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Metal matrix syntactic foams produced by pressure infiltration – the effect of infiltration parameters

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Abstract: Metal matrix syntactic foams (MMSFs) were produced by pressure infiltration. Two parameters of the infiltration process (pressure and time) were varied and the infiltrated length was measured as the function of infiltration parameters in order to get data for the implementation of pressure infiltration as mass-production of MMSFs similar to injection mould casting, especially in the short infiltration time range (<10 s). The infiltrated length was found to be linear function of pressure and square-root function of time.. The effect of the infiltration parameters on the microstructure and mechanical properties of MMSFs were investigated by optical microscopy and standardised compression tests. The microscopic images were used to qualify the pressure infiltration and showed that more than one combination of infiltration parameters can be found for successful production of a part with given required dimensions. Considering the compression tests, the main characterising properties were mapped as function of infiltration parameters. The registered values showed dependency on the infiltration parameters and indicated that a given infiltration length produced by higher pressure and shorter time has better mechanical properties. The infiltrated specimens were isotropic, anisotropy was not observed in the reference measurements.

Keywords: metal foam; infiltration; compression test; microstructure

1. Introduction

Metal matrix syntactic foams (MMSFs) are particle reinforced composites consisting of low weight metal matrix (Al or Mg for instance) and hollow spheres in closely or randomly packed structure [1, 2]. The hollow spheres' material is usually some sort of ceramic (SiO_2, and/or Al_2O_3) or metallic (pure iron or steel) and they are available in commerce [3-7]. MMSFs are made for various reasons, for example to produce energy-saving lightweight components, collision and vibration dampers or core material for sandwich composites applied as hulls.

There are two common ways to produce MMSFs; both of them use the matrix in liquid state. On one hand MMSFs can be made by stir casting. In this case the matrix material is melted, overheated and the pre-heated hollow spheres are added in small amounts during continuous stirring [8-16]. The advantages of this process are simplicity and low cost. Shortcomings are the potential fracture of the hollow spheres due to the mechanical mixing and the lower volume fraction of the hollow spheres compared to the theoretically possible. On the other hand MMSFs can be made by infiltration. In the case of wetting matrix-reinforcement systems (e.g. Al matrix and Fe hollow spheres) the infiltration can be spontaneous and gravity casting can be successfully used to produce MMSFs [17-20]. If the matrix-reinforcement system is non-wetting (e.g. Al matrix and Al_2O_3 hollow spheres) a threshold pressure is needed to initialise infiltration, most commonly inert gas pressure is used [21-29]. Gas pressure infiltration is similar to low pressure hot chamber injection mould casting and it is important as possible industrial scale production method of MMSFs. Gas pressure infiltration has three main parameters: infiltration pressure, time and temperature.

The infiltration pressure is forcing the molten matrix metal into the gaps between the hollow spheres. It is normally significantly higher than the threshold pressure because along the path of the molten metal the infiltration pressure decreases due to viscous and form drag like in usual flows. Asthana et al. [30] studied the infiltration of metal matrix composites (MMCs) and found that the infiltrated length is linearly proportional to the pressure. The same was also derived by Garcia-Cordovilla et al. [31] for packed ceramic particulates and liquid metals. According to these equations with sufficiently high pressure any size of MMSF can be produced theoretically. However an upper limit exists: namely the fracture strength of the hollow spheres. If the pressure exceeds this limit the molten metal can fill up the hollow spheres and the foam structure is lost.

A few review papers were published about the effect of infiltration time [30-34]. All of these papers present theoretical considerations to derive the infiltrated length as square root function of time. Besides the infiltration pressure and time the equations depend on the dynamic viscosity (η), the surface tension (γ), the contact angle (Θ) and geometrical dimension (for example the radius (r) in the case of straight capillaries). The existing models use common and serious simplifications:

- Geometrical simplifications to get analytically solvable closed formulas (regular spatial distribution of capillaries instead of random distribution, permeability and tortuosity of the capillaries etc.).
- The chemical reactions between the reinforcement and matrix is neglected (the reactions generally reduces the surface tension and wetting angle).
- The time dependence of the wetting angle, the air resistance and the gravitational force are neglected.

