Tensile Properties of Glass Microballoon-Epoxy Resin Syntactic Foams

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ABSTRACT: The effect of hollow glass particle (microballoon) volume fraction in the range of 0.3–0.6 on the tensile properties and fracture mode of syntactic foams is characterized in the present research. Sixteen types of syntactic foams have been fabricated and tested. Four types of glass microballoons, having 220, 320, 380, and 460 kg/m^3 density, are used with epoxy resin matrix for making the syntactic foam samples. These foams contain 30, 40, 50 and 60% microballoons by volume. All types of microballoons have the same size but different wall thickness, which reflects as a difference in their density. It is observed that the tensile strength increases with a decrease in the volume fraction of microballoons. All types of syntactic foams showed 60–80% decrease in the tensile strength compared with that of the neat resin. The foams containing low strength microballoons showed lower tensile modulus compared with that of the neat resin, but the presence of high strength microballoons led to an increase in the tensile modulus of the composites.
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Key words: composites; foams; mechanical properties; tension; microballoon

INTRODUCTION

Hollow particles are used in a variety of applications ranging from piezoelectric transducers and sound absorption to fabrication of lightweight composite materials for aeronautical and marine structures.[1] Hollow glass particle (microballoon) filled polymeric composites, known as syntactic foams, have been studied over the past two decades for a variety of mechanical properties. The microstructure of a syntactic foam is shown in Figure 1, where glass microballoons are embedded in epoxy resin matrix. These lightweight materials are known for their high compressive strength, dimensional stability, and low moisture absorption compared to other types of foams.

Syntactic foams are extensively studied in the published literature for compressive, flexural, and hygrothermal properties.[2–5] However, studies on tensile strength of these materials are scarce.[6–8] Most of the applications of syntactic foams were limited to the marine structures, where the light weight of these materials could be used to obtain high buoyancy. These applications, where hydrostatic compression is the principal applied load on the material, gained advantage from high strength and energy absorption characteristics of these materials under compressive loading conditions.

It is observed that the compressive properties of syntactic foams can be effectively modified either by changing the microballoon volume fraction in the foam[2] or by choosing microballoons of different wall thicknesses.[9] The strength of microballoons depends on their wall thickness. It is observed that the compressive strength shows an almost linearly increasing trend with increase in the syntactic foam density. However, the total energy absorption decreases because of a decrease in the fracture strain in higher density foams.

Interest in utilizing the advantage of low density of syntactic foams in other applications such as aerospace structures and sports equipment has made it necessary to characterize these materials for tensile loading and study various parameters affecting their properties. The existing studies on the tensile properties of syntactic foams were carried out on foams containing low microballoon volume fractions (V_{mb}).[6,7,10] It was found that the tensile modulus increases with decrease in V_{mb}. The present work characterizes syntactic foams containing high V_{mb} (0.3–0.6). The selection criterion for microballoons makes it possible to directly relate the microballoon properties with the tensile strength and modulus of syntactic foams. The present study selectively studies the effect of microballoon density and volume fraction on the tensile strength and modulus of syntactic foams.

Selection of microballoons and porosity calculations

Hollow particles can be characterized based on their wall thickness (ω). A parameter named "radius ratio" is defined for hollow particles as

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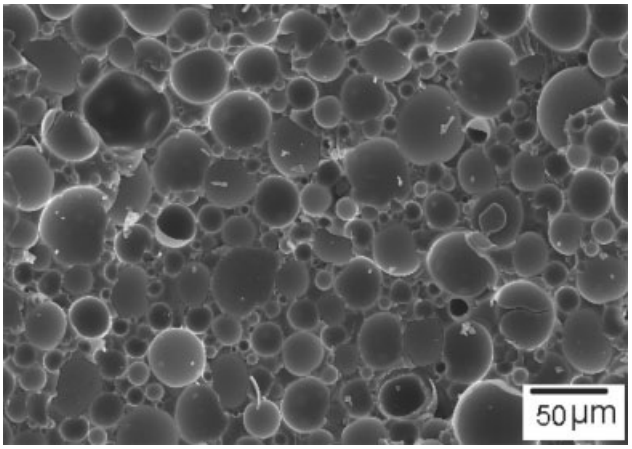


