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The effect of particle shape on mechanical properties of perlite/metal syntactic foam

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Abstract: In a previous work, a natural porous volcanic glass, expanded perlite (EP), was introduced for fabrication of cost-efficient metallic syntactic foams. Perlite metal syntactic foams (P-MSF) were produced by counter gravity infiltration of a packed bed of porous expanded perlite (EP) particles with molten aluminium. In the current study, the effect of EP particles shape on the mechanical and structural properties of foams were investigated. The irregular shape and coarse surface of raw EP particles were turned to near spherical shape with smooth surface using a tumbling process. Foams containing rounded EP particles showed higher mechanical strength at a constant density. The superior mechanical properties of foams with rounded EP particles are likely due their regular positioning and less structural defects.

Keywords: Metal matrix composites; syntactic foam; cell shape; microcomputed tomography; mechanical properties.

1. Introduction

The mechanical response of metallic foams under compression depends largely on structural and microstructural characteristics of the cell walls. It is well established that the mechanical strength of metallic foams increases with the foam density [1]. Since higher density of metallic foams is usually not desirable due to weight constraints, other approaches have been investigated to improve the mechanical properties. Refining the microstructure of cell walls by heat treatment improves the mechanical properties in case of heat treatable materials [2]. The effect of cell size on the mechanical properties of metallic foams has been a controversial issue. In a previous work, we showed that number of cells across the sample diameter does not have a direct effect on the mechanical properties of the foams when a minimum of 7 cells is considered. However, superior microstructural characteristics and geometrical homogeneity of the cell-wall obtained by using smaller EP particles enhance the mechanical properties [3].

Structural irregularity proved to have an important effect on mechanical properties of foams. According to Fazekas et al., irregularity is caused by cells with large size differences or cells with irregular shapes [4]. There are some 2D numerical investigations on the effect of wall geometry, wall thickness distribution, and geometrical imperfections on mechanical properties of cellular solids with low density [4-10]. Fazekas et al. showed that homogeneous cell structures result in higher compressive strength compared to that of perturbed structures [4]. However, Li et al. reported that the foam stiffness increases as the foam becomes more irregular [8]. In a comprehensive 3D numerical analysis, Simone et al. showed that the thickness distribution of cell walls has a significant effect on the stiffness of foams.

According to the authors, there is an optimum ratio between the solid fraction of joints and struts in which the maximum stiffness is achieved [10]. Zargarian et al. reported similar findings [7]. Most of these studies are focused on simplified porous patterns which limits their validity for actual configurations. Moreover, the effect of cell morphology was often disregarded. Cho et al. applied finite element method to investigate the effect of pore shape on mechanical properties of porous metals [11]. Comparing ellipsoidal and spherical pore shapes, they found that models with ellipsoidal pores have inferior mechanical properties compared to models containing spherical pores.

So far, there is a limited number of experimental studies on the effect of pore shape on mechanical properties of metallic foams. It is difficult to control the pore shape and structural homogeneity in manufacturing processes using gas releasing agent or direct blowing. In a recent study, Lehmhus et al. modified the pores shape of aluminium closed cell foams by annealing the TiH₂ blowing agent for 4 h at 500 ^oC. They observed significant increases in mechanical properties of foams produced by heat treated blowing agent due to a high level of structural homogeneity [12]. However, the structural modification can be easily achieved in the case of replicated foams or syntactic foams. In such foams, the porosity is established by a space holder which either is removed from the structure (replicated foams) or remains in the structure (syntactic foams). Bekoz et al. investigated the effect of carbamide space holder shape on mechanical properties of replicated aluminium foams. They found that foams with spherical pores have superior mechanical strength [13]. According to the authors, the foams with irregular pores had compressive yield strengths between 20 MPa and 92 MPa depending on density of foams (0.83-1.66 g/cm³). This range increased to 25 MPa - 112 MPa in the case of foams with spherical pores and same density range. Jiang et al. produced open cell foam with infiltration of elongated and spherical carbamide space holders. They also reported that the foams with spherical pores have higher compressive strength [14]. However,

to the author's knowledge there is no experimental study in the literature focusing on the effect of space holder shape in case of metallic syntactic foams at the time of work.

