

Mechanical properties of iron hollow sphere reinforced metal matrix syntactic foams

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Abstract. Metal matrix syntactic foams (MMSFs) were produced by low-pressure inert gas infiltration technique. Matrixes of the produced syntactic foams were Al99.5, AlSi12, AlMgSi1 and AlCu5 respectively, and each was reinforced by pure Fe based hollow. The produced blocks were investigated by optical and scanning electron microscopy. The microstructural investigations revealed proper infiltration with small amount of unwanted voids and an effective and thin interface layer between the matrix materials and the reinforcing spheres. The produced MMSFs were also tested under quasi-static compression loading to get characteristic mechanical properties. The test results showed that the MMSFs with iron spheres have outstanding mechanical properties compared to 'conventional' foams.

Introduction

Metal matrix syntactic foams (MMSFs) are special foams consisting of a metallic matrix and a set of hollow spheres. The matrix material is usually a light-weight alloy (aluminum in most cases), but Fe [1] and Zn [2] are also applied for experiments. Most commonly the hollow sphere inclusions are built up from oxide ceramics (Al₂O₃ and/or SiO₂), but pure iron hollow spheres are also available on the market (Globomet grade spheres from Hollomet GmbH). MMSFs have been investigated in many aspects, but the publications commonly refer to the microstructural and mechanical testing besides the production methods. Regarding the microstructure, the most important part of the MMSFs are the interface layers between the matrix material and the reinforcing hollow spheres, because this layer is the responsible for proper load transfer between the constituents. Due to this distinguished role, the interface layer and the load transfer mechanisms were studied in details [3-6]. The mechanical properties of the MMSFs have been studied in compressive loading mode, most commonly. They have excellent mechanical damping and energy absorbing capability that makes them beneficial as collision dampers or as basement of (vibrating) machines, machine tools. Besides the measurements [7], the compressive properties have been predicted [8] too and the compressive curves were analyzed according to the failure mechanisms of the MMSFs [9]. The tribological properties of MMSFs are also in the focus of interest [10-12]. Dry sliding properties as well as slurry erosive wear and wear in corrosive medium was investigated [10-12]. Due to the promising expected properties the main aim of our paper is to give numerical results about the characteristic properties under compressive loading.

Materials and experimental methods

Production of the composites

Throughout our experiments, pure aluminum (Al99.5) and various aluminum alloys; AlSi12, AlMgSi1 and AlCu5 were used as matrix materials for the synthesis of MMSFs. For all the MMSFs, Globomet grade iron hollow spheres were used as reinforcing material, manufactured by Hollomet GmbH. The reinforcement material was studied in detail, because that should be the main load-bearing component. The diameters of 700 hollow spheres were measured on a stereomicroscope (Fig. 1a), the average diameter of the hollow spheres was found to be 1.9 mm.

The density of the hollow spheres was $\rho=0.4 \text{ gcm}^{-3}$. Proper surface adhesion of the hollow spheres is crucial for the production of high quality composites; therefore the surface of the hollow spheres was studied by scanning electron microscopy (SEM). The SEM image (Fig. 1b) clearly shows that the wall of the spheres is porous, which, due to a high surface area, results in excellent contact between the matrix material and the hollow spheres.

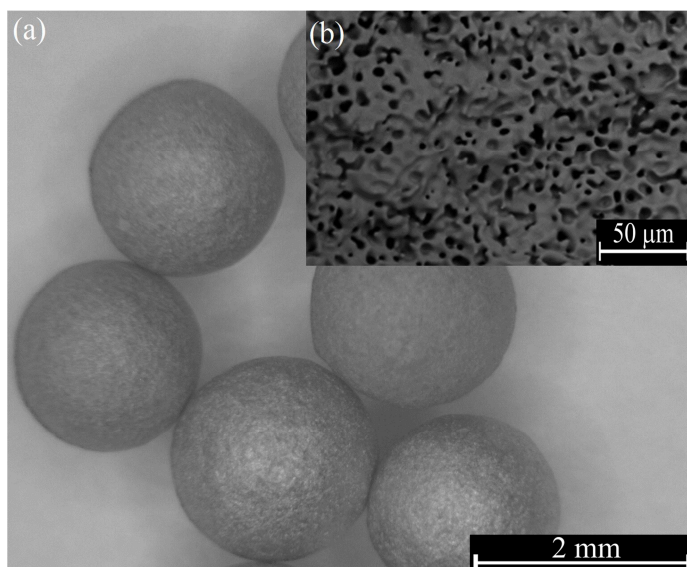


Figure 1. (a) Stereomicroscopic image of the iron hollow spheres (b) SEM image about the wall of an iron hollow sphere

