Synthesis and Compressive Characterization of Aluminium-SiCp Composite Foam by Stir-casting Technique Using Dual Foaming Agent

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Abstract – Aluminium Composite foams is produced by melting Aluminium alloy (LM6) containing foaming agent(s) and vigorous stirring. TiH₂ is a common agent for this. As TiH₂ decomposes into Ti and gaseous H₂ when heated above about 465°C, large volumes of hydrogen gas are rapidly produced, creating bubbles that leads to a closed cell foam. A novel Strategy to enhance the mechanical properties of Al-MMC foams is discussed here, and it is demonstrated that titanium hydride (TiH₂) in the form of 10-15 μ m diameter particles can be pre-treated by selective oxidation to produce more uniform foams having better

mechanical properties. It is found that the mechanical properties of the foams and the uniformity of cell size distribution is improved when the foam is blown with an optimized mixture of CaCO₃ and pre-treated TiH₂.

In order to define the relationship of mechanical properties with relative density of this material, correlations which uniquely defines the compressive behaviour of this modified Al-MMC foam has been developed.

Index terms: Al-Si MMC foam, mechanical behaviour of cellular materials, Dual foaming agent

I. INTRODUCTION:

Metal foam is a type of cellular solids, having a combination of properties such as high stiffness with very low density and a capability to absorb impact energy. These unique combinations of properties indicate various potential applications such as packaging materials for protection sensitive devices, machinery enclosures, automobiles, and as sound absorbing material under difficult situations. Mechanical testing of aluminium foams is a prerequisite for any application. The study of compressive properties of metallic foams is necessary as its major applications are primarily loadbearing and energy absorption. Even aluminium foams whose main properties are functional require minimal mechanical properties to prevent damage or failure. The compressive stress-strain diagram of metal foam as defined by Gibson and Ashby [1] consists of three distinct regions namely linear elastic region, collapse region and densification region. Fig. 1 shows a representative stress – strain curve of metal foam under compressive loading. The first zone (linear elastic zone) is recorded up-to small strain (about 2-3%). The second zone i.e. plateau region, continues up to about 70% of strain, characterized by a small slope of the stress-strain curve. In some cases the curve is even horizontal. In second zone collapsing of cell continues till the foam behaves like a solid material. The third zone (densification zone) shows a rapidly increasing stress, here the cell walls become pressed together and the material attains bulk-like properties.



Figure 1: Stress strain curve for metal foams [1].

The main aim of the present investigation is to determine the compressive characteristics of the closed cell Aluminium Metal Matrix Composite (Al-MMC) foams developed in the laboratory. The outcome of the experimental investigation are compared with the established theoretical models developed by different researchers [1-9] so that it can be further referred for different industrial applications.

II. SYNTHESIS OF AL-SiCp COMPOSITE FOAM

The material under investigation is closed cell aluminium foam, manufactured through liquid metallurgy route in the Foundry Laboratory of Jadavpur University, Kolkata, using aluminium alloy (LM6: consisting of 0.1% Cu, 0.1% Mg, 0.13% Si, 0.6% Fe, 0.5% Mn, and trace amount of Zn, Pb, Sn and rest Al). The aluminium alloy used is of density (ps), 2.7gm/cm3, having compressive elastic modulus (Es) of 69 GPa and compressive yield strength (σ_s) of 120 MPa. The ingot is melt in a tilting resistance furnace. The formation of foam requires a high liquid-metal viscosity which is achieved by the addition of Silicon Carbide (SiC) particulate to the melt. The amount of Aluminium is 1 kg. 5% SiC (pre-heated) are added to the melt, which also increases the mechanical strength of the foamed component. For homogeneous mixture of SiC in Al matrix, continuous stirring is required. The achieved high viscosity allows liquid Aluminium to be stable at a temperature of TiH2-decomposition (465°C) which is much lower than the freezing temperature of liquid Aluminium.

The homogeneous Al-SiC mixture is then poured into a pre heated mold (which is fitted with a stirring arrangement) after removal of slag as much possible. 2.5% blowing agent (Titanium Hydride) is added to the mold. TiH₂ begins to decompose into Ti and gaseous H₂ when heated above about 465°C. By adding titanium hydride particles to the aluminum melt, large volumes of hydrogen gas are rapidly produced, creating bubbles that leads to a closed cell foam structure. It is needed to stir the mold with constant speed for good foaming.



Figure 2: Pouring of Al-SiC melt into mold.

As TiH₂ is a very costly material, so, manufacturing of Al-SiCp foam by this method is not so cost effective. The solution to this problem is Calcium Carbonate (which is very cheap in cost). So, instead of adding 2.5% TiH₂, a dual foaming agent (2% CaCO₃ and 0.5% - 1.0% TiH₂) is added separately and this produces same result with minimum cost. Addition of Ca in Al matrix slightly changes the mechanical properties but it is nearly identical.

