

MANUFACTURING OF ALUMINUM FOAMS USING THE SINTERING DISSOLUTION PROCESS

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ABSTRACT

This work reports the processing of aluminum (Al) foams with pore size in the range from 1 to 2 mm and minimum densities of 0.56 g/cm³. The foams were manufactured using a powder metallurgy method with a space holder (also known as Sintering Dissolution Process, SDP), carbamide as space holder particles and using a device with controlled atmosphere in the sintering step to avoid the quickly oxidation of metal. The foams obtained showed a porous structure with regular pore size and pores distribution. The plots of stress σ vs strain ϵ indicated σ_{\max} values between 61.39 and 0.04 MPa (0.0 and 80 %vol. of CH₄N₂O, respectively). On the other hand, the foam porosity increased from 11.84 to 79.44 % (0.0 and 80 %vol. of CH₄N₂O, respectively). The results are interpreted in terms of increase the SHP content in the sample.

Keywords: *Aluminum foams, Sintering dissolution process, Cellular metals*

RESUMEN

Este trabajo reporta la fabricación de espumas de aluminio con tamaños de poro en el rango de 1 a 2 mm y densidades mínimas del orden de 0.56 g/cm³. Las espumas fueron fabricadas usando el proceso de sinterización–disolución, con carbamida como material generador de poros y empleando un dispositivo con atmósfera controlada en la etapa de sinterización para evitar la oxidación del metal. Las espumas obtenidas mostraron una estructura porosa con una distribución de poros y tamaño de poro regular. Las gráficas de esfuerzo σ vs deformación ϵ indicaron valores de esfuerzo máximo σ_{\max} entre 61.39 MPa y 0.04 MPa (0.0 y 80 %vol. de carbamida, respectivamente). Por otra parte, la porosidad de la espuma incremento de 11.84 a 79.44 % (0.0 a 80 %vol. de carbamida, respectivamente). Los resultados son interpretados en términos del aumento en el contenido de carbamida en la muestra.

Palabras Clave: *Espumas de aluminio, Proceso de sinterización–disolución, Metales celulares*

1 INTRODUCTION

The technology of powder metallurgy (PM) has been highlighted as a source in the making of new materials and light parts providing a reduction of production costs mainly in the automobile industry. Currently, the PM has resulted to be competitive regarding the casting, forging and machining processes particularly employed in the manufacture of complex metallic parts, and which can be made using the PM processes. Nowadays, the PM processes have been used to fabricate porous metals. These porous metals (also called cellular metals) with high porosity have been developed to use them as new functional materials, since these materials present a unique combination of physical and chemical properties which can be derived from their cellular structure [1]. Porous 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 [2,3]. Cellular metals can be manufactured through a great variety of methods, including processes with the metal in the solid, liquid and gaseous state. Commonly used methods based on the solid state involve the incorporation of a removable space holder phase; e. g. a conventional powder metallurgy (PM) methods, involving removable particles blended with those of metal's [4,5]. Zhao and Sun [6] developed a technique to manufacture open–cell foams at low cost using the PM route, known as the sintering and dissolution process (SDP). To date, the SDP method has been used to obtain foams, which widely show good properties and interconnected pores (important for any application where a large superficial area of the foam is necessary). The SDP method consists of four stages: i) Mixing metal powder with the Space Holder Particles (SHP), ii) Compacting the mixture to obtain a green compact, iii) Dissolution of the SHP with an appropriate solvent to obtain a cellular metallic structure, and iv) Sintering of the cellular metallic structure to produce metallurgical bond among the metallic powders. In the present work, the SDP route is employed to fabricate porous aluminum (with increasing porosity to obtain closed pore and open pore foams) with regular pore size and good mechanical properties, using Al powders and carbamide as SHP.

2 EXPERIMENTAL TECHNIQUES

The granular $\text{CH}_4\text{N}_2\text{O}$ and Al–foams morphology was characterized using a digital camera (Sony 3D) to determine the obtained granules' form and pore distribution. Scanning Electron Microscopy (Jeol JSM 6610 HLV) operated at 20 keV was used to determine the quality of the metallurgical bond among the metallic powders. The mechanical properties of the specimens, measured through compression tests, were conducted on an MTS–810 materials testing machine with a crosshead speed of 0.1 mm/min. For each specimen, the weights of the initial Al powders, $\text{CH}_4\text{N}_2\text{O}$ granules, Al/ $\text{CH}_4\text{N}_2\text{O}$ green compact and the resultant Al foam before and after sintering processes were measured using an Ohaus balance with an accuracy of 0.1 %. Using these data, the green compact porosity P_c , and foam porosity P_f , were determined through the equations 1 to 3 [7,8]. Where, ρ_c is the green compact density, ρ_{th} is the green compact theoretical density, ρ_{Al} is the Al density, W_{Al} is the Al weight fraction in the mixture, ρ_{SHP} is the carbamide density and ρ_f is the foam density. The densities of green compact and foams were determined using the conventional equation $\rho=M/V$ (M=mass, V=Volume).

