

Morphological analysis of SSM Al–4.5 wt.% Cu measured by the rheocast quality index

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Abstract

There is a direct relationship between grain size obtained from macrostructure characterisation and the globule size and shape factor, obtained from microstructure characterisation: the smaller the grain size, the smaller the globule size and shape factor. Due to this a rheocast quality index ($RQI = \text{globule size}/(\text{grain size} \times \text{shape factor})$) was used to evaluate the Al–4.5 wt.% Cu alloy produced by strain induced melting activation (SIMA). The structure that presents the smallest grain size, the smallest shape factor, and the most homogeneous and globular size of the primary phase, has the best RQI and consequently the best behaviour in the semi-solid forming. Four different as-cast structures were cold rolled to obtain 20 and 40% deformation and were partially melted at 635 °C at holding times of 5 min. Both, macro- and microstructure were characterised.

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1. Introduction

Only rheocast structures produced by cold deformation followed by partial melting (SIMA—strain induced melting activation), or those produced by spray casting, present a completely spherical shape [1,2]. Rheocast structures, which present minor grain size, minor shape factor, and most homogeneous and globular size of the primary phase, have the best behaviour in the semi-solid forming [3,4]. Despite, these routes being the most expensive to obtain rheocast structures, these materials are the most suitable for thixo-forming. This paper presents the rheocast quality index (RQI) characterisation method as a powerful tool in the semi-solid characterisation area.

The most common raw material production methods—mechanical or electromagnetic stirring, ultra-refining and super-cooling—assure a very refined equiaxial dendrite structure. This structure, when re-heated to the semi solid state, presents several three-dimensional interconnections within the primary phase branches. Ito et al. [5] presented these connections in 1992. Due to these interconnections, some difficulties to correctly characterise these materials were reported [6,7]. Some authors focused their characterisation work on the shape and size of the primary phase in microstructure; others used different shape factors as well as

globule/grain size determination [8–10]. But, few researches were focused on the macrostructure characterisation. There is a significant difference between macrostructure and microstructure for the A356 alloy, as shown in previous work [11–14], and this difference could help in the rheocast characterisation. The relationship between macro- and microstructure shows that the ratio between the size of the primary phase and the size of the grain can help to quantify the several interconnections mentioned above. Small and more globular grains tend to have few branches and a more globular shape. In this case, a grain in the macrostructure tends to have the same size and shape as the grain in the microstructure, i.e. one grain is one globule. The most common structures present a quasi-globular or rosette-shaped grain. This paper will show the difference between grain and globule by comparing the macro- and the microstructure of the Al–4.5 wt.% Cu rheocast alloy produced by SIMA.

2. Experimental procedure

The thixotropic raw material used in this experiment was the AA2014 alloy (Al–4.5 wt.% Cu–0.20 wt.% Si–0.30 wt.% Fe), prepared by SIMA in the Thixo-forming Laboratory at Unicamp. The as-cast initial structures of the alloy Al–4.5 wt.% with four different grain sizes were produced by controlling cooling rate during solidification, using

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different pouring temperatures (750 and 700 °C) and mould temperature (200 and 20 °C). For this alloy the *liquidus temperature* is 645 °C and the *solidus temperature* is 628 °C, as determined by DTA analysis. From these four starting materials, rheocast structures with different grain sizes were produced by partial melting via isothermal treatment, for 5 min at 635 °C (sufficient temperature to produce 40% solid fraction, according to Scheil's equation in the following ways: (a) direct from the four as-cast conditions; (b) from a condition where the four as-cast materials were previously solubilised at 500 °C/1 h and cold-rolled to 20% true deformation and (c) from a condition where the four as-cast materials were also previously solubilised at 500 °C/1 h and cold-rolled to 40% true deformation. The resulting rheocast samples were micro- and macroscopically characterised by optical microscopy to obtain the dendritic arm spacing and grain size of the as-cast material as well as the globule size, shape factor, secondary phase content, secondary phase entrapped inside the primary phase and grain size of the as-rheocast material. For globule/grain size determination, the intercept average method was used.

