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Compression properties of syntactic foams: effect of cenosphere radius ratio and specimen aspect ratio

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Abstract

The present work is aimed at characterizing syntactic foams for flatwise (specimen aspect ratio of 0.5) properties and investigating the effect of change in the internal radius of cenospheres. The density and mechanical properties of the syntactic foam can be changed while keeping cenosphere volume fraction and particle–matrix interfacial area the same by using cenospheres of same outer radius but different inner radius. Five types of cenospheres, with the same mean outer radius but a different internal radius, have been selected for the fabrication of syntactic foams. ASTM C 365-94, a standard for the flatwise compressive properties of sandwich cores, is followed in the present work. The results obtained in the study are compared with the results of edgewise (specimen aspect ratio of 2) compressive properties evaluated in earlier work. Results show an increase in compressive strength and modulus with decrease in internal radius of cenospheres. The peak compressive strength and modulus were measured to be higher for the specimens tested in flatwise orientation compared to that in edgewise orientation. Varying only one parameter, the internal radius of cenospheres, helped in understanding the role of cenospheres and matrix resin in deformation and fracture process of syntactic foams.

Keywords: A. Foams; A. Particle-reinforcement; D. Mechanical testing

1. Introduction

Close cell structured polymeric foams such as syntactic foams are made by mixing hollow particles called cenospheres in a matrix material [1–3]. Syntactic foams give advantage of low moisture absorption and high compressive strength compared to the open cell structured foams. Some other advantages of syntactic foams are high energy absorption during deformation and high damage tolerance [4]. Greater design flexibility and wide range of properties can be achieved by choosing appropriate materials as cenospheres and matrix material [2]. These advantages make syntactic foams a popular choice for core material in sandwich structured composites [5] for marine and aerospace applications.

Several experimental and analytical studies are available on compressive [6–11], impact [12,13] and hygrothermal [14–16] properties of syntactic foams. Studies on viscoelasticity [17], fire performance [18], effect of polymer cure cycle [19] and microstructural characterization [20–22] of syntactic foams are also found in the published literature. Some studies on syntactic foam core sandwich composites are also available [23]. In many of these studies the effect of change in cenosphere volume fraction on mechanical properties of syntactic foams is investigated. However, a great advantage offered by the possibility of keeping the cenosphere volume fraction constant, but changing the syntactic foam density by changing the cenosphere internal radius is not found studied in the published literature. The present study explores this possibility by investigating the change in compressive properties of syntactic foams due to the change in cenosphere internal radius. The matrix resin system, cenosphere material and volume fractions are kept the same in all the five types of syntactic foams in this study. Hence, changes in the compressive strength and deformation or fracture pattern can be attributed to a change in one parameter only, i.e. the cenosphere internal radius. Therefore, a better understanding of the compressive properties of syntactic foams can be obtained by such an investigation. The syntactic foam specimens are tested in

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flatwise compression mode in this study in accordance to ASTM C 365-94 standard. The specimen aspect ratio (height/width) is maintained at 0.5 for such tests. Results of these tests are compared to the results obtained by the authors in their earlier research [24] for tests in accordance to ASTM D 695-96 on specimens of a different aspect ratio, 2, which are termed as edgewise compression. Results of compression tests of both types are also compared here to develop a better understanding of deformation and fracture pattern of syntactic foams. This comparison also helps in highlighting the effect of specimen aspect ratio on the compressive strength and modulus.

2. Radius ratio

Several modeling approaches for particulate composites can be found in the published literature. These approaches range from empirical or semi-empirical relations [25] to rigorous mathematical models [26–33]. Modeling parameters for particulate composites include mechanical properties of matrix, cenospheres and interface. Some other parameters such as particle volume fraction, particle size and shape also appear in the models. It is possible to use these models for cenosphere filled composites such as syntactic foams only if cenospheres do not fracture during deformation and the failure mode is either interfacial failure or matrix fracture. However, when cenospheres tend to fracture during a deformation process, the stress state in the syntactic foams may be considerably different to that of the composite containing solid particles of the same size, shape and volume fraction. This difference is based on the internal radius of the cenosphere, which is not considered in these particulate composites' models.