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

$$\rho_{th} = \rho_{Al} * W_{Al} + \rho_{SHP} * (1 - W_{Al}) \quad (2)$$

$$P_f = 1 - \frac{\rho_f}{\rho_{Al}} \quad (3)$$

3 RESULTS AND DISCUSSION

The metal powder used in the present study to produce the main framework of the foams was Al powder with 99.0 % purity and particle size of $\sim 74 \mu\text{m}$ provided by “*Stannum de México S. A.*” (Fig. 1a). As it can be seen in the figure, the particles had a roughly equiaxed shape. In order to increase porosity and to create an interconnected structure, granular $\text{CH}_4\text{N}_2\text{O}$ provided by “*Droguería Cosmopolita S. A. de C. V.*” with particle size in the range from 1 to 2 mm was selected as the SHP (Fig. 1b). The aim to employ an SHP with a variable size range is that the stiffness and the strength of foams increase with multi-size cells [9].

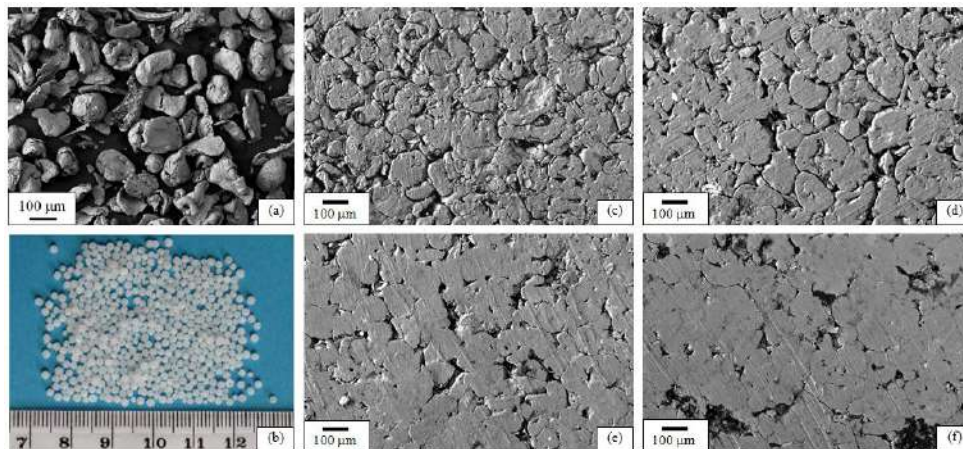


Figure 1. a) SEM secondary electron images of Al powders with a size of $\sim 74 \mu\text{m}$, b) optical image of granular $\text{CH}_4\text{N}_2\text{O}$ with a size range from 1 to 2 mm, and SEM secondary electron images showing the superficial bonding of compacted aluminum particles sintered at temperatures of: c) $530 \text{ }^\circ\text{C}$, d) $560 \text{ }^\circ\text{C}$, e) $590 \text{ }^\circ\text{C}$ and f) $620 \text{ }^\circ\text{C}$.

The total weight of Al and $\text{CH}_4\text{N}_2\text{O}$ mixture was $\sim 4.5 \text{ g}$ in all experiments. The mixing of the Al powders and $\text{CH}_4\text{N}_2\text{O}$ granulates was performed in an agate mortar for 30 min. With the aim of ensuring good adhesion of Al powders, 2 % vol. of ethanol was sprayed on $\text{CH}_4\text{N}_2\text{O}$ granules before the mixing stage to obtain a sticky surface. Then, the mixture was put into a steel mold and uniaxially pressed at 400 MPa for all samples to produce cylindrical green compacts with $\sim 16 \text{ mm}$ in diameter and $\sim 13 \text{ mm}$ in length. The $\text{CH}_4\text{N}_2\text{O}$ fraction of the green compact was dissolved by two-time immersion in a water bath at $25 \text{ }^\circ\text{C}$ for two hours. In all cases the dissolution stage of the process was carried out before the sintering stage. Specimens containing 100 % vol. of Al powders were sintered at temperatures from $530 \text{ }^\circ\text{C}$ to $620 \text{ }^\circ\text{C}$ (with increments of $30 \text{ }^\circ\text{C}$ each experiment) for 180 minutes, employing a controlled atmosphere device [10], in order to determine the optimum sintering temperature, T_s . Following this procedure, T_s was found