With the above mentioned restrictions Washburn [32] analysed the dynamics of capillary flow and found that for simple cases like straight capillaries it is possible to derive closed form formulas to predict the infiltrated length as the function of pressure and time. However in more complicated cases these formulas would not apply and the infiltration length could only be determined by experiment. Semlak and Rhines [33] studied the rate of capillary rise of liquid metals in porous-metal bodies consists of parallel capillary tubes. Asthana et al. [30] studied non-reactive particle reinforced MMC systems and found that, a comprehensive theoretical framework suitable for rationalizing all the observed features of pressure-infiltrated MMCs is lacking due to the extremely complex physicochemical and hydrodynamic nature of the process. In the case of complex problems direct measurements could be more practical. Garcia-Cordovilla et al. [31] investigated non-reactive ceramic particulate-liquid metal systems. Although the discussed results showed much progress in the

process of infiltration many questions remained unsolved. For example the nature and reason of the observed incubation period in the infiltrated length-infiltration time diagrams. Kaptay [34] discussed some aspects of high-temperature capillarity and provided an extended set of mathematical expressions connecting different phenomena relevant to production of MMCs with interfacial energies. Muscat and Drew [35] investigated the kinetics of infiltration of molten Al in TiC preforms. Short incubation periods in the infiltrated length – infiltration time diagrams were observed. In the case of reactive matrix-reinforcement systems Kevorkijan [36] experimentally monitored the dynamics of the infiltration process in the time range of 100-3600 s. The reinforcements were SiC, Si_3N_4, AlN, Mg_3N_2, TiO_2 and fused silica, while the matrix was standard A356-T6 aluminium alloy (7 wt% Si and 0.3 wt% Mg). It was found that the infiltration length increased linearly with the square root of time. Eustathopoulos et al. [37] studied the effect of oxygen-wetting transition in metal/oxide systems. From the analysis, it was shown that in the presence of oxygen a definite decrease of the contact angle (Θ) could be observed due to the adsorption of oxygen-metal clusters at the metal/oxide interface. In Al-SiO_2 systems it can be explained by the decomposition of SiO_2, while in the case of Al-Al_2O_3 system the rupture of the oxide layer on the melt cause a ~40° decrese in the contact angle (Θ).

Finally the infiltration temperature (as third infiltration parameter) has only indirect effect on the infiltrated length through the temperature dependent properties (η, γ and Θ). Higher temperatures can also initialise or fasten possible chemical reactions.

In summary the results of existing infiltration models give satisfactory predictions on the infiltrated length as the function of pressure, time and temperature in the case of simple systems, however large deviations from the predicted results can be observed in case of reactive systems having complex geometry, changing permeability and short infiltration times, such in the case of MMSFs. Therefore the first aim of this paper is to determine

infiltrated length values through experimental methods as function of infiltration pressure and time at constant temperature in order to ensure base data for the implementation of low pressure hot chamber injection mould casting as industrial scale production method of MMSFs. The measurements have been performed in the short infiltration time range (<10s), in order to analyse the nature of the so called 'incubation period' and to get results from this range typically lacking or not detailed in the professional literature.

Additionally the produced MMSFs should be qualified both on the microstructure scale (to qualify the infiltration process itself) and on the aspects of mechanical properties (to ensure design parameters for engineers). The overall microstructure of MMSFs is usually investigated by optical microscopy. Extended studies apply scanning electron microscopy (SEM) with energy dispersive X-ray spectrometry (EDS) in map and line scan modes to investigate the interface layer, that responsible for load transferring from the matrix to the reinforcement [38-40]. Considering the mechanical characteristics mainly the compressive properties have been studied. Palmer et al. [21] performed compressive tests on Al based MMSFs reinforced with 45 μm or 270 μm hollow spheres. The MMSFs with smaller hollow spheres showed higher compressive strength. Rohatgi et al. [24] also investigated the size effect of the hollow spheres and confirmed the same relationship. Balch et al. [41-42] investigated Al matrix MMSFs and they have found that, MMSFs can ensure higher mechanical properties than conventional metallic foams. Kiser et al. [43] investigated the mechanical response MMSFs under compression loading. Extremely high energy absorption capacity was observed compared to conventional metal foams. Wu et al. [44] established a new method to predict the compressive strength of MMSFs. Tao et al. [45-47] investigated the mechanical properties and failure mechanisms of MMSFs with monomodal and bimodal distribution of hollow spheres. The bimodal foams have the advantages of a flat plateau regime, high plateau stress and good ductility. Zhang et al. [25] studied the mechanical

