

# Sintered foams from Ni-based superalloy metallic powders

I. VIDA-SIMITI\*, N. JUMATE, E. BRUJ, G. THALMAIER, O. NASCA

*Department of Materials Science and Technology, Technical University of Cluj-Napoca, 103-105 Muncii Ave., 400641 Cluj-Napoca, Romania*

Hollow spherical, 125 to 500  $\mu\text{m}$  particles of NiCrSiB powders obtained through argon gas atomization were used. The metallic foam structure was obtained by sintering the hollow particles in vacuum,  $10^{-5}$  Torr at 950°C. To shape the macro porous open-cell network inside the structure, space holders were used. Thus, bi-porous metallic foams were obtained with closed-cells due to hollow particles and open-cells formed by the space holders. Optical and electronic microscopy was performed to illustrate the microstructure of the bi-porous metallic foams. Thermal conductivity, thermal expansion and sound damping capabilities of the samples were investigated.

(Received July 30, 2010; accepted August 12, 2010)

*Keywords:* Metal foams, Bi-porous structures, Hollow particles, Porous materials

## 1. Introduction

Cellular solids are materials with high porosity which are divided into distinct cells [1].

Porous metals, with porosity as high as 90%, have been drawing much attention for their unique properties. The metallic foams can also be used as a damping material or as sound absorber. The large specific area of porous metals is another important characteristic; they can be used as heat exchangers if pore connectivity is ensured [2].

Sintered hollow sphere foams have a certain volumetric fraction of enclosed pore space inside the spheres, but also have interstitial porosity between the spheres appearing as a mixed open/closed cellular solid [3].

Heat transfer in the foam, as in cellular solids in general, may be considered as a composition of single pore size level mechanisms such as thermal radiation from the solid elements to each other, thermal conduction within the solid elements, and thermal conduction in the air and solid-to-fluid convective heat transfer [4].

Sound absorption together with mechanical stiffness and strength at low weight makes an attractive combination. Automobile floors and bulkheads are examples of structures, their primary function is to carry loads, but if this is combined with sound absorption the product quality is enhanced [5].

Metallic foams have been considered in various acoustic applications. On one hand, the unique structure of these materials can provide good sound absorption. On the other hand, their acoustic properties can be combined with other characteristics of the metallic foams (i.e. thermal and chemical stability, mechanical properties) to make them more attractive than common sound absorbers such as mineral wool or polymer foams [6].

Compared with glass wool and polymer foams, the advantage of using metal foams as a sound absorber is

obvious: self-supporting due to its stiffness and strength, fire retardance, low moisture absorption, and superior impact energy absorption capabilities [7].

These materials can be considered biphasic: porous phase and solid phase. The solid phase represents the structure of the sample and can be a polymer, metal or ceramic that forms the solid matrix and provides the load bearing capability to different external loadings. The porous phase consists of closed pores and open pore network surrounding the solid phase matrix. For the cellular solids, foams respectively, the porous phase does not represent defects, but has the functional role of: weight reduction, sound and vibration damping, etc. [8]

This paper aims at studying the possibilities of achieving bi-porous structures (open and closed porosities), with low densities and very good acoustic and thermal properties by powder metallurgy route.

## 2. Materials and experimental method

Ni-based superalloy hollow spherical powders were used, with the following chemical composition 70.19% Ni, 7.08% Cr, 12.34% Fe, 6.73% Si, and 3.66% B, (NiCrSiB).

Four granulometric fractions were used: +125-200  $\mu\text{m}$ , +200-315  $\mu\text{m}$ , +315-400  $\mu\text{m}$  and +400-500  $\mu\text{m}$ .

To form the metallic foam structure's macro porosity, polystyrene particles of 2 to 3 mm in diameter were used. The melting point of the polystyrene particles is at 240 °C, while the thermal decomposition temperature is 300 °C.

NiCrSiB particles and the polystyrene particles were mixed together and homogenized for 10 minutes together with an organic binder. The organic binder used was polyvinyl alcohol (PVA), a hydrophilic synthetic polymer soluble in water, with a melting point at 180-190 °C, and a decomposition temperature of 200 °C.

The resulted mixture was poured and gently pressed into alumina dies. The samples were then sintered in vacuum,  $10^{-5}$  Torr for 30 minutes.

The sintering temperatures were 900, 950 and 1000°C.

For the complete decomposition of the polyvinyl alcohol and the polystyrene particles, two plateaus, each of ten minutes during the sintering cycle at 210°C and 370°C, respectively, were necessary, as presented in Fig. 1.

