

Hollow Glass Microspheres Filled Aluminum Syntactic Foam Made Through Stir-Casting Technique



Kirti Chaware, G. Dixit

Abstract: *Hollow Glass Microspheres (HGM) in the range of 10–20 vol. % were used as space holders for making syntactic aluminum foam having density 2.28–2.49 gm/cc using stir-casting technique. Aluminium alloys have already established their exceptional ability to sketch out the material for required properties. Aluminium alloy and its composites are one of the widely used materials for aeronautics and marine applications where high strength is expected from a low density material. So, an effort is made to enhance this lightness of aluminium alloy LM13 without affecting its strength by reinforcing it with Hollow Glass Microspheres (HGM). This synthesized syntactic foam, was characterized in terms of density and compressive deformation behaviour. It was noted that the syntactic foam behaves like a high strength aluminium foam under compressive deformation exhibiting flat plateau region in the stress–strain curves. The plateau stress of syntactic foam decreases with Hollow Glass Microspheres volume fraction vis-à-vis porosity.*

Keywords: *Hollow Glass Microspheres, Compressive Strength, Stir Casting, Aluminium Alloy, LM13, Syntactic Foam.*

I. INTRODUCTION

Syntactic foams are being used since 1960s and at that time, these foams were used for deep sea applications as buoyancy aid materials [1]. Syntactic foams are also one of the composite materials, synthesized by dispersing the particles of hollow geometry known as micro-bubbles, micro-balloons, or microspheres into a ceramic, polymer or metal matrix. Presence of these hollow particles results into a composite with higher strength to weight ratio because of low density and lower coefficient of thermal expansion. Such foams exhibit excellent combination of mechanical and physical properties which may be of great importance for various specific and tailored applications [1]. Such as core in sandwich structures, for packaging / fire proof, crash safety, damping panels and under-water buoyant structures [1–5]. One of the most remarkable property of syntactic foam is their high damage tolerance because of their high energy absorbing potential.

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As compared to other foams syntactic foams shows high specific stiffness, improved strength and high acoustic and mechanical damping capacities as well [2,3].

In the view to achieve improved and desired mechanical properties, syntactic foams are primarily developed by two techniques.

In the first method the volume fraction of the dispersed particles that is micro-balloon in the syntactic foam structure is changed whereas in the second technique, micro-balloons of different size or wall thicknesses are used to fill the syntactic foam structure.

Syntactic foams compressive properties mainly depend on the properties of micro-balloons while the tensile properties depend on the properties of material for matrix that holds the micro-balloons together.

It has been observed in the literature that the syntactic foams filled with ceramic particles and the volume fraction of this ceramic phase is comparatively high in the matrix, shows excellent thermal insulation and the coefficient of thermal expansion is also observed to be low [6–8].

By far the literature of aluminium alloy syntactic foams are concerned, the quasi-static compression behaviour with a focus on energy absorption capability of these foams, has attracted few researchers in the past to carry out the study [2–6,9–11]. Ramachandra and Radhakrishna reported that by adding the micro-bubbles, density of composites reduces and the mechanical properties enhances as they synthesized aluminium based metal matrix syntactic foams (MMSFs) by dispersing micro-bubbles by stir casting method [12].

In the present research work, an endeavour has been made to synthesize Aluminium LM13-HGM syntactic foam as no research has been observed during the literature review about the development and characterization of syntactic foam having matrix of LM13 aluminium alloy with space holder as the hollow glass microspheres.

Hence, it was thought to be an interesting project to study their compressive behavior and density so as to understand its worthiness for such applications. This work imbibes the density and compressive behaviour of novel syntactic foam developed by dispersing hollow glass microspheres with varying volume concentrations in the LM13 Aluminium alloy matrix, resulting in syntactic foams with three different densities. These composites behave like solid foams in general and their behaviour under compression changes along with composite density.

II. MATERIALS AND METHODS

A. Matrix Material

LM13 alloy was found suitable and chosen to be the matrix material because of industrial viability, and its chemical composition is given in Table 1.

Owing to its good resistance against wear, low coefficient of thermal expansion along with good bearing properties, various uses and applications of LM13 alloy are not limited to pulleys (sheaves), pistons for diesel and petrol engines, and other automotive engine parts operating at elevated temperatures. Another impressive characteristic of LM13 alloy is its high resistance to corrosion under atmospheric conditions. It possesses good fluidity due to which it can be cast into comparatively thin sections. LM13 approximately melts in the range of 525-560 °C whereas its typical pouring temperature is 700 °C but depending on the mould configuration the temperatures may range between 670-780°C.