response of Al matrix MMSFs with low-cost porous ceramic spheres of diameters between 0.25 and 4 mm. They found that the amount of energy absorption was mainly determined by the volume fraction of Al and to a lesser extent by the mechanical properties of the ceramic spheres. Mondal et al. [11] measured considerably higher plateau stress on MMSFs containing 30–50 vol% hollow spheres than in the case of conventional Al foams. Rabiei and O'Neill [18] produced MMSFs reinforced by steel hollow spheres, that displayed superior compressive strength and energy absorption capacity. By compression and low-velocity impact tests Castro et al. [48] confirmed the MMSFs as potentially beneficial materials in high energy absorption applications. Peroni et al. [49-50] investigated the mechanical behaviour of MMSFs made of hollow glass microspheres mixed in an iron matrix. Compared to other types of metal foams it showed greatly increased quasi-static compressive strength. Besides the compressive behaviour other mechanical properties have been also studied: the wear properties of MMSFs was examined on pin-on-disc apparatus and showed much better wear behaviour, than the pure matrix [12, 15, 16, 51]. On the other hand the creep resistance has been also studied on aluminium matrix syntactic foams [52].

According to above mentioned mechanical investigations the second aim of this paper is to map the standardised compressive properties (DIN 50134 [53]) of the produced MMSFs as the function of infiltration parameters.

2. Materials and methods

2.1 Materials

Ceramic hollow spheres of SL300 grade from Envirospheres Pty. Ltd. [4] were used as reinforcement. The average diameter, wall thickness and density of the hollow spheres were 150 μm, 6.75 μm and 0.692 g/cm^3 respectively. The wall of the hollow spheres was built up from oxide ceramics (33 wt% Al_2O_3, 48 wt% amorphous SiO_2 and 19 wt% mullite ($3Al_2O_3 \cdot 2SiO_2$)). AlSi12 alloy (Al4047) was used as matrix material; it contained: 12.830

wt% Si, 0.127 wt% Fe, 0.002 wt% Cu, 0.005 wt% Mn, 0.010 wt% Mg, 0.007 wt% Zn and the remaining is Al. This measured composition is in the range of the standardised nominal values [54]. The eutectic aluminium alloy was chosen because it has the lowest melting point (~575 °C) among the aluminium alloys and the high amount of Si helps to suppress the possible chemical reaction (Eq. 1) between the matrix and the SiO_2 content of the hollow spheres.

$$4Al_{(liq)} + 3SiO_{2(sol)} \rightarrow 2Al_2O_{3(sol)} + 3Si_{(sol)} \quad \text{(Eq. 1)}$$

At first glance this reaction is beneficial, because it transforms the amorphous SiO_2 to crystalline Al_2O_3 (better mechanical properties), but during the transformation the wall of the hollow spheres are harmed and (at least partially) dissolved into the matrix. The driving force of the above diffusion reaction is the Si concentration mismatch between the matrix and the wall material of the hollow spheres [38-40, 55]. By choosing the high Si amount Al alloy as matrix the reaction can be avoided and the system can be considered non-reactive [56].