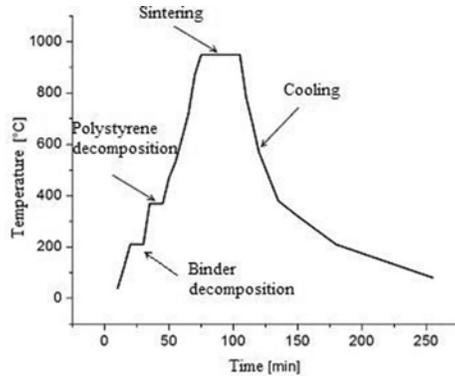


Fig. 1. The optimized sintering cycle.

At 900°C the sintering necks between the NiCrSiB particles were not completely formed to give the bi-porous metallic foams good stiffness and high strength, while at 1000°C the samples were over sintered. The optimum sintering temperature was found to be at 950°C.

The samples obtained were characterized by means of optical, scanning electron microscopy. Thermal conductivity, linear expansion and sound damping capability were also determined.

The thermal conductivity was measured using the stationary temperature field method on a thermal conductivity experimental setup (Fig. 2).

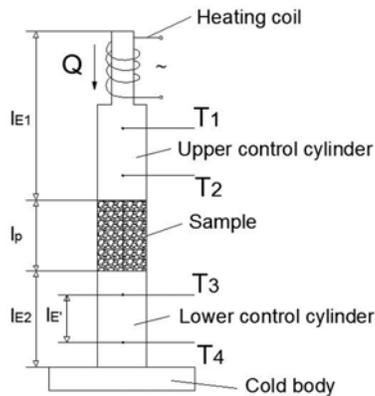


Fig. 2. Thermal conductivity experimental setup.

The experimental setup consisted of: The upper control cylinder with the length  $l_{E1} = 50$  mm and thermal conductivity  $\lambda_E$ , made of copper, with an AC powered coil wrapped around it, which generates a controlled quantity

of heat  $Q$ . The lower control cylinder also made of copper and  $l_{E2} = 50$  mm in height, is connected to a cold body to ensure a good heat transfer throughout the sample ( $L_P$  - length of sample). The sample is fixed between the upper and the lower control cylinders. The temperature throughout the upper control cylinder is measured with two thermocouples ( $T_1$  and  $T_2$ ) in two points of the cylinder at a distance of  $l_E' = 45$  mm between them. The temperature on the lower control cylinder is measured in the same way as in the upper control cylinder but with the  $T_3$  and  $T_4$  thermocouples. In order to minimize the heat losses, the experimental setup was insulated from the environment. The thermal conductivity was calculated using the following relation:

$$\lambda = \frac{l}{2} \lambda_E \frac{l_p}{l_E} \frac{(T_1 + T_3) - (T_2 + T_4)}{(T_2 - T_3)}, [\text{W/mK}] \quad (1)$$

Thermocouples  $T_2$  and  $T_3$  are situated at a small distance from the sample and for the results to be accurate corrections need to be applied to the measured temperatures.

The linear expansion coefficient was determined using an Ulbricht-Weiss dilatometer on samples with the length  $l_0 = 40$  mm, in the temperature range of 20 – 400°C. The calculation formula is:

$$\alpha = \frac{l}{l_0} \frac{\Delta l}{\Delta T} \quad (2)$$

where:  $l_0$  is the initial length of the sample;

$\Delta l$  – sample expansion;

$\Delta T$  – temperature range.

Acoustic attenuation properties of bi-porous metal foams were determined in the frequency range of 100-9000 Hz. Measurements were made by transmission method on an acoustic setup.

### 3. Experimental results and discussions

**3.1. Densities and porosity** of the obtained samples are given in Table 1. The porosity  $P$  from Table 1 (porous phase) is formed of:  $P = P_1 + P_2 + P_3$ , where:

- $P_1$  is the porosity formed by micro pores (between the sintered powder particles);
- $P_2$  is the porosity formed by macro pores (formed by the polystyrene particles);
- $P_3$  is the closed porosity of the hollow particles.

Table 1. Density and porosity of the obtained samples as function of particle size range.

