Manufacturing and investigation of aluminium matrix bimodal metal foams

B Leveles$^{1,2*}$, A Kemény$^{1,2}$, I N Orbulov$^{1,2}$

$^1$Budapest University of Technology and Economics, Faculty of Mechanical Engineering, Department of Materials Science and Technology, Budapest, Hungary
$^2$MTA–BME Composite Metal Foams Research Group, Budapest, Hungary

E-mail: borbala.leveles@edu.bme.hu

Abstract. Metal matrix foams (MMFs) are mostly used in the automotive industry due to their high specific energy absorbing capacity and relatively low weight. During this research, bimodal metal matrix foams were produced, where the filler was ceramic hollow spheres (CHSs) made of high-purity alumina with the nominal diameters of $\Omega_{d_1} = 7$ mm and $\Omega_{d_2} = 2.4$ mm. The hollow spheres of different sizes were used in 1:1, 2:1 and 4:1 volume ratio, which after mixing can be considered as uniformly distributed. Al99.5 was infiltrated between the CHSs with low pressure to create a cellular material. The manufacturing parameters have a significant influence on the properties of the finished metal matrix foam. Different preheating temperatures, melt temperatures, infiltrating pressures and time was applied to achieve proper wetting and filling. Samples were evaluated based on macro images and microscopic photographs.

1. Introduction

Cellular materials in nature appeared to increase the stability of a more massive structure. Preferably, the selection was made in nature to reduce weight while maintaining or increasing strength. Metal matrix foams are mostly used in the luxury automotive, aerospace, and marine industries because of their high energy absorbing capacity and relatively low weight.

In the literature, MMFs are sorted in the class of hybrids as composites, sandwich structures and lattice materials. MMFs, in which the cells are formed by a second phase, their distribution within the material can be considered homogeneous, and their size is close to uniform, are called metal matrix syntactic foams (MMSFs). Usually, the matrix material is a lightweight metal, and the filler material is a hollow sphere or expanded material that allows the presence of air [1].

Various manufacturing technologies can produce MMFs. Three of these are widespread: powder metallurgy, stir casting and infiltration [2–4]. The most common procedure is infiltration, which may be gravity-, or pressure-assisted, depending on the wetting ratio [5,6]. In the latter technology, the matrix material is poured between the hollow spheres in a molten state, applying pressure to increase wetting. By changing the infiltration pressure, time, and temperature parameters, the quality of the given syntactic foam can be adjusted. Steel infiltration was shown in Yang and his colleagues’ research, where many unsuccessful samples were created due to low preheat temperature [7–12].

Bimodal materials exhibit two distinct characteristics/distributions in one property and are nearly the same in all of the other. By bimodal metal matrix foams, the literature means MMSFs filled with CHSs of two different diameters. The proportion of CHSs of different diameters may be different from 50-50 wt. % as shown in Figure 1. [13–15].
Figure 1. Bimodal distribution with two different filler ratio

Tao et al. investigated aluminium matrix syntactic foams filled with bimodal ceramic microspheres. In their research, the fabricated bimodal metal matrix foams had up to 10% higher porosity and 8% higher onset strain of densification compared to MMSFs. 6082 Al alloy was used with fine (75-125 µm) and coarse (250-500 µm) ceramic microsphere filler [14].

Orbulov et al. published a paper on the compressive characteristics of bimodal aluminium syntactic foams. AlSi12 alloy was used with smaller (150 µm) and larger (1425 µm) Globocer CHSs. The mechanical properties of the bimodal samples laid between the pure small and pure large CHS filled MMSFs, and the compressive strength could be reliably estimated by the rule of mixtures [16].

The goal of this research is the manufacture of bimodal metal foams to create a biphasic material with reduced density and higher specific compressive strength properties compared to homogeneous solid continuum material. Currently, such materials can only be manufactured under laboratory conditions without accurate knowledge of mixing methods and casting parameters. Once all condition factors become known, bimodal metal foam casting can also be industrialised with any form of shaping capability. This paper aims to cast and study the microstructure of bimodal metal foams to qualify different mixing ratios and determine their manufacturability.

2. Materials and methods

In MMSFs the matrix material absorbs and distributes the load, and through the boundary layer, the hollow spheres are kept in place and bear a part of the load. Al99.5 (1050 Al) was used as matrix material in this research to characterise the properties of bimodal metal matrix foams.

The second phase most commonly used for MMSFs are porous ceramic or hollow metal spheres. The fillers of the produced bimodal metal foams were the same in their base material and density, but two different average diameters. The CHSs are made from Al2O3 C795 according to EN 60672-3:1999 with nominal diameters of Ød1=7 mm and Ød2=2.4 mm and obtained from Hollomet GmbH. The specific properties of these CHSs are detailed in a previous research [17].