In recent studies, we introduced expanded perlite (EP) [15] and pumice [16] as novel filler materials to produce aluminium syntactic foams. Results showed that the EP particles do not directly contribute to the mechanical response of foams because of their low mechanical strength. However, using smaller EP particles results in a refined aluminium microstructure with smaller grain size and more uniform foam geometry which improve the mechanical properties [3]. In the current study, the effect of pore shape on compressive strength and structural properties of EP/aluminium syntactic foam is investigated.

2. Materials and Methods

2.1. Samples preparation

Expanded perlite (EP) was supplied by Australian Perlite Company. Syntactic foams were produced by counter gravity infiltration of a packed bed of EP particles with molten A356 aluminium alloy. The chemical composition of constituents and a detailed description of the infiltration process can be found in [15]. Samples were subjected to a T6 heat treatment comprising solution treatment at 540 °C for 16 hrs followed by quenching in water at room temperature and aging at 160 °C for 10 hrs. In order to investigate the effect of particle shape on the mechanical properties of EP/A356 syntactic foams, EP particles were spheroidized using a simple rotary tumbling machine. The machine container was filled to two third of its volume and rotated with the speed of 60 rpm for 5 hours. Raw and rounded EP particles with the size of 2-2.8 mm were used in this study. To this end raw particles with a slightly larger initial size were filled in to the tumbling machine container in order to obtain spheroidized EP particles of the desired size.

2.2. Structural investigations

The roundness of particles was investigated by optical image analysis. To this end, high resolution digital photographs were taken from a large number of raw and rounded particles dispersed on a black surface. Subsequent thresholding was applied on the images using Image J software (http://imagej.net) to create binary black (background) and white (particles) images. The segmented images were then subjected to 2D analysis using a purpose-written Matlab code. The code measures the aspect ratio of the projected particles by dividing the shortest with the longest axis. A higher aspect ratio, with a maximum possible value of 1, indicates a more circular projection. Ellipsoid particles are more likely to rest on their longer side and thus this method is likely to yield lower values for particle sphericity.

The structure of syntactic foams was investigated by micro computed tomography (μ CT). μ CT scans were prepared using an Xradia MicroXCT-400 machine with a Hamamatsu L8121-03 X-ray source and a constant voxel size of 35.32 μ m. The selected acceleration voltage was 140 kV and the current 70 μ A. Due to its low density, perlite is transparent and appears as dark areas in μ CT images. The raw μ CT images of each sample were subjected to thresholding process and segmented based on the actual volume fraction of metal. The thresholding constant was selected through an iterative process so that the volume fraction of white voxels (metal) matched the volume fraction of aluminium in the scanned sample. The procedure to determine the volume fraction of aluminium based on the mass of particles and metal in the foams is explained in [15].

2.3. Mechanical testing

Compression tests were conducted following the ISO-13314 standard on a uni-axial computer-controlled 50 kN Shimadzu testing machine. A constant crosshead speed of 3 mm/min corresponding to an initial strain rate of 10^{-3} s⁻¹ was used in all tests. Stress-strain

curves were obtained by dividing the recorded load and displacement by the initial surface and height of samples, respectively. The unloading slope, yield stress, plateau stress, and the energy absorption of foams were derived from the stress strain curves based on the ISO 13314 standard [17].

3. Results

3.1. Structural properties

Unexpanded perlite rock is extracted in mines and crushed to small fragments with polyhedral shapes. After heating and subsequent expansion, the irregular shape of expanded perlite particles is a projection of the shape of the unexpanded particles. The as-received expanded perlite particles have irregular elongated, platy, or near spherical shapes (see Figure 1 a). Expanded perlite has a highly porous structure with about 95% internal porosity. Microstructural investigations showed that pore walls have a low average thickness (500 nm) [15] and are thus fragile and have a low mechanical strength. As a result, abrasion takes place in a short time after exposure of EP particles to the tumbling process. This removes the angular sections, corners, and edges and results in a rounded shape (see Figure 1 b). Moreover, the rough surface of raw EP particles becomes smoother as small protrusions are removed. During the tumbling process, the elongated particles break into smaller particles and most of the platy particles are crumbled completely and subsequently removed as dust. Domokos et al. [18] showed that regardless of the initial shape, tumbled particles would change to spheres in two stages. First, particle edges rapidly round without any change in axis dimensions until the shape approximates an ellipsoid. Subsequently, abrasion is driving the particles to become spherical and the axis dimensions are slowly reduced [18][18][18].

