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Experimental analysis of the effect of control factors on aluminium foam produced by powder metallurgy

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Abstract. Aluminium foams, produced by the powder metallurgy route, have a high potential for use in weight-sensitive construction parts. The stiffness to weight ratio is the main criterion for material selection in light-weight design. The aim of this study is to evaluate the properties and to optimize the control factors of a powder technique, developed at the Fraunhofer Institute, starting from aluminium powders with titanium hydride as the foaming agent. During the experimental work, many samples were made following the principles of the design of experiment. Then a study of the morphology and distribution of cells and of the walls of the cells was carried out and relative density was measured. Mechanical properties of foam samples were studied by quasi-static compression tests. The final evaluation of the most important features has been made using ANOVA statistical analysis.

Key words: aluminium foam, powder metallurgy, foaming agent, design of experiments.

1. INTRODUCTION

Due to the strong demand of the transport industry for lower operating costs, higher payloads, improved environmental compatibility as well as increased passenger safety and comfort, aluminium foams have become more and more important during the last few years. They have been identified as a new class of materials of great interest due to their unique combination of properties, caused by their cellular structure and metallic behaviour. In particular, the automotive

industry sees a great potential in the use of aluminium foams in light-weight constructions, for crash energy absorption and noise control. Applications are also to be expected in other areas as in aerospace, ship, railway, biomedical and building industry, and also in filters, heat exchangers, silencers, flame arresters and for water purification [1]. Different manufacturing methods can be distinguished according to the state of the starting metal: liquid, powdered or ionized.

This study is focused on a powder technique with a foaming agent. This process, developed at the Fraunhofer Institute for Manufacturing and Advanced Materials, belongs to the processes starting from metal powders as raw material with an addition of the titanium hydride as the foaming agent [2]. The reference technique was first applied in 1963 [3]. This process permits the production of near net-shaped parts with complex geometries as well as 3D-shaped sandwich structures with a foamed core layer [4]. Nowadays a huge number of studies on the process are available [1], but the knowledge on correlations between the final properties of the foamed parts and the process parameters is still not complete.

The aim of this study is to evaluate the properties and to optimize the process parameters of this powder technique by means of a statistical approach. During the experimental work many samples have been made following the principles of the design of experiments (DOE) and the final evaluation of the most important features (relative density and compression strength) was carried out by ANOVA statistical tool.

2. EXPERIMENTAL

The process is based on mixing aluminium (Al), titanium hydride (TiH_2) and silicon carbide (SiC) powders to obtain an homogeneous mixture, followed by compression into a preform (foamable precursor material), and foaming of the preformed sample in a furnace.

2.1. Powders

In the present work, selection of the aluminium powders was a very difficult task. In fact, during the initial screening, four types of Al powders from different suppliers were tested as shown in Table 1.

Table 1. Specification of the powders

Powder	Manufacturer	Purity	Size
Al	Baker	purified	44 μm
Al	Riedel-de-Haen	93%	fine
Al	Carlo Erba	95%	n. spec.
Al	Pometon	99.5%	45–150 μm
TiH_2	Riedel-de-Haen	97.5%	40 μm
SiC	Aldrich	n. spec.	37 μm

The problems were associated with the size of the powders: too small particles are not suitable to obtain foam. Accordingly, Al powders produced by Pometon were suitable. The content of the blowing agent (TiH_2) was in the range of 0.5–2 wt%. This wide range (different from [5]) was chosen due to the mentioned problems with fine Al powder (causing hydrogen loss during precursors heating).

Moreover, in order to improve the stability of metal foams, usually ceramic particles were added. Literature data report that ceramic particles inhibit melt flow, after increasing the viscosity, thus decreasing the rate of cell coarsening and drainage of liquid through the structures [6]. The tested amount of SiC was in the range of 0–10 wt%.

2.2. Mixing

This was the second process step carried out through mixing of the metal powders with the blowing agent in glass mixer to ensure the homogenization of components.

