ANALYSIS OF MICROSTRUCTURES OF A5052 ALUMINIUM THIXOTROPIC SHEETS OBTAINED BY PARTIAL MELTING

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Abstract*. The semi solid process, or thixoforming, is one of the recent techniques in metal forming, that has been attracting much interest of the cientifical community and the industries in general. The slurries utilised in the semi solid process present time dependence of the viscosity, known as thixotropy. This work presents an analysis of the microstructures of A5052 aluminium thixotropic sheets. To obtain the thixotropic structures, rolled sheets were submitted to partial melting. The influence of thixocast temperature and holding time in the characteristics of the material, obtained by partial melting, was analysed. The parameters utilised were: 627ºC for 5 and 10 minutes, and 634ºC, for 5 and 15 minutes, in sheets of 0.8 mm; 624°C for 5 and 10 minutes, and 631°C for 5 and 15 minutes, in sheets of 2.0 mm; and 625, 630 and 635°C for 15 minutes in sheets of 4.0 mm thickness. The sheets had their microstructure characterised; results showed high homogeneity of the structure throughout the test piece. The variations of time and temperature were not significant to promote differences in the thixotropic structure.*

Keywords*: semi-solid, aluminium, microstructure.*

1. INTRODUCTION

The semisolid process, or thixoforming, is one of the recent techniques of metal forming that has attracted much interest in the scientific community as well as in the industries in general. The advantages presented for this process are already well known, like the production of parts near its final form (*near net shape*) or parts with more complex geometry in a unique stage

The semisolid process is based on the different microstructure that the rheo or thixocast material presents. A rheo or thixocast material, in the semisolid state, is constituted of globular solid dispersed in liquid. This characteristic gives to the semisolid slurries different viscosity of the slurries obtained by conventional solidification. For solid fractions of up to 60%, a behavior similar to the liquid can be obtained, under certain tensions. This makes possible casting processes, as injection and compression, to be carried through minor temperatures and less turbulence in the molds, when compared to the conventional casting (material in the liquid state). In rest, however, rheo or thixocast slurries can behave as solid, and are able to be handled. Rheo or thixocast semisolids, with high solid fraction, can be formed by mechanical processes as forging and extrusion, presenting the advantage to need lower tensions when compared to conventional processes in the solid state.

Due the importance of the microstructure, the microstructural evolution of the materials in the semisolid state has been widely studied, therefore characteristics as final globule size affects directly the viscosity properties, beyond important parameters in the thixoforming process, as heating cycle until the semisolid range, maintenance time in the partial melting temperature, among others. The present work brings a detailed study of the microstructures obtained in partial melting treatments, for the attainment of thixocast slurries, at different times and temperatures.

2. EXPERIMENTAL PROCEDURE

As raw material for the production of thixotropic material, sheets of 0.8 and 2.0 mm thickness of A5052 (aluminum-magnesium alloy) submitted to H36 treatment, and sheets of 4.0 mm of the same alloy, submitted to treatment O, were used. The H36 nomenclature means that the alloy was cold worked with area reduction of 75% during the rolling process, and after that, it was stabilized. The nomenclature O means that the alloy was annealed after rolling.

The sheets initially were characterized in relation to their microstructure. For that, samples for metallographic analysis were prepared following the procedure: grinding in abrasive papers of 220, 320, 400, 600, 800 and 1200 mesh; polishing with diamond paste of 6, 1 and 0.25 μm; etching in solution: 15 ml HF, 10 ml H3PO4 and 60 ml of distilled water, for approximately 20s. The samples were analyzed in optical microscope Neophot 32/Zeiss and in scanning electron microscope JEOL in which microanalysis (EDS) were carried through for determination of the composition of the intermetallic phases.

2.1. Differential thermal analysis

Differential thermal analysis was used in this work for the determination of temperatures *solidus* and *liquidus* of the alloy, to know these temperatures is essential for working in the semisolid state. This analysis was realized in samples of sheets with 0.8; 2.0 and 4.0 mm thickness, in an equipment NETZSCH model STA 409. The samples were heated, in a controlled way, from 350 to 750ºC, in a velocity of 5ºC/min. In the cooling, it was used the same velocity of 5ºC/min, with a controlled temperature since 750 until 400ºC.

2.2. Samples dimensions

Samples were cut from sheets, with the dimensions indicated in Table 1, for thixocasting, that is, to obtain the thixotropic globular structures and posterior microstructural analysis.

Table 1 - Dimensions of samples prepared for thixocasting of A5052 alloy, from sheets with different thicknesses.