Table 1: Elements in LM13 Aluminium alloy

Elements	Si	Mg	Cu	Fe	Ti	Cr	Ni	M _n	Al
Wt.%	12.1	1.2	0.8	0.8	0.02	0.07	0.9	0.2	Bal.

B. Micro Balloon Material

Hollow Glass Microspheres, also called bubbles, microbubbles or microballoons, and are typically made out of soda lime - borosilicate glass blend formulation which provides low density with other useful benefits such as high heat and chemical resistance. These hollow glass microspheres, typically possess rigid walls with thickness of about 10% of the diameter of the sphere. Generally the thickness of wall of these hollow glass spheres determines the crush strength and, as expected, the higher the density of the sphere, the higher the crush strength. Some other impressive properties offered by these light weight soda lime borosilicate hollow glass microspheres are that they are chemically stable, non-combustible, nonporous, and have excellent water resistance.

Some important physical properties of soda lime – borosilicate, hollow glass microspheres used as space holder material for developing the concerned syntactic foam as per the data of supplier 3M™ is given in Table 2.

Table 2: Properties of Hollow Glass Microspheres

Appearance	White Powder
True Density	0.6 g/cc
Softening Point	600 Degree Celsius
Crush Strength	27,000 MPa
Size	18 microns

C. Composite Fabrication

The studied HGM reinforced LM13 alloy syntactic foam is developed using permanent mold die casting assisted with melt-stirring technique. An innovative two-step blending method, preheating of particles, rationally chosen stirrer

velocity, and melt degassing using hexachloroethane tablets (C₂Cl₆) have assisted to achieve steady dispersion of hollow glass microspheres [13-18]. The ingot-shaped LM13 alloy was previously split into smaller parts and then kept inside a resistance furnace of 2 kW Power working at 230 V, in a graphite crucible and superheated to 700 °C. Commercially available Hexachloroethane tablets (C₂Cl₆) were used to degas, the molten metal in order to reduce the defects of casting namely porosity, blow holes and voids. Before adding the HGM particles to the melt the temperature of the melt was reduced and maintained at 650°C. In order to remove loose scales, residues and moisture, the reinforcing HGM particles of size 18 microns were preheated to 200 °C for 2 hours. Stirring of molten metal was carried out by a motorized stirrer operated at speed of 550 - 600 rpm in order to generate a whirlpool. The HGM particles are discharged to the molten metal whirlpool at a rate of 15 - 20 g/minute through a funnel. In the view to avoid contamination, the chromium steel blades mechanical stirrer with zirconium coating was used. The stirring was maintained at a low rate of 300 rpm for about 15 min to obtain adequate dispersion of HGM particles. On completion of stirring process the molten mix of metal and reinforcing particles was at last poured into a preheated mold of cast iron, at a pouring temperature of 650 °C and then allowed to cool and solidify at ambient temperature. Three dissimilar composites with different percentages of HGM namely, HG10 (10 v% HGM), HG15 (15 v% HGM) and HG20 (20 v% HGM) were fabricated to study the effect of HGM reinforcement on the density and compressive behaviour of these combinations of matrix and reinforcement. Unreinforced LM13 alloy was also cast for comparison purpose. The stir casting experimental set up and process details is given in Fig. 1 and Table 3 respectively.

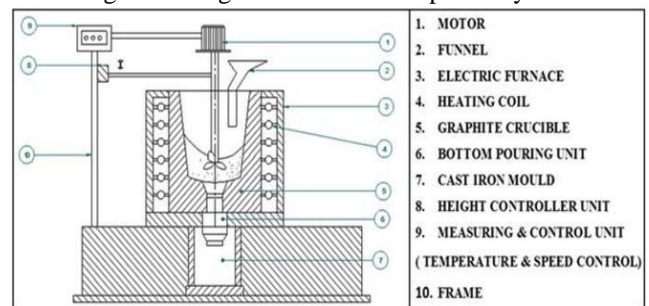


Fig. 1. Schematic of Stir Casting Setup [20]

Table 3: Stir Casting Process Parameters

Impeller Type	Placement of Stirrer	Span of Stirring	Speed of Stirring	Metal Pouring Temperature
3 blades fan type made from chromium steel and coated with Zirconium	Depth of ~2/3 calculated from the bottom of crucible	~15 min	550 - 600 rpm	650 °C

D. Density of Materials and Volume Fraction of Hollow Glass Microspheres

Density, which in both physical and everyday terms simply refers to a concentration of something within a given defined space, usually means "mass density," and thus it refers to the amount of matter per unit volume. In addition, the concept of composite density is important.

