APPLICATION OF SEMI-SOLID TECHNOLOGY FOR THE MANUFACTURING OF THE OPEN-CELL METALLIC FOAMS

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Abstract: *Although the main processing routes of metallic foams fabrication are based in foaming of liquid metals, open-cell porous materials can be produced by several manufacturing techniques. The technology of semi-solid processing was recently introduced in the scenario, and has already showed to be an interesting option for the production of open cell metallic materials (previous work by the authors). The main advantage of this process is related to non-Newtonian behavior of the semi-solid metal which allows high flow to low applied stress, making it possible the infiltration of removable pre-forms. This work investigates the infiltration of semi-solid thixotropic AA2011 aluminium alloy into pre-forms of sintered and non sintered NaCl particles with different sizes, by thixoforming processing. Processing parameters are investigated and obtained products are characterized: internal architecture of porous, by tomography and processing of images; physical properties such as actual and relative densities; thermal conductivity and diffusivity. Results show a great potential for application of semi solid state technology for the fabrication of porous metals.*

Keywords: *cellular metals, open-cell metallic foams, thixoforming, AA2011 aluminium alloy.*

1. INTRODUCTION

The cellular metals are a new class of materials which have as main features interesting and unique combinations of physical and mechanical properties, such as: high stiffness associated with low specific weight; high resistance to compression associated with the capacity to absorb energy in impacts events. Furthermore, the cellular metals are also effective for sound absorption and mechanical vibrations absorption (SIMANCIK, 2002).

The cellular metals can be mainly classified as metal sponges with open cell or metal foams with closed foam. The metal sponges have high surface area (as walls of cells) with high electrical and thermal conductivity, which enable applications such as; support of catalysts, heat exchangers, filters for high temperatures, consumable electrodes and absorbers electromagnetic (WADLEY, 2002; CURRAN, 2003). On the other hand, the metal foams due to its high resistance to compression, low density and the ability of high plastic deformation for low and constant stress (ASHBY et al., 2000), find application in the automotive industry as absorbers of impact, as well as acoustic and thermal insulators providing greater comfort to passengers (BERG, MAYSENHÖLDER and HAESCHE, 2003). In the Figure 1 it is showing the typical structure of foam and sponges metallic.

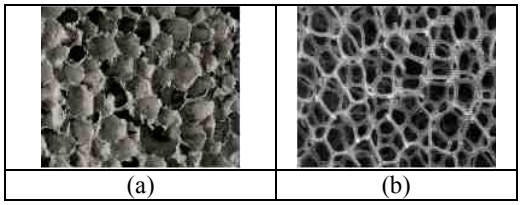

Figure 1: Structure typical of cellular metals; (a) metal foam – closed cells; (b) metal sponges – open cells.

The most of the cellular metals manufacturing process are based on processing liquid state or powder metallurgy. In the liquid state the foams are produced by application of the gases into the melt which promote the formation of liquid bubbles or by addition of the foaming agents in melt; these agents release gas during its dissociation in the liquid metal this way leading to formation of bubbles. Furthermore, metal sponges can be produced in the liquid state by infiltration of the liquid metal in space holders which are removed after process by washing with water or by infiltration in a polymeric sponges structure; this process it is known as replication process and the polymeric structure can be removed by burn in a furnace. The powder metallurgy also can be used in the metal foam manufacturing process by mix of metal powder and foaming agents, compaction of mixture and foaming inside the furnace. It is also possible the compaction of the metal powder and spaces holders and after compaction its sintering at an appropriate temperature; the space holders are removed by washing or mechanical vibrations.

In recent years it begun to be explored a new route by manufacturing of metal foam which utilize of the semi solid technology to produce cellular metals. The technology of semi solid process has been explored in research in the recent

decades. This technology began in 70 decade in Massachusetts Institute of Technology during measures experiments of viscosity of Pb-Sn alloy in the interval *liquidus/solidus*. These results showed which the shear stress increases slightly with decrease of temperature below the *liquidus* line, whereas, in conventional solidification occurs unlike where the formation of dendrites cause a strong reduction of the flow of material even for low fractions solid. This occurs because the presence of globular solid inside liquid metal, thus, leading to its non-Newtonian and thixotropic behavior (FLEMINGS, 1991). The Figure 2 has shown a typical globular structure. Non-Newtonian behavior of the semi-solid metal slurry allows high flow to low applied stress and the tixotropic behavior is related the possibility of maintaining of a flow non-turbulent regime during the force application. These characteristics mean that this technology is very promising for producing of cellular metal.