	Sheets thickness		
Dimensions of samples (mm)	0.8 mm	2.0 mm	4.0 mm
	100x100; 200x200	200x200	100x100

2.3. Method used to obtain thixotropic structures

To obtain sheets with thixotropic globular structure, it was employed the method of controlled partial melting. In this method, the material is submitted to thermal treatments in a temperature superior to *solidus* temperature, in order to promote the secondary phases melting and globularization of the primary phase.

Based on the values of the temperatures *solidus* and *liquidus* obtained by differential thermal analysis, of the chosen alloy, different temperatures of treatment, for different times, were tested. It was searched to obtain globular structures with different globule sizes. Samples of rolled sheets (thickness of 0.8 and 2.0 mm) and of annealed sheets (thickness of 4.0 mm) were submitted to controlled partial melting.

The sheets of thickness 0.8 mm were treated at 627ºC during 5 and 15 minutes, and at 634°C during 5 and 10 minutes. These temperatures correspond to 44 and 62% of liquid fraction in the slurry, respectively. For sheets of 2.0 mm thickness, experiments were made at 624ºC during 5 and 15 minutes, and 631ºC during 5 and 10 minutes. These temperatures correspond, respectively, to 45 and 64% of liquid fraction. The samples of sheets of 4.0 mm were treated at 1 temperature: 630ºC during 15 minutes. This temperature corresponds to 21% of liquid fraction.

2.4. Equipments used for treatment of partial melting

Due the different sample sizes (100 x 100 mm and 200 x 200 mm), different furnaces and devices especially constructed for the introduction and maintenance of the samples in its interior were used, during treatment of controlled partial melting.

For thermal treatments of samples 100 x 100 mm, it was used a resistive furnace, programmable and with digital control of temperatures. The samples were inserted in the furnace with aid of a device especially constructed to prevent its deformation or warping during the treatment. Such device consists of 2 steel plates of 120 x 120 mm and thickness 5.0 mm, held in one of the sides, as indicated in Figure 1.

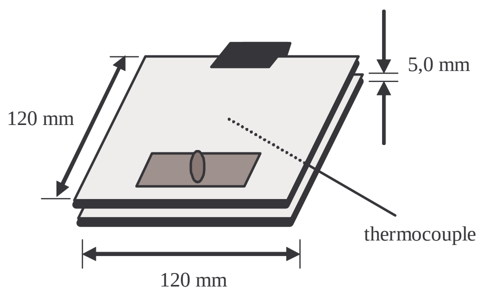

Figure 1 – Device used for the thermal treatments of controlled partial melting in samples of 100 x 100 mm.

For thermal treatments of samples 200 x 200 mm, another furnace, also resistive, was used and especially adapted for these experiments. The furnace, rectangular and leaked in the center, is presented in Figure 2. The resistances are located in all the walls. The chamber openings were closed with isolating plates.

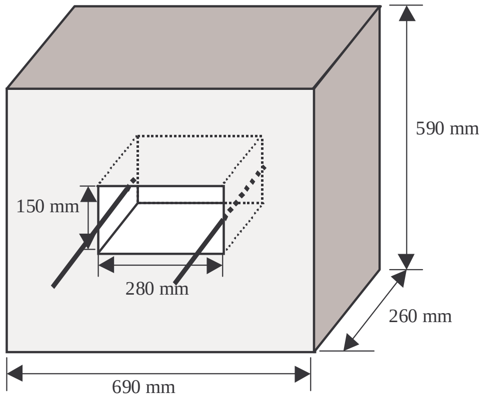

Figure 2 - Scheme of the furnace used in the preparation of thixocast sheets: dimensions 200 x 200 mm.

2.5. Experimental procedure for the obtention of thixotropic structure

The procedures used for the experiments of partial melting were similar, independent of the furnace utilized, that is, independent of the sample's dimensions. Initially the furnace was heated until the desired temperature of treatment, measured with a thermocouple located in the center of the sample. When the temperature was reached, the sample was introduced, in the interior of the steel plates. At this moment, it was initiated the injection of argon in the interior of the furnace. During the treatment, the temperature was monitored for a thermocouple type K, located at the center of the sample. At the end of the established time, the device was removed and the sample cooled in water.

2.6. Metallography of thixocast sheets

For each tested condition (temperature and time), the sheets were cut in 8 regions, and observed in optical microscope Neophot 32/Zeiss. One objective was to characterize the homogeneity of the structure in all the extension of the sheet. The metallographic technique was similar to that used for the rolled and annealed samples. The microstructures were characterized by the following parameters: average diameter of the globular phase and relative amount of secondary phase. These parameters were measured through the program of analysis of images Leica Q500MC connected to the optical microscope Neophot 32/Zeiss.

