Characterization of syntactic foams and their sandwich composites: modeling and experimental approaches

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CHARACTERIZATION OF SYNTACTIC FOAMS AND THEIR SANDWICH COMPOSITES: MODELING AND EXPERIMENTAL APPROACHES

A Dissertation
Submitted to the Graduate Faculty of the Louisiana State University and Agricultural and Mechanical College in partial fulfillment of the requirements for the degree of Doctor of Philosophy in The Interdepartmental Program in Engineering Science

by

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ABSTRACT
Hollow particle filled polymers known as syntactic foams are lightweight and highly damage tolerant. Syntactic foams are used as core materials in sandwich composites. The use of such materials in aeronautical and space structures make it necessary to understand their characteristics for various environmental and loading conditions. The first part of the present work takes modeling and finite element analysis approach to understand and predict the deformation behavior of syntactic foams. Contact analysis is performed on single particle models by the finite element analysis approach. In the second part extensive experiments are carried out to characterize syntactic foams for hygrothermal and compressive properties, and syntactic foam core sandwich composites for compressive and flexural properties. Flexural tests are carried out in three and four point bending and short beam shear configurations. Syntactic foams are tested in three different specimen sizes and orientations to characterize them as per the recommendations of various ASTM standards. Effect of specimen aspect ratio on the measured mechanical properties can be determined by such an approach. The effect of change in the internal radius of hollow particles, called cenospheres, on mechanical properties is studied for all these loading conditions. Five different types of cenospheres are selected for the study of the internal radius dependence of mechanical properties of syntactic foams and their sandwich composites. All selected types of cenospheres have the same outer radius, however, their internal radius is different. Hence, difference in mechanical properties of syntactic foams is caused due to a difference in only one parameter, the cenosphere internal radius. Such unique approach made it possible to identify the individual contribution of matrix resin and cenospheres in the mechanical properties of syntactic foams. Specimen deformation behavior and fracture features are correlated to deformation curves obtained during the testing.
1 INTRODUCTION

The terminology used in this dissertation is in accordance with ASTM D 883 – 00 [1], D 3878 – 01 [2] and ASTM C 274 – 99 [3] standards, which describe standard terminology for plastics, composites and structural sandwich constructions respectively.

1.1 Composite Materials

Increasing performance demands for modern technology applications make it necessary to look for new materials. It is difficult to achieve high and strict performance standards using any one material, hence new materials are fabricated by combining two or more conventional materials. These materials named as composite materials give unique combination of properties, which cannot be obtained from any single conventional material. A formal definition of composite materials given by ASM Handbook [4] is “macroscopic combination of two or more distinct materials, having a recognizable interface between them”. Composites are normally made by incorporating some reinforcement such as fibers in a bulk material known as matrix.

Some of the main advantages of composite materials are high strength, modulus, bending stiffness and chemical resistance. Properties of composites can also be tailored according to specific design requirements, directional and spatial properties.

Defining a composite material needs information on three aspects

1. Matrix material: e.g. polymer, metal or ceramic.
2. Reinforcements: e.g. continuous or discontinuous fibers or particles.
3. Structure: e.g. laminated or sandwich.

At the present composite materials have found wide spread applications in aeronautics and space sector due to their lightweight and high strength. Almost all modes of transportation and sports equipment also use considerable amount of composites.

1.2 Sandwich Structured Composites

Sandwich structured composites are a special class of composite materials which have become very popular due to high specific strength and bending stiffness. Low density of these materials makes them especially suitable for use in aeronautical, space and marine applications.

Concept of sandwich structured composite materials can be traced back to as early as the year 1849 AD [5] but potential of this construction could be realized only during Second World War. Developments in aviation posed requirement of lightweight, high strength and highly damage tolerant materials. Sandwich structured composites, fulfilling these requirements became the first choice for many applications including structural components. Now their structural applications spread even to the ground transport and marine vessels.

1.2.1 Definition

Sandwich composites comprise of two thin but stiff face sheets attached on either side of a lightweight, thick slab known as core. Many variations of this definition are available but the key factor in making this type of materials remains the lightweight core, which reduces the
overall density of the material and stiff skins providing strength. The structure of sandwich composites is shown in Figure 1.

Integral bonding between skins and core prevents the interfacial failure under the applied load enhancing the flexural properties of sandwich composites. There is no general rule about the relationship between the thickness of skin and core. It depends on the application and required properties. Major advantage of sandwich structured composites is the possibility of tailoring properties by choosing appropriate constituting materials and their volume fractions.

1.2.2 Components in Sandwich Composites
Sandwich composites primarily have two components namely, skin and core as shown in Figure 1. If an adhesive is used to bind skins with the core, the adhesive layer can also be considered as an additional component in the structure. The thickness of the adhesive layer is generally neglected because it is much smaller than the thickness of skins or the core. The properties of sandwich composites depend upon properties of the core and skins, their relative thickness and the bonding characteristics between them.

1.2.2.1 Core
Based on the performance requirements, large numbers of materials are used as core [6]. Popular core materials can be divided into three classes as described below.

1. Low density solid materials: open and closed cell structured foams, balsa and other types of wood.
2. Expanded high-density materials in cellular form: honeycomb, web core.
3. Expanded high-density materials in corrugated form: truss, corrugated sheets.

High-density materials used for the purpose of making expanded core include aluminum, titanium and various polymers. The structure of the core material affects the interfacial contact area between skins and the core. Expanded high density materials normally provide much smaller contact area compared to the solid low density materials. The choice of appropriate structure for core provides additional parameter to design a sandwich composite as per given specifications or service conditions.

The use of cores like closed cell structured foam gives some distinct advantages over open cell structured foams and cores. The specific compressive strength of close cell structured foams is much higher. They also absorb less moisture than open cell structured foam.

![Figure 1 The structure of a sandwich composite.](image-url)
1.2.2.2 Skins
A wide variety of materials are available for use as skins. Sheets of metals like aluminum, titanium and steel and fiber reinforced plastics are some of the common examples of skin materials. In case of fiber reinforced skins, the material properties can be controlled directionally in order to tailor the properties of the sandwich composite. Fiber reinforced polymers are used widely as skins due to their low density and high specific strength. Another advantage offered by the use of polymer composites in skins is that the same polymer can be used to make the skin and the core. Cross-linking of polymer between core and skin would provide adhesion strength level equal to the strength of the polymer. This provides possibility of making the skin an integral part of the structure eliminating the requirement of the adhesive. When an adhesive is used to bond the skin and the core together, selection of adhesives becomes very important, as they should be compatible with both the skin and the core materials. The adhesion must have desired strength level and should remain unaffected by the working environment.

In case of metallic components, welding or brazing is used as a means of binding the core and skins together. Use of adhesives is also possible but is limited to such cases where one or more of the components cannot withstand heat.

Choice of skins is important from the point of view of the work environment as this part of the structure comes in direct contact with the environment. Corrosion, heat transfer characteristics, thermal expansion characteristics, moisture absorption and other properties of the whole sandwich composite can be controlled by proper choice of skin material. In most cases both skins of the sandwich are of the same type, but could be of different type depending upon specific requirements. Difference may be in terms of materials, thickness, fiber orientation, fiber volume fraction or in any other possible form.

1.2.2.3 Properties of Sandwich Composites
Main advantage of any type of composite material is the possibility of tailoring their properties according to the application. The same advantage also applies to sandwich composites. Proper choice of core and skins makes sandwich composites adaptive to a large number of applications and environmental conditions. Some general characteristics of sandwich composites are described below:

1. Low density: choice of lightweight core or expanded structures of high-density materials decrease the overall density of the sandwich composite. Volume of core is considerably higher in the sandwich composite compared to the volume of skins so any decrease in the density of the core material has significant effect on the overall sandwich density.
2. Bending stiffness: this property comes from the skin part of the sandwich. Due to a higher specific stiffness sandwich composites result in lower lateral deformation, higher buckling resistance and higher natural frequencies compared to other structures.
3. Tensile and compressive strength: the z-direction (Figure 2) properties are controlled by the properties of core and x and y directions (Figure 2) properties are controlled by properties of skins.
4. Damage tolerance: use of flexible foam or crushable material as core makes sandwich material highly damage tolerant structure. For this reason foam core or corrugated core sandwich structured materials are popular materials in packaging applications.

1.2.2.4 Advantages of Sandwich Composites

Some of the advantages of sandwich composites are:
- Tailoring of properties according to requirements.
- Large available choice of constituents for core and skins.
- Low density leading to saving of weight.
- High bending stiffness.
- Higher damage tolerance.
- In-situ fabrication.
- Good vibration damping capacity.

1.2.2.5 Limitations of Sandwich Composites

There are current limitations that can be overcome through the development of new materials and manufacturing methods. Some of these are:
- Higher thickness of the sandwich composites.
- Higher cost of sandwich composites compared to conventional materials.
- Processing is expensive.
- Difficult to join.
- Difficult to repair, if damaged.

1.2.2.6 Applications of Sandwich Composites

There are several applications that require materials of low density, high strength and high damage tolerance. Due to their lightweight, sandwich composites are widely used in various kinds of vehicles used for air, ground or sea transportation. Some of the main areas of applications of sandwich composites are listed below.

1. Structural applications: aircraft, spacecraft, submarine, ships and boats, surface transport vehicles, building materials etc.
2. Packaging materials.
3. Thermal and electrical insulation.
4. Storage tanks.
Innovativeness is essential in finding new combinations of core and skin materials and new ways to use them in various applications where conventional materials have already reached their performance limits.

1.3 Particulate Composites

Particulate composites are used as core materials in sandwich composites. Incorporation of particulate fillers in epoxies leads to several useful properties such as reduced density, increased impact strength, desired magnetic and electrical properties, high damage tolerance and reduced cost. These properties make particulate composites suitable for use in weight sensitive applications such as aircraft structures and damage prone applications such as packaging. Sandwich structures having particulate filled composite materials as core particularly give the advantage of high specific compressive strength and bending stiffness.

1.3.1 Filler Materials

A variety of particles are used as fillers in composites [7]. Purposes of using fillers range from reducing the cost of expensive polymeric components to modification in strength, magnetic, electrical or fire retardant properties and change in density. Large number of materials can be selected as fillers for the polymers, which include particles of minerals, metals, ceramics, polymers and also some industrial wastes [8]. Some common examples of filler materials are particles of alumina, silica, hollow and solid particles of glass, wood chips, flyash and carbon black. Selection of materials is mainly based on the desired properties of the composite.

The shape of the filler particles plays important role in determining the properties of the composite, hence, particles are normally classified based on their shapes. Some of the common particle shapes are spherical, cubical, blocks, flaky and fibrous. The surface area is different for the same volume of these shapes, which affects the size of the interfacial region between the particle and the matrix resin. For each of these shapes the stress concentration factor would be different due to their different corner radius of curvature and aspect ratios. Spherical particulate fillers are more popular compared to the other types.

Use of hollow particles, known as cenospheres, has increased considerably in recent years in the production of core materials of low density and high damage tolerance. Such low density materials are classified as close cell structured foams and are known as “syntactic foams”. Density values of syntactic foams can be tailored over a wide range by changing the material or density of cenospheres.

1.3.2 Syntactic Foams

Syntactic foams are known for their high specific compressive strength, low moisture absorption and excellent damping properties. They are used as core materials in sandwich composites for weight sensitive structural applications. Syntactic foams are multi-functional composite materials due to their broad range of mechanical properties coupled with vibration damping characteristics, fire performance and ability to be fabricated in functionally graded configurations. These materials were developed in the 1960s as buoyancy aid materials for deep sea applications [9]. Presently they are used in aircraft, spacecraft and ship structures [10].
One of the major advantages of syntactic foams is their ability to be designed and fabricated according to the physical and mechanical property requirements of the application. Depending upon the service conditions the matrix resin can be chosen from a wide range of thermosetting and thermoplastic resins. Similarly, cenospheres of polymer, ceramic or metal can be chosen [11, 12]. Other parameters that can be adjusted are the volume fractions of the matrix and cenospheres in the structure.

There are two methods of changing the density of syntactic foams to directly influence their properties. The first method is to change the volume fractions of matrix and cenospheres in the structure. The second method is to use cenospheres of different internal radius but the same outer radius and the same volume fractions of matrix resin and cenospheres. The second method gives great design flexibility as any change in properties of syntactic foam can be related to just one parameter, the internal radius of cenospheres. Considering the applications of syntactic foams in aeronautics and space applications it is important to establish the effect of the internal radius on the mechanical properties of syntactic foams. In the present work syntactic foams having glass cenospheres in polymeric matrix are analyzed and discussed.

1.3.2.1 Structure of Syntactic Foams

Syntactic foams have two phases in their structure, namely matrix resin and cenospheres. Structure of syntactic foam can be observed in the scanning electron micrograph presented in Figure 3. The micrograph shown in this figure is taken from the as-cut surface of syntactic foam specimen. Cenospheres embedded in the matrix resin are visible in the structure. The two phase structure is schematically shown in Figure 4a. During the fabrication of syntactic foams some air is inevitably trapped in the structure and is present as open cell structured porosity. This entrapped air, termed as “voids”, makes syntactic foams three phase materials. Three phase structure is schematically shown in Figure 4b. In the present work the fabricated syntactic foams have three phase structures.

Figure 3 Scanning Electron Micrograph showing structure of as-cut surface of syntactic foam.
Figure 4 Schematic representation of (a) two phase and (b) three phase structures of syntactic foam.
2 LITERATURE SURVEY

2.1 Analytical Modeling

Syntactic foams are hollow particles (cenospheres) filled materials. These materials are very similar to the solid particle filled composites in the structure. Hence, one possibility is to apply models developed for the solid particulate composites to syntactic foams and check their validity. Some of these models have already been tested for syntactic foams by other researchers. A few relevant models are discussed here.

It is observed that large number of analytical models have been aimed at semi-empirical approach where models are developed based on the experimental results. In such approaches, it is difficult to eliminate the requirement of some empirical constants. However, recent approaches have gone beyond such attempts and have presented rigorous models to predict the mechanical behavior of syntactic foams. Some of the important theories are discussed below.

2.1.1 Early Efforts

Some of the earlier theories give simple relation between tensile strength of filled and unfilled epoxies. One such relation presented by Nielsen [13] is given by Equation 1.

\[
\varepsilon_c = \varepsilon_m (1 - \phi^{1/3})
\]

where \(\varepsilon_c\) and \(\varepsilon_m\) represent strains in the composite and the matrix material and \(\phi\) is the filler volume fraction. Lavengood et al [14] have tried to experimentally verify this model. These types of simple models neglect shear effect around the particle and also the Poisson’s ratio effect. Precise calculation cannot be performed using these models, however, they formed a base for the development of more complex models having greater number of parameters.