A Matlab code was used to measure the aspect ratio of individual particles by measuring the shortest and longest axes of each particle projection. The processed images are shown in **Figure 1** and the shortest and longest particle axes are marked by red lines. Comparing the processed images of raw and rounded EP particles, one can see how the tumbling process results in near spherical shape with smooth surface. The surface protrusions and uneven shape of the raw particles can be easily observed in the 2D processed images.





Figure 1. a) raw EP particles, b) rounded EP particles. The smallest and larges axes of particles are marked in processed images.

Different approaches have been proposed to describe the shape of particles [19]. In this paper, the sphericity of the particles is determined by their aspect ratio (i.e. the ratio between the minor and major axis). **Figure 2** shows the relative frequency of aspect ratios for 3 independent observations of raw and rounded particles. Each measurement analysed the shape of approximately 100 particles. The relative frequency denotes the number of occurrences of a ratio divided by the total number of particles. The average aspect ratio (standard deviation) is 0.80 (0.09) for rounded particles and 0.70 (0.11) for raw particles. As mentioned above a higher aspect ratio in the case of rounded particles indicates that the particles are more spherical. A lower standard deviation of rounded particles further means that the particle shapes are more homogeneous.



Figure 2. Normalized frequency of occurrence of EP particles aspect ratio

Figure 3a illustrates foams containing raw and rounded EP particles. Surface sections of the images are enlarged for a better comparison. One can see the irregular shape of the pores on the surface of the foam with raw EP particles. A considerable amount of elongated or plate like pores are observable. In some areas, tight contact between the flat surfaces of EP particles prevents proper infiltration during casting (see marked areas in **Figure 3** a). This causes the coalescence and formation of larger pores. This structural defect is referred to as missing cell wall in literature [6]. In comparison, foams with rounded EP particles show a more uniform surface with regular near spherical pores. There is no evidence of missing cell walls on the surfaces of the tested samples. A likely explanation is that the rounded EP particles have point-like contact areas leaving uniform and wide gaps for melt infiltration.

Figure 3 b and c show 3D reconstructions of the metallic phase of foams containing raw and rounded EP particles. All foams have an open cell structure due to the contact between neighbouring EP particles. Missing cell walls and an irregular cell morphology are more frequent in the 3D construct of foams with raw EP particles. Some areas containing missing cell walls are marked with red ovals. The considerable irregularity of angles between cell struts (see lines marked in black in **Figure 3** b) could be a result of the irregular packing of particles due to their non-uniform shapes. In contrast, the foam containing rounded EP particles shows a relatively uniform distribution of near spherical pores. Cell struts mainly form angles of 120° at the nodes and angles of 90° occur occasionally (see marked struts in **Figure 3** c). This is a characteristic of spherical packing systems. The angle between the cell struts depends on the coordination number of particles. The particles in body cantered cubic structure have a coordination number of 8 which results in an angle of 90 degrees between the struts. In face centred cubic and hexagonal packing systems the angle between the struts is 120° (coordination number 12). Because of slight differences in particle size and shape, the packing system may change in different layers which causes different coordination numbers and thus changes in the angle between the struts.

The 3D images also reveal that the cell faces in foams with raw EP particles are serrated. The reason is the coarse surface of the raw EP particles with lots of protrusions (see **Figure 1** a). In comparison, the rounded EP particles have a smooth surface that results in a smoother surface of the cell walls.



a





b

Figure 3. a) Light photographimages of foams containing raw and rounded EP particles, b and c) 3D construct of foams with raw and rounded EP particles respectively.