Figure 1 The microstructure of syntactic foam containing 60% microballoons by volume.

$$\eta = \frac{r_i}{r_o} \tag{1}$$

where r_i and r_o are the internal and outer radii of the hollow particle, as schematically represented in Figure 2. The parameter η is related to the microballoon density (ρ_{mb}) and the material that the microballoon is made of and is given by the equation

$$\eta = \left(1 - \frac{\rho_{mb}}{\rho_g}\right)^{\frac{1}{3}} \tag{2}$$

where ρ_g is the density of the microballoon material, which is glass in the present case. It can be observed that η is inversely proportional to the density and wall thickness of microballoons. If r_o is the same, any difference in the ρ_{mb} is caused by a difference in their η. In the present study, four types of microballoons with nearly the same particle size are chosen. Table I lists density, particle size, and η for the four types of soda lime borosilicate glass microballoons selected in the present study. The density of glass material of microballoons is taken as 2540 kg/m^3 in calculating r_i, ω, and the cavity size. The density was measured by using a crushed microballoon sample in a Quantachrome Ultrapyc pycnometer.

In the present study, η is the primary consideration while selecting different types of microballoons. Prior studies on the tensile testing of syntactic foams have varied V_{mb} in syntactic foams to vary the foam density and mechanical properties. While this approach is effective in varying the density of syntactic foams, it falls short of directly relating the mechanical properties of syntactic foams with the microballoon properties for one reason—the microballoon-matrix interfacial area also changes with changing volume fraction. Since interfacial strength between particles and matrix is an important parameter in defining the strength of composites, an approach that can keep the interfacial area also constant is needed to derive a direct correlation between the syntactic foam and microballoon properties. Hence, the approach adopted in the present work relies upon selecting microballoons having the same size range and mean particle size but different densities. A difference in the r_i or η of the same size microballoons causes a difference in ρ_{mb} and strength. Another problem that occurs if the size of microballoons in syntactic foams varies, is that the difference in their curvature leads to a difference in the stress concentration generated because of the presence of these particles. High stress concentration factor in the syntactic foams containing high V_{mb} can cause failure at lower applied stress.

Syntactic foams contain two types of porosities, namely microballoon porosity ($V_{p,mb}$) and matrix porosity ($V_{p,m}$), as shown in Figure 3. The hollow volume enclosed within microballoons gives rise to microballoon porosity, which is the desired closed cell porosity to reduce the density of the foam material. The volume fraction of the cavity in the microballoon structure ($V_{c,mb}$) can be given as

$$V_{c,mb} = \eta^3 \tag{3}$$

The calculated $V_{c,mb}$ in each type of microballoons used in the present study are given in Table I. The volume fraction of the desired closed cell microballoon porosity in syntactic foams ($V_{p,mb}$) is defined as

$$V_{p,mb} = V_{mb} \times \eta^3 \tag{4}$$

The second type of porosity arises because of the entrapment of air in the syntactic foam structure during the foam synthesis steps and is referred as the matrix porosity, as shown in Figure 3. The structure and morphology of the matrix porosity depends on its volume fraction. When the volume fraction of the matrix porosity ($V_{p,m}$) is small and it is well distributed, it also forms a closed cell structure. However,

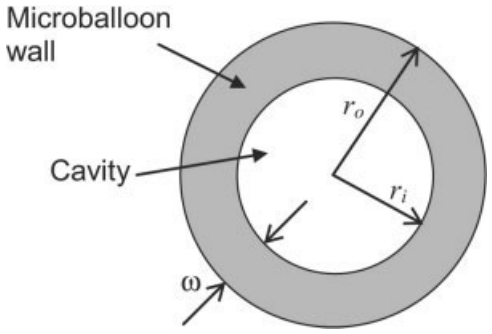


Figure 2 Notations used to define various physical parameters for microballoons.

