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Main process parameters for manufacturing open-cell Zn-22Al-2Cu foams by the centrifugal infiltration route and mechanical properties



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HIGHLIGHTS

G R A P H I C A L A B S T R A C T

- Open-cell Zn-22Al-2Cu foams were manufactured by centrifugation process.
- NaCl particles have been used as space holder.
- The mechanical properties can be improved when the relative density is modified.
- The minimum centrifugal force varied for each NaCl particle size of the preform.
- The relative density decreases when the pore sizes increases.



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ABSTRACT

Parameters of centrifugal infiltration process for manufacturing open-cell Zn-22Al-2Cu foams with sphericalshape porosity and different pore sizes from 0.85 to 0.42 mm were experimentally determined. The experimental results showed that the most favorable temperature both of heating of the NaCl preforms and of pouring (or infiltration) was 600 °C, while the minimum centrifugal forces varied for each NaCl particle size of the preform. Minimum centrifugal forces of 11, 16 and 25 N were required to fully form open-cell Zn-22Al-2Cu foams with pore size of 0.85 mm and 0.42 mm, respectively.

The compressive mechanical properties of the open-cell Zn-22Al-2Cu foams increased when the pore size decreased. The higher mechanical parameters; Elastic modulus E, yield strength σ_y , and plateau stress σ_{pl} were obtained for the open-cell foam with the smaller pore size (0.42 mm).

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1. Introduction

In recent years, the attention of several researchers has been focused on the development and characterization of porous metal structures as metallic syntactic foams or composite foams (MMSFs), in which hollow

* Corresponding author. *E-mail address:* gelikmart@gmail.com (A. Sánchez-Martínez). Porous metal structures as closed-cell and open-cell foams show a n hollow combination of several properties such as high strength-to-weight ratio high energy and sound abcorption capacity large specific surface

to control the pore size and shape.

ratio, high energy and sound absorption capacity, large specific surface, thermal control and excellent vibration reduction capacity [6]. These properties allow using the porous metal structures as shock and impact

spheres are dispersed in a continuum matrix [1–5], and conventional

metallic foams, in which sacrificial space-holders offers the possibility

absorbers, dust and fluid filters, engine exhaust mufflers, heat exchangers, flame arresters, catalyst supporters, among others. There are several methods to produce open-cell foams [7]. Among these, the infiltration casting process offers a high degree of control over the foam topology. This method produces an interconnected cellular structure or open-cell foam by infiltrating molten metal under pressure through an open pore preform (negative of the foam) prepared with bonded granular particles (space holders). NaCl particles are usually used as space holders for low melting point alloys. After the solidification process, the NaCl particles are leached in water [8,9]. Since the methodology proposed by Polonsky et al. [10] for the manufacture of open-cell foams, many devices and preform designs composed by NaCl particles have been developed [11–14].

The infiltration pressure, pouring temperature ($T_{pouring}$) and the design of the preform (material, size, porosity, etc.) are the main variables for the replication casting process [15,16]. Therefore, the possibility of controlling these parameters in several ways with different mechanisms has led to a wide variety of devices development to produce metallic foams. In these devices, the infiltration pressure is commonly applied by means of a gas or pistons [17,18].

However, the infiltration pressure generated in a molten metal by centrifugal force has not been used to manufacture open-cell foams. The centrifugal infiltration casting has been successfully used as an effective process to manufacture high quality metal-matrix composites (MMCs) [19,20]. The centrifugal casting process requires simple and economical casting equipment and can be suitable for fabricating open-cell foams of Al/Al-alloys and Zn/Zn-alloys.

Zn-22Al-2Cu alloy is a kind of superplastic material that is having further attention on the field of porous metallic structures, because it combines the properties of superplasticity with the energy absorption and vibration reduction capacities [21]. Neither the manufacture of open-cell Zn-22Al-2Cu foams by the centrifugal infiltration process has been studied nor the process parameters necessary to obtain the desired pore sizes and relative density. Therefore, the aim of this work is to determine the adequate experimental process parameters for the production of open-cell Zn-22Al-2Cu foams by the centrifugal infiltration route, and establish the relationship between the mechanical properties as a function of the pore size.

