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Epoxy-glass Microballoon Syntactic Foams for Blast Mitigating Applications

A.V. Ullas[!,@], P.K. Sharma[!], P. Chandel[#], P. Sharma[#], D. Kumar[#], D. Kumar[@], and P.K. Roy[!,*]

[!]*DRDO-Centre for Fire, Explosive and Environment Safety, Delhi -110 054, India*
[@]*Department of Applied Chemistry and Polymer Technology, Delhi Technological University, Delhi -110 042, India*
[#]*DRDO-Terminal Ballistic Research Laboratory, Chandigarh - 160 003, India*
[*]*E-mail: pk_roy2000@yahoo.com*

ABSTRACT

Polymeric syntactic foams refer to a class of cellular material created using preformed hollow spheres bound together with a polymeric matrix. These cellular materials possess exceptional ability to respond against high impact dynamic loads. This paper is an attempt to fabricate polymeric syntactic foams of epoxy containing hollow glass microballoon at varying loading (40 % - 60 %) and explore their potential towards blast mitigation. The tensile, compressive and flexural strength were found to be inversely proportional to the microballoon loading in the quasi-static regime. The strain rate sensitivity of the foams was confirmed by performing high strain rate studies using split hopkinson pressure bar. The flow stress of these foams was found to increase with increasing strain rates. The syntactic foams were subjected to controlled transient blast loadings using a shock tube. The samples remained intact and no strain was observed on the strain gauge, even under a blast load of ~ 90 psi, which clearly highlight their potential as core materials for blast mitigating applications.

Keywords: Syntactic foams; High strain rate; Blast load; Shock tube

1. INTRODUCTION

Syntactic foams belong to a subclass of composite cellular materials prepared by dispersing hollow micro balloons in a matrix of polymer, ceramic or metal[1,2]. The presence of a large proportion of gaseous phase in these composites lead to significant weight reduction, and impart special mechanical properties which vastly differs from the bulk materials. These cellular composites are finding diverse applications ranging from deep sea to space vehicles[3] especially as energy absorbing core materials in sandwiched structures[4]. The presence of soft condensed matter helps reduce the damage arising from impact and other transient loads[5]. Although, both open and closed cell foams have been effectively utilised in their ability to resist blast/dynamic loads[6,7], the latter owing to the presence of entrapped air, forbears additional deformation at high strain rates and increase the compressive strength and energy absorbing ability of the foams[8].

Glass microballoons have been extensively used for syntactic foam manufacturing because of their high strength and low density[9]. Additionally, these microspheres act as independent energy absorbing loci on being subjected to compressive loads. Potential applications of syntactic foams as core materials, utilising their ability to act as energy absorbers, include them being used in the underbelly of tanks to attenuate the blast effects arising out from anti tank mines or other such applications where the ability to mitigate the effects of explosion are vital. Sandwich cores have found place in the literature[8,10], most important of them being balsa and foam cores in various naval applications such as surface ship deck structures and in boat hulls[11,12].

Rigid polymeric foams, usually prepared by the chemical reaction of isocyanates with polyols, are not strong enough to withstand blast loads and therefore mandate reinforcement[13]. Also, the processing of polymer-based foams is an involved process with interfacial phenomena and fluid dynamics being very critical[14,15]. In the field of defence technologies, in view of their excellent mechanical properties syntactic foams have been reported to withstand dynamic loads[16]. It is worth mentioning that blast effects, fall into the category of high impact dynamic loading which are characterised by the occurrence of high amplitude and high frequency waves which act on the structures for a very short period of time[17].

The aim of this study is to develop epoxy based syntactic foams and evaluate their behaviour both at quasi static and high strain rates using a Split Hopkinson pressure bar. Further, their response on being subjected to controlled transient loading has been studied on a shock tube facility. The aim is to explore the potential of these syntactic foams as core materials for energy absorbing applications.