2.2 Equipment and experimental method

An infiltration system has been designed and assembled to determine infiltrated length values as the function of infiltration parameters. The main part of the system is the pressure infiltration equipment (Fig. 1). The whole chamber has been built from 316L grade stainless steel. The main part is the upper tube (#14), its outer diameter, length and wall thickness is 20 mm, 250 mm and 1 mm respectively. The tube was fulfilled with SL300 hollow spheres and the spheres were tapped rigorously to achieve ~64% volume fraction and randomly closed packed equal spheres (R-CPES) structure [2, 57]. The packed tube was closed on both ends by meshes (#3). The hollow spheres in the tube were heated by a Kanthal heating-spiral (#16) up to 625 °C (50°C above the melting point of the AlSi12 matrix), it was continuously monitored by three thermocouples (TC2…4) in order to prevent freezing of the matrix during infiltration. The heating spiral was covered by heat insulator mat (#15) and it was separated from the tube by special rings made from refractory material (#2). The tube was fitted into an

upper cup (#5) and it was fixed by an upper plate (#17), three threaded rods (#13) and three wing nuts (#1). A lower tube (#11) was fixed to the upper cup (#5). A previously made preform of the matrix was situated in a crucible (#7), the crucible was placed in the pressure vessel (tube #6) and it was positioned by a space holder (#10). The temperature of the matrix material was continuously monitored by a thermocouple (TC1). The pressure vessel was closed and insulated by the upper (#5) and lower (#8) cups and refractory insulators (#9 and #12) respectively. During the tests Ar gas was used as pressurising agent. Three gas inlets (#4) with inner diameter of 8 mm were connected to the upper cup (#5). The assembled equipment was placed in the computer controlled infiltration system (Fig. 2). The equipment (E) and the matrix within it were heated by an induction coil (C, Power-Trak 15-96). A data acquisition system (DAQ) collected the signals of the thermocouples (T) and a pressure transducer (P), while controlled four high performance valves. One valve (V) was set to continuously fill the puffer to the previously set infiltration pressure from the Ar tank through a pressure regulator (R). Three high flow-rate valves (V1…3) were responsible to let the infiltration pressure into the equipment.

In the beginning of the process the equipment was continuously flushed by Ar gas and the heating of the matrix and upper tube started. After the matrix melted and the whole equipment reached the infiltration temperature (625 °C) the high performance valves were opened for the previously set infiltration time. After the infiltration the equipment was removed from the system and cooled down to room temperature. Subsequently the upper tube was opened and the infiltrated length was measured. The tests were performed at constant infiltration temperature (625 °C) and within the pressure and time ranges of 250…750 kPa (in 250 kPa steps) and 1…9 s (in 2 s steps) respectively. Five measurements were done and averaged with each parameter settings. After measuring the length, plate specimens for microstructural

investigations and cylindrical specimens for compression tests were manufactured from the infiltrated rods.

In the case of such short time tests it is essential to calculate (or at least estimate) the time necessary for the adequate pressure built up in the equipment. There are routes in the theory of transient gas dynamics to reach this aim, but for now a CFD model of the designed system and numerical solution is considered to be acceptable. The system was modelled basically by two vessels. A smaller one (0.5 dm^3) was used as the infiltration equipment (0 kPa at 700°C), while a larger one (25 dm^3) was used as the puffer (250 kPa at 25°C in initial state). In order to be strict and conservative the vessels were connected by only one 1 m long adiabatic cable having 8 mm inner diameter (there are three of them in reality), the drag in the cable was also considered, but the resistance of the high flow-rate valves (V1…3) were neglected. This model showed that the required gas pressure could build up within 50 ms (Fig. 3).

Light microscopic observations were done on polished specimens taken from the infiltrated rods along and perpendicular to their longitudinal axis. The polishing was done on an automatic grinding/polishing machine in five steps: (i) grinding on P320 SiC paper (22N, 220 min^{-1}; 1 min, counter direction), (ii) polishing with 6 μm diamond suspension (27N, 150 min^{-1}, 15 min, counter direction), (iii) polishing with 3 μm diamond suspension (27N, 150 min^{-1}, 6 min, counter direction), (iv) polishing with 0.05 μm SiO_2 suspension (27N, 125 min^{-1}, 3 min, compliance direction) and vibration polishing with 0.05 μm SiO_2 suspension on Buehler Vibromet 2 (6N, 60 min, 80% amplitude).

For the investigation of the interface zone between the hollow spheres and the matrix line-EDS) measurements were performed on the polished specimens by a Phillips XL-30 type SEM equipped with an EDAX Genesis EDS analyser. The excitation voltage was 20 kV, 100 points were measured along the lines, and each point was excited for 20 s with 35 μs detector acquisition rate.