2. Experimental procedures

2.1. Alloy preparation

Zinc alloy with a composition of Zn-22 wt.% Al, 2 wt.% Cu alloy was fabricated using elements of commercial purity; Zn (99.995%), Al (96.2%) and Cu (99.95%) in an electrical resistance furnace. The melt was degassed with Ar to reduce the hydrogen level. The molten alloy contained in a graphite crucible was poured into an iron mold. The chemical composition was analyzed by optical emission spectroscopy (OES) on the as-cast ingots. At least 10 measurements were performed on each ingot to determine the average composition. The experimental composition of the alloy was 21.92 ± 2 wt.% Al, 1.79 ± 0.2 wt.% Cu, and the balance Zn.

2.2. Infiltration casting

The equipment used for the manufacture of the open-cell Zn-22Al-2Cu foams consists of two electrical resistance furnaces and a centrifugal device, Fig. 1a. The infiltration process was carried out in a cylindrical steel crucible designed in two parts with the following dimensions: inner diameter 6 cm, outer diameter 7 cm, and height 15 cm, Fig. 1b. The crucible was hermetically closed in the bottom part with a removable circular cover of diameter = 10 cm.

Open-cell Zn-22Al-2Cu foams were produced using porous preforms constituted by NaCl particles (space holders) of spherical shape. In order to manufacture spherical particles, commercial NaCl particles with a cubic shape were molten, atomized with air, and solidified in flight to retain their spherical shape. The NaCl particles were sieved and separated in three groups. Sieves with the following ASTM numbers: 16–25, 25–35, and 35–45, corresponding to 1.18–0.71 mm, 0.71–0.5 mm and 0.5–0.35 mm openings, were used. The NaCl particle sizes were classified here as (A) 0.85 mm, (B) 0.65 mm and (C) 0.42 mm, considering the average size of each group. Fig. 2 shows the morphology and size of such NaCl particles.

The NaCl particles were loaded into the crucible in order to form the preform with dimensions: diameter 6 cm and height 10 cm. Prior to the infiltration process, the steel chamber was introduced into an electrical



Fig. 1. a) Centrifugal infiltration equipment b) cylindrical steel chamber used for the infiltration process (upper part) and its position in the rotor of the centrifuge (lower part).



Fig. 2. SEM-images of the NaCl particles used in the manufacture of the open-cell Zn-22Al-2Cu foams: a) (A) 0.85 mm, b) (B) 0.65 mm and c) (C) 0.42 mm.

resistance furnace (1) in order to sinter the preform using a temperature of 680 °C (120 °C below the melting point of the NaCl [22]) for 3 h. This sintering condition was taken from Reference [23]. Fig. 3 shows digital and SEM images of a representative sintered preform, where the attachment between NaCl particles can be observed.

When the sintering time was completed, the furnace temperature was decreased until the preheating temperatures of preforms (T_{pref}), which were varied from 400 °C to 600 °C.

Simultaneously to the sintering process of the preforms, ingots of Zn-22Al-2Cu alloy were melted in another electrical resistance furnace (2). Pouring (or infiltration) temperatures ($T_{pouring}$) between 500 and 600 \pm 5 °C were used for the infiltration casting process. When the infiltration temperature was reached, the molten alloy was poured into the steel chamber on top of the sintered and preheated preform. Afterwards, the crucible was extracted from the furnace and quickly placed in the rotor of the centrifugation system for the infiltration process. For this process, centrifugal forces (F_c) from 11 to 25 N were tested. After 5 min of spinning the centrifuge was stopped. When the formed Zn-22Al-2Cu alloy–NaCl composite was completely solidified, it was extracted from the crucible, and then machined to obtain samples for analysis. Finally, the NaCl particles were completely dissolved in H₂O using an ultrasonic equipment.

The characterization of the porosity and cell structure was carried out by optical microscopy and scanning electron microscopy using a JEOL 6300. The percentage of porosity Pr (%) of the foams was calculated using the relative density, ρ_{Rel} (defined as the foam density ρ^* between metal matrix density $\rho_{solid} = 5.4 \ g/cm^3$) in the following expression: Pr (%) = (1-relative density) \times 100. The density of the open-cell Zn-22Al-2Cu foams ρ^* was determined by calculating their volume from their known external dimensions and measuring their mass.