2. EXPERIMENTAL

2.1 Materials and Methods

Cycloaliphatic epoxy resin (Ciba Geigy, Araldite CY 230; epoxy equivalent 200 eq g^{-1}), hardener (HY 951; amine content 24 eq kg^{-1}) and Hollow glass microballoons (K46, 3M) were used for the preparation of syntactic foams. The HGMs used have a true particle density of 0.46 g/cm^3, mean diameter of 40 µm (wall thickness 1.29 µm) and isostatic

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crushing strength of 6000 psi.

2.2 Preparation of Syntactic Foams

Epoxy syntactic foams containing varying loadings of hollow glass balloons (40 %v/v, 50 %v/v, and 60 %v/v) were prepared as per the procedure reported previously[3]. In brief, theoretically calculated amounts of HGM was added to the epoxy resin and the contents were mechanically mixed and degassed followed by introduction of calculated amounts of triethylene tetramine. The viscous slurry hence obtained was transferred to silicone moulds and cured for 48 h at room temperature to prepare specimens for mechanical testing.

The mass of HGM to be added was calculated as follows:

$$\frac{Mass\ of\ HGM}{Mass\ of\ Syntactic\ foam} = \frac{\rho_{HGM} \times \Phi_{HGM}}{\rho_{HGM} \times \Phi_{HGM} + \rho_m \times \Phi_m}$$

where ρ and Φ refer to the density and volume fraction of the constituent respectively, and the subscripts *HGM*, and *m* refer to microballoons and epoxy respectively. For the purpose of calculation, density of HGM and epoxy have been assumed to be 0.46 g cc^{-1} and 1.17 g cc^{-1}[3], respectively. The syntactic foams were designated as *Sx*, where *x* refers to the microballoon loading (%v/v). For example, S40 refers to a formulation containing 40 % HGM and 60 % epoxy. The air-void porosity trapped within the foam was determined as the ratio of the difference in the theoretical (ρ_{th}) and experimental densities (ρ_{ex}) to theoretical density[2], as per the standard procedure.

$$\text{Void volume } (\%) = \frac{\rho_{th} - \rho_{ex}}{\rho_{th}} \times 100$$

The theoretical density was calculated using the standard rule of mixtures.

$$\rho_{th} = \rho_{HGM} \times \Phi_{HGM} + \rho_m \times \Phi_m$$

The experimental density (ρ_{ex}) was determined experimentally by averaging the mass : volume ratio of five specimens per sample as per the standard method ASTM D1622–98.

2.3 Characterisation

The thermal behaviour was investigated using Perkin Elmer Diamond STG-DTA under N_2 atmosphere in the temperature range 50-600 °C. A heating rate of 10 °C/min and sample mass of 5.0 ± 0.5 mg was used for each experiment. Rheological studies were performed for HGM-epoxy formulations using an Anton Paar Rheometer (MCR 102) using 25 mm disposable aluminium parallel plates. Shear rate sensitivity studies were performed, while varying the same over 0.1 s^{-1} to 1 s^{-1} under isothermal conditions (30 °C). The morphology of fractured surface was studied using a scanning electron microscope (Zeiss EVO MA15) under an acceleration voltage of 1 kV. Samples were mounted on aluminium stubs and sputter-coated with gold and palladium (10 nm) using a sputter coater (Quorum SC7620) operating at 10-12 mA for 120 s.

2.4 Mechanical Testing

Mechanical testing was carried out using Universal Testing Machine (International Equipment) at ambient temperature. For compression testing, standard cylindrical specimens (12mm diameter, 6 mm thick) were compressed at a rate of 1.3 mm min^{-1}. Five specimens of each composition were tested and the load-displacement data obtained from the tests was used to obtain stress-strain curves for calculation of compressive strength. Toughness, which refers to the area under compression stress-strain curve, was quantified as an index of the amount of energy absorbed by the foams[18].

$$\text{Energy absorption} = \frac{1(\rho_c \times \epsilon_c)}{2} + (\rho_c \times \varepsilon_c)$$

where ρ_c and ε_c refer to the compression yield strength and crushing strain of the foam, respectively.

Flexural testing of the samples was performed under three point bending mode as per ASTM D790. For this purpose, specimens of standard dimensions (127 mm length x 12.5 mm width x 3.5 mm thickness) were prepared and subjected to a deformation rate of 2 mm min^{-1} while maintaining a span length of 60 mm.