2.1.2 Influence Zone Theory

This theory developed by Liu et al [15] argues existence of an influence zone around each filler particle. The strength of the material in this influence zone is considered to be more than the strength of the matrix material. This model assumes a gradual change in the modulus of the matrix with respect to the distance within the influence zone. In the influence zone, the modulus and tensile strength values are given by Equations 2 and 3 respectively.

\[
E_I = K E_m
\]

and

\[
\sigma_I = K \sigma_m
\]

where \(K = (r + r')/a\)

Where \(E\) and \(\sigma\) represent the tensile modulus and the tensile strength respectively. Subscripts I and m represent influence zone and matrix respectively. Symbols \(r\) and \(r'\) represent radius of particle and measurement distance in the influence zone respectively. It is attempted to establish that the influence zone is different from the interfacial zone and has much greater thickness. Observation of influence zone is experimental and there is no proposed way to calculate it.
theoretically. This theory holds good only for low filler volume fractions where interaction between the influence zones is absent. However, in case of high filler volume fraction in the composite the influence zones of the particles will overlap. This will eventually give rise to a situation where modulus and strength of the polymeric matrix material at any point is greater than the original modulus and strength, which is in contrast with the experimental data.

2.1.3 Adhesion Bond Theory
This theory proposed by Papanicolaou and Bakos [13] assumes rigid filler particles as infinite number of coaxial cylinders. Direction of major axis of the cylinders is assumed to be in the direction of the applied load. This divides the problem into two parts, calculation of shear stress in the particle-matrix interface and the shear stress between coaxial cylinders assumed within the particles. The final relation obtained in their work is given by Equation 5.

\[
\sigma_p = \frac{E_f - E_m}{E_m} \sigma_m \left[ 1 - \frac{2(\tan^{-1}(e^{kr}) - \pi/4)}{k^2 r^2} \sinh(kr) \right]
\]

or

\[
\sigma_p = B \sigma_m
\]

where

\[
B = \frac{E_f - E_m}{E_m} \left[ 1 - \frac{2(\tan^{-1}(e^{kr}) - \pi/4)}{k^2 r^2} \sinh(kr) \right]
\]

The parameter \( k \) is defined as:

\[
k = 2G/[r^2 \ln 2(E_f - E_m)]^{1/2}
\]

where \( \sigma_p \) is the tensile strength of composite having filler, \( \sigma_m \) is the tensile strength of unfilled polymer, \( E_f \) is the modulus of the filler particle, \( E_m \) is the modulus of the matrix material and \( r \) is the radius of the filler particle. The model developed in this article for tensile strength of particulate composites is in very good agreement with some of the experimental data over a wide range of particle volume fractions (0-60%), but shows deviation for the other data set for the same volume fraction range. Good agreement is obtained for glass particles-epoxy system having solid particles of intermediate diameter (62 \( \mu \)m) and cenospheres of large diameter (147 \( \mu \)m). Deviations are observed for systems containing solid glass particles having small (21 \( \mu \)m) and large (216 \( \mu \)m) diameter and cenospheres of small diameter (77 \( \mu \)m).

Validity of adhesion bond theory for syntactic foams is a matter of concern, as dividing cenosphere particles into cylindrical components is not practical. In case they are divided into such components, body of the cylinders has to be imaginary and shear stresses along the length would be zero. Only the shear stress terms for cylinder-matrix interface will exist in such case. For cenospheres it is not sufficient to look only at the outer radius if the cenospheres fracture, wall thickness is important in drawing any conclusion. It is of equal importance to see if the particle fractured in the process of deformation or not. Cenospheres having low wall thickness can fracture under secondary tensile forces, which is highly unlikely for thick walled cenospheres or solid particles of any size.
2.1.4 Concentration Decrease/Void Addition (CD/VA) Theory

In the concentration decrease theory, proposed by Anderson et al [17-19], changes in internal stresses are related to the debonding process through the modified first law of thermodynamics given by Equation 9.

\[ \delta Q + \delta W = \delta U + G_c \delta A \quad 9 \]

where \( \delta Q \), \( \delta U \) and \( \delta W \) are the net heat transferred into the system, the net external work done on the system and the net internal energy in the system respectively. The term \( G_c \delta A \) represents the surface energy dissipated. Energy released is shown to be related to the variation in the material stress and strain by Equation 10.

\[ 2G_c \delta A/V_0 = \sigma_{ij} \delta \varepsilon_{ij} + \delta \sigma_{ij} \varepsilon_{ij} \quad 10 \]

where the subscript \( c \) represents the current filler concentration (after removal of debonded particles), \( \sigma_{ij} \) and \( \varepsilon_{ij} \) are components of stress and strain respectively and \( V_0 \) is the volume of composite.

The assumptions implied in this approach are that the particles do not fracture and debonding or matrix failure leads to the final failure of the composite. Assumption of debonding as main mode of failure makes this theory highly dependent on interfacial strength, which is not a practical approach. This assumption is valid in case the interfacial strength is more than the strength of the matrix material, matrix failure would prevail. However, this is not a good assumption for cenosphere filled materials where fracture of particles can start well before the fracture of the interface or the matrix.

Combined CD/VA theory gives a more practical approach. According to this theory, the debonded particle is replaced by an equivalent void. Final form of this model is given by Equation 11.

\[ -2 G_c/V_0 (dA/dc) = \varepsilon^2_{11} (dE/dc) \quad 11 \]

Fracture of thin walled cenospheres is possible under secondary tensile stresses in the lateral direction; hence, this assumption can lead to valid results. However, in energy balance equations an additional term is required for the energy consumed in the fracture of cenospheres. Experimental verification of this model is carried out by Wong et al [20]. Experimental results are compared with the values calculated by using this theory for two types of particle surface coatings (denoted as T and U). Close agreement is observed for coating type T with both, small and large particle size, but only in large volume fractions. With another type of coating U good agreement is found only with small particle size at low volume fractions. For all other cases large deviations between experimental and calculated values are observed. In this study particles are solid glass beads. Observation that the strength values decrease with an increase in particle size is consistent with the experimental data obtained from other sources. Comparison of results shows strong dependence of results on interfacial strength. The observed difference may be due to the change in the stress concentration factor at the particle location due to debonding. The
change will be significantly different for the small and the big particles due to a difference in their diameter (radius of curvature).

2.2 Experimental Studies

Mechanical properties of syntactic foam can be tailored over a very wide range depending on the requirements of the application. Suitable matrix materials can be selected from a vast range of polymeric resins and hardeners. Cenospheres of polymers [21, 22], metals [23] or ceramics [10] can be chosen according to the specific requirements. Additional reinforcement can also be added to syntactic foams by incorporation of fibers in continuous or discontinuous form [24, 25].

Syntactic foams have been gaining popularity as core materials in sandwich structured composites. Some experimental studies [26, 27] characterizing syntactic foam core sandwich composites could be found published in the literature. The syntactic foam core sandwich composites are used as buoyancy aid material in ships and boats. They are also used in aircraft and spacecrafts [28] due to their lightweight.

Several experimental and analytical studies are available on compressive [29, 30], impact [31-33] and hygrothermal [34-36] properties of syntactic foams. Viscoelasticity [22], fire performance [37] and effect of polymer cure cycle [38] can also be found studied in the published literature. Syntactic foam core sandwich composites have also been studied by some researchers for compressive [26, 27], impact [39, 40] and flexural [41] properties.

None of these studies investigate the effect of the cenosphere internal radius on the mechanical properties of syntactic foam and use this parameter as a means of changing the syntactic foam density. In most of these studies either just one type of syntactic foam is fabricated and tested for mechanical properties or the effect of change in the volume fraction is investigated [42]. Choice of constituting materials for syntactic foams is so large that it is difficult to find many studies using the same constituents. Hence, direct comparison of the results from different experimental studies is not possible.
3 RESEARCH OBJECTIVE
The objective of the present research is to develop an understanding for the deformation and fracture behavior of closed cell structured foams such as syntactic foams. The mechanical and hygrothermal properties of syntactic foams are investigated for potential use as core in the sandwich structured composites for weight sensitive applications. The fracture pattern and mechanical properties of syntactic foam core sandwich composites are also evaluated for a variety of loading conditions.

The proposed research covers three approaches namely Analytical Modeling, Finite Element Analysis and Experimental studies to characterize syntactic foams for various mechanical properties. In two-phase syntactic foams contribution of each constituent in the deformation and fracture process is identified using a unique approach of changing only one parameter of one phase. A parameter named \( \text{Radius Ratio} \) is defined in this study as the ratio of the internal to the outer radius of cenospheres. The deformation behavior, fracture pattern and mechanical properties of syntactic foams and their sandwich composites are analyzed and evaluated for a variation only in Radius Ratio keeping all other parameters the same.

3.1 Analytical Modeling
The deformation and fracture behavior of syntactic foams depends upon the properties of cenospheres. Following are the main objectives of the modeling in this study:

1. Categorize cenosphere filled composites.
2. Develop a modeling approach for various categories.
3. Determine the material deformation and fracture behavior based on the developed approach.

The stress concentration factor approach is adopted to model the deformation and failure characteristics of syntactic foams. It is assumed that cenospheres under study are hard and brittle and are spherical in shape.

Variation of different parameters and their effects on such systems are to be discussed in this study. The parameters included in the modeling approach are the strength of particle and internal radius of cenospheres.

3.2 Finite Element Analysis
Contact analysis is performed using finite element analysis (FEA) method to analyze the effect of the interfacial strength and Radius Ratio, \( \eta \), on the deformation behavior of syntactic foams. The analysis is performed on a two-dimensional model. The contact analysis is aimed at finding out the effect of interfacial strength between the matrix and cenosphere on the debonding strain of the material. The debonding strain values obtained for various interfacial strengths are used to determine the fracture mode of the material. Friction between cenosphere and the matrix material is taken as the measure of the interfacial strength and is varied from 0 to 0.9.

3.3 Experimental
The objective of the experimental study is to investigate and establish the deformation and fracture behavior of syntactic foams and syntactic foam core sandwich composites. The effect of
change in cenospheres density, by means of changing cenosphere $\eta$, on mechanical properties of syntactic foams and their sandwich composites is explored extensively by taking five different types of cenospheres. The following approaches are adopted to achieve this objective.

1. Selection of appropriate constituent materials, e.g. matrix resin, hardener, diluent, cenospheres etc.
2. Development of a fabrication process to fabricate syntactic foams and sandwich composites of consistent quality. Fabrication of syntactic foam slabs and syntactic foam core sandwich composites.
3. Testing of fabricated materials. Physical and mechanical tests that would be conducted based on the appropriate ASTM standards for the testing of syntactic foam and sandwich composites as listed in Table 1 and Table 2 respectively.

Table 1 ASTM Standards selected for the testing of syntactic foams.

<table>
<thead>
<tr>
<th>Property</th>
<th>ASTM Standard Number</th>
<th>Standard Title</th>
</tr>
</thead>
</table>

Table 2 ASTM Standards selected for the testing of syntactic foam core sandwich composites.

<table>
<thead>
<tr>
<th>Property</th>
<th>ASTM Standard Number</th>
<th>Standard Title</th>
</tr>
</thead>
</table>

*Flexural properties have been measured in Three-point bending, Four-point bending and Short beam shear configurations.
4 RESULTS AND DISCUSSION

4.1 Analytical Approach
Stress Concentration factor (SCF) approach is developed for cenosphere filled materials. Relation between SCF and Radius Ratio is developed to classify the cenosphere filled materials in two different classes based on Radius Ratio.

4.1.1 Stress Concentration Factor
The SCF is defined as the ratio of maximum stress ($\sigma_{\text{max}}$) in the material due to presence of any inhomogeneity to the stress away from that inhomogeneity, termed as nominal stress ($\sigma_{\text{nom}}$).

\[ k = \frac{\sigma_{\text{max}}}{\sigma_{\text{nom}}} \]

where $k$ represents the SCF.

Local stress can be higher than the applied stress due to stress concentration caused by any inhomogeneity in the material. In the case of a spherical particle the maximum stress due to the stress concentration is achieved at the surface of the particle and is three times the stress present away from it. For particles or inhomogeneities of other shapes it can be even higher. SCF for a variety of discontinuities and for different loading conditions can be found in the published literature [43, 44].

4.1.2 SCF in Particle Filled Epoxies
A single particle system under unidirectional tensile stress is considered in this study. Figures 5a and 5b show composite material systems having a solid particle and a cenosphere embedded in the matrix, respectively. The particles in the present study are assumed to be spherical in shape. The parameters that are studied here are the strength of particle, interfacial strength between the particle and the matrix resin and also the differences arising due to the particle being hollow or solid.

Figure 5 Assumed single particle system and coordinate system for analytical study.
The deformation and fracture characteristics of particulate composites can be divided into two categories. These are

1. Interface fractures before the particle fractures,
2. Fracture of particle.

If debonding is the first failure mechanism the void created due to debonding determines the SCF. If particle fractures during deformation of the composite the change in SCF depends on the shape and dimensions of the region occupied by the particle fragments. Both these conditions are analyzed and discussed below.

4.1.2.1 Failure of the Interface
When the failure mode is the particle-matrix interfacial failure, an ellipsoidal shaped void is formed around the particle. The void geometry and the size remain the same for cenosphere or solid particles and are schematically shown in two-dimensional representation in Figures 6a and 7a, respectively. Here ellipsoid reduces to ellipse in two-dimensional representation. Convention used for dimensions and the loading direction for the ellipse is shown in Figure 8. For elliptical shaped void the SCF can be given as shown in Equation 13.

\[
k = \left(1 + 2 \frac{a}{c}\right)
\]

where \(2a\) is the axis of the ellipse perpendicular to the direction of tensile stress and \(2c\) is the axis in the direction of the tensile stress.

