Enhancing significantly the damping response of Mg using hollow glass microspheres while simultaneously reducing weight

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Abstract

Lightweight composite materials possessing higher damping capabilities are of great interests to material designers satisfying ever changing demands in automotive, aerospace and marine sectors. Besides having lowest density in metals regime, magnesium exhibits superior mechanical properties. Specific properties can still be enhanced by reducing the density further with development of magnesium based syntactic foams. Present work deals with processing and experimental characterization of glass microballoon (GMB) reinforced magnesium (Mg) composites. Hollow glass microspheres (5, 15 and 25 wt.%) reinforced magnesium syntactic foams were synthesized in magnesium matrix using the disintegrated melt deposition (DMD) method and their damping properties are investigated. The addition of glass microspheres enhanced the damping and loss factors by 370% and 12.5 times respectively for the highest filler loading as compared to pure magnesium. Further, increase in damping is correlated with microstructural changes arising due to the presence of the hollow glass microspheres. Elaborate discussion is presented on underlying mechanisms and different phases formed during processing. Copyright © 2017 VBRI Press.

Keywords: Magnesium, syntactic foam, glass microsphere, damping behaviour.

Introduction

Damping is a measure of materials ability to prevent or absorb vibrations and dissipate the energy efficiently during mechanical disturbances [1]. Usage of high damping capability materials are essential in maintaining the structural integrity in automotive, aerospace and marine structures, heavy machineries and electronic components owing to dynamic loading conditions and temperature variations during their service life [2-4]. Increased demand for lightweighting, drives the interest for developing newer utilitarian low density materials to achieve substantial weight savings, increased payload capacity and overall reduction in fuel consumption. Efforts are being pursued rigorously in developing lightweight high performance materials that are tailored to exhibit good mechanical properties and high damping [5, 6]. Owing to their lower densities, Aluminium ($\rho = 2.7$ g/cc) and Magnesium ($\rho = 1.74$ g/cc) are most sought after materials in design of several dynamic structures in semiconductor equipment, aerospace and defence sectors [7].

Magnesium is more preferred as it has the lowest density of any structural metal along with high specific strength [7]. Material selection in dynamic loadings is primarily based on specific stiffness (E/ρ) , replacing magnesium with aluminium results in lower values of mass and inertia as both metals have similar specific stiffness. Further, damping capacity of pure magnesium is almost 10 times higher than that of pure Al [8] and 100 times higher as compared to 316 stainless steel [9]. These exceptionally higher damping values of magnesium helps in dissipating energy effectively in automotive and electronic components, minimizing fretting damages [10]. Recently magnesium has gained considerable interest in development of lightweight biodegradable implants due to lower modulus, biocompatibility and bioresorbability with no toxicity post implantation [11]. Hence, developing biomedical implants and fixation devices with high damping capacity aids in mitigating vibrations caused during ambulation suppressing stresses developed at the bone/implant interface to achieve better osseointegration [12]. However, complex processing routes, lower corrosion resistance and mechanical

properties limits wide scale adaptability **13**, **14**]. This fact necessitates efforts to be put in towards enhancing mechanical properties of magnesium based material systems.

Magnesium matrix composites (Mg-MMCs) are the potential materials systems designed by addition of TiC, SiC, Al₂O₃, B₄C, SiO₂, graphite powder, carbon fibers and metastable reinforcements into magnesium matrix to realize improvements in both, strength and damping capabilities [2, 9, 12, 15-17]. Moreover, addition of such hard, high damping reinforcements in the matrix can modify the microstructure of metals and alloys altering energy dissipation sources [2]. The overall damping enhancement of magnesium alloys and composites can be attributed to several mechanisms like microstructural defects, porosity, intrinsic damping capability of the particles, thermo-elastic damping, grain boundary damping, thermal mismatch between the particulate and the matrix, and interface damping [5]. Understanding the underlying mechanisms becomes essential in development of newer materials like in magnesium foams.

Cellular metallic materials (metal foams) are special class of materials that are gaining increased interest due to same or higher damping properties in comparison with their fully dense structure [18]. High damping properties of metal foams can be attributed to the increased inhomogeneities near the cell walls in the matrix, enhanced interaction between the microscopic defects and the macroscopic cell walls under cyclic loads and modified microstructures of the matrix [19, 20]. Porous magnesium and their alloys are expected to be superior in energy decapitations as compared to their aluminium counter parts due to unique compressive deformation mechanism and inherently higher damping values of magnesium [20, 21]. Also, damping capacity of the porous metals can be further strengthened if the dislocation density in the cell walls is increased [22]. Higher dislocation damping capability of magnesium makes it to be the most promising material system which can be exploited further with advent of syntactic foams [20]. Porous foams also called as open cell foams lacks in strength and modulus which has limited their application areas. Whereas, syntactic foams are closed cell foams having superior mechanical properties due to closed cell structures. In these foams, instead of embedding air/gas voids, hollow particles are dispersed uniformly in the matrix. Syntactic foams offer an interesting and wide spectrum of properties, like lower densities, higher energy absorption and better vibration dampening capacity. It has the potential to replace most of the conventional metallic foams based on its specific performance [23].