3. RESULTS AND DISCUSSIONS

3.1. Initial microstructures

Figure 3 presents typical microstructure of sheets 0.8 mm in the rolled state. It can be observed that the microstructure is formed by a matrix of the phase α and by intermetallic phases elongated and oriented in the rolling direction. The composition of the intermetallic phases was analyzed by scanning electron microscope. It was observed that the majority of them is formed by a great amount of iron, around 20 to 30%, and other elements as silicon and chromium.

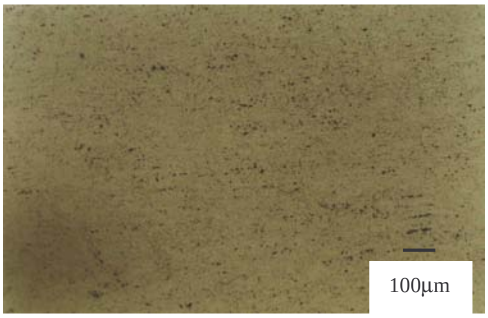

Figure 3 – Microstructure of the sheet 0.8 mm, alloy A5052, in the rolled state.

The microstructure of sheet 2.0 mm, in the rolled state, can be observed in the Figure 4. The sheets of 2.0 mm were acquired with the same treatment, that is, H36, as described previously, and can be observed that the structure is similar to the sheet 0.8 mm thickness. In this structure, the grain boundaries are more visible and present certain orientation. The secondary phases present less orientation. As well as in the sheet of 0.8 mm, the intermetallic phases were analyzed in relation to the composition and the results were similar, however with the phases presenting greater percentage of iron, varying of 40 to 50%, and it was also detected the presence of manganese and chromium.

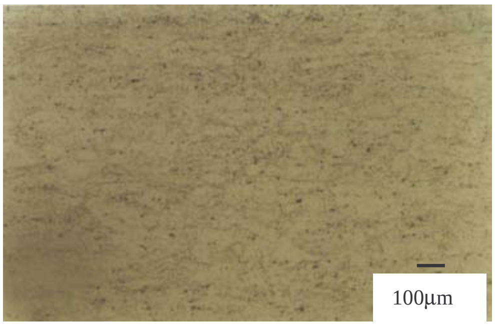

Figure 4 - Microstructure of the sheet 2.0 mm, alloy A5052, in the rolled state.

The microstructure of the sheet 4.0 mm thickness (Figure 5), in the annealed state, differs from the other sheets, thickness 0.8 and 2.0 mm. The grains are clearly visible and recrystallized: they do not present preferential orientation. Great amount of intermetallic phases in the sample is also observed, with similar composition to the samples of 0.8 and 2.0 mm, that is, phases with high percentage of iron and small presence of other elements as manganese, silicon and chromium.

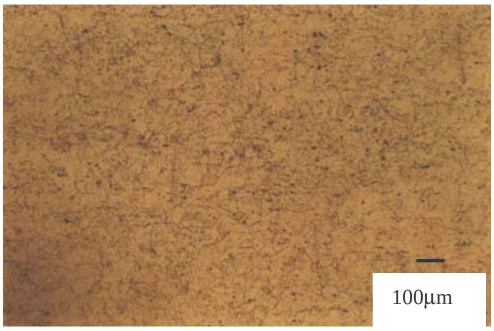

Figure 5 - Microstructure of the sheet 4.0 mm, alloy A5052, in the annealed state.

3.2. Thixocast structures

It only will be presented the microstructures of a sample treated at 634ºC, during 10 minutes, thickness 0.8 mm; of a sample treated at 624ºC, during 5 minutes, thickness 2.0 mm; and of a sample treated at 630ºC during 15 minutes, thickness 4.0 mm, because it is not viable to present all the microstructures obtained. A Table with the results of all the parameters obtained by the microstructures will be presented too.

3.2.1. Thixocast structures obtained in sheets 0.8 mm thickness and dimensions 200 x 200mm

Thixocast samples, submitted to different conditions of temperature/time, were cut for analysis of its structure in different regions. It was searched to analyze the homogeneity of the structure produced in the thixocasting treatments.

The Figure 6 presents the regions analyzed in a sheet 200 x 200 mm. The distances of (a) and (b) of the edges of the sheet are equal to the distances of (d) and (c); (e) and (f); (h) and (g), respectively.