---

![Figure 6 Cenosphere filled system showing (a) debonding and (b) particle fracture.](image-url)
The void formed due to the fracture of interface is free to enlarge in the direction of tensile stresses. However, as the particle is still intact, there will be no change in the dimensions in the transverse directions. Equation 13 can be re-written using the boundary conditions in the x-direction as shown in Equation 14.
$$k = \left(1 + \frac{r_0}{c}\right)$$  \hspace{1cm}  \text{(14)}

When the particle does not fracture, deformation and debonding patterns remain symmetric to the axis of the applied stress, the z-axis. Hence, the three-dimensional problem can be reduced to two dimensions for modeling. Symmetry along Z-direction can be applied to the two-dimensional solution to derive a three-dimensional solution. The equation of the ellipsoidal shape created by debonding can be given as Equation 15.

$$\left(\frac{x}{a}\right)^2 + \left(\frac{y}{b}\right)^2 + \left(\frac{z}{c}\right)^2 = 1$$  \hspace{1cm}  \text{(15)}

where the dimensions of the ellipsoid in x, y and z direction are denoted as a, b and c and the relation between them is given as

$$a = b < c$$  \hspace{1cm}  \text{(16)}

for the given loading conditions. Equation 15 can be reduced to two-dimensional equation of ellipse as given below in Equation 17 and the corresponding parametric equations for the surface of the ellipse can be given by Equations 18 and 19.

$$\left(\frac{x}{a}\right)^2 + \left(\frac{z}{c}\right)^2 = 1$$  \hspace{1cm}  \text{(17)}

$$x = a \cos \alpha$$  \hspace{1cm}  \text{(18)}

$$z = c \sin \alpha$$  \hspace{1cm}  \text{(19)}

In the given condition when particles do not fracture, no change is possible in the x and y dimension of the void. Hence, following conditions can be applied to Equation 15 based on this consideration

$$a = b = r_0$$  \hspace{1cm}  \text{(20)}

The x and y dimensions do not depend on the applied stress and the strain in the material as the particle does not fracture. The z dimension tends to increase from the initial value of r₀, which is the radius of the particle and depends on the strain in the material. The condition for the third dimension can be given by Equation 21, where third dimension is expressed as a function of applied stress.

$$c = r_0 + f(\sigma)$$  \hspace{1cm}  \text{(21)}

where for small strain in the material the conditions can be modified as given below in Equation 22.
\[ c = r_0 + \varepsilon r_0 \]
\[ c = r_0 (1+\varepsilon) \]  

If the volume fraction of the filler is low enough so that the stress fields of different particles do not interfere with each other, the system can be modeled as a single particle system. Based on such an assumption the solutions for the internal elliptical crack problems can be applicable for this situation. Kassir and Sih [44] have presented solutions for such problems where Equation 17 can be taken as the equation of the elliptical inhomogeneity and the boundary conditions given in Equations 20 and 21 can be applied to it.

4.1.2.2 Failure of Particle

When a particle fractures, there is a sudden change in the dimensions and the shape of the region previously occupied by it. This reflects as a sudden change in the SCF at the point of particle fracture irrespective of the fact that particle-matrix interface has failed or not. The sudden jump in the stress value may lead to localized failure of the matrix material in dynamic loading conditions. Considerable differences are observed in the local stress condition due to the fracture of cenospheres and solid particles. These differences are identified and analyzed below.

4.1.2.2.1 Fracture of Cenospheres

Fracture of cenospheres exposes the volume enclosed within them and forms a void in the matrix. This additional volume is now available for the matrix material surrounding the particle to occupy. The situation arising from the fracture of cenospheres is schematically shown in Figure 6b.

Suppose the internal radius of the cenosphere is \( r_1 \) and the outer radius is \( r_0 \) as shown in Figure 8. The Radius Ratio, \( \eta \), for the cenosphere is defined as given by Equation 23.

\[ \eta = \frac{r_1}{r_0} \]

Cenosphere wall thickness can be related to \( \eta \). Keeping the outer radius the same, a decrease in wall thickness increases \( \eta \). Calculation of \( \eta \) requires knowledge of the outer radius of the cenosphere and the true particle density. Both these quantities can be experimentally determined and \( \eta \) can be calculated. The volume occupied by the particle before fracture (\( V_0 \)) is

\[ V_0 = \frac{4}{3} \pi r_0^3 \]

A cenosphere has hollow space within and the volume of the material used in making the cenosphere wall (\( V_m \)) can be given by Equation 25.

\[ V_m = \frac{4}{3} \pi (r_0^3 - r_1^3) \]

Equation 25 can be written in the form of \( \eta \) as given by Equation 26
The volume that the cenosphere debris occupies after its fracture depends on $\eta$. The higher values of $\eta$ lead to thinner cenosphere walls. Hence, even if the outer radius of the cenospheres is the same, the volume occupied by debris, called effective volume ($V_E$), after cenospheres fracture depends on their $\eta$ values. Fragments generated due to the crushing of the particle do not exist in the form of dense compact mass. These fragments are subjected to rotational movement due to shear stresses and linear movement in favorable direction due to local tensile and compressive stresses. 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 $V_m$. On further crushing, these fragments tend to break into smaller pieces and become more and more compact. The volume occupied by fragments is termed as “Effective Volume”, represented by $V_E$. If the cavity to particle diameter for the cenospheres is very high (close to 1), the volume of the fragments generated is extremely small compared to the volume of the cavity. However, if this ratio is low enough the effective volume of debris due to the presence of voids between the fragments may be more than the volume of the cenosphere before fracture. Due to a mismatch between the various fractured pieces, $V_E$ is always more than the volume $V_m$. However, the stress state in the material after the cenosphere fractures depends on the difference between cenosphere volume ($V_c$) and $V_E$.

A lower bound to the volume occupied by debris can be determined. It is assumed that the fragments of the cenospheres are spherical in shape and all fragments are of same size. The volume of material constituting the cenosphere can be equated to the volume of the material constituting spherical fragments. Random close packing factor for the spheres of equal size is approximately given as 0.64 [17]. The effective volume $V_E$, is given by Equations 27 and 28.

$$V_m = \frac{4\pi}{3} r_0^3 \left(1 - \eta^3\right)$$  

27

$$V_E = 6.54 r_0^3 \left(1 - \eta^3\right)$$  

28

The fracture of a cenosphere leading to the generation of smaller particles gives rise to a situation which can be visualized as an ellipsoidal void in the matrix material filled with unbonded smaller particles. This is similar to having an ellipsoidal void in the matrix as observed in the debonding failure without the fracture of the cenosphere, however, now the matrix does not have constraint in x and y directions. Hence, Equation 15 represents the shape of the void in this case also, but the boundary condition given by Equations 20 and 22 take the form of Equations 29 and 30.

$$a = b < r_0$$  

29

$$a = b = f_1(-\nu\sigma)$$  

30

where $\nu$ is the Poisson’s ratio. For the third axis the condition can be given as
These conditions are valid as long as the effective volume is less than or equal to the volume of the cenosphere before fracture.

\[
\frac{V_E}{V_0} \leq 1
\]

\[
\frac{1}{0.64}(1 - \eta^3) \leq 1
\]

\[
\eta^3 \geq 0.36
\]

where \( \eta \) is the Radius Ratio and is defined by Equation 23.

\[
\eta \geq 0.71
\]

The value of \( \eta \) at which transition in deformation is observed is termed as \( \eta_{cr} \), which is equal to 0.71 according to Equation 35. At this value of \( \eta \) the volume of debris generated due to the cenosphere fracture will be equal to the volume of the particle before fracture.

Similar to the case when the particle does not fracture, solutions for the elliptical crack can also be applied to this case. However, the boundary conditions to the equation of ellipse, Equation 17, would be defined as given by Equations 29 and 30. The values of elastic constants for the inhomogeneity in this solution should be taken as zero. This will lead to a situation where the matrix is able to deform without any constraint from the particle or the fragments. The general equations given by Kassir and Sih [44] are applicable for this case also and can accommodate any loading condition and direction.

When \( \eta \) is less than 0.71, the effective volume of debris becomes more than the volume of the particle before fracture. It should be noted that the solid particle could be taken as a special case of cenospheres where size of the void is zero

\[
r_1 = 0
\]

which means the Radius Ratio

\[
\eta = 0
\]

A comparison between the stress conditions generated in the composite containing cenospheres having \( \eta < 0.71 \) and \( \eta > 0.71 \) reveal remarkable differences. The situation arising from the fracture of cenospheres having \( \eta < 0.71 \) is schematically shown in Figure 7b, which in fact, is also the case of solid particles. Apart from the applied stress, the stress generated due to the increased effective volume of the cenosphere having \( \eta < 0.71 \) after its fracture must also be
taken into account now. Hence, the effective stresses in the material will be the net sum of the applied stress and the stress due to increase in the effective volume of the particle. Suppose the applied stress is $\sigma_0$ and the internal stress generated due to the increased effective volume of the particle is $\sigma'$. The total stress in this case will be

$$\sigma = \sigma_0 + \sigma'$$  \hspace{1cm} (38)

The increased value of stress would lead to additional strain in the material. The additional stress generated due to the increase in effective volume of the particle would depend on the size of the ellipsoidal shape generated, which in turn depends on $\eta$. The strain generated due to the increase in effective volume, $\varepsilon'$ is inversely proportional to $\eta$ as shown in Equation 39.

$$\varepsilon' \propto \frac{1}{\eta}$$  \hspace{1cm} (39)

$\varepsilon'$ is dependent on the properties of the matrix material. This additional component of the strain can be calculated by using the value of $\sigma'$ and the modulus of the matrix. The relation to find the dimensions of the ellipse after the fracture of the particle would contain an additional term due to the effect of the increased effective volume. Dimensions of the ellipse in the direction of applied tensile stress and the transverse direction for small strain value can be given as Equations 40 and 41, respectively.

$$c' = c(1 + \varepsilon + \varepsilon')$$  \hspace{1cm} (40)

$$a' = a(1 - \nu \varepsilon + \varepsilon')$$  \hspace{1cm} (41)

Based on Equations 40 and 41, it can be concluded that the stress concentration factor shows a sudden change from a value of 3 when the spherical particle is intact, to a value of

$$k = \left(1 + \frac{a(1 - \nu \varepsilon + \varepsilon')} {c(1 + \varepsilon + \varepsilon')} \right)$$  \hspace{1cm} (42)

The SCF given by Equation 42 can be used to find the value of maximum stress in the material.

### 4.1.2.3 Conclusions

The SCF approach for cenosphere filled composites based on the ratio of the inner radius to the outer cenosphere radius, termed as Radius Ratio ($\eta$), is developed and discussed in the study. This approach is extended to the solid particle filled composites by assuming the cavity diameter to be zero. The approach is divided into two different cases. In the first case where the particle-matrix interface fails before the particle fracture, the boundary conditions are determined to solve the deformation problem. The second case where the cenosphere fracture during the deformation process is sub-divided based on $\eta$. A critical value of $\eta$ is found as 0.71. If cenospheres having $\eta \geq 0.71$, tend to fracture before the interface fails, it is found that the problem reduces to a simple case of the matrix having an ellipsoidal void. However, fracture of cenospheres having $\eta < 0.71$ leads to an increase in the effective particle volume, which results in an additional
component of stress. Value of this additional component directly depends on the value of $\eta$. Relations are developed to calculate the SCF for such conditions.

4.2 Finite Element Analysis

It is theoretically established in the previous section that a cenosphere having $\eta$ below a critical value, $\eta_{cr}$, behaves like a solid particle, especially when the cenosphere tends to fracture during the deformation process. Packing mismatch among the fragments generated due to the fracture of these particles can lead to an effective increase in the volume of the debris. Fracture of a cenosphere having $\eta$ more than 0.71 leads to a situation of having a void in the matrix, whereas fracture of cenosphere having $\eta$ less than 0.71 generates compressive stresses in the adjacent matrix material. To understand and predict the effect of $\eta$ on the deformation behavior of cenosphere filled composites FEA can be a very effective technique. The effect of interfacial strength between cenosphere and matrix, which is not found discussed in the published literature on syntactic foams extensively, can also be analyzed by this technique at the same time.

4.2.1 Cenosphere Filled Polymers

There are four major parameters in the modeling of cenosphere filled polymers. The first and the second are the mechanical properties of cenospheres and matrix resin respectively. The third parameter is the interfacial strength between cenosphere and the matrix. The fourth parameter is the volume fractions of various raw materials in the structure of the syntactic foam. Mechanical properties of cenospheres involve parameters such as the cenosphere size and density. Cenosphere size can be a measure of contact surface area between the matrix and the cenosphere. For constant volume fractions of cenospheres and matrix the interfacial area can be increased by decreasing the cenosphere size. Density of cenospheres varies based on the internal radius, which is included in the modeling in the form of $\eta$.

4.2.2 FEA Parameters

Contact analysis is performed using FEM software ANSYS™. In this analysis a two-dimensional model of a cenosphere in the matrix resin is constructed and meshed with 8-node PLANE82 elements. Initially 4-node elements were used for mesh, however, results were not reliable as some of the elements become very stiff. Three different mesh sizes were tested with 8-node elements to check the convergence of the model, based on which medium mesh size is selected. Total 1029 nodes are present in the model. Newton-Raphson method is selected to solve the problem. Contact element is generated between the cenosphere and the matrix resin. The cenosphere surface is selected as target in contact pair due to its higher stiffness and matrix surface is selected as contact surface. Properties of contact elements are considered to be the same as the properties of the matrix material. In the analytical approach the critical Radius Ratio, $\eta_{cr}$, is identified as 0.71. Hence, first the analysis is carried on a 2-dimensional model with cenospheres having Radius Ratio equal to 0.71. In the second stage of analysis, cenospheres having $\eta$ values of 0.9 and 0.95 are constructed and analyzed.

The coefficient of friction at the cenosphere-matrix contact surface is considered to be a measure of the interfacial strength in this study. Both the friction and the interfacial strength, resist the relative motion between the cenosphere and the matrix resin. Hence, among the available choice of parameters in this FEA method, coefficient of friction seems to be the most reasonable choice. If coefficient of friction is taken as zero, it corresponds to a situation where there is no interfacial
bonding between the cenosphere and the matrix resin. However, the coefficient of friction value of 1 represents the case where there is perfect bonding between the cenosphere and the matrix resin and there is no relative movement. Mechanical properties of the matrix are assigned to the contact pair also. Hence, in the case of perfect bonding the interface will show yielding or failure at the yield of failure level for the matrix. Appropriate boundary conditions and degree of freedom (DOF) couplings are defined in the model to translate the strain in the loading direction to the strain along the cenosphere surface due to Poisson’s Ratio effect.

Model of syntactic foam that is used in the FEA is shown in Figure 9. The DOF in the y-direction is coupled for nodes along Line 1 and Line 2 shown in Figure 9. For two nodes shown as coupled nodes 1 and 2, all DOFs are coupled. It is necessary to couple these nodes to translate friction into strain in the material. Pressure is applied along Lines 1 and 2. Line 3 in Figure 9 shows the location of contact pair. A meshed model and the contact pair developed in ANSYS™ are shown in Figures 10 and 11 respectively. Symmetry boundary conditions are also applied to facilitate the three-dimensional axi-symmetric expansion of the model along the y-axis after the analysis.