Many materials naturally consist of, or are manufactured from, a mixture or elements or structural molecules, each with their own density. If the ratio of individual materials in the composite under study is known along with their individual densities, then it is possible to determine the composite density of the developed material as a whole. A solid sphere of mass *M* and radius *R* with its mass spread evenly throughout the sphere and a solid sphere of mass *M* and radius *R* with its mass concentrated almost entirely in a thin outer "shell" have the same density.

In the concerned study three sets of syntactic foams were developed each with a different volume fraction of HGM (*f_g*). This means that in each set of developed syntactic foam the HGM volume fraction (*f_g*) and the volume fraction of aluminium alloy LM13 (*f_m*) is different. And this is the base of this study, as the changes in the *f_g* will result in syntactic foams with different porosity that will further affect the compressive behaviour of these syntactic foams.

E. Compressive Strength Testing



Fig. 2. BiSS Slow Strain Rate Test System

Quasi-static compression test was performed on the developed syntactic foam at ambient temperature using BiSS Slow Strain Rate Test System as shown in Fig. 2. These tests were performed in accordance with ASTM-E9-95 at Advanced Centre for Material Science at IIT, Kanpur (U.P), India. Strain rate for these tests was set at 0.1/s. Samples prepared for this test are cylindrical in shape with length of 15 mm and diameter of 10 mm. Prior to testing, the friction between the compression test plates and the specimen surface was reduced by mechanically polishing the specimens surfaces and coating them with thin layer of molybdenum sulphide for lubrication. During the testing, data was recorded for load-displacement and then standard methodology was

used to convert it to stress–strain graph. Set of five samples were tested for each density set of syntactic foam.

III. RESULTS AND DISCUSSION

A. Density and Porosity

The density of HGM (ρ_g) is noted to be 0.6 gm/cc whereas the density of the aluminium alloy LM13 matrix material (ρ_m) is 2.7 gm/cc. Further the density of the synthesized syntactic foam (ρ_s) can be calculated on the basis of the rule of mixtures. The volume fractions f_g and f_m in the form of percentile contribution must be converted to the decimal number by dividing it by 100, then only it can be used in the equation below.

$$\rho_s = \rho_g f_g + \rho_m f_m$$

Once the density of the developed syntactic foam (ρ_s) is calculated on the basis of the rules of mixtures by using the above equation. Then the relative density (ρ_r) can be calculated by using the following relation.

$$\rho_r = \rho_s / \rho_m$$

Now, the porosity fraction (f_p) can be given as,

$$f_p = 1 - \rho_r$$

Three sets of f_g that is 10 v%, 15 v% and 20 v% is used for synthesizing syntactic foam. This variation in f_g leads to varying porosity fraction in the developed syntactic foam. The densities and the porosity fraction (f_p) for different sets of syntactic foams are reported in Table 4. The thermal expansion coefficients of aluminum alloys is considerably larger than that of HGM and this could lead to the thermal residual stress vis-à-vis generation of higher extent of dislocation. There is also a greater possibility of relaxation of residual stress through localized elasto-plastic deformation of the HGM shells, and sinking of dislocation at the HGM–matrix interface, because of mechanical bonding. With the increase in HGM content, the HGM–matrix interface area increases, and therefore more amount of dislocations get annihilated at the interface. Thus, the dislocation density as well as the residual stress within the matrix in syntactic foam remain invariant to HGM content and matrix strengthening due to HGM addition is negligible [19].

Table 4: Density and Porosity of Syntactic Foam

HGM Volume Fraction	Syntactic Foam Density	Relative Density	Porosity Fraction
10 v%	2.49	0.92	0.08
15 v%	2.38	0.88	0.12
20 v%	2.28	0.84	0.16

The density and porosity are the two inversely proportional properties in foam structures and syntactic foams. Higher the density lower the porosity and lower the density higher the porosity in the material and this correlation of density and porosity can be clearly observed in Table 4. Lowering of density reduces the mass per unit volume and the weight per unit volume also gets reduced. Thus the material gets lighter and lighter as the density goes on decreasing.



But this lightness of the material should not be at the cost of strength as far as the mechanical and structural applications are concerned. So, it becomes necessary to see the variation in the compressive strength with the decrease in the density of the syntactic foam with change in the HGM volume fraction. Thus, in order to check the balance of density and compressive strength of the developed syntactic foam, a graph has been plotted in Fig. 3 between peak compressive stress and relative density.