The structures obtained at 634ºC during 10 minutes are presented in the Figure 7. It was observed, that for these conditions, the structure is already totally modified, formed by primary globular phase α, involved for a second phase, that is formed by precipitation in the solid state at the regions of α enriched of Mg.

The high velocity of cooling imposed in thixocasting can make possible the precipitation of secondary phase in the boundaries of the primary phase, at temperatures superior to the line *solvus* of the phase diagram. Thus, at ambient temperature, it is possible to have greater amount of secondary phase that previewed in conditions of slow cooling.

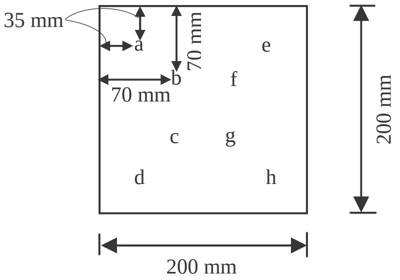

Figure 6 - Representative scheme of the regions of analysis of the thixocast sheet’s microstructures, dimensions 200 x 200 mm.

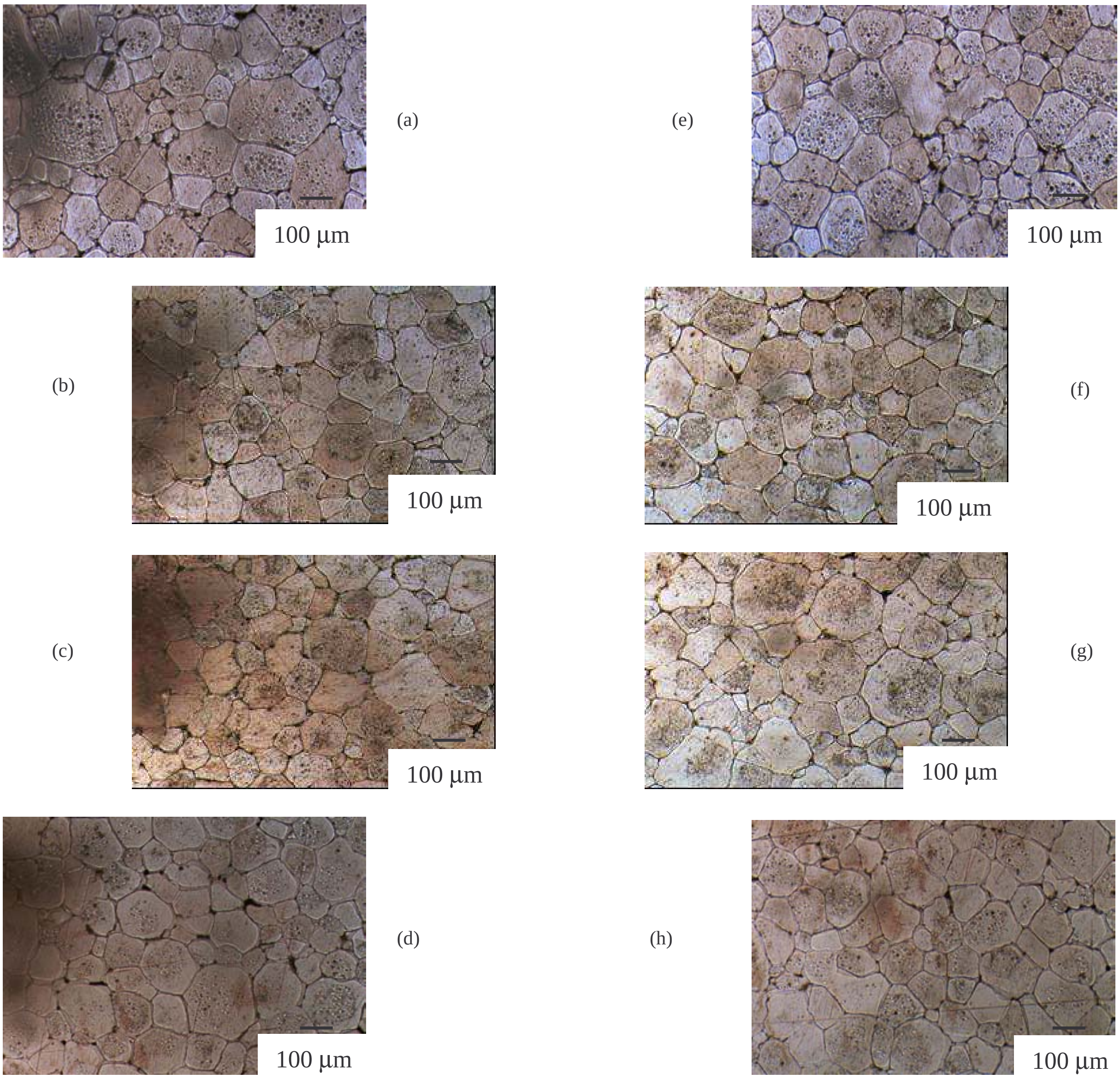

Figure 7 – Typical microstructures of a A5052 aluminium alloy sheet, 0.8 mm thickness, treated at 634ºC during 10 minutes, in different regions. Sample with dimensions 200 x 200 mm, (a-h) regions of analysis.