Recent review articles have detailed the synthesis methods, microstructures and mechanical properties and applications of magnesium and aluminum matrix syntactic foams [24, 25]. Engineered hollow spheres of SiC and Al₂O₃ [26, 27] and inexpensive fly ash cenospheres [28-31] have been used as fillers in developing syntactic foams. Processing challenges in magnesium composites pose difficulties in developing

lightweight foams of them. Further, to the authors best of knowledge, damping behaviour of such magnesium foams is not available in the literature. Volume fraction of hollow microballoons, phases formed during processing, energy dissipation mechanisms involved due to enclosed porosity might influence damping properties significantly in Mg based foams. Thereby, in the present work, hollow glass microballoons are reinforced in Mg matrix and investigated foe damping characterization. Recently, AZ91D/SiC hollow particle reinforced syntactic foams investigated for damping properties are [**26**]. Higher damping capacity is observed as compared to matrix alloy due to dislocation based (presence of SiC hollow particles) and elastothermodynamic damping. Nevertheless, this study dealt with an Mg alloy increasing processing complexities and quantification of additional phases formed.

Reinforcing pure Mg with glass microballoons using DMD route of processing is quite challenging and interesting task, hence adopted in this work. Mg/GMB foams are investigated for damping response and the influence of filler loading analyzed. GMB is varied in Mg matrix by 5, 15 and 25 wt.% during DMD processing route. Pure Mg samples are also casted for comparison. Structure-property correlations are elaborately discussed with micrography.

Experimental

Materials

Magnesium in turnings form (ACROS Organics, New Jersey, USA) with 99.9% purity is used as matrix material. Hollow 'GMB particles with a mean particle dia. 11 μ m having density of ~ 1.05 g/cc procured from Sigma Aldrich, Singapore, are used as reinforcing filler.

Processing

DMD technique is used to synthesize Mg/GMB syntactic foams. Pure Mg turnings with weighed amounts of GMB's depending on the foam composition are heated to 750 °C in argon atmosphere. Uniform dispersion of hollow GMB's in Mg matrix is ensured by stirring the slurry at 465 rpm for 5 min [**32**]. Thereafter, the molten metal is bottom poured into a metallic mould in the presence of argon gas flowing at 25 lpm flow rate to disintegrate the melt steam. Cylinder of 40 mm diameter is obtained which is subsequently trimmed to required dimensions to conduct damping tests. All the samples are coded with Mg-XX convention, where XX represents weight % of GMB. FIVE replicates are tested of each composition and the average values are reported.

Characterizations

Density measurements are performed in accordance with Archimedes' principle on FIVE samples. Distilled water is used as the immersion fluid. The samples were weighed using an A&D ER-182A electronic balance having an accuracy of ± 0.0001 g.

The coefficients of thermal expansion (CTE) of the pure magnesium and foam samples are determined by measuring the displacement of the samples as a function of temperature in the temperature range of 50-350 $^{\circ}$ C using an automated TMA PT1000 thermo-mechanical analyzer.

Microstructural characterization studies are conducted on metallographically polished samples to investigate morphological characteristics and presence of second phases, if any. A Scanning Electron Microscope (JEOLJSM-6010) equipped with an Energy Dispersive Spectrometer (EDS) is used to perform micrography.

Elastic modulus and damping characteristics of magnesium and their foams with dimension of $\Phi7\times60$ are estimated using the resonant frequency and damping analyser (IMCE, Belgium) as outlined in ASTM E1876-09. The sample is freely excited by a light impact to bending vibrations of the first mode at low amplitudes. Two polymer wires support the sample, which is placed in the nodes of the vibration mode, to avoid background damping. The specimen vibration is recorded by a microphone. The software detects the resonance frequency for calculating the elastic modulus and the decay of the amplitude for calculating the loss factor.