The main loading mode of foam materials is the compression; therefore compression tests were performed on cylindrical specimens according to DIN 50134 standard [53]. The diameter and the height of the specimens were 14 and 21 mm respectively (H/D = 1.5). The longitudinal axis of the specimen coincided with the infiltration direction of the pieces. Reference measurements on specimens (Ø10×15 mm, due to dimensional restricts) with longitudinal axis perpendicular to the infiltration direction of the pieces were also done in order to get information about the potential anisotropy of the infiltrated specimens. The tests were performed on a MTS 810 type machine in a four column tool at room temperature. The surfaces of the tool were grinded and polished. The specimens and the tool were lubricated with anti-seize material. The test speed was 0.15 mm/s, which ensured quasi-static compression. Five specimens were compressed until 50% engineering strain from each MMSF type to get representative results.

3. Results and discussion

3.1 Infiltrated length

The measured and averaged infiltrated lengths and their scatters are plotted in Fig. 4. The scatter was relatively small, not higher than 10 mm. In order to predict the infiltrated length in the whole parameter range a nonlinear surface, defined in Eq. 2 was fitted on the points.

$$L = Apt^B \qquad \text{(Eq. 2)}$$

Where p is the infiltration pressure in kPa, t is the infiltration time in s, A and B are fitting parameters with the values of 0.10972 and 0.49727 respectively. With the equation of the surface the infiltrated length can be easily determined within the measured ranges and in a reverse case, when a desired infiltration length is given, one can evaluate satisfying pressure and time parameter pairs. To investigate the effect of time and pressure independently sections were made along constant time (Fig 5a) and constant pressure values (Fig. 5b). The

pressure has linear effect on the infiltration length (Fig. 5a). Lines with $L_{|p=0}=0$ intersection were fitted on the measured points (Eq. 3). The fitting parameters are listed in Table 1.

$$L = ap_{|t,T=const.} \quad \text{(Eq. 3)}$$

There are two limits for the infiltration pressure on the production side: (i) the threshold pressure as lower limit and (ii) the crush strength of the hollow spheres as upper limit. The latter is usually given by the providers (45 000 kPa for SL300 grade) and is much higher than the usual infiltration pressures. The threshold pressure (p_{th}) can be calculated according to Bárczy and Kaptay [1], Trumble [19] or Rohatgi [22] for example. Concerning Kaptay's model the expression of the threshold pressure can be written as:

$$p_{th} = \max\left[\frac{\sigma_{l/g}}{R}\left[\frac{\sqrt{3}}{3}\frac{\pi}{\varepsilon_s}\left(\frac{h}{R}-1-\cos\Theta\right)+\frac{\sqrt{3}}{3}\frac{\pi}{\varepsilon_s}\left(\frac{h}{R}-2.63-\cos\Theta\right)\right]\right] \quad \text{(Eq. 4)}$$

where

$$\varepsilon_s = 1 - C\left|2\frac{h}{R}-\left(\frac{h}{R}\right)^2\right| - C\left|2\left(\frac{h}{R}-1.63\right)-\left(\frac{h}{R}-1.63\right)^2\right| \quad \text{(Eq. 5)}$$

in Eq. 4 and 5 p_{th} is the threshold pressure, R is the average diameter of the hollow spheres, $\sigma_{l/g}$ is the interfacial energy between the infiltrating liquid (the Al melt) and the surrounding gas (air), ε_s is the ratio of the liquid/gas interface and the total cross-section area, C is a coefficient for the volume fraction of the hollow spheres (C=0.822 for R-CPES model [2]), h is the vertical position of the infiltrating melt front, θ is the wetting angle between the material of the hollow spheres and the infiltrating material. Trumble defined a hydraulical radius, which depended on the shape and the volume fraction of the fillers and calculated the threshold pressure by using the Young-Laplace equation [19].