Observing the microstructures of Figure 7, some considerations can be made. In a generalized manner, the regions are similar, proving the homogeneity of the structure throughout the sample. The size of globules varies in a analyzed region, it can be seen globules of 20 to 120 μm in a same region. Some regions are, apparently, more globular than others, and also seem to present less dispersion in the size of globules. It was observed the formation of secondary phase in the globule boundaries, in all regions. It is possible to observe, in all regions, the presence of secondary phase, in the globules interior, in the format of small globules. The origin of them can be attributed to liquid retention during the globularization process of the structure. This liquid, when fastly solidified, was transformed into a phase α, saturated of Mg, that precipitates in the form of Mg_5Al_8, when reaches the line *solvus*. This phenomenon is common in thixocast structures.

The transformation of the original structure from deformed to globular occurs through a sequence of events: recovery and internal recrystallization of the deformed structure, with the subsequent formation of low angle boundaries; liquid formation around the boundaries; "wetting" of the recrystallized boundaries and separation of them by the liquid penetration, and finally the growth of these isolated grains in the liquid.

If the boundary formed presents high energy, that is, if the superficial energy of the grain boundary is superior to twice the superficial energy solide/liquide, the system tends to reduce the internal energy promoting the penetration of liquid, destroying the boundaries and forming two new surfaces solid/liquid. In this way, the recrystallized grain is separated and starts to grow equiaxially in the liquid, constituting the thixocast slurry.

The results of average globular size and secondary phase fraction are presented graphically in Figures 8 and 9.

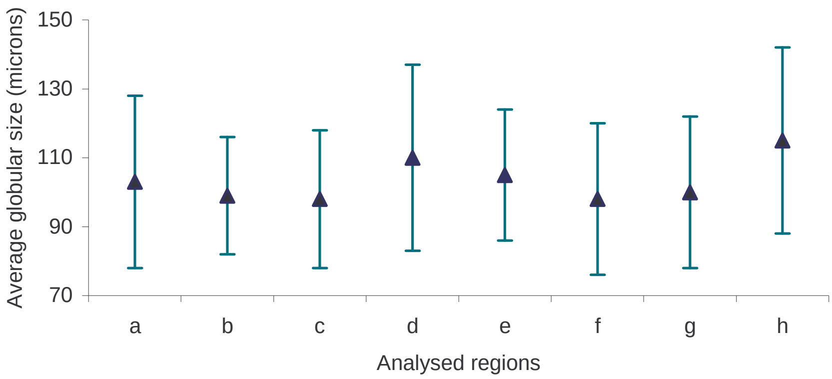

Figure 8 - Values of the average globular diameter in different regions of sample 200 x 200 mm; 0.8 mm thickness, thixocast at 634ºC during 10 minutes.

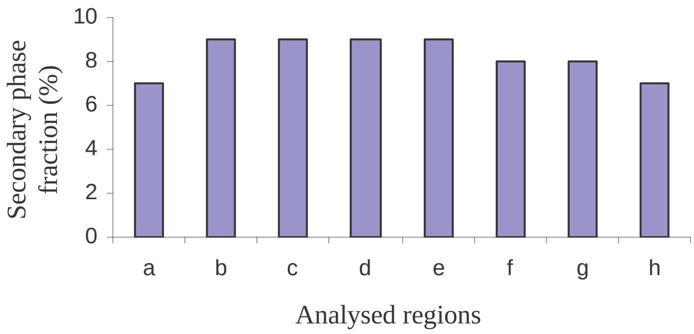

Figure 9 - Values of the secondary phase fraction in different regions of sample 200 x 200 mm; 0.8 mm thickness, thixocast at 634ºC during 10 minutes.

Analyzing the graphic of Figure 8, the homogeneity of the structure is observed, with the average diameters varying from 98 to 115 μm in all regions, the greatest diameters obtained in all the treatments for this thickness and dimensions of sample. It occurred a small increase in the average globular size in relation to the treatment at the same temperature with less maintenance time. The increase of the time carried to the globules coalescence and consequent increase of size. An increase in the dispersion of dimensions values is also observed, probably due to the contact between globules.

