Syntactic Foam Composites of Epoxy-Allyl Phenol-Bismaleimide Ternary Blend—Processing and Properties

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ABSTRACT: Syntactic foams based on a reactive polymer blend of Epoxy-Allyl Phenol-Bismaleimide were processed using hollow glass microballoons. The effect of microballoon concentration on the mechanical and thermophysical properties of the foam composites was investigated. The mechanical properties, specific strength, coefficient of linear expansion, and density of the foam composites showed decreasing trend with increase in microballoon concentration whereas the specific heat showed a marginal increase. The temperature dependence of the thermophysical properties revealed an increase in coefficient of linear expansion, whereas the specific heat was more or less insensitive to the test temperature. Syntactic foams of fixed filler loading (50% by weight) were processed using a blend of two types of microballoons of different shell thickness. The mechanical properties increased proportional to the concentration of the higher shell thickness microballoon. At moderate filler load microballoon breakage dominated the failure mode. The compositional dependency of the mechanical properties was corroborated by SEM analysis of the fracture surfaces. © 2007 Wiley Periodicals, Inc. J Appl Polym Sci 105: 3715–3722, 2007

Key words: syntactic foam; epoxy-allylphenol-bismaleimide blend; reactive blend; thermomechanical properties

INTRODUCTION

Syntactic foams are moderately strong, lightweight materials that find application in products for marine, aerospace, and automotive industries. The very low density of the foam is achieved through the incorporation of hollow microballoon fillers in the polymeric binder matrices. The hollow spheres may be made of metals, polymers, or ceramics. The matrix material may be thermosetting resins such epoxy, polyimide, phenolic, cyanate esters, or thermoplastic resins such as polyethylene, polystyrene. The properties of the resulting foam composition are dictated by the filler type, its volume fraction and the quality of the binder matrix system.[1–9] The micro porous material has got many additional advantages like lesser anisotropy and mechanical property enhancement including specific strength, shear strength, impact resistance, and energy absorption.[1,3,10] Unlike most other foams, syntactic foams are materials whose density before curing is the same as that after curing.[2,5,6] Such predictability is advantageous in the manufacturing process in aerospace structure. Syntactic foams are normally used as cores in sandwich structures as they possess superior properties compared to other core materials.[2–4,6–9,11]

There is current interest on mechanical characterization of syntactic foams. The elastic behavior of syntactic foam has been studied by Bardella et al.[12] An experimental–numerical investigation into the tensile, compressive, and fracture behavior of prefabricated syntactic foams have been briefly reported by Rizzi et al.[13] Medhat et al.[14] have reported an experimental and numerical study on dynamic fracture of functionally or compositionally graded epoxy syntactic foams. Sankaran et al.[15] observed that the incorporation of small percentage of Single Wall Carbon Nanotube (SWCNT) improved the storage shear modulus of the epoxy-amine system by 50% while in the case of Multi Wall Carbon Nanotube (MWCNT) the reported increase was 24%. Other studies on mechanical properties of syntactic foams include their dynamic mechanical properties,[3] fracture toughness,[5] micro structural failure modes under compression and three-point bend test,[11] failure mechanism under compression,[4,6] fiber reinforced syntactic foams,[7–9,16] elastic moduli,[10] adiabatic shear failure,[17] and thermo elastic behavior,[18] short beam three-point tests,[19] flexural strength of nanoclay reinforced hybrid syntactic foams[20] and modeling on mechanical behavior.[13]

Recently, we have reported a high performance composite system based on epoxy, allyl phenol, and bismaleimide[21] blend resin system that is superior in respect of high temperature performance in comparison to the corresponding epoxy-phenol system. The present study focuses on the use of this epoxy-allyl

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TABLE I
The Sources of Raw Materials of EPB System

Diallyl Bisphenol A (DABA)	Synthesized from bisphenol A and by a known procedure[23]
Epoxy resin, EPN 1139	Supplied by Ciba Geigy, Mumbai, equivalent weight 185.2
Triphenylphosphine (TPP)	Supplied by E-Merck, India and used as received
Bismaleimide (BMI-P)	Synthesized by a reported procedure[24]

phenol-bismaleimide (EPB) ternary blend, as matrix in the processing of syntactic foams. As the properties of the syntactic foams depend on the matrix properties, it is of interest to investigate the properties of the syntactic foams derived from this blended matrix system. Though syntactic foams of epoxy, bismaleimide etc. have been reported,[22] those based on polymer blends have been rarely reported. This article examines the mechanical and thermophysical properties of syntactic foams of EPB matrix with different types of fillers and varying filler loading. SEM analysis of the fracture surface has been done to study the failure modes.

