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PRODUCTION OF RHEOCAST SLURRIES BY PARTIAL MELTING THROUGH ALTERNATIVE THERMOMECHANICAL TREATMENTS

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Abstract. *The production of alloys with rheocast structure, in which the primary phase consists of globular particles, by partial melting, involves grain recrystallization and secondary phases melting. In the process known as SIMA (Strain Induced Melt Activation), the raw material is cold deformed by rolling in two steps, making it less efficient. This work investigates the production of rheocast Al-3.35wt%Cu alloy by alternative thermomechanical treatments. Two treatments are performed: Recrystallization and Partial Melting (RAP) and Overaging (OAT) processes. In the first, as-cast dendritic alloy is cold-deformed at room temperature and then heated to a constant temperature in the mushy zone, to obtain the rheocast structure. In the second, the alloy undergoes a solution and precipitation heat treatments, before cold-deformation, with the aim of obtaining precipitated particles of secondary phase with size and interparticle spacing favourable to increase the nucleation rate of recrystallized grains during heating of the alloy. Results show that the two routes produce globular structures, with grain size reduction of 700 and 900% for RAP and OAT, respectively, with respect to initial grain sizes. In general, OAT process results in more rounded and smaller globules in the rheocast structure than RAP.*

Keywords*: semi-solid alloys, rheocasting, thermomechanical treatments, aluminium alloys.*

1. INTRODUCTION

The increasing tendency for near-net-shape forming of industrial parts, taking advantage of cost reduction and quality improvement, is turning thixoforming and thixocasting into viable technologies - which includes forging, injection and extrusion moulding (Kenney et al, 1988), and with possibilities in deep drawing (Adamiak & Robert, 1998) - for light alloys, steel and tool steel.

Although the widely stated potential of thixoforming processes, industrial production of thixotropic materials is limited to the electromagnetic stirring process, in which the molten alloy undergoes shear stresses during solidification, resulting in a microstructure of fragmented dendrites and rosette shaped particles, which must be submitted to heat treatment to promote the required spheroidization. Other proposed processes like mechanical stirring (Flemings *et* *al.*, 1976), SCR process (Kiuchi & Sugiyama, 1992) and passive stirring (Fiorini *et al.*, 1994) were not competitive because of lower productivity or difficulties in the process control.

Another approach for the production of rheocast alloys is the solid state route, by partial melting of deformed dendritic materials (Robert & Kirkwood, 1988), where the deformed material is heated up so that recrystallization takes place before reaching the semi-solid temperature range. At temperatures higher than *solidus*, the liquid formed can penetrate in recrystallized grain boundaries, causing grains detachment and resulting in a spheroidized microstructure. However, when the holding time is excessive, coarsening phenomena gives rise to undesirable grain growth. The major advantage of this route is that it neither requires expensive equipment nor posterior globularization heat treatments. In the method patented as SIMA (Young et al, 1983) cast material is extruded at high temperature to fragment the dendritic structure and then cold-deformed above a critical strain, before the heating step.

The purpose of this work is to study the behaviour of an Al-Cu alloy after being submitted to simpler thermomechanical treatments, which involve only one deformation step, and to analyse the influence of initial grain size, ageing conditions and cold work level on the final characteristics of the resulting rheocast. According to Loué & Suéry (1995), the initial grain size shouldn't affect the morphology and final grain size of rheocast slurries, provided that enough strain has been supplied. On the other hand, working with Al-4.5wt%Cu alloy, Zoqui & Robert (1998) obtained results that show a dependence between initial and final grain sizes in rheocast produced from raw material submitted to 40 % of true strain.

The main parameter that influences the final microstructure of a rheocast material is thought to be the degree of cold deformation. If a critical value is surpassed, over which there is dislocation density saturation, the morphology and final grain size are independent on strain level (Robert & Kirkwood, 1988) in the raw material.

2. EXPERIMENTAL PROCEDURES

An aluminium-copper alloy, whose chemical composition is shown in Table 1, with *Solidus* and *Liquidus* temperatures of 906 K and 915 K, respectively, determined by DTA, was chilled and sand cast to produce samples with two different initial grain sizes.

Table 1**.** Chemical composition of Al-Cu alloy utilised in the experiments.