to be 620 °C, since at this temperature the best particle bonding was observed in the SEM. At temperatures of 530 and 560 °C poor particle bonding was found (Figs. 1c–f) and at a temperature of 590 °C a little particles–bonding was observed. As temperatures below 620 °C did not result in substantial bonding, the temperature of 620 °C was selected for all subsequent sintering treatments. Figure 2 shows the series of Al foams containing from 100 to 20 %vol. of Al powders, formed using granular CH₄N₂O after the sintering process. Macroscopically, the samples presented a regular CH₄N₂O distribution, which results in Al foams with a highly homogeneous framework, as it is required for reliable measurements of mechanical properties in this kind of material. The sintering processes of the green foams were carried out in two steps and using the device with controlled atmosphere proposed in [10]: i) the device containing the sample was put into a vertical electrical furnace and the temperature was increased from 25 to 250 °C at a heating rate of 10 °C/min; the samples were held at 250 °C for 60 min to eliminate the remaining CH₄N₂O in the green compact, and b) the temperature was increased from 250 to 620 °C at a heating rate of 10 °C/min and the samples were held at this temperature for 180 min to form a metallurgical bond among the Al powder particles. Finally, the Al foams were finely polished with a silicon carbide paper in order to obtain a length/diameter ratio of 0.8 [11]. The final dimensions of the Al foams were also measured to determine their volume and density.

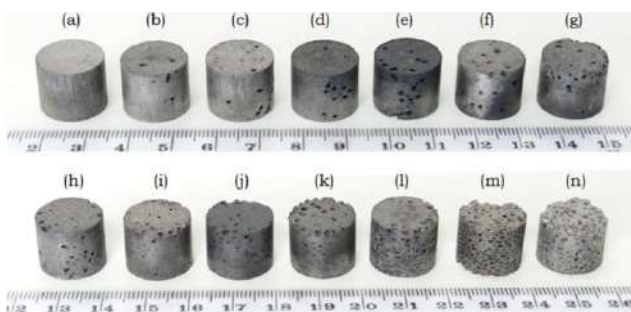


Figure 2. Image of an aluminum foams series after sintering process: a) 100, b) 80, c) 75, d) 70, e) 65, f) 60, g) 55, h) 50, i) 45, j) 40, k) 35, l) 30, m) 25 and n) 20 %vol. of Al powders.

Figure 3a shows the changes in porosity P_c , and density ρ_c , of the green compact samples as a function of the CH₄N₂O concentration. In this plot, ρ_c decreased with increasing SHP content. The decreasing in ρ_c is explained by the lower value of the density of CH₄N₂O (1.34 g/cm³) compared to Al (2.7 g/cm³); as the proportion of SHP in the compact increases, the density decreases. On the other hand, the P_c quickly increases until 15.5 % with increments in the SHP up to 45 %vol. Contents of SHP higher than 45 % produce a change relatively steady in P_c with a slight variation of approximately 1.8 %. This could be associated with a better packing of different size particles achieved at 45 %vol. of SHP (the amount of porosity in the green compact is strongly related to the packing efficiency of the Al–Al powders, CH₄N₂O–CH₄N₂O particles and Al–CH₄N₂O powders and particles, respectively; or it could be due to a higher degree of compaction implying a mechanical weakening of the CH₄N₂O, and as a consequence, a deformation of the SHP, which produces a decreasing on the empty volume inside the sample. Figure 3b shows, as a function of the CH₄N₂O content, the changes in the porosity, P_f and foam density, ρ_f of Al foams after the SHP dissolution and sintering processes. P_f increases while ρ_f decreases, both linearly with SHP content, as it would logically be expected. Although the main contribution to the P_f value is from the SHP, the P_f is greater than the SHP added content. Until

increments of 50 % vol. of SHP, the P_f appears to be around ~ 8 % more than the amount of space holder content (i.e. for a $\text{CH}_4\text{N}_2\text{O}$ content of 20 % at the green body stage the resulting foam has a porosity of ~ 30 %, for content of 30 % the porosity is ~ 38 % and so on). On the other hand, when the SHP amount is increased, the P_f slowly increases (4 % approximately) compared to the initial tendency (i.e. for a $\text{CH}_4\text{N}_2\text{O}$ content of 55 % at the green body stage, the resulting foam has a porosity of around ~ 60 %, for content of 65 % the porosity is around ~ 69 % and so on). This tendency can be caused by the voids present among the Al particles due to imperfect packing, producing a small contribution to the porosity. The effects of $\text{CH}_4\text{N}_2\text{O}$ volume fraction on the mechanical properties of the processed Al foams were assessed through compression tests.