2.1. Mixing

During this research, bimodal metal foams were made with two different diameter ceramic hollow spheres as filler. The peculiarity of bimodal metal matrix foams lies in the mixing ratio of the two sized hollow spheres. First, the packing fractions of different mixing ratios of the spheres were calculated...
from theoretical equations [18] which has not led to total success, because the produced foams had lower packing ratios. During the producing, three mixing ratios were tested, with the ratio of large to small hollow spheres of (1:1) 50-50%, (2:1) 67-33% and (4:1) 80-20% distribution. Theoretical space fills do not differ significantly, 72.8%, 71.9% and 69.7% respectively, therefore macrostructure studies have determined the optimum and substantiality of the mixing ratios.

2.2. Manufacturing of bimodal foams
Two hollow spheres of different diameters were measured in volume ratio (and also in weight ratio due to the equal density) to the intended mixing ratio. The hollow spheres were mixed in a larger container until it was approximately uniformly distributed. After mixing, the CHSs were poured in a pre-welded and drilled, blocked hollow section into which an Al$_2$O$_3$ layer was pre-placed to prevent the flow of the molten matrix material and a steel mesh to prevent the spheres from moving, in the arrangement shown in Figure 2.

The ceramic container was preheated to the required high temperature (~100 °C below the melting point of aluminium) and then maintained to uniform heat. During this time, the matrix material was melted in an ING 1IF-10 induction furnace to allow rapid melting. For performing low-pressure infiltration, an insulated pipe was designed and manufactured, which was connected to a high-pressure argon bottle.

![Figure 2. Schematic cross-section drawing of the infiltration layout](image)

![Figure 3. Failed MMSF production (left) and proper casting (right)](image)

During the research, numerous bimodal metal matrix foam infiltration process was performed, some of which have failed until the proper infiltrating parameters were set. Table 1. shows the parameters to proper infiltration process with the used materials. A production was considered defective if the matrix material did not fill the space between the hollow spheres, where the aluminium did not reach the bottom of the crucible and froze too quickly, as shown in Figure 3., or that, due to too low viscosity or too high pressure, it passed completely through the hollow spheres and spilt at the bottom of the container.

The specimens were cut out using an automatic cutting machine, with a diamond particle cutting wheel, which, due to its hardness, was able to cut the block into a smooth plane cube without problems. As a result, from the 56×56×140 mm blocks (Figure 3. right) 56×56×50 mm sized cubic samples were produced.
Table 1. Infiltration parameters for proper production of bimodal MMSFs from Al99.5

| Preheat (°C) | Preheat time (min) | Infiltration pressure (bar) | Infiltration time (s) | Matrix temperature (°C) |
|--------------|--------------------|----------------------------|----------------------|------------------------|
| 550          | 45                 | 3                          | 5                    | 720                    |

The surface of the resulting specimens is essential for sanding and polishing for macro and microstructural studies. Due to the different hardness of the matrix and filler and the wall porosity of the latter, this proved to be a difficult task. Unfortunately, the peeling ceramic pieces constantly caused smaller scratches on the aluminium surface, necessitating the use of a polishing technique.

3. Results and discussion

The average density of the produced bimodal metal matrix foams was 1.62±0.03 g/cm³. It should be noted that besides the hollow spheres at the edges of the blocks more aluminium is present, and the ratio of the damaged hollow spheres also increases the value of the measured density compared to the theoretical density. This measured value is 0.2 g/cm³ higher than the theoretical 1.4 g/cm³, which is a reasonable rate.

3.1. Filler ratio determination

The success of producing MMSFs can first be determined by examining the actual filling rate of the CHSs and comparing it with the theoretical value. When the maximum is approached, the casting is effective. The most obvious method is to inspect the filler distribution on a flat section visually. In the first approach, the number of large and small hollow spheres were evaluated, and in the second approach, the ratio of the coverage area of all hollow spheres to aluminium (thus surface ratio). The first evaluation method has not yet yielded results. The solution for determining the filler ratio of the hollow spheres was software image analysis, for which ImageJ and Adobe Lightroom software were used with several methods. On average, Figure 4. shows a measured ratio of 55–67%, which has a significant deviation from the theoretical value of 69–73%. Identification of the proportion of small and large hollow spheres was tested with more advanced software, with the help of neural networks, but due to the inaccuracies of the scratches and grinding this method did not work flawlessly.

A proper solution to measure the filler ratio is with computer tomography (CT) measurements in 3D, with which the voids can be measured with high accuracy [19]. However, the filler ratio excludes the walls of the CHSs, so their volume must be added to the cumulative void volume. Another disadvantage is that only the perfect spheres can be measured (small–blue, large–green); the ones on the borders and the spheres that are partly filled with matrix are left out (Figure 5.). From this measurement, ~50% of
the bimodal metal matrix foam is made up of voids, so a calculated filler ratio is around 66%. This measurement takes quite some time to evaluate, so easier methods are needed to be developed.