TABLE I
Density, Wall Thickness, and Cavity Size for Microballoons Used in the Fabrication of Syntactic Foams

Microballoon density (kg/m^3)	Radius ratio η	Outer radius (μm)	Inner radius (μm)	Wall thickness (μm)	Microballoon cavity volume (%)
220	0.9702	17.5	16.98	0.52	91.3
320	0.9561	20	19.12	0.88	87.4
380	0.9474	20	18.95	1.05	85.0
460	0.9356	20	18.71	1.29	81.9

higher $V_{p,m}$ can give rise to an interconnected pore structure leading to open cell porosity. The matrix porosity is undesired and should be kept to the minimum level, because the presence of matrix porosity can lead to reduction in the foam strength and modulus.[7,11] The matrix porosity can also lead to an increase in the moisture absorption because moisture can diffuse in the foam specimens and can be accumulated in the matrix porosity regions.[4] The parameter $V_{c,mb}$ defines the microballoon strength, hence, control over r_o and η has already accounted for the presence of porosity in the foam structure. However, the matrix porosity is entrapped during the processing step and acts as stress concentration sites in a random manner. An estimate for $V_{p,m}$ can be derived by accounting for the difference between the theoretical density (ρ_t) calculated using rule of mixtures and the measured density (ρ_m).[12]

$$V_{p,m} = \frac{\rho_t - \rho_m}{\rho_t} \quad (5)$$

During the syntactic foam synthesis process some microballoons fracture, exposing the cavity enclosed within them. The exposed cavity can get filled up with the resin, increasing the density of the composite. Hence, eq. (5) actually accounts for the difference in the density increased because of the microballoon fracture and decreased because of the entrapment of matrix porosity. If the processing is carried out carefully and fractured microballoon fraction is very small, then $V_{p,m}$ calculated by eq. (5) can be approximated as the matrix porosity. Some of the previous studies have shown measured density of syntactic foams to be higher than their theoretical densities, despite the presence of matrix porosity, which leads to a conclusion that there was excessive failure of microballoons in those foams during synthesis.[6] Most previous studies have reported $V_{p,m}$ in the range of 2–10% in syntactic foams, depending upon the foam composition and processing conditions.

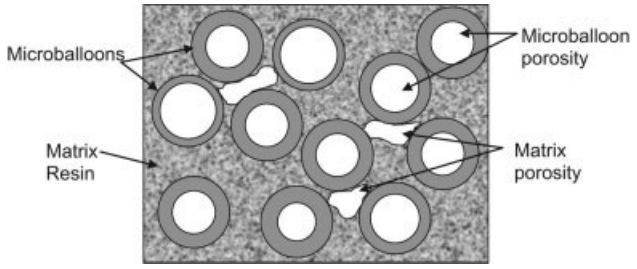


Figure 3 Schematic structure of syntactic foams showing microballoon and matrix porosities.

The microballoons have been used in the as supplied condition, without any surface treatment. It is known that the surface treatment can improve the matrix-microballoon bonding, leading to improved tensile strength of composites. However, one of the main focuses in the present study is to keep all the experimental parameters the same for all syntactic foams containing the same V_{mb} and change only one parameter, which is η. Any inconsistency in the surface treatment of the microballoons will affect the tensile test results, making the comparison meaningless.

EXPERIMENTAL

Constituting materials

DGEBA based epoxy resin DER 332 manufactured by DOW Chemical was used with an amine based hardener DEH 24 as the matrix resin system. A diluent C_{12}-C_{14} aliphaticglycidylether was used in the 5% by volume quantity to reduce the viscosity of the epoxy resin and facilitate mixing and wetting of microballoons.

Sixteen types of syntactic foams were fabricated in the present study using four types of microballoons in four different volume fractions each. These microballoons, supplied by 3M under the trade name of Scotchlite, have average true particle densities of 220, 320, 380, and 460 kg/m^3, respectively. The V_{mb} was varied from 30 to 60%. The compositions of these foams are presented in Table II. Neat resin samples were also prepared and tested under the same conditions. Neat resin density measured from these samples was used in the rule of mixture for the calculation of theoretical density of syntactic foams.