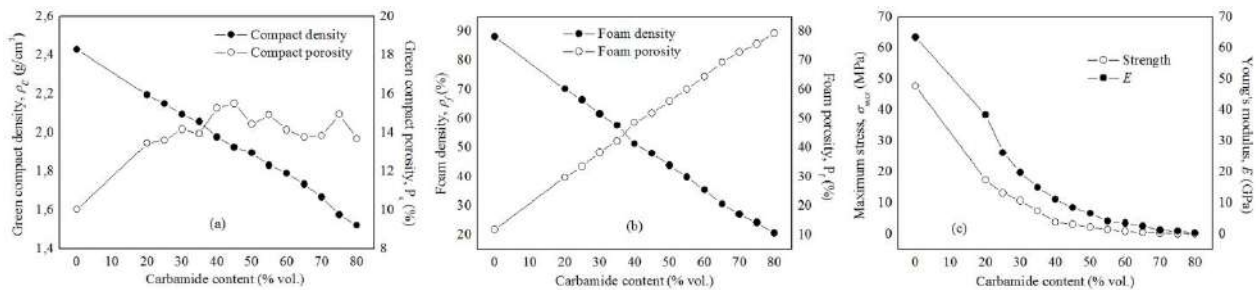


Figure 3. Plots of: a) changes in the ρ_c and P_c , b) changes in the ρ_f and P_f , and c) changes in the σ_{max} and E . Three as a function of the $\text{CH}_4\text{N}_2\text{O}$ content.

Figure 3c shows the plot of maximum stress σ_{max} , and young's modulus E , both vs deformation ϵ , for the foams processed from 0.0 to 80 % vol. of SHP. The specimen with 100 % vol. of Al powders showed a $\sigma_{max}=47.77$ MPa and $E=63.42$ GPa. These values are close to the reported of 55 MPa and 69 MPa values, respectively, for the pure Al obtained through casting processes [12]. This obtained values indicate a good metallurgical bond among particles, caused by the uniaxial compression step and sintering at the correct T_s , previously determined. In this plot, the σ_{max} decreases as the SHP content increases. The value of $\sigma_{max}=47.77$ MPa for the specimen with 100 % vol. of Al powders decreases to $\sigma_{max}=0.04$ MPa for the specimen with 20 % vol. of Al powders (when the SHP content increase from 0.0 to 80 % vol.). Similarly to previously seen trends in other foams [13] this diminishing in σ_{max} is attributed to the increasing of foam porosity caused by the SHP increasing, which implies a lower material amount in the sample and consequently a lower area supporting the load. Note that the σ_{max} value for the sample without space holder particles (100 % vol. of Al powders) also supports this trend, since the value of 47.77 MPa is lower than the expected σ_{max} value of 55 MPa for the pure obtained Al through casting processes. This is due to a significant amount of porosity generated by the inclusion of void spaces among the metal particles, which implies a lower area supporting the load, and consequently a reduction in σ_{max} value for the 100 % vol. of Al powders sample. Finally, The E decrease from ~ 63.4 to 0.29 GPa (0.0 to 80 % vol. of $\text{CH}_4\text{N}_2\text{O}$, respectively). Similarly, this tendency is caused by the lower area supporting the load as the SHP amount is increased.

4 CONCLUSION

Metallic foams containing from 20 to 100 % vol. of Al powders were successfully produced by the sintering dissolution process and utilizing carbamide as space holder particles. The T_s of 620 °C was the optimum temperature to produce Al foams with good metallurgical bond among its

particles as it is observed in the SEM images, which indicates that the device's internal atmosphere was successfully controlled. A highly porous structure was obtained for the sample with 80 %vol. of carbamide which resulted to be ~81 %. Also, good mechanical properties were observed in the strength values obtained through compression tests, indicating that the T_s was properly selected. The results presented here indicate that the use of SDP is an excellent method to obtain Al foams with homogeneous pore distribution and good mechanical properties.

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