The increase of the globular size in slurries rheo or thixocast maintened at high temperatures is attributed to the mechanisms of coalescence and "Ostwald ripening". The coalescence phenomenon leads to the formation of a globule with greater dimensions from two or more globules with minor dimensions. The globules, in contact, tend to lose the separation boundaries by diffusion. The mechanism of Ostwald, however, promotes the growth of greater globules from minor globules.

The minor globules tend to liberate solute that migrates in the liquid and is incorporated by the greater globules. For high solid fractions in the slurry, as in the present case, mechanisms of coalescence are preponderant and they lead to a increase in the dispersion values of the average globule diameter, as observed by Robert, 1993.

3.2.2. Thixocast structures obtained in sheets 2.0 mm thickness and dimensions 200 x 200mm

The microstructures obtained in the treatments at 624ºC during 5 minutes are presented in Figure 10.

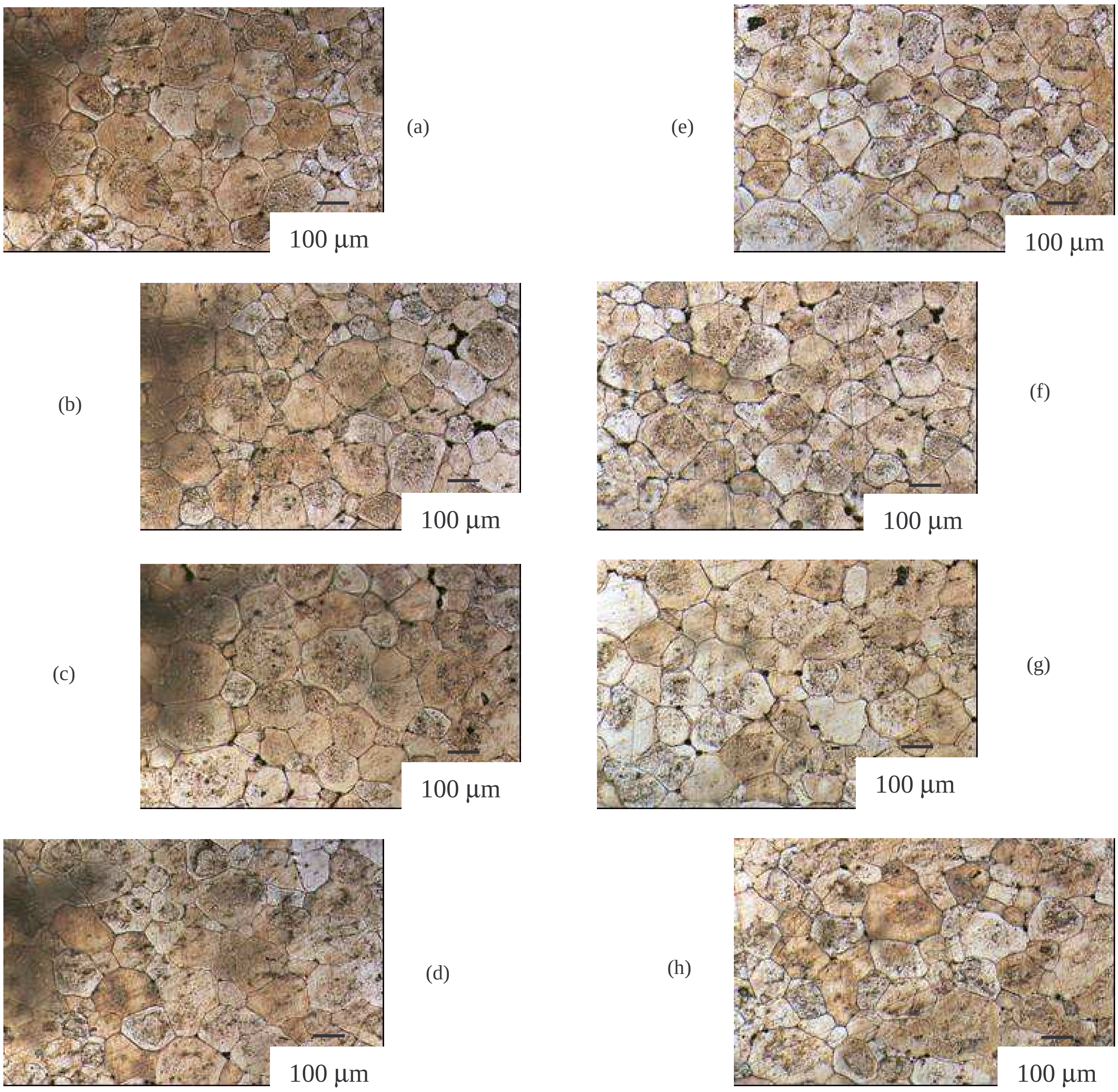

Figure 10 – Typical microstructures of a A5052 aluminium alloy sheet, 2.0 mm thickness, treated at 624ºC during 5 minutes, in different regions. Sample with dimensions 200 x 200 mm, (a-h) regions of analysis.