EXPERIMENTAL

Materials

The source and chemical structure of the raw material of EPB blend system is given in Tables I and II. AR acetone (Nice Chemicals, Cochin, India) was used as received. Glass microballoons K-37 and K-25, supplied by 3M Company, US were used as fillers. The particle size distribution and properties of glass microballoon, as provided by the manufacturer are listed in Table III.

Preparation of EPB system

EPB matrix system was prepared by the reactive blending of epoxy novolac (EPN), 2,2′-diallyl bisphenol A (DABA) and 2,2-bis 4-(4 maleimidophenoxy) phenyl propane (BMI-P) in their stoichiometric equivalent ratios (1 : 1 : 1). EPN and DABA were weighed in their stochiometric equivalent ratio along with 0.5 wt % of triphenyl phosphine (TPP). To this, calculated amount of BMI-P was added so as to get a stoichiometric EPB blend. This ternary blend was dissolved in AR acetone for uniform mixing of the components. The acetone in the mixture was re-

TABLE II
Structure of Raw Materials of EPB System

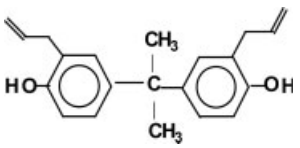
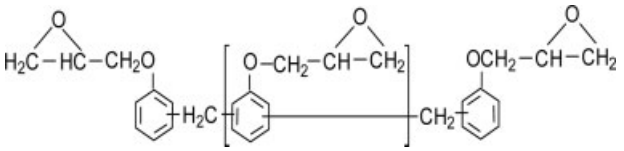
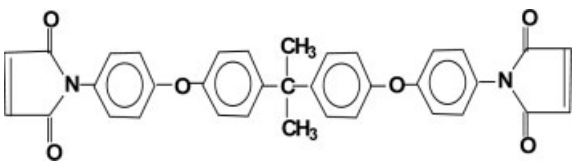
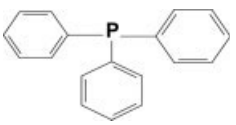
Materials	Structure
2,2′-Diallyl bisphenol (DABA)	
Novolac Epoxy (EPN)	
2,2-bis 4-(4 maleimidophenoxy) phenyl propane (BMI-P)	
Triphenyl phosphine (TPP)	

TABLE III
Particle Size Distribution and Properties of Microballoons

Microballoon type	Microballoon size distribution (μm, by volume)			Effective top size (μm)	Target fractional survival (%)	Average true particle density (kg/m^3)
	10th percentile	50th percentile	90th percentile			
K-25	25	55	90	105	90	250
K-37	20	45	80	85	90	370

moved by heating the system in vacuum oven at 60°C and the resulting neat matrix was characterized by FTIR, DSC, and rheological analysis. The properties of the matrix system is reported by us in an another study.[25]

Preparation of syntactic foam

The foam composites were prepared by mixing known weights of EPB system and calculated amount of microballoon so as to get compositionally graded foam composites with varying weight percentages of microballoon. The highly viscous ternary blend was first dissolved in AR acetone and to this required quantity of microballoon was added and mixed well to disperse it uniformly. Mixing was done gently to avoid the breaking of microballoon. The mixture was kept at ambient temperature overnight for the removal of acetone. It was then compression-molded to the required thickness following the cure schedule: 100°C (1/2 h), 150°C (1/2 h), 175°C (1/2 h), 200°C (1/2 h), and 250°C (5 h). The cure schedule was optimized using the data obtained from dynamic and isothermal rheological analysis and DSC thermograms.[25] Foam composites with mixtures of microballoon (K-37 and K-25), were prepared in a similar way by varying the weight ratio of the two types of microballoons in the composite system containing a total of 50 wt % filler. The densities of the foam composites were determined from the weight of foam samples of known volume.