Element	Cu	Si	Mg	Fe	Mn	Zn	Ni	Ti	Pb	Sn
Content (wt%)	3.35	0.06	0.005	0.08	0.001	0.001	0.01	0.008	0.005	0.001

Part of the samples has undergone solution, at 820 K for 2 h, and precipitation, at 653 K, heat treatments. The coarse grained alloy was heat treated for 6 h and the fine grained alloy for 25 h, for precipitation. Such parameters were determined according to results obtained in preliminary experiments of recrystallization of overaged samples, on searching for the times that provided smaller grain sizes. Figure 1 shows recrystallized grain sizes obtained from coarse and fine raw material with respect to precipitation times. It can be observed that the curve for coarse alloy reaches a minimum after 6 h of heat treatment, while the recrystallized grain size produced from refined alloy decreases continuously up to 25 h of treatment. No longer treatment times were investigated.

Samples with dendritic and overaged microstructures were then submitted to 45 % and 80 % of true strain by compression and to rheocasting by partial melting at the constant temperature of 908 K. Average heating rate was .92 K/s. There was no holding time at this temperature, so that samples were chilled as soon as they reached it. Schemes of the thermomechanical treatments employed are shown in Fig. 2.

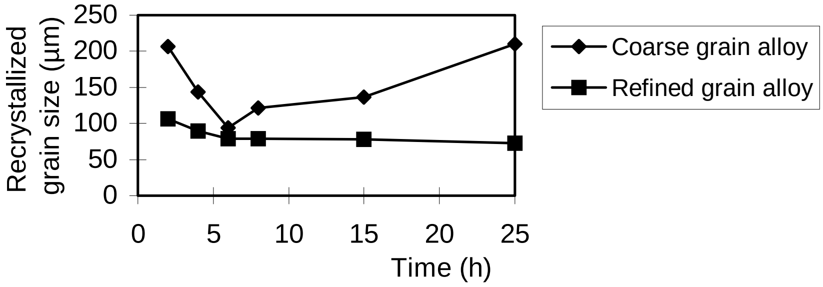

Figure 1 - Average diameter of recrystallized grains obtained from coarse and refined alloys treated at 893 K, with respect to overaging time.

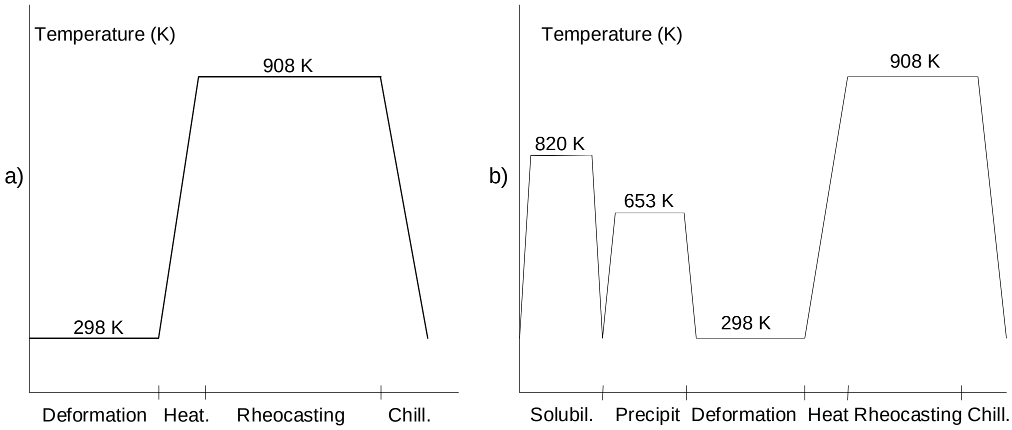

Figure 2 - Schematic drawing showing the thermomechanical treatments employed: a) recrystallization and partial melting (RAP); b) overaging treatment (OAT).

Optical and scanning microscopy were used to evaluate results. Microprobe analysis was carried out in as-cast and rheocast structures. Quantitative metallography was applied to measure grain sizes, while shape factor, defined as the factor between the larger and the least dimensions of each particle, and solid fraction were quantified by image analysis.