$$p_{th} = \frac{2\sigma_{l/g}\cos\Theta}{r_h} = \frac{12\lambda(1-V_{mb})\sigma_{l/g}\cos\Theta}{V_{mb}2R} \quad \text{(Eq. 6)}$$

where r_h is the hydraulical radius, V_{mb} is the volume fraction of the hollow spheres (64 vol%) and λ is a shape factor (λ=1 for spheres). Similar was done by Rohatgi et al., but they defined an effective distance between the hollow spheres. The base equation was the Young-Laplace equation again [22].

$$p_{th} = \frac{2\sigma_{l/g}\cos\Theta}{r_e} = \frac{2\sigma_{l/g}\cos\Theta V_{mb}}{2R(1-V_{mb})} \qquad \text{(Eq. 7)}$$

where r_e stands for the effective distance. The required threshold pressures were calculated by the interpreted models with typical parameters (R=150 μm, $\sigma_{l/g}=860\cdot10^{-3}$ Nm^{-1}, this corresponds to oxidized surface for aluminium alloys, Θ=140…160 and 160°). In this range the models developed by Trumble and by Rohatgi et al. predict somewhat smaller threshold pressures than the Kaptay model as it is shown in Fig. 5a by vertical dashed lines (for Θ=150°). In summary the threshold models gave rather different results - but in the same magnitude - due to their different approaches. The Kaptay model is fully theoretical and works with the more or less well defined (R-CPES) geometry, while the Trumble and Rohatgi models are based on semi experimental approaches and affected by the definition of hydraulic and effective radius. The hydraulic radius is defined as the ratio of pore volume and pore surface area and although it is dimensionally correct, it has no fundamental basis on correctly describing even the average pore size [19]. The effective radius is defined as inter particle distance and calculated by multiplying the average hollow sphere diameter by the ratio of the matrix and hollow sphere volume fraction respectively [22]; that has also not so obvious relationship to the real geometry of the pores. Considering the problems above the best way to determine the threshold pressure for a given system is measurement. In order to validate the models preliminary measurements were done with constant infiltration time (5 s) and relatively small pressure steps (25 kPa increments from 50 to 250 kPa). The results were plotted in Fig. 5a by blue ○ marks. In the case of the investigated material pair the Kaptay model seems to give closest prediction.

In Fig. 5b the effect of infiltration time can be investigated at constant infiltration pressures. As it is presented in the Introduction the professional literature is rather divided in the question: what is the correct form of the curves in short infiltration time region? The theoretical predictions suggest that the curve has square-root shape from the very beginning of the infiltration, while the published measurements show an exponential shape tail in the first period of the diagram [30-35]. This period is generally called 'incubation time', and usually related to the pressure built up in the infiltration chamber or to the existence of some kind of chemical exchange reaction between the reinforcement and the matrix in the case of reactive material pairs. Considering this dual problem the most proper way is to measure the infiltration characteristics in the short time region as it is done and presented here in Fig. 5b. The two mentioned problems were eliminated by the equipment (designed for quick pressure build-up) and the applied non-reactive material pair (AlSi12 – Al_2O_3 based ceramic) respectively. The plotted average results and their scatter obey the following equation (Eq. 8).

$$L = bt^{c}_{|p,T=const.} \qquad \text{(Eq. 8)}$$

where b and c parameters were determined by non-linear curve fitting, and their values are listed in Table 2. The exponent c≈0.5 and the length – time curves are near to the square root function in the short (1…9 s) infiltration time range. This behaviour confirms the theoretical predictions. In summary the above analysed results can be applied to determine the required infiltration parameter values for a part with given geometrical dimensions. Moreover they can lead to the successful implementation of hot chamber low pressure mould casting as time and cost effective MMSF production method.