Figure 9 Details of the model used for finite element analysis.
4.2.3 Results and Discussion

In the first part of the analysis the coefficient of friction is varied from 0 to 0.9 in the intervals of 0.1. For each of these cases the strain at which interfacial separation starts and the maximum and the minimum strains in the matrix material are noted. An important finding of the experimental results, discussed in Section 4.3, is that the strain at peak compressive stress is independent of the cenospheres $\eta$. It is found that for all five types of cenospheres taken in the experimental study, the strain at peak stress exists within a narrow range around 3%. Hence, a comparison of strain value is necessary to determine the failure mode in the material.

In this study the nodal solutions obtained for various friction coefficient values are compared. Axi-symmetrically expanded model and one of the nodal solution plots for stress in the x-direction are shown in Figures 12 and 13, respectively.

In the analysis, about 8% compression of the specimen is achieved as a result of the applied pressure. It is observed that for higher values of friction coefficient, debonding between cenosphere and matrix takes place at higher strain. Results show that for a friction coefficient value of 0.3 the specimen strain is about 3%, which lies within the experimentally obtained strain.
range. It is also observed that much higher strain is required for debonding when friction coefficient of 0.9 is used. It is noted that the maximum compressive strain in the y-direction at the contact pair varies between 0.179% and 0.135% for 0 and 0.9 friction coefficient values respectively. Corresponding tensile strains in the x-direction are between the values of $9.49 \times 10^{-2}\%$ and $7.34 \times 10^{-2}\%$ respectively. These are also the values of strain at the cenosphere surface, as the contact element is shared by both matrix and cenosphere. If this strain in the cenosphere exceeds the cenosphere fracture strain, the cenosphere would fracture before the failure of the interface.

Nodal stress solutions in the x-direction show that the maximum stress changes from 0.9 MPa to 3.3 MPa for variation in friction coefficient from 0 to 0.9. Compressive stress in the y-direction varies between 4.04 and 4.16 MPa in the same range of friction coefficient. It must be noticed that the incorporation of brittle rigid particles brings the strength of the polymer down as they act as stress risers in the material. However, the gain from incorporation of cenospheres is the increased energy absorption during compression. This is evident in the compression test stress-strain behavior, which shows an extended region of constant stress up to 8-10% strain after peak stress.
The next set of FEA is run for cenospheres having $\eta$ value of 0.95 and 0.9 with friction coefficient of 0.3 applied to the contact elements. These cases are close to the $\eta$ values of cenospheres selected for the experimental study. In these cases it is observed that the strain in the cenosphere exceeds 1% due to their much lower wall thickness compared to the cenosphere having $\eta$ value of 0.71. Glass cenospheres have fracture strain of 1% or less. Hence, both cenosphere fracture and debonding, would exist in the actual material at the same time contributing to the final failure.

4.2.4 Conclusions
Based on the FEM contact analysis performed on a single cenosphere containing polymer, which is a syntactic foam type material, it is concluded that
1. The first part of the analysis is carried out on a cenosphere having $\eta$ value of 0.71. For the friction coefficient value of 0.3 the strain at the point of debonding between cenosphere and the matrix material is within the experimentally found strain for peak strength.
2. For models having cenospheres Radius Ratio of 0.9 and 0.95 the analysis is carried out at a coefficient of friction of 0.3. The strain on the surface of the cenosphere in this analysis is about 1%. This can lead to the fracture of cenospheres along with interfacial failure.

4.3 Experimental Study
Relevant ASTM standards have been followed for the experimental work in this study. ASTM standards have been given preference over any other standards that may exist for similar kind of materials and testing conditions.

4.3.1 Raw Materials
Details of raw materials used for the fabrication of syntactic foams are given below.

4.3.1.1 Matrix Resin
Based on a comparative study of various commercially available epoxy resins, D.E.R. 332, a di-epoxy resin, manufactured by DOW Chemical Company is selected for the study. This resin is called as diglycidyl ether of bisphenol A (DGEBA). This resin has lower viscosity compared to other similar resins, which would help while mixing large volume fraction of cenospheres. Chemical name of D.E.R. 332 resin is 2,2-bis[4-(2’3’ epoxy propoxy) phenyl] propane. Average epoxide equivalent weight of the epoxy resin is 174. This resin has high purity, has lack of polymer fractions and provides uniform performance as per the manufacturer’s recommendation. The chemical formula of this resin is presented in Figure 14.

```
CH2 — CH — CH2 — O — C — C — O — CH2 — CH — CH2
     |     |     |     |     |     |     |
     CH3   O   CH3
```

Figure 14 Chemical formula of the DGEBA resin used as the matrix material.
4.3.1.2 Diluent
Large volume fraction of cenospheres can be mixed in the resin properly if the viscosity of the resin is low. Hence, a diluent is added to lower the viscosity of the resin mix. Adding diluent C_{12}-C_{14} aliphatic glycidyl ether, commercially known as ERISYS-8 in 5% by weight quantity brings down the viscosity of the resin from about 4 N-s-m^{-2} at 20°C to about 2 N-s-m^{-2}. This diluent is supplied by CVC Specialty Chemicals and its chemical formula is shown in Figure 15. As per the supplied datasheets by the manufacturer of this compound, addition of up to 5 wt % of this diluent increases the tensile strength and modulus of epoxy resins. Average equivalent epoxide weight (EEW) of the diluent is 285.

4.3.1.3 Hardener
Triethylene tetramine (TETA), a polyfunctional aliphatic amine, is used as a curing agent. This chemical is commercially known as D.E.H. 24 and manufactured by DOW Chemical Company. The chemical formula of TETA is C_{6}H_{18}N_{4}, which is shown in Figure 16. The amines react with the epoxy group through the active amine hydrogen. Molecular weight of this hardener is 146.4 and weight per active hydrogen is 24.4. Aliphatic amine based hardeners cure at room temperature, provide excellent chemical and solvent resistance to the polymer solutions and retain physical properties in the long term.

4.3.1.4 Cenospheres
Five different types of borosilicate glass cenospheres are used for the fabrication of syntactic foam specimens in this study. These cenospheres are manufactured and supplied by 3M under the trade name “Scotchlite”. Distribution of outer diameter of all types of cenospheres is nearly the same, but the internal diameter is different. This causes a difference in the density of different types of cenospheres. Particle size distribution of cenospheres supplied by the manufacturer is given in Table 3. Average cenosphere density, crush strength, calculated average wall thickness and η based on cenosphere densities are given in Table 4. The number in the cenosphere type denotes the cenosphere density.

4.3.1.5 Glass Fabric
Glass fabric is used to fabricate skins of sandwich composites. E-glass fabric with epoxy compatible coating is used for this purpose. This fabric has plain weave pattern and 93 g/m² weight.

\[
\text{ROC H}_2 \text{CH CH}_2 \text{O} \]

Figure 15 Chemical formula of Aliphatic Glycidyl Ether. Here R = C_{12} to C_{14}.

\[
\text{NH}_3 \rightarrow \text{CH}_2 \rightarrow \text{CH}_2 \rightarrow \text{NH} \rightarrow \text{CH}_2 \rightarrow \text{CH}_2 \rightarrow \text{NH} \rightarrow \text{CH}_2 \rightarrow \text{CH}_2 \rightarrow \text{NH}_3
\]

Figure 16 Chemical Structure of hardener molecule.
4.3.1.6 Mold
Two stainless steel molds having inner dimensions of $229 \times 229 \times 13 \text{ mm}^3$ are used for casting the syntactic foam slabs. No vacuum or pressure is applied during the casting or curing of syntactic foam slabs to avoid fracture of cenospheres during the fabrication process.

4.3.1.7 Mold Release Agent
Dow Corning 111 Sealant and Lubricant is used as a release agent in the molds. This lubricant is silicone based white translucent gel. Selection of this release agent is based on its service temperature range of $-40$ to $204^\circ \text{C}$ and bleed characteristics, $0.5\%$ in 24 hrs at $200^\circ \text{C}$. Specific gravity of this release agent is 1.0.

4.3.2 Materials Processing and Fabrication

4.3.2.1 Raw Material Calculations
In the matrix material epoxy resin to diluent ratio is kept at 19:1 parts by weight. Average equivalent epoxide weight (EEW) of the diluent is 285. EEW of a mixture of resin and diluent can be calculated using Equation 43.

\[
\text{EEW} = \frac{100}{\frac{w_1}{\text{EEW}_1} + \frac{w_2}{\text{EEW}_2}}
\]

Table 3 Particle size distribution of cenospheres.

<table>
<thead>
<tr>
<th>Cenosphere Type</th>
<th>Particle Size Distribution (µm)</th>
<th>Effective Top Size</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10th</td>
<td>50th</td>
</tr>
<tr>
<td>S22</td>
<td>20</td>
<td>35</td>
</tr>
<tr>
<td>S32</td>
<td>20</td>
<td>40</td>
</tr>
<tr>
<td>K37</td>
<td>20</td>
<td>40</td>
</tr>
<tr>
<td>S38</td>
<td>15</td>
<td>40</td>
</tr>
<tr>
<td>K46</td>
<td>15</td>
<td>40</td>
</tr>
</tbody>
</table>

Table 4 Physical properties of cenospheres.

<table>
<thead>
<tr>
<th>Cenosphere Type</th>
<th>Average True Particle Density (kg/m$^3$)</th>
<th>Pressure for Min. 80% Fractional Survival (MPa)</th>
<th>Average Wall Thickness (µm)</th>
<th>Radius Ratio η</th>
</tr>
</thead>
<tbody>
<tr>
<td>S22</td>
<td>205</td>
<td>2.76</td>
<td>1.26</td>
<td>0.922</td>
</tr>
<tr>
<td>S32</td>
<td>320</td>
<td>13.79</td>
<td>1.86</td>
<td>0.907</td>
</tr>
<tr>
<td>K37</td>
<td>370</td>
<td>20.68</td>
<td>2.17</td>
<td>0.891</td>
</tr>
<tr>
<td>S38</td>
<td>380</td>
<td>27.58</td>
<td>2.23</td>
<td>0.888</td>
</tr>
<tr>
<td>K46</td>
<td>460</td>
<td>41.37</td>
<td>2.74</td>
<td>0.863</td>
</tr>
</tbody>
</table>
where \( w_1 \) and \( w_2 \) are the weight fractions of the resin and the diluent respectively. \( \text{EEW}_1 \) and \( \text{EEW}_2 \) are the average equivalent epoxide weights of resin and diluent respectively. For a 95 wt% resin and 5 wt% diluent mixture the EEW is calculated as shown in Equation 44.

\[
\text{EEW} = \frac{100}{\frac{95}{174} + \frac{5}{285}} = 177.5 
\]

Hydrogen functionality: 6
Amine H Equivalent Weight: \( \frac{146.3}{6} = 24.383 \)
Phr of amine for pure resin: \( 24.383 \times \frac{100}{174} = 14.01 \)
Phr of amine for 95-5 resin mix: \( 24.383 \times \frac{100}{177.5} = 13.74 \)

Phr represents parts per hundred parts of resin. As per the calculation, 13.74 parts of hardener are mixed with 100 parts of resin-diluent mixture. For the selected combination of epoxy resin and hardener the curing schedule is to gel at room temp and post cure at 100°C for 1-2 hrs.

Determination of cenospheres and resin volume fractions is based on a conservative estimate of the interstitial spaces between cenospheres. It is required to have the maximum possible volume fraction of cenospheres in the syntactic foam structure to obtain the minimum possible density using selected cenospheres. However, if sufficient quantity of resin is not used, the void content in the syntactic foam structure will increase, which is undesired. For spherical particles of the same size the random close packing factor is 0.65 [45]. For spheres of different sizes the packing factor may be 0.9 or even higher. Hence, it is assumed that if proper mixing and wetting is achieved, 35 % resin by volume would fill all the spaces between cenospheres if they are present in random close packed arrangement. Hence, the cenosphere volume fraction in the fabricated syntactic foams is maintained at 0.65 in all types of syntactic foam slabs.

### 4.3.2.2 Fabrication Process

Low viscosity of the resin system is desired to ensure uniform mixing and complete wetting of cenospheres. Hence, the resin and the diluent are mixed together and heated to 50°C to further reduce the viscosity of the mix. Subsequently, the hardener is mixed followed by cenospheres addition to this resin system mixture. This mixture is then cast in molds and allowed to cure. All the fabricated slabs are cured for 36 hrs at room temperature and then post cured for 3 hrs at 100±3°C.

To fabricate sandwich composite slabs four layers of glass fabric are laminated on either side of the fabricated syntactic foam slabs. Epoxy resin system, which is used to fabricate syntactic foam, is used to fabricate skins also. Hand lay-up followed by vacuum bagging process is used to laminate skins directly on the syntactic foam slabs. In the sandwich composites the core to the skin thickness ratio is measured to be 15:1. Fabricated sandwich panels are cured at room temperature for at least 96 hrs before trimming and cutting.
4.3.2.3 Density Measurement
To measure the density of the fabricated syntactic foam material standard ASTM C 271-94 [46] is followed. This standard is for measuring the density of sandwich core materials. This standard is selected considering the intended use of the fabricated syntactic foam slabs as core material in sandwich composites. Results of density calculation of fabricated syntactic foam specimens are shown in Table 5. The density values are obtained by measuring dimensions and weight of at least 8 pieces of $25 \times 25 \times 12.5$ mm$^3$ dimensions.

4.3.3 Testing Parameters

4.3.3.1 Hygrothermal Tests
For the moisture absorption study ASTM D 5229-92 standard [47] is followed. After careful study of all the recommended procedures, the procedure “B” described in this standard is considered suitable for this study. According to this procedure specimens should be immersed in the moisture till they reach saturation stage. Criteria specified for the saturation stage is that the change in the weight should be less than 0.1% in 7 days. Specimens subjected to hygrothermal tests are referred as “wet” specimens here.

The hygrothermal experiments are conducted at two different temperatures, room temperature and 70°C. In addition to that, the tests are carried out in DI water and also in salt water. Instant ocean™ synthetic sea salt is used to make sea water like composition of the salt water. High temperature specimens are kept in an oven that had air circulation facility and temperature is maintained at 70±3°C. Specimens are kept in closed container to minimize the evaporation losses. Evaporation of water would change the salt concentration in the salt water, which is undesirable. Specimens are briefly taken out periodically for weighing. Before weighing the specimens, excess surface water is wiped off and specimens are left in air for about 5 minutes. After the specimens reached a stage of moisture absorption saturation as defined by the ASTM standard, compression test is conducted on the specimens.

4.3.3.2 Ultrasonic Imaging
Ultrasonic imaging (UI) of syntactic foam specimens is carried out using Physical Acoustic Corporation’s water immersion type system UltraPAC™ with Ultrawin™ software. Wet and dry syntactic foam specimens are scanned using UI technique. High damping and scattering of ultrasonic waves is observed due to the presence of substantial amount of air in the structure of

Table 5 Density and void content of fabricated syntactic foam slabs.