3.2. Mechanical properties

Figure 4 shows the quasi-static compressive stress-strain curves of foams containing either raw or rounded EP particles. All of the graphs exhibit the well-known compressive response of metallic foams which comprises three distinct regions; the elastic region in which the stress increases sharply, the plateau region which shows a gradual increase in stress over a prolonged strain range, and the onset of densification where the stress increases sharply again.

The foams with even slightly higher density exhibit higher stresses. The density of foams may vary due to uneven shape of particles that affects the particles packing density. A previous study showed that the mechanical properties of EP/aluminium syntactic foam is controlled by the matrix and EP particles do not have a direct effect on the mechanical properties [20]. Therefore, the higher volume fraction of matrix material in case of the foams with higher densities explains their higher strength. The foams with rounded EP particles (full lines) show considerably higher stresses compared to the foams containing raw particles (dashed lines) with similar densities. For a better comparison, curves of two types of the foams with identical densities (1.05 g/cm^3) are shown with thick black lines (see Figure 4).

In a previous study [3], it was shown that the shape of the compressive stress strain curves depends on the macroscopic deformation behaviour of the material and the way of formation and collapse of deformation bands. Hence, the similarity between the profiles of the curves in both types of foams might be an indicative of identical deformation behaviour. This has been approved by visual observation of the samples during the compression.



Figure 4. Stress strain curves of foams containing raw and rounded EP particles at different densities.

Figure 5 a-d show the mechanical properties of the foams. Rounded EP particles result in a slightly lower density of foam. This is most likely due to a higher packing density compared to raw particles. The effective mechanical properties of the foam samples were derived on the basis of the ISO 13314 standard. The plateau stress (σ_{pl}) is the mean stress between 20% and 40% of macroscopic strain. The sample with minimum density was compressed first and its plateau stress was measured. The remaining samples were unloaded at 70% and reloaded at 20% of this initially measured plateau stress. Their elastic unloading gradient was then obtained as the secant line of the resulting hysteresis loop. The absorbed energy was calculated using the integral $W = \int_0^{50\%} \sigma d\varepsilon$ where ε is the macroscopic strain. Error bars have been added based on an uncertainty analysis of the experimental measurements.

The graphs clearly show that foams containing rounded EP particles have superior mechanical properties at the same density. For instance, the unloading slope, 1% offset yield stress, plateau stress, and energy absorption of foams with rounded EP particles are

respectively 38%, 24%, 14%, and 19% higher than those of foams containing raw EP particles at the same density of 1.05 g/cm³. One can see that the increase in mechanical properties is more significant in the case of the unloading slope and 1% offset yield stress. Both values are related to the early stages of deformation where differences of the initial geometries are still preserved.



Figure 5. Mechanical properties of foams with raw and rounded EP particles: a) unloading slope b), 1% offset yeild stress, c) plateau stress, d) energy absorption 50%.

4. Discussion

In a previous study [3], it was shown that the mechanical properties of the P-MSF depend on the EP particle size and resulting microstructure of the matrix alloy. In general, smaller EP particles result in a modified microstructure of cell struts, i.e. smaller grain size and reduced casting defects, and therefore higher mechanical strength of the foam. [3]. The microstructure of the matrix alloy can also be modified by the subsequent heat treatment of the sample in order to improve the mechanical properties of the foam [21]. However, in the current work both raw and rounded EP particles have the same average size (2.4 mm). Furthermore, the produced foams undergo identical casting and thermal treatment to ensure an identical micro-structure and that differences in their mechanical properties are due to the EP particles shape.

The findings presented in the previous section are in good agreement with experimental [14] and numerical studies [11]: foams with near spherical pores exhibit superior mechanical properties. According to Cho et al. [11], the pore aspect ratio has a major effect on mechanical performance of the foams. Their finite element analysis showed that models with spherical pores have considerably higher strength when compared to ellipsoidal pores. According to those authors, high level of localized deformation at sharp edges of the pores results in premature failure of the cell wall. Following the same behaviour, the more ellipsoidal raw EP particles (see Figure 2) could result in considerable reduction in mechanical strength of the structure. The μ CT observations (**Figure 3**b) further revealed that the cells in foams with raw EP particles have a rough surface due to protuberances and structural defects which are better observable in 2D projections of the particles shape (see **Figure 1**a). This surface roughness may trigger local stress concentrations that further reduce the strength of cells in raw EP foams.