Characterization of the syntactic foams

Thermal characterization

The specific heat and linear expansion of these composites were determined using DSC (ASTM D 968-82) and Thermo mechanical analysis (TMA) techniques (ASTM E 831-82). Samples of size 4 × 4 × 4 mm^3 were used for TMA.

Mechanical characterization

The mechanical characterization of the syntactic foams were done in a Universal Testing Machine Instron Model 4202. The foam materials of different microballoon content were cut into specimens of required size for tensile, compressive and flexural property evaluation. Tensile tests were carried out using dumbbell specimen conforming to ASTM D- 638 at a crosshead speed of 5 mm/min. No extensometer was used for strain measurement. Compressive properties were evaluated using specimens of dimension 10 × 10 × 15 mm^3 conforming to ASTM D-695 at a cross head speed of 2 mm/min. Flexural properties were evaluated by three point bending using rectangular specimens (5 × 13 × 100 mm^3), a span-to-depth ratio of 16 : 1 and a crosshead speed of 2.3 mm/min as specified in ASTM D-790-80. Five specimens were tested for each type and average value is reported.

RESULTS AND DISCUSSION

Effect of microballoon weight percentage on mechanical properties

The mechanical properties of the syntactic foams depend on its density which in turn depends on the resin/microballoon ratio. For syntactic foams, generally the strength properties are found to follow the same trend as its density.[7] The foam composites were fabricated using the EPB matrix system and varying concentrations of hollow glass microballoon (K-37) and their properties were evaluated. The mechanical properties of the foam composites are found to vary significantly with increase in concentration

TABLE IV
Tensile Properties of EPB Foam Composites of Varying Microballoon Content

Microballoon content (wt %)	Tensile strength (MPa)	Elongation (%)	Modulus (MPa)	Density (kg/m^3)
40	25 ± 1.0	2.1 ± 0.1	1490 ± 12	600 ± 10
50	18 ± 0.8	1.8 ± 0.1	1340 ± 13	540 ± 13
60	16 ± 0.7	1.8 ± 0.1	1315 ± 12	500 ± 12
70	10 ± 1.5	1.8 ± 0.1	1040 ± 15	440 ± 14

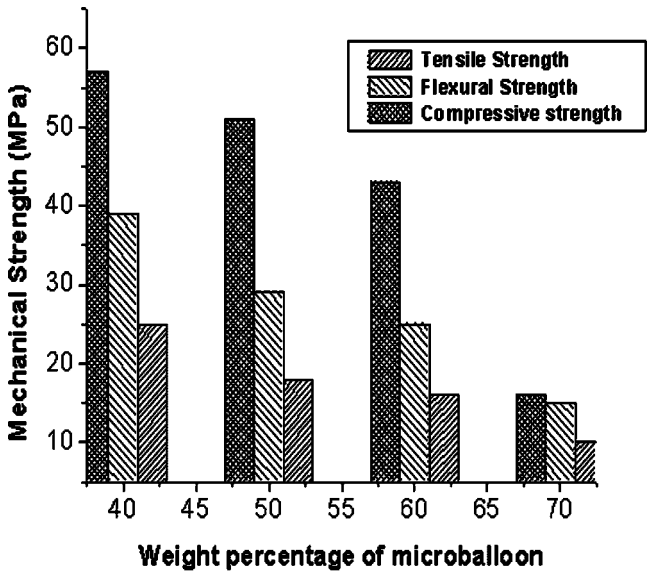


Figure 1 Effect of microballoon (K-37) concentration on the flexural, compressive, and tensile strength of the syntactic foam.

of microballoon. The syntactic foams also showed a gradual decrease in density with increase in microballoon concentration because of the incorporation of low-density filler (Table IV). The density values reported are the average of the duplicate test results. In all cases, the density measured was very close to theoretically predicted from the weights and densities of its constituents and using the rule of mixtures relationship.[8] The strength and modulus of these systems, under all the three loading environments (tensile, flexural, and compressive) were found to decrease with increase in filler concentration. The effect of microballoon weight percentage on the tensile, compressive, and flexural properties of the foam composite is shown in Figure 1. The failure mode was found to be brittle in the case of tensile and flexural tests and under compression, it was different. Thus, at lower microballoon concentration, the failure was mainly due to the interface failure, i.e., debonding between matrix and microballoon. At higher microballoon concentration, the weak microballoons are not properly protected by the binder matrix thereby reducing the strength as a result of poor interfacial interaction between the matrix and the microballoon. The tensile properties of the foam materials with varying concentration of microballoon are given in Table IV along with the deviations obtained for the test results. The tensile strength and modulus of the system were found to decrease on enhancing the filler concentration, whereas the elongation was quite independent of the composition.