2.3. Mechanical properties

Cylindrical-shape foams with dimensions: 20 mm in diameter and 16.6 mm in height were machined for the compression tests. Based on previous studies [24,25] the sample height was minimized to obtain

stress–strain curves with a large strain, including linear elasticity, collapse plateau and densification. The tests were conducted at room temperature using a universal testing machine (Instron 5500R) at a constant crosshead speed of 0.5 mm/min, corresponding to a nominal strain rate of $3 \times 10^{-3} \text{ s}^{-1}$. The stress-strain data are reported in terms of engineering stress and strain. For each pore size at least three samples were tested and the average mechanical parameters (e.g. Yield strength σ_y , Young's modulus E and plateau stress) are reported.

3. Results and discussion

3.1. Infiltration parameters

Fig. 4(a-i) shows open-cell foams samples of the Zn-22Al-2Cu alloy manufactured by the centrifugal casting process at different infiltration parameters, using preforms constituted by spherical particles of NaCl.

The open-cell Zn-22Al-2Cu foams were initially manufactured using a centrifugal force (F_c) of 11 N, a pouring temperature (T_{pouring}) of 500 °C (45 °C above the melting point of the alloy) and a preform constituted by NaCl particles of (A) 0.85 mm size, which was heated to a temperature (T_{pref}) of 400 °C prior to the infiltration process. From Fig. 4a, it can be observed that the open-cell foam was partially formed at these infiltration parameters. Increasing the pouring temperature (T_{pouring}) at 600 °C (145 °C above the melting point of the alloy) to increase the fluidity of the molten metal through preform, a higher volume of open-cell foam was obtained, Fig. 4b. When the heating temperature of the preform (T_{pref}) was increased to 500 °C (using the same pouring temperature T_{pouring} (600 °C) and centrifugal force F_c (11 N), a complete formation of the open-cell foam was achieved, Fig. 4c.

Fig. 4d shows the open-cell foam manufactured with a preform constituted by a minor size of NaCl particles (B) 0.65 mm) using the previous optimal infiltration parameters. As can be observed, these parameters were not sufficient to fully form the open-cell foam. Increasing the heating temperature of the preform (T_{pref}) at 550 °C, a higher amount of molten metal was infiltrated. However, part of the molten metal was solidified on top of the open-cell foam, Fig. 4e. The complete



Fig. 3. a) Digital image of sintered preform and b) SEM-image of sintered NaCl particles.



Fig. 4. Open-cell Zn-22Al-2Cu foams manufactured varying the infiltration parameters: NaCl particle size, pouring temperature ($T_{pouring}$), heating temperature of the preforms (T_{pref}) and centrifugal force (F_c).

498

Table 1Optimal infiltration parameters for the different pore sizes.

Sample pore size (mm)	Centrifugal force Fc (N)	Infiltration pressure P (KPa)	Preheating temperature T _{pref} (°C)	Pouring temperature T _{pouring} (°C)
(A) 0.85	11	0.61	500	600
(B) 0.65	16	0.94	550	600
(C) 0.42	25	1.42	600	600

formation of the open-cell foam was achieved by increasing the centrifugal force (F_c) at 16 N, with the same heating temperature of the preforms (T_{pref}) (550 °C) and pouring temperature $T_{pouring}$ (600 °C), Fig. 4f.

Fig. 4g shows the open-cell foam manufactured with a preform formed by fine NaCl particles of (C) 0.42 mm size. As expected, the last optimal parameters were not enough to form this type of opencell foam. To attain the formation of the open-cell foam with this fine pore size, first the heating temperature of the preforms (T_{pref}) was increased to 600 °C (the same as the pouring temperature ($T_{pouring}$) 600 °C) and the centrifugal force (F_c) was maintained at 16 N. Although with these process parameters the open-cell foam was not completely formed, the molten metal was infiltrated in higher amount, Fig. 4h. Increasing the centrifugal force (F_c) to 25 N the open-cell foam was fully formed as can be observed in Fig. 4i.

Table 1 summarizes the optimal infiltration parameters found for each pore size. In general, the experimental results suggest that a decrease in the NaCl particle size of the preforms require increasing the infiltration parameters such as the pouring temperature ($T_{pouring}$), the heating temperature of the preforms (T_{pref}) and the centrifugal force (F_c). The maximum pouring temperature ($T_{pouring}$) used to infiltrate the preforms was limited to 600 °C (145 °C above the melting point) because at higher temperatures the formation of slag (oxidation of the molten metal) increases during the centrifugal process, which can obstruct the liquid metal flow through the NaCl preform.