3. RESULTS AND DISCUSSION

Fig. 3 shows macro and microstructures of sand and chilled cast samples of Al-3.35wt% produced as raw material for rheocast experiments. Fully equiaxial grains are observed in both cases, with typical dendritic microstructure. Due to the different cooling conditions employed, grain sizes and secondary arm spacing differ. These values are presented in Table 2.

Table 2 - Grain sizes and secondary arm spacing values of as-cast Al-3.35 %Cu structures obtained in different moulds.

Mould type	Grain size (µm)	Sec. spacing (µm)
Chill-cast	403 ± 35	40 ± 6
Sand-cast	1290 ± 125	102 ± 10

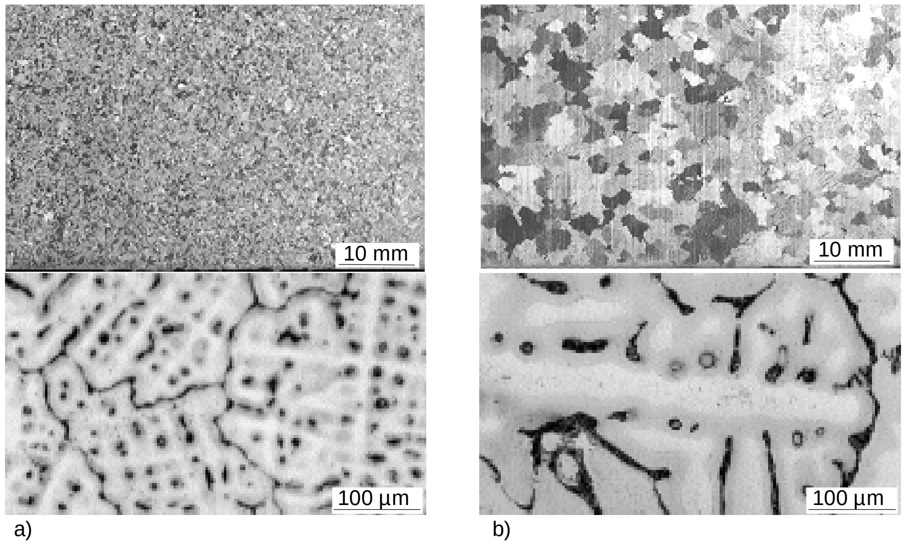

Figure 3 - Macro and microstructures of Al-3,35%Cu alloy: a) chill-cast; b) sand-cast.

Microssegregation of Cu due to solute rejection during solidification can be observed through microprobe analysis across dendritic arms of the as-cast alloy. Figure 4 shows a SEM micrography of the coarse alloy, showing the points of microanalysis, and distribution profiles for Cu. The average Cu contents in primary phase is 3.2 ± .1 %, with maximum and minimum values of 5.0 % and 1.62 % respectively. Applying a distribution index suggested by Pires (1998), a result of 61 is obtained, which attests the great heterogeneity of Cu distribution in a dendritic alloy.

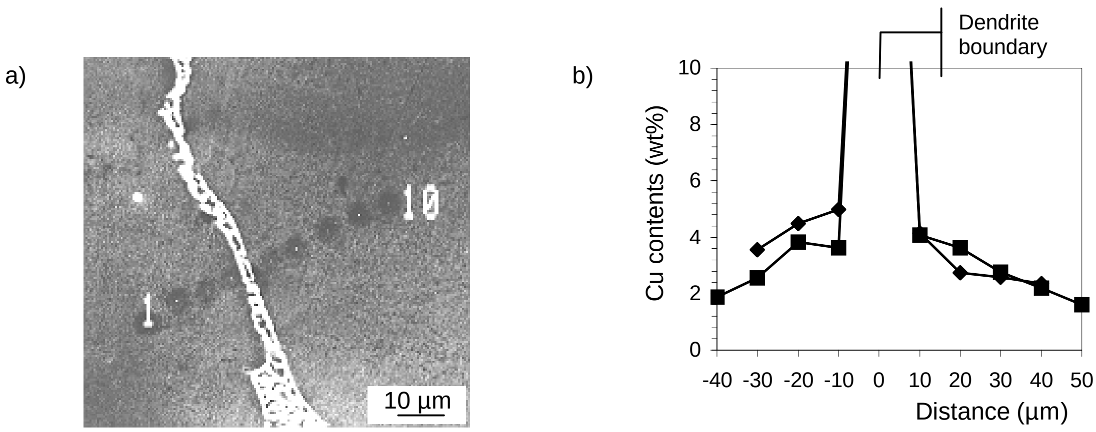

Figure 4 - Microanalysis of as-cast Al-3,35%wtCu coarse alloy: a) typical SEM micrography showing points of analysis; b) distribution profiles of Cu with respect to the distance from dendrite boundary (point 0).