The most widely studied cellular metals and developed are based on aluminum and its alloys, other metals such as Fe, Cu, Pb and Zn can also be used, provided that the processing technique used is adequate. The metal structure of a cell typically consists of between 70 to 95% of pores and the main reason for their use is their high strength and stiffness, low weight and lower density with respect to the bulk material.

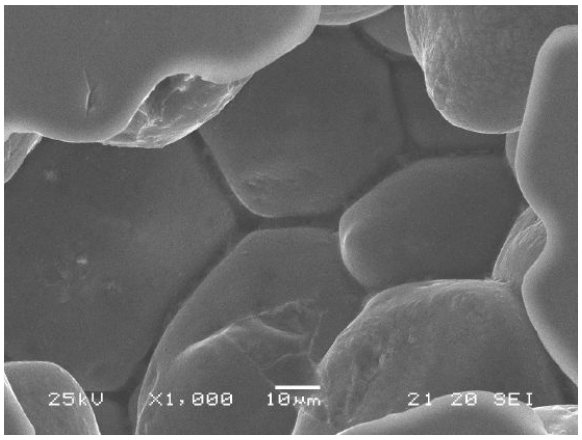

Figure 2: Globular structure of Al–3,0wt%Si–0,5wt%Mg thixocasting alloy and rewarmed to a fraction solid of 45%. (BENATI, 2008).

The cellular structure of porous metals is the key factor that determines its properties; this fact makes it essential to its structural characterization. The main objective is to characterize the structural parameters and understand their influence on the properties of cellular metals (KRISZT, 2002). The cellular metals are structurally characterized by its cell type, closed (foam) or open (sponge), relative density, size of the cell, shape of the cell, cell thickness and anisotropy (ASHBY et al., 2000).

2. MATERIALS AND METHODS

The alloy used for the manufacture of sponges was the aluminum alloy AA2011, belonging to the series 2XXX (worked alloys), and its basic chemical composition in accordance with the Metals Handbook (Murray, 1985) is Al - 5.5 wt% Cu, as show in Table 1. This alloy presents a reasonable range of temperatures between *solidus* and *liquidus* lines allowing an appropriate control of operating parameters in semi solid state. Furthermore, the alloy is AA2011 widely used in automobile industry for the manufacture of various components.

Table 1: Chemical composition of the alloy AA2011 (% by weight) used in the work.

Element	Cu	Bi	Fe	Si	Mn	Zn	Cr	B	Al
%	5,29	0,47	0,39	0,27	0,02	0,08	0,01	~0,001	Balance (~93%)

The determination of the range of temperatures between the *liquidus* and *solidus* lines is crucial for defining the processing parameters in the semi-solid state, or to obtain the slurry with globular structure used in the manufacture of metal sponges. To determine this range the curves were recorded for heating and cooling during the solidification of the alloy AA2011 by testing of differential scanning calorimetry (DSC). It was obtained for the alloy in study from the DSC curves and the use of dedicated software the curve of variation fl x T (liquid fraction x temperature). From this curve it was possible to determine the values of T liquidus (100% liquid) as equal to 658° C and T solidus (100% solid) as equal to 538 ° C and thus the range of solidification of the alloy (Tl -Ts) as 120 º C. The range of solidification has

revealed to be quite broad, favorable for the treatment of control melting partial (FPC). A wide range of solidification allows greater flexibility and control of parameters of tixoforming.

The microstructure of the alloy AA2011 was examined by optical microscopy in the state as extruded. The characteristics of the original microstructure is important for defining the parameters of the thixocasting, for example, a refined microstructure requires a shorter time at a temperature T above the solidus line for the occurrence of globularization when compared to more coarse microstructures. Figure 3 shows the microstructure of the alloy AA2011 after preparing metallography. This microstructure presents a non-dendritc microstructure with small grains almost equiaxiais - this is due to the occurrence of recrystallization during the extrusion process. Within the primary α phase can be observed the presence of precipitates of the θ phase ($CuAl_2$), formed by precipitation in the solid state during cooling after extrusion. The grains size of the microstructure have reduced with an average diameter of 13.2 ± 1.9μm, with some variations in the growing direction of the central samples, this is due to the thermal regime in the conformation, where the interior tends to cool more slowly experiencing increased growth of recrystallised grains.