The SEM pictures of the flexurally failed surfaces showed almost uniform distribution of broken microballoons [Fig. 2(a,b)]. This indicates the likely initiation of failure by the breakage of weak microballoon. Karthikeyan et al.[9]observed in their studies that the process involving resin fracture and resin-microballoon debonding rather than crushing/breaking of microballoon dominated the tensile failure of syntactic foams. The matrix serves as the load-bearing phase in the composite whereas the microballoon only provides lightweight and minimal strengthening effect.[5] The reduction in the amount of load bearing matrix resin outweighs the increase in stiffness produced by the higher microballoon concentra-

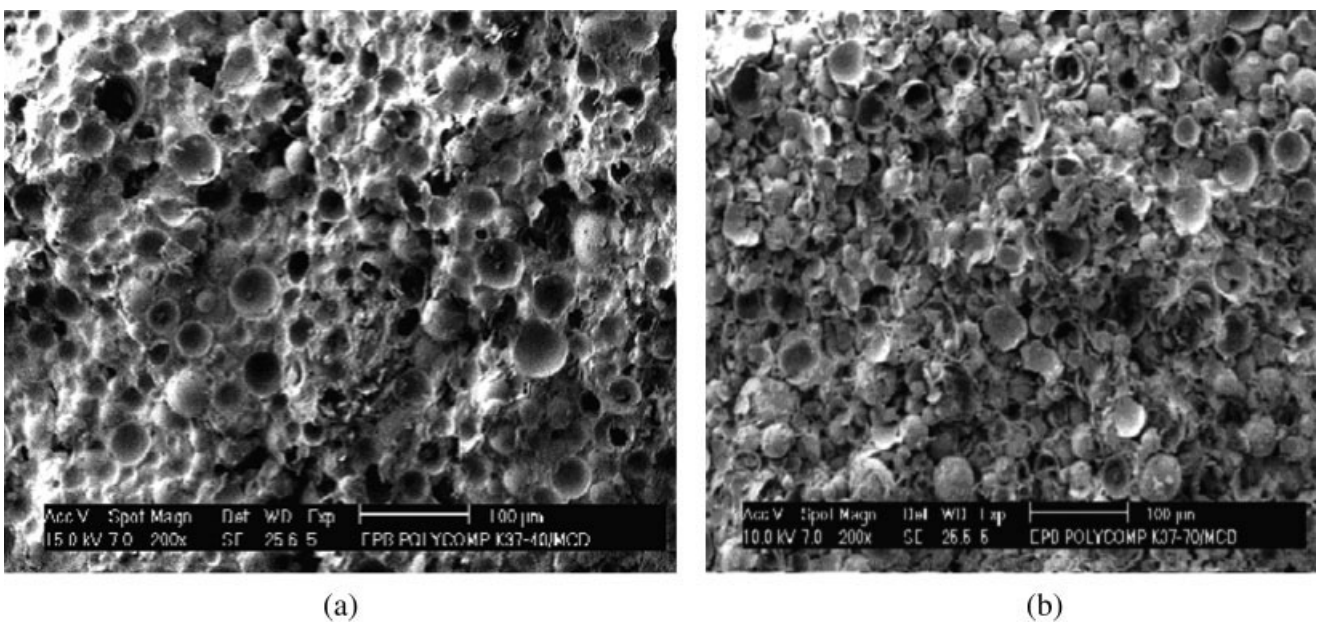


Figure 2 SEM pictures of syntactic foam composite with different microballoon concentrations (a) K-37 (40%) and (b) K-37 (70%).