It can be observed a structure formed by globules of the primary phase α, involved by a second phase, similar to the structures obtained in sheets 0.8 mm thickness. The globules present certain variation of size in a determined region, but the regions are very similar. The structure presents homogeneity in all regions analyzed. A small formation of pores can be noticed. This structure is similar to the structures obtained in the previous treatments with sheets 0.8 mm thickness, confirming the good results with the controlled partial melting

3.2.3. Thixocast structures obtained in sheets 4.0 mm thickness and dimensions 100 x 100mm

In order to verify the structural homogeneity in thixocast sheets of dimensions 100 x 100 mm, samples of the treated sheets were removed, in different regions, as indicated in Figure 11. The distances of (b), (c) and (d) to the edges of the sheet are equal to the distances of (a) to the edges.

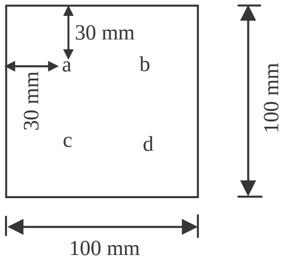

Figure 11 - Representative scheme of the regions of analysis of the thixocast sheet’s microstructures, dimensions 100 x 100 mm.

The microstructures of the different analyzed regions are presented in Figure 12. Typical globular structure of thixocast material can be observed. The globules present size variation in a determined region and they are in the same range of globules diameter obtained in the structures of sheet 0.8 and 2.0 mm thickness. They also present small globules of secondary phase in the interior of the primary phase. Comparing the diverse regions, great homogeneity in the sample is observed. The pores formation is not observed.

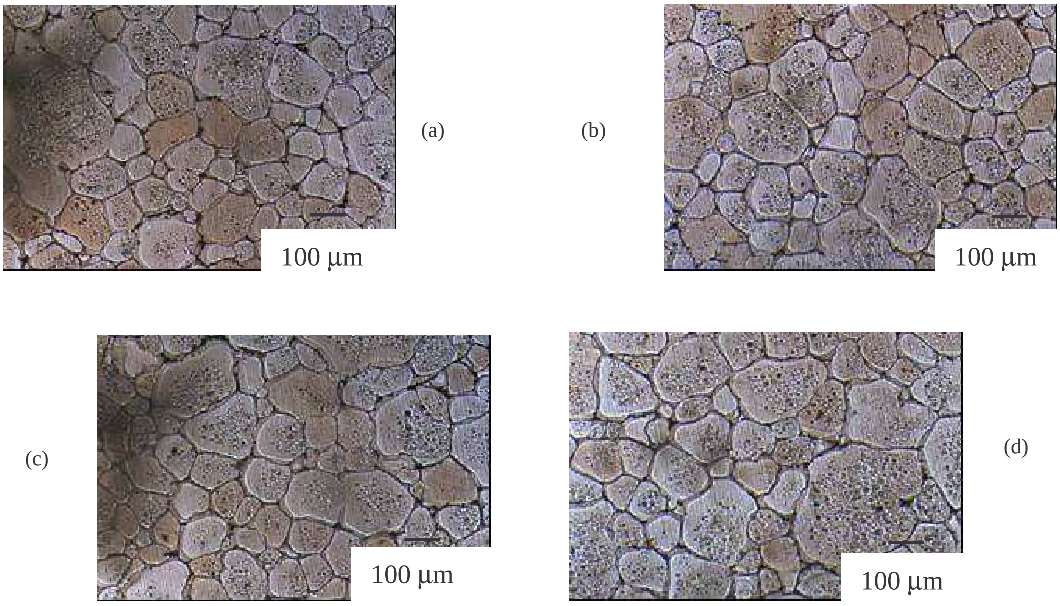

Figure 12 – Typical microstructures of a A5052 aluminium alloy sheet, 4.0 mm thickness, treated at 630ºC during 10 minutes, in different regions. Sample with dimensions 100 x 100 mm, (a-d) regions of analysis.