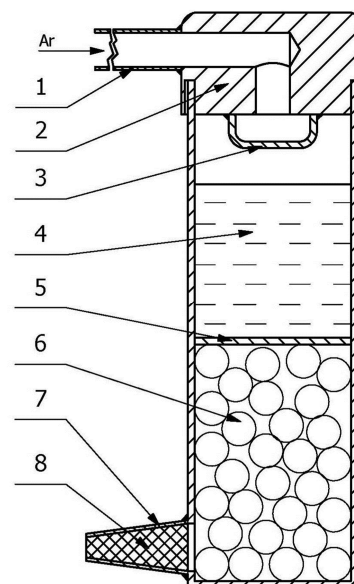


Figure 2. Sketch of the infiltration chamber: 1. gas inlet, 2. closing hatch, 3. gas deflector plate, 4. metal, 5. mesh, 6. hollow spheres, 7. exhaust 8. Al_2O_3 mat

For the synthesis of the low-pressure infiltration was used. The casting mold used throughout the production is shown in Fig. 1. For a casting mold, we utilized a hollow section piece that was closed at the bottom by welding. In addition, an exhaust pipe was welded to the side of the hollow section, which was filled with Al_2O_3 mat in order to prevent the spillage of molten aluminum upon the application of infiltration pressure. The inner side of the mold was coated with graphite spray, and was filled with the iron hollow spheres approximately to the half. The hollow spheres were closely packed by the application of mechanical vibration, and were locked in place by a metal mesh. The casting mold was pre-heated to $\sim 300^\circ\text{C}$, and was filled with the molten matrix material at $\sim 700^\circ\text{C}$. Then the mold was sealed and inert gas (Ar) was applied for 30 s at 4 bar pressure. The apparatus was then externally cooled by water. The specimens made with precipitation hardenable alloy matrix materials (AlMgSi1 and AlCu5) were investigated in two different heat treated conditions. First solution heat treatment (specimens marked with O) was applied. According ASTM standard 1 hour of heat treatment at 520°C , followed by rapid cooling in water. The second was precipitation hardening (specimens marked with T6), whereby additional heating after the solution heat treatment was applied at 170°C for 14 hour. The specimens were labeled as follows: matrix material-reinforcement (heat treatment if applied). For example, the name AlCu5-GM(O) corresponds to MMSF made by AlCu5 matrix material and a Globomet (GM) reinforcement that was subjected to solution heat treatment (O).

Investigation techniques

Density is an important property of metal foams; it was determined by Archimedes method on the compressive test specimens. The density of the metal foam was also estimated using calculations ($\rho_{\text{calculated}}$) by using the average 1.9 mm diameter, 0.4 gcm^{-3} density and $\sim 65\%$ space filling for the reinforcement spheres. The results of the density measurements are shown in Table 1.

The quality of the infiltration was examined by metallographic techniques. For this purpose, a representative piece from the produced sample was cut out and mounted in synthetic resin. The steps for the metallographic sample preparation were as follows: wet grinding with SiC P80, 160, 320, 600, 1200 and 2500 grade grinding papers. Subsequently, automatic polishing steps were

applied first with a 3 μm grain size diamond suspension for 40 minutes, followed by polishing with a 1 μm grain size diamond suspension for 30 minutes, and finally polishing with a 0.05 μm grain size $\gamma\text{-Al}_2\text{O}_3$ suspension for 20 minutes with 15 N force for all the polishing steps respectively.

Molten aluminum is a highly reactive material, and can dissolve the walls of the iron spheres during infiltration; therefore an interface layer was created between the two materials. The thickness of this layer was measured by a Phillips XL-30 scanning electron microscope and linear energy dispersive X-ray spectroscopy (line EDS). For the EDS analysis an EDAX Genesis detector was used with 20 kV accelerating voltage, 35 μs detector amplification time and 15000 ms excitation time for each point along the line respectively. The electron beam spotsize was only approximately 50 nm, therefore it was neglected in the analysis of the transition zones.