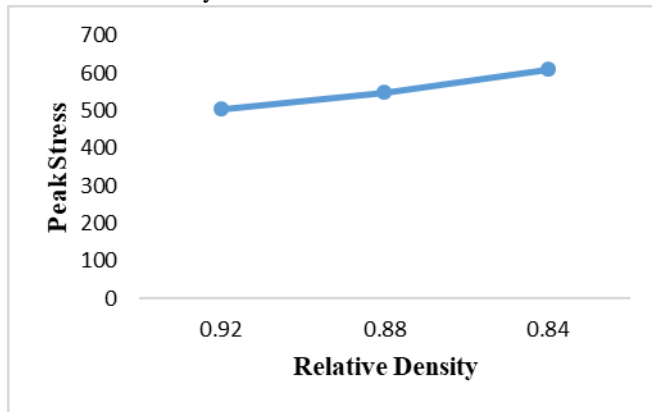


Fig. 3. Variation of Peak Compressive Stress with Relative Density.

B. Compressive Behaviour

Compressive stress strain curves of syntactic foam with varying porosity vis-à-vis HGM content is shown in Fig. 4 and Fig. 5. Stress strain curves for the syntactic foams having 10 v% and 20 v% of HGM are only reported in the Fig. 4 and Fig. 5. It is done for simplification, because the stress strain curve for 10 v% and 15 v% are graphically similar to each other. The stress strain curve for 10 v% and 15 v% of HGM shows a linear portion at the initial stage indicating the elastic portion of the graph. Just after this there is a small flat region where the strain rises at almost constant stress and this is the plateau region where the plastic deformation and compaction of the foam takes place. But, this plateau region is comparatively small for 10 v% and 15 v% stress strain curves as compared to the stress strain curve for the 20 v% HGM. Also this plateau region is not followed by the densification strain as in the 20 v% stress strain curve, instead there is a sharp drop in the stress just after the small plateau region. This behaviour is observed because the volume fraction of HGM is relatively low as compared to the volume fraction of matrix resulting in low porosity fraction. So, there is not enough porosity to show a longer compaction region and densification region. However, improvement in the compressive strength is observed as compared to pure matrix material but as far as the stress strain curves for 10 v% and 15 v% HGM are concerned the compression behaviour of the samples is more like solid than foam. The stress-strain curve for 20 v% HGM clearly depicts five regimes (I) a linear portion at the initial stage indicating elastic portion, (II) just after yielding there is a small flat region, (III) after regime II, sharp drop of stress with strain and then again increases with increase in strain indicating a well-defined yield point, (IV) a plateau region where the plastic deformation and compaction of the foam take place at almost constant stress level and (V) densification region beyond the densification strain where the SF starts

densifying after compaction and plastic deformation. This indicates that from 20 v% onwards the compressive behaviour of the samples of syntactic foam shifts from the solid mode to foam mode. The syntactic foam with 20 v% of HGM clearly behaved like foam during the compression test and all the standard regions of compression in the stress strain curve that is elastic region, plateau region and densification region are available. One of the most important difference observed in the stress strain curves of the three sets of syntactic foams is the increase in the strain for peak stress from 24% to 78% for 10v% and 20 v% of HGM respectively. It is because the deformation behaviour of 10 v% and 15 v% syntactic foams is almost like a solid whereas the syntactic foam with 20 v% HGM showed the compressive behaviour of foam leading to much higher strain at the peak stress as compared to the other two sets of syntactic foams.