To explain the difference caused by a variation in the internal radius a parameter called the radius ratio, η, is introduced and defined by Eq. (1).

$$\eta = \frac{r_i}{r_0} \quad (1)$$

where r_i is the internal radius and r_0 is the outer radius of the cenosphere. Volume of material, V, composing the cenosphere can be represented in terms of η by Eq. (2).

$$V = \frac{4}{3}\pi r_0^3(1 - \eta^3) \quad (2)$$

Changing the value of η does not change any other modeling parameters such as cenosphere surface area and cenosphere/matrix interfacial strength. However, change in η changes the mechanical properties of the cenosphere. Therefore, its effect on the internal stress state must be understood thoroughly.

Fracture of any brittle particles under compression gives rise to fragments. These fragments are subjected to rotational movement due to shear deformation and linear movement in favorable direction due to local tensile and

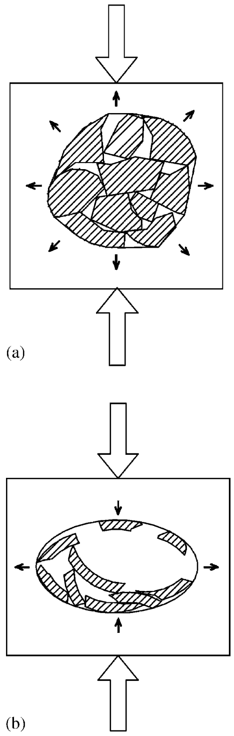

Fig. 1. Effect of the particle fracture on the surrounding matrix (a) solid particle or cenosphere having $\eta < \eta_{cr}$ and (b) cenosphere having $\eta > \eta_{cr}$.

compressive displacement. The relative movement of fragments with respect to each other leads to a mismatch between them and causes fragments to occupy more volume than the particle before fracture as shown in Fig. 1(a). The volume occupied by the particle before fracture and by debris after its fracture are represented as V_P and V_E, respectively. The ratio of V_P and V_E determines the additional stresses generated in the surrounding matrix material. In the case of cenospheres, the volume of debris generated depends on η. The higher η is, the smaller will be the volume of debris for the same outer radius. Hence, the effective volume, V_E, can be represented by Eq. (3).

$$V_E = \kappa V \quad (3)$$

where κ is termed as mismatch parameter and is a measure of hollow space between particle fragments. Eq. (3) can be written in terms of η as Eq. (4).

$$V_E = \kappa V_P(1 - \eta^3) \quad (4)$$

It is assumed that on further compression after initial fracture of the particle, further crushing will take place and the fragments finally take form of smaller solid spherical particles. It is also assumed that these new particles are present in a 'random close packed' arrangement. In such a case κ can be related to the random close packing factor for equal size spheres, which is 0.65 [34]. A critical value of η, termed as η_{cr}, can now be found where the volume of cenosphere before fracture is equal to the volume of debris generated after its fracture. In their previous work [35–37] the authors have calculated η_{cr} to be 0.71.

For syntactic foams containing cenospheres with $\eta < 0.71$, the volume occupied by the debris generated due to cenosphere fracture would be more than the volume of the cenosphere before fracture. This is because of the mismatch between various debris particles. Hence, additional stresses will be generated in the matrix in such a case as shown in Fig. 1(a). For the fracture of cenospheres having $\eta_{cr} > 0.71$, material system will become similar to a polymer with internal voids. This situation is shown in Fig. 1(b). Changes in the local stress states can lead to stress concentration or stress relieving and affects the compressive strength of the syntactic foam. All the cenospheres selected to fabricate syntactic foams in this study have $\eta > \eta_{cr}$. Such a choice causes the effect of cenosphere fracture on the internal stress state to be of similar type for all types of syntactic foams and makes the comparison of results more meaningful. The present study analyzes the deformation and fracture characteristics of syntactic foams with varying η and experimentally demonstrates the η dependence of the compressive properties.

3. Materials and processes

Constituent materials selected for the fabrication of syntactic foams, the fabrication process details and the compression test parameters are described in this section.