3.3. General results about the production of thixocast sheets

The Table 2 presents the values of each parameter studied (average globule diameter and secondary phase fraction) in function of the thickness and dimensions of the sample and the conditions of thixocasting.

In relation to sheets with greater dimensions (200 x 200 mm), the homogeneity of the structure is particularly interesting; therefore it proves the viability of the production of homogeneous thixocast materials in great parts, with care of the uniformity of temperature distribution. Small formation of pores was observed, although the furnace atmosphere had been controlled with argon. Two reasons are probable: due to the size of the furnace, the control of the atmosphere was not so efficient, and the proper contraction of the liquid phase when transformed to solid leading to pores formation.

Analyzing the studied parameters of thixocasting, time and temperature, it can be observed that, in the ranges of values used in this work, they do not have significant influence in the size of globules. It could be observed a trend of the greater times of treatment leading to greater globules, with exception to the treatment made in sheets 0.8 mm thickness, temperature 627ºC, whose the time of 5 minutes must not have been enough for all liquid formation, leading to a reduction in the size of the globule with the increase of time.

In relation to the temperature, it can be observed a very small trend of greater temperatures leading to greater globules, maybe by the stimulation to phenomena of mass transport needed to thickening mechanisms; the thickening by boundaries coalescence is preponderant when high solid fractions are present, as in the case of the produced samples.

Table 2 - Obtained values of average globule diameter and secondary phase fraction in function of the thickness and dimensions of the sample and the conditions of thixocasting.

Thickness and dimensions of the samples (mm)	Thixocasting conditions (temperature and time)	Average globular diameter (μm)	Secondary phase fraction (%)
0.8 - 200 x 200	627°C / 5 minutes	90 ± 3	7
	627°C / 15 minutes	88 ± 3	7
	634°C / 5 minutes	94 ± 3	10
	634°C / 10 minutes	103 ± 6	8
2.0 - 200 x 200	624°C / 5 minutes	98 ± 4	9
	624°C / 15 minutes	101 ± 3	9
	631°C / 5 minutes	97 ± 4	9
	631°C / 10 minutes	103 ± 5	9
0.8 - 100 x 100	627°C / 15 minutes	87 ± 6	9
	634°C / 10 minutes	112 ± 9	11
4.0 - 100 x 100	630°C / 15 minutes	112 ± 7	12

Obs.: The dispersion values of the average globular diameter relate to the dispersion of the average values in each case, not between the measured diameters.

A certain difference in the dispersion values of the globules size is observed, when comparing the sheets 0.8 and 2.0 mm thickness, as evidenced in the measures presented in Table 2. The sheets 2.0 mm thickness, in all the times and temperatures studied, present in a generalized manner, dispersion values of globule diameter greater than the values of sheets 0.8 mm thickness, perhaps by greater temperature variation in the interior of sheets with greater thickness.

The secondary phase fraction, in all the cases, was superior to the previewed theoretically by the phase diagram, probably due to difficulty of Mg dissolution in the phase α during the fast cooling. The high gradients of solute in the phase α could lead to the precipitation of secondary phases at higher temperatures, that is, the fast cooling could deviate the *solvus* line to the left.

In relation to samples 0.8 mm thickness and dimensions 100 x 100 mm, it can be observed that very small modifications happened, in comparison to the samples with greater dimensions, obtained in the same conditions. But a trend to the increase of the average globule diameter and the secondary phase fraction in the thixocast structure obtained at 634ºC during 10 minutes, was observed. These values can be° a result of better control of the temperature in the samples, due to smaller dimensions of the samples and the furnace too.

According to Pires, 1998, the influence of time and temperature in the obtention of thixocast structures are interdependent. Short times at temperatures next to *solidus* are not sufficient for all liquid formation, influencing in the final globules diameter. At higher temperatures, shorter times are indicated, therefore greater times lead to an excessive growth of globules, characteristic not desired. In the present work, the variations of time and temperature had not been significant to promote differences in the thixocast structure.

4. CONCLUSIONS

In a generalized manner, the results obtained are very promising, indicating the production possibility of thixocast structures in sheets. The thixocasting of sheets had not been done until the moment due to difficulty of integrity maintenance of the material in the mushy state, mainly in the case of sheets with small thickness. Another difficulty would be related to the distribution of temperature in the sheet, which could lead to a heterogeneous structure. The results obtained in sheets of different thicknesses show the possibility of semi-solid sheets obtention as well as homogeneous structure throughout the sheet.

The results show that a small variation of process parameters (time/thixocasting temperature; thickness and dimensions of sheets) did not produced important distinctions between the thixocast structures.

5. REFERENCES

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