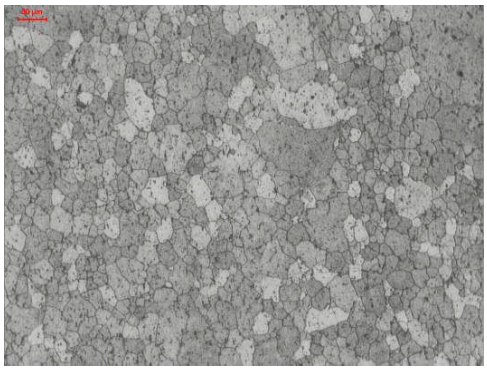

Figure 3: Microstructure of AA2011 alloy used in the manufacture of open-cell metal foam.

It was utilized as space holders particles of NaCl and this was classified and separated into 3 groups of values; fina ($ø_1$) particles with diameter between 1.0 mm and 1.6 mm; average ($ø_2$) - particles with diameter between 1.6 mm and 3.15 mm; course ($ø_3$) - particles with a diameter greater than 3.15 mm. The NaCl space holders pre-forms were produced by different conditions: from free particles of salt or subjected to heating to sintering. The conditions to produce the space holders pre-forms are shown in the table 2.

Table 2: Conditions used for the manufacture of pre-porous forms of NaCl

Conditions	Granulometry	Weight of salt (g)	Pre-load (kN)	T sinterization (ºC)	t sinterization (min)
1	Course	38	-	-	-
2	Average	38	-	-	-
3	Fine	38	-	-	-
4	Fine	45	5	785	210

Initially the extruded bar of the alloy AA2011 was machined and cut to obtain disks of dimensions 43.5mm of diameter by 10mm or 6mm thickness. In a infiltration standard procedure of pre-forms for the production of porous cylinders (Al open metal foam) the disk of 6mm thickness was placed at the base of the matrix and on it was placed space holders pre-form and then on it was positioned the disc with 10mm thickness. In the Figure 4 is presented the assembly scheme of the thixoforming test. After assembly of the test the Al massive discs and the space holders pre-form were heated at 640°C, temperatures between the *solidus/liquidus* lines, to promote the modification of its initial structure (extruded) to globular. After the atop disc reach the temperature at 640°C this was maintained for a time of 4 minutes to ensure the complete globularization of the primary phase of the slurry and the temperature homogenization; it was removed after 4 minutes the atop thermocouple and turn on the press hydraulic machine. It was monitored the applied stress with the use of the load cell and data acquisition system in an acquisition frequency of 4Hz and a total of 300 points. After this step the samples were subjected to successive washing in warm water to remove space holders pre-form. In this work were produced 7 samples for each non-sintered condition and 3 samples for sintered condition.