The tensile properties were determined as per ASTM method D638 using a Universal Testing System (International equipments) at ambient temperature. The dumb bell shaped specimens used for tensile testing were 165 mm long, 3 mm thick, and 13 mm wide along the centre of the casting for syntactic foams. The samples were subjected to a cross head speed of 10 mm min^{-1}.

2.5 High Strain Rate Testing

The mechanical response of the samples under high strain rates was investigated using a Split Hopkinson Pressure bar. The basic principle of SHPB is based on one-dimensional wave propagation in elastic bars, and since it is not possible in practice, the theory is adopted with certain approximations. The setup at Terminal Ballistic Research Laboratory (TBRL), Chandigarh comprises of two high strength maraging steel with yield strength 1750 MPa, diameter 20 mm and length 2000 mm. A projectile (300 mm length, 20 mm dia) is made to hit the cylindrical foam sample sandwiched between the two bars to produce different strain rates varying between 1376 s^{-1} - 2574 s^{-1}. Strain gauges of 120 Ω, 90° tee rosette precision strain gauges designated as EA-06-125TM-120 were used. For wave shaping a 1.5 mm OFHC copper wave shaper was used.

In all the experiments, cylindrical specimens with length to diameter (L/D) ratio of 0.5 were used, with the diameter of the specimen being 12 mm. This ensures the impact on full cross-section of the specimen and also permits the specimen to expand along radial direction within the cross-sectional area of the bars after the compressive load is applied.

2.6 Shock Tube Test

The dynamic response of the syntactic foams was evaluated by subjecting disc shaped specimens to controlled blast loadings on a shock tube. A shock tube assembly essentially comprises of a long rigid cylinder, sub-divided into a high-pressure driver section and a low pressure driven section, which are separated by a mylar diaphragm. The shock tube, used for the present study has an overall length of 8.7m;

divided into 1.7 m driver and 7 m driven sections. A cylindrical disc of syntactic foam specimen (12 cm diameter, thickness 1 cm) was sandwiched between two aluminium sheets (0.25 mm thickness), held under simply supported boundary conditions in front of the driven section. Pressurised air is used as the driver gas, the driven gas being ambient air. The rapid release of gas due to diaphragm rupture results in the creation of a shock wave, which travels down the driven tube to impart dynamic blast loadings. A circular region of 10 cm diameter was effectively subjected to blast loading, as pictorially shown in Fig. 1. Pressure sensor (113B21, PCB make), placed at a distance of 11.3 cm from the sample was used to monitor the pressure profile during the testing. Strain gauge (HBM:K-LY4-1-11-120-3-2) was affixed on the blast–opposing face of syntactic foam specimen to measure sample deflections.

The signal recorded on the strain gauge installed on the opposite face of the blast is indicative of the extent of deformation in the syntactic foam sample due to blast loading. A control set of experiments was also performed, which comprised of two aluminium sheets with no foam between.

3. RESULTS AND DISCUSSION

3.1 Rheological Studies

The effect of introducing glass micro balloons on the rheological behaviour of the resin is of particular interest for processing of the foams. The shear rate dependence of the formulation is presented in Fig. 2. Unfilled epoxy resin exhibits a viscosity of ~ 1000 mPa.s at 30 °C (shear rate =0.1 s^{-1}) and inclusion of microballoons lead to a considerable increase in its viscosity, the extent of which is proportional to microballoon loading(~10^5 mPa.s, 40 per cent microballoon loading). The viscosity-shear rate profiles of all the formulations are clearly indicative of the shear thinning nature of the composition.

3.2 Void age in Syntactic Foams

The ratio of experimental and theoretical densities was used to quantify the void age in each sample, and the results are presented in Table 1. As expected, an increase in the microballoon loading lead to a decrease in the density of the foams.