One of the microstructural features of rheocast alloys is the low microssegregation within globular primary phase, due to higher diffusion rates in the temperatures required for partial melting. Figure 5 shows Cu distribution in rheocast structures obtained by RAP and OAT with respect to the distance from grain boundaries (point 0). A flat profile inside the globules can be observed. The average Cu contents in the primary phase of the OAT rheocast is 1.9 % and 1.5 % in the RAP, lower than in the as-cast alloy because of the presence of approximately 25 % of liquid, where its solubility is higher. The distribution index provides values of 37 and 28 for RAP and OAT rheocasts, respectively, much lower than the index found for the as-cast alloy.

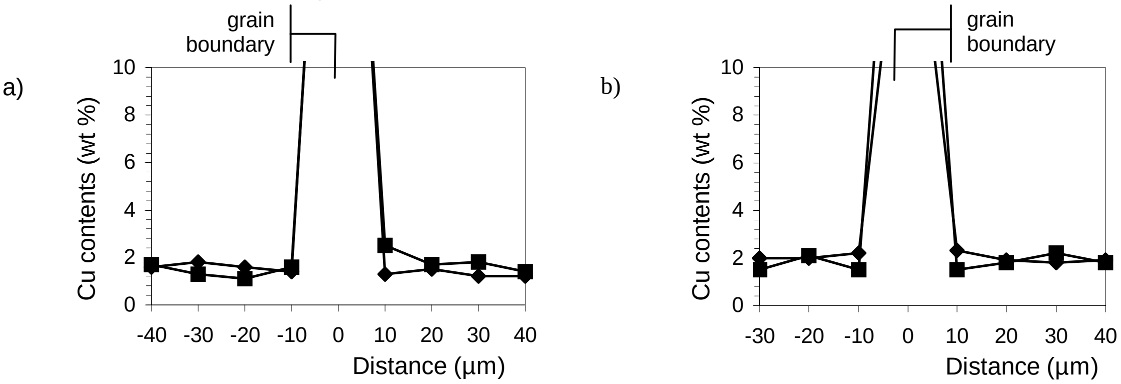

Figure 5 - Cu contents in the primary phase of rheocast alloys with respect to distance from grain boundaries (point 0): a) RAP, b) OAT.

3.1 RAP thermomechanical treatment

Typical microstructures of Al-3.35wt%Cu alloy rheocast samples, obtained by RAP process, starting from different conditions, are presented in Fig. 6.