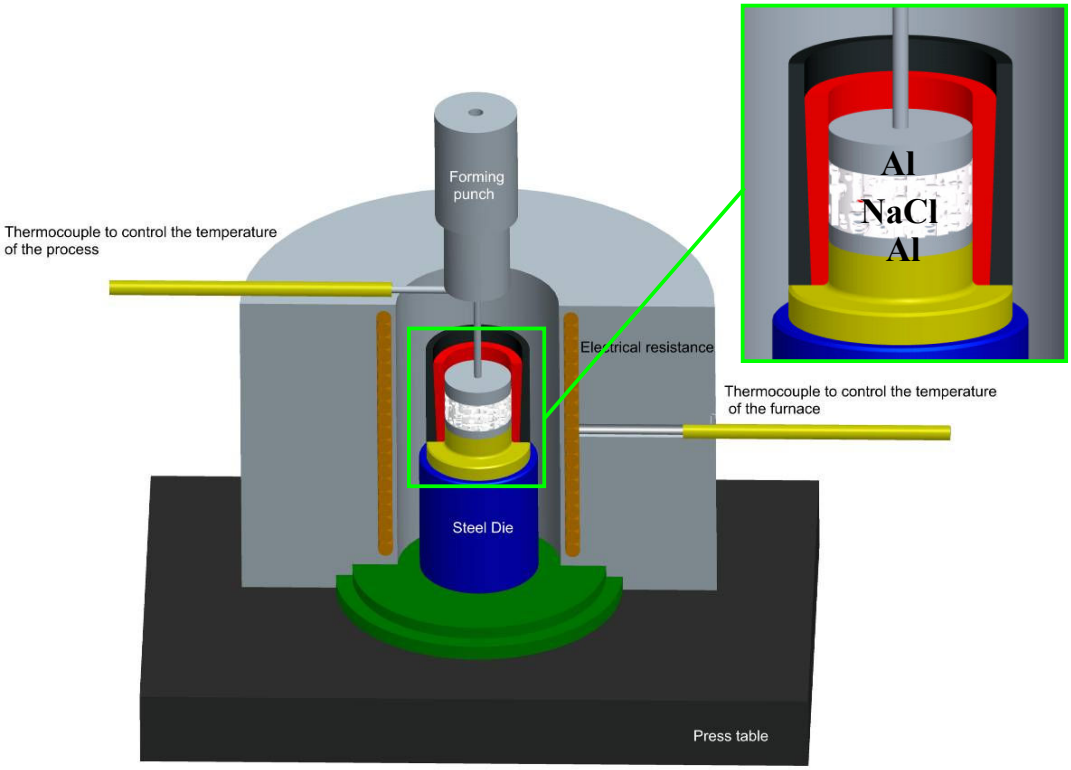

Figure 4: Schematic illustration of the thixoforming process to produce open metal foam.

The characterization of the cellular architecture was performed qualitatively and quantitatively. It was used a helical tomography, ELSCINT manufacturer, model HELICAT FLASH for qualitative analysis. The optical microscopy with dedicate software was used to quantitative analysis.

To calculate the actual and relative densities on all the samples had their characteristic dimensions (diameter and height) measured using digital caliper accurate to 0.01 mm. Then the samples were weighed on a digital scale accurate to 0.001 g.

The mean values of volumes and weights were used to calculate the actual density ρ_r, Equation 1, and the relative density ρ / ρs, according to Equation 2. It was adopted to calculate the relative density the value of 2.82 g/cm^3 as being the density of the AA2011 alloy (BRAY, 1992).

$$\rho_{real} = \frac{m}{V} \tag{1}$$

$$\rho_{rel} = \frac{\rho}{\rho_s} \tag{2}$$

ρ = cellular metal density (g/cm^3)
m = sample mass (g)
v = volume of sample (cm^3)
ρ_s = massive metal density (g/cm^3)

The thermal conductivity λ, normalized thermal conductivity λ/λs, thermal diffusivity are thermal behavior characteristics of which differ in cellular material with the relation massive metal. These thermal characteristics were estimated by Equations 3, 4 and 5.

$$\lambda \approx \lambda_s \left(\frac{\rho}{\rho_s} \right)^q \tag{3}$$

$$\lambda_n = \frac{\lambda}{\lambda_s} \tag{4}$$

$$a = \frac{\lambda}{C_p \rho} \tag{5}$$

λ = thermal conductivity of the cellular metal (W/m.K)
λ_s = thermal conductivity of the massive metal (W/m.K)
λ_n = thermal conductivity normalized
ρ = cellular metal density (g/cm^3)
ρ_s = massive metal density (g/cm^3)
q = correction factor (value adopted 1.7)

3. RESULTS AND DISCUSSION

In the Figure 5 shows examples of open metal foam obtained of the thixoconformation of the AA2011 alloy. These samples were produced by space holders pre-forms of fine, medium and coarse particle size, moreover, these were represented by the letters F, M and G; and sintered pre-forms of fine particles were represented by letter FS. The temperature of tixoforming used was suitable for the production of open-cell metal foam; thus indicating the presence in the thixotropic slurry of a liquid fraction suitable for infiltration, this liquid fraction requires reduced stress (around 3MPa) to the slurry infiltration. According to the general aspects observed, it can be considered that all samples produced by infiltration of pre-forms in different conditions, exhibit acceptable quality and which the process has also acceptable repeatability.

FS F M G

Figure 5: Pictures of sponges produced by tixoforming of metal alloy AA2011 in removable pre-forms in the different conditions: F = fine particles pre-forms; M = average particles pre-forms; G = coarse particles pre-forms, FS = fine particles sintered pre-forms.

It was utilized X-ray tomography for the internal mapping of the all samples. In Figure 6 is shown pictures of sections of open-cell metal foam produced in different conditions of the space holders pre-forms. It can be observed in all cases the interconnection of pores throughout the sample volume which characterizes as open-cell metal foam and the complete removal of the space holders pre-forms.