2.3. Compaction

Powder compaction may be performed using different techniques (hot pressing, cold isostatic pressing, extrusion) [4]. In our experiments, powder compaction was performed adding about 15 g of the mixture (depending on the composition) in a 32 mm diameter steel die, followed by cold compaction by uniaxial pressing. This is a fair simplification of the process in comparison to others methods. Compaction in the range of 100–700 MPa was obtained with a MATEST E157 hydraulic motorized press.

2.4. Foaming

Foaming was carried out in a pre-heated electric furnace at different temperatures in the range of 620–950 °C. Foaming operation took place in normal atmosphere and the mould was opened only at the top. In the furnace, the mould was placed on a ceramic plate, excluding temperature gradient in vertical direction as suggested in [7]. Careful control of heating conditions during foaming is essential to obtain quality foams. The difficulty is that the liquid foam is thermodynamically unstable and conditions change constantly during foaming [8]. Due to that, when the mould is filled with foam, it must be suddenly cooled down below its melting point to stabilize the structure. Air quenching leads to foam collapse, therefore water was preferred for cooling. Moreover, in air-cooled samples the foam structure shows pronounced cracking of cell walls that is not the case by water quenching [9]. In the present work, foaming was stopped at a moment, which depended mainly on the furnace temperature.

3. RESULTS AND DISCUSSION

Variables, influencing the foaming process, are the TiH_2 and SiC content, mixing time, compaction tool, pressure, sintering temperature and time, mould features and the cooling procedure. Each variable influences the output foam quality. In the initial screening, a large number of samples was tested by changing parameters in wide ranges. As a result it was found that although many control factors influence the process, the output quality depends mainly on three primary control factors: SiC fraction, compaction pressure and sintering temperature. Influence of these reference factors on the cells area, cell wall thickness, linear expansion, relative density and plateau stress was investigated. A standard approach of DOE is to use the full factorial method that requires a total of 27 experimental runs if there are 3 factors to be investigated and each consists of 3 different levels (high, medium and low). Table 2 shows the levels of factors in this systematic investigation. For each combination of the parameters, three replications have been carried out. The quantity of Al was fixed at 15 g and TiH_2 at 1 wt%.

3.1. Characterization of cell morphology

After screening, different samples were made in the second experimental step, following the principles of DOE to study mechanical properties and the evolution of the foam in time. The last measurement, called *ex situ*, was carried out interrupting the foaming by quenching. By varying the time span from start to quenching, a series of samples was obtained reflecting various stages of foam evolution (Fig. 1). Samples were cut with a diamond blade to minimize cell damage.

Table 2. Experimental factors and levels

Factors	Level		
SiC content, wt%	0	2.5	5
Pressure, MPa	310	370	430
Temperature, °C	750	800	850

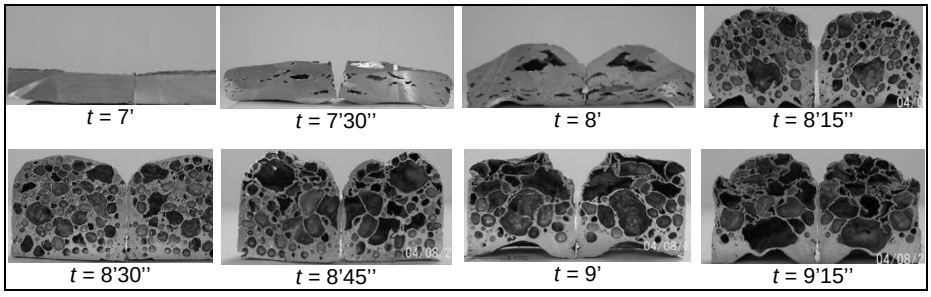

Fig. 1. Foam structure evolution (SiC 2.5%, p = 370 MPa, T = 800 °C).

The linear expansion (LE) was then calculated as [10]

$$LE = \frac{h_f - h_0}{h_0}, \tag{1}$$

where h_f and h_0 are the specimen height after and before foaming.