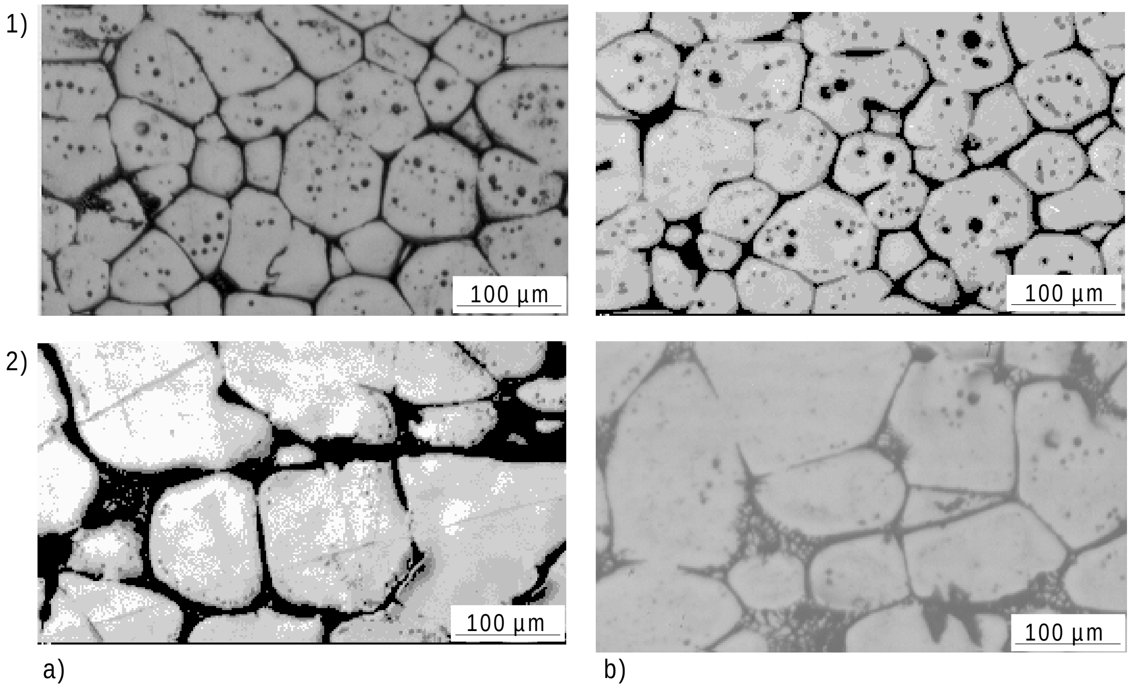

Figure 6 - Rheocast microstructures obtained by RAP: 1) from originally refined alloy; 2) from originally coarse alloy; a) submitted previously to 45 % true strain; b) submitted previously to 80% true strain.

Structures obtained from initially refined alloy show globular primary Al-α, even for less deformed material, with average 1,5 %Cu in solution and entrapped pools of $CuAl_2$. Fe and Si, the main impurities, were found to be concentrated in pools and in the second phase with eutectic composition in the interglobular regions.

For coarse grained alloy the primary phase of semi-solid material is not completely spherical, mainly for the lowest degree of previous deformation. This incomplete spheroidization is due to longer diffusion distances involved and the short time supplied (no holding time at 908 K was allowed) for coarsening phenomena to be effective where recrystallization does not play the main role. The samples submitted to lower deformation present fragmented dendritic arms, in an initial globularization process, where thicker second phase surrounding α particles reveals original grain boundaries. $CuAl_2$ pools are mainly present inside the primary phase of the initially refined alloy, since the second phase was more homogeneously distributed in the original fine dendritic structure. While there is no qualitative evidence of the influence of deformation level on the microstructure, the role of initial grain size is predominant in the globularization process.

The average globule size, shape factor and solid fraction of rheocast samples obtained by RAP process, with no holding time at the rheocasting temperature, in all tested conditions, are presented in Table 3. Results show a great reduction in grain/globule size in rheocast material compared to initial as-cast alloy, indicating efficient recrystallization. It is observed that grain size of rheocasts obtained from RAP is statistically independent on degree of previous deformation, but globule size of rheocasts produced from refined alloy are 39 % smaller than from coarse alloy, showing a significant influence of initial grain size on the process.

Results of shape factor show that there are limits to use RAP process to obtain rheocast materials with well rounded primary phase. Only the refined alloy was successful with this respect, at any degree of deformation, with an average shape factor of 1.65. In the case of coarse alloy, there wasn't complete globularization, resulting in higher values of shape factor.

As the rheocasting temperature was kept constant, so was the solid fraction value, which is in reasonable agreement with Scheil equation (70 %). Solid fraction after 300 s of rheocasting treatment is of the order of 72 %, nearer from equilibrium condition given by Scheil equation. The reduction in solid fraction as holding time increases is due to melting of solute rich regions, to reach the equilibrium conditions at the considered temperature.

Table 3 - Average grain sizes, shape factors and solid fractions of rheocasts obtained by RAP, for all tested conditions, with no holding time at rheocasting temperature.