Furthermore, the higher homogeneity of the structure and lower structural defects in the case of foam with rounded EP particles improve the mechanical strength of the foam both in elastic and plastic regions. Silva et al. [22] investigated the effects of structural irregularities by numerical investigation of two dimensional ordered honey-comb (i.e. the angle of 120° between the struts) and irregular voronoi structures. They observed that defect-free structures with random variations in cell wall arrangement are approximately 20-40% weaker than periodic structures of the same density. Their findings have been confirmed by Ajdari et al. who observed a 27% decrease in the yield stress in the case of irregular structure [6]. According to Silva et al., a higher magnitude of localized stress in irregular structures leads to failure at a lower effective stress than in the periodic case. The same reasoning can be used to explain the approximately 24% higher 1% offset yield stress of the P-MSFs containing rounded EP particles. Silva et al. further showed that defects, such as missing cell walls, significantly reduce the strength of the structure [22]. This could be a further reason for lower strength of the foams with raw EP particles that have a higher amount of missing cell struts (see Figure 3a). Interestingly, finite element analysis results [6] showed that the structures with a higher level of irregularity are more sensitive to missing walls. Foams with raw EP particles exhibit both a more irregular structure and a higher number of missing cell walls when compared to shaped EP foams thus explaining the difference in mechanical properties.

Considering Figure 4 the overall shape of the stress-strain curves is essentially similar for all samples. This is attributed to identical macroscopic deformation behaviour of foams containing raw and rounded EP particles, i.e. formation and collapse of the deformation bands. The visual observation of the current foam samples during the compression test revealed similar macroscopic deformation of all samples (i.e. raw and rounded) irrespective of their particle shape. On account of the findings of Silva et al. [22], structural (in)homogeneity and structural defects are unlikely to have significant effects on the macroscopic deformation behaviour of investigated foams. They suggested that the irregularity of the structure predominantly affects *local* deformation in foams, e.g. the deformation of individual or a number of neighbouring cells. A larger number of deformation sites result in a lower overall stress in the case of irregular structures. However, they further stated that it is unlikely to change the *macroscopic* deformation patterns. High local stresses caused by irregularity are rapidly decreased by deformation and result in the shifting of deformation to weak struts elsewhere in the structure. For the same reason, missing cell struts do not macroscopically alter the deformation pattern of the structures. According to Silva et al., when one or two cell struts were removed (i.e. defects were introduced) in volumes close to an active deformation band, this band did not shift. Individually dispersed defects typically do not sufficiently weaken the surrounding strong cells to generate a new deformation band. Instead, weaker cells elsewhere in the structure collapse [22]. According to their findings, a critical concentration of defects is required to ensure that the deformation band invariably passed through the defects [22]. This could explain the fact that despite reducing the strength of the material, the missing cell struts in the foam with raw EP particles do not change the macroscopic deformation behaviour of the foam when compared to the foams containing rounded EP particles.

5. Conclusions

The effect of EP particle shape on the structural and mechanical properties of EP/Aluminium syntactic foam was investigated. A rotary tumbling machine was used to turn the irregular shape of raw EP particles to a near spherical geometry. Foams with a density of 1.05 g/cm³ containing raw and rounded EP particles were subjected to quasi-static compression testing. Usage of rounded EP particles increased the unloading slope, 1% offset yield stress, plateau stress, and energy absorption by 38%, 24%, 14%, and 19%, respectively.

A likely explanation for these improvements is geometry changes of the aluminium matrix. Image possessing and micro computed tomography (μ CT) investigations revealed a regular distribution of near spherical pores in the case of foams with rounded EP particles. In contrast, an increased level of cell struts misalignments, missing cell walls, and pore surface roughness was observed for foams containing raw EP particles. A significant reduction in the number of such defects is the likely explanation for the superior properties of foams containing rounded EP particles.

Conflicts of Interest: The authors declare no conflict of interest.

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