Results and discussion

Microstructure

Micrography is carried out on as cast samples of Mg foams and is presented in Fig. 1. Micrography reveal uniform distribution of intact glass microspheres, secondary phase particles and presence of micro-voids at the particles/magnesium matrix interface. GMB/secondary particles are fairly well distributed and the magnitude of secondary phase formation is observed to increase with higher filler loadings. The hollow glass microspheres are noted to be having 8-13 µm in outer diameter and exhibited good variety of morphologies within the microstructure as seen from Figure 1a. Limited reaction zones are also clearly evident along the walls of the glass spheres, which could potentially lead to strong interfacial bonding between the foam constituents. Few GMB are seen to be fractured (Fig. 1b) during processing and further owing to embrittlement creeping in because of interface reactions between the Mg matrix and GMB's.

A careful investigation of the chemical reaction products during DMD process is also carried out by analyzing the presence of surface chemical elements using EDS (Figure 1d). Mg has a high reaction activity, therefore, when glass microspheres are added to the Mg melt, it is possible to undergo a chemical reaction between Mg elements and silica phase present in GMB. Thermodynamic computation indicates the following chemical reaction likely to happen:

$$\begin{array}{ll} 2Mg + SiO_2 \rightarrow Si + 2MgO & (1) \\ 2Mg + Si \rightarrow Mg_2Si & (2) \end{array}$$

EDS reveal presence of Mg_2Si as a secondary phase in Mg/GMB foam (**Fig. 1d**). Mg₂Si formed, can grow from the particle shell into the matrix.



0 1 2 3 4 5 6 Full Scale 137564 cts Cursor: 3.766 (373 cts)

Fig. 1. Representative microstructure of Mg-15 foam: (a), HGM showing uniform wall thickness (b), Mg₂Si dendrites present in the Mg matrix (c) and EDS result indicating the presence of Mg₂Si as secondary phases.

Diffusion of Si atom to Mg alloy might be cause for formation of Mg₂Si having two different morphologies, dendrite crystals and polygons. Mg₂Si dendrites formed are in the size range of 3-5 μ m. Very few GMB's are fractured owing to processing route followed as seen from micrograph in **Fig. 1a**. Mg matrix occupies the space created due to microballoon fracture and may compromise overall density of the foam.

Density measurement

Values of experimental densities based on the Archimedes Principle are presented in **Table 1**. The results show that addition of glass microsphere particles led to a significant reduction in density (13.4%). Also, it can be noticed that the experimental density values are lesser than that of theoretical ones, indicating GMB particles are intact in Mg matrix. It is also observed that, Mg/GMB foams synthesized in this study exhibit the densities closer to polymer based composites like in PE-30% fiber glass composite (1.429 g/cc) [**33**]. These facts make Mg foams suitability and usage in wider applications.



Fig. 2. Damping characteristics of Mg/GMB foams with the fitted curves for the evaluation of attenuation coefficients.

Elastic modulus and damping characteristics

Fig. 2 shows a set of amplitude-time plots of representative samples and Table 2 lists damping loss rate, damping capacity and elastic moduli of Mg and their foams. The vibration signal from each sample is recorded in terms of amplitude vs. time in free vibration mode. The results clearly indicate that the amplitude and time taken to stop the vibration are different for each materials and addition of glass microspheres significantly enhances the damping characteristics of pure Mg. It can be seen from **Fig. 2** that the amplitude decreases gradually for pure Mg as against steeper fall in Mg foams. With the addition of 5 and 15 wt.% GMB's in Mg matrix, time taken to damp the vibrations is reduced significantly from 0.52 to 0.22 and 0.18 s, respectively. Most of the vibrations are ceased in less than ~0.1 s with highest filler loading.

Material	Theoretical density (ρ _{ct}) (g/cc)	Measured density (ρ _{ce}) (g/cc)	Matrix porosity (vol.%)	CTE (μ/°C)
Mg	1.738	1.7014±0.002	2.1	27.1
Mg-5	1.686	1.6739±0.015 (↓1.6%)	0.72	24.2 (↓10.7%)
Mg-15	1.588	1.5597±0.010 (↓8.3%)	1.78	22.7 (↓16.2%)
Mg-25	1.502	1.4723±0.018 (↓13.4%)	1.98	21.2 (↓21.7%)

* ($\downarrow x\%$) indicates the decrease in the property with respect to pure Mg by x%.

