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Processing and characterization of open-cell aluminum foams obtained through infiltration processes

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Abstract

This work reports the fabricating of open-cell Al foams which were manufactured through infiltration processes employing a device with controlled atmosphere for the fusion and infiltration processes of the metal, which is separate from the heating furnace. The device allows controlling its internal atmosphere with an inert gas to protect the metal of the fast oxidation, avoiding the employment of a controlled atmosphere furnace (a furnace of this kind incorporate the heating and the control atmosphere systems itself). The metal used for the present study was Al in ingot form with 99.8 % purity. Equiaxial NaCl granules in three ranges of particle sizes were chosen as space holder particles (SHP). The range I from 4.7 to 4.0 mm, the range II from 4.0 to 3.3 mm, and the range III from 3.3 to 2.0 mm. The obtained foams showed a highly porous structure with interconnected cells in all cases. The maximum porosity and minimum density values resulted to be ~73.7 % and ~0.71 g/cm³, respectively. This work demonstrates that it is possible to use NaCl with different particle sizes in the manufacturing of Al foams with high porosity, low density and roughly 100 % interconnected cells employing a device with controlled atmosphere and an electrical furnace, both separated from one another. The results are interpreted in terms of NaCl particle sizes and its content in the samples.

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1. Introduction

The term "foam" has been defined as the uniform dispersion of a gaseous phase in a liquid or a solid phase [Banhart (1999)]. In these two cases, each gas bubble is separated by a liquid or solid wall, thus the gas bubbles are entirely isolated and are not consistently interconnected to each other. Although, the term "foam" is utilized only for the dispersion of gas bubbles in a liquid, this foam morphology can be preserved by the solidified liquid metals in framework form. During the fabrication processes of the material, a liquid metallic foam is generated, which finally is solidified and known as "metallic foam". In these cases, the expression "metallic foam" generally refers to a solid foam. Nevertheless, the solid foam is a special case that is more commonly designated as "cellular solid" [Banhart (2001)]. The cellular solids (cellular metal in the case of the metals), can be classified as closed cell metals (metallic foams) and open cell metals or interconnected cell metals (metallic sponges) according to its topology. This kind of materials with high porosity have been developed to be used as new functional materials, since these materials present a unique combination of physical and chemical properties which can be derived from their cellular structure [Davies and Zhen (1983)]. Cellular metals show increasing potential for applications in a wide range of structural and functional products, due to their exceptional mechanical, thermal, acoustic, electrical and chemical properties [Gibson and Ashby (1997); Banhart and Eifert (1997)]. Metallic foams can be manufactured by a great variety of methods, including methods with the metal in the solid, liquid and gaseous state. The most commonly used method based on the liquid state is the infiltration process (replication process, RP), which involves the infiltration of a liquid metal within a space-holder-particles (SHP) bed. The fabrication route to produce cellular metals mainly consists of five steps:

- *Pre-form molding*: a leachable SHP with specific form is put into a defined form mold.
- *Pre-form Sintering*: the mold containing the SHP is put into the furnace to sinter the SHP and to produce a leachable stiff pre-form of interconnected particles.
- *Metal melting*: after the SHP pre-form is cooled, the metal is put on the SHP pre-form and introduced into the furnace again to melt the metal.
- *Metal infiltration*: the liquid metal is infiltrated in the stiff SHP pre-form to fill its empty voids.
- *Pre-form leaching*: after the SHP/Liquid metal system is taken out of the furnace to solidify the metal, and the SHP are leached with an adequate solvent a metallic sponge with interconnected cells is obtained.

In the present work, the infiltration process is employed to obtain aluminum foams (porous aluminum with interconnected cells) with high porosity, low density and using equiaxial NaCl particles as SHP in three ranges of particle sizes. The range I from 4.7 to 4.0 mm, the range II from 4.0 to 3.3 mm, and the range III from 3.3 to 2.0.