<table>
<thead>
<tr>
<th>Cenosphere Type</th>
<th>Corresponding Syntactic foam type</th>
<th>Syntactic Foam Density (kg/m$^3$)</th>
<th>Void Volume Fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>S22</td>
<td>SF22</td>
<td>493</td>
<td>6.1</td>
</tr>
<tr>
<td>S32</td>
<td>SF32</td>
<td>545</td>
<td>9.1</td>
</tr>
<tr>
<td>S38</td>
<td>SF38</td>
<td>569</td>
<td>10.0</td>
</tr>
<tr>
<td>K37</td>
<td>SF37</td>
<td>575</td>
<td>10.0</td>
</tr>
<tr>
<td>K46</td>
<td>SF46</td>
<td>650</td>
<td>5.9</td>
</tr>
</tbody>
</table>
syntactic foam in the form of 65% of hollow spheres by volume [48]. Such a high volume fraction of air in the material makes it necessary to use lower frequency for obtaining meaningful scans. Hence, 2.25 MHz frequency transducers having spherical focus are used in pulse-echo mode to scan the specimens. Ultrasonic transducers have diameter and focal length of 0.5 and 1.5 inch respectively. All the UI scans are carried out at a sampling rate of 15.625 MHz.

4.3.3.3 Mechanical Tests
For the compression testing MTS 810 Material Test System with computer controlled data acquisition system is used. Teststar II software is used for data acquisition. Load-displacement data obtained from the machine is used for the calculation of modulus and compressive strength. Stainless steel platens are fixed in the hydraulic grips of the testing machine to carry out the compression tests. Appropriate MTS machine fixtures are used for all types of bending tests as recommended by relevant ASTM standards. The number of specimens tested, specimen dimensions and cross head displacement velocity are reported in the respective sections of each mechanical test.

4.3.4 Hygrothermal Testing of Syntactic Foam

4.3.4.1 Moisture Absorption Study
Some comprehensive references are available [49-52], which explain the mechanism and effect of moisture absorption in epoxy resins and unidirectional fiber reinforced composites. However, results of moisture absorption in syntactic foams cannot be compared directly with that of matrix resin or unidirectional fiber reinforced composites for many reasons. In case of syntactic foams tested in this investigation, volume fraction of resin is only 0.35. The resin is present in the form of a thin film around the cenospheres, which are inert to the attack of moisture. Hence, moisture absorption would be in specific channels in the random network of resin present in the structure. Presence of air voids and some broken cenospheres in syntactic foams would lead to increase in moisture absorption in specific directions.

The usefulness of UI for syntactic foam type of materials is highlighted in some published studies [53, 54]. UI is very effective in obtaining the moisture absorption pattern within the specimen. Absorption of moisture changes the density and mechanical properties of the matrix material, which causes a change in its attenuation coefficient. It must be remembered that the specimens tested in this study are compression test specimens where surface area of the side walls is nearly half of the total surface area of the specimens. Hence, moisture intake is substantial from all surfaces. When specimens are scanned by UI, the effect of moisture absorption from the top and the bottom surface would be the same throughout the thickness. However, absorption from the side wall will lead to the generation of difference in the attenuation coefficient across the cross section of the specimen, giving the water absorption pattern. UI scan of a dry SF46 specimen is shown in Figure 17. It can be observed that the intensity changes only close to the edges of the specimen. This figure would serve as a reference for the scans of wet samples.

Moisture absorption trends for all the specimens tested in this study are shown in Figure 18. The values shown in Figure 18 are average values based on moisture absorption by four samples.
under each type of test condition. It is evident from Figure 18 that the moisture absorption at low temperature reaches equilibrium in about 500 hrs. However, it takes much longer time at 70°C to attain equilibrium. For both types of syntactic foams equilibrium is reached in about 1200 hrs at higher temperature. The experiments are continued for one additional week after the equilibrium conditions specified in the selected ASTM standard are reached. Large difference in the moisture absorption tendency is observed with the change in temperature. It is observed that for both types of syntactic foams water absorption is less than 1% at room temperature. At high temperature water absorption increased to about 10 fold for SF46 specimens and about 5 to 7 fold for SF22 specimens.

UI C-scans of a low temperature DI water immersed SF46 type specimen are shown in Figure 19 and 20. These figures show change in amplitude of the ultrasonic waves in the test specimens. Location of the specimen is marked by a box in Figure 19 for better visualization. Figure 19 shows average pattern of the water absorption throughout the thickness of the specimen. Due to the presence of moisture only to a certain depth in the specimen, the wave pattern observed in A-scan shows a front surface reflection peak and then a second peak at the end of the moisture absorbed layer. Depth of moisture absorption is not the same at every point in the specimen; hence, the location of second peak also changes continuously while scanning the specimen. According to the wave pattern observed in A-scan the location of data acquisition is adjusted in such a way that second peak lies within it to get a profile of moisture absorption shown in Figure 20. UI C-scan for a high temperature DI water immersed SF22 specimen is shown in Figure 21. Comparison of Figures 17, 19-21 shows considerable difference in the extent of moisture penetration. Dry region in high temperature immersed specimens is smaller compared to the low temperature immersed specimens as observed in these figures. Similar difference is observed in the salt water immersed specimens also.
Figure 18 Result of moisture absorption study on syntactic foam on room temperature and 70°C (a) for the entire experiment time span and (b) initial part of the graph enlarged for better visualization.
Figure 19 Ultrasonic imaging scan of a low temperature DI water immersed SF46 specimen.

Figure 20 Ultrasonic imaging C-scan profile of a DI water immersed SF46 specimen at some depth from surface.
Some general conclusions can be drawn based on the graph shown in Figure 18. First, specimens immersed in the salt water absorbed less moisture than the same type of specimens immersed in DI water. The second, SF22 specimens absorbed more water than the SF46 specimens. The first observation is consistent with the results published by other researchers [35]. Presence of salt ions in the water interferes with the diffusion of water in the syntactic foam specimens. Ionic species of salt being considerably bigger in size have much slower diffusion rate compared to the diffusion rate of ionic species of water. Additionally, deposition of salt can take place near the pores in the material, which further reduces the diffusion rate of water and salt ions in the material.

The second observation that the SF22 syntactic foam absorbs more water than the SF46 syntactic foam needs very close attention as both types of syntactic foams have same matrix resin and particle-matrix volume fraction. The only difference between these foams is the cenosphere wall thickness. However, borosilicate glass particles are inert to moisture attack, hence, such a large difference in the moisture absorption is not expected. It should be noted that the slope of the moisture absorption curves for SF22 specimens is much higher than that for SF46 specimens. This means that higher number of pores are either open to the surface or present near the surface. Diffused water accumulates in these pores in the initial stage of immersion, leading to steeper slope of the curves for SF22 samples in the beginning of the study.

At higher temperature the difference in the water absorption characteristics of two types of foams can be related to an additional factor, which is the strength of cenospheres. According to the compressive strength values for the cenospheres given in Table 4, K46 types of cenospheres are much stronger than S22 type of cenospheres. Due to the hygrothermal strain gradient in the matrix material some of the thin walled cenospheres can fracture exposing the hollow space existing within them. This makes additional space available for the water to accumulate in the specimen. If the assumption of cenosphere fracture is correct, then it should reflect as sharp
decrease in the compressive strength of the specimens immersed in high temperature bath compared to specimens immersed in the low temperature bath. Compression test results of these specimens will be helpful in determining the validity of this argument.

4.3.4.2 Compressive Strength

4.3.4.2.1 Test Parameters
Constant cross head displacement rate of 0.5 mm/min is used for the compression testing of the specimens as per the recommendation of the standard ASTM 365 – 94 [55]. Three samples of each type of syntactic foams are tested for compressive properties. Test specimens have dimensions of 25×25×12.5 mm³ for length, width and height respectively.

4.3.4.2.2 Results and Discussion
Results of compression test of dry specimens of SF22 syntactic foam (Figure 22) are compared to the results of specimens that are subjected to hygrothermal studies (Figures 23-26). Similarly, stress-strain curves for SF46 syntactic foam are shown in Figure 27 and Figures 28-31 for dry and wet specimens, respectively.

From the comparison of trends observed in stress-strain curves it can be noticed that the shape of the curves is the same for dry and moisture absorbed samples for both types of syntactic foams. These curves show trends that are similar to the characteristics of elastic-perfectly plastic materials. After the elastic region, stress becomes nearly constant for considerable strain. In some cases slight decrease in stress may be observed due to origination of crack near the corners of the specimens. This type of behavior is more common for SF46 syntactic foam, which has much higher rigidity compared to the SF22 syntactic foam. However, after the elastic region, stress becomes nearly constant during further compression. This plateau region is referred as densification stage. Cenospheres fracture under compressive stresses and the compressing material occupies the hollow space exposed. These events lead to an overall increase in the density of the material. Hence, this stage is referred as densification stage. At the end of the densification stage stress starts increasing again. Some of the samples are compressed to strain as high as 60% to observe the trend of the curve in this region.

Figure 22 Stress Strain curves for dry SF22 type syntactic foam samples.
Figure 23 Stress-Strain curves for SF22 Syntactic foam in DI water at room temperature.

Figure 24 Stress-Strain curves for SF22 Syntactic foam in salt water at room temperature.

Figure 25 Stress-Strain curves for SF22 Syntactic foam in Deionized water at 70°C.
Figure 26 Stress-Strain curves for SF22 Syntactic foam in salt water at 70°C.

Figure 27 Stress Strain curves for dry SF46 type syntactic foam samples.

Figure 28 Stress-Strain curves for compression test of SF46 Syntactic foam in Deionized water at room temperature.
Figure 29 Stress-Strain curves for compression test of SF46 Syntactic foam in salt water at room temperature.

Figure 30 Stress-Strain curves for SF46 Syntactic foam in Deionized water at 70°C.

Figure 31 Stress-Strain curves for SF46 Syntactic foam in salt water at 70°C.
An important difference can be noted while comparing stress-strain curves of dry and hygrothermal samples. In case of dry samples, compression tests are stopped around 10-15% strain for both types of syntactic foams. At such strain level several cracks originated in the materials and propagated to substantial length to cause failure. Figures 32a and 32b show SF22 and SF46 type dry syntactic foam samples respectively, where substantial amount of fragmentation can be observed in the sidewalls of the specimens. Origination of vertical cracks is a typical fracture features in such materials, which are indicated in these figures by arrows. These cracks originated due to the brittle nature of the matrix and the effect of secondary tensile stresses in transverse direction. However, wet samples do not show this type of behavior. The wet samples are compressed uniformly to about 35% strain in SF22 syntactic foam and about 25% strain in SF46 syntactic foam. Even after such a high value of compression, no drop in stress is observed and the stress remains nearly constant in the plastic deformation region, referred as plateau region. At the end of the plateau region increasing trend is observed in the stress value of the curves and samples could be further compressed to over 40% strain. Figure 33 and 34 show wet SF22 and SF46 samples respectively, where cracking and fragmentation cannot be observed even after high degree of compression. These observations are similar for specimens immersed in DI water and salt water.

Comparison of peak compressive strength of dry and wet samples is shown in Figure 35. No significant difference is observed in the peak strength of low temperature hygrothermal samples compared to the dry samples for both types of syntactic foams. Slight increase in the compressive strength is observed for SF22 type and slight decrease is observed for SF46 specimens. The difference in compressive strength values in both cases is less than 10% compared to the dry samples. However, the compressive strength of specimens immersed in high temperature water showed much lower values compared to the dry and low temperature wet specimens. It is observed that the compressive strength of these samples has decreased by about one-third compared to the dry samples of the same type. In the moisture absorption section it was expected that if cenosphere fracture due to hygrothermal strain gradient caused by the high temperature, the compressive strength of high temperature wet specimens will show substantial decrease. Hence, the experimental observation of marked decrease in the compressive strength validates this argument.

Figure 32 Compressive fracture features of dry syntactic foam samples (a) SF22 type (b) SF46 type. Vertical cracks are marked by arrows.
Figure 33 Compressive fracture features of SF22 type syntactic foam subjected to salt water immersion at room temperature.

Figure 34 Compressive fracture features of SF46 syntactic foam subjected to DI water immersion at room temperature.
Comparison of compressive modulus of dry and various types of wet specimens is shown in Figure 36. It can be observed that the modulus values of all types of syntactic foams are affected severely due to the presence of moisture in the specimen. For both types of syntactic foams values of compressive modulus have reduced by about half in case of room temperature wet specimens and by about two-third in case of high temperature wet specimens. For SF22 specimens decrease in the compressive modulus for low temperature DI water and salt water is calculated to be 49 and 51% respectively. The decrease in the compressive modulus for high temperature specimens is observed to be 65 and 68% respectively. For SF46 syntactic foams decrease in the compressive modulus is measured to be 48, 64, 57 and 60% for low temperature DI water, low temperature salt water, high temperature DI water and high temperature salt water immersed specimens respectively.

These observations can be attributed to two factors, which are the moisture content in the specimen and possibility of material property degradation. According to Figure 18 moisture content in the high temperature specimens is much higher compared to the respective low temperature samples. Considerable decrease in modulus without any change in the compressive strength value for low temperature specimens reveals that moisture has infused in the matrix resin leading to its plasticization. Due to plasticization, syntactic foam specimens could be compressed to a very high degree of strain without generation of cracks. Strength as well as modulus has been reduced in high temperature tested specimens, which indicates towards occurrence of some additional events in the material.
Effect on Modulus

<table>
<thead>
<tr>
<th>Material and Test Type</th>
<th>SF22 Type</th>
<th>SF46 Type</th>
</tr>
</thead>
<tbody>
<tr>
<td>Dry</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Low Temp, DI Water</td>
<td></td>
<td></td>
</tr>
<tr>
<td>High Temp, DI Water</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Low Temp, Salt Water</td>
<td></td>
<td></td>
</tr>
<tr>
<td>High Temp, Salt Water</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Figure 36 Effect of temperature and moisture on the compressive modulus of syntactic foams.

One possibility is the fracture of cenospheres due to the hygrothermal study prior to the compression tests. Due to the fracture of cenospheres, not only the strength and modulus of the syntactic foam would go down, but further moisture absorption would also increase. Considerable increase in moisture absorption during hygrothermal study verifies and validates this possibility. In addition to this, it must be remembered that the volume fraction of resin in the structure is only 0.35. Hence, only a very thin film of resin would be there around cenospheres. Hygrothermal strains may cause rupture of the resin film in some places, which would reduce the strength of the syntactic foam specimens. Additional factor contributing to the increase in the matrix strains and contributing to the failure is the expansion of entrapped air voids due to the increase in temperature. A combined effect of these three factors results in the reduction of both strength and modulus of high temperature hygrothermal syntactic foam specimens.