Initial grain size (μm)	Degree of previous deformation (%)	Rheocast globule size (μm)	Rheocast shape factor	Rheocast solid fraction (%)
403 ± 35	45	75 ± 6	1.7 ± .6	75 ± 2
	80	74 ± 5	1.6 ± .7	76 ± 2
1290 ± 125	45	126 ± 9	2.8 ± .5	75 ± 3
	80	117 ± 8	2.0 ± .6	77 ± 2

3.2 OAT thermomechanical treatment

When using overaged starting material, rheocasts with globular primary phase surrounded by liquid of eutectic composition are obtained in all tested conditions, as shown in Fig. 6. There is a great amount of $CuAl_2$ particles inside the globules which may be attributed to precipitation during cooling after rheocasting treatment, or to the growth of precipitate particles which were not dissolved during heating. Some big pools can also be observed, mainly in the structure produced from coarse material submitted to the lower deformation. These pools can be formed as a consequence of coarsening phenomena of Al-α particles, that prevailed to some extent.

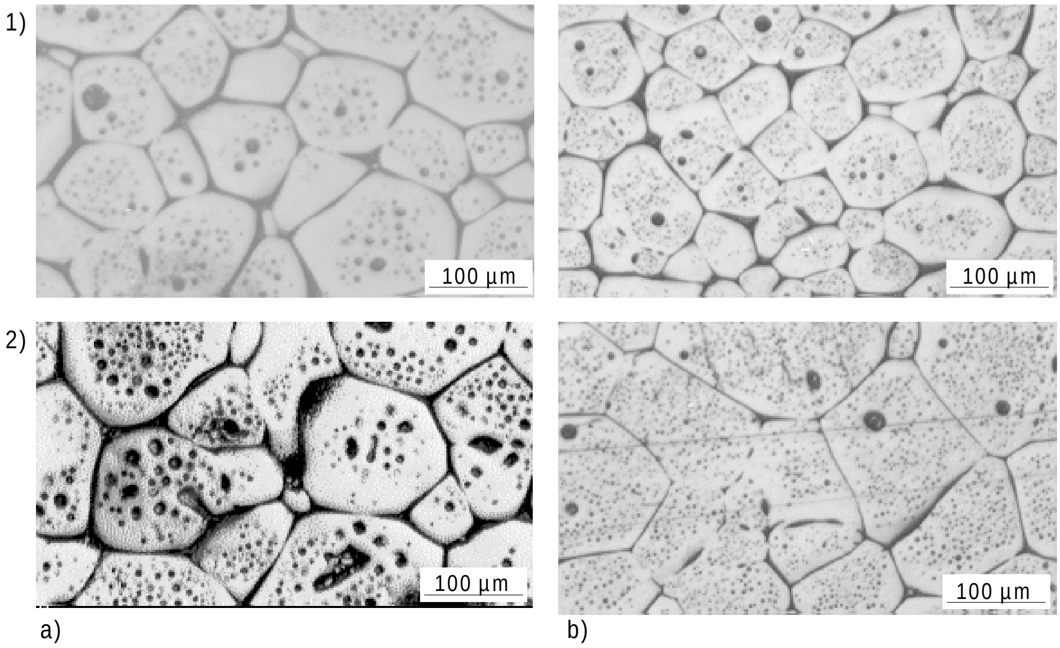

Figure 6 - Rheocast microstructures obtained by OAT: 1) refined alloy; 2) coarse alloy; a) 45 % true strain; b) 80% true strain.

Therefore, the secondary phase in rheocast material is located either at grain boundaries with eutectic composition and as entrapped liquid, when produced from dendritic as-cast alloy, or at grain boundaries and as precipitate particles in the primary phase, when produced from non-dendritic overaged alloy. These $CuAl_2$ precipitates, confirmed by SEM analysis, are found all over the primary phase except in an annular region surrounding the grain boundaries. It can be observed that these areas are larger where the boundaries are thicker.

Fig. 7 shows a SEM micrography of rheocast material obtained by OAT of a fine grained alloy previously deformed 80 % where this precipitates depleted area can be seen. This area is the result of copper diffusion to the grain boundaries. Very similar structure was shown in recent work (Braccini et al, 1998), after holding samples of spray deposited Al-10%Cu in the mushy zone.