Fabrication process

Resin and diluent were mixed and heated to 50°C to further decrease the viscosity of the resin system. Then

TABLE II
Composition of the Fabricated Syntactic Foams

No.	Microballoon density (kg/m^3)	Microballoon volume fraction (%)	Sample name	Measured density (kg/m^3)
1	220	30	SF220–30	849
2		40	SF220–40	735
3		50	SF220–50	611
4		60	SF220–60	549
5	320	30	SF320–30	875
6		40	SF320–40	781
7		50	SF320–50	670
8		60	SF320–60	611
9	380	30	SF380–30	887
10		40	SF380–40	800
11		50	SF380–50	694
12		60	SF380–60	624
13	460	30	SF460–30	932
14		40	SF460–40	846
15		50	SF460–50	732
16		60	SF460–60	645
17	None	None	Neat Resin	1,160

the hardener was mixed, followed by microballoon addition. The mixture was slowly stirred until a uniform slurry was obtained, which was cast in stainless steel molds to obtain syntactic foam slabs. Cast foam slabs were cured for 24 h at room temperature and post cured at 100°C for 3 h.

Density measurements

The theoretical density values for all types of foams are presented in Table III, based on the rule of mixtures. ASTM standard C271–94 was adopted to measure the density of all fabricated specimens.[13] The weights and volumes of at least five pieces of $25 \times 25 \times 12.5$-mm^3 size specimens were measured to calculate the foam density. The measured density values are provided in Table II. The difference in the measured and calculated densities is used to calculate the matrix porosity content of the specimens using eq. (5), which is listed in Table III.

TABLE III
Total Porosity in Various Syntactic Foams

Sample name	Theoretical density (kg/m^3)	Matrix porosity (%)	Microballoon porosity (%)	Total porosity (%)
SF220–30	878	3.3	27.4	30.7
SF220–40	784	6.2	36.5	42.7
SF220–50	690	11.4	45.7	57.1
SF220–60	596	7.9	54.8	62.7
SF320–30	908	3.6	26.2	29.8
SF320–40	824	5.2	35.0	40.2
SF320–50	740	9.4	43.7	53.1
SF320–60	656	6.9	52.4	59.3
SF380–30	926	4.2	25.5	29.7
SF380–40	848	5.6	34.0	39.6
SF380–50	770	9.8	42.5	52.3
SF380–60	692	9.9	51.0	60.9
SF460–30	950	1.9	24.6	26.5
SF460–40	880	3.9	32.8	36.7
SF460–50	810	9.6	40.9	50.5
SF460–60	740	12.9	49.1	62

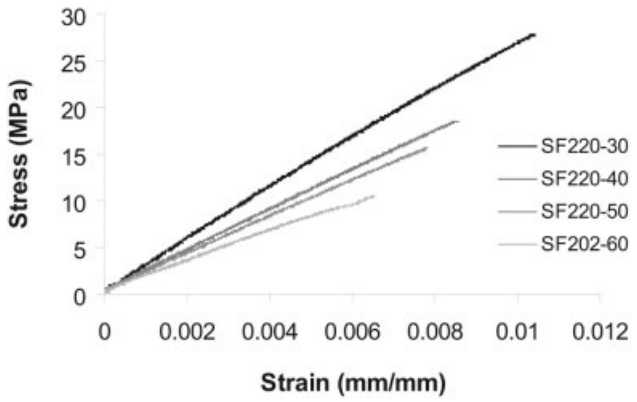


Figure 4 Representative stress–strain curves for SF220 type syntactic foams containing different volume fraction of microballoons.

Tensile testing

A computer controlled Instron 4467 mechanical test system was used for conducting the tests. The grip alignment was checked before carrying out the tests. The tests were carried out at a crosshead displacement speed of 0.2 mm/min. An extensometer with 25 mm gauge length was used to collect the strain data. The load-strain data collected from the tests were used to calculate the tensile strength and modulus of the specimens. At least five specimens of each type of composite were tested and average values are reported. The presence of matrix porosity can cause premature failure of some of the specimens. The strength values for such specimens were lower by a minimum of 30% compared to that for other specimens. After inspecting the fracture surface for the presence of matrix porosity, the results for such specimens were discarded. To compute the elastic moduli, the strain range of 0–0.002 mm/mm was considered. Although the stress–strain curves were straight line until fracture, the lower strain rate range was considered for consistency. Fracture of microballoons will not be significant in the lower strain region. Hence, this lower strain range is expected to represent the region where actual elastic deformation is taking place without microballoon fracture.