2. Experimental techniques

The NaCl particles and aluminum foams topology was characterized through digital images to visually determine the form and size of NaCl particles and the obtained cell size and form (Sony 3D digital camera). The cells interconnection was characterized through Scanning Electron Microscopy SEM (Jeol JSM 6610 HLV). For each specimen, after the melting and solidification processes of metal, the weights and dimensions of the Al/NaCl compact were measured to determine their volume and density. Similarly, the weights of NaCl content in the Al/NaCl compact were quantified after the NaCl leaching, and the final weight and dimensions of the resultant aluminum foams were also measured to determine their volume and density. The weights were registered using a balance Ohaus with a maximum capacity of 310 g and readability of 0.001 g. Using these data, the Al/NaCl compact density ρ_c , aluminum foam density ρ_f , compact porosity P_c , and foam porosity P_f , were determined through the equations 1 to 4 [Bafti and Habibolahzadeh (2010); Jee et al. (2000)].

$$\rho = \frac{m}{V} \quad (1)$$

$$P_c = 1 - \frac{\rho_c}{\rho_{th}} \quad (2)$$

$$\rho_{th} = (\rho_{Al} * W_{Al}) + [\rho_{NaCl} * (1 - W_{Al})] \quad (3)$$

$$P_f = 1 - \frac{\rho_f}{\rho_{Al}} \quad (4)$$

Where, M=mass, V=volume, ρ_{th} is the theoretical compact density, ρ_{Al} is the aluminum density, W_{Al} is the aluminum weight fraction in the compact and ρ_{NaCl} is the NaCl density.

3. Experimental procedure

Aluminum foams were prepared through infiltration process employing the new method proposed by [Sandro Baez Pimiento et al. (2013)]. This new method consists in employing a device with controlled atmosphere [Sandro et al. (2013)] separated from the heating furnace for the fusion and infiltration processes of the metal. The device allows to control its internal atmosphere with an inert gas to protect the metal from the fast oxidation, avoiding the employment of an expensive controlled atmosphere furnace (a controlled atmosphere furnace incorporates the heating and atmosphere control systems itself). The device is internally composed by a metallic container provided of a hole in its bottom part and a metallic mold. Fig. 1 shows the container/mold system, in which the metallic container with the aluminum is put on the metallic mold containing the NaCl particles bed, and both strongly adjusted to one and other. After that, the system container/mold is put inside the device with controlled inert gas atmosphere. Finally, the device with controlled inert gas atmosphere is sealed and it is put inside of a conventional vertical electrical furnace opened in its upper side to process the metal foams.

The foams were fabricated with aluminum ingots with 99.9 % purity provided by “*Metales Águila S. A. de C. V.*” and using NaCl particles as SHP in three ranges of particle sizes provided by “*Droguería cosmopolita S. A. de C. V.*”. The range I from 4.7 to 4.0 mm showed in the Fig. 2(a), the range II from 4.0 to 3.3 mm showed in the Fig. 2(b) and the range III from 3.3 to 2.0 mm showed in the Fig. 2(c). The metal was employed to produce the main framework of the aluminum foam, and the NaCl particles with an equiaxial shape were used to generate the internal cells. Each range of NaCl particle size was put into a metallic mold until it was totally filled. The aluminum mass was placed inside of a metallic container provided with a hole in the central part of the bottom.

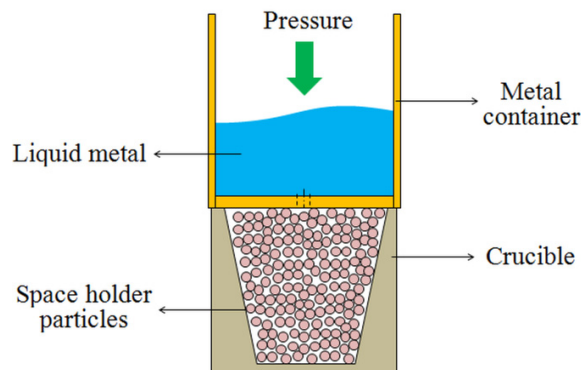


Fig. 1. Image of the configuration container/mold employed to process open-cell aluminum foams in this work.



Fig. 2. Digital images showing the NaCl particles sizes used in the processing of the aluminum foams: a) NaCl particle size from 4.7 to 4.0 mm (range I), b) NaCl particle size from 4.0 to 3.3 mm (range II), and c) NaCl particle size from 3.3 to 2.0 mm (range III).