3.3 Thermal Properties

TG-DTG traces (under inert atmosphere) of syntactic foams are presented in Fig. 3. The profile associated with the degradation of epoxy was found to remain unaltered, except for the substantial increase in the char content, due to the presence of hollow microballoons. The thermogravimetric trace of neat

Table 1. Experimental and theoretical densities of syntactic foams

Sample designation	Density (kg/m^3)		Voids (% v/v)
	Experimental	Theoretical	
S40	859.3 ± 7	886	3.0
S50	775.3 ± 4	815	4.8
S60	712.7 ± 5	744	4.2

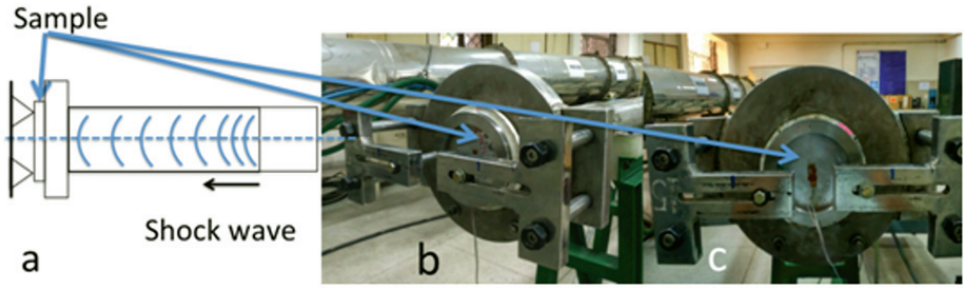


Figure 1. (a) Representation of the specimen loading on shock tube, (b) Specimen placement, and (c) Front view.

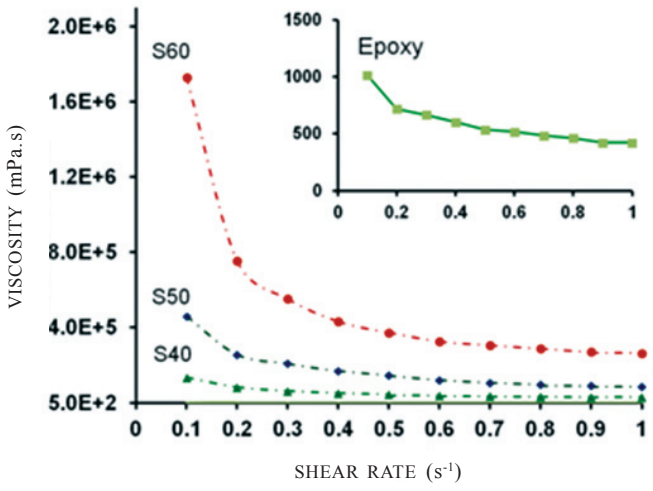


Figure 2. Effect of increasing shear rate on viscosity of syntactic foam formulations.

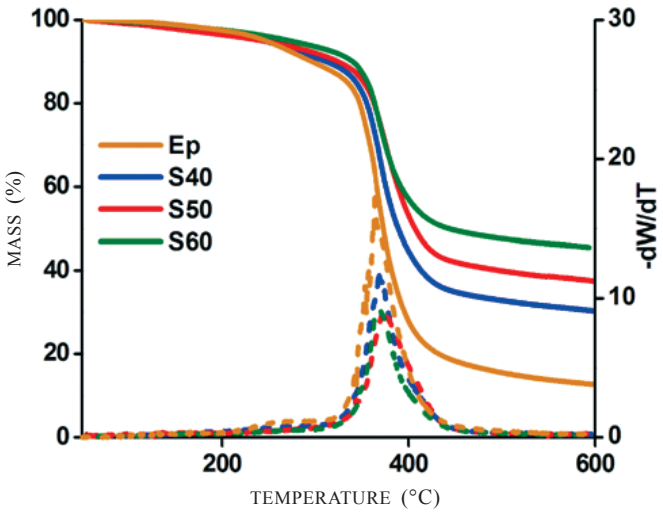


Figure 3. TG-DTG traces of syntactic foams with varying microballoon loading.

epoxy is also included in the figure for ready reference. For syntactic foams with 40 per cent microballoon loading, a char content of 29 per cent was observed (at 600 °C), a major fraction (~21%), being the incombustible glass microballoons.