3.1. Constituent materials

Epoxy resin D.E.R. 332 and hardener D.E.H. 24 are selected to fabricate the syntactic foam slabs. These materials are manufactured by DOW Chemical Company. The volume fraction of matrix resin is maintained at 0.35. The viscosity of the selected resin at room temperature is about 4 N s m^{-2}. It is difficult to properly mix and wet the cenospheres if the resin viscosity is this high. Hence, a diluent C_{12}–C_{14} aliphatic glycidyl ether is added in 5% by weight quantity.

Five types of cenospheres have been selected from 3M's Scotchlite product range. According to the material property data provided by the manufacturer all types of cenospheres have nearly the same outer radius distribution and mean outer radius as given in Table 1. However, there is a difference in the internal radius of cenospheres, which reflects as the variation in the density values. Cenosphere wall thickness and η are calculated using the true particle density values and are presented in Table 1. In this table the cenosphere type is the manufacturer's code for the product where last two digits relate to the true particle density value of the cenospheres.

3.2. Fabrication process

Resin, diluent and hardener are heated to 50 °C to further lower the viscosity and then mixed together. Cenospheres are added to this mixture and hand stirred gently using wooden stirrers to minimize cenosphere damage. This mixture is cast in stainless steel molds of size $229 \times 229 \times 13$ mm^3. Mold surfaces are coated with silicon grease to ensure easy removal of foam slab after curing. Foam slabs are cured for 36 h at room temperature, 25 °C, and then post cured at 100 ± 3 °C for 3 h. Compression test specimens are cut from these slabs using a diamond blade tile saw. This saw and the blade are manufactured by MK Diamond Products Inc, CA, USA. Cutting speed of the blade is 3450 rpm in this saw.

Since the fabrication route involves mechanical mixing of materials, some air is entrapped in the material system giving rise to open cell structure porosity [2]. This entrapped air is termed as voids. Density of foam slabs is measured in accordance with the standard ASTM C 271-96. Weight and dimensions of at least sixteen pieces of $25 \times 25 \times 13$ mm^3 size foam pieces are measured to calculate the foam density values. Void volume of the fabricated syntactic foam slabs is calculated using Eq. (5).

$$V_{voids} = V - \left(\frac{V_{f,c} \times W_{slab}}{\rho_c} + \frac{V_{f,m} \times W_{Slab}}{\rho_m} \right) \quad (5)$$

where V and V_{voids} represent volume of the slab and voids, W_{Slab} represents the weight of the slab and ρ_c, ρ_m, $V_{f,c}$ and $V_{f,m}$ represent densities of cenospheres and matrix material and volume fractions of cenospheres and matrix material, respectively. The approach of taking large number of small foam pieces for the density and the volume fraction measurements also gives information on void distribution

Table 1
Properties of cenospheres

Cenosphere type	Cenosphere size distribution (μm)			Average true particle density (kg/m^3)	Average wall thickness ((m)	Radius ratio η
	10th percentile	50th percentile	90th percentile			
S22	20	35	60	205	1.26	0.922
S32	20	40	75	320	1.86	0.907
K37	20	40	80	370	2.17	0.891
S38	15	40	75	380	2.23	0.888
K46	15	40	70	460	2.74	0.863

Table 2
Density and void content of fabricated syntactic foam slabs

Cenosphere type	Corresponding syntactic foam type	Syntactic foam density (kg/m^3)	Void volume fraction(%)
S22	SF22	493	6
S32	SF32	545	9
K37	SF37	570	10
S38	SF38	575	10
K46	SF46	650	6

in the slabs. It is found that voids are distributed uniformly. The measured densities and calculated void volume fractions of the fabricated syntactic foam slabs are given in Table 2. In this table the code used as the syntactic foam nomenclature contains two letters, SF, which refer to 'syntactic foam' and two numbers, which are taken from the corresponding cenosphere type.