TABLE V
Flexural and Compressive Properties of EPB Composites of Varying Microballoon Content

Microballoon content (wt %)	Flexural strength (MPa)	Flexural modulus (MPa)	Compressive strength (MPa)	Compressive modulus (MPa)
40	39 ± 1.0	3890 ± 11	57 ± 2.5	1460 ± 11
50	29 ± 1.3	3330 ± 12.5	51 ± 1.0	1430 ± 13
60	25 ± 1.8	3020 ± 10.5	43 ± 1.8	1405 ± 16
70	15 ± 1.5	2190 ± 15.0	16 ± 2.5	960 ± 18

tion. At lower microballoon volume fraction, the failure dominated mainly in the matrix resin. At this stage the breakage was found to be caused by both the interphase failure and the breakage of the microballoons of higher size [Fig. 2(a)]. Most of the smaller size microballoons were intact because of their intimate contact with sufficient quantity of the matrix resin and resulting in higher bond strength. At high microballoon concentrations of the order of 70%, the failure occurred mainly because of microballoon breakage [Fig. 2(b)] due to poor wetting of microballoon by resin.

The flexural and compressive properties of the EPB foam composites are given in Table V. The stress-strain curves obtained for the compression test, shown in Figure 3, reveal that the failure is not of brittle nature. After attaining the maximum strength, a slow rate of decrease in strength with increase in strain was observed in all cases. A special feature observed in this case is that at strain values of the order of double the strain at maximum strength, the material can still withstand almost 50% of its maximum load. The initial high slope of the compression stress-strain curve corresponds to the elastic deformation of the foam and the low slope/plateau regime is easily related to crushing/densification of microspheres. Unlike the trend observed in fiber reinforced composites, where kink banding, shear failure, matrix/fiber, interface failure are common failure modes in compression tests, the failure in these syntactic foam composites was crushing failure at the upper and lower surfaces of the specimen in contact with the platens of the compression test fixture, whereas no visual cracks were observed in the other areas. The initial modulus values under

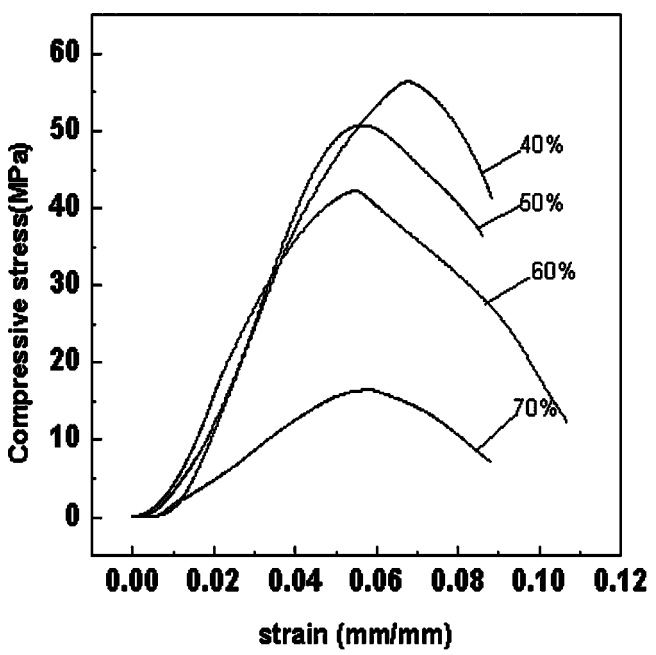


Figure 3 Effect of K-37 microballoon concentration on the compressive behavior of EPB syntactic foam composites.

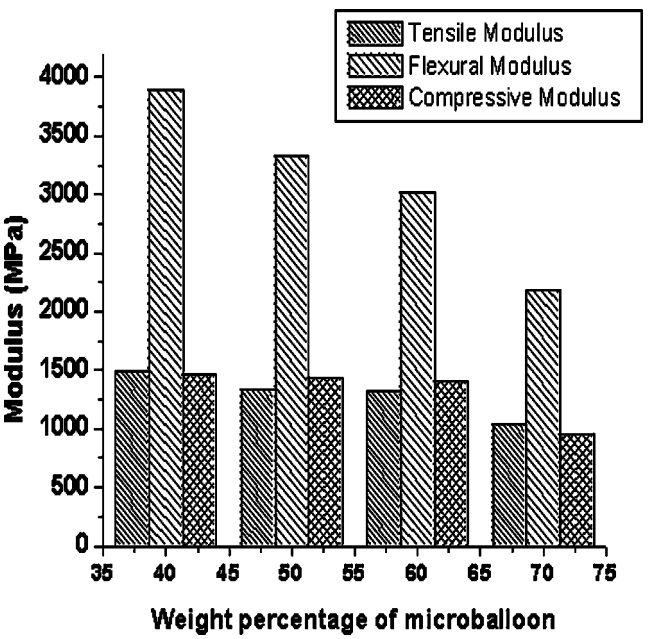


Figure 4 Effect of microballoon (K-37) concentration on the modulus of the syntactic foams.