3.4 Mechanical Properties

3.4.1 Quasi-static Testing

The effect of increasing microballoon content (40% v/v - 60 % v/v) on the mechanical properties of syntactic foams under quasi-static conditions is presented in Fig. 4. The stress-strain curves (compressive mode) are also included (Fig. 4(a)). In general, the lower compressive strength of microballoons (41 MPa) in comparison to neat epoxy (138 MPa)[19] is expected to result in a reduction of compressive strength, which is very well evidenced from our studies (Fig. 4(b)). It is to be noted that under low microballoon loading, the hollow constituents are well embedded within the resin. During compression, substantial energy is absorbed by the matrix as well as the microballoon-epoxy interface prior to the fracture of microspheres[20]. With increasing microballoon loading, the contact between the microballoons increases due to the reduction of resin content, leading to localised aggregation which leads to further reduction in the compressive properties.

Mechanical response was also determined in tensile and flexural mode, the results of which are included in Fig. 4(c). The precipitous drop in stress after the end of elastic region was indicative of the brittle failure in the sample. Flexural strength was also found to be inversely proportional to the microballoon loading. However, it is to be noted that under flexure, failure occurs primarily due to matrix fracture and is less dependent on failure of microballoons[21]. The tensile properties, which depend primarily on the response of the matrix rather than on the filler i.e. glass microballoons[2], are presented in Fig. 4(d). It can be seen that the tensile properties are also inversely proportional to the microballoon loading[22]. It is to be noted that the properties of syntactic foams are strongly dependant on the wetting and interfacial bonding between microballoon and the resin[23]. Further improvement is possible by surface treatment of the glass microballoons[24,25].

The microstructure of syntactic foams was studied using a scanning electron microscope. The SEM image of syntactic foam specimen (S40) post quasi-static testing (both compressive and tensile) is presented in Fig. 5. Embedded hollow glass microballoons are observed in the image. During compression, glass microballoons undergo extensive crushing which results in the failure of the foams (Fig. 5(a)). On the other hand, tensile failure is brought about by the failure of matrix (Fig. 5(b)). Flexural failure occurs due to a combination of tensile and compressive forces and results in microballoons debonding as well as matrix failure.

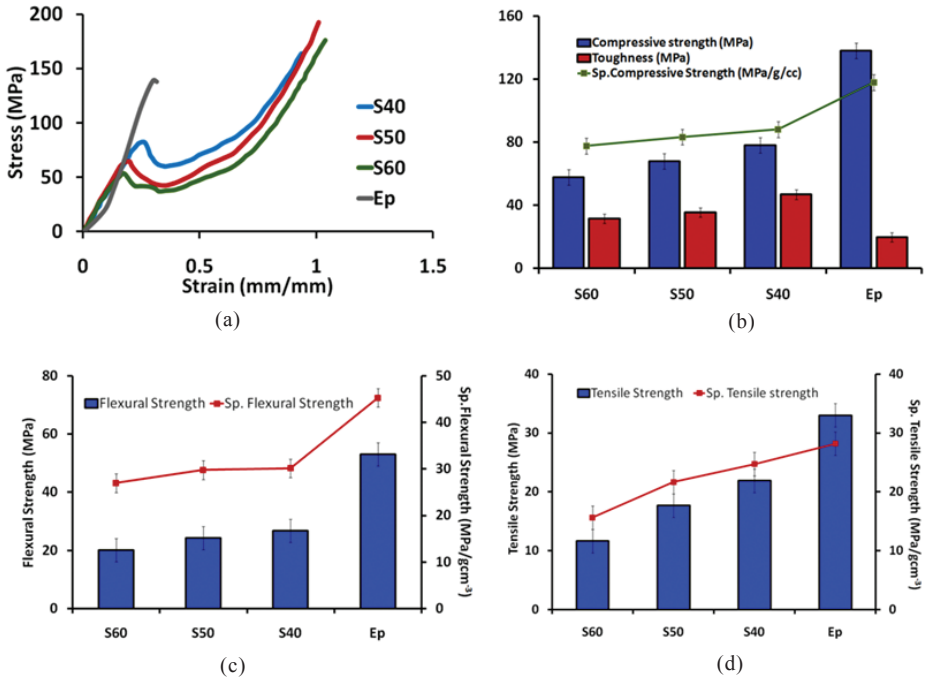


Figure 4. (a) Stress-strain curves (Compressive mode), b) variation of compressive, (c) flexural and (d) tensile properties of syntactic foams as a function of microcapsule loading.