Generally, the primary loading mode on MMSFs is compression. Therefore compression tests were done by a hydraulic MTS 810 type universal material testing machine. For the compression tests, cylindrical test specimens of $D=14$ mm diameter were machined, with height to diameter (H/D) ratios of H/D=1, 1.5 and 2. In total, 18 pieces per composite were compressed (6 per slab). Double lubrication with Loctite anti-size lubricant was applied to the specimens. The test objects were compressed with a constant velocity depending on the H/D ratios. For H/D=1, 1.5 and 2, the compressing speeds were 0.1 mms^{-1} , 0.15 mms^{-1} and 0.2 mms^{-1} , respectively. The measured force – height reduction curves were corrected for alignment error, than the plots were averaged by Origin software, and were converted to stress-strain plots. The evaluation of the diagrams were performed in accordance with the ruling DIN 50134 standards. The monitored properties were the compressive strength, yield strength, plateau strength, structural stiffness, and absorbed energy values.

Results and discussion

The results of the density measurements are shown in Table 1. It is clear that the measured density values were always very close to the calculated values. The small difference between them was caused most probably by improper infiltration. In Fig. 3a the white arrow shows a hollow sphere with damaged wall and therefore filled by the matrix material, the black arrow shows the lack of matrix material: an unwanted porosity between the hollow spheres.

Table 1. Theoretical and measured density values of the MMSFs

Sample MMSF densities (gcm^{-3})	Al99.5-GM	AlSi12-GM	AlMgSi1-GM	AlCu5-GM
Calculated ($\rho_{\text{calculated}}$)	1.62	1.60	1.62	1.76
Measured (ρ_{measured})	1.41	1.42	1.60	1.72

As Fig. 3.b shows the spheres had not significant impact on the microstructure of the matrix material. It has polygonal grains, but near to the spheres the number of silicon rich and magnesium rich (in case of AlSi12 and AlMgSi1 matrix respectively) precipitations had increased. The chemical composition along a line from the matrix was measured perpendicular to the wall of a sphere. For example in Fig. 4, the oxygen content clearly increased in the wall of the spheres, because of the slight oxidation during the preheating process. In the right side of the image the two discursive lines shows the transition layer thickness. This value varies between 4-11 μm for all the samples respectively. In the case of the AlCu5 and AlMgSi matrices a local enrichment of Cu and Mg was detected alongside the spheres. In summary coherent, thin transition layers are present between the matrix and reinforcement material in all samples.

Regarding the compression tests, the more intensively deformable syntactic foams with iron hollow spheres have different shaped compressive curves, compared to more rigid syntactic foams with ceramic hollow spheres. There was no initial stress peak, so they do not have a pronounced compressive strength: the curves constantly step over to the plateau region, because the metal spheres have higher plastic deformation ability, in contrast to the ceramic spheres' rigid behavior. Regarding the characteristic mechanical properties, the structural stiffness (S) show higher values when the H/D ratio is higher (Fig. 5). As it is expected, the precipitation hardened alloys showed higher structural stiffness values than the ones with solution heat treatment. The AlSi12-GM(O) and AlCu5-GM(O) specimens showed higher stiffness values than the specimens of Al99.5(O) and

AlMgSi1(O) matrix. The structural stiffness values varied between 1000-7000 MPa. Higher aspect ratios caused also resulted in higher scatters, because there are infiltration errors in the syntactic foams, bigger specimens causing higher probability of serious errors.