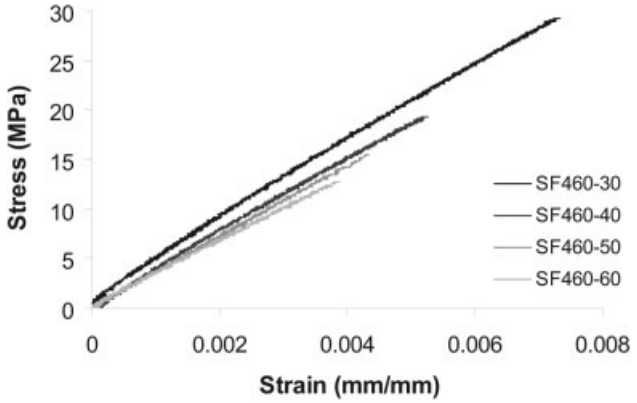


Figure 5 Representative stress–strain curves for SF460 type syntactic foams containing microballoons in various volume fractions.

TABLE IV
Tensile Strength and Modulus of Syntactic Foams

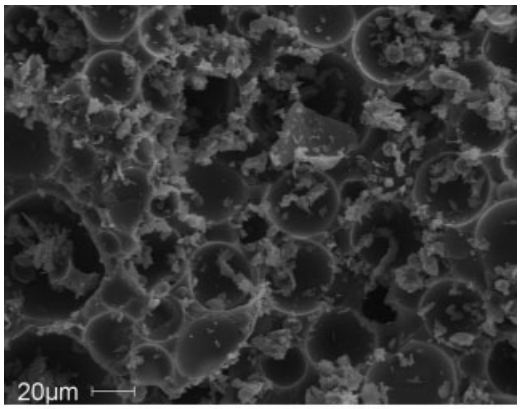
No.	Sample name	Tensile strength (MPa)	Modulus (MPa)
1	SF220–30	17.6 ± 0.8	2,490 ± 197
2	SF220–40	14.2 ± 1.7	2,368 ± 218
3	SF220–50	12.4 ± 2.0	1,910 ± 145
4	SF220–60	11.0 ± 1.5	1,880 ± 61
5	SF320–30	19.2 ± 0.9	2,938 ± 80
6	SF320–40	19.0 ± 1.5	2,963 ± 118
7	SF320–50	14.1 ± 1.7	2,960 ± 173
8	SF320–60	13.6 ± 0.7	2,623 ± 205
9	SF380–30	23.2 ± 1.2	3,260 ± 106
10	SF380–40	20.2 ± 0.5	3,482 ± 218
11	SF380–50	14.6 ± 3.7	2,867 ± 67
12	SF380–60	14.1 ± 0.7	3,002 ± 53
13	SF460–30	25.1 ± 1.9	3,700 ± 126
14	SF460–40	20.7 ± 1.2	3,641 ± 121
15	SF460–50	15.6 ± 1.2	3,615 ± 190
16	SF460–60	12.8 ± 1.6	3,491 ± 99
17	Neat Resin	57.2 ± 2.6	2,752 ± 92

RESULTS AND DISCUSSION

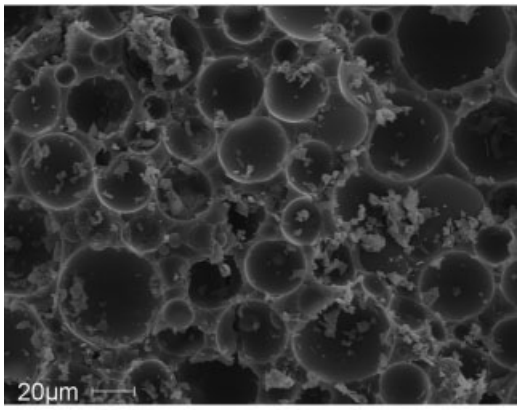
Two types of comparisons need to be carried out on the strength and modulus values of syntactic foams. In the first step, a comparison of tensile properties of foams containing microballoons having the same η in different V_{mb} is carried out. In the second step, tensile properties of foams containing microballoons of different η in the same V_{mb} are compared.

The representative stress–strain curves for SF220 and SF460 syntactic foam specimens are presented in Figures 4 and 5, respectively. These curves show linear stress–strain relationship immediately followed by brittle fracture. The stress–strain curves for other types of syntactic foams also showed similar features. The tensile curves are remarkably different from the compressive stress–strain curves, which show an elongated stress-plateau region representing their exceptional energy absorption capabilities.[2,14]

The calculated tensile strength and modulus values are listed in Table IV. Compared to the tensile strength of neat resin specimens, the strength is lower by 60–80% for all types of syntactic foams. With an increase in V_{mb} in the range of 0.3–0.6, the decrease in the strength is observed to be on the order of 25–60% for



(a)



(b)

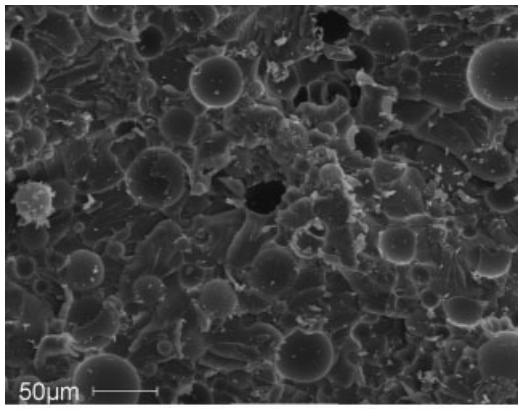
Figure 6 Fracture surface of SF220 type syntactic foams containing (a) 30% and (b) 60% microballoons.

various types of foams. Matrix being the continuous phase in syntactic foams, it acts as the load bearing phase in syntactic foams as suggested by Wouterson et al.[15] They also tested similar kinds of microballoons in epoxy resin systems. The matrix-microballoon interface does not appear to be very strong in these composites, and the presence of higher volume fraction of microballoons only reduces the volume fraction of epoxy resins in the structure, causing the lower strength of syntactic foams. Hence, with a decrease in the volume fraction of the matrix resin in the material structure, the strength of the composite is observed to decrease.[16]

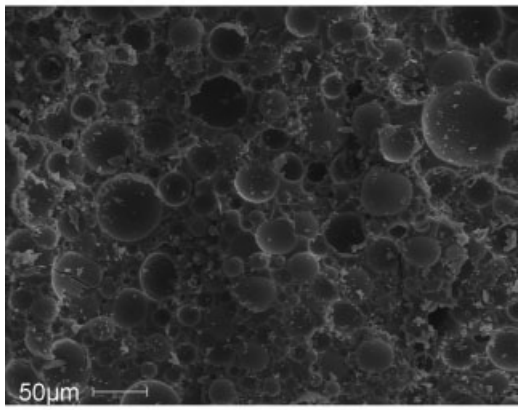
The modulus of SF220 syntactic foams increases with decrease in V_{mb} as per the results presented in Table IV. However, for other types of foams there is no significant change in the modulus with respect to V_{mb} over the experimental range of V_{mb} from 0.3 to 0.6. The difference in the modulus of SF460–30 and SF460–60 foams is only about 5.5%, which is within the experimental variation rage. However, for SF220–30 foams the modulus is about 25% higher than that for SF220–60 foams. Compared to the modulus of the neat resin specimens, SF220 type foams show a decrease in the range of 10–30% depending upon V_{mb}. However, SF320, SF380 and SF460 type foams show increase of about 4, 15, and 31% compared to the modulus of the neat resin.

Huang and Gibson had observed a trend similar to that of SF220 foams, where tensile modulus decreased with increase in V_{mb}.[6] Several analytical models have been validated using their data.[7,13] Their foams were made of lower density microballoons and higher strength matrix resin. Inclusion of weak particles is known to reduce the strength and modulus of composites because part of the stronger phase is replaced by a weaker phase. It is also possible that the fracture of lower strength microballoons takes places during the tensile testing, making the foams with high η microballoons dependent on the V_{mb}. The higher strength microballoons, used in other types of foams in this study, do not fracture during the tests in any significant amounts and these foams are relatively insensitive to V_{mb}. Numerical and analytical studies by Bardella and Genna showed that it is possible to obtain an increase in the modulus of syntactic foams only if microballoons are of sufficiently high strength.[7]

The microstructures of the fracture surfaces of SF220–30 and SF220–60 syntactic foams are shown in



(a)



(b)

Figure 7 Fracture surface of SF460 type syntactic foams containing (a) 30% and (b) 60% microballoons.