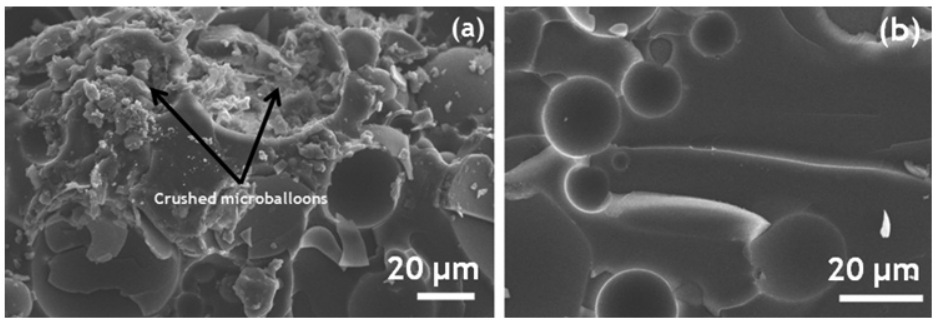


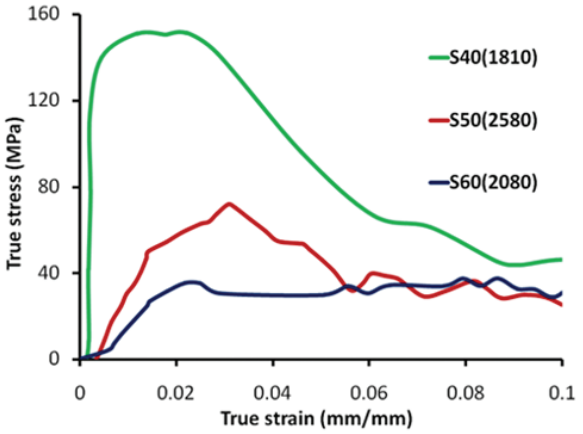
Figure 5. (a) Compressive and (b) tensile fracture features of epoxy syntactic foam.

3.4.2 High Strain-rate Studies

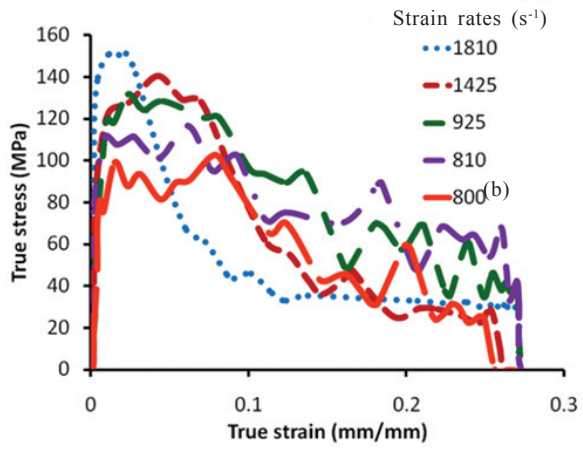
The effect of increasing strain rates on the compressive behaviour of syntactic foam was quantified using Split Hopkinson Pressure bar setup, which consists of input and output bar sandwiching a short specimen[26,27]. The details of the setup are presented in the published paper[19]. Pressurised air propels a projectile, which strikes at one end of the input bar. A compressive stress wave is generated, which upon hitting the specimen, is partially transmitted and reaches the output bar while a part of it is reflected back to the input bar. In the present case, this propagation of the compressive wave leads to an irreversible plastic deformation in the specimen. The reflected pulse propagates as a tension wave, while the transmitted pulse remains in compression. The wave signal responses are quantified using

strain gauges attached on the input and output bars. The stress – strain profile obtained for different samples are presented in Fig. 6. Representative curve for each loading is presented for the sake of comparison of the results. It is to be noted that under high strain compressive testing, the strain rate rapidly increases during the initial stages of loading; therefore it was not attempted to estimate the Young's modulus from the strain gauge data. As in the case of quasi-static testing, the flow stress (maxima) decreased with increasing microballoon loading (38MPa S60 (ε =2080 s^{-1}), 71 MPa, S50 (ε =2580s^{-1})and 151 MPa, S40, (ε =1810s^{-1}).