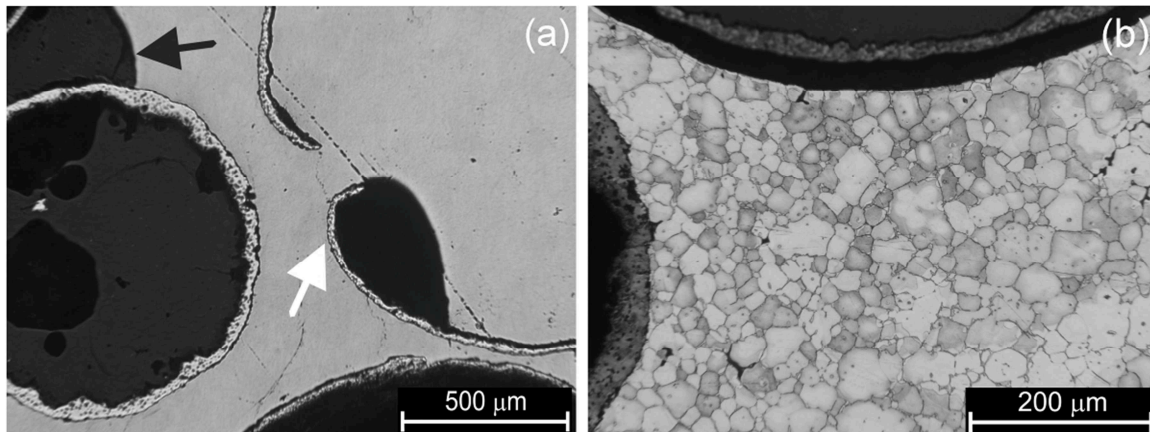


Figure 3. Light microscope images of MMSFs (a) Al99.5-GM(O), polished sample and (b) AlCu5-GM(O), etched sample

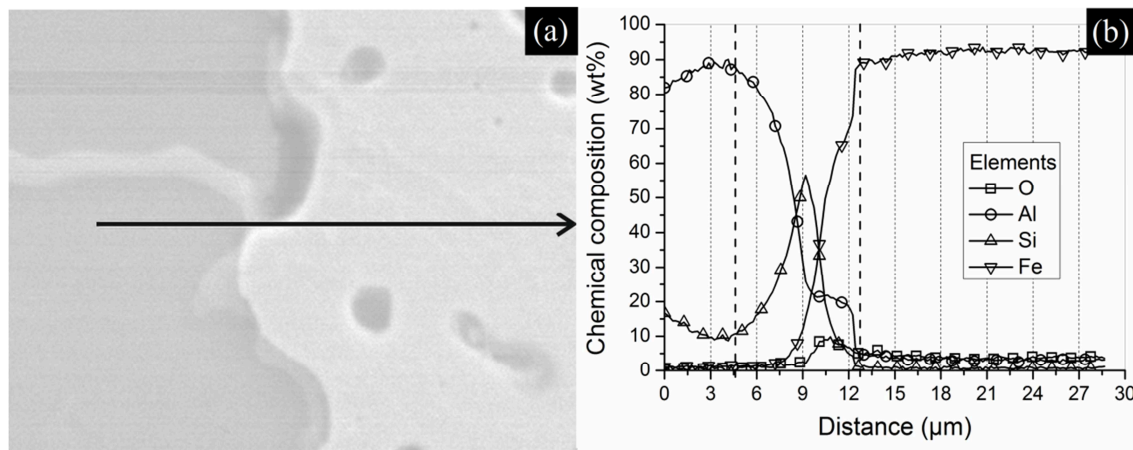


Figure 4. (a) SEM image of a polished AlSi12-GM(O) sample with the line for the EDS measurement, (b) plot of the EDS measurement along the black arrow

In the yield strength (σ_Y) values (Fig. 6) no definitive relation could be found with the aspect ratio; the values within the sample types were within the scatter for each H/D ratio. The T6 heat treated specimens showed significantly higher yield strength values, approximately by 30%, than the specimens with solution heat treatment. Compared to the samples with pure aluminum matrix (~ 20 MPa) about four times higher yield strength can be achieved with heat treated AlCu5 matrix (~ 80 MPa). The plateau strength ($\sigma_{plt.}$) values (Fig. 7) had a slight dependence on the H/D ratio, in the case of higher specimen the values were higher (except the AlMgSi1 (O) specimen). The matrix strength also had influence on $\sigma_{plt.}$ and σ_Y , with higher matrix strength, causing higher plateau strength values. The difference can be significant, for example the plateau strength of Al99.5 matrix (~ 40 MPa) can be tripled by AlCu5 matrix (~ 120 MPa). As it is expected, the T6 heat treated specimens also had higher plateau strength values than the solution heat treated ones. The mechanical energy absorbed up to 1% plastic deformation ($W_{1\%}$) is the limit value of the maximum energy that the MMSF can withhold without macroscopic fractures. This energy was found to be inversely proportional to the aspect ratio (Fig. 8), but it should be noted that energy values were mostly in the scatter range. The effect of the matrix material and heat treatment on the $W_{1\%}$ and $W_{50\%}$ (overall mechanical energy absorbed up to 50% plastic deformation, Fig. 9) values showed the same trends like the yield strength, plateau strength and structural stiffness respectively. The $W_{50\%}$ values varied within wide ranges from $\sim 2000 \text{ MJm}^{-3}$ in the case of Al99.5 matrix up to $\sim 6000 \text{ MJm}^{-3}$ in the case of AlCu5 matrix with T6 heat treatment. During the compressive tests different failure mechanisms can be observed in the case of iron spheres reinforced compared to the MMSFs with ceramic spheres, there was no crack plane and the specimens had plastic deformation