TABLE VI
Specific Mechanical Properties of the Syntactic Foams

Specific mechanical properties (MPa/kg/m^3) × 10^3	Microballoon (wt %)			
	40	50	60	70
Specific tensile strength	42	33	32	23
Specific compressive strength	95	94	86	36
Specific flexural strength	65	54	50	34

TABLE VII
Effect of Microballoon (K-37) Weight Percentage on the Specific Heat of the EPB Foam Composite

Microballoon content (wt %)	Specific heat at different test temperatures (cal/g/°C)			
	70°C	80°C	90°C	100°C
40	0.37 ± 0.01	0.37 ± 0.01	0.37 ± 0.01	0.38 ± 0.01
50	0.38 ± 0.01	0.38 ± 0.01	0.35 ± 0.01	0.32 ± 0.01
60	0.41 ± 0.01	0.39 ± 0.01	0.36 ± 0.01	0.33 ± 0.01
70	0.25 ± 0.01	0.25 ± 0.01	0.23 ± 0.01	0.22 ± 0.01

TABLE VIII
Effect of Microballoon (K-37) Concentration and Temperature on the Coefficient of Thermal Expansion of the Syntactic Foams

Micro balloon content (wt %)	Linear expansion coefficient ($°C^{-1}$) at different temperature ranges (10^{-5})		
	30–50°C	50–100°C	150–200°C
40	2.5 ± 0.05	4.4 ± 0.07	5.8 ± 0.08
50	2.2 ± 0.05	3.6 ± 0.06	3.7 ± 0.06
60	1.7 ± 0.04	2.4 ± 0.04	2.9 ± 0.05
70	1.1 ± 0.03	1.5 ± 0.04	2.5 ± 0.05

tensile, compressive and flexure modes were found to follow a decreasing trend with increase in microballoon concentration. This trend is clearly depicted in Figure 4. The density of the foam composites also followed the same trend. The specific tensile, compressive, and flexural strength values also decreased with increase in microballoon concentration. (Table VI).

The syntactic foams derived from EPB blend systems showed higher mechanical properties compared to epoxy[26] and cyanate ester.[2] Epoxy syntactic foams exhibited a compressive strength of 51 MPa for a density of 570 kg/m^3, which is almost equal to that of EPB syntactic foams for a density of 540 kg/m^3. The compressive modulus values for EPB syntactic foams were also found to be higher than that for epoxy syntactic foams.[26] The tensile, flexural, and compressive properties reported[2] for cyanate ester syntactic foams were found to be inferior to that obtained for EPB syntactic foams of comparable composition.

Effect of microballoon concentration and temperature on specific heat and coefficient of thermal expansion of the syntactic foams

The thermophysical properties like the specific heat and linear expansion of composites are very important properties for foam composite intended to be used as a thermal insulating structures. The specific heat and linear expansion of these composites were determined using differential scanning calorimetry and thermomechanical analysis respectively, following the standard ASTM techniques. Since the microspheres are distributed randomly throughout the polymer matrix, the thermophysical properties are to be isotropic.[22] The two important factors which affect the thermophysical properties of syntactic foams are concentration of microballoon and temperature.