The mechanical response of the specimens under high strain rate was found to follow the same order as under quasi-static testing, however a distinct difference in terms of the absence of a stress plateau was clearly evidenced. In line with the quasi-static tests, decrease in microcapsule loading leads to a proportional increase in the maximum flow stress. The strain rate sensitivity of a representative formulation (S40) is presented in Fig. 6(b). In line with previous studies[28], the flow stress was found to increase proportionally with increasing strain rates. The fracture mechanics of syntactic foams has been extensively studied, wherein significant dependence of the compressive failure upon the strain rate has been reported[28].



(a)



(b)

Figure 6. (a) Stress-strain profile of syntactic foams under high strain rates (~1380-2580 s^{-1}) and (b) effect of increasing strain rate on a representative syntactic formulation (S40).

The failure has been reported to occur via varied mechanisms. At low strain rates, the foams have been reported to undergo shear cracking due to crushing of the microballoons whereas at higher strain rates, the failure occurs primarily due to crack propagation in the direction of compression.

3.4.3 Shock Tube Studies

The dynamic response of the syntactic foams was evaluated by subjecting circular disc shaped specimens to controlled transient loadings on a shock tube. The pressure profile along with the peak pressure, impulse as well as the response of the syntactic foam in terms of strain deformation is presented in Table 2.

It can be seen that the control set (aluminium sheets) undergo extensive deformation due to blast loadings (35.5 psi). In comparison, the syntactic foam specimens do not undergo any deformation even when subjected to larger over pressure. A photograph of the S40 specimen post-blast loading (~89.6MPa) is presented in Fig. 7. It can be seen that the sample is intact, which clearly highlights the potential of syntactic foams, as core material in sandwich configuration for blast mitigating applications.

S40 syntactic foams were repeatedly subjected to blast pressures of ~90 psi. The post-blast visual of the sample exposed to 5 repetitive shock loadings is presented in Fig. 8. It can be seen that the sample underwent chipping, with no visual evidence of microballoon crushing.

4. CONCLUSIONS

Polymeric syntactic foams were prepared by introducing

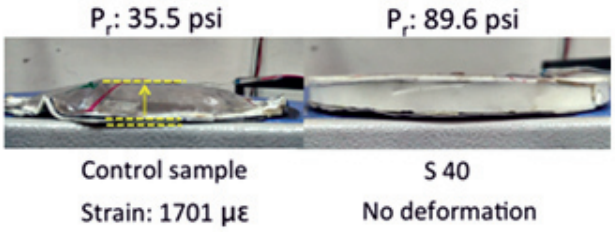
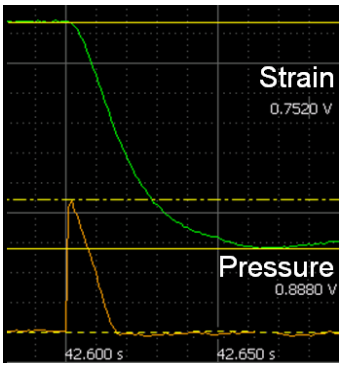
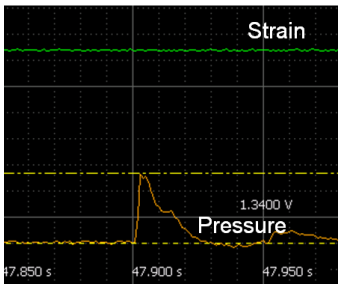
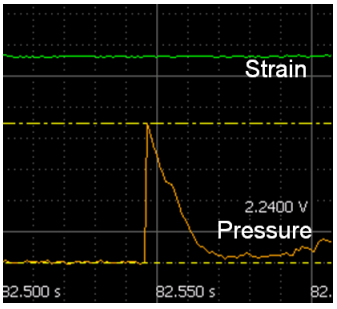


Figure 7. Post-blast visuals of control and S40 syntactic foam.