On the other hand, it is well known that the preforms must be preheated to prevent the premature solidification of the liquid metal and increase its fluidity during the infiltration process, especially for low infiltration pressures [7]. Although the preforms constituted by NaCl particles could be heated at temperatures near 800 °C, the optimal heating temperature (T_{pref}) found in this work was 600 °C, equal to the

maximum pouring temperature ($T_{pouring}$). Results suggest that for the manufacture of open-cell Zn-22Al-2Cu foams, both temperatures could be set at 600 °C, while the centrifugal force should be changed according to the NaCl particle size of the preform.

The NaCl particle size of the preforms acted as a scaling parameter for the centrifugal force (F_c). This means that the NaCl particle sizes of 0.85 mm, 0.65 mm and 0.42 mm required minimum centrifugal forces (F_c) of 11, 16 and 25 N, respectively, to fully form the open-cell Zn-22Al-2Cu foams. Therefore, for the manufacture of open-cell foams with preforms constituted by smaller NaCl particle sizes than 0.42 mm, higher centrifugal forces than 25 N must be used.

J.F. Despois et al. [26] have reported that using a minimum infiltration pressure (threshold pressure) in the manufacture of Al foams, the molten metal is infiltrated in the preform only through the large spaces located between NaCl particles. This condition favors the interconnection of the pores due to the large contact area between NaCl particles. However, when the infiltration pressure is increased, the molten metal is gradually infiltrated through the narrower regions. Consequently, the density and the properties of the metallic structure are increased.

In order to estimate the minimum infiltration pressure as function of the NaCl particle size, the following equation was used [27]:

$$P = \frac{6V_f \gamma \text{Cos}\theta}{d_f (1 - V_f)} \tag{1}$$

where, V_f is the volume fraction of the preform ($V_f = 1 - Pr$), d_f diameter of the spherical particles of NaCl, γ is the surface tension of the alloy ($\gamma = 0.572 \text{ N m}^{-1}$), θ the contact angle (5 deg.). The last two data (surface tension and contact angle) were obtained from data available for similar Zn-Al alloys [28,29]. The results are shown in Table 1-column 3.

The minimum centrifugal force (corresponding to the minimum infiltration pressure) obtained for each NaCl particle size in this work could be increased to modify the relative density of open-cell Zn-22Al-2Cu foams and consequently their properties. Nevertheless, it must be considered that the maximum pressure infiltration (generated by the rotation of the liquid metal) should be less than those used in the manufacture of metal-matrix composites (MMCs) for the same route.



Fig. 5. Open-cell Zn-22Al-2Cu foams with different pore sizes: a) (A) 0.85 mm, b) (B) 0.65 mm and c) (C) 0.42 mm and interconnected metallic structures (upper part).

Table 2

Densities, porosities and cell-strut thickness ($\pm 0.01~\text{mm})$ of open-cell Zn-22Al-2Cu foams.

Sample	Density of sponges (g/cm ³)	Relative density	Pr (%)	Average cell-strut thickness (mm)
(A) 0.85 mm	1.73	0.32	68	0.76
(B) 0.65 mm	1.94	0.36	64	0.52
(C) 0.42 mm	2.21	0.41	59	0.4

3.2. Cell structure

Fig. 5 shows samples of open-cell Zn-22Al-2Cu foams with three pore sizes: a) (A) 0.85 mm, b) (B) 0.65 mm and c) (C) 0.42 mm. In the upper part of this figure, SEM-micrographs of their metallic structures are shown. From these images, it can be observed that the foams have a homogeneous distribution of the porosity with shape and size equivalent to those of the space holders (NaCl particles) used in the preforms. Besides, these images clearly show that the foams have open porosity completely interconnected.

Table 2 shows the experimental parameters used to calculate the percent of porosity Pr (%). In addition, the cell-strut thickness of the open-cell foams is shown. From this table, it can be seen that the Pr (%) increased as the pore size increased. On the other hand, when the pore size decreased, the relative density and the open-cell foam density were increased. Relative density variations of the foams due to rotational speed (infiltration pressure) were not analyzed in the present work. Open-cell foam with pore size of (C) 0.4 mm and relative density of 0.41 had the smallest cell-strut thickness (0.4 mm).