3.3. Compression testing

Two ASTM standards, C 365-94 and D 1621-94, are found applicable for the flatwise compression testing of syntactic foams, which are specified for the sandwich cores and rigid cellular plastic type of materials, respectively. A comparison reveals that the specimen sizes recommended in these standards are the main difference. ASTM D 1621-94 recommends cross sectional area between 2580 and 23,200 mm^2 and a height of 25.4 mm. For close cell structured foams such as syntactic foams ASTM C 365-94 recommends a smaller specimen size, with a cross sectional area of 625 mm^2 and no specific height. Based on the consideration that the fabricated foam slabs have thickness of about 13 mm and that in further experiments sandwich composites having syntactic foams as core would be fabricated and tested, ASTM C 365-94 is selected in this study. Specimens in this study have dimensions of $25 \times 25 \times 12.5$ mm^3 for length, width and height, respectively, in accordance with ASTM C 365-94.

Compression tests are carried out using a MTS 810 Material Test System. This machine is attached to a computerized data acquisition system. Stainless steel platens are fixed in the hydraulic grips of the testing machine to carry out the compression tests. Setup of compression tests is shown in Fig. 2. Constant crosshead movement rate is maintained at 0.5 mm/min as recommended by the selected ASTM standard. Six specimens of each type of syntactic foam are tested. Load–displacement data is obtained from the tests and is used to determine the compressive strength and modulus.

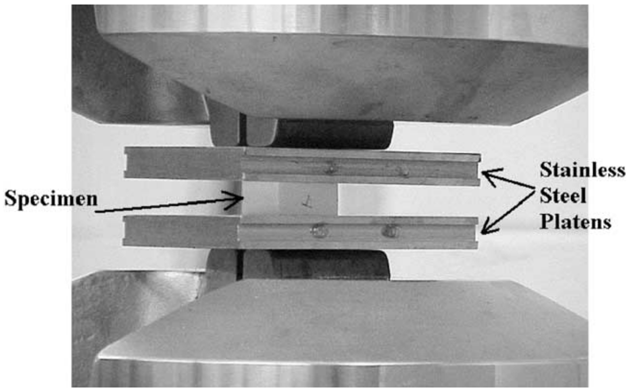

Fig. 2. Flatwise compression test setup.

4. Results and discussion

4.1. Flatwise compression tests

In the five types of cenospheres selected in the present study η varies from 0.863 to 0.922. Change in compressive modulus and average peak compressive strength of syntactic foam with change in η is given in Table 3. Strong dependence of modulus on η is evident from the values. Increase in compressive modulus from 1550 to 2640 MPa is observed with a decrease in η from 0.922 to 0.863. Peak compressive strength also shows increasing trend with decrease in η and changes from 30 to 72 MPa within this range of η. These results demonstrate strong influence of cenosphere η on the modulus and peak compressive strength of syntactic foam materials, which can be understood by studying the deformation and fracture pattern of the specimens in detail.

Some representative stress–strain curves for the flatwise compression testing of syntactic foams are shown in Fig. 3. For all types of syntactic foams the trend of the stress–strain curves is similar and corresponds to the trend observed by the authors in their previous work [6] and also by others [11]. It is observed that the stress decreases by about 10–20% after reaching a peak value. Peak stress denotes the point of crack initiation. After this decrease, the stress becomes nearly constant for further compression. This constant stress region is referred as the plateau region or densification stage. This is the stage when cenospheres are crushed exposing their internal hollow volume. Cenosphere debris and matrix resin occupy this volume while getting compressed. The plateau region for all of the syntactic foam samples extends beyond 10% strain without any further decrease in stress. No definite fracture point is observed in the flatwise compression of syntactic foams. The reason for this observation is described as follows. In any particulate system, all particles do not have exactly the same diameter. Particle diameter varies over a range of values. Similarly, cenospheres in the fabricated syntactic foams have a distribution of outer radius and η values as given in Table 1. Hence, the strength of various cenospheres of one type is expected to vary over a range of

Table 3
Comparison of compressive properties of syntactic foam specimens tested using two different specimen dimensions

Syntactic foam type	Radius ratio	Compressive modulus (MPa)		Peak compressive strength (MPa)	
		Flatwise	Edgewise	Flatwise	Edgewise
SF22	0.922	1550 ± 50	1220 ± 70	30 ± 2	32 ± 3
SF32	0.907	2025 ± 60	1531 ± 60	38 ± 2	40 ± 3
SF37	0.888	2195 ± 70	1845 ± 70	53 ± 2	54 ± 3
SF38	0.891	2395 ± 50	1965 ± 90	63 ± 3	56 ± 2
SF46	0.863	2640 ± 60	2221 ± 50	72 ± 3	64 ± 4

values depending on their inner and outer diameter. In any part of the foam structure, if the stress value rises above the fracture strength of any cenosphere, then that cenosphere fractures. If the mixing of cenospheres in the matrix resin is carried out properly, cenospheres of all different sizes and η values are distributed randomly in the syntactic foam structure. In such a condition there is no preferred fracture plane in the material. Hence, the specimens sustain large strain without showing any definite fracture point in the stress–strain curves.