The plateau region of the stress strain curve for 20 v% HGM is almost flat and depicts apparently no strain hardening during the plastic deformation of the syntactic foam. It can also be clearly observed from this curve that the stress lowering took place at a critical strain of approx. 0.16 after yielding and the stress reaches its lowest point in the curve at a strain of about 0.20. As in the present study, the average size of the HGM is same for all sets of syntactic foams. So, the extent of stress reduction after yielding may be a measure of either of the two phenomenon. First is the degree of agglomeration or tendency of collapse or breakage of HGM shells during yielding. In this the HGM's in the matrix of syntactic foam begins to deform elasto-plastically ensuing the strain hardening of matrix. But this strain hardening may not be that significant because of the mechanical bonding between the HGM and the matrix material at its interface. And this interface itself acts as the site for dislocation sink that inhibits the matrix strain hardening and is expected to be very marginal. Secondly, in the due course of time, the HGM shells starts to shear and fracture, resulting in the loss of modulus that subsequently decreases the stress. Now the fraction of HGM that collapsed or broke begins to compact and again the yielding of matrix begins when the level of stress reaches to the extent where the next series of HGM begins to shear and crack. The deformation in the syntactic foam is highly inhomogeneous and localized because of its highly porous nature. It has been observed throughout the plateau region that the stress varies in an oscillating manner. This oscillating nature of stress is the effect of collapse of the HGM shells and shearing of the matrix around the HGM followed by the compaction of pores as all this takes place simultaneously during the deformation in the plateau region. After yielding, at the initial stage, major volume fraction of HGM may get sheared, broken and collapsed leading to higher degree of stress reduction. During the process of deformation, matrix also gets deformed and this deformation of matrix may lead to the strain hardening because of this a steady increase in the stress is expected along with the strain during the deformation. But the stress does not rise with the strain as expected, instead it oscillates about the average stress value that is plateau stress in the plateau region.

This behaviour is attributed to the breakage and collapse of HGM shells and its densification in the due course nullifies the strain hardening effect and is supposed to be responsible for decrease in the values of stress. Whereas a marginal stress hardening takes place when the matrix gets deformed after the densification of localized broken and collapsed HGM shells and this strain hardening is responsible for the increase of stress in the oscillating stress behaviour of plateau region.

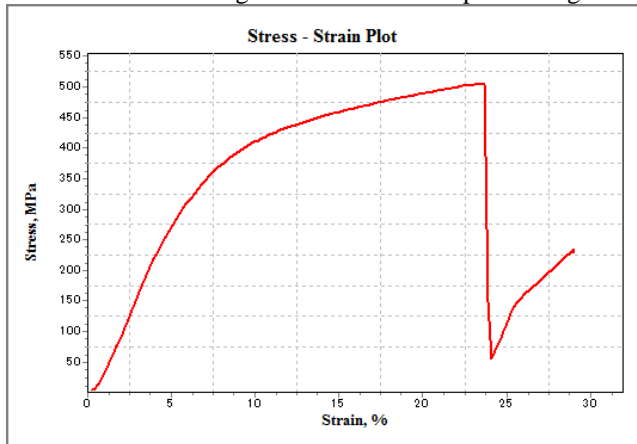


Fig. 4. Stress Strain Curve for 10 v% HGM.



Fig. 5. Stress Strain Curve for 20 v% HGM.

During the compression test, it was observed that the deformation initiates from the corner region of the specimen's surface in contact with the compression platen. This deformation progresses at an angle of approx. 45° along the plane of shear towards the specimen's centre at the middle of the sample. During the course of deformation the sheared zone progresses layer by layer. The maximum deformation is observed to be associated with the planes of shear and at the centre of the samples [19].

IV. CONCLUSION

- Hollow glass microspheres are capable of retaining their shape and size during the process of mechanical stirring and so it can be used effectively to synthesize the hollow glass microsphere filled aluminium alloy syntactic foam.
- Syntactic foam with up to 20 v% hollow glass microspheres was synthesized successfully by using the permanent mould die casting assisted with melt-stirring technique.
- Syntactic foam's density has decreased with rise in the volume fraction of hollow glass microspheres resulting in per unit weight reduction. But this lightness in material is in

balance with the compressive strength of this hollow glass microspheres filled aluminium alloy syntactic foam.

- Compressive behavior of syntactic foams with 10 v% and 15 v% of hollow glass microspheres is almost similar to a solid material whereas the syntactic foam with 20 v% of hollow glass microspheres showed a foam like compressive behavior. This means, at least 20 v% of hollow glass microspheres is required to develop an effective syntactic foam with aluminium alloy matrix.
- The strain at peak stress during compression for up to 15 v% of hollow glass microsphere is around 24% but as soon as the compression behavior changes from solid like to foam like for the syntactic foam with 20 v% of hollow glass microsphere, then the strain at peak compressive stress reaches around 78%.
- The compressive deformation behavior of the developed syntactic foam is found to be similar to that of conventional low density aluminium alloy foam. However, the plateau stress of syntactic foam is comparatively higher than the conventional aluminium foams.
- Compressive strength of the developed syntactic foam was observed to improve significantly with increase in the hollow glass microspheres content.
- Hollow glass microspheres can be effectively used with the aluminium alloy matrix for the development and production of syntactic foams with superior compressive strength at low cost for applications where good strength and stiffness is needed from a light weight material.

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