3.3. Mechanical properties

The influence of the pore size on the mechanical properties of the open-cell Zn-22Al-2Cu foams had also been analyzed in the present work. Fig. 6 shows representative compressive stress–strain curves of the open-cell foams with different pore size. It can be seen that the curves exhibit three characteristic regions: (1) an initial linear elastic region at very low strain (smaller than 0.05) without the presence of peak stress, (2) an extended plateau region with a nearly constant flow stress and (3) a densification region (registered between 0.5 and 0.7% strain) where the flow stress increased steeply. It is clear that the compression curves in the plateau region show a smooth behavior, without the



Fig. 6. Compressive stress-strain curves of the open-cell Zn-22Al-2Cu foams with different pore sizes.

presence of serrations. This behavior was attributed to a uniform plastic deformation of the foams due to uniform pore morphology and the high mechanical strength of the cell strut material. According to M. Taherishargh et al. [30], the uniform plastic deformation was caused by the formation of multiple active deformation bands and high strain hardening of the cell struts during the plastic deformation of the foams.

The experimental mechanical properties obtained from the compressive test are summarized in Table 3. It was observed that all mechanical parameters; elastic modulus E, yield strength σ_y , (the 0.2% offset criteria was used to identify the initial linear elastic behavior and the yield stress) and plateau stress σ_{pl} (average value of the stress measured between the elastic region and the densification region) showed the tendency of increasing when the pore size was decreased.

The reduction of the pore size increased the relative density and decreased the cell strut thickness of the open-cell ZnAlCu foams; consequently the mechanical properties were also increased. For instance, the higher mechanical parameters; Elastic modulus E (0.56 GPa), yield strength σ_y (8.6 MPa) and plateau stress σ_{pl} (19.3 MPa) were obtained for the open-cell foam with the smaller pore size of (C) 0.42 mm (relative density, $\rho^*/\rho_s = 0.41$). A similar behavior was reported in a previous study on the open cell Mg foams carried out by C.E. Wen et al. [31]. They found that the mechanical properties were increased when the porosity and pore size were decreased. This behavior was attributed to the hardening capacity of the cell struts of the foams. According to the aforementioned reference [30], the hardening capacity of the cell strut was higher in the case of foams with small pore size (thin thickness) because of a refined microstructure. This produces an effective hardening of the metal matrix of the foams causing high stress in the plastic region.

In general, the mechanical properties results were consistent with those reported for the open-cell ZA22 alloy foams with irregular pore morphology [23] and closed-cell ZA22 alloy foams which were generally preferred for structural applications [32]. From a practical point of view, the mechanical properties obtained in the open-cell Zn-22Al-2Cu foams, with pore sizes from 0.85 mm to 0.42 mm, could be acceptable for a lot of functional and structural applications.

4. Conclusions

Open-cell Zn-22Al-2Cu foams with pore sizes of 0.42 mm, 0.65 mm and 0.85 mm were successfully fabricated by centrifugal infiltration process using preforms constituted by NaCl particles with spherical shape.

The suitable heating temperature of the preforms was 600 °C, which it is the same to the pouring alloy temperature, while the minimum centrifugal force varied for each NaCl particle size of the preform. Minimum centrifugal forces of 11, 16 and 25 N were required to fully form opencell Zn-22Al-2Cu foams with pore size of 0.85 mm, 0.65 mm and 0.42 mm, respectively.

When the pore size decreased, the relative density of the open-cell Zn-22Al-2Cu foams increased and on the other hand, the cell strut size decreased; consequently their mechanical properties were increased. The higher mechanical parameters; Elastic modulus E (0.56 GPa), yield strength σ_y (8.6 MPa) and plateau stress σ_{pl} (19.3 MPa) were obtained for the open-cell foam with the smaller pore size (C) 0.42 mm (relative density, $\rho_{Rel} = 0.41$).

 Table 3

 Mechanical parameters of the open-cell Zn-22Al-2Cu foams.

Sample	Yield strength σ_y^* (MPa)	Elastic modulus E (GPa)	Average plateau stress σ_{pl} (MPa)
(A) 0.85 mm	4.8	0.27	8.4
(B) 0.65 mm	6.5	0.29	13.5
(C) 0.42 mm	8.6	0.56	19.3

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