4.3.4.2.3 Conclusions

Based on this study the conclusions that are drawn are summarized below,

1. Moisture absorption of SF22 and SF46 syntactic foams is below 1% at room temperature. At 70°C SF22 syntactic foam absorbed 6.7 and 2.5% moisture in DI and salt water, respectively. It is observed that SF46 syntactic foam absorbed 3.9 and 1.9% water at 70°C.

2. Considerable decrease in modulus is observed in wet samples compared to the dry samples of same type of syntactic foam. For SF22 specimens this decrease is 49, 51, 65 and 68% respectively for low temperature DI water, low temperature salt water, high temperature DI water and high temperature salt water. For SF46 syntactic foams decrease in modulus is measured to be 48, 64, 57 and 60% for low temperature DI water, low temperature salt water, high temperature DI water and high temperature salt water respectively.
3. No significant difference is observed in the peak compressive strength of low temperature specimens compared to the dry specimens. However, high temperature specimens showed 36, 33, 34 and 31% decrease for SF22 DI water, SF22 salt water, SF46 DI water and SF46 salt water immersed samples compared to the corresponding dry samples.
4. Cenosphere fracture takes place due to hygrothermal stresses contributing to high temperature moisture absorption and is verified by substantially reduced compressive strength of the material.

4.3.5 Edgewise Compression Tests of Syntactic Foams – Small Specimens

4.3.5.1 Test Parameters
Test standard ASTM D 695 – 96 [56] is selected for the compression testing of syntactic foams. This standard is for unreinforced and reinforced rigid plastic type of materials. The compression test specimen cross section is 12.7×12.7 mm² and height is 25.4 mm. Rate of cross head movement is maintained at 1.3 mm/min. At least five specimens of each type of syntactic foam are tested and average compressive strength and modulus values are presented and analyzed here.

4.3.5.2 Results and Discussion
Stress-strain curves for all types of specimens tested in this study are shown in Figures 37a-e. These figures show that the scatter in the results is very small for various samples tested for each type of syntactic foam. Values of modulus and peak strength calculated from these graphs are given in Table 6. It is evident from the results that the modulus and the peak strength of the syntactic foam increase with an increase in cenosphere wall thickness. Modulus and peak strength have almost doubled for 6.4% increase in cenosphere η. The average particle diameter of all types of cenospheres is within a very close range, hence, large difference in the mechanical property values can be directly attributed to the cenosphere η. These trends indicate that there is a strong dependence of mechanical properties of syntactic foams on the cenosphere wall thickness.

The shape of stress-strain curves shown in Figures 37a-e is quite similar to that observed by some other researchers [30]. A drop in the stress value is observed at the end of the elastic region. Thereafter the stress becomes nearly constant or shows decreasing trend but with very small

<table>
<thead>
<tr>
<th>Syntactic Foam Type</th>
<th>Modulus (MPa)</th>
<th>Peak Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF22</td>
<td>1219.6</td>
<td>31.8</td>
</tr>
<tr>
<td>SF32</td>
<td>1531.1</td>
<td>39.4</td>
</tr>
<tr>
<td>SF37</td>
<td>1844.5</td>
<td>54.0</td>
</tr>
<tr>
<td>SF38</td>
<td>1964.5</td>
<td>56.3</td>
</tr>
<tr>
<td>SF46</td>
<td>2220.5</td>
<td>64.4</td>
</tr>
</tbody>
</table>
Figure 37 Stress-strain curves for the edgewise compression testing of syntactic foams of type (a) SF22 (b) SF32 (c) SF37 (d) SF38 and (e) SF46.
slope. This region is known as plateau region or densification stage. Damage tolerant characteristics of syntactic foams are attributed to this plateau region. Fracture mechanism in this region is the crushing of cenospheres. Hollow space existing within the cenospheres is exposed due to their crushing. This space is occupied by the cenosphere debris and matrix material while getting compressed. These events lead to compaction or densification of the material.

Specimen fracture behavior is closely monitored during the process of compression. The compression test setup is shown in Figure 38. Fracture sequence of syntactic foam specimens is shown in the sequence of photographs in Figure 39a-c, which is captured for SF38 specimen. It is observed that the initial fracture takes place under the effect of shear stress. This is evident due to the generation of shear cracks from the corners of the specimen. In the Figure 39a, these cracks can be observed in the bottom of the specimen and are labeled as “A”. Shear cracks grow to a substantial length and form wedge shaped fragment at the bottom or top of the specimen. The wedge shaped fragment can be clearly noticed in the bottom of the specimen in Figure 39a. The tip of the wedge shaped fragments acts as stress riser in the specimen. Localized stress concentration at the wedge tip along with secondary tensile stresses acting in the lateral direction leads to the formation of a vertical crack. This crack causes vertical splitting of the specimen. In Figure 39a the onset of a vertical crack, labeled as “B”, can be noticed at the wedge tip while the shear cracks have propagated to their full length. In Figure 39b the vertical crack grows further and finally in Figure 39c it grows to the full height of the specimen leading to complete splitting and failure.

Specimens shown in Figures 40a-d are of SF22, SF32, SF37 and SF48 type of syntactic foams respectively. In all these figures fracture features of specimens are quite similar to the features observed in Figure 39. However, it is noticed that as the cenosphere \( \eta \) decreases, vertical splitting becomes more and more prominent. Stiffness of the material is a factor affecting the formation of vertical crack. Higher stiffness values lead to higher level of secondary tensile stresses in the material and cause early formation of the vertical crack. In SF46 specimens the vertical splitting became so prominent that the test is stopped at around 5 to 6% strain due to sudden drop in stress around this value. For SF37 and SF38 samples the plateau region is
observed till about 6-8% strain followed by decreasing trend. Whereas, for the SF22 and SF32 type of samples the plateau region continued to over 8% strain without drop in stress. However, for most of the specimens of all types of syntactic foams the peak strength is observed in the range of 2.8-3.5% strain. Within this range of strain values no general pattern for strain at the peak stress could be found and related to the cenosphere $\eta$ for different types of syntactic foams. This fact strongly indicates that the critical strain at which peak strength is observed does not depend on the type of cenospheres and can primarily be attributed to properties of the matrix material.

Scanning electron microscopy is performed to observe the fracture features of SF46 specimens. Fracture features observed on the surface of shear cracks are shown in Figure 41. Large amount of debris can be observed in this micrograph. Some unbroken cenospheres can also be observed.
Figure 40 Failure of (a) SF22, (b) SF32, (c) SF37 and (d) SF46 type of syntactic foams.

Figure 41 Features of fracture on shear cracks on SF46 syntactic foam specimen.
under the debris in this figure. From this figure it appears that the damage is limited to the crack surface only. Fracture features observed on the vertical crack surface are shown in Figure 42. In this micrograph debris of broken cenospheres is not present. Tensile failure leading to generation of this surface has occurred primarily due to the fracture of the matrix material. Close observation of this surface shown in Figure 43 reveals features such as debonding between matrix and cenospheres and plastic deformation and fracture marks on the matrix resin surface. Such plastic deformation marks are not observed on the compressive failure surface. Hence, it is concluded that under compressive stresses cenospheres fracture and under tensile stresses the matrix deformation and fracture are the primary failure mechanisms. Similar features are observed in specimens of other types of syntactic foams also.

![Figure 42](image1.jpg)

Figure 42 Tensile fracture features as observed on the vertical crack surface.

![Figure 43](image2.jpg)

Figure 43 Hi-magnification micrograph taken on tensile crack surface. Debonding between cenosphere and matrix fracture pattern can be observed.


4.3.5.3 Conclusions

Effect of wall thickness of cenospheres on the compressive properties of syntactic foams is investigated in this section. Experimental results of compression testing of syntactic foams are summarized as follows:

- Trends of stress-strain curves are similar for all types of syntactic foams.
- Compressive strength and modulus increase with decrease in cenosphere $\eta$.
- For a decrease in cenospheres $\eta$ from 0.922 to 0.863, the modulus increases from 1219.6 to 2220.5 MPa and the peak strength is increased from 31.8 to 64.4 MPa.
- The strain at peak stress in stress-strain graphs is independent of cenosphere wall thickness.
- Fracture features for all types of syntactic foams are similar. It is observed that the initial fracture mode is shear cracking followed by vertical splitting of specimens in the direction of applied load under secondary tensile stresses.

4.3.6 Edgewise Compression Tests of Syntactic Foams - Bigger Specimens

4.3.6.1 Test Parameters

Constant crosshead velocity of 1.3 mm/min is maintained during the test in accordance to the ASTM D 695 – 96 [56] standard. Test specimens have cross section area of 25.4×12.5 mm$^2$ and 25.4 mm Height. Six specimens of each type of syntactic foam are tested.

4.3.6.2 Results and Discussion

Syntactic foams behave like rigid plastics under compressive loading conditions. Due to the presence of brittle glass cenospheres in the structure syntactic foams tend to form fine powder on crushing under compression. In these materials crack propagation is generally slow and is associated with extensive crushing of cenospheres on the compressive fracture planes.

In the compression testing of smaller syntactic foam specimens in the previous sections, it is observed that the specimen collapses soon after the crack origination. Dimensions of fragments generated due to the crack formation are not enough to support themselves. For this reason one dimension of the specimen’s cross section is chosen to be larger than the other dimension. The effect of shear stresses and shear cracks is much less on the bigger surface area sidewalls. The cracks in this case are not able to propagate to cause complete fracture of these walls by the time the fracture under the secondary tensile stresses takes place on the smaller surface area sidewalls of the specimen. This ensures that the specimens do not collapse immediately after initiation of crack and can be compressed to higher strain values. In further experimental work, sandwich composites would be fabricated using syntactic foam as core material. These sandwich composites would be tested for edgewise compressive properties. Testing syntactic foam specimens of the dimensions taken in this study would facilitate direct comparison of results.

Fracture mode of SF22 and SF46 syntactic foam specimens is shown in Figures 44a and 44b, respectively. In both figures, it can be observed that the vertical crack originated under lateral secondary tensile stresses is very prominent. Shear cracks can also be noticed near the corners of the specimens. Initial failure takes place under shear mode, followed by vertical cracking. Onset of the vertical cracking leads to a sudden drop in the stress-strain curves of the material.
However, the specimen does not collapse after the origination of vertical crack. The final specimen failure depends on the further shear cracking of the fragments generated due to the vertical cracking. Extended plateau region in the stress-strain curves denotes uniform stable crushing of the specimen. However, extent of this region is highly dependent on the crack pattern in the material, which may be affected by localized factors such as presence of voids or localized concentration of cenospheres or matrix material.

Extensive crushing of cenospheres on the shear crack planes, observed in Figures 44a and 44b, leads to the formation of powder like mass. However, fracture mode is mainly matrix failure under secondary tensile stresses along the vertical crack plane. Hence, generation of powder like mass is generally absent on such crack planes unlike compression fracture planes.

Some of the stress-strain curves for SF32 and SF46 syntactic foams are shown in Figure 45. Stress-strain curves for other types of syntactic foams also exhibit similar trends. Compressive modulus values of various samples within one type of syntactic foam are very close to each other. However, peak compressive strength shows some variation. Average peak compressive strength and compressive modulus values obtained from these curves are given in Table 7. Steady increase in peak compressive stress and compressive modulus is observed with decrease in cenosphere $\eta$. It is observed that for a decrease in cenosphere $\eta$ from 0.922 to 0.863 the modulus increased from 1160.6 MPa to 2269.5 MPa, and the peak compressive strength increased from 32.9 MPa to 64.5 MPa. For this range of $\eta$ values the wall thickness increased from 1.26 to 2.74 $\mu$m.

It must be noted that the same matrix resin system is used for the fabrication of all types of syntactic foam slabs and all types of cenospheres are made of same material. Hence, the difference in the compressive strength and modulus values can only be attributed to the cenosphere $\eta$. 

Figure 44 Specimen of (a) SF22 and (b) SF46 syntactic foam after compression test.
Figure 45 Stress-Strain curves for (a) SF32 and (b) SF46 syntactic foams.

Table 7 Compressive Modulus and strength of syntactic foam tested in edgewise orientation using bigger specimen.

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Radius Ratio (\eta)</th>
<th>Compressive Modulus (MPa)</th>
<th>Peak Compressive Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF22</td>
<td>0.922</td>
<td>1160</td>
<td>32.9</td>
</tr>
<tr>
<td>SF32</td>
<td>0.907</td>
<td>1370</td>
<td>37.5</td>
</tr>
<tr>
<td>SF37</td>
<td>0.891</td>
<td>1471</td>
<td>49.6</td>
</tr>
<tr>
<td>SF38</td>
<td>0.888</td>
<td>1940</td>
<td>50.5</td>
</tr>
<tr>
<td>SF46</td>
<td>0.863</td>
<td>2270</td>
<td>64.5</td>
</tr>
</tbody>
</table>
4.3.6.3 Conclusions
Based on the experimental study conclusions are drawn that a decrease in cenosphere $\eta$ from 0.922 to 0.863 has led to an increase in

1. Peak compressive strength from 32.9 to 64.5 MPa and modulus from 1160.6 to 2269.5 MPa.
2. Specimen fracture pattern is not found affected by change in $\eta$.

Specimen fractures mainly under secondary tensile stresses, which lead to vertical splitting. Pieces of specimen generated due to vertical cracking tend to fracture under shear mode and constitute final failure of the specimen.

4.3.7 Flatwise Compression of Syntactic Foams

4.3.7.1 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 size recommended in these standards is the main difference. ASTM D 1621 – 94 recommends minimum cross sectional area of 2580 mm$^2$ and 25.4 mm height. For close cell structured foams such as syntactic foams ASTM C 365 – 94 recommends a smaller specimen size, which is 625 mm$^2$ cross section and no specific height. Based on the consideration that the fabricated syntactic foam slabs have half inch thickness and 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 25×25×12.5 mm$^3$ for length, width and height, respectively, in accordance with ASTM C 365 – 94.

Setup of compression tests is shown in Figure 46. Constant crosshead movement rate is maintained at 0.5 mm/min as suggested in 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 carry out the calculations for modulus and compressive strength.

![Figure 46 Flatwise compression test setup.](image-url)
4.3.7.2 Results and Discussion

In earlier part of the experimental investigation another standard ASTM D 695 – 96, which is for the compression testing of rigid plastics, is followed. This standard recommends specimens having aspect ratio of 2 to be tested. 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. The polymer material system used as matrix in the syntactic foam is also tested for compressive properties as per both selected standards. Compressive modulus and compressive yield strength values of polymer material system are presented in Table 8.