The properties of metal foams depend on many morphological features, such as pore size distribution, cell wall curvature, defects, etc.[3][•] Although the exact interrelationship between properties and structure is not yet sufficiently known, one usually assumes that a uniform distribution of convex pores free of defects is highly desirable. The task for the experimentalist is to produce such

structures. A short look at existing foams shows that there is still much potential for development since these often tend to be irregular [4].

Thus, the foam fabricated by this method are usually non-uniform which leads to inferior mechanical properties. The reason for this can be non-adoption of TiH₂ to the melting range of the alloy to be foamed. This is avoided by pre treatment of titanium hydride (TiH₂) in the form of 10-15 μ m diameter particles by selective oxidation.

The pre treatment of TiH_2 was first introduced by B. Matijasevic-Lux and J. Banhart for manufacturing of Aluminium foam through powder metallurgy [10]. The same method is followed here for melt route also.

TiH₂ powder supplied by LOBA chemical, India (purity 98.9%) was used in this study. The powder was supplied in the "untreated" state. Pre-treatments of the TiH₂ powder were carried out isothermally at various temperatures (450, 480, 510 °C) and times (30, 60, 120 and 180 min) under air in a chamber furnace. For heating, the ceramic crucible (with required amount of TiH₂) is placed into a volume chamber muffle furnace and is left there for the time specified. After pre-treatment all powders were gently homogenized by tumbling in a container.

Hydrogen starts to be released from TiH₂ at about 405 - 470 °C with some variations between powders of different origin. However, most of TiH₂ powder starts decomposing at 465°C.

As heating is carried out under air, an oxide layer grows which is roughly 100 nm thick [10] after 180 min at 480°C and contains an outer shell of TiO₂ and an inner shell of Ti₃O.[10]. Pre-treatment under air also reduces the amount of hydrogen and shifts the temperature of decomposition by 160°C. Using pre-treated TiH₂ for foaming Al alloys delays foaming and leads to a more uniform distribution of rounder pores. The best parameters found are close to 60 min at 480°C. It is noted that at higher pre-treating temperature (510°C), the amount of available hydrogen is not sufficient to produce uniform foam (Fig.4).



Figure 3: Comparison of stress-strain curves (compression) of Al foam using untreated and pre-treated TiH₂.





(c)

Figure 4: Optical microscopy image of the Al-MMC foam samples at 10x magnification. (a) using untreated TiH₂, (b) TiH₂ treated at 480°C for 60 Minutes, (c) TiH₂ treated at 510°C for 60 minutes.

III. EXPERIMENTAL PROCESS

Aluminium foam is specially designed for load bearing applications; therefore efforts are made to evaluate the compressive properties of the developed Al-MMC foam. Compressive tests are performed on Al-MMC foams of various relative densities. All the compression tests are performed in a computer controlled 100KN compressive testing machine. The specimens for compressive tests are of 10x10x20 mm sizes. The specimen surfaces are polished before the test and during test the surfaces are greased to reduce friction. Most of the specimen tested exhibit a significant drop of the stress after the end of the elastic deformation at different strains. This is because of failures in the structure of the foam (fractures of cell walls) at several locations, manifested in the form of sudden stress drop. The yield stress (σ_{Pl}) are determined from the curves and the results is presented in Table 1. The scatter in the experimental results can be explained from the fact that Al-MMC

foams are produced by random dispersion of gases in the melt, as such structure i.e. cell size and cell shape will vary. Such nonuniformities in the structure of the material yield a scattered result. It is observed that the use of Dual foaming agent not only reduces the cost, but also it enhances the mechanical properties. The mechanical properties are even better (definite plateau region) if pre-treated TiH₂ is used (Fig. 2). And the optimum pre treatment temperature is 480°C, holding time is 60 minutes.

Untreated TiH2		TiH2 at 450C 60		TiH2 at 480C 60		TiH2 at 510C 60	
	Yield		Yield	16 (Yield		Yield
RD	Strength	RD	Strength	RD	Strength	RD	Strength
	(MPa)		(MPa)		(MPa)		(MPa)
0.1522	2.4	0.1162	1.7	0.1015	2.1	0.1272	2
0.1718	1.8	0.119	1.75	0.1176	2.5	0.1225	1.9
0.1937	2.3	0.143	2.1	0.1265	2.7	0.147	2.3
0.1988	2.4	0.1624	2.8	0.132	2.8	0.153	2.4
0.2	2.5	0.1832	2.7	0.15	3.2	0.179	2.8
0.2015	2.6	0.19	2.9	0.158	3.4	0.188	2.9
0.2111	2.67	0.2047	3.1	0.187	3.9	0.2005	3.1
0.2151	2.9	0.21	3.2	0.198	3.9	0.205	3.2
0.2177	2.1	0.2125	3	0.2015	3.95	0.211	3.3
0.2267	3	0.22	3.3	0.215	4.05	0.22	3.4

Table 1: Yield strength of Al-MMC foams of different relative density.