3.2 Microstructure

Beside the production of the desired part shape its quality should be assessed by light microscopy observations on polished specimens (see section 2.2). The typical micrographs are presented in Fig. 6. Along the vertical and horizontal axis of Fig. 6 the infiltration pressure

and time is increasing respectively. The light phase is the AlSi12 matrix and the black spots represent the hollow spheres and the (usually unwanted) voids between them. Dark circles with light phase inside are hollow spheres into which the matrix could penetrate due to breakage. In ideal case the hollow spheres can be clearly separated in the figure and the amount of unwanted voids and fulfilled spheres tends to zero. Two trends can be observed in the diagram at first glance: the quality of infiltration is better in the case of higher infiltration pressure and longer infiltration time. However the demands for both can raise serious requirements on the equipment side of the process: higher pressures require more robust structure and high performance sealings, while the longer time needs proper matrix addition and tool tempering in order to avoid the freezing of the matrix (and to lower the amount of unwanted voids).

Another important question is the interface layer between the hollow spheres and matrix. This interface layer is the responsible for the load transfer from the matrix to the reinforcing hollow spheres. If the interface layer is too brittle and thick (due to some kind of chemical reaction between the constituents), or too weak (due to insufficient bonding) then the load transfer cannot be effective enough. Therefore the interface layers should be precisely investigated as it is done in the case of experimentally produced MMSFs [39, 40]. The typical SEM micrograph and chemical composition along the indicated line are shown in Fig. 7. The interface layer is relatively thin and the outer wall of the hollow sphere is well defined and unharmed, this indicates limited chemical reaction (see Eq. 1). Precipitations cannot be observed in the micrograph, or in the chemical composition. The transition between the composition of the matrix and the wall material is sharp but smooth. In summary all of the above mentioned observations suggest optimal interface layer. However the load transfer capability can be measured and qualified only by mechanical tests.

3.3 Density and mechanical properties

The ideal density of the produced MMSFs can be estimated by the rule of mixtures (Eq. 9).

$$\rho_{MMSF} = V\rho_{HS} + (1-V)\rho_{AlSi12} \quad \text{(Eq. 9)}$$

where ρ_{MMSF} is the estimated optimal density of the MMSF, ρ_{AlSi12} is the density of the AlSi12 matrix (2.680 g/cm^3 [54]), ρ_{HS} is the density of the reinforcing hollow spheres (0.692 g/cm^3) and V is the volume fraction of the reinforcement (64 vol%). With the above values the ideal density of the MMSF is 1.408 g/cm^3, respectively. The densities of the produced MMSF samples were determined by Archimedes' method and plotted against the infiltration pressure and time in Fig. 8. The density of the specimens remained below the ideal density (this indicates imperfect infiltration and unwanted void content) but converges to the ideal with the infiltration pressure. Higher densities were also measured in some cases that indicate infiltrated hollow spheres as it can be observed near to the right-top corner in Fig. 6.

The main loading mode of the MMSFs is the compression. The tests were done according to DIN50134 standard [53]. The engineering stress – engineering strain curves were registered and subsequently analysed as it is shown in Fig. 9. The stress –strain curves were divided into typical sections [58, 59]. In the first section (from point A to B) the specimens were deformed elastically only. In this section the hollow spheres remained unharmed; there were no cracks at all. The slope of the first part is defined as structural stiffness (S (MPa), dashed black line in Fig. 9) [53]. The stiffness is one of the characterising properties of the MMSFs. In the vicinity of point A the deviation from the fitted dashed line can be caused by the internal sliding of the material or by the springs and the natural movement of the sliding parts of the tool. In the second section from point B to C the plastic deformation of the matrix began. The load transfer between the matrix and the hollow spheres increased to its maximum, but the spheres remained still unharmed. At the end of this section at point C the stress reached the compressive strength (σ_c (MPa)) at the fracture strain (ε_c (%)). These parameters are also important characterising properties, because they show the load bearing capacity of the

MMSFs directly. At point C the first crack appeared in the specimen. This first rupture was very thin and only one row of the hollow spheres was cracked. The plane of the crack closed 45° with the horizontal direction, because in the case of uniaxial loading the maximum shear load appears in this direction (see inset micrograph). The stress suddenly dropped to point D due to the reduced load bearing capacity caused by the fracture of the hollow spheres and the movement of the recently formed specimen halves. From point D to E the fracture band expanded and the crack became thicker and thicker. This deformation phenomenon consumed significant strain and mechanical energy due to the fracture of the ceramic hollow spheres and due to the plastic deformation of the matrix. The absorbed mechanical energy (W (J/m^3)) is the fourth main characterising parameter of the MMSFs, as it indicates the damping and protecting capability of the MMSFs against a blast, collision or simple vibration. This part – also called plateau region – absorbs lot of energy, because it is relatively long and has high stress value. It is worth to note that the shape of the diagrams after point D can be ascending or constant (usually ascending because the densifying material needs higher force to be deformed). It may contain larger drops or local maximums due to secondary cracks. The process ended at 50% engineering strain when the densification was almost completed (see inset micrograph) and the test stopped (point E in Fig. 9).