The metallic container with aluminum was placed on the metallic mold with NaCl to guarantee the permanent contact of NaCl particles. The mold/container system was introduced in a vertical electrical furnace to melt the aluminum at 700 °C for 30 min. When the aluminum was melted, the NaCl particles bed was infiltrated with the liquid aluminum by gravity. After the infiltration process, the mold/container system was cooled to obtain a Al/NaCl compact. The amount of NaCl in the compact was leached by immersion in a water bath at 25 °C for two hours, to generate the aluminum foams with interconnected cells. Finally, to determine the NaCl volume in each foam, the water leaching was evaporated on a warming plate, later, the salt was dried in a stove at 105 °C until a constant weight was reached. The NaCl content for each obtained aluminum foam resulted to be 53.1 %vol for the size particle range I, 54.1 %vol for the size particle range II and 56.4 %vol for the size particle range III.

4. Results and discussion

Fig. 3 shows the obtained aluminum foams through infiltration process and which were manufactured using equiaxial NaCl particles with particle sizes in the range I (left) and the range II (right), respectively. Fig. 3(a) shows the obtained as-cast compacts Al/NaCl, the Fig. 3(b) shows the compacts Al/NaCl after the machined processes and Fig. 3(c) shows the aluminum foams after the NaCl leached processes. In the samples, a relatively uniform distribution of cells is observed. This homogeneous cell distribution results in an aluminum foam with a highly homogeneous framework as it is required for reliable measurements mechanical properties in this kind of materials.

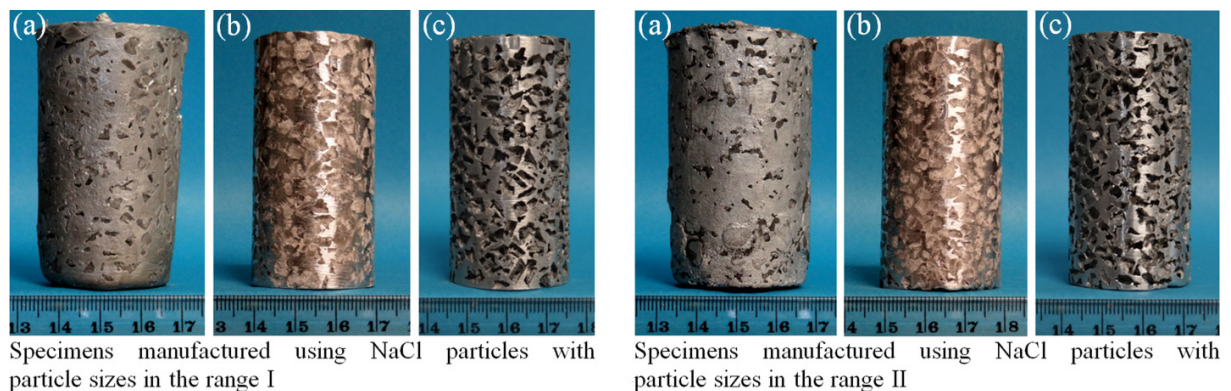


Fig. 3. Digital images showing the obtained aluminum specimens after the infiltration process: a) obtained as-cast Al/NaCl compacts, b) obtained Al/NaCl compacts after the machined processes, and c) obtained aluminum foams after the NaCl leached processes.

Fig. 4 shows the cells interconnection and obtained cells form in the foams using NaCl particles in the range II. The regions enclosed by the yellow lines display the approximate form of the equiaxial NaCl particles employed to produce the aluminum foams. It is seen that the cell size in the samples and the NaCl particles size match quite

closely. The image clearly shows the interconnectivity with a second cell which is enclosed by the red circle. On the other hand, this second cell is interconnected with a third cell which is enclosed by a green circle. Both interconnections corresponding to a black zone in the image, generating an open cell structure with practically all cells communicating with each other.