Yield Strength Vs pre-treatment temp. of TiH2



Fig. 4: Variation of Yield Strength w r t heat treatment of TiH2

Comparison with existing theoretical models of compressive behaviour

Gibson and Ashby [1] proposed a model for closed cell metal foam of density ρ_0 and made from solid aluminium alloy of density ρ_s with a structure consisting of cubes of solid struts and plates, in which the struts meet at their midpoints,. When closed cell foam is deformed the cell edges bend, and the cell wall carry membrane stresses. The contribution from cell wall stretching to the overall stiffness and strength of the foam is represented by a term which is linear in the relative density, (**RD**= ρ_0/ρ_s), while the contribution from cell edge bending is non-linear in the relative density. Thus according to Gibson and Ashby [1], the yield stress of metallic foam σ_{pl} in compression is related to the yield stress of the cell wall material σs as:

(3)

where, φ is the "distribution constant", is the fraction of solid in the foam which is contained in the cell edges; the remaining fraction (1- φ) is in the faces. And RD is the Relative Density.

$$RD \le \varphi \le 1 \tag{2}$$

Gibson and Ashby [1] developed this model without considering the distribution of the cell-size, cell edge and cell-wall thickness, or any other the irregularities present in the structure of the closed cell metal foam.

Similar efforts have been made by various researchers. Andrew et al. [5] and Simone and Gibson [6] generalized Eq. (1) as:

Lu and Ong [7] described compressive behaviour of closed-cell aluminium foams as,

Tzeng and Ma [8] described compressive behaviour of closed-cell aluminium foam developed by them as,

Grenestedt [9] described the compressive behaviour of closed-cell aluminium foam considering the cell wall imperfections as,

$$\frac{\sigma_{\Theta^{*}}}{\sigma} = 0.3 \text{ RD}$$
 (6)

The normalized compressive yield stresses are measured by finding the ratio of the measured compressive yield stress of the Al-MMC foams (Table 1) to the compressive yield stress of the solid aluminium alloy. The normalized compressive yield stress of the Al-MMC foams is then compared with the models. Fig. 4 shows the normalized yield strength ($\sigma_{pl} * / \sigma_s$) of closed-cell aluminium for different relative density (RD = ρ_0/ρ_s) based on various theoretical models. All the models excepting that defined by Eq. 6 is developed assuming that cell edges and faces are uniform, they have also neglected morphological defects like curves and wiggles. The normalized compressive yield strength of the Al-MMC foam is also plotted in Fig. 4. It is evident from Fig. 4 that the properties of closed cell Al-MMC foams are well below that suggested by models of closed cell Al-MMC foams. The theoretical results seem to be higher compared with the current work. Such difference can be attributed to the fact that the model assumes identical cube cells, without considering the distribution of the actual cell shape, cellsize, cell edge and cell-wall thickness or the different imperfections (broken cell-walls, cell-edge and wall curvature etc.).

However Gibson and Ashby model defined by Eq. 1 with high value of φ =0.7 is much closer to the experimental values [12]. High value of φ means fraction of solid in the foam which is contained in the cell edges is higher than the remaining fraction (1- φ) in the cell walls. However none of the model could exactly define the compressive deformation behaviour of the material developed. Hence an empirical equation is used to fit the experimental data to correlate the normalized yield stress of the developed Al-MMC foam with its relative density (Fig. 5). The developed correlations for foam using untreated TiH₂ and pre-treated TiH₂ are:

a. Using Dual foaming agent, CaCO₃ & TiH₂ (untreated):

$$\frac{\sigma_{p^{t^{*}}}}{\sigma} = 0.11 \text{ RD}$$
 (7)

b. Using Dual foaming agent, CaCO₃ & TiH₂ (heat treated 450°C, 60 mins):

$$\frac{\sigma_{pl^*}}{\sigma} = 0.125 \,. \, \text{RD}$$
(8)

c. Using Dual foaming agent, CaCO₃ & TiH₂ (heat treated 480°C, 60 mins):

$$\frac{\sigma_{\mu^*}}{\sigma} = 0.175 . \text{ RD}$$
 (9)

d. Using Dual foaming agent, CaCO₃ & TiH₂ (heat treated 510°C, 60 mins):