After the heating treatment, the samples were cut and the areas around the previous position of the thermocouple were characterised for macro- and microstructure. Firstly, for microstructure, the samples were polished up to 6 µm and electro-chemically etched with 800 ml ethanol, 140 ml water and 60 ml perchloric acid (8.5 V and 1 A), for 15 s. After this, the same samples were chemically etched with 10 g NaOH in 90 ml water at 80 °C for 15 s, followed by 50 ml of HCl and 50 ml of water for macrostructure. Microstructure characterisation used a Neophot 55 microscope. Macrostructure characterisation used a Bausch & Lomb StereoZoom 6 stereo-microscope. The contrast between grains was obtained using different light sources with yellow and blue colour filters [14].

The image analysis system was used to delineate the branches in the microstructure and the grains in the macrostructure. It was possible to make the following measurements: linear intercept average and shape factor (inverse of sphericity). The shape factor used was

$$\text{Shape factor} = \frac{P_{\alpha}^2}{4\pi A_{\alpha}} \quad (1)$$

where A_{α} and P_{α} represent the area and the perimeter of the primary phase in the microstructure, respectively. Macro- and microstructure were compared using the RQI [11–13] defined by:

$$\text{RQI} = \frac{\text{globule size}}{\text{grain size} \times \text{shape factor}} \quad (2)$$

where the measured globule is the size of the primary phase in the microstructure and the grain size is defined by the macrostructure. For these measurements, the intercept linear average method was used. Macrostructure characterisation demands a better comprehension with regard to the interpretation of the real grain size. The criteria adopted were to

manually connect those separated grains that presented exactly the same tone and were beside each other. Grains that presented the same tone, but were separated by one branch with a different tone, were counted as separate grains. This evaluation was repeated, until 200 grains in several images, were counted.

3. Results and discussions

Table 1 shows the results obtained for the macro- and the microstructure for all the tested samples. The smaller the original dendritic grain size, the smaller the as-rheocast structure obtained, as expected. The re-heating of the Al–4.5 wt.% Cu at the semi-solid range, promoted the melting of the secondary phase (eutectic $\alpha + \text{CuAl}_2$) present in the Al- α boundary. Under a such circumstance, the thermodynamic condition of minimum solid/liquid surface energy is achieved by changing the dendritic morphology of the solid phase to a spherical type. The dendritic condition as well as the cold deformed dendritic structure can produce, in this way, the rheocast structure.

For a clear and direct comparison, the macro- and the corresponding microstructure of the samples can be seen in Figs. 1 and 2. In the present research, only the structure that presented the smallest starting grain (dendrite) as well as the consequently small rheocast structures will be shown to exemplify the characterisation procedure, i.e., the as-cast and the as-rheocast structures produced from the lower pouring temperature (700 °C) and the lower mould temperature (20 °C). Both macro- and microstructure characterisation were used for all samples because only with both these techniques can the real state of the structure be shown.

Fig. 1 shows the macro- and the microstructure of one of the starting material condition (dendrite structure presenting a grain size of $39 \pm 5 \mu\text{m}$ and a secondary dendrite arm spacing of $7 \pm 2 \mu\text{m}$, see Table 1). The structure presents homogeneous grain size and very refined dendrite morphology surrounded by the eutectic. From these raw materials all rheocast materials were produced by re-heating at semi-solid state (with and without previous deformation by cold rolling).

Fig. 2 shows the rheocast material produced from the material presented in Fig. 1. Starting with macrostructure comparison (see Table 1), it is possible to see that the isothermal heat treatment at 635 °C was much more effective to produce the rheocast structure, in the as-cast structure deformed 40%, especially in the structure with the smallest initial grain size. The explanation for this is the recrystallisation phenomenon that occurs before the semi-solid range. This recrystallisation phenomenon generates small new grains. The melting of the grain boundaries occurs in those small grains, leading to their detachment as individual globules. The higher the deformation, the faster the recrystallisation and the smaller the final rheocast grain size: in this case a rheocast grain

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