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# A novel solid state method for manufacturing Al foams by over solution heat treatment

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ABSTRACT

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#### 1. Introduction

Metallic foams have increased their applications due to the exceptional properties derived from their structures [1]. The most important methods for manufacturing these materials include infiltration of liquid metal, powder metallurgy and liquid processes [1]. In the search of new manufacturing processes, the present work introduces a new solid state method using heat treatments at temperatures higher than the melting point of the second phases. For these alloys a challenge has been to control the solution heat treatment for avoiding pores formation due to the localized melting of low melting point second phases [2,3]. Nevertheless, the extensive porosity formation (since now a not desired effect) could be used as an extra manufacturing method in order to obtain metallic foams and widespread the applications of these alloys. Al-Si-Cu-Mg alloys are excellent candidates for the study of the foams formation through the above mentioned method due to their microstructure, including  $\alpha$ -Al, Mg<sub>2</sub>Si, Al-Si eutectic, Al<sub>2</sub>Cu and Al<sub>5</sub>Mg<sub>8</sub>Cu<sub>2</sub>Si<sub>6</sub> (Q) [2]. Solution heat treatment is particularly suitable for alloys with high magnesium content, fact that has led to investigate the effect of the addition of this element. There are some previous works about the solution heat treatment of conventional Al-Si-Cu-Mg alloys [2,3], even for high Mg contents (between 0.5 and 7.0 wt%) [4,5], being the optimal

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http://dx.doi.org/10.1016/j.matlet.2016.03.060 0167-577X/© 2016 Elsevier B.V. All rights reserved. reported solution temperature close to 500 °C. The increase in the quantity of Mg could help in obtaining the desired second phases for the foams manufacturing. Thus, the main objective of the present work is to obtain metallic foams using a solid state method through modifications on the microstructure of Al-Si-Cu-Mg alloys. Keeping this purpose in mind, over solution heat treatments were performed on three modified Al alloys with Mg contents of 7, 9 and 11 wt%.

#### 2. Experimental

The present study shows a new manufacturing process of foams of Al alloys by the modification of the

conventional solution heat treatment of three alloys Al-6Si-3Cu-xMg (x=7, 9 and 11 wt%). Over-heating

the alloys during 6 h at 520, 560 and 600 °C allowed to obtain foams with maxima porosities of 40% and

pore sizes from 20 to 150 µm, originated due to the localized melting of second phases. The character-

istics of the foams highly depended on the Mg content and the heat treatment temperature.

As first step for manufacturing the Al foams, three experimental Al-6.0Si-3.1Cu-xMg (x=7%, 9% and 11%) alloys were obtained as precursors, casting cylinders with 2 C. In diameter and 15 cm in height using a Leybold-Heraeus induction furnace with Ar atmosphere. Fig. 1a shows the basics of the new manufacturing process. Conventional solution heat treatment temperature (T) is always  $< T_{lm1}$  ( $T_{lm1}$  is the melting temperature of the second phase with the lowest melting temperature). In this new process the temperatures must be higher than the melting temperatures of the second phases ( $T_{lm1}$ ,  $T_{lm2}$ ,  $T_{lm3}$ ... $T_{lmn}$ ), in order to obtain foams with different porosities. According to the selected temperature ( $T_{foam1}$ ,  $T_{foam2}, T_{foam3}...T_{foamn}$ ) we can obtain from foam 1 (with pores formed due to the localized melting of the phase 1) to foam n(with pores formed due to the localized melting of the phases from 1 to n). In order to determine  $T_{lm}$ , the as-cast alloys were sectioned in cubic samples (1.0 cm per side) and analyzed using







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Fig. 1. (a) Representation of the heat treatments used for manufacturing the foams; and (b) DTA curves showing the melting points (*T<sub>im</sub>*) of the phases for the as-cast alloys.

Differential Thermal Analysis (DTA). This allows to determine the transition temperatures of the different phases, related to the formation of the porosity due to their localized melting (besides phase transformations). DTA study was performed between 300 and 700 °C in a TA Instruments SDT Q-600 calorimeter. Fig. 1b shows the obtained DTA curves, where three endothermic peaks can be observed for each alloy, changing according to the chemical composition, and corresponding to  $T_{lm1}$ ,  $T_{lm2}$  and  $T_{lm3}$  in Fig. 1a. The first endothermic peaks appear at 508, 509 and 506 °C for the alloys with 7, 9 and 11 Mg wt%, respectively. The second endothermic peaks were obtained at 541, 538 and 542 °C; while the last peaks appear at 585, 588 and 581 °C. These peaks could be originated due to the incipient melting of Al<sub>2</sub>Cu+Al<sub>5</sub>Mg<sub>8</sub>Cu<sub>2</sub>Si<sub>6</sub> and eutectic Al-Mg<sub>2</sub>Si, according to literature [6-8]. Three temperatures (520, 560 and 600 °C; respectively T<sub>foam1</sub>, T<sub>foam2</sub> and  $T_{foam3}$  in Fig. 1a) were selected in order to obtain three different foams for each alloy. Samples of about  $1.0 \times 1.0 \times 0.5$  cm were heat treated in a forced-air furnace during 6 h followed by air-cooled. Superficial and bulk analyses of the heat treated alloys were carried out by cutting the samples in two symmetrical parts.