It is observed that the strain at peak stress for all types of syntactic foams is close to 3%. Two component structure of syntactic foams consists of cenospheres and matrix resin. Different types of foams possess different sets of cenosphere properties whereas all the foams possess the same properties of matrix resin. Hence, the observation that the peak stress occurs at the same strain value for all types of syntactic foams indicates that it is independent of cenosphere η.

To establish the dependence of strain at peak stress on the properties of matrix material compression tests of matrix resin (unreinforced) are carried out. These tests are also carried out in flatwise and edgewise orientations keeping the specimen size the same as that for the syntactic foams specimens. Stress–strain curves for the compression tests of the unreinforced polymer are shown in Fig. 4 and their compressive strength and modulus values are presented in Table 4. The general trends of these curves show that under edgewise compression there is a distinct plateau region, whereas no such plateau region is found under flatwise compression. The main reason for such an observation is that it is easier for the edgewise compression specimens to deform laterally under secondary tensile stresses due to a higher specimen aspect ratio, whereas such a deformation is highly restricted in flatwise orientation. From these curves it can be observed that the yield strain for the matrix polymer is approximately 3.5%. These observations indicate that the failure initiation in syntactic foams does not depend on the strength of cenospheres and is related primarily to the properties of the matrix polymer.

4.2. Effect of specimen aspect ratio

By using a different specimen aspect ratio there are significant differences in the specimen fracture behavior and compressive properties. Specimen deformation and fracture pattern in both types of specimens is studied with respect to the specimen dimensions and compared here to explain the differences measured in the modulus and peak compressive strength. Stress–strain curves for SF37 and SF46 foam specimens tested in accordance to ASTM D 695-96 standard (edgewise compression) are shown in Fig. 5(a) and (b), respectively. Other types of syntactic foams also show similar trends in their stress–strain curves. A comparison of these curves with corresponding flatwise compression curves in Fig. 3 reveals some differences in their general characteristics. In case of edgewise compression curves, the stress decreases sharply after the peak stress value. The decrease in stress is in the range of 25–50%, which is significantly higher than about 10–20% decrease under the flatwise conditions. It is also observed that most specimens

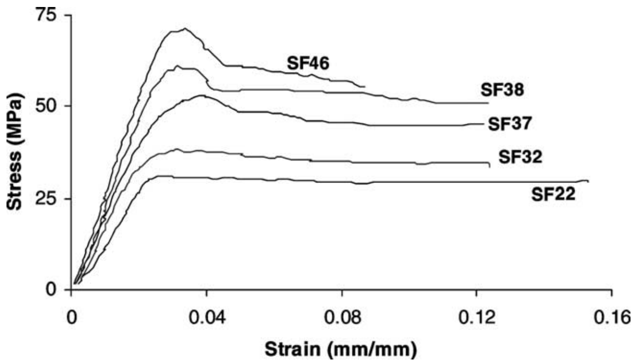

Fig. 3. Stress–strain curves for the flatwise compression testing of syntactic foams.

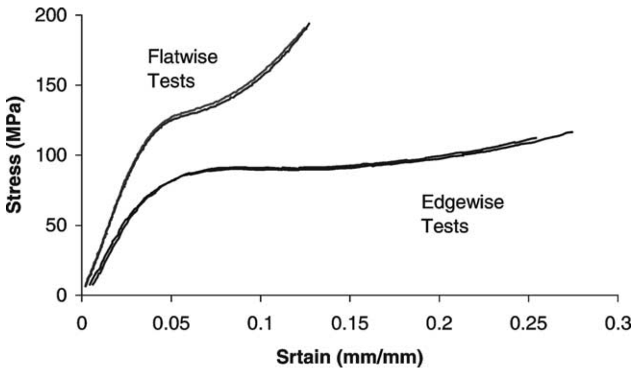

Fig. 4. Flatwise and edgewise compression test results of unreinforced matrix polymer system.