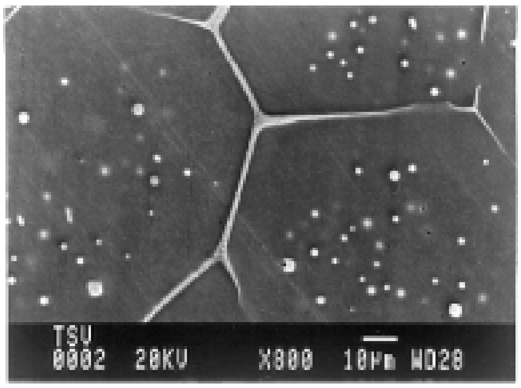

Figure **7 -** SEM micrography of a rheocast Al-3.35wt%Cu, produced by OAT from a refined alloy, submitted to 80 % of previous deformation, showing precipitates free areas.

Rheocasts produced by OAT process presented globular structures in all cases, generally with smaller grain sizes when compared to that obtained by RAP in the same conditions. The reductions in grain sizes, if compared to initial grain sizes of the raw material, are 476 % for refined alloy and 1295 % for coarse alloy.

Table 4 shows results on average globule size, shape factor and solid fraction of rheocast samples obtained by OAT process, in all tested conditions. Unlikely RAP, in this process the globule size is dependent on initial grain size and, at less extent, on degree of deformation. The average globule size in the rheocast undergoes a reduction of 24 % when grain size of raw material is decreased, and of 14 % when deformation is increased, with better performance for initially refined alloy (17 %), against 11 % for coarse grained alloy, showing that rheocasting of overaged alloys is more effective with initial smaller grains. As already explained, recrystallization stimulated by precipitate particles plays an important role in OAT process at any condition. The best rheocast quality (smaller and more rounded primary phase in the semi-solid) was obtained in the most favourable initial conditions: higher strain level, smaller grain size and bigger size of $CuAl_2$ precipitates produced after 25 h of precipitation heat treatment.

Table 4 - Average grain sizes, shape factors and solid fractions of rheocasts obtained by OAT, for all tested conditions, with no holding time at rheocasting temperature.

Initial grain size (μm)	Degree of previous deformation (%)	Rheocast globule size (μm)	Rheocast shape factor	Rheocast solid fraction (%)
403 ± 35	45	77 ± 5	1.7 ± .5	77 ± 2
	80	63 ± 4	1.7 ± .5	76 ± 3
1290 ± 125	45	98 ± 5	2.0 ± .8	75 ± 2
	80	87 ± 4	1.9 ± .8	75 ± 3

The shape factor of rheocasts produced by OAT shows that good globularization occurred for all tested conditions and don't seem to depend on degree of deformation in the raw material, for a given initial grain size.

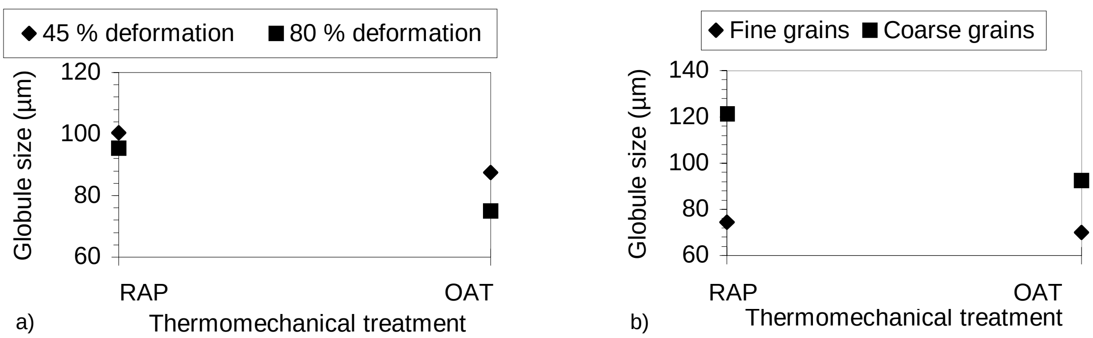

Figure 8 - Influence of thermomechanical treatments on average globule diameter of rheocasts produced by RAP and OAT from different conditions in the raw material: a) different strain levels; b) different initial grain sizes.