3.2. Wall porosity determination

Based on microstructure studies, a relation was established between the ceramic hollow spheres and the aluminium matrix. In particular, the wall porosity of the spheres is worth investigating as it affects the ability to make cohesion with aluminium, but on the other hand, it gives a chance of failure. The porosity of the hollow spheres can be observed from polished cross sections at high magnification on optical microscopic images.

Similarly to the determination of filler distribution, wall porosity was also measured by image analysis. From the binary image, the percentage coverage of porous portions can be determined as a proportion of areas. On average, a porosity of 40-60% was observed. In Figure 6., metal microscope image made on Olympus PMG-3 illustrates the porosity of the spherical wall more closely.

![Figure 6. An approximate solution to measure the wall porosity](image)

3.3. Boundary layer thickness

With energy dispersive X-ray spectrometry (EDS), the transition between the two materials can be examined more closely. For determining boundary layer thickness, line composition analysis was performed on a Zeiss EVO MA 10 scanning electron microscope (SEM) EDAX Z2 analyser module. The purpose of the measurement was to investigate the relation between the hollow spheres and the aluminium matrix, which is critical for structural integrity. The relation can be adhesive and cohesive. In the case of adhesion bonding, the bond depends only on the geometric properties of the surface of the CHSs. With cohesive relation, there is chemical bonding between the materials. The two phenomena often overlap, resulting in a complex relationship. To examine the boundary layer, linear EDS measurement was applied perpendicular to the transition between the hollow sphere and the matrix material. The points on the line EDS profile show the actual chemical composition in wt.%. Measurements were made on polished cross-sections starting from the wall of a hollow sphere, through the matrix material and closing on the wall of another hollow sphere (Figure 7.).

The measurement data can be used to determine the distribution of the elements in the matrix material and in the hollow spheres. The transition between the lines thus obtained determines the thickness of the boundary layer. The layer thickness was measured to be $12.50 \pm 3.22 \, \mu m$, which shows a strong bond between the CHSs and the Al99.5 matrix material.
4. Conclusions
The following conclusions can be drawn from the measurements:

- CT measurements can result in determining the accurate void volume, but there is a need for simpler methods to approximate the filler ratio of bimodal MMSFs. Cross-section image analysis could be a solution to this problem if a measurement method is created, which decreases the deviation of the results.
- The measured filler ratio was ~66% so a higher ratio can be achieved with the investigated sized bimodal CHSs than with uniformly sized spheres.
- The difference from the theoretical value of the filler ratio can be explained with the high wall porosity of the CHSs, so molten aluminium can enter some spheres by cracking the thinner parts of the walls.
- There should be further investigations on the mixing methods of the different sized spheres to create an even lower density foam and approach closer to the theoretical filler ratio. The imperfect CHSs complicate this purpose.
- The line EDS measurements prove that proper infiltration parameters were defined for Al99.5/Al2O3 bimodal foams because a relatively thick boundary layer was formed between the CHSs and the matrix.

References
[1] Gupta N and Rohatgi P K 2018 4.15 Compr. Compos. Mater. II A 364–85
[2] Neville B P and Rabiei A 2008 Mater. Des. 29 388–96
[3] Manoj et al 2018 Mater. Sci. Eng. A 731 324–30
[4] Ginsztler J et al 2013 Mater. Sci. Test. Informatics VI 729 68–73
[5] Castro G and Nutt S R 2012 Mater. Sci. Eng. A 553 89–95
[6] Dobránszky J et al 2008 Mater. Sci. Test. Informatics IV 589 137–42
[7] Eustathopoulos N et al 2001 Mater. Sci. Eng. A 300 34–40
[8] Rabiei A and O’Neill A T 2005 Mater. Sci. Eng. A 404 159–64
[9] Palmer R A et al 2007 Mater. Sci. Eng. A 464 85–92
[10] Orbulov I N 2013 Mater. Sci. Eng. A 583 11–9
[11] Bárczy T and Kaptay G 2009 Mater. Sci. Forum 473–474 297–302
[12] Castro G and Nutt S R 2012 Mater. Sci. Eng. A 535 274–80
[13] Tao X F and Zhao Y Y 2012 Mater. Sci. Eng. A 549 228–32
[14] Tao X F et al 2009 Mater. Des. 30 2732–6
[15] Brouwers H J H 2006 *Phys. Rev. E - Stat. Nonlinear, Soft Matter Phys.* **74** 1–15
[16] Orbulov I N et al 2019 *Compos. Part A Appl. Sci. Manuf.* **124** 105479
[17] Kemény A and Károly D 2019 *Acta Mater. Transylvanica* **2** 27–31
[18] Brouwers H J H 2013 *Phys. Rev. E - Stat. Nonlinear, Soft Matter Phys.* **87** 1–8
[19] Kozma I et al 2014 *Period. Polytech. Mech. Eng.* **58** 87–91