Figure 2 shows LE as a function of the furnace holding time; foaming started after about 7 min and was completed after 8 min and 30 s (in this way the best structure was obtained). Beyond this time the foam collapses for two reason: drainage (i.e. the flow of the molten metal, driven by gravity) and coalescence (i.e. merging to form a larger cells).

Figure 3 shows the dependence of LE on the temperature and pressures. Each column represents the mean value of 3 samples. As expected, LE is in the range of 200–450% and it increases with pressure (foaming agent is well trapped) and decreases with temperature (too extensive drainage).

The samples showed in Fig. 1 were prepared with the aim to investigate morphology of the foam: the cell area, cell wall thickness and bubble sphericity. This investigation was carried out by measuring the "equivalent" diameter of the cells (i.e. the diameter of the circle with the same area of the bubble) and by assigning a value to the "circularity parameter" C expressed by [11]:

$$C = 4\pi A / P^2. \tag{2}$$

Here A and P are the area and the perimeter of bubbles, respectively. Evidently $C = 1$ for circular cells. Small bubbles with uniform size and spherical shape guarantee better mechanical properties and the implication of shape is very important in determining the yielding susceptibility of the cells [12]. Moreover, deformation behaviour is greatly affected by cell wall thickness [13]. Morphological parameters were measured in 2D cross section of the sample by using a software tool. Figures 4 and 5 show the cell area distribution for sample obtained

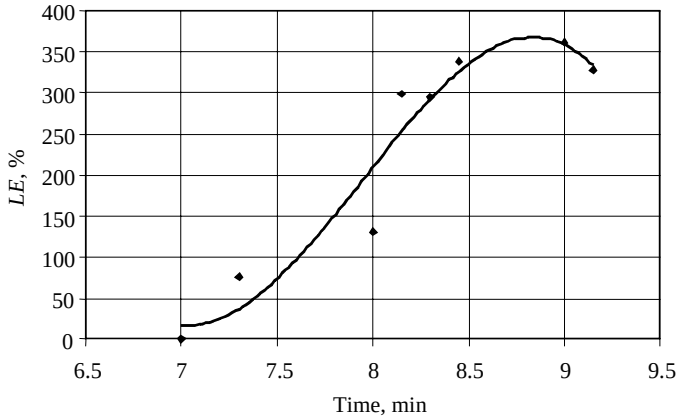

Fig. 2. Linear expansion as a function of the furnace holding time.

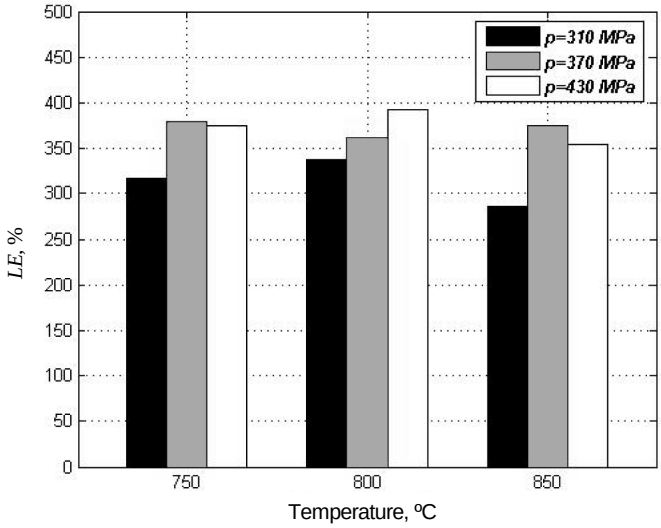

Fig. 3. *LE* vs temperature and pressure.