The sinusoidal damped vibration equation is expressed as [34],

$$y(t) = Ae^{-kt}\sin(\omega t + \varphi)$$
(3)

where 't' is the time; 'A' is the initial amplitude; 'k' represents the decay constant which is material dependent property; ' ω ' denotes angular frequency; ϕ is the phase angle and frequency, 'f' is the number of cycle per time unit equals $\omega/(2\pi)$. Damping factor is estimated as,

$$Q^{-1} = k/\pi f \tag{4}$$

Damping loss rate is a function of sample weight (m) and the average ratio of two adjacent peaks from amplitude-time plot. Damping loss rate is expressed as,

$$\partial = \frac{1}{m} \ln \left(\frac{y_1}{y_2} \right) \tag{5}$$

Resonant frequency and damping analyser (IMCE, Belgium) is used to conduct the damping test. The damping loss rate (L) which is the ability of a material to absorb vibration [9], showed an increase with the addition of glass microspheres (**Fig. 3a**) and Mg-25 exhibited the maximum value of ~108.2 (~12.5 times greater than that of pure Mg). Damping loss rate as a function of weight percent (W_r) of the hollow reinforcement follows a linear fit and can be expressed using Eqn.6 as,

$$L = 9.6373 + 3.9278X \text{ (wt.\%)} (R^2 = 0.9989)$$
(6)





Fig. 1. Linear fitted curves for: (a) Damping loss rate, and (b) Damping capacity as a function of filler content.

Damping capacity (Q^{-1}) of Mg also seen to be increasing with the increasing filler loadings (Figure 3b) and is expressed using Eqn. 7. Mg-25 Highest damping capacity of 0.002560 (~370% rise as compared to pure Mg) is shown by Mg-25 foam.

$$Q^{-1} = 0.0006 + 8E - 05X \text{ (wt.\%)} (R^2 = 0.993)$$
 (7)

A more qualitative evaluation of damping would be by considering the attenuation coefficients. In magnesium and its alloys, the amplitude of free vibration gradually decreases with increase in time and the difference lies in the steepness of the curve which is quantified by the attenuation coefficient. In this study, the material vibrates at a resonant frequency when excited by cyclic external force and when this external force is removed, the resonant-vibration dampens gradually. Then, amplitude of a damping vibration, A(t), can be expressed as

$$A(t) = A_o e^{(-\alpha)t} + C$$
(8)

where, 't' is the time after removal of the external force, 'A₀' denotes the amplitude at t=0 and ' α ' represents the apparent attenuation (damping) coefficient (which depends on the damping capacity of materials) and 'C' is the fitting coefficient. An increasing trend in α is clearly evident from **Fig. 2** with increasing amount of 'GMB's. A notable enhancement in the value of α from 7.365 to 86.04 is observed in case of Mg-25 foam as compared to pure Mg demonstrating significant rise in foams damping capability.

Foams exhibit lower modulus with increasing GMB content (**Table 2**). Uniform distribution of reinforcement, relative moduli difference of the constituents and interfacial bonding between them prompts higher elastic moduli [**12**, **35**]. Syntactic foam modulus is found to be much lower than that of the matrix material in case of lower moduli microballoons. Nevertheless, it is interesting to note that, the damping capacity of the Mg foams increases with the decreasing density and Young's

modulus, whereas in case of monolithic alloys the damping capacity decreases. Similar observation are noted in Invar (FeNi36) matrix syntactic foams reinforced with GMB's [23]. This unique behaviour of Mg foams helps in tailoring structural and functional properties. Specific properties might yield superior performance as compared to other metallic systems affirming Mg foams feasibility in weight sensitive applications. In Higher damping for Mg/GMB foams might be due to presence of a plastic zone, increase in dislocation density and due to other damping sources, such as defects and porosities.

Table 2. Elastic modulus and damping characteristics of Mg-glass microsphere syntactic foams.

Material	Damping loss rate	Damping capacity	Elastic modulus (GPa)
Mg	8.6 ± 0.4	0.000546 ± 0.000032	43.3 ± 0.16
Mg-5	31.2 ± 0.7 (*3.6)	0.00098 ± 0.000017 (↑79%)	$\begin{array}{c} 42.56 \pm 0.28 \\ (\downarrow 1.7\%) \end{array}$
Mg-15	67.3 ± 1.9 (*7.8)	0.001631 ± 0.00042 (†198%)	$\begin{array}{c} 41.10 \pm 0.18 \\ (\downarrow 5.1\%) \end{array}$
Mg-25	108.2 ± 2.3 (*12.5)	$\begin{array}{c} 0.002560 \pm 0.0005 \\ (\uparrow 368\%) \end{array}$	$\begin{array}{c} 39.85 \pm 0.12 \\ (\downarrow 7.9\%) \end{array}$

* (*x) indicates the increase in the property with respect to pure Mg by x times. ($\uparrow x\%$) and ($\downarrow x\%$) indicates the increase and decrease in the property with respect to pure Mg by x%, respectively.