Particle size range [ $\mu\text{m}$ ]	$\rho$ [ $\text{g}/\text{cm}^3$ ]	P [%]
+125-200	3.18	65
+200-315	2.63	71
+315-400	2.22	76
+400-500	1.71	81

### 3.2. Microscopic analysis

In the images presented in Fig. 3, the structures of the obtained samples can be seen. In Fig. 3.a, one can see an overview of the sample with macro pores of 2 - 3 mm into the body of the sintered structure. Fig. 3.a represents a detailed optical image, in section, where the sintered structure of hollow particles and a macro pore formed by removing the porous agent can be seen. In Fig. 3.b and 3.c, SEM images details of cross-section are presented. One can see the micro pores between the sintered particles and the internal pores from the hollow particles. In Fig. 3.d, a SEM image is presented, representing two sintered hollow particles from the bi-porous structure of the metal foam and the sintering neck formed between them.

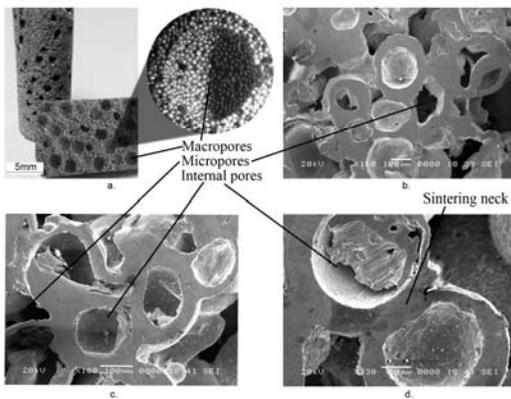


Fig. 3. Images of the bi-porous metal foam structures.

In Fig. 3.d one can also see that the thickness of the hollow particle's walls is between 10 and 50  $\mu\text{m}$  for the particles with mean diameter of 400  $\mu\text{m}$ .

### 3.3. Thermal properties

The dependence between thermal conductivity, temperature and porosity is shown in Fig. 4, 5 and Table 1.

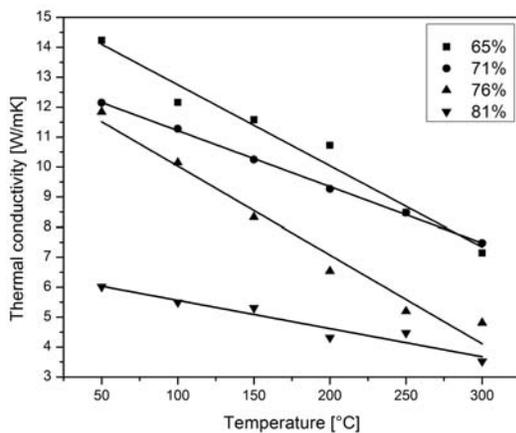


Fig. 4. The dependence between thermal conductivity and temperature for samples having different porosities.

The type of heat transmission is primarily thermal conduction at the interparticle necks level, macro pores, pores between the powder particles and closed pores of the hollow particles. The thermal conductivity,  $\lambda$ , decreases with temperature due to the way of heat transmission through the solid metal phase. With the temperature increase, the thermal agitation of atoms in crystalline networks of material also increases and thus the heat flux through the sample decreases (Figs. 4, 5). The porous phase growth caused by increasing the powder particle size makes the thermal conductivity decrease. Therefore, the thermal conductivity is much lower in porous phase than in solid phase.

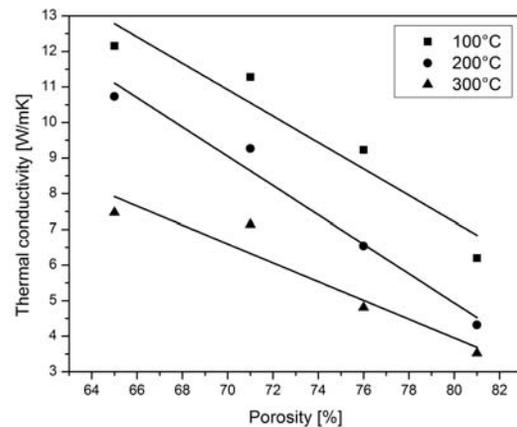


Fig. 5. Thermal conductivity as function of porosity and temperature.

The thermal expansion coefficient,  $\alpha$ , calculated for the samples for each powder fraction is given in Table 2.

Table 2. Thermal expansion coefficient function of porosity for the sintered samples.

Particle size range [ $\mu\text{m}$ ]	P [%]	$\alpha \cdot 10^{-6}$ [ $\text{K}^{-1}$ ]
+125-200	65	18.74
+200-315	71	18.25
+315-400	76	18.21
+400-500	81	18.19

From the data presented in Table 2, it can be seen that the linear expansion coefficient is constant and does not depend on the sample's porous structure, or on the porosity [10]. Thermal expansion and the macroscopic changes in the linear dimensions of samples are produced by increasing the average distance between atoms at increasing temperature. Linear expansion coefficient is a

material constant and depends on the nature of atomic species and not on the porous structure.