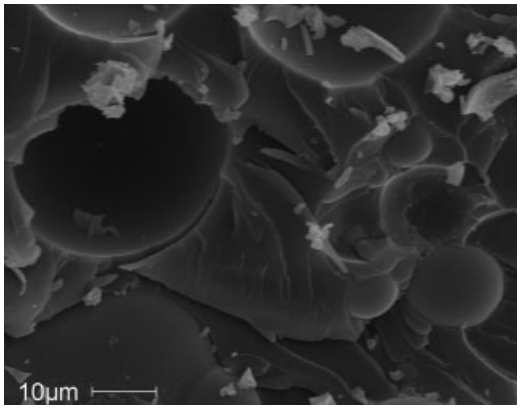


Figure 8 Deformation and fracture marks on the epoxy resin matrix in a SF46 syntactic foam having 30% microballoons by volume.

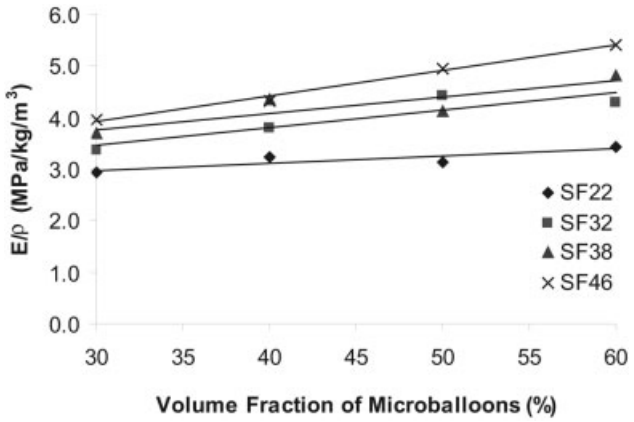


Figure 10 Comparison of specific modulus (E/ρ) of syntactic foams.

Figures 6(a) and 6(b), respectively. The fracture features observed in these micrographs can be compared with those observed in Figures 7(a) and 7(b), respectively, for SF460–30 and SF460–60. It was observed that fracture of SF220-type specimens generates considerably higher amount of debris compared with SF460-type foams. Such difference was noticed during the testing also when the fracture of SF220 was accompanied with formation of notably higher amount of powder compared to that of SF460 foams. Deformation and fracture marks can be observed clearly in the fractographs for SF460 foams, whereas the microballoon debris can be observed in the fractographs for SF220 foams. The fracture surface of a SF460 syntactic foam specimen is presented in a higher magnification micrograph in Figure 8, which shows deformation and fracture marks on the epoxy resin matrix.

Weight sensitive applications raise concern about the specific strength and modulus of syntactic foams. The specific strength of syntactic foams is plotted in Figure 9. The specific strengths of SF220 and SF320 foams do not show any variation over V_{mb} range of 0.3–0.6. A decreasing trend is observed for SF380 and SF460 foams but these values are also spaced closely and the trend can be considered to be nearly constant. For most specimens tested in this study the specific strength values are within the range of 0.020–0.026 MPa/(kg m^3).

Several applications of syntactic foams are in the form of sandwich composites for making structural parts or large structures.[17–19] The weight of a component can be minimized if E/ρ, E/ρ^2, and E/ρ^3 are increased for the same axial stiffness of a beam, bending stiffness of a beam, and bending stiffness of a plate. Several recent studies have focused attention on the bending properties of syntactic foams.[20,21] For all types of composites, these three parameters are calculated and presented in Figures 10–12. Compared to the values of 2.4 MPa/(kg m^3), 2.04×10^{-3} MPa/(kg m^3)^2, and 1.76×10^{-6} MPa/(kg m^3)^3 for neat resin, the E/ρ, E/ρ^2, and E/ρ^3 parameters for all types of syntactic foams are several times higher as shown in these