The specific heat values of the syntactic foam composites with varying microballoon content were determined at different temperatures (Table VII). The specific heat showed a marginal increasing trend

TABLE IX
Effect of Microballoon Blend Composition (K-37 : K-25) on the Properties of EPB Syntactic Foam Composites (50% Filled)

Property	K-37 : K-25 weight ratio			
	100 : 0	60 : 40	40 : 60	0 : 100
Tensile strength (MPa)	18 ± 1.0	15 ± 1.0	13 ± 1.1	10 ± 1.0
Tensile modulus (MPa)	1338 ± 13	1260 ± 11	1200 ± 11	837 ± 12
Compressive strength (MPa)	51 ± 1.5	30 ± 1.6	24 ± 1.5	14 ± 1.5
Compressive modulus (MPa)	1375 ± 13	897 ± 14	653 ± 15	621 ± 11
Flexural strength (MPa)	30 ± 0.8	28 ± 0.7	26 ± 0.6	12 ± 0.9
Flexural modulus (MPa)	3331 ± 13	3176 ± 14.5	2886 ± 16	1833 ± 18
Density (kg/m^3)	540 ± 13	470 ± 12	440 ± 11	390 ± 15

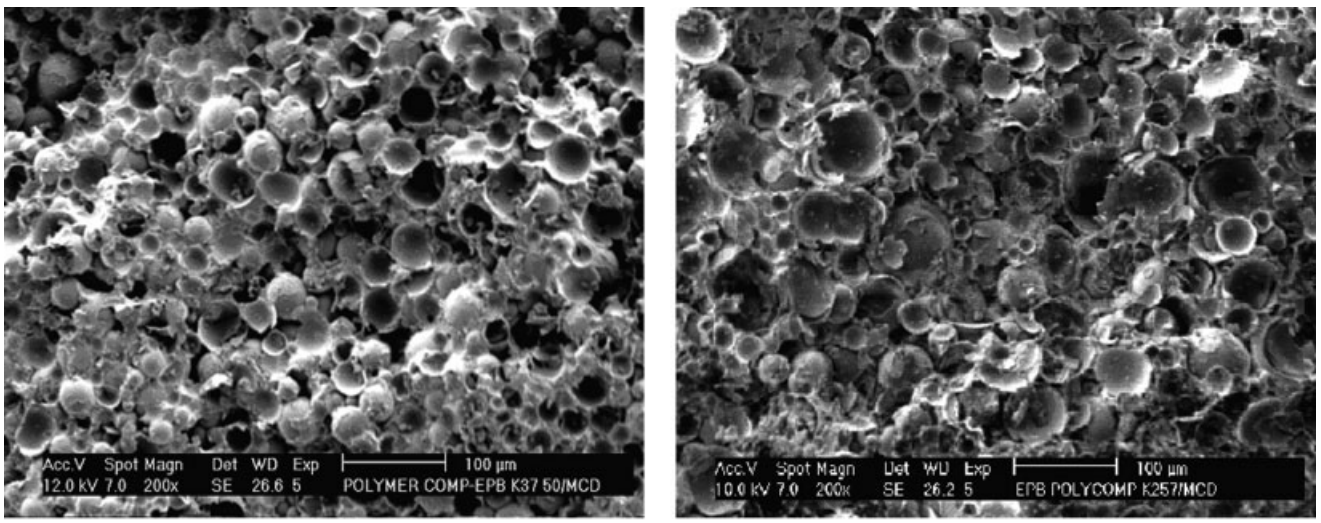


Figure 5 The SEM pictures of the failed surfaces of the EPB foam composites (50% filled) with a) K-37 and b) K-25 microballoon.

with increase in microballoon concentration up to about 60%. At the highest microballoon concentration the specific heat value showed a reduction. The temperature dependence of the specific heat was evaluated up to 100°C. Specific heat was found to be more or less insensitive to the test temperature.

The effect of microballoon composition and temperature on the coefficient of linear expansion of the foam composite is given in Table VIII. The expansion was found to show a systematic decrease with increase in microballoon concentration. At higher temperature regime the relative expansion was more for a given composition. The expansion was less pronounced for higher filler loading. This implies that matrix's role is dominant in thermal expansion of the foam.

Effect of shell thickness of microballoon

Syntactic foams were fabricated using a mixture of K-37 and K-25 microballoon fillers at a total filler content of 50% by weight. The proportion of the two was varied to get different K-37 : K-25 blend ratios (100 : 0, 60 : 40, 40 : 60, and 0 : 100). These composites were also characterized for their thermomechanical properties and the results are summarized in Table IX.