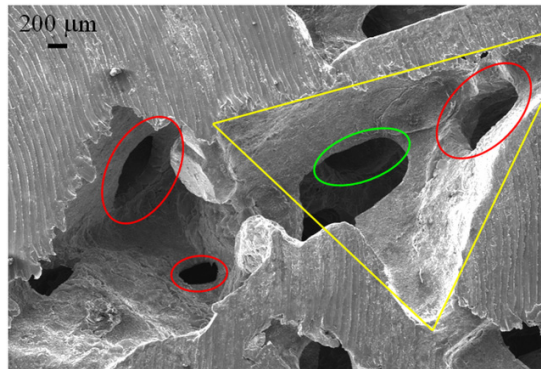


Fig. 4. SEM secondary electrons image of aluminum foams processed with NaCl particle size in the range II (from 4.0 to 3.3 mm) showing the equiaxed cells left by leached NaCl and enclosed by the yellow lines, interconnectivity with a second cell which is enclosed by the red circle, and interconnectivity with a third cell which is enclosed by a green circle.

Fig. 5 shows, as a function of the NaCl content in the compact, the changes in the ρ_{th} , ρ_c and ρ_f . In this plots, the ρ_{th} , ρ_c and ρ_f decreased with the increasing NaCl content. The diminishing in the ρ_{th} is explained by the lower value of the NaCl density (2.165 g/cm^3) compared to aluminum density (2.7 g/cm^3). Given that the proportion of SHP in the compact increases, the ρ_{th} decreases. On the other hand, the diminishing in the ρ_c results to be lower than the ρ_{th} , this reduction is caused by two reasons:

- The lower density value of the NaCl compared to aluminum.
- The empty spaces found inside the compact and located around the contact points among NaCl particles.

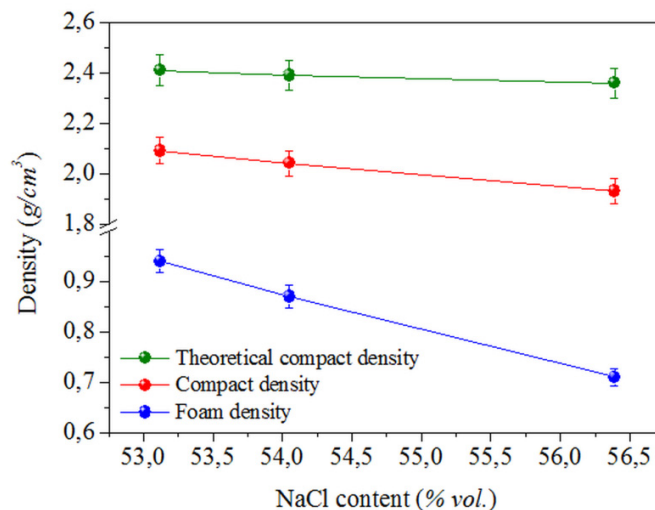


Fig. 5. Specimens density variations as a function of NaCl content: (a) theoretical compact density ρ_{th} , (b) compact density ρ_c , and (c) foam density ρ_f .

These empty spaces are generated because the liquid metal does not wet completely the NaCl particles caused by its superficial tension and consequently the liquid metal cannot infiltrate these small empty spaces inside the NaCl bed. The average value of the empty spaces in the aluminum foams obtained results to be 6.5 % for the NaCl particle size in the range I, 7.3 % for the NaCl particle size in the range II, and 8.4 % for the NaCl particle size in the range III. Given that the NaCl particle size decreases the contact points among NaCl particles are increased, which implies that the small empty spaces around these contact points inside the NaCl bed are increased too. As the liquid metal cannot infiltrate entirely these small empty spaces, the free spaces are increased as it was mentioned above in the second reason. Fig. 5 shows the fast decreasing in the ρ_f , which is attributed to two empty spaces contributions:

- The empty spaces found inside the Al/NaCl compact.
- The empty spaces left by the NaCl particles after their leaching.

The minimum obtained ρ_f value was 0.71 g/cm^3 , which corresponds only to 26.3 % of the aluminum density (2.7 g/cm^3).