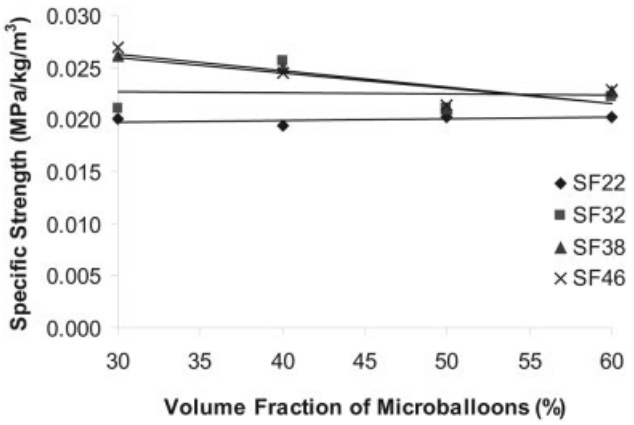


Figure 9 Comparison of tensile strength/weight ratio (specific strength) for various types of syntactic foams.

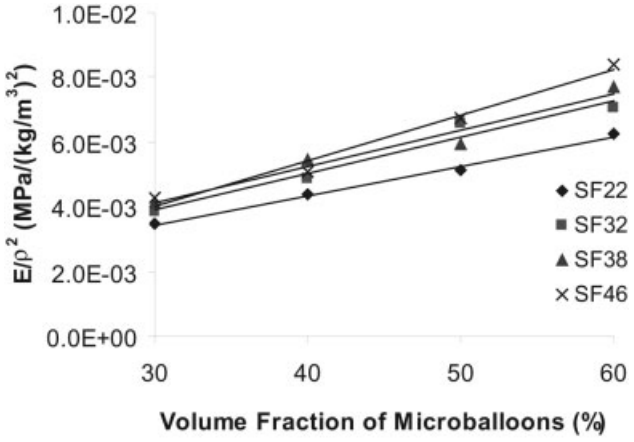


Figure 11 Comparison of E/ρ^2 for various types of syntactic foams.

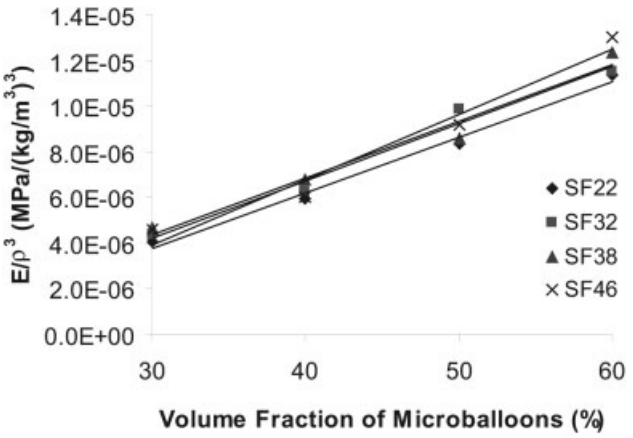


Figure 12 Comparison of E/ρ^3 for various types of syntactic foams.

figures. It is observed that these parameters are severely affected by the V_{mb} in the material. For the highest V_{mb}, giving rise to the lightest materials tested in this study, E/ρ, E/ρ^2, and E/ρ^3 are also the highest. Hence, use of syntactic foams can lead to substantial weight savings in structural applications. To obtain plates containing high bending stiffness, SF220 foams can be used effectively as they provide the same level of stiffness with the minimum weight.

Increase in the V_{mb} leads to a higher fracture strain in compressive failure mode, which is contrary to the tensile test results, where an increase in the V_{mb} seems to have led to a decrease in the failure strain of the material. The matrix material is the load bearing phase under tensile loading conditions in syntactic foams tested in the present study, and the V_m reduces with increasing V_{mb}. This difference causes the decrease in the failure strain at increased V_{mb} under tensile loading conditions. An improvement in the matrix-microballoon interfacial strength can result in improvement in the tensile strength of syntactic foams.

CONCLUSIONS

An experimental investigation is carried out to characterize the tensile properties of syntactic foams. Microballoon density and volume fraction are taken as variables in this study. The tensile modulus of syntactic foams containing low density microballoons is found to increase with decrease in the microballoon volume fraction in syntactic foams. For foams containing higher density microballoons, there was no significant effect of volume fraction on the modulus. The modulus was found to increase with the microballoon density. The tensile strength was found to increase with microballoon density and decrease with increase in the volume fraction of microballoons having the same density.

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