Some representative stress-strain curves for the flatwise compression testing of syntactic foams are shown in Figure 47. For all five types of syntactic foams the trend of the stress-strain curves is similar. It is observed that the stress decreases by about 10-20% after reaching a peak value, that denotes the point of crack origination in the specimens. After this decrease, the stress becomes nearly constant for further deformation. 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 crushed. The plateau region for all of the syntactic foam samples, except SF46 type, extends beyond 10% strain without any further decrease in stress. The SF46 specimens show the end of the plateau region at 6-8% compression followed by negative slope in the stress-strain curve. No definite fracture point is observed in the flatwise compression of syntactic foams. The reason for this observation is described as follows. Actually cenospheres having a large distribution of outer radius and η exist in the fabricated syntactic foams. The strength of these cenospheres also varies over a wide range of values. In any part of the syntactic foam structure, if the stress value rises above the fracture strength of any cenosphere, then that cenosphere tends to fracture. If the mixing of cenospheres in the matrix resin is carried out properly, cenospheres of all different sizes and η values will be distributed randomly in the syntactic foam structure. In such a condition there will be no preferred compression or fracture plane. Hence, the specimens sustain large deformation without showing any definite fracture point.

It is observed that the strain at peak stress for all types of syntactic foams is close to 3%. In the two-component structure, one component, i.e. cenospheres, is different in different types of syntactic foams. The matrix resin is the same for all types of syntactic foams. Hence, the observation that the peak stress occurs at the same strain value for all types of syntactic foams concludes it is independent of cenosphere properties. To examine the dependence of strain at peak stress on the properties of matrix material compression tests of matrix resin (unreinforced)

<table>
<thead>
<tr>
<th>Specimen Orientation</th>
<th>Compressive Modulus (MPa)</th>
<th>Compressive Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Flatwise</td>
<td>3363</td>
<td>126</td>
</tr>
<tr>
<td>Edgewise</td>
<td>2321</td>
<td>90</td>
</tr>
</tbody>
</table>
Figure 47 Stress-Strain curves for the flatwise compression testing of (a) SF22 (b) SF32 (c) SF37 (d) SF38 and (e) SF46 syntactic foam.
are carried out. Flatwise and edgewise compression test results for the unreinforced polymer are shown in Figure 48. It can be observed that the yield strain for the matrix polymer is approximately 3.5%, which is close to the strain for the peak stress observed for syntactic foams. These observations lead to the conclusion that the elastic deformation of syntactic foams does not depend on the strength of cenospheres and is related primarily to the properties of the matrix polymer.

In the five types of cenospheres selected in the present study \( \eta \) varies from 0.863 to 0.922. Change in compressive modulus and peak compressive strength of syntactic foam with change in \( \eta \) is given in Table 9. Strong dependence of modulus on \( \eta \) is evident from the values. Increase in compressive modulus from 1548 MPa to 2640 MPa is observed with a decrease in \( \eta \) from 0.922 to 0.863. Ultimate compressive strength also shows increasing trend with decrease in \( \eta \) and changes from 30.3 MPa to 71.7 MPa within this range of \( \eta \). These results demonstrate strong influence of cenosphere \( \eta \) on the modulus and ultimate compressive strength of syntactic foam materials.

![Figure 48 Flatwise and Edgewise compression test results of unreinforced matrix polymer system.](image)

Table 9 Comparison of compressive properties of syntactic foam specimens tested in flatwise and edgewise orientations.

<table>
<thead>
<tr>
<th>Syntactic Foam Type</th>
<th>Radius Ratio</th>
<th>Compressive Modulus (MPa)</th>
<th>Peak compressive Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>( \eta )</td>
<td>Flatwise</td>
<td>Edgewise</td>
</tr>
<tr>
<td>SF22</td>
<td>0.922</td>
<td>1548</td>
<td>1220</td>
</tr>
<tr>
<td>SF32</td>
<td>0.907</td>
<td>2025</td>
<td>1531</td>
</tr>
<tr>
<td>SF37</td>
<td>0.888</td>
<td>2195</td>
<td>1845</td>
</tr>
<tr>
<td>SF38</td>
<td>0.891</td>
<td>2395</td>
<td>1965</td>
</tr>
<tr>
<td>SF46</td>
<td>0.863</td>
<td>2640</td>
<td>2221</td>
</tr>
</tbody>
</table>
4.3.7.3 Conclusions
Compression test results are related to the cenosphere Radius Ratio, $\eta$. It is concluded that the

1. Peak compressive strength increase with decrease in $\eta$.
2. Compressive modulus value increases with decrease in $\eta$.
3. Strain at peak compressive stress does not depend on $\eta$.
4. The plateau region observed in the stress-strain curves is attributed to the fracture of cenospheres during compression of syntactic foams.

4.3.8 Effect of Specimen Aspect Ratio on Syntactic Foam Compression

4.3.8.1 Discussion
Change in the compression test specimen aspect ratio reflects in the specimen fracture behavior and measured compressive properties. Specimen deformation and fracture pattern in both types of specimens is studied carefully with respect to the specimen dimensions and compared here to explain the difference in the modulus and peak compressive strength values. Stress-strain curves for syntactic foam specimens tested in accordance to ASTM D 695 – 96 standard (edgewise compression) are shown in Figure 37. A comparison of these curves with corresponding flatwise compression curves in Figure 47 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% for most of the specimens, which is significantly higher than the drop of about 10-20% observed for the flatwise compression. It is also observed that most specimens do not show uniform crushing after reaching the ultimate stress, which leads to absence of the plateau region in the stress-strain curves. The tests had to be stopped at 6-8% strain due to sudden drop in the stress value unlike the flatwise compression tests, which showed plateau region till 10-15% compression. These variations can be related to the crack origination pattern in the specimens.

Edgewise and flatwise compression test specimens of syntactic foams are shown in Figures 49a and 49b respectively. Figures 50 and 51 show schematics of the fracture mechanism of the specimens for edgewise and flatwise orientations respectively. Fracture pattern of these specimens is compared to understand the differences observed in the trends of the stress-strain curves. In both cases shear cracks originate from the specimen corners. These 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 Figure 50a. As the strain increases in the specimen, due to the stress concentration and secondary tensile stresses acting normal to the applied load, the specimen shows barreling effect as indicated in Figure 50b. This leads to vertical splitting of the specimen as observed in Figure 50a. Location and extent of the shear and tensile cracks is very critical for the final failure in such orientation. This is the reason that considerable variation is seen in the peak stress values and trends of the stress-strain curves after ultimate stress is reached. In flatwise tests also the shear cracks originate, however, they do not generate any stress riser in the specimens as evident from the specimen fracture mechanism shown in Figures 51a and 51b. Wedge shaped fragments from in this orientation also. However,
Figure 49 Syntactic foam specimens tested under (a) flatwise and (b) edgewise compression.

Figure 50 Fracture mechanism of syntactic foam tested under edgewise orientation.

Figure 51 Fracture mechanism of syntactic foam tested under flatwise orientation.
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. It must be noted that the modulus depends on the elastic properties of the material. 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 of cenospheres and matrix resin, which causes comparable results in edgewise and flatwise orientations.

Compression tests of unreinforced polymer, which is used as matrix in syntactic foams, are helpful in understanding the effect of lateral expansion and 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 Figure 52 where specimen marked as “A” is an undeformed specimen and the specimen marked as “B” is subjected to about 25% 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 Figure 53.

![Figure 52 Specimens of matrix polymer subjected to the edgewise compression.](image)

![Figure 53 Specimens of matrix polymer subjected to the flatwise compression.](image)
In this figure the specimen marked “A” is undeformed and the specimen marked as “B” is subjected to about 13% compression. The effect of barreling in this specimen is not as prominent as observed in edgewise compressed specimen shown in Figure 52. The syntactic foam specimens also exhibit the similar behavior. This difference reflects in the modulus values of the syntactic foams obtained from the tests.

4.3.8.2 Conclusions
The compression tests are conducted in flatwise and edgewise specimen orientations. Comparison of results leads to the conclusion that the peak compressive strength and compressive modulus of syntactic foams are dependent on specimen aspect ratio and cenosphere \( \eta \). 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. Ultimate compressive strength values measured in edgewise compression show strong dependence on the crack origination pattern.
3. Strain at ultimate 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.

4.3.9 Edgewise Compression Testing of Syntactic Foam Core Sandwich Composites

4.3.9.1 Test Parameters
Compression tests of sandwich composites are conducted in edgewise orientation in accordance to the ASTM C 364-94 standard. A fixture for MTS machine, shown in Figure 54, is designed and fabricated for the tests as suggested by the ASTM standard. This fixture supports the specimen near the loaded ends to prevent early buckling failure.

Figure 54 MTS machine fixture designed and fabricated for compression testing of sandwich composites.
Compression test specimens have dimensions of 50×14.5×50 mm³ as length, width and height respectively. The crosshead movement rate is maintained at 0.5 mm/min throughout the tests in accordance with the ASTM standard. Six specimens of each type of sandwich composites are tested in this study. Load-displacement data is obtained from the test machine and used to develop stress-strain graphs.

4.3.9.2 Results and Discussion

Stress-strain curves for a few specimens of five types of syntactic foam core sandwich composites subjected to the compression test in this study are shown in Figure 55. General features of these curves can be noted in Figure 56, which is a stress-strain curve for SFS38 specimen. The elastic region can be divided into three parts in most of these curves, except for S22 Sandwich composites, as indicated in Figure 56. Slope of these three regions is calculated and presented in Table 10 as Modulus I, II and III respectively. Curves for the SFS22 specimens showed only two stages, hence, Modulus II value for this set is not calculated. Occurrence of three stage elastic region is explained based on the observed specimen deformation behavior. It is also observed that the ultimate compressive strength of these specimens is observed in the range of 3-4% strain. The specimens could be compressed to about 8% strain before complete fracture except SFS46 specimens, which showed complete fracture around 5% strain.

It is observed during the compression of sandwich composites that skins are the first to fracture. The fracture direction is usually normal to the applied load. Core has much higher fracture strain compared to skins, hence, skins tend to fracture first. One of the SFS22 specimens is shown in Figure 57a and b to show skin fracture. Figure 57a shows onset of skin fracture and Figure 57b shows final form of the fracture. Fracture of skins is caused by the combined effect of true compression and shear stress component of the applied load. Secondary tensile forces in lateral direction that generate barreling in the specimen cause skins to bulge outwards. The strain in lateral direction generates shear component in the specimen. The ends of the specimens are clamped in the compression fixture, which further enhances the barreling effect and shear component. Modulus I values, presented in Table 10, are measured before any of the skins fracture and represents elastic modulus of the whole specimen. This value is more than modulus II and III values. It is observed that both skins do not fracture at the same time. This leads to limited stress relief in the specimen. As one of the skins has already fractured and only the other one is bearing the load at this stage, the modulus value recorded is lower than the first stage. High aspect ratio and constrained ends make the barreling effects more prominent which reflects in the form of lower modulus values. At the end of the second stage, the second skin also fractures. From this point onwards the entire load is taken up only by the foam core. Relative strengths of skin and core determine the number of stages observed in the stress-strain curve. If the compressive strength of the core is lower than that of the skin, the core tends to fracture before the skin fractures. In such a case, only two stages would be visible in the curve. It must be noted that fracture in the core is mainly under the secondary tensile forces and shear component of compressive stresses. Evidence of this is drawn from the fact that the crack in syntactic foam core is observed to originate in the direction of compressive stress as shown in Figure 58. The specimen shown in Figure 58 is SFS32 sandwich composite.
Figure 55 Stress strain curves obtained for compression testing of (a) SFS22 (b) SFS32 (c) SFS37 (d) SFS38 and (e) SFS46 sandwich composites.
Figure 56 A typical stress-Strain curve presented to show the general characteristics of such curves for sandwich composites.

Table 10 Compressive strength and modulus of syntactic foam core sandwich composites.

<table>
<thead>
<tr>
<th>Sandwich Type</th>
<th>Sandwich Composite (MPa)</th>
<th>Peak Compressive Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Modulus I</td>
<td>Modulus II</td>
</tr>
<tr>
<td>SFS22</td>
<td>1595.0</td>
<td>---</td>
</tr>
<tr>
<td>SFS32</td>
<td>2064.0</td>
<td>1719.0</td>
</tr>
<tr>
<td>SFS37</td>
<td>2198.2</td>
<td>2073.0</td>
</tr>
<tr>
<td>SFS38</td>
<td>2120.2</td>
<td>1879.3</td>
</tr>
<tr>
<td>SFS46</td>
<td>2585.1</td>
<td>2375.3</td>
</tr>
</tbody>
</table>

Figure 57 Fracture of SFS22 sandwich composite (a) onset of skin cracking (b) final fracture features.
Effect of $\eta$ of cenospheres used in fabrication of syntactic foam on the edgewise compressive properties of sandwich composites is shown in Figure 59. Modulus I, II and III values are plotted in this graph. Compressive modulus values of syntactic foam material obtained from separate set of experiments are also shown in this graph. The testing for syntactic foam specimens is performed in accordance with the standard ASTM D 695 – 96. As a general observation, decrease in compressive modulus is observed with an increase in cenosphere Radius Ratio parameter. A comparison of modulus values reveals that the modulus III values calculated from the stress-strain curves for sandwich composite are lower than the corresponding syntactic foam modulus. In the earlier part of the discussion, Modulus III values are assigned to the syntactic foam core material. This discrepancy can be associated with the higher aspect ratio of the sandwich specimens.

Figure 59 Compressive modulus values for syntactic foam and their sandwich composites.
Peak compressive strength values of sandwich composites are presented in Table 10. The ultimate compressive strength of SFS38 sandwich composite is more than that of SFS37 sandwich composite. However, the difference in the strength values is only about 4%. Such a small difference is not sufficient to draw any conclusion about SFS37 foam being stronger than SFS38 syntactic foam for two reasons. First, the reported strength values are average for six specimens of each type. Difference in the strength values of these six samples of each type of sandwich is more than 4%. Second, about 10% voids by volume are presented in these foam core slabs. Difference in the distribution of voids in various specimens can also lead to such a small difference in strength values. The peak strength values for sandwich composites are compared with the ultimate compressive strength of syntactic foam material presented in Table 7. It is observed that the peak compressive strength values of sandwich composites are 1.2 – 6.5% higher than the corresponding values for corresponding syntactic foam material. It is observed that both the skins are thin and fracture before the fracture of syntactic foam core and during later stages of compression foam core is the load bearing component in the specimen. Hence, the ultimate compressive strength of sandwich composite is comparable to the core compressive strength.