Figure 5: Normalised yield strength (σ_{pl}/σ_s) of aluminium foam of different relative density RD (ρ_0/ρ_s)

Similarly, the elastic modulus E^* of the foam is related to the elastic modulus of the cell wall material Es according to Gibson and Ashby [1]:

$$= \phi^2 RD^2 + (1 - \phi). RD$$
 (11)

Similar efforts have been made by Andrew et al. [5] and Simone as:

$$= 0.33 \text{ RD}^2 + 0.32 \text{ RD}$$
 (12)

The correlation of normalized elastic modulus and relative density as presented by Kraynik [13] is:

$$= 0.311 \, \text{RD}$$
 (13)

The normalized compressive elastic modulus are also measured and compared with the existing theoretical models. Similar to the findings of the normalized compressive yield stresses of the developed material and the theoretical model. The theoretical results seem to be higher compared with the current work. Such difference can be attributed to the fact that the model assumes identical cube cells, without considering the distribution of the actual cell shape, cell-size, cell edge and cell-wall thickness or the different imperfections (broken cell-walls, cell-edge and wall curvature etc.).

Untreated TiH2		TiH2 at 450C 60 mins		TiH2 at 480C 60 mins		TiH2 at 510C 60 mins	
RD	Elastic Modulus (GPa)	RD	Elastic Modulus (GPa)	RD	Elastic Modulus (GPa)	RD	Elastic Modulus (GPa)
0.1522	0.0158	0.1162	0.0066	0.1015	0.0062	0.1272	0.0097
0.1718	0.0274	0.119	0.0073	0.1176	0.0116	0.1225	0.0082
0.1937	0.0472	0.143	0.0161	0.1265	0.0158	0.147	0.0181
0.1988	0.0529	0.1624	0.0278	0.132	0.0189	0.153	0.0216
0.2	0.0545	0.1832	0.0467	0.15	0.0326	0.179	0.0427
0.2015	0.0562	0.19	0.0546	0.158	0.0407	0.188	0.0528
0.2111	0.0693	0.2047	0.0753	0.187	0.0832	0.2005	0.0699
0.2151	0.0761	0.21	0.084	0.198	0.1061	0.205	0.077
0.2177	0.0795	0.2125	0.0884	0.2015	0.1143	0.211	0.0873
0.2267	0.0959	0.22	0.1026	0.215	0.1506	0.22	0.1047

 Table 2: Compressive elastic modulus of Al-MMC foams of different relative density.

A set of empirical equations are used to fit the experimental data for Untreated TiH₂ and pretreated TiH₂ are:

a. Using Dual foaming agent, CaCO₃ & TiH₂ (untreated):

$$- = 1.1 \text{ RD}^{4.5}$$
 (14)

b. Using Dual foaming agent, CaCO₃ & TiH₂ (heat treated 450°C, 60 mins):

$$-$$
 = RD^{4.3} (1.5)

c. Using Dual foaming agent, CaCO₃ & TiH₂ (heat treated 480°C, 60 mins):

$$-$$
 = 1.5 RD^{4.25} (16)

d. Using Dual foaming agent, CaCO₃ & TiH₂ (heat treated 510°C, 60 mins):

$$= 1.1 \text{ RD}^{4.35}$$
 (17)



Figure 6: Normalized Compressive Elastic Modulus (E^*/E_s) of aluminium foam of different relative density RD (ρ_0/ρ_s)

An important aspect of deformation mechanism can be inferred from Eq. 7-10, is that the linear density term dominates. This implies that cell wall stretching is the more significant mechanism during plastic deformation of this type of closed cell foams.

It can be interpreted from Eq. 14-17, that the non linear relative density term dominates, implying that cell wall bending is the more significant mechanism during the elastic deformation of this type of closed cell foams.

It is also observed that heat treatment of TiH₂ at 480°C for 60 minutes results in higher slope, which implies higher compressive strength for same RD value, thus, more usable foam (Fig. 3).

 TiH_2 is also pretreated for different time durations other than 60 minutes, but considerable enhancement in the mechanical properties are not found.

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IV. CONCLUSIONS

Uniaxial compressive tests are carried out find the compressive properties of Al- MMC foam. The material properties calculated based on existing theoretical models yield result which is higher compared with the experimentally measured properties of the developed material. The theoretical models therefore, inadequately describe the compressive properties of the developed Al-MMC foam. The reasons for this can be attributed to the fact that the model assumes identical cube cells, which is far from the actual case. An important observation that can be made from the comparisons of the experimental data is: pretreating of TiH₂ at 480°C for 60 minutes produces best result. In order to define the relationship of different compressive properties with relative density of this material, correlations which uniquely defines the compressive behaviour of this material has been developed. Such correlations are very much essential in order to properly design and application of this material.

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