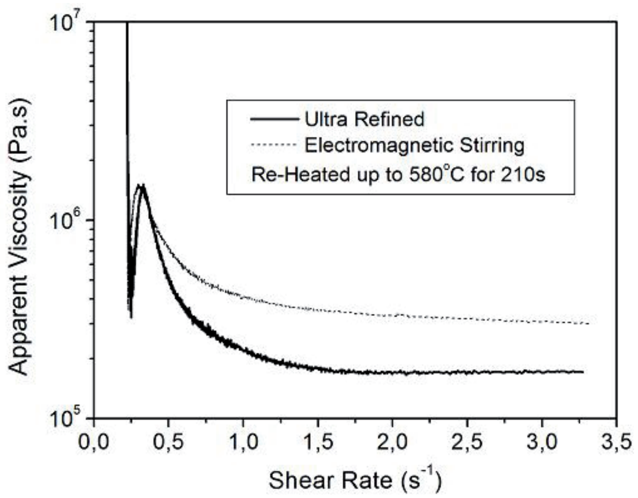

Fig. 13. Apparent viscosity x shear rate of A356 alloy in the semi-solid state produced from ultra-refined material (intensive initial nucleation) and from electromagnetic stirred material (fragmented dendrites)

Results also show that semi-solids produced by different routes present distinct flow behaviour: viscosity is higher for the slurry produced from electromagnetic stirred than the slurry produced from ultra-refined materials. Considering that at the testing conditions the semi-solids present similar globules sizes and solid fractions, as observed in their microstructures, the obtained results on viscosity can be explained by the influence of the solid globules interconnections.

To quantify the globules interactions, measurements of the contiguity factor (C) were made. This factor can be calculated using the equation proposed by Gurland [15], given by:

$$\frac{2N\alpha\alpha}{2N\alpha\alpha + N\alpha\beta} \qquad (3)$$

where $N\alpha\alpha$ is the number of $\alpha-\alpha$ intersections and $N\alpha\beta$ the number of $\alpha-\beta$ intersections; α and β are the primary and secondary phases respectively.

Results on contiguity values are 0.60 ± 0.16 in the slurry produced from stirred condition and 0.53 ± 0.02 in the slurry produced from ultra-refined material. These values indicate higher degree of globules interactions in the first case, where the globular morphology of α phase is a result of coarsening mechanisms acting upon fragmented dendrites.

On the other hand, when producing a semi-solid from refined material, where no dendritic formation was allowed in the first place, the resulting globules are more independent, once formed from individual crystals. Some degree of interaction can be expected as coalescence of touching globules with low degree of crystal misorientation can occur.

Therefore, results show that the processing route followed for the production of a semi-solid thixotropic material can play a significant role in its flowing behaviour. Processes interfering in the initial nucleation rate are more able to produce slurries with less agglomerated and interconnected solid phase and as a consequence, with low viscosity.

3.6. Flow behaviour – thixoforging tests

Figure 14 shows results on variation of applied force x time during thixoforging tests of semi-solid A356 alloy produced by the different techniques investigated.

It can be observed in all cases very low values of maximum forces required to complete filling of the mould with the material in the semi-solid state. The behaviour of the curves F x t are similar in all cases: there is no significant resistance to flow during the initial stages of the pressing operation, when material flows freely, with no friction with die walls. This first stage lasts for about 4s, when friction with walls starts to difficult the flow and forces start to increase. It can be observed that this second stage initiates earlier for the semi-solid produced from electromagnetic stirred raw material, indicating more difficulty for this slurry to flow, when compared to the other slurries produced from techniques not involving dendritic formation in the first place.

Values of maximum required forces are similar for slurries produced from raw material obtained by techniques that stimulate the formation of great number of nuclei in the structure during solidification or recrystallization. Among these techniques, it is not possible to distinguish results obtained for cooling slope and recrystallization. Both structures must present similar conditions of negligible globules interactions, resulting in the very small value of required maximum force.

A slight higher force, but still very low, is required to complete the mould filling when using semi-solid produced from raw material obtained by solidification in conditions such to promote intensive chilling. In this case, some dendritic growth could be present, if the chilling effect is not extended to the whole liquid. The presence of dendrites, even with fine dimensions, could result in less independent globules in the semi-solid.

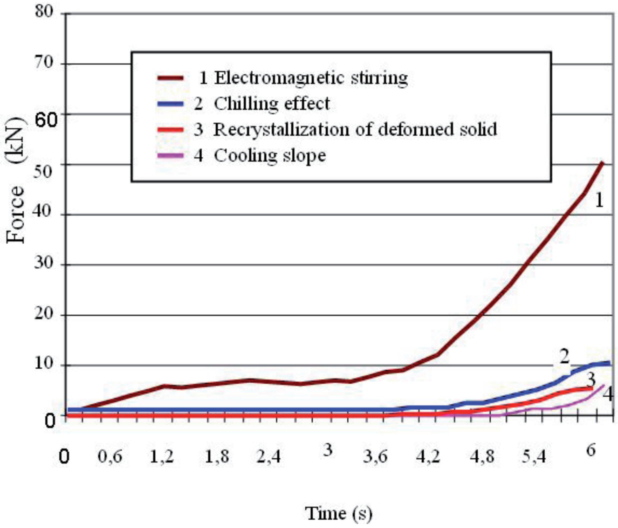

Fig. 14. Force x time during thixoforging of A356 alloy in the semi-solid state produced from raw material obtained by different techniques

Electromagnetic stirred material produces thixocast slurry with different behaviour, as observed. Applied forces already increases soon at the beginning of the forging process, in the first stage; then increasing rate decreases, characterising a second flow stage and finally rate increases again in a third stage. This behaviour is related to the internal structure of the slurry: the solid fraction is more interconnected, requiring forces at certain extent to break this interaction and to start the flow in the first stage. Once broken globules interconnections, flowing is facilitated, resulting in the reduction on the force rate gradient observed in the second stage of the curve F x t. However, as liquid is rejected from the flow front – a phenomenon observed in previous work [2], solid interaction in the slurry can build up again. This, together with the friction with die walls could result in the high increasing rate of force observed in the last stage of the forging operation.

4. Conclusions

Results show that the microstructure of a semi-solid thixotropic metallic alloy depends on the processing route followed for its production, mainly in the feature related to the interconnection of the globular solid particles of the primary phase. Processes interfering in the initial nucleation rate or recrystallization rate are more able to produce slurries with less agglomerated and interconnected solid phase (lower values of the contiguity factor parameter) than processes based on globularization of fragmented dendritic structures.

The flow behaviour of slurries produced by re-heating of different initial structures, evaluated by viscosity measurements and required forces for filling a mould in a thixoforging operation showed to be also dependent on the processing route followed to produce the semi-solid condition. Semi-solids produced by cooling slope, chill and recrystallization techniques presented better flowing behaviour than that produced by stirring technique. The result was attributed to the higher degree of solid globules interconnections in the semi-solid in the last case.

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