Hence, conversion of syntactic foam into sandwich composite by attaching thin glass fabric skins leads to significant increase in the modulus of the structure. If the skin thickness is sufficient, then skins would not fracture before the fracture of core material and would lead to an increase in ultimate compressive strength also.

4.3.9.3 Conclusions
From the edgewise compression tests of syntactic foam core sandwich composites following conclusions are drawn.

1. Compressive modulus of syntactic foam core sandwich composites increases with decrease in cenospheres $\eta$.
2. Stress-strain curves for syntactic foam core sandwich composites are divided in three parts and modulus values are calculated for each of these parts. Due to the fracture of both skins during compression, third stage corresponds to the compression of foam core material only. Modulus of this stage is found to be same as the modulus of syntactic foam material tested separately.

It is observed that skins fail first under compressive loading conditions and the shear component of the applied compressive stress. Syntactic foam core fractures under the effect of secondary compressive stresses in the lateral direction.

4.3.10 Flexure Test of Sandwich Composites

4.3.10.1 Flexural Test Parameters
ASTM C 393 – 94 [57] standard is followed to characterize the flexural properties of the sandwich composites. In accordance with this standard three-point bending, four-point bending and short beam shear strength (SBSS) tests are conducted. Specimen dimensions for all types of tests are given in Table 11. The thickness of specimens is equal to the thickness of the fabricated
Table 11 Test parameters for the flexural testing of sandwich composite specimens.

<table>
<thead>
<tr>
<th>Type of Bending Test</th>
<th>Width (mm)</th>
<th>Span Length (mm)</th>
<th>Crosshead Displacement Rate (mm/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Three-Point</td>
<td>25.4</td>
<td>175</td>
<td>2</td>
</tr>
<tr>
<td>Four-Point</td>
<td>25.4</td>
<td>175</td>
<td>2</td>
</tr>
<tr>
<td>SBSS</td>
<td>25.4</td>
<td>55</td>
<td>1</td>
</tr>
</tbody>
</table>

sandwich composites. Four specimens of each type of sandwich composite are tested in each type of the three bending test. Three and four-point bending setups are shown in Figures 60a and 60b respectively. SBSS test setup is similar to the three-point bend test except the use of different specimen span length.

4.3.10.2 Results and Discussion

Bending test presents a case where stress varies across the thickness, shown in Figure 61 by the plane “CNT”, of the specimen in three-point bend test. Stress changes from compression at the point where the loading anvil touches the specimen, marked as point “C”, to tension on the

![Figure 60](image)

Figure 60 Flexural test setup for (a) Three-point and (b) four point bend tests on MTS test system. In SBSS test the loading scheme is similar to the three-point bend test.

![Figure 61](image)

Figure 61 Schematic of sandwich composite bending.
opposite surface of the specimen, marked as point “T”. In addition, shear stresses act along the length of the specimen. The core or skins can fracture under these three types of stresses depending upon their properties under such stresses. Additionally, the interface between the skins and the core can also fracture under shear stresses. Hence, crack origination locations and propagation directions will help in determining the types of stresses causing failure.

The load-displacement curves for each kind of syntactic foam core sandwich composites for three and four point bending and SBSS tests are shown in Figures 62, 63 and 64, respectively. Some of the general observations from these curves are listed here and will be explained in the following sections.

1. The load decreases sharply after the end of the elastic region due to failure initiation in sandwich composites.
2. Some of the samples show complete fracture, whereas some others show a plateau region after this sharp decrease in the load.
3. Variation in displacement value at which peak load is observed for various types of syntactic foams is considerable.
4. The failure originates on the tensile side.

Within the elastic region of the load-displacement curves, where no damage is induced, the responses of specimens to the applied loads are quite similar. This is visible in the form of nearly constant slope in the elastic region of the load-displacement curves for different types of syntactic foams, as shown in Figures 62 to 64. It is observed that the failure starts in the form of crack origination on the tensile side of the specimen as displacement increases. This crack tends to grow towards the compression side of the specimen as shown in Figure 65. On further loading, the skin of the sandwich composite that is on the tensile side tends to fracture, causing the final failure of the specimen. Localized fracture of cenospheres takes place at the location of maximum compressive stress when the specimen is being loaded. However, it is not significant enough to lead to the final failure of the specimen. It is observed here that the entire specimen fractures at the instant of skin fracture. This happens because the load transferred to the core due to the skin fracture is more than the tensile strength of the core.

Appearance of plateau region in the load-displacement curves is based on the crack length in the syntactic foam core before final failure. During the loading process, deformation also takes place in the compression side of the specimen. Crushing of foam in the compression side while the crack propagates actually causes the plateau region in the load-displacement curves. Plateau regions were observed consistently in the compression test experiments of the syntactic foam material. It is observed here that the load-displacement curves show longer plateau regions for specimens showing longer cracks. Three specimens subjected to the three-point bend test are presented in Figure 66. Considerable difference can be observed in the crack lengths of the three specimens shown in this figure. The crack length of the specimen marked as “A” in Figure 66 is considerably larger than the specimen marked as “C”. Therefore, much longer plateau region is observed in the load-displacement curve of the specimen “A” compared to the specimen “C”.

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Figure 62 Load-displacement curves for three-point bend test of (a) SFS22 (b) SFS32 (c) SFS37 (d) SFS38 (e) SFS46 sandwich composites.
Figure 63 Load-displacement curves for four-point bend test of (a) SFS22 (b) SFS32 (c) SFS37 (d) SFS38 (e) SFS46 sandwich composites.
Figure 64 Load-displacement curves for short beam shear test of (a) SFS22 (b) SFS32 (c) SFS37 (d) SFS38 (e) SFS46 sandwich composites.
Figure 65 (a) Fracture origination in tensile side of sandwich (b) a closer look at the crack.

Figure 66 Three-point bend test specimens. Difference in crack lengths should be noticed.
Two of the specimens subjected to the four-point bend test are shown in Figure 67. This figure also demonstrates considerable crack length variation. It must be noticed that in case of four-point bend tests two cracks are observed in each specimens, each under a loading anvil. Factors such as localized interfacial strength between skin and core, presence of voids or excess concentration of resin or cenospheres near the fracture zone cause variations in the crack lengths. The same factors causing localized stress concentration zones lead to variation in displacement values at peak load in different specimens.

Core Shear Stress ($\tau$) and Skin Bending Stress ($\sigma$) values are calculated for each type of sandwich composite based on experimental data. Equation 45 is used for the calculation of $\tau$ for all types of bend tests. For the calculation of $\sigma$, Equation 46 is used for three-point bend and SBSS tests and Equation 47 for four-point bend test. The flexural test results are presented in Table 12.

![Figure 67 Four-point bend test sandwich composite specimens. Two cracks in each specimen can be noticed.](image)

<table>
<thead>
<tr>
<th>Sandwich Composite Type</th>
<th>Three-point bending ($\tau$) (MPa)</th>
<th>Three-point bending ($\sigma$) (MPa)</th>
<th>Four-point bending ($\tau$) (MPa)</th>
<th>Four-point bending ($\sigma$) (MPa)</th>
<th>Short Beam Shear Strength ($\tau$) (MPa)</th>
<th>Short Beam Shear Strength ($\sigma$) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SFS22</td>
<td>1.77</td>
<td>172.2</td>
<td>3.19</td>
<td>155.2</td>
<td>6.07</td>
<td>185.3</td>
</tr>
<tr>
<td>SFS32</td>
<td>1.68</td>
<td>163.8</td>
<td>3.13</td>
<td>152.1</td>
<td>5.04</td>
<td>154.0</td>
</tr>
<tr>
<td>SFS37</td>
<td>1.70</td>
<td>165.0</td>
<td>3.05</td>
<td>148.4</td>
<td>4.90</td>
<td>149.8</td>
</tr>
<tr>
<td>SFS38</td>
<td>1.74</td>
<td>169.4</td>
<td>3.07</td>
<td>149.6</td>
<td>4.87</td>
<td>148.8</td>
</tr>
<tr>
<td>SFS46</td>
<td>1.71</td>
<td>166.0</td>
<td>2.96</td>
<td>144.0</td>
<td>4.01</td>
<td>122.7</td>
</tr>
</tbody>
</table>
\[
\tau = \frac{P}{(d + c)b} \tag{45}
\]
\[
\sigma = \frac{PL}{2t(d + c)b} \tag{46}
\]
\[
\sigma = \frac{PL}{4t(d + c)b} \tag{47}
\]

Here \( P \) is the ultimate load, \( L \) is the span length, \( b \) is the sandwich width and \( d \), \( c \) and \( t \) represent the thickness of sandwich, core and skin respectively.

Careful examination of \( \tau \) and \( \sigma \) values in Table 12 leads to two general observations:

1. No general trend can be observed in the values for the three and four point bending tests in relation to \( \eta \).
2. Decrease in \( \tau \) and \( \sigma \) is noted with decrease in \( \eta \) for SBSS test.

From the first observation it is clear that \( \eta \) does not affect the bending properties of syntactic foams in three and four point bend tests. In the specimen failure pattern it is observed that the final failure is due to the fracture of sandwich skin on tensile side. Hence, these values actually reflect the fracture strength of the skins.

However, results of the SBSS test show a definite decreasing trend in \( \tau \) and \( \sigma \) values as \( \eta \) decreases. This is due to the pronounced effects of shear stresses in the foam core. It can be observed in Table 12 that \( \tau \) for SBSS test is about three times and 1.5 times more than for three-point and four point bend tests respectively. This is due to the smaller specimen span lengths. The high value of shear stress causes deformation and fracture of the syntactic foam core in shear mode. Two specimens subjected to the SBSS tests are presented in Figure 68.

![Figure 68 SBSS specimens. Fracture along the central neutral axis should be noticed. Core cracking in tensile is also visible.](image)
In addition to the tensile cracks (along the line “CTN” shown in Figure 61) in syntactic foam core, shear fracture along the neutral axis (the line “ANB” shown in Figure 61) can also be observed. The total crack lengths in these specimens are considerably larger than three and four point bend tests. In the load-displacement graphs of the SBSS tests most of the specimens show large plateau region after initial failure because of such large crack lengths.

In SBSS tests, strength of cenospheres plays an important role. Stress relieving due to the fracture of cenospheres is an important phenomenon in deciding the final strength of the sandwich material. With a decrease in $\eta$, cenosphere wall thickness increases and cenospheres become stronger. Lower strength cenospheres, such as SF22, tend to fracture under the applied stresses, relieving the stresses in the sandwich structure. Higher strength cenospheres, such as S38 or K46 type, do not fracture at lower levels of stress leading to greater stress concentration in the material around them. Hence, the final fracture takes place at lower stress levels for such specimens. The evidence for stress concentration effect can be obtained from the load-displacement curves in the form of lower fracture displacement for SFS46 sandwich composites compared to SFS22 type. The load-displacement curves shown in Figure 64 confirm that the fracture displacement for the SFS22 sandwich composites is around 1.5 mm whereas for SFS46 type specimens is less than 1 mm. It must be noted that the cenosphere volume fraction in all types of syntactic foam materials is the same. Hence, the change in the properties of the syntactic foam can be related to $\eta$, the only parameter that is changing.

4.3.10.3 Conclusions

The following conclusions are drawn based on the flexural tests of five types of syntactic foam core sandwich composites in this study.

1. Core shear stress ($\tau$) and skin bending stress ($\sigma$) in three-point and four-point bend tests are not affected by cenosphere radius ratio ($\eta$). This is because specimens fracture on the tensile side and the tensile properties of the matrix resin used in syntactic foam core dominate mechanical properties of such materials. Such a behavior is observed because cenospheres do not fracture under tensile loading conditions and fracture is limited to the matrix resin. Hence, while designing the syntactic foam type of materials, optimum volume fractions of constituting materials can be chosen to obtain the required tensile or bending properties, and then foam density can be decided upon by choosing cenospheres of appropriate $\eta$.
2. In SBSS tests, $\tau$ and $\sigma$ show decrease with decrease in $\eta$ due to prominent shear stresses in the specimen due to the smaller specimen aspect ratio. Considerably long shear cracks are observed along the specimen neutral axis in the SBSS tests. Such a long crack growth is reflected in the load-displacement curves as a long plateau region.
3. Fracture originated in the tensile side of the syntactic foam core and it progressed towards the compressive side in all types of sandwich composites.
5 SUMMARY

Analytical and FEM work is performed to develop better understanding of the deformation and fracture mechanism of syntactic foams. Stress concentration factor approach is adopted in the analytical modeling of syntactic foams. A clear dividing line is established about the fracture characteristics of cenospheres depending on their Radius Ratio, $\eta$. The critical value of $\eta$ termed as $\eta_{cr}$ is calculated to be 0.71. It is established that in a composite the fracture of cenospheres having $\eta$ less than $\eta_{cr}$ causes stress state similar to that caused by solid particle fracture. Stress states are found to be considerably different for the fracture of cenospheres having radius ratio value more than $\eta_{cr}$. To make the direct comparison of experiment results meaningful, all types of selected cenospheres had radius ratio value more than the $\eta_{cr}$. Calculation of fracture strain using FEM contact analysis and comparison of FEM results with experimental results show that the interfacial strength between matrix and cenospheres can be assumed as friction coefficient 0.3. Extensive experimentation is carried out to gather sufficient information to draw conclusions about the response of syntactic foams and their sandwich composites under various loading and environmental conditions. Syntactic foams are characterized for hygrothermal and edgewise and flatwise compressive properties, whereas sandwich composites are tested for edgewise compression, three-point bending, four-point bending and short beam shear properties. Detailed conclusions drawn from these studies are given in their respective sections. In brief, the compression test results show strong dependence of compressive strength and modulus of syntactic foams and sandwich composites on the cenosphere $\eta$. With a decrease in $\eta$ compressive strength and modulus values increase. Core shear stress and skin bending stress of sandwich composites in three and four-point bend tests were found to be independent of cenosphere $\eta$ as the fracture stress depends on the mechanical properties of the skin material. However, in short beam shear test core shear stress and skin bending stress were found to be dependent on the cenosphere $\eta$ because of prominent shear stresses in the core material.
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VITA

Nikhil Gupta was born on July 23, 1973 in Jaipur, India. He finished his high school education in first class with distinction from Adarsh Vidya Mandir school, Jaipur in 1990. Nikhil joined Malaviya Regional Engineering College, affiliated to the University of Rajasthan, Jaipur, in 1991 for Bachelor of Engineering in the department of Metallurgical Engineering. He graduated from there in first class with distinction in 1996. He joined the prestigious Indian Institute of Science, Bangalore, to continue his education in the field of materials. Nikhil graduated, