



8th International Conference on Porous Metals and Metallic Foams, Metfoam 2013

Heat treatment of aluminium foam precursors: effects on foam expansion and final cellular structure

Jaime Lázaro^a, Eusebio Solórzano^a, Miguel Angel Rodríguez-Pérez^a*, Olaf Rämer^b,
Francisco García-Moreno^b, John Banhart^b

^a Cellmat Laboratory Condensed Matter Physics Department, University of Valladolid, Paseo de Belén 7, Valladolid, E-47011, Spain

^b Technische Universität Berlin, Hardenbergstrasse 36, Berlin, D-10623, Germany

Abstract

Aluminium foams produced by the powder metallurgical route present an initial –anisotropic– semisolid expansion that has a detrimental effect on the foamability and on the subsequent quality of the final cellular structure. The present work proposes a method oriented to modify the precursors' microstructure and overcome this problem. It is based on the use of heat treatments below the solidus temperature, and has been tested in AISi10 (0.8 wt.%TiH₂) commercial precursors from Alulight Company (Austria). The experimental results obtained have demonstrated that the thermal treatment induces notable changes in the microstructure of the precursor material. Then, these changes produce a reduced and smoother semisolid expansion and improve the quality of the final cellular structure, in terms of higher pore circularity and less amount of defects.

© 2014 Elsevier Ltd. This is an open access article under the CC BY-NC-ND license

(<http://creativecommons.org/licenses/by-nc-nd/3.0/>).

Peer-review under responsibility of Scientific Committee of North Carolina State University

Keywords: Aluminium foam; powder metallurgy; heat treatment; texture; semisolid expansion.

1. Introduction

Among all the routes to produce aluminium foams (Banhart (2001)), the most studied and commercially exploited is the powder metallurgical route (Yu et al. (1998)). However, this route is also known to present an initial, anisotropic, expansion in the solid and semisolid state that has a detrimental effect on the foamability and hence on the quality of the cellular structure (Lazaro et al. (2013)). This early expansion is caused principally by the mismatch between the TiH₂ decomposition and the melting point of the aluminium (or any of its alloys), something

* Corresponding author. Tel.: +34 983-18-40-35; fax: +34-983-42-31-92

E-mail address: marrod@fmc.uva.es

that produce elongated cracks in the solid precursor material and variable pore anisotropy and defects in the final foam. In addition, it has been recently discovered that these cracks, and their evolution (expansion), are directly correlated with microstructural anisotropy of the precursor material, induced during the powder compaction step (Lazaro et al. (2013), Lazaro et al. (2013)).

Several strategies have been proposed in the last years in order to suppress or reduce the magnitude of the initial expansion (low melting point alloys (Lehmhus and Busse (2004)), treated TiH_2 (Matijasevic-Lux et al. (2006)), compaction applying vacuum (Jimenez et al. (2009)), etc.) and/or its anisotropic behavior (isotropic processing (Weise et al. (2003)), foaming under pressure (Garcia-Moreno and Banhart (2007)), etc.). All of them exhibit a notable improvement on the precursor foamability and the quality of the cellular structure. However, these strategies are not a final solution to the problem by themselves, and in fact, they also present some limitations when considering the production at industrial scale. Therefore, other possible methods to overcome the mentioned problems in the PM route need to be considered and analyzed.

In this sense, in the PM industry (not foam industry) it is already known and well accepted that annealing (heat-treatment) tends to homogenize and improve the microstructure and the inter-particle bonding in the compacted material (Jones (1960)). However, this idea has not been still applied or studied in detail in the case of aluminium foam precursors, probably due to the difficulties associated with the control of the early decomposition of the TiH_2 .

For this reason, this investigation attempts, for the first time, the evaluation of the effects of this post-processing technique on the following aspects: the aluminium foam precursor's microstructure, the foamability of the precursors and the quality of the cellular structure of the foams produced.

2. Experimental

2.1. Materials

Aluminium foam precursors used in this investigation were supplied by Alulight Company (Austria) in the form of extruded strips (E-S) of $20 \times 5 \text{ mm}^2$ cross section. This commercial material is based on a mixture of Al and Si powders (10 wt.%), containing 0.8 wt.% of TiH_2 powders.

2.2. Heat treatments

For each heat treatment, a piece of, as-received, precursor strip ($L \sim 15 \text{ cm}$) was introduced inside a pre-heated furnace at the selected temperature, and held there during a fixed period of time. Afterwards the precursor was extracted from the furnace and allowed to cold down to room temperature.

Selected temperatures for the heat treatments ranged from $400 \text{ }^\circ\text{C}$ to $575 \text{ }^\circ\text{C}$ and the holding times were between 0.25 hours and 10 hours. It can be appreciated that the solidus temperature of the alloy ($\sim 577 \text{ }^\circ\text{C}$) was never reached, thus keeping the precursor in the solid state. However, as the treatment temperature was increased, the holding time was reduced. By doing this we tried to avoid too much gas generation (and/or possible losses) and the premature expansion due to excessive softening of the material.

2.3. Characterization

After performing the heat treatments, the precursors were analyzed in order to evaluate the possible changes induced in the macro (porosity), meso (crack generation), and micro-structure (changes in crystallographic texture). Their foamability was studied by using X-ray radiography (Garcia-Moreno et al. (2004)). Finally, the quality of the solid cellular structure was also analyzed.

3. Results and discussion

3.1. Effect on precursor's microstructure

3.1.1. Initial porosity

The first and more observable consequence of the heat treatment is a change of precursor's density. Fig. 1 shows the measured porosity values ($1 - \rho/\rho_s$), after each heat treatment. It can be appreciated that below or close to 500 °C the precursor's porosity is very low and comparable to the as-received material. However as treatment temperature increases the porosity also increases, being this tendency more clear for the higher temperatures (higher slope). As it should be expected, near the solidus temperature (565 and 575 °C) the porosity generation is so quick that, in fact, an already foamed material ($p > 30\%$) can be obtained. The error bars have not been included in the figure to allow a better visualization of the trends, but they are in the order of 1-4%, thus suggesting a high reproducibility level on this parameter with the different treatments applied.

3.1.2. Crack generation

The mentioned change in the density has its origin in the cracks generated due to the early TiH_2 decomposition while the material is still in the solid state (Lazaro et al. (2013)). The aspect of these cracks after four different treatments (2 temperatures and 2 periods) is presented in Fig. 2.

For low treatment temperatures (up to 535 °C) the cracks exhibit an elongated shape in the extrusion direction (Fig. 2a). The cracks tend to increase in size with the holding time inside the furnace, but keeping the elongated shape (Fig. 2b) (Lazaro et al. (2013)). However, the morphology of the cracks changes completely when the treatment temperature is set closer to the solidus temperature of the alloy. Although some elongated cracks can also be found, the tendency is to obtain smaller and rounded cracks (some of them marked with circles in Fig. 2c). This can be better appreciated when the holding time increases (Fig. 2d).

These differences can be understood taking into account the reduction of the mechanical properties of the precursor material when the temperature reaches values closer to the solidus temperature. This would allow the metal microstructure to better adapt to the generated internal gas pressure and derive in more circular initial cracks than when working at lower temperatures, where the matrix strength is still high.

3.1.2. Texture changes

The previous results suggested that also the metal microstructure, and in particular the inter-particle bonding, should have suffered changes during the heat treatment. Assuming that the intrinsic crystallographic anisotropy (or texture) of PM precursors and their behavior during semisolid expansion are connected through the inter-particle strength and its distribution (a conclusion recently proposed in a previous work by the authors (Lazaro et al. (2013))), the texture of the material should have also changed during the process.

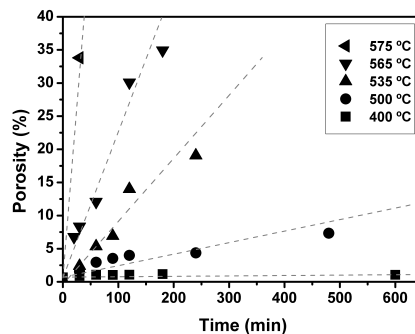


Fig. 1. Porosity induced into the precursor material after the heat treatment.

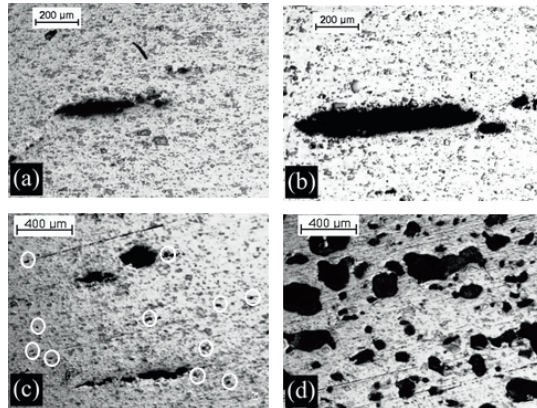


Fig. 2. Aspect of the cracks generated into the precursor due to the heat treatments. (a) 535 °C, 2h; (b) 535 °C, 4h; (c) 565 °C, 1h; (d) 565 °C, 2h.

In order to evaluate this hypothesis, texture measurements were performed on selected samples. For that, a Bruker diffractometer (mod. D8 Discover) was used, and the collected data analyzed by using Multex software. The changes in the pole figure corresponding to the Al_{fcc} 220 peak (the most characteristic in this type of materials (Lazaro et al. (2013))) is shown in Fig. 3.

A first observation indicates a similar pattern in both figures. This suggests that the treatment does not change the type of texture of the material, contrary to what happens commonly during post-processing of metals by annealing or rolling (Eddahbi et al. (2007)). This effect is probably associated to the oxide network present in the precursors, which would allow only the annealing recrystallization inside and/or the union points between the constituent powder particles.

However, the two pole figures show some small differences. On one hand, the intensity of the whole pattern, and especially at the peak zones, suffers a reduction after the heat treatment, something suggesting a reduction of texture. In fact, the quantitative analysis done over the collected data has shown an increase of the isotropic fraction (non-texturized precursor volume fraction) of more than 10%. In particular the change goes from 51 vol.% in the as-received material, to 65 vol.% in the precursor after 1h at 565 °C. On the other hand, the symmetry of figure 3b is a clear indicator of higher homogeneity. Thus, assuming the hypothesis mentioned before (Lazaro et al. (2013)), the inter-particle bonding should have become more homogeneous (isotropic) inside the precursor, something that agrees with the change in the aspect of the generated cracks (figure 2c-d).

3.2. Effect on foamability

Figure 4 presents the results of the radiography foaming tests. The aspect of the expanding precursor (cross section view) during the semisolid state ($T \sim 580^\circ\text{C}$) can be appreciated in figure 4a. The as-received material exhibits higher expansion in both height and weight directions (but non growth in the extrusion direction, as already known (Lazaro et al. (2013))). However as the treatment temperature increases, the initial expansion in the semisolid phase is reduced tending to a more isotropic behavior, at least laterally. In fact, in the case of precursor treated at 565 °C for 1 h, the early –semisolid– growth takes place only in height. This change in the initial expansion could be associated to gas losses during the treatment (thought cracks near the surface), but also to a possible ‘treatment’ of the TiH_2 that would delay its decomposition. On the other hand, the changes induced in the microstructure would cause that the gas released inside the treated samples, during foaming heating, would tend to go to the generated cracks, therefore not creating new ones and not expanding the damage as it happens in the untreated sample.

Regarding the later expansion in the liquid state (figure 4b) it can be observed that the as-received material presents larger pores. For the precursor treated at 535 °C for 2h, the expansion give as a result a foam high lower expansion. In addition this sample presents un-foamed parts at the top and bottom, as a consequence of the mentioned possible gas losses through the interconnected cracks near the surface during the heat treatment. Finally, in the case of the precursor treated at 565 °C for 1h, the resulting foam exhibit an expansion similar to the as

received material in magnitude, but with more spherical appearance. In addition, the internal structure presents more and smaller pores, which in principle seems to be more circular (see next section for the quantification of this observation).

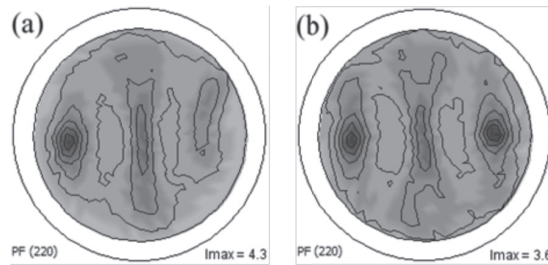


Fig. 3. Pole figure (Al_{220}) of the precursor material. (a) As-received samples; (b) After 1h at $565\text{ }^{\circ}\text{C}$.

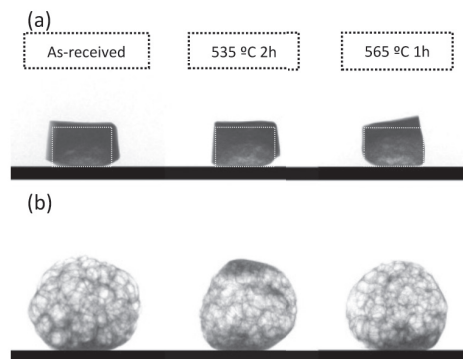


Fig. 4. Radioscopy images (cross section view) at two stages of the foaming process. (a) Semisolid expansion; (b) Final -liquid- expansion. Initial precursor shape is also indicated ($5 \times 10\text{ mm}^2$).

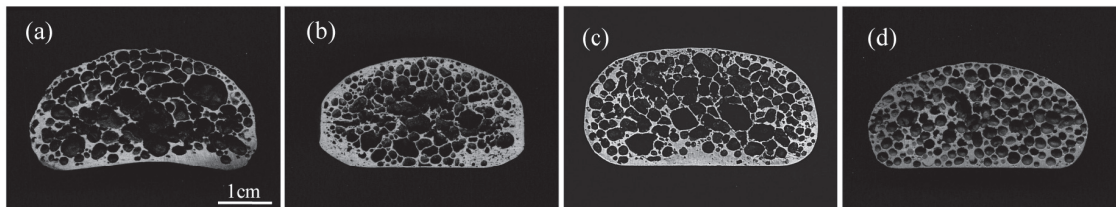


Fig. 5. Cross section of the foams produced after different heat treatments. Same foaming conditions: $710\text{ }^{\circ}\text{C}$, 9min. (a) As-received material; (b) $500\text{ }^{\circ}\text{C}$, 8h; (c) $535\text{ }^{\circ}\text{C}$, 2h; (d) $565\text{ }^{\circ}\text{C}$, 1h.

3.3. Effect on cellular structure

In order to evaluate the final cellular structure, one piece of each type of treated precursor ($L \sim 4\text{ cm}$) was foamed under the same conditions. It was introduced inside a pre-heated furnace at $710\text{ }^{\circ}\text{C}$, held there during 9 minutes and then extracted and cooled down, by using compressed air at 5 bar, until complete solidification. The characterization of the cellular structure was done by image analysis software after cutting the samples in half. The foams produced with as-received precursor (figure 5a) exhibits the typical defects for this type of materials: large and/or elongated pores, missing cell walls, etc. After the treatment of the precursor material at $500\text{ }^{\circ}\text{C}$ during 8h, the final foam (figure 5b) exhibits smaller pores, although with similar irregular shape, and also defects. In addition, it is also possible to detect the presence of an un-foamed zone near the surface due to the mentioned gas losses. For

precursors treated at 535 °C during 2h, the expansion derives in a structure with less external skin and less amount of missing cell walls (figure 5c). However the pores still exhibit an anisotropic or irregular shape. Finally, when foaming the precursors treated at higher temperature (1h at 565 °C) the resulting foam (figure 5d) presents a cellular structure with higher quality. First, it can be observed that no un-foamed (or even drainage) zones appear, consequence of the better crack nucleation during the treatment: small and round cracks that do not connect and therefore do not promote gas loss to the exterior. On the other hand, the pore size is also smaller, in comparison to the rest of foams shown in figure 5, and the shape of the pores exhibit much higher circularity (C) ($C > 0.85$ in Fig. 5d, while $C < 0.7$ in Fig. 5a).

4. Conclusions

The applicability of heat treatments to PM aluminium foam precursors has been demonstrated in the present study.

Results obtained have shown that heat-treated precursors exhibit a more homogeneous and isotropic microstructure.

In addition, a reduced and smoother semisolid expansion has been observed, in comparison with as-received materials, something which clearly influences the later expansion in liquid state.

Improvement effects are particularly clear in precursors pre-heated at 560°C for 1 hour. For these samples, a better cellular structure in terms of pore morphology and amount of defects has been observed as a consequence of the more homogeneous foaming behavior.

This low cost post-processing method could be used alone or together with other known strategies to improve the quality and production of PM aluminium foams.

Acknowledgements

Financial support from the Spanish Ministry of Science and Innovation and FEDER funds (MAT2009-14001 CO2-01 and MAT 2012-34901) and the Junta of Castile and Leon (project VA174A12-2) are gratefully acknowledged

References

- Banhart, J., 2001. Manufacture, Characterization and Application of cellular metals and metallic foams. *Progress in Materials Science* 46, 559-632.
- Eddahbi, M., Jimenez, J.A., Ruano, O.A., 2007. Microstructure and creep behaviour of an Osprey processed and extruded AlCuMgTiAg alloy. *Journal of alloys and Compounds* 433, 97-107.
- Garcia-Moreno, F., Banhart, J., 2007. Foaming of blowing agent-free aluminium powder compacts. *Colloids and Surfaces A* 309, 264-269.
- Garcia-Moreno, F., Fromme, M., Banhart, J., 2004. Real time X-ray radioscopy on Metallica foams using a compact microfocus source. *Advance Engineering Materials* 6, 416-420.
- Jimenez, C., Garcia-Moreno, F., Mukherjee, M., Goerke, O., Banhart, J., 2009. Improvement of aluminium foaming by powder consolidation under vacuum. *Scripta Materialia* 61, 552-555.
- Jones, W.D., 1960. *Fundamental Principles of Powder Metallurgy*, Edward Arnold Ltd, London.
- Lazaro, J., Laguna, E., Solorzano, E., Rodriguez-Perez, M.A., 2013. Effect of Microstructural Anisotropy of PM Precursors on the Characteristic Expansion of Aluminium Foams. *Metallurgical and Materials Transactions B* 44, 984-991.
- Lazaro, J., Solorzano, E., de Saja, J.A., Rodriguez-Perez, M.A., 2013. Early Anisotropic Expansion of Aluminium Foam Precursors. *Journal of Materials Science* 48, 5036-5046.
- Lehmhus, D., Busse, M., 2004. Potential New Matrix Alloys for production of PM Aluminium Foams. *Advanced Engineering Materials* 6, 391-396.
- Matijasevic-Lux, B., Banhart, J., Fiechter, S., Görke, O., Wanderka, N., 2006. Modification of titanium hydride for improved aluminium foam manufacture. *Acta Materialia* 54, 1887-1900.
- Weise, J., Stanzick, H., Banhart, J., 2003. Semi-solid processing of complex-shaped foamable material. In: *Cellular Metals: Manufacture, Properties and Applications*, Editors: J. Banhart, Fleck, N.A., Mortensen, A., MIT-Verlag, Berlin, 169-174.
- Yu, C.J., Eifert, H.H., Banhart, J., Baumeister, J., 1998. Metal foaming by a powder metallurgy method: Production, properties and applications. *Materials Research Innovations* 2, 181-188.