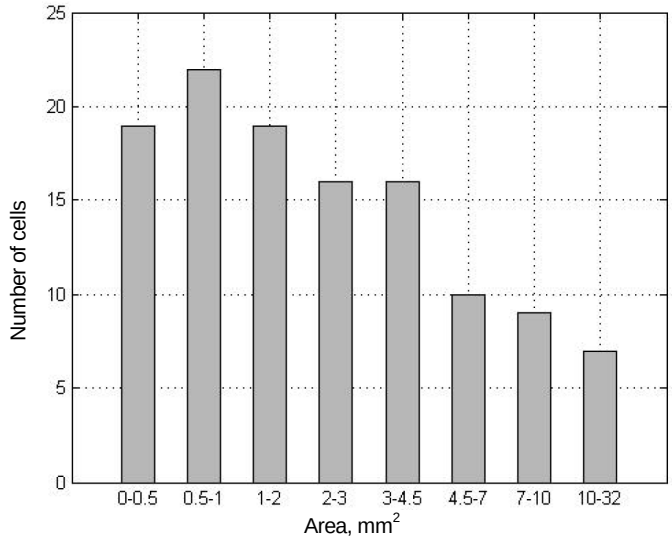

Fig. 4. Cell area distribution (SiC 2.5 wt%, p = 370 MPa, T = 800 °C).

at $t = 8'30''$. As can be seen, the distribution of the size of the pores is very wide. The values of the areas, determined for about 120 cells, are in the range of 0.22–32 mm^2 with a mean value of 3.77 mm^2; the wall thickness range is 0.10–1.82 mm (mean value 0.63 mm). Equivalent diameter range is 0.53–6.4 mm and the mean value 1.85 mm is slightly larger than given in [14]. The circularity parameter was in the range of 0.33–0.91 with mean value 0.62. This result implies that there are many cells of irregular shape and contour.

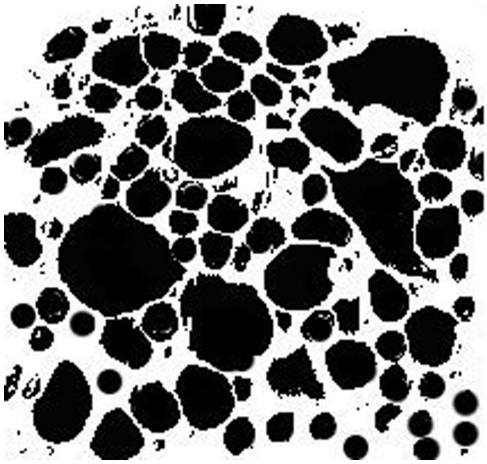

Fig. 5. Treated image (SiC 2.5 wt%, p = 370 MPa, T = 800 °C).

3.2. Mechanical tests

After checking the foam morphology, mechanical tests were carried out. Relative density and compressive strength were selected as the two main parameters for evaluation. Relative density of metal foams, defined as the ratio of the foam density to the base material density [14], is an important parameter describing the material and it can be controlled by adjusting the process parameters. Moreover, relevant properties, in particular the compressive strength of metal foams can be approximated on the basis of the relative density of the foam.

The relative density has been evaluated taking in account the content of 2.5 and 5 wt% of SiC (density of 3.1 g/cm^3). The measured relative density range was 0.23–0.31.

Compressive strength is the second evaluation criterion because it is relevant in the context of energy absorption of the foam. Figure 6 shows typical compression test samples (diameter 30 mm) prepared by cutting.

In fact, after foaming all surfaces of the samples were covered by a film that was partially removed. The tests were carried out using an INSTRON machine (model 5869) in quasi-static conditions, following the standard test method for compressive properties of metal foams [15], prepared by Cimat Company (taking into account ASTM and DIN standards). Stress was evaluated as the load per total area of the specimen, including porosity, as suggested in [16]. Likewise, strain, defined as a nominal value for the foam structure, is not the real strain experienced by the cell walls. Compressive deformation curves for different SiC contents are shown in Figs. 7 and 8. It is seen that the stress values and plateau stress of the foam, containing SiC, are higher than those of pure Al foam at the same density. The difference in stress values decreases for large values of strains. Literature reports different methods for the determination of the compression strength [17]. In our case the strength value at 40% of deformation was selected