In general the results show that the quality reproducibility of the cellular material produced is greater in the case of the ease of infiltration the semi-solid metal, this is in the case of using of more course space holders pre-forms. In this case the incidence of infiltration defects is less frequent and is greater homogeneity of the structure obtained.

The possible filling failures possibly occur due to compaction of space holders in the case of not sintered pre-forms or due to the occurrence of a greater contact area between the sintered particles in the case of sintered pre-forms; both events hinder the infiltration of semi-solid sllury in the voids of the particles of the space holders;

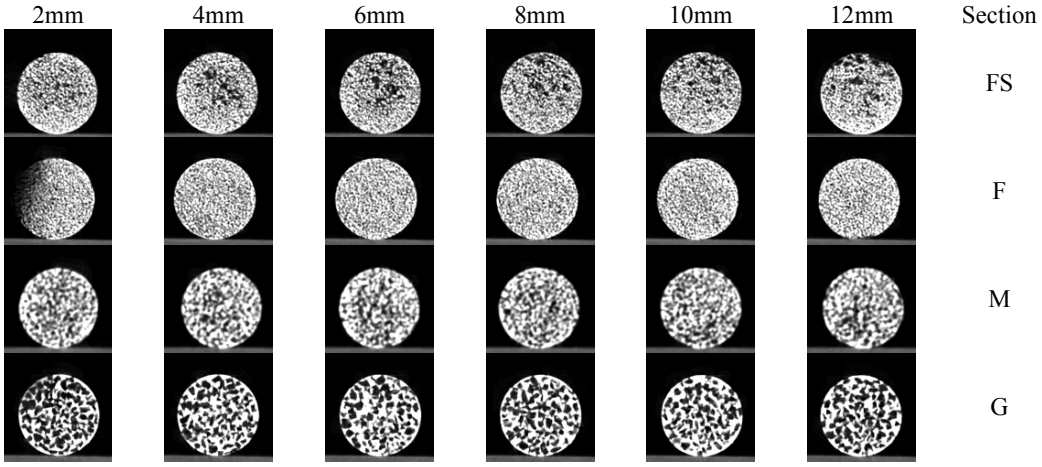

Figure 6: Images of parallel sections obtained by X-ray tomography of open-cell metal foam fabricated by infiltration of alloy AA2011(12 sections of 2mm along the samples); (FS) fine particles sintered pre-forms; (F) fine particles pre-forms; (M) average particles pre-forms; (G) coarse particles pre-forms.

The architecture of the open-cell metal foam produced was quantitatively characterized with respect to the thickness of cell walls. It was utilized for these images of longitudinal central section of the samples which were processed and analyzed by using of images analysis software. With the images processed the following parameters were measured to characterize quantitatively the architecture of samples produced: average thickness of wall cell and diameter of Feret. The images of cellular metals walls in all the conditions are shown in the Figure 7.

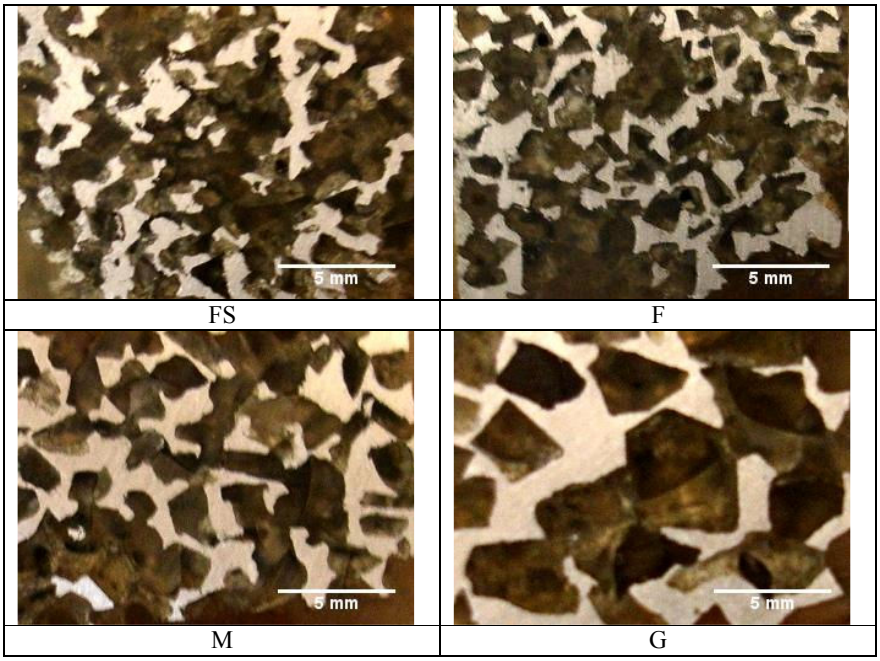

Figure 7: Images of the central longitudinal section of typical cellular metals of the alloy AA2011 produced by infiltration in different conditions of space holders pre-forms.