Figure 8(a) shows the influence of the thermomechanical treatments on average globule diameters of rheocasts. Samples previously submitted to 45 and 80 % of deformation present grain size reductions of 13 % and 21 %, respectively, when obtained by OAT with respect to RAP process.

The effect of thermomechanical treatments, taking into account initial grain sizes, can be observed in Fig. 8(b). Only the coarse grained alloy produces significant globule diameter reduction when rheocasting by OAT with respect to RAP. This is due to the behaviour of refined alloy submitted to 45 % of cold-work, which showed no important changes between the different treatments, probably because recrystallization of an overaged alloys is little sensitive to low degrees of deformation (Humphreys, 1977).

Figure 9 shows the influence of thermomechanical treatments on globule diameter for all analysed parameters. The best absolute result was obtained with OAT process applied to refined alloy submitted to 80 % of deformation, while the best performance was obtained when using coarse grained alloy, submitted to 45 or 80 % of deformation.

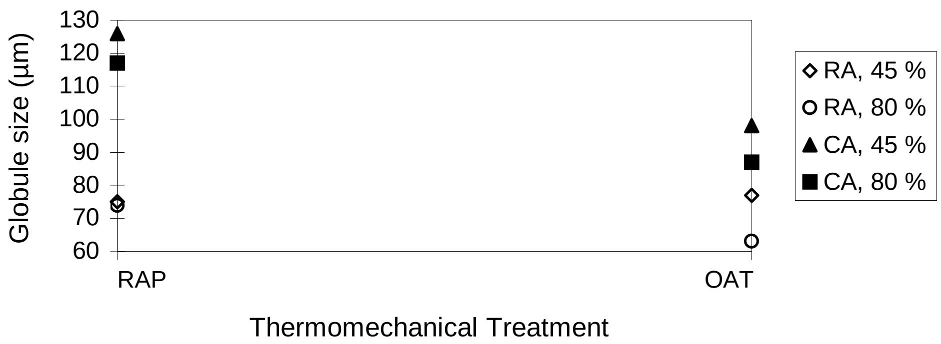

Figure 9 - Effect of thermomechanical treatments on average globule diameter of rheocasts produced by RAP and OAT processes, for all analysed conditions. RA refers to refined raw material and CA to coarse raw material.

Table 5 shows the average globule size variation of rheocast structures obtained by OAT with respect to RAP rheocast structures, for each experimental condition. It can be seen that the choice between OAT and RAP processes is not so significant when using alloy with refined grain as starting material for rheocast. Both processes produce good results, although the utilisation of raw material with 80 % of previous deformation produces rheocasts with grain size 15 % smaller if OAT process is chosen instead of RAP.

Table 5 - Globule size variation of rheocast structures obtained by OAT with respect to RAP. RA refers to refined raw material and CA to coarse raw material.

Experimental condition	Globule size variation (%)	Experimental condition	Globule size variation (%)
CA, 45 %	22	RA, 45 %	-3
CA, 80 %	26	RA, 80 %	15

On the other hand, for initially coarse raw material, the main role in producing rheocasts with smaller grain sizes is played by the type of process employed. In this case, the degree of initial deformation plays only a small part in reducing the final grain size in rheocast. Therefore, when utilising coarse raw material, the most indicated rheocast process is OAT.

4. CONCLUSIONS

Results show that rheocast structures of Al-3.35%wtCu can be produced by partial melting of the alloy either in cold deformed (RAP) or overaged (OAT) conditions. However, resulting rheocast characteristics differ according to the process employed. Reductions in globule size with respect to initial grain size are 700 % and 870 %, for RAP and OAT respectively. Initial grain size influences the morphology and globule dimensions of the primary phase of rheocasts obtained by both processes, but previous degree of deformation influences only the globule dimensions of rheocast produced by OAT, which decreases with increasing deformation. In general, OAT process produces rheocast material with more homogeneous structure and smaller globule size, of the order of 26 %, when comparing to structures obtained by RAP.

In rheocasts produced by RAP, pools of original eutectic phase are present inside the Al-α particles, mainly in originally refined raw material, while in OAT rheocasts, $CuAl_2$ precipitates are distributed in the primary phase particles. Therefore, previous overaging treatment can be advantageous in the production of better quality rheocast material, mainly when starting with coarse grained raw material.

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