The CTE of GMB and magnesium is 8 [36] and 27.1 μ / °C respectively. This distant difference of thermal expansion coefficient between Mg and GMB might induce high residual stresses around the filler in the Mg matrix, resulting in the formation plastic deformation zones at the particle/matrix interface. Plastic zone damping proposed by Carreno-Morelli et al. [37] discusses dependency of damping on the volume fraction of plastic zone and the strain amplitude. Progressive increase in the energy dissipation of pure Mg matrix is attributed to the higher amount of plastic zone around GMB's and further, at higher filler loadings effects are multifold resulting to such a rise in damping capacity of foams. Thermal mismatch between the constituents leads to higher dislocation density in matrix. The increase in dislocation density is given as follows [38],

$$\rho = \frac{9.6 \,\Delta \alpha \Delta T V_p}{bd} \tag{9}$$

where, ' $\Delta \alpha$ ' is the thermal expansion coefficient mismatch between matrix alloy and the filler, ' ΔT ' is the difference between working and final temperatures, 'b' is denoted by the Burger vector, ' V_p ' represents volume fraction, and 'd' is the diameter of reinforcement. CTE difference between GMB and Mg is around 19.1µ/ °C. Formation of dislocation density can be quite significant at the interface and it increases with increasing GMB content. For magnesium-based materials, increased dislocations are favourable for damping enhancement as

dislocation pinning contributes to higher damping [10]. In addition, the crystal structure will be distorted locally at the matrix/reinforcement interface due to the presence of two-dimensional defects at the interface [12]. Thereby, atoms may slip up at the interface resulting in flexible dislocation movement leading to higher damping response [39].

Further, formation of harder Mg₂Si secondary phases poses higher constraints to localized matrix deformation leading to increased elastic strain energy dissipation sources. The origin of these dislocations can also be attributed to the difference in CTE values of Mg and Mg₂Si (7.5 μ / °C) [40] and leads to the higher stresses in Mg matrix around Mg₂Si phase. These high stresses are partially relieved during solidification by dislocation formations. The amount and the distribution of Mg₂Si phase might markedly influence the dislocation network in Mg foams with subsequent increase in dislocation density with higher content of Mg₂Si. Presence of Mg₂Si phase enhances the damping capacity of Mg alloys [8, 41].

In addition, it has also been observed that defects play an important in tailoring damping properties. Chung [42] suggested that defects may shift the locations during vibration, acting as internal friction resources leading to higher damping capacities. Though each pore of syntactic foams is reinforced by the stiff shell of the hollow particle, microscopic pores still exist in the walls of these particles [43]. Further, micro-cracks might get formed during cooling from melt temperature due to the differing shrinkage behaviour of matrix and glass spheres [44]. As a result, damping mechanism is dominated by internal friction in small cracks and dislocation movement due to stress concentration in certain areas of the foam structure. Further, more defects such as interfacial and the particle boundary arise from a rise in glass microsphere content and can lead to more microplasticity deformation [45] owing to stress concentration around them, improving the damping behaviour of Mg/GMB foams. Relative displacement of GMB's is relatively easier at the regions wherein matrix porosity exists, further increasing the damping capacity. Matrix porosity augments the damping capacity due to the heterogeneous stress-strain distribution causing stress concentrations resulting in higher dislocation movements [22]. From Table 1, it can be seen that increase in the addition of glass spheres increased the porosity levels of Mg/GMB foams. Highest damping capacity is observed for Mg-25 foam which has maximum matrix porosity levels as seen from Table 1. The hollow GMB particles, are expected to have higher damping capacity than solid particles and damping performance of syntactic foams is found to be comparable or superior than those of commercially available alloys and conventional metal matrix composites reinforced with solid particles. Mg/GMB foams presented in this study has the capability to replace commercial magnesium alloys and MMCs where density and damping behaviour are the material selection criterion.

Conclusions

Based on the present study, following conclusions may be drawn:

- 1. Mg matrix syntactic foams with hollow glass microsphere reinforcement can be successfully synthesized using disintegrated melt deposition technique and Mg-25 foam exhibited an average density of 1.47 g/cc similar to polymers signifying its weight saving potential.
- 2. The developed syntactic foams were more dimensionally stable as compared to pure Mg.
- 3. Damping loss rate of Mg-25 foam increased by 12.5 times when compared to pure Mg and exhibited a linear relationship with increasing amount of reinforcement.
- 4. Damping capacity of Mg-25 foam increased by 370% when compared to pure Mg and exhibited a linear relationship with increasing amount of reinforcement.

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