Table 4
Compressive properties of polymer used as syntactic foam matrix material

Specimen orientation	Compressive modulus (MPa)	Compressive strength (MPa)
Flatwise	3360 ± 40	125 ± 3
Edgewise	2320 ± 40	90 ± 2

do not show large strains after reaching the peak stress, which leads to an absence of the plateau region in the edgewise stress–strain curves. The edgewise compression tests had to be stopped at a strain of 6–8% due to sudden drop in the stress value unlike the flatwise compression tests, which showed plateau region till 10–15% strain. These variations in the specimen deformation pattern and stress–strain curves can be associated with the crack initiation pattern in the specimens.

Failed edgewise and flatwise compression test specimens of syntactic foams are shown in Fig. 6(a) and (b), respectively, to show the general characteristics of the fracture patterns. Figs. 7 and 8 show schematics of the observed fracture pattern of the specimens for edgewise and flatwise orientations, respectively. Fracture patterns of these specimens are compared to understand the differences observed in the characteristics of the stress–strain curves. In both cases shear cracks originate from the specimen corners as the first fracture activity, which is consistent with the authors' observations in earlier studies [6]. These cracks originate due to the deviatoric component of the applied

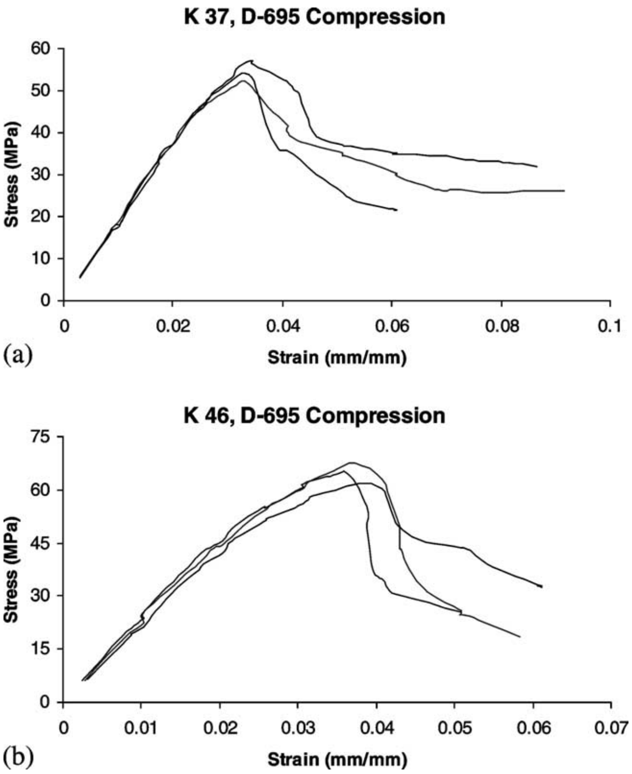

Fig. 5. Stress–strain curves for edgewise specimens of (a) SF37 and (b) SF46 syntactic foams.

(a)
(b)