In the first case traversals parallel lines were drawn across the section (cutting cell walls) whose length was considered the value of wall thickness. Were taken about 25 values for each area evaluated in a total of 2 fields per sample. In the second case the Feret diameter is defined as the diameter of the circle whose area equals the area of the surface measure. Were taken automatically about 30 values through software-editing images in each area observed for a total of 2 fields per sample. Table 3 presents the average values of cell wall thickness and Feret's diameter to open-cell metal foam obtained in all test conditions. It can be seen a trend of increasing cell wall thickness and Feret diameter with increasing the particle size of the space holders used, this is possibly due to the ease of infiltration of the thixotropic slurry in larger particulate of space holders. The analysis of the values obtained also allows to observe that

the variation of the structure is less sensitive to the use of fine particles sintered or non-sintered pre-forms of which the use of pre-forms of particles of different sizes.

Table 3: Average of the cell wall thickness and the Feret's diameter for the samples of open-cell metal foam of the alloy AA2011 produced by thixoforming on pre-forms of the NaCl particles in different conditions.

Type of space holders pre-forms	Cell wall Thickness (mm)	Average Feret's diameter (mm)
Sintered Fine	1,1±0,5	2,6±1,7
Fine	1,2±0,5	2,9±1,5
Average	1,4±0,5	3,3±1,6
Coarse	2,1±0,9	5,4±1,5

The cell material properties depends, besides its architecture which includes distribution and size of voids, thickness and continuity of cell walls and also the specific properties of the walls themselves, these are responsible for the cohesion of the material. For this reason it was analyzed the microstructure of the alloy AA2011 constituent of the walls of the cellular metal. Typical microstructures obtained for the cellular metals produced from pre-forms in different conditions are presented in Figure 8.

The cellular metals microstructure is formed in all cases of α-phase with equiaxiais dendrites or rosettes morphology and phase θ ($CuAl_2$) in the contours, moreover, it can be observed that the rosettes size is about 10x higher (around 160μm) than the average size of grains in the extruded alloy (original condition). This microstructure is not a typical structure of thixotropic slurry and indicates which in the thixoforming operation there is separation of liquid and solid phases and being preferentially infiltrate the liquid fase.

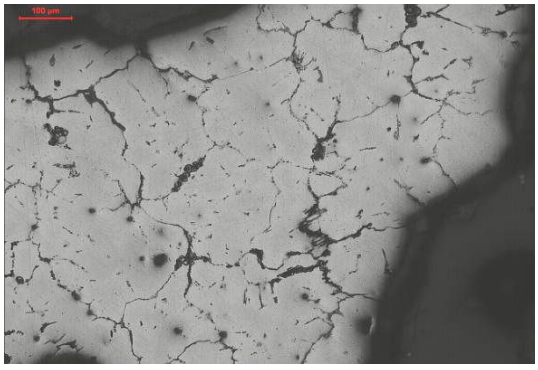

Figure 8: Microstructure of the wall cell of cellular metals manufactured by tixoforming of AA2011 alloy in same condition (temperature and time).

The relative and real densities calculated of the cellular metal are directly related to their physical and mechanical behavior. Table 4 presents the values of real and relative density and porosity for all the samples produced in all the conditions of manufacture.

Table 4: Data table with the values of real and relative density and level of porosity for all conditions.