in all cases. This observation is supported by the metallographic examinations of compressed specimens in Fig. 10. A high level of plastic deformation of the spheres and matrix material can be observed. The spheres crashed (black arrow) because of the pressure, and the matrix tried to follow the deformation of the spheres (white arrow).

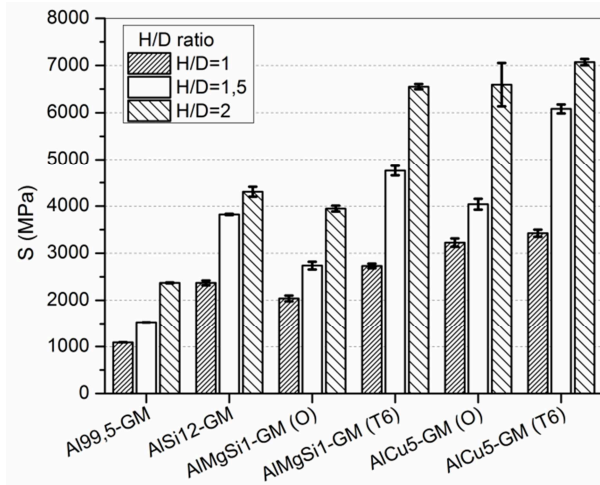


Figure 5. The structural stiffness values

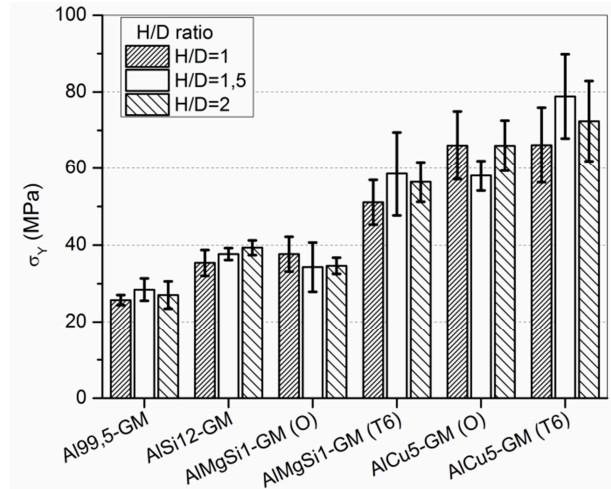


Figure 6. The yield strength values

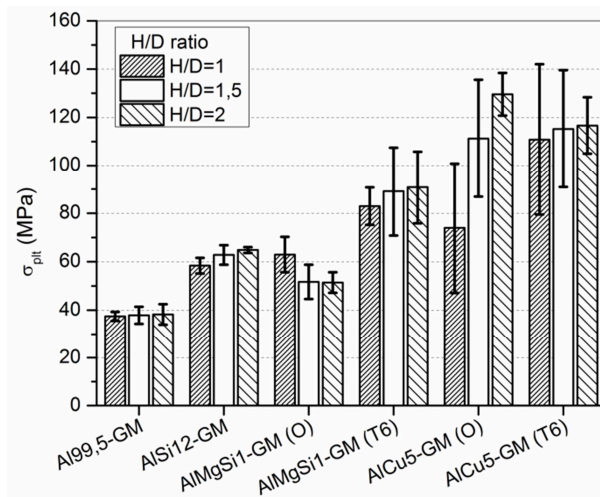


Figure 7. Plateau strength values of the composites

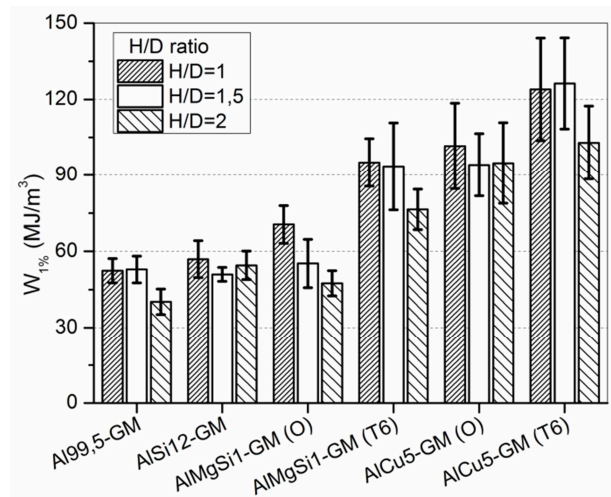


Figure 8. Absorbed energy values (up to 1% plastic deformation)

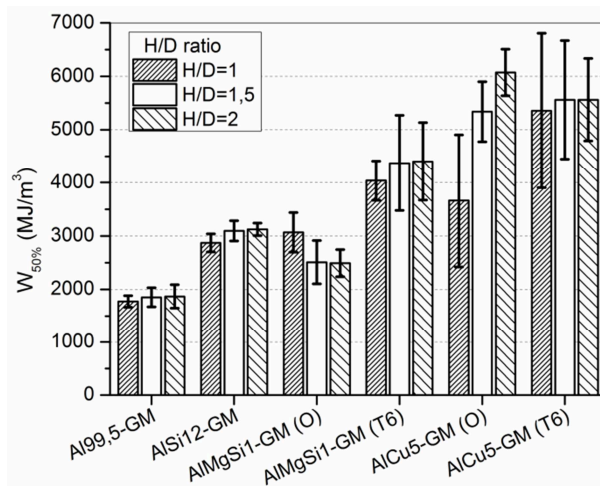


Figure 9. Mechanical energy absorbed up to 50% plastic deformation

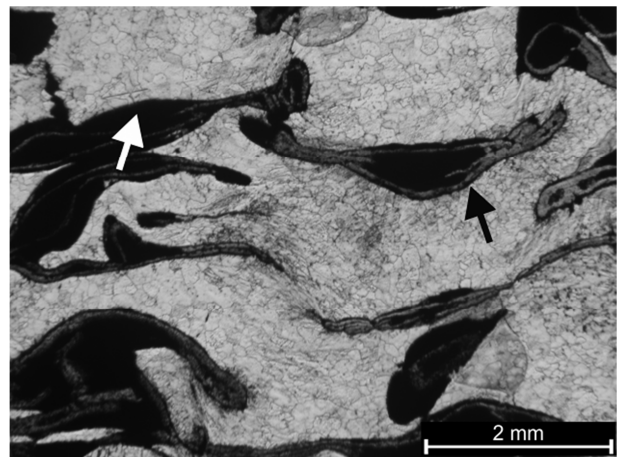


Figure 10. Microscopic image of compressed AlCu5-GM(O) polished and etched specimen

Summary

From the above detailed experiments and investigations the following conclusions can be drawn:

1. Low-pressure infiltration proved to be an effective method for the production of metallic hollow sphere reinforced AMSFs. The produced AMSFs have low unwanted void content, their densities are close to the theoretical ones with small scatter, and the whole procedure is well repeatable.
2. The microstructural investigations showed good cohesion between the matrix material and the hollow spheres that is also confirmed by the presence of a 4-11 μm thick interface layer.
3. The quasi-static compressive tests confirmed outstanding mechanical properties. The structural stiffness and the whole absorbed energy values were in linear relationship with the aspect ratio, while the plateau and yield strength were independent from it. The mechanical energies absorbed up to 1% deformation was inversely proportional to the aspect ratio.

Compared to the data available in the professional literature; the produced AMSFs have significantly better mechanical properties than conventional metallic foams.

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