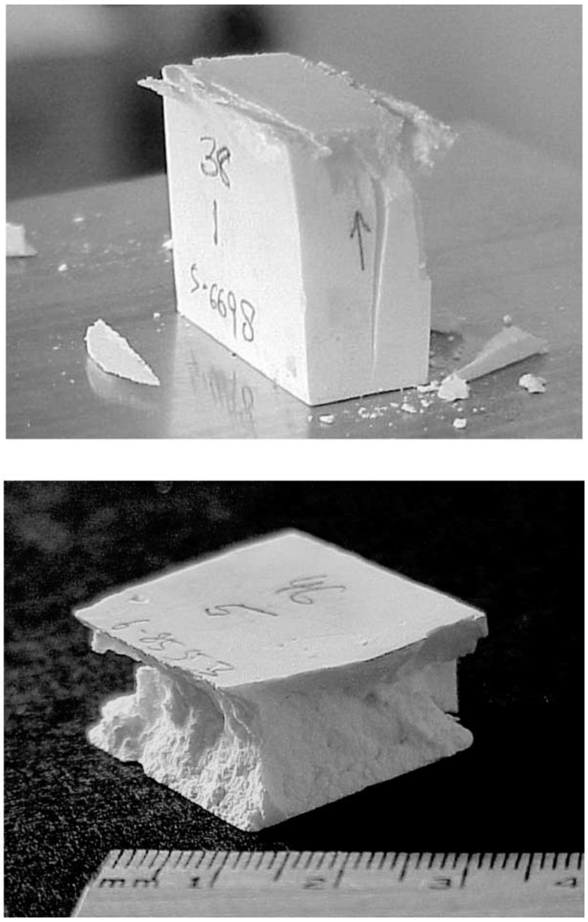

Fig. 6. Syntactic foam specimens compression tested under (a) edgewise and (b) flatwise orientation.

compressive stress. The shear cracks tend to form wedge shaped fragments in the specimens. In edgewise orientation, the wedge shaped fragments lead to stress concentration locations in the specimen along one edge of the fragment. The end of this edge on the face of the specimen is marked as 'A' in Fig. 7(a). As the strain increases in the specimen, due to the stress concentration, secondary tensile stresses acting normal to the applied load and the restraining effect of the compression fixture platens, the specimen shows barreling effect as indicated in Fig. 7(b). This leads to vertical splitting of the specimen as observed in Fig. 6(a). The extent of barreling depends on cenosphere η also. Fracture of cenospheres exposes the hollow space existing within them, which is now available for the compressing material to occupy. For higher η cenospheres more new space will be available for the compressing material, leading to lower lateral expansion. However, for syntactic foams having lower η cenospheres lesser new space will be exposed and the effect of secondary tensile stresses will be higher.

Location and size of the shear and tensile cracks is very critical for the final failure in edgewise orientation. This is a possible reason that considerable variation is observed in the peak stress values and trends of the stress–strain curves after peak stress is reached. The fracture mechanism of

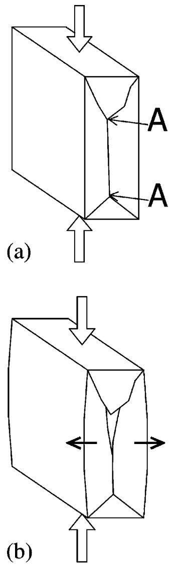

Fig. 7. Fracture mechanism of syntactic foam compressed under edgewise orientation.

flatwise specimens is shown in Fig. 8(a) and (b). In these figures it can be observed that the shear cracks originate in the specimens giving rise to wedge shaped fragments in the specimens in a manner similar that observed in the edgewise orientation. However, these fragments form in the sidewalls of the specimens and a large part of the specimen is not affected by their formation and separation. Comparison of results reveals that there is 15–25% difference in the modulus of syntactic foams in edgewise and flatwise orientations, whereas the peak compressive strength values show about 2–11% difference. The modulus depends on the elastic properties and volume fractions of the constituent materials. Hence, the modulus is affected by a possibility of lateral expansion due to the presence of large free surface compared to the specimen–platen contact area in edgewise orientation. The peak compressive strength depends on the mechanical properties

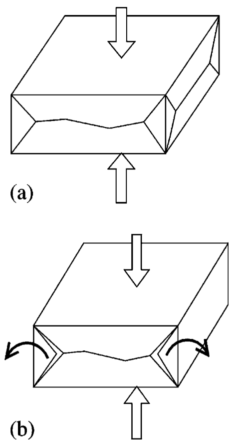

Fig. 8. Fracture mechanism of syntactic foam compressed under flatwise orientation.