Fig. 6 shows, as a function of the NaCl content, the variations in the P_f and P_c . The increase observed in the P_c is mainly associated with the diminishing in the NaCl particle size. As the particle size decreases, the contact point among NaCl particles increases, the empty spaces found inside the compact and located around these contact points are increasing too and consequently the liquid metal is not able to completely wet the NaCl particles due to its superficial tension and the liquid metal cannot infiltrate these small empty spaces inside the NaCl bed. The changes in the P_f of the aluminum foams, after the dissolution processes, indicated a continuous enhance with the increase in the NaCl content. In this case, a higher NaCl content produces a higher empty space in the foam when the NaCl is leached, and consequently, the porosity is enhanced, as it would logically be expected. On the other hand, in the Fig. 6 it is observed that the porosity difference between samples with high NaCl content (56.4 %vol.) and low NaCl content (53.1 %vol.) is enhanced when the NaCl content is increased. This tendency is attributed to the sum up of two contributions:

- The increase in the empty spaces found around NaCl particles, which increases with the diminishing of NaCl particle size and with the increase of NaCl content.
- The empty spaces left by the NaCl particles after their leaching which enhance with the increase of NaCl content.

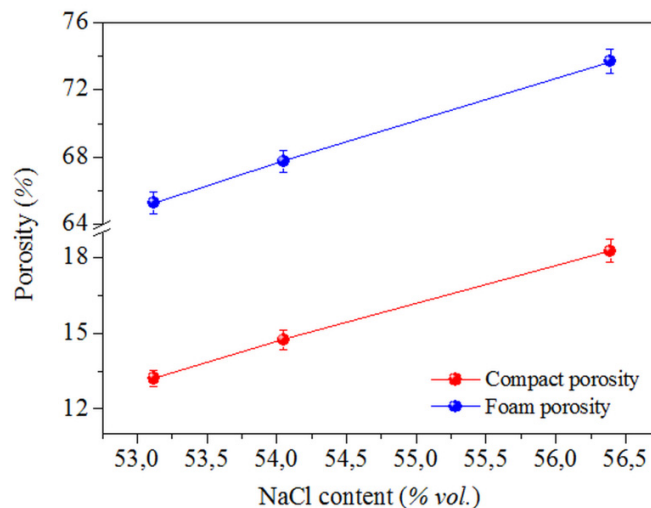


Fig. 6. Specimen porosity variations as a function of NaCl content: a) compact porosity P_c and foam porosity P_f .

The obtained aluminum foams presented densities between 0.71 and 0.94 g/cm³ and porosities between 65.3 and 73.7 %. The porosity values are higher to reported porosity results in the literature. In particular [Banhart (2001)] reported porosity values $\leq 65\%$ to metal foams obtained through casting around space holders processes when Al, Zn, Pb and Cu are employed as based metals to generate the metal framework. The highest obtained porosity in the aluminum foams processed in our laboratory, employing NaCl as space holder, can be attributed to the interconnection among cells. The interconnection among cells is caused by the permanent contact among NaCl particles and its immobility when the metallic container with aluminum is placed on the metallic mold with NaCl particles, as it is showed in the Fig. 1. A higher contact among NaCl particles involves a higher interconnection among cells and consequently a higher porosity when the NaCl particles are leached. The high interconnection among cells (roughly 100 %) was quantified through NaCl volume variation, before and after its leaching process. The difference between these volumes results to be $\sim 0.08\%$ in all cases (~ 0.031 grams) the average value of a NaCl particle with the lower particle size (e.g. particle size in the range III) is ~ 0.025 . This small value in the volume variation implies a great and almost total NaCl leaching into the sample, which indicates that roughly 100 % of NaCl was removed; hence the $\sim 100\%$ of interconnected cells in the sample is present. On the other hand, the high interconnection among cells is possible since the NaCl particles in the mold were not budged due to the contact between the metallic container with aluminum, and the NaCl-filled metallic mold guarantees the permanent contact among NaCl particles.

5. Conclusions

Open-cell aluminum foams containing porosities between ~ 65.3 to $\sim 73.7\%$ and minimum densities of 0.71 g/cm³ were successfully produced through the infiltration of liquid aluminum into a NaCl particles bed. A highly interconnected porous structure was obtained for all samples. This high interconnectivity among cells was caused by the contact among NaCl particles in the bed, which was promoted by the placement of the metallic container with aluminum on the metallic mold with NaCl particles in the configuration used in the present study. This configuration avoided the NaCl particles movement due to densities effects between aluminum and NaCl particles. The reached open cell structure is important for applications where the matter or energy transportation and the superficial area of the metal is essential. The results presented indicate that the use of infiltration processes are an excellent method to obtain aluminum foams with high porosity and a homogenous cells distribution.

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