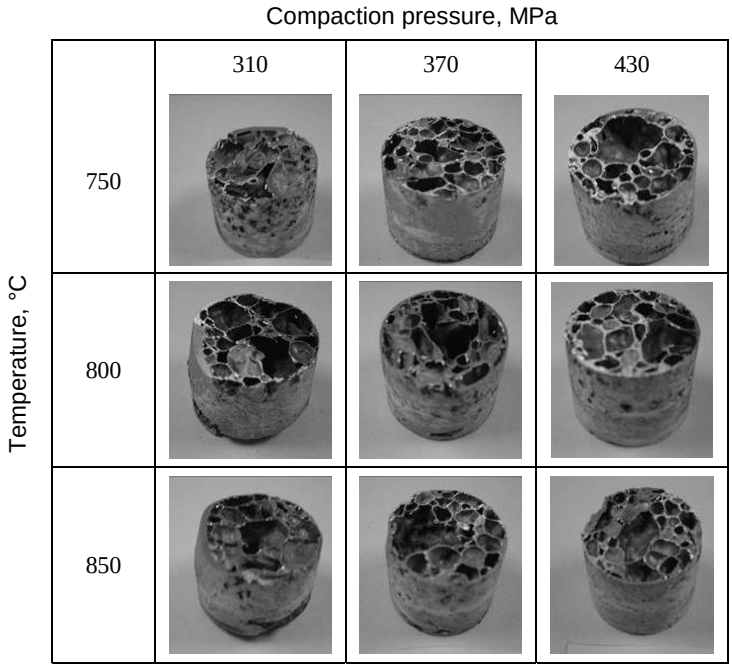

Fig. 6. Samples (2.5 wt% SiC) machined for mechanical tests.

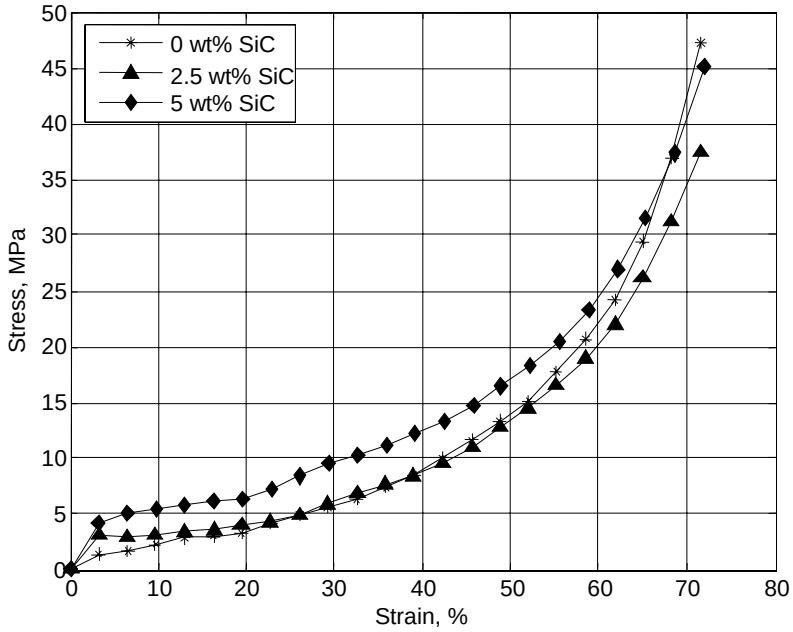

Fig. 7. Stress-strain diagram (samples made at p = 370 MPa, T = 800 °C, relative density 0.3).