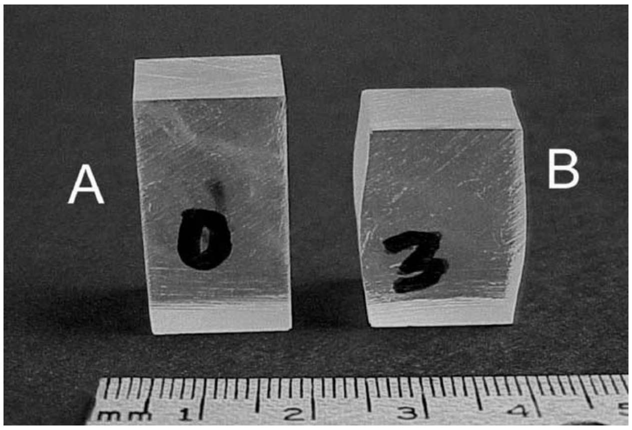

Fig. 9. Specimens of matrix polymer subjected to the edgewise compression.

of cenospheres and matrix resin, which causes comparable results in edgewise and flatwise orientations.

Compression tests on the matrix polymer are helpful in understanding the effect of lateral expansion and of secondary tensile stresses. In edgewise tests of the polymer specimens the height is twice the width. Hence, the effect of the lateral expansion is considerable and is observed in the form of barreling of the specimen. This is visible in Fig. 9 where specimen marked as 'A' is an undeformed specimen and the specimen marked as 'B' is subjected to about 25% strain under compression. Barreling can be observed in specimen 'B' in this figure. Flatwise compression test specimens have much smaller aspect ratio compared to the edgewise compression specimens. Hence, the lateral expansion is restricted as evident from Fig. 10. In this figure the specimen marked 'A' is undeformed and the specimen marked as 'B' is subjected to about 13% strain under compression. The effect of barreling is not as prominent as observed in edgewise compressed specimen shown in Fig. 9. The syntactic foam specimens also exhibit the similar behavior. This difference reflects in the modulus values of the syntactic foams obtained from the tests.

The unique approach of varying cenospheres η while keeping all other parameters the same made it possible

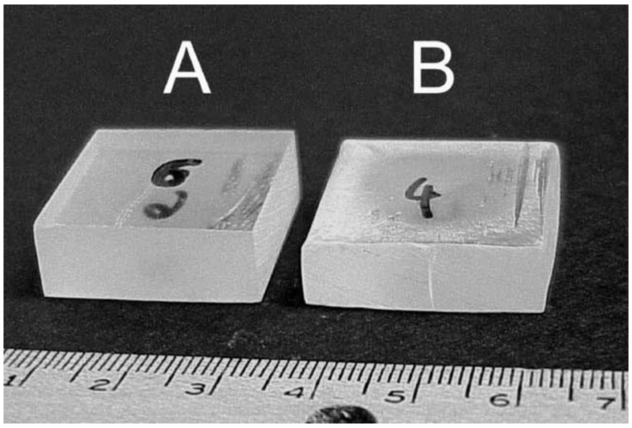

Fig. 10. Specimens of matrix polymer subjected to the flatwise compression.

to relate the variations in the compressive properties and fracture pattern of syntactic foams to just one parameter.

5. Conclusions

Compression tests are carried out on five types of syntactic foams having the same matrix material and cenosphere volume fraction. Such an approach is useful in designing syntactic foam materials for applications where a particular cenosphere volume fraction is defined. The strength and the density of syntactic foams can still be changed by selecting cenospheres of correct η value. The compression tests are conducted in flatwise orientation. The test results are related to the only parameter that is varying in this study, i.e. cenosphere radius ratio, η. It is concluded that with a decrease in η peak compressive strength and modulus increase. The present approach made it possible to conclude that the strain at peak compressive stress does not depend on η and is a property that comes from the matrix resin. The plateau region observed in the stress–strain curves is attributed to the fracture of cenospheres during compression of syntactic foams.

Comparison of results with edgewise compression test results leads to the conclusion that the measured peak compressive strength and modulus of syntactic foams are dependent on specimen aspect ratio. The following conclusions are drawn based on this study.

1. Specimens tested in edgewise orientation have lower values of compressive modulus compared to that of the flatwise specimen orientation because of lateral expansion and barreling.
2. Peak compressive strength values measured in edgewise orientation show strong dependence on the crack origination pattern.
3. Strain at peak compressive stress is about 3% for all types of syntactic foam specimens tested in both flatwise and edgewise orientations.
4. There is no specific fracture point on flatwise compression of syntactic foams.

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