The as-cast and the heat treated alloys were polished using standard metallographic techniques and characterized by Optical (OM) and Scanning Electron Microscopy (SEM), using respectively a Nikon EPIPHOT 300 OM, and a JEOL JSM 7600F SEM operated at 20 kV, with a BRUKER XFlash6130 energy dispersive X-ray spectrometer (EDX) attached. The alloys were also analyzed by X-ray diffraction (XRD), using a Siemens 400 X-ray diffractometer, with CuK $\alpha$  radiation at 30 kV and 25 mA.

#### 3. Results and discussion

#### 3.1. Analysis of the as-cast alloys

Fig. 2a–c shows OM micrographs of the as-cast alloys. The increase in Mg content led to significant microstructural modifications. For the alloy with 7 Mg wt% (see Fig. 2a) four second phases can be observed, according to the EDX analysis: Al-Si eutectic, Al-Mg<sub>2</sub>Si, blocky Al<sub>2</sub>Cu and Al<sub>5</sub>Mg<sub>8</sub>Cu<sub>2</sub>Si<sub>6</sub>. For the alloy with 9 Mg wt% (see Fig. 2b) microstructure shows the presence of primary Mg<sub>2</sub>Si particles besides the eutectic Al-Mg<sub>2</sub>Si. In this case Al<sub>2</sub>Cu is not only as blocky particles but also as eutectic. For the alloy with 11 Mg wt% (see Fig. 2c) the quantity of Cu-rich phases is higher than for the alloys with lower Mg content. Besides, a higher content of Al-Si eutectic was observed, and Mg<sub>2</sub>Si appears as Chinese-script. XRD corroborated these results.

#### 3.2. Analysis of the over-heat treated alloys

Fig. 3a–i shows the porosities induced by the heat treatments. A wide range of pore percentages and sizes were obtained (note that all micrographs are at the same magnification). Fig. 3a, d and g show the microstructures for the alloys over-heat treated at 520 °C (porosities  $\sim 3 \pm 1\%$ ; average equivalent diameters  $\sim 20 \pm 2 \mu$ m). Fig. 3b, e and h show the microstructures obtained after over-heat treatments of 560 °C, presenting porosities significantly higher than for the alloys treated at 520 °C in this case, the porosity reached a maximum of 18.5% for the alloy with 7% Mg ( $\sim 8\%$  for the other alloys). For this temperature, the equivalent diameter of the pores also significantly increased, reaching a maximum of  $\sim 58 \pm 9 \mu$ m for the alloy with 7% Mg ( $\sim 38 \mu$ m for the other alloys). Finally, for the alloys treated at 600 °C porosities



Fig. 2. Optical micrographs of the as-cast alloys for Mg wt% of: (a) 7%, (b) 9%, and (c) 11%. Second phases are indicated.



Fig. 3. Optical micrographs showing the porosity originated in the alloys with 7% Mg (a-c), 9% Mg (d-f) and 11% Mg (g-i) over solution heat treated during 6 h at temperatures of: 520 °C (a,d,g), 560 °C (b,e,h) and 600 °C (c,f,i).

and pore sizes also significantly increased (see Fig. 3c, f and i). For this temperature, two kind of pores were observed for the alloys with lower Mg contents: i) pores with aspect ratios near 0.9 and equivalent diameter of  $115 \pm 25 \,\mu$ m; and ii) elongated pores with aspect ratios between 3 and 8, as large as 700  $\mu$ m. The formation of these elongated pores was avoided for the alloy with 11 Mg wt%, because have aspect ratios of 0.8, with pore sizes of  $150 \pm 20 \,\mu$ m and a porosity of  $\sim 40\%$  (see Fig. 3i).

At 600 °C, a much higher amount of liquid is produced due to the melting of more secondary phases, according the endothermic peaks shown in Fig. 1b. After the heat treatment process two main effects were observed in the sample: (i) the formation of high amount of porosity, and (ii) phase transformations. The first effect, i.e. large porosity, is shown in Fig. 3a-i, and has been already reported for these alloys at temperatures as low as 505 °C [3], however, it was considered as a harmful effect. In this work, a greater amount of porosity was obtained using higher heat treatment temperatures, turning around an adverse or undesired effect into a manufacturing method for producing metallic foams. Pores formation could be attributed to the shrinkage generated by localized peritectic reactions after melting of specific second phases. This second effect, i.e. phase transformations (mainly Q into Al<sub>2</sub>Cu), was observed through XRD and EDX analyses, and will be published in detail elsewhere. It is important to remark that due to the low cooling rate no significant differences were observed throughout the microstructure of the samples, and that no liquid phase was lost from the samples during the process (not flows but solidifies into another phase). Besides the microstructure, other characteristics of the foams, such as density and mechanical properties will be further determined.

#### 4. Conclusions

An innovative solid state method to fabricate Al foams was successfully developed by over solution heat treatment of Al-Si-Cu-Mg alloys, obtaining foams with maximum porosities of  $\sim 40\%$  and pore sizes of  $\sim 150~\mu m$ . The morphology of the foams highly depends on the heat treatment temperature and on the secondary phases (Al<sub>2</sub>Cu, Al<sub>5</sub>Mg<sub>8</sub>Cu<sub>2</sub>Si<sub>6</sub>, Al-Si eutectic and Mg<sub>2</sub>Si) present in the alloys, that were used as precursors. It is though that the localized melting of these phases caused the pore formation. The increase in the Mg content led to significant microstructural modifications in these precursor alloys.

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