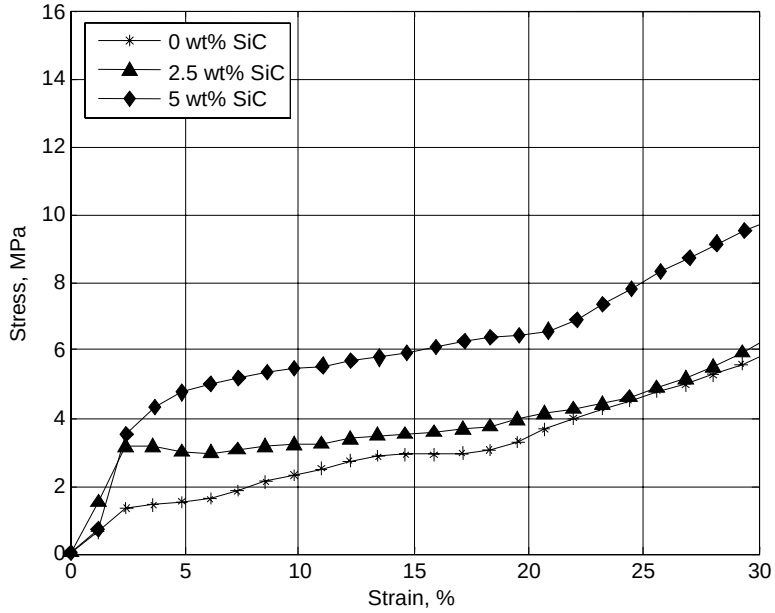

Fig. 8. Enlarged stress-strain diagram of Fig. 6.

for all curves and, as expected, the observed values are in the range from 6.1 to 14 MPa. Only two samples (carried out by 310 MPa, 750 °C, 5% SiC and 310 MPa, 850 °C, 5% SiC) showed different features for stress and density (Fig. 9).

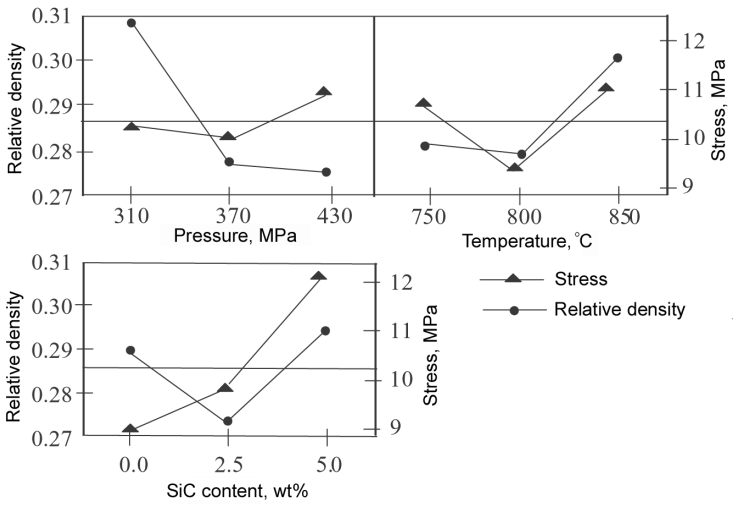

Fig. 9. Main effect plot for relative density and stress.

ANOVA method (ANalysis Of VAriance) was used to corrrelate the parameters on relative density and compressive stress. As a result it was found that the only parameter, affecting the relative density, was pressure (95% confidence interval). Only SiC content had a little influence. The effects of individual factors were plotted using MINITAB software (Fig. 9). The optimal parameters can be easily recognized. Realizing that low relative density and high compressive strength is the best compromise, best set-up parameters are the following: pressure 430 MPa, temperature 750 °C and SiC about 3 wt%. The last result agrees with another study on this subject [18]. The sample with these parameters shows also high LE value (430%).

Correlation between the density (ρ) and compressive strength (σ) of foams has been in [19] described as

$$\frac{\sigma}{\sigma_{\mathrm{Al}}} = N\left(\frac{\rho}{\rho_{\mathrm{Al}}}\right)^{n}, \tag{3}$$

where N and n are constants. The authors found an average value for n exceeding 1.42 instead of 1.5, conventionally assumed in the literature (Fig. 10). Differences may be caused by heterogeneities, related to uneven density distribution and imperfections in the microstructure.