The results revealed that the strength properties viz. tensile strength, flexural strength, and compressive strength and the respective modulus values decreased with increase in concentration of K-25 microballoon filler. For the same concentration of microballoon, foam composites with K-37 microballoon gave higher density owing to its higher shell thickness and true density. The strength and the corresponding specific strength of the K-37 composites were significantly higher than those of K-25 composite. Since the breaking strength values are proportional to the shell thickness as well as the crushing strength values of the two types of microballoons, the microballoon breakage is confirmed as the cause for the foam failure at this filler loading (50%). The SEM pictures [Fig. 5(a,b)] of the failed surfaces of the foam composites also confirmed the same. The effect of microballoon blend ratio on the strength of syntactic foams (containing both K-25 and K-37) is shown in Figure 6 with the corresponding deviations

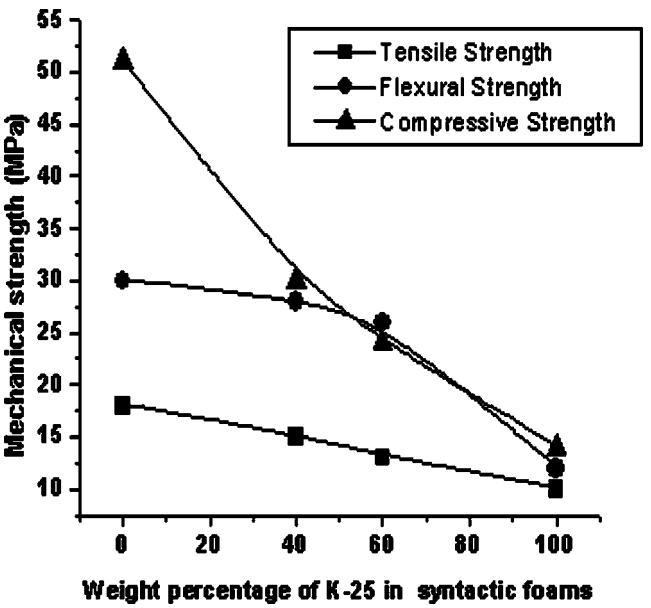


Figure 6 Effect of microballoon (K-25) concentration on the strength of syntactic foams containing both K-25 and K-37 microballoons.

TABLE X
Effect of Microballoon Blend Composition (K-37 : K-25) on the Specific Heat of EPB Syntactic Foam Composites (50% Filled)

K-25 in the blend (wt %)	Specific heat (cal/g/°C)			
	70°C	80°C	90°C	100°C
0	0.38 ± 0.01	0.37 ± 0.01	0.35 ± 0.01	0.33 ± 0.01
40	0.40 ± 0.01	0.39 ± 0.01	0.37 ± 0.01	0.35 ± 0.01
60	0.38 ± 0.01	0.38 ± 0.01	0.36 ± 0.01	0.34 ± 0.01
100	0.32 ± 0.01	0.32 ± 0.01	0.30 ± 0.01	0.30 ± 0.01

obtained for the different test results. The density values also showed systematic decrease with decrease in concentration of K-37 microballoon in the blend. The specific heat values of EPB syntactic foam composites with K-37 and K-25 microballoon blends (50% filled) are given in Table X. The specific heat was found to be insensitive to the blend composition and the test temperature.

CONCLUSIONS

The syntactic foams based on EPB matrix system were found to have superior mechanical properties in comparison to epoxy and cyanate ester based foam composites of comparable composition. The addition of hollow microballoon to the EPB system resulted in the reduction in strength and modulus of the material in tension, compression and flexural modes. The density also showed systematic decrease with increase in microballoon concentration in the foam composites. The specific heat showed a slight increasing trend with increase in microballoon concentration up to about 60% filler loading, whereas the linear expansion showed a systematic decrease with increase in microballon concentration and an increase with temperature. The strength and the corresponding specific strength and density of the K-37 based composites containing microballoon of higher shell thickness and crushing strength were significantly higher compared to those of K-25 foams. Since the breaking strength values are proportional to the shell thickness as well as the crushing strength values of the two types of microballoons, the microballoon breakage is confirmed as the cause for the foam failure. The mechanical properties of foam composites can be tuned by regulating the relative concentration of microballoon of differing crush strengths for a given composition. The good mechanical strength and low density imply the potentiality of these foam materials for use in moderately load-bearing insulative structures.

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