Figure 8. Post-blast visuals of S40 syntactic foam after 5 repeated blast loadings (P_r~90psi).

Table 2. Response of syntactic foam under blast loading conditions

Sample	ΔP (Driver pressure-driven pressure)	Pressure/ strain profile	Peak pressure (psi)	Sample response
Control 2 Aluminium sheets	2		35.5	Extensive deformation observed. Strain: 1701 με
S40 (1cm) sandwiched between 2 Aluminium sheets	5		53.6	No strain observed. Sample intact post-blast
S40 (1 cm) sandwiched between 2 Aluminium sheets	15		89.6	No strain observed. Sample intact post-blast

K46 hollow glass microballoons at varied loading (40 %v/v - 60 %v/v) into a cycloaliphatic epoxy resin. Rheological studies were performed to establish the dependence of viscosity on increasing shear rates. Increasing the loading of glass microballoons led to a proportional increase in the viscosity of the formulation, however all the formulations exhibited a shear thinning behaviour. The zero shear viscosity increased from ~ 10^3 MPa.s to 10^5 MPa.s at 30 °C as the microballoon loading was increased to 40 %v/v. Mechanical properties of the foamed composites was evaluated both at quasi-static (tensile, compressive and flexure) and high strain rates. The difference in the crushing strength of glass microballoons (41 MPa) and cured epoxy (138 MPa) reflected in terms of a proportional decrease in the mechanical properties with increasing filler loading. The strain rate sensitivity of the foams was confirmed by high strain rate studies using a split Hopkinson pressure bar. In line with quasi-static tests, the flow stress (maxima in stress strain curve) for compositions containing 40 per cent loading was substantially higher 151 MPa as compared to 38 MPa (60 per cent loading). Syntactic foams (S40) were subjected to controlled transient blast loadings on a shock tube, the intensity of which was varied by varying the pressure gradient between the driver and driven sections. Specimens with 40 per cent microballoon loading were found to withstand blast loads of the order of ~90 psi, while control samples underwent extensive deformation (1701 με at ~36 psi),which clearly highlight the potential of the syntactic foams as a blast mitigating material, especially as a core material in sandwich configuration.

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CONTRIBUTORS

Mr A.V. Ullas is a doctoral research fellow in Department of Applied Chemistry & Polymer Technology, Delhi Technological University. His research interests include development of polymeric syntactic foams for blast mitigating applications. He has been involved in the Preparation and testing of syntactic foam specimens.

Prof. D. Kumar is presently heading the Biotechnology department in DTU. He has vast research experience in the field of conducting polymers, polymer composites, etc. Dr. D.Kumar has to his credit over 170 research papers in elite journals. He has provided technical guidance on the research paper

Dr Prasun Kumar Roy joined DRDO in the year 2000 and is presently working as Scientist 'F' in CFEES. His areas of research include development of polymeric coatings, syntactic foams and degradable polymers. He has been involved in planning the experimentsfordevelopment of syntactic foams.

Mr Pankaj Sharma joined DIAT, Pune in the year 2002. He is presently working as Scientist 'E' in CFEES, DRDO. He is involved in the shock tube testing of syntactic foam samples.

Mr Prince Sharma is working as Scientist 'E' in Terminal Ballistic research laboratory, Chandigarh, DRDO. He has contributed in high strain rate testing of syntactic foam samples.

Mr Prince Chandel is working as Scientist 'E' in Terminal Ballistic research laboratory, DRDO. He has contributed in high strain rate testing of syntactic foam samples.

Mr Devendra Kumar is working as Scientist 'C' in Terminal Ballistic research laboratory, DRDO. He has contributed in high strain rate testing of syntactic foam samples.