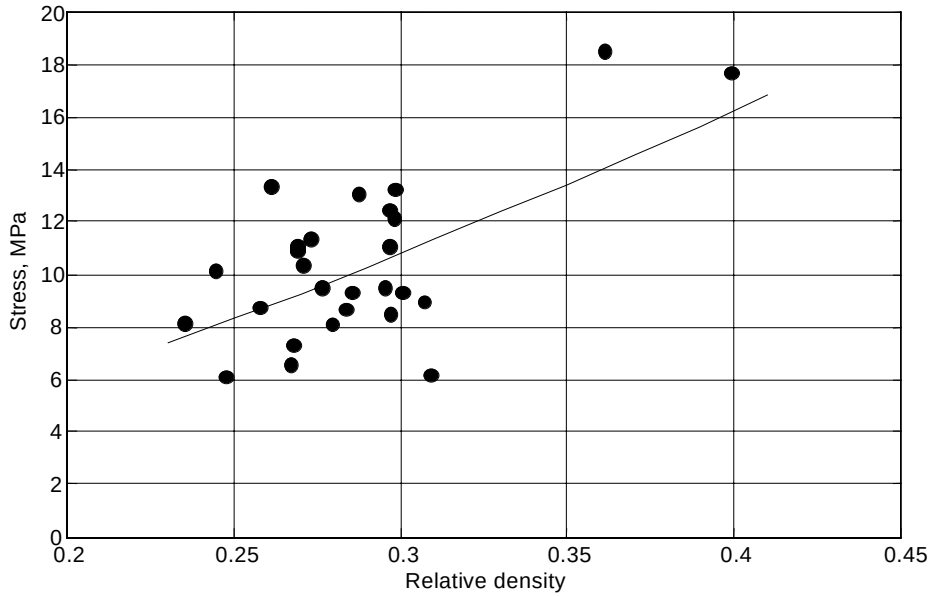

Fig. 10. Stress-density law.

4. CONCLUSIONS

A statistical approach was applied to investigate the influence of the main control factors on the linear expansion, relative density and compressive strength of an aluminium foam. Using DOE and ANOVA analysis technique we were able to identify the most significant factors that influence the foams: compaction pressure, temperature and SiC content. The optimal manufacturing parameters were obtained as follows: $p = 430$ MPa, $t = 750\,°C$ and SiC = 3 wt%. Experiments showed that it is possible to manufacture Al foams also in the absence of SiC particles even if the samples show low quality in terms of linear expansion. Moreover, the selection of raw materials is very important (particularly the size of Al particles to prevent hydrogen losses). In our experiments simple uniaxial cold pressure was used in foam preparation and this is a fair simplification of the process in comparison to other methods. It is likely that data scatter by experiments is caused by the heterogeneities in the regions with variable density distribution.

Many other design and manufacturing features might influence the production of foams. This will be the subject of future investigations.

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Kontrollparameetrite mõju eksperimentaalne uurimine pulbermetallurgiameetodil valmistatud vahtalumiiniumile

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Pulbermetallurgia teel valmistatud vahtalumiiniumil on hea kasutuspotentsiaal eelkõige kaalutundlikes konstruktsioonides, kus jäikuse ja massi suhe on materjali valiku põhiline kriteerium. Artikli eesmärgiks on hinnata ja optimeerida Fraunhoferi instituudis loodud alumiiniumipulbrist ja vahumoodustajast – titaanhüdriidist – vahtalumiiniumi pulbertehnoloogia kontrollparameetreid. Eksperimentide kavandamisel on kasutatud katsete planeerimise metoodikat. On uuritud kärgstruktuuri morfoloogiat, kärgede jaotust ning nende seinapaksust; kriteeriumiks on vahtalumiiniumi tihedus ja survetugevus. Põhiliste iseärasuste hindamisel on kasutatud ANOVA statistilist analüüsi.