### 3.4. Acoustic properties

Sound energy loss, or sound absorption in porous materials is caused mainly by air friction during its oscillation in the material pores. For effective sound absorption the material must have a porous structure while the pores must be open to the outside and communicate among them so as not to obstruct sound wave penetration inside the material. The degree of sound attenuation in the sample was determined using the following formula:

$$A = 20 \log \left( \frac{p_1}{p_2} \right), [\text{dB}] \quad (3)$$

where:  $p_1$  is the incident sound pressure wave;  
 $p_2$  is the sound pressure wave through the sample.

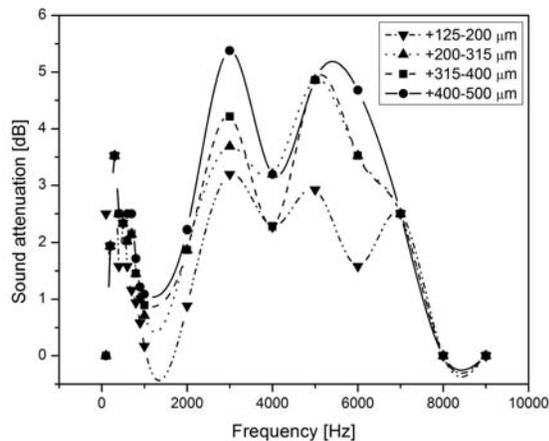


Fig. 6. Sound attenuation as a function of the frequency.

One can see from Fig. 6 that the sound attenuation is a function of frequency and porosity, and also depends on the powder particles size. Sound attenuation capability was found to be better for higher porosity samples. Maximum attenuation of 5.4 dB was provided by the sample with porosity of 81% at the frequency of 3000 Hz.

### 4. Conclusions

Bi-porous metal foam structures with low densities between 3.18 and 1.71 [g/cm<sup>3</sup>], and high porosities between 65 and 81 [%] were obtained.

A linear dependence of thermal conductivity with the measured temperature was observed. The type of heat transmission was primarily conduction at the interparticle necks level. The macro pores created by the polystyrene particles throughout the sample act as thermal insulators.

The linear expansion coefficient in the temperature range of 25 - 400°C did not depend on particle size and the macro pores from the sample.

Sound attenuation was found to be a function of frequency and porosity, and also dependent on the powder particles size.

The possible applications for the bi-porous metallic foams obtained could be in the fields of: sound and heat insulation, sound absorption, mechanical damping, catalytic support, heat exchangers.

### Acknowledgements

This work was supported by CNCIS – UEFISCSU, project number PNII – IDEI 749/2009.

### References

- [1] C. Veyhl, R. Winkler, M. Merkel, A. Ochsner, Defect and Diffusion Forum **280-281**, 85 (2008).
- [2] M. Hakamada, Y. Yamada, T. Kuromura, Y. Asao, Y. Chen, H. Kusuda, M. Mabuchi, Advanced Materials Research **15-17**, 416 (2007).
- [3] T. X. Yu, D. Karagiozova, Z. Y. Gao, Key Eng. Mater. **340-341**, 389 (2007).
- [4] Th. Fend, O. Reutter, J. Sauerhering, K. S. do Couto Aktay, R. Pitz-Paal, S. Angel, ECTP, Bratislava, September 4-8, (2005).
- [5] T. J. Lu, A. Hess, M. F. Ashby, J. Appl. Phys. **85**, 11 (1999).
- [6] L. P. Lefebvre, J. Banhart, D. C. Dunand, Adv. Eng. Mater. **10**(9), 775 (2008).
- [7] L. Gibson, M.F. Ashby „Cellular Solids”, 2nd ed., Cambridge, UK, (1997).
- [8] I. Vida-Simiti, J. Optoelectron. Adv. Mater. **8**(4), 1479 (2006).
- [9] G. Matei, E. Bicsak, N. Jumate, Proc. Int. Conf. RoPM’96, Ed. UTPress, Cluj-Napoca, Romania, 195-198 (1996).
- [10] M. F. Ashby, A. G. Evans, N. A. Fleck, L. J. Gibson, J. W. Hutchinson, H. N. G. Wadley, Metal Foams: A Design Guide, Butterworth-Heinemann, USA, (2000).

\*Corresponding author: Vida.Simiti@stm.utcluj.ro