Generally the structural stiffness, compressive strength, fracture strain and the area under the whole curve – which gives the total absorbed energy during the test – is used to characterise the compressive behaviour of the MMSFs. Therefore the effect of the infiltration pressure and time on these properties were investigated and pursued in Fig. 10. In Fig. 10a the average structural stiffness is shown; its alternation is similar to the trend average density. The unwanted voids worsen the overall cohesion in the composite. In the vicinity of the voids the interface layer between the hollow spheres and the matrix is lacking, therefore there is no load transfer and the matrix material can deform freely. As the amount of voids decreased by the

increasing of the infiltration pressure and time, the structural stiffness became higher and higher. As the density was increased by the infiltration the pressure, it has linear effect on the stiffness too. Fig. 10b shows the alternation of the compressive strength above the infiltration pressure –time plane. The compressive strength again follows the trends of the density, similar to the structural stiffness, but with higher scatter. The pressure has – again – linear effect on the strength, but the influence of time has no typical trend. In Fig. 10c the fracture strains are shown. The strain increasing proportionally with the pressure and the time has almost no effect on the fracture strain. Finally, the absorbed mechanical energies are plotted in Fig. 10d. The absorbed energy varies similarly to the compressive strength, because it has direct effect on the area under the stress – strain curves [58, 59]. In order to analyse the potential anisotropy of the infiltrated pieces, reference measurements were done perpendicular to the infiltration direction of the infiltrated specimens. However due to the almost perfect spherical shape of the reinforcement particles and because of the almost complete infiltration no significant deviations can be measured. The monitored results were within the scatter ranges of the specimens with coinciding longitudinal axis. In summary the mechanical properties alter linearly with the infiltration pressure and increase with the infiltration time. These outstanding mechanical properties (among metallic foams) and the possibility of the application of injection mould casting as production method makes the MMSFs good choice for low weight structural parts with high mechanical absorbtion capability.

4. Conclusions

From the investigations and results above the following conclusions can be drawn:

- An equipment for the measurement of the infiltrated length as the function of infiltration pressure and time has been successfully developed. The equipment is ideal to operate in the short infiltration time range (<10 s).

- In the short infiltration time range the infiltrated length is proportional to the infiltration pressure and square-root function of time.
- The infiltration method has an upper and a lower pressure value as process limits. The upper limit connects to the crush strength of the hollow spheres. The lower limit is the threshold pressure, for this best estimation was given by Kaptay and confirmed by the measurements.
- The density and the mechanical properties alters linearly with the infiltration pressure and increase with the infiltration time. The specimens were isotropic, no anisotropy was observed in the monitored mechanical properties.
- The successful application of short time pressure infiltration technique and the measured outstanding mechanical properties confirm the possible application of low pressure hot chamber injection mould casting as economic and effective production method of MMSFs.

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Figure captions

Figure 1. Schematic view of the equipment (longitudinal section)
Figure 2. Arrangement of the measurement setup
Figure 3. Pressure build-up transient in the infiltration equipment
Figure 4. The measured infiltrated lengths (black dots) above the pressure - time plane and the fitted surface (Eq. 2)
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Figure 9. Typical stress –strain diagram of MMSFs (p= 750 kPa, t= 5 s)
Figure 10. Structural stiffness (a), compressive strength (b), fracture strain (c) and absorbed energy (d) of the infiltrated MMSFs

Table captions

Table 1. Fitting parameters for infiltrated length as the function of infiltration pressure
Table 2. Fitting parameters for infiltrated length as the function of infiltration time