Journal of Achievements in Materials and Manufacturing Engineering

VOLUME 40
ISSUE 2
June
2010

Thixoinfiltration: a new approach to produce cellular and other low density metallic materials

M.H. Robert*, A.F. Jorge, F. Gatamorta, R.R. Silva
Faculty of Mechanical Engineering, State University of Campinas, SP, Brazil
* Corresponding author: E-mail address: helena@fem.unicamp.br

Received 11.03.2010; published in revised form 01.06.2010

Manufacturing and processing

ABSTRACT

Purpose: the work presents an innovative approach for the production of cellular metallic materials as well as low density metal matrix composites, by using thixoforming techniques; thixotropic semisolid metal is infiltrated into removable and non-removable space holder preforms. Different kinds of preforms are tested to obtain open cell material (sponges), syntactic foams and low density composites. Products are evaluated concerning relative density and mechanical behavior under compressive stresses.

Design/methodology/approach: Al alloy AA2011 was infiltrated in the semisolid state into preforms of sintered NaCl particles, sintered glass spheres, vermicular ceramic particles and porous ceramic granulates. After solidification, preforms were either removed by leaching (NaCl) resulting in open cell cellular product, or not (all others), resulting in composites of low density. Tomography tests were used to observe internal quality, and semi-static and dynamic compression tests were performed to evaluate the deformation ability of the material.

Findings: results show that thixoinfiltration is a simple and low cost technique to produce different types of low density, porous material. Open cell material as well as syntactic foams and low density composites can be produced with reliable internal quality and dispersion of cells and reinforcement. Composites containing porous reinforcements can present some mechanical characteristics of the conventional cellular metals.

Research limitations/implications: as all new developments, the complete understanding of the influence of processing variables upon the final quality of the product, as well as its consistency, must be provided before the technology can be widely used commercially.

Practical implications: the technique can represent an alternative, low cost processing route for the fabrication of sponges, foams and low density composites, which can avoid restrictions and operational difficulties of presently available manufacturing processes based in liquid manipulation or powder sintering methods.

Originality/value: infiltration of appropriate preforms by thixotropic metallic alloys to produce low density composites and cellular material is a new technique under development by the proposing group at FEM/UNICAMP.

Keywords: Casting; Cellular material; Composites; Thixoforming

Reference to this paper should be given in the following way:
M.H. Robert, A.F. Jorge, F. Gatamorta, R.R. Silva, Thixoinfiltration: a new approach to produce cellular and other low density metallic materials, Journal of Achievements in Materials and Manufacturing Engineering 40/2 (2010) 180-187.

1. Introduction

In the last two decades, cellular metallic materials have been increasingly produced and utilized in different sectors such as building and architecture, mechanical and chemical industries, for biomedical purposes among others, due to their unique combination of properties: low density, high impact absorption, damping properties, sound and thermal insulation, etc [1-3]. Such properties intend to fulfil growing demands by the society for environmental friendly materials and processing techniques, besides increasing requirements of quality and performance of engineering artefacts [4-6]. Cellular metals can be classified, according to the Rio summit in 1992, as an ecomaterial category 4 – Materials for society and human health, sub-category III.b - Materials for reducing human health impact [7]. In particular as human implants, cellular metallic materials can represent a new approach to the usual plastic formed Ti implants [8].

Different kinds of cellular structures are available, which can be classified according to the arrangement of empty space and metallic walls delimitating them, or the architecture of the structure. Indeed, open cell (sponges), closed foamed cell (foams), syntactic foams are typical products, presenting different cells arrangement [9,10]. At this point, a particular kind of syntactic foam can be considered, those presenting low density particulates as cells. This material, in fact a low density composite, could present some properties of conventional foams and worth to be investigated.

Several processing routes are already available for the production of cellular metals [10, 11] and also for the production of metal matrix composites [12], some based on simple concepts and involving simple operational methods and others dealing with complex techniques which may present difficulties concerning processing control.

In previous work [13-14] it was shown the feasibility of using SSM technology to produce open cell porous Al alloys by infiltration of the metal in the thixotropic semisolid condition, into a porous preform of removable space holder particles. The use of SS metals implies in lower temperatures when comparing to liquid processing, resulting in higher operational control and costs savings.

The present work aims to contribute to the development of the SSM route to produce cellular metallic materials, widening its application to different sorts of low density metallic materials, such as sponges, syntactic foams and syntactic filled-cell foams. Additionally investigates the mechanical behavior, under compression, of obtained products.

2. Experimental procedures

In the experiments it was used the alloy AA 2011, with basic composition Al-5.3%Cu, 0.27%Si, 0.4%Fe (wt %).

As the alloy was processed in the semisolid condition, it was mandatory the previous knowledge of the *solidus* and *liquidus* temperatures (T_s and T_l), in order to properly define the thixoprocessing window. For this purpose differential scanning calorimetry (DSC) tests were performed in different conditions. As values of T_s and T_l are very sensitive to DSC conditions [15], values obtained during heating cycle at 5ºC /min, which are close to the practical conditions used in the thixoinjection experiments, were considered. So determined solidification interval ranges between T_s = 575ºC and T_l = 653ºC.

As space holder agents, four types of materials were used: NaCl particles, hollow glass microspheres, foamed ceramic granulates (cinasite) and expanded vermicular ceramic particulates (vermiculite). All these materials are stable in semisolid AA2011 in the processing conditions.

Sodium Chloride: NaCl particles with poliedric geometry and three distinct size distributions were used: fine (average dimensions ranging from 1.6mm > ϕ_F > 1.0mm), medium (3.15mm > ϕ_M > 1.6mm) and coarse (ϕ_C > 3.15mm). Porous space among particles vary from 0.6 a 2mm.

After processing, preform was leached out by dissolution in hot water. As a result, samples with open and interconnected cells (sponges) were obtained. Complete removal of the salt particles was assured.

Hollow glass microspheres: preforms of sintered hollow glass microspheres were kindly provided by the Fraunhofer Institute for Manufacturing Technology and Applied Materials Research (IFAM, Bremen, Germany). Average spheres diameter and size of empty space among spheres are 27±14μm and 49±13μm, respectively. Fig. 1 shows glass microspheres in the preform used in the thixoinfiltration tests.

After the alloy solidification, resulting product is a syntactic foam, containing the microspheres as cells.

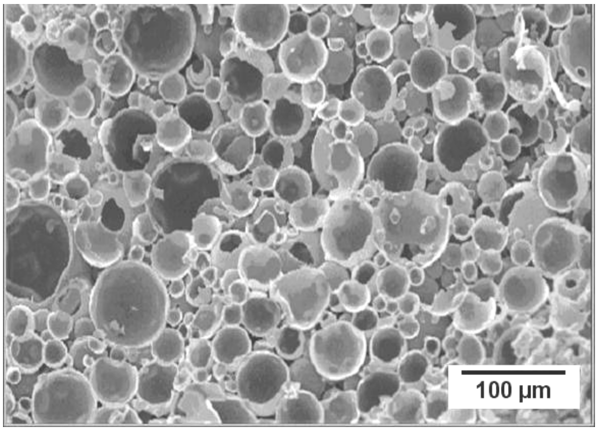


Fig. 1. Preform of hollow glass microspheres used in the thixoinfiltration experiments (IFAM - Bremen, Germany)

Foamed ceramic granulate (cinasite): particles with irregular geometry (spheroidish) and three distinct dimensions were used: fine (1.6mm > ϕ_F > 1.0mm), medium (3.15mm > ϕ_M > 1.6mm) and coarse (5mm > ϕ_C > 3.15mm). The particles are porous, with density of 0.85g/cm^3. A mass ratio of 3:1 of the alloy to the granulates was used.

Chemical composition of the porous granulate is shown in Table 1; while Fig. 2 shows its general aspect. After processing resulting product is a metal matrix composite.

Table 1.
Typical composition of the porous ceramic granulates (cinasite) and expanded vermiculite particles used in the experiments

Type of material	cinasite	vermiculite
Element	% wt	%wt
Silica (SiO_2)	57.6 ~ 66.8	38 ~ 46
Ferric Oxide (Fe_2O_3)	9.6 ~ 3.9	6 ~ 13
Alumina (Al_2O_3)	19.4 ~ 18.9	10 ~ 16
Calcium Oxide (CaO)	3.4 ~0.9	1 ~ 5
Mg Oxide (MgO)	2.6 ~1.7	16 ~ 35
Alkalies (Na_2O)	5.6 ~ 4.6	5.6 ~ 4.6
Potassium Oxide (K_2O)	-	1 ~6
Titanium Dioxide (TiO_2)	-	1 ~3
Water (H_2O)	not detected	8-16
Other	-	0.2 ~12

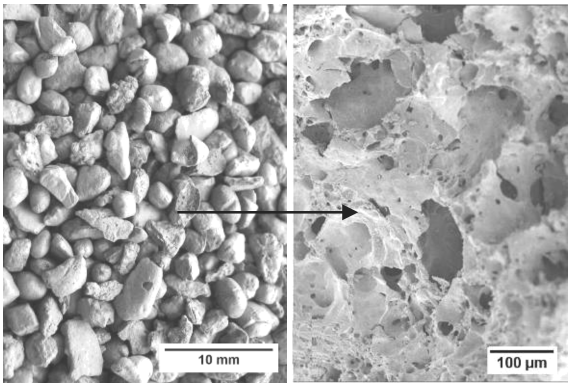


Fig. 2. Porous ceramic granulates used in thixoinfiltration experiments to produce low density composites

Expanded vermiculite: vermiculite is a hydrated magnesium-aluminum-ironsilicate with lamellar structure. After expansion, its density ranges between 0.08 to 0.14g/cm^3. Size of particles vary between 1.6mm and 5mm. A mass ratio of 17:1 of the alloy to the vermiculite particles was used in the experiments.

Chemical composition of the expanded vermiculite is shown in Table 1, while the general aspect of particles can be observed in Fig. 3.

After processing, the resulting product is a metal matrix composite.

Semisolid AA2011 samples were produced by heating the alloy from fine, equiaxial grains with diameter from 16 to 120µm, to temperatures within the solidification range. After reaching the predefined temperature (T_{tixo}), the material was kept for 5 min to promote complete spheroidization of the solid phase. In order to reduce the final dimensions of the globular primary phase present in the thixotropic material, it was also used pre-deformed (80% cold deformation by compression) samples. Heating temperatures (T_{tixo}) were defined to promote liquid fractions in the thixotropic semisolid from 0.2 to 0.4.

For thixoinfiltration tests, the preform was placed between two layers of solid AA2011 alloy and the assembly was heated to the chosen T_{tixo}. As the thixotropic condition was reached, pressure was applied to promote the metal infiltration into the the preform. Fig. 4 illustrates the experimental assembly.

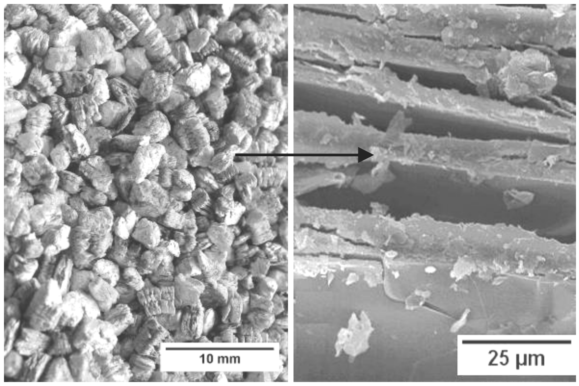


Fig. 3. Expanded vermiculite particulates used in thixoinfiltration experiments to produce low density composites

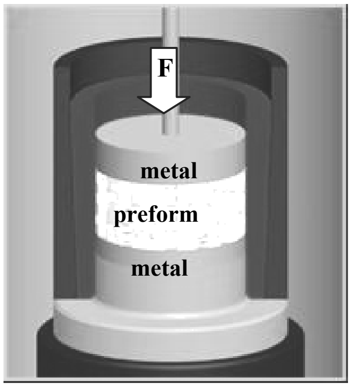


Fig. 4. Schematic representation of assembly utilized for infiltration of thixotropic semisolid metal into a preform of space holder particles, to produce porous metallic material

Obtained products were characterized concerning density by Archimedes method and He picnometry; porosity and reinforcement distribution was analyzed by tomography images of parallel sections taken each 1.5mm. Samples were submitted to uniaxial semi-static compression tests in an universal servo-hydraulic equipment; lateral external surfaces were kept free for lateral expansion. Dynamic compression tests were performed in a drop test equipment.

3. Results and discussions

3.1. Thixotropic structure

A typical structure of the AA2011 alloy produced by heating the material to a temperature within *solidus*/*liquidus* range is shown in Fig. 5. In the particular case presented in the photo, the material was submitted to cold deformation before heating. It can be seen the typical thixotropic constitution of globular α-phase (solid at T_{tixo}) and fine boundaries of the secondary phase $CuAl_2$

(liquid at T_{tixo}). Globules diameter ranges from 14 to 98μm. This structure is obtained by separation of recrystallized grains due to liquid penetration in the grain boundaries at high temperatures. This particular semisolid was used for infiltration into the preforms with the finest porosity (glass microspheres preforms). Similar kind of structure was obtained by heating to the same T_{tixo} the alloy with fine equiaxial grains, without any previous additional deformation; however, in this case, coarser α-phase globules were obtained (20 - 40μm) due to growth of the original grains in the liquid environment during spheroidization treatment. Slurries with higher glolubes size were used for infiltration tests into preforms with coarser porosity.

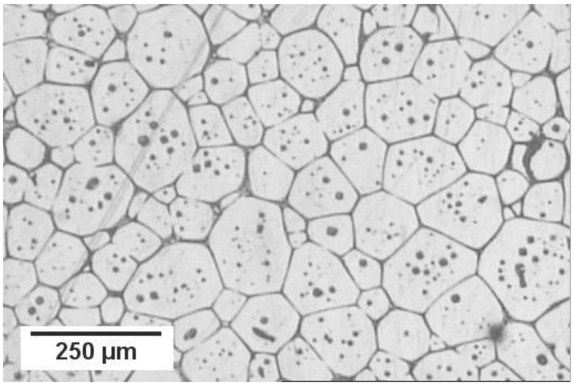


Fig. 5. Microstructure of thixotropic AA2011 used for infiltration into space holders preforms to produce porous material. Heating conditions: 630 ºC/5min, from cold deformed condition

In all cases, dimensions of solid globules in the thixotropic semisolid alloy were compatible with the dimensions of porosity to be infiltrated in the preforms. Indeed, complete filling of the porosity in the preforms by the semisolid could be achieved in most of the cases; some defects due to lack of total infiltration were detect only when using low liquid fraction in the semisolid associated with very small porosity in the preform. Low values of pressures, ranging from 2 to 10MPa, were required for total infiltration, due to the high flowing behavior of the thixotropic material.

3.2. Products obtained by thixoinfiltration into removable preforms

Fig. 6 presents general aspect of a typical product obtained by infiltration of semisolid AA2011 into a preform of NaCl particles. The material is characterized as open cell, with cell size according to the dimensions of the NaCl particles in the preform. Finer NaCl particles led to thinner cell walls in the sponge, as the porosity was smaller and better distributed in the preform in this case, when compared to performs containing coarser NaCl particles.

Tomography analysis indicates good internal quality of the product, given by the ability of the semisolid to infiltrate and fill the porosity of the preform. In rare cases, when using performs made from the finest NaCl particles, infiltration was not perfect, resulting in internal defects in the product. The influence of thixoinjection parameters in the infiltration ability of semisolid into NaCl performs was investigated elsewhere [13, 14].

Infiltration pressure required ranged from 3 to 4 MPa, increasing as porosity of the preform decreases. Such pressure is low enough to prevent any damage of the preforms.

Also in Fig. 6 are presented the general aspect of the open cell architecture of the product and the microstructure of the cell wall, presenting α phase with globular morphology, as in the slurry previously to the infiltration.

Relative density of obtained products ranged from 0.25 to 0.35 (0.58 a 0.97±0.02g/cm^3), increasing as the size of NaCl particles used as space holder increases.

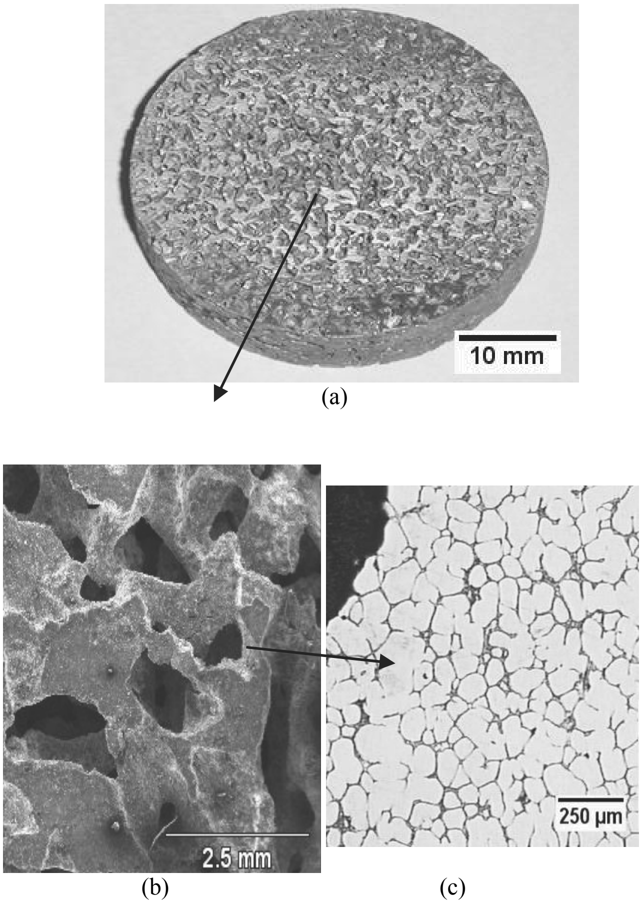


Fig. 6. Typical sample produced by thixoinfiltration of AA2011 alloy into a preform of removable NaCl particles; (a) general aspect; (b) general aspect of the architecture; (c) microstructure of cell walls

Stress x strain curves in semi-static compressive tests of samples of different densities are presented in Fig. 7; and results on mechanical properties are listed in Table 2.

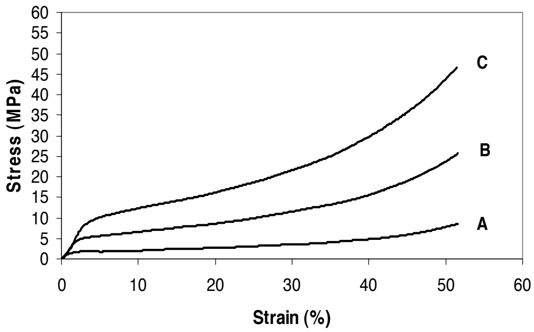


Fig. 7. Stress x strain curves in semi-static compression tests of open cell AA2011 with different densities, produced by thixoinfiltration into preforms of removable NaCl particles

Table 2.
Mechanical behavior under semi-static compression, of open cell AA2011 produced by thixoinfiltration into NaCl preforms

Density (g/cm^3)	Rel. density (ρ_{cel}/ρ_{bulk})	E (MPa)	EN_a (J/g)	σ_{pl} (MPa)	ε (%)
0.58	0.21	133	1.1	3.0	39
0.78	0.28	249	2.8	9.1	36
0.89	0.32	299	3.7	17.6	29

E: Young's modulus; σ_{pl} : plateau stress (average stress in the plateau region); ε: densification strain (deformation until the beginning of the densification stage); EN_a: absorbed energy.
Bulk AA2011: $\rho_{bulk} = 2.82\ g/cm^3$; $E = 82.10^3$ MPa.

It can be observed in all cases, typical behavior of a cellular material. Deformation curves show three distinct regions: short period of elastic deformation followed by a long plateau of plastic deformation with negligible stress increase, due to the collapse of porous, and a final stage, after densification of the material, when further deformation requires significant stresses. Young's modulus and plateau stress increase significantly with increasing density of the samples; therefore, in this case, when thicker cell walls are present. Densification strain tends to decrease due to higher resistance imposed by the thick metallic cell walls in the material with higher density.

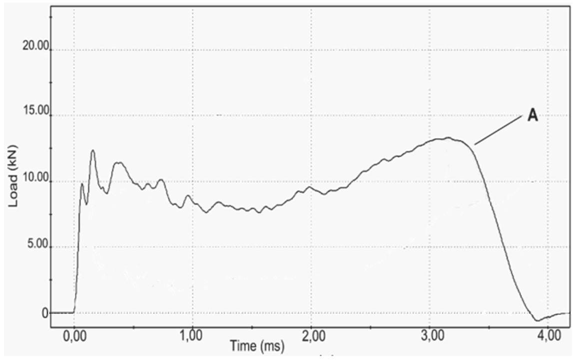
The area under the stress x strain curve, given by equation (1), is the absorbed energy during compression. Ideal energy absorbers should present a long flat stress-strain curve.

$$E = \int_{\varepsilon_1}^{\varepsilon_2} \sigma(\varepsilon)\, d\varepsilon \qquad (1)$$

E = absorbed energy in a deformation interval from ε_1 and ε_2.

When dealing with absorbers for human protection, as in vehicle safety applications, it is necessary to choose a cellular material with the longest plateau stress with values bellow that which would cause injure. Therefore, in this kind of application, the absorber must retain the kinetic energy of the moving car without reaching its densification strain. Calculated absorbed energy in compression tests of the cellular AA2011 produced using NaCl space holders, results in values from 1 to 6 J/g. These values lies within the range of absorbed energy/mass for most commercial cellular metallic absorbers, which can vary from 0.1 to 40J/g for circa 25% deformation [15].

Results on dynamic compression tests (impact experiments) are presented in Fig. 8 and Table 3. High plastic deformation with no significant increase in the applied load is observed, and therefore high capacity to absorb the kinetic energy in impacts.



(A) 0.94g/cm^3

Fig. 8. Typical load x time curve in dynamic compression test of open cell AA2011 produced by thixoinfiltration into preforms of removable NaCl particles

Table 3.
Mechanical behavior under dynamic compression tests of open cell AA2011 produced by thixoinfiltration into NaCl performs

Density (g/cm^3)	Rel. density (ρ_{cel}/ρ_{bulk})	σ_{pl} (MPa)	EN_a (J/g)
0.94	0.33	28.3	5.2

The influence of architectural features and density on the mechanical behaviour is clear: increasing density, cells walls thickness and porous dimensions promoted by increasing dimensions of space holder particles result in decreasing of plastic deformation and increasing of plateau stress.