Type of space holders pre-forms	Density (g/cm^3)	Relative Density	Porosity (%)
FS	**0.71±0.15**	**0.25±0.05**	**75±5**
F	**0.88±0.04**	**0.31±0.01**	**69±1**
M	**0.89±0.09**	**0.32±0.03**	**68±3**
G	**0.94±0.02**	**0.33±0.01**	**66±0,8**

It can be observed in all cases a significant reduction in the density of the material with respect to the massive material: the value of density obtained is around 30% of the massive allow density which means the presence of about 70% of voids in all cases. The average values obtained range from 0.71 to 0.94g/cm^3 for the actual density, 0.25 to 0.33 for the relative density (ρ/ρs) and 66 to 75% for content of voids. For this massive alloy the actual density is of 2.82g/cm^3 (BRAY, 1992). The open-cell metal foams obtained are defined, therefore, in the classification of cellular metals commonly used for porous products containing the content voids of at least 70%. It also can be observed that, in

general, the dimensions of the cellular metals produced in different conditions are little distinguishable from each other with tendency to increase in density with increasing the size of the space holders dimensions.

The cellular metals produced were evaluated for their thermal behavior by means of theoretical estimate proposed by Ashby (2000). Calculations of properties of thermal conductivity, diffusivity and normalized conductivity were performed by using the Equations 3, 4 and 5, and these calculations are shown in table 5. The thermal conductivity (λ) is the physical property that characterizes the ability of a material to transfer heat - for the AA2011 alloy used for the manufacture of the cellular metals - the thermal conductivity is 151W/m.K (BRAY, 1992). Now the normalized thermal conductivity ($\lambda/\lambda s$) is the ratio of cellular metals conductivity and massive alloy conductivity. And the thermal diffusivity (a) is a physical property that indicates how the flow of heat spreads through the material and is obtained by dividing its thermal conductivity by its volumetric specific heat - for the AA2011 alloy the thermal diffusivity is $0.84^{-4}m^2/s$ (KULKARNI, 2005). Estimated theoretical calculations show that the thermal conductivity of the cellular metals is significantly lower (10x) to the massive alloy, while the thermal diffusivity is significantly higher (4x). The values for these properties tend to increase with increasing of space holders particle that causes the increase of metal walls thickness and the size of voids.

Table 5: Estimated values of thermal conductivity (λ), normalized thermal conductivity ($\lambda / \lambda s$) and thermal diffusivity (a) of the cellular metals of alloy AA2011 produced from of space holders pre-forms in different conditions.

Type of space holders pre-forms	**Relative density (ρ/ρ_s)**	**λ (W/m-K)**		**λ/λ_s**		***a* (m²/s)**	
		Min. (q=1.8)	**Máx. (q= 1.6)**	**Min.**	**Max.**	**Min.**	**Max.**
FS	0.23	13.0	15.8	0.09	0.10	$2.9.10^{-4}$	$3.5.10^{-4}$
Standard deviation	***0.03***	***4.8***	***5.4***	***0.03***	***0.04***	***$1.1.10^{-4}$***	***$1.2.10^{-4}$***
F	0.31	18.5	22.0	0.12	0.15	$4.1.10^{-4}$	$4.9.10^{-4}$
Standard deviation	***0.01***	***1.4***	***1.5***	***0.01***	***0.01***	***$0.3.10^{-4}$***	***$0.4.10^{-4}$***
M	0.32	19.2	22.8	0.13	0.15	$4.2.10^{-4}$	$5.0.10^{-4}$
Standard deviation	***0.03***	***3.5***	***3.9***	***0.02***	***0.03***	***$0.8.10^{-4}$***	***$0.8.10^{-4}$***
G	0.33	20.9	24.7	0.14	0.16	$4.7.10^{-4}$	$5.5.10^{-4}$
Standard deviation	***0.01***	***0.8***	***0.9***	***0.01***	***0.01***	***$0.2.10^{-4}$***	***$0.2.10^{-4}$***

4. CONCLUSIONS

The thixoconformation process is efficient for the production of open-cell metal foam of the alloy AA2011 because allows the filling of voids between NaCl particles of space holders pre-forms of different sizes in the sintered or non-sintered conditions.

In general, all the cellular metals obtained in different conditions of pre-forms have acceptable quality. The quality consistency obtained depends of the processing condition: the use of pre-forms of space holder particles of average and coarse sizes provide more consistent quality, that is, in these cases there is better repeatability of the process.

The cellular metals present , in all cases, the cells with geometry and dimensions compatible with the geometry and particle size of NaCl used to make pre-form.

Increasing the particle size of the space holders leads to increased cell wall thickness, density, thermal diffusivity and of thermal conductivity.

Thus the product of the thixoconformation may have great thermal applications with products that require low thermal conductivity and high diffusivity, such as heat exchangers.

6. ACKNOWLEDGMENTS

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