Increasing dimensions of porous, even with simultaneous increase in cell walls thickness and density (samples produced from coarser NaCl particles), result in increase of the absorbed energy in compression. Therefore, the influence of density alone must be taken carefully as architectural parameters can be significant in the materials behaviour. This observation reinforces the necessary attention when choosing a cellular material for a specific application, as in the case of impact absorbers for passengers protection as mentioned previously, when high ability of energy absorption must be associated with low plateau stress.

3.3. Products obtained by thixoinfiltration into non-removable preforms

Thixoinfiltration of AA2011 alloy into hollow glass microspheres: a typical product obtained by infiltration of semisolid AA2011 into preforms of hollow glass spheres is presented in Fig. 9. The material is characterized as syntactic foam with the microspheres as closed cells. Tomography analysis indicates good internal quality of products in most of the cases. Rare situations did not promote complete filling of the preform, particularly those when low liquid fraction is present in the semisolid.

The presence of fine solid globules in the semisolid metal produced by recrystallization route, used in this case, provided appropriate infiltration ability even into the small porosity of the preform. Infiltration pressures required ranged from 4.4 to 5.6 MPa, depending on the liquid fraction in the semisolid. These pressures are lower than the necessary to promote the fracture and collapse of the glass spheres as previous tests showed that preform collapses at 12.5 MPa. However, some spheres do suffer rupture during tixoinfiltration, causing metal penetration and filling of the space, as observed.

Relative density of 0.55 was obtained; this value, although not too low as far as general foams are concerned, is typical for syntactic foams, where cell wall can be formed by a material of different nature. In the present case, the filling of part of the hollow microspheres works to increase the expected density.

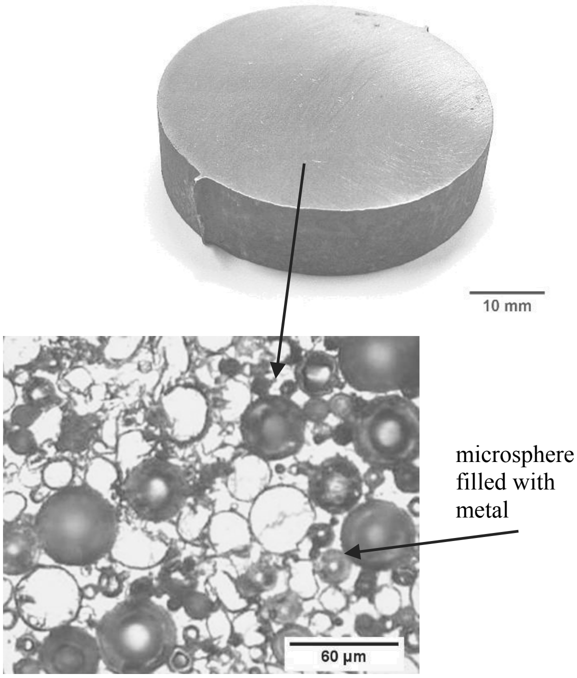


Fig. 9. Typical sample produced by thixoinfiltration of AA2011 alloy into a preform of hollow glass microspheres

Typical stress x strain curve in semi-static compressive tests is presented in Fig. 10, and results on mechanical properties are listed in Table 4. Also in this case high plastic deformation is observed and a deformation plateau can be identified. However, the higher density of the material, when compared to the sponges produced by thixoinfiltration in NaCl preforms, leads to higher values of stress plateau and Young modulus, and lower densification strain.

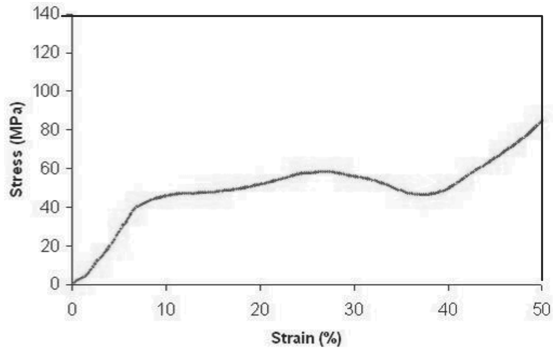


Fig. 10. Typical stress x strain curve in semi-static compression tests of syntactic foam produced by thixoinfiltration of AA2011 into preform of hollow glass microspheres

Table 4.
Mechanical behavior under semi-static compression tests of syntactic foam produced by thixoinfiltration of AA2011 into preform of hollow glass microspheres

Density (g/cm^3)	Rel. density (ρ_{cel}/ρ_{bulk})	E (MPa)	σ_{pl} (MPa)	EN_a (J/g)	ε (%)
1.56	0.55	600	49	12.5	39

Absorbed energy is high but it must be considered, in case of passengers protection, the high value of the stress plateau. Dynamic compression tests (impact in drop test) were also performed and results show an ability of energy absorption around 6x higher than the bulk material.

<u>Thixoinfiltration of AA2011 into expanded ceramic granulates</u>: Fig. 11 presents general aspect of typical product obtained by infiltration of semisolid AA2011 into a preform of expanded ceramic granulates.

Infiltration pressure required for full infiltration ranged from 7 to 10MPa, increasing as granulate size decreases. No lack of infiltration was detected in any case, as tomography analysis indicated. It could be observed in all cases, a homogeneous distribution of the ceramic porous particles throughout the metallic matrix. Relative densities of products vary from 0.35 to 0.6, increasing as dimensions of the ceramic particles increases. The density of the products lies within or close to that considered typical for cellular materials.

Dynamic compression tests (impact in drop test) results are presented in Fig. 12 and Table 5. Curves produced by samples of different densities show a plateau of plastic deformation typical of porous materials; however, the plateau occurs at relatively high strain levels. Moreover, the absorption of energy in the impact, however lower than the observed for the other cellular materials produced, is considerable, taking in account the range of 0.1 and 40J/g at 25% deformation, generally taken for metallic cellular absorbers, as indicated by Ashby [15] and mentioned previously.

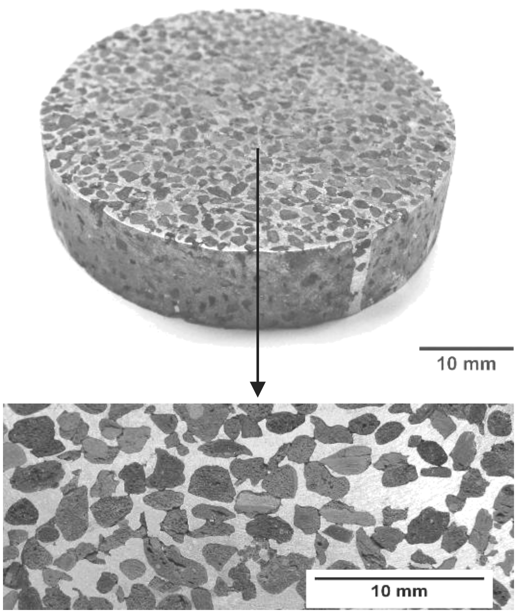
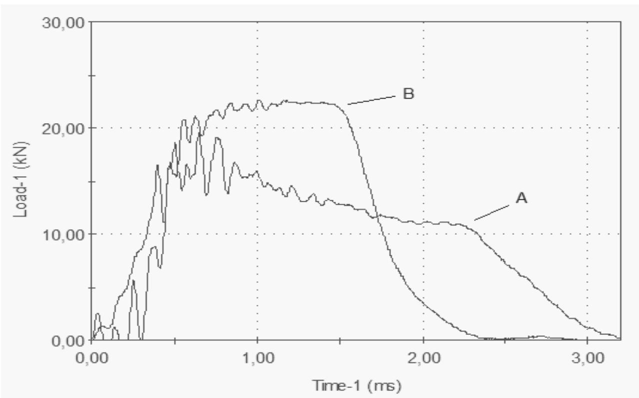


Fig. 11. Typical composite produced by thixoinfiltration of AA2011 alloy into preforms of expanded ceramic granulates



(A)1.00g/cm^3 (B) 1.34g/cm^3

Fig. 12. Load x time curves in dynamic compression tests of composites with different densities, produced by thixoinfiltration of AA2011into preforms of expanded ceramic granulates

Table 5.
Mechanical behavior under dynamic compression tests of low density composites produced by thixoinfiltration of AA2011into preforms of expanded ceramic granulates

Density (g/cm^3)	Rel. density (ρ_{cel}/ρ_{bulk})	σ_{pl} (MPa)	EN_a (J/g)	ε (%)
1.00	0.35	37	0.97	20.8
1.34	0.47	70	1.09	11.0

As a lightweight porous reinforcement, the expanded ceramic particles contribute for the low density of the produced material; on the other side, the high stiffness and brittle characteristic of such particles can lead to the high stress levels observed. It can be also observed that increasing density leads to lower deformation and high plateau stress, as expected.

Thixoinfiltration of AA2011 alloy into vermiculite particulates: Fig. 13 presents typical product obtained by infiltration of semisolid AA2011 into a preform of vermiculite particulates. Relative density of products varies from 0.42 to 0.39, increasing as dimensions of vermiculate particulates increase, which could be attributed to the higher infiltration ability of the semisolid metal when coarser particles are used. However, no visible infiltration defects were detected by tomography analysis, indicating good internal quality of the product in all cases. Further studies on microporosity distribution could clarify the influence of particles dimension in the infiltration ability and consequent density of the material. An acceptable distribution of the ceramic particles in the metallic matrix is observed as well as the presence of metal within the lamellar structure of the particulates.

Infiltration pressure required was in the order of 2MPa, increasing as porosity of the preform decreases. In rare cases when higher pressures were applied, compacting of the ceramic particles during processing was observed.

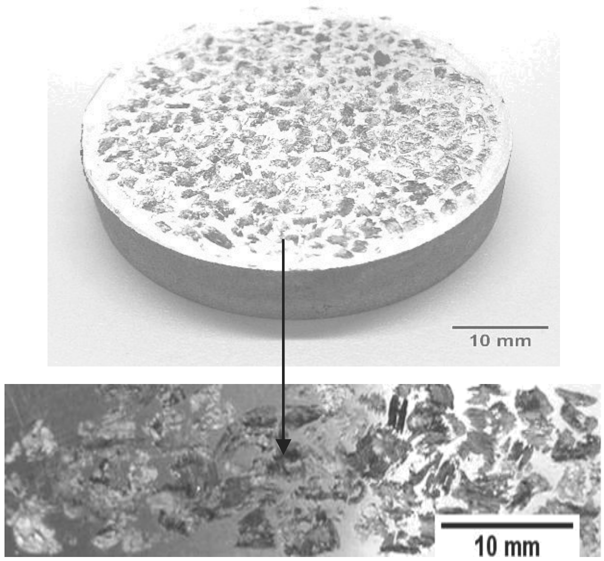
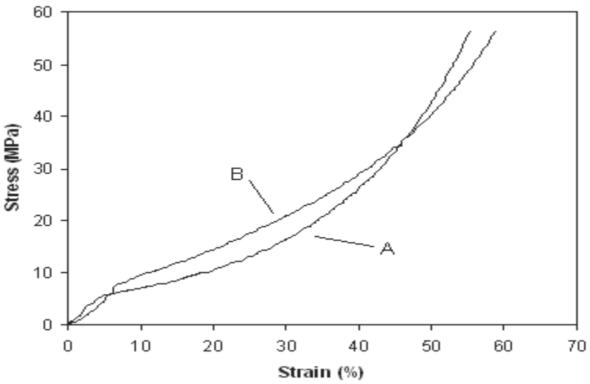


Fig. 13. Typical composite produced by thixoinfiltration of AA2011 alloy into preforms of expanded vermiculite particulates

Typical results of semi-static compressive tests of samples of different densities are presented in Fig. 14 and Table 6. In this case the typical plateau of plastic deformation usually present in cellular materials is not clearly defined, although a considerable plastic deformation is achieved at low stresses.



(A)1.1g/cm^3 (B) 1.2g/cm^3

Fig. 14. Stress x strain curves in semi-static compression tests of composites with different densities, produced by thixoinfiltration of AA2011 into preforms of expanded vermiculite

Table 6.
Mechanical behavior under semi-static uniaxial compression of low density composites produced by thixoinfiltration of AA2011into preforms of expanded vermiculite

Density (g/cm^3)	Rel. density (ρ_{cel}/ρ_{bulk})	*E* (MPa)	$\sigma_{\varepsilon(30\%)}$ (MPa)
1.1	0.39	120	16
1.2	0.42	122	21

$\sigma_{\varepsilon(30\%)}$: stress at 30% strain

Densification occurs more gradually due probably to the presence of the metal infiltrated into the particles. Young modulus values are similar to those obtained for low density sponges produced by thixoinfiltration into NaCl performs. Although difficult the determination of the energy absorbed during deformation, the high ability of plastic deformation at low stresses can suggest that this low density composite could be suitable for applications as absorbers. Moreover, the presence of ceramic particulates can attribute other interesting properties to this low density composite.

4. Conclusions

Results show the feasibility of the application of semisolid technology to produce open cell and closed cell syntactic foams, as well as low density metal matrix composites by thixoinfiltration of the alloy into removable preforms of NaCl particles or non removable preforms of hollow glass microspheres and ceramic porous particulates. The process is easily handled and controlled, and flexible as far as variables are concerned, allowing the production of a wide range of materials and architectures.

Concerning the materials produced, results show that composites containing porous reinforcements can present some mechanical characteristics of the conventional cellular metals. Taking in account this behavior associated to the low density and other specific properties inherent to composite materials, more attention must be paid to this new kind of material.

Acknowledgements

The authors want to thank financial support from CAPES, FAPESP, CNPq; and also Dr. Sergio Luis M. dos Santos, from the Radiology Dept., General Hospital, UNICAMP, who kindly performed the tomography examination. Authors want also to thank Dr. Jorg Weise, from IFAM/Bremen, Germany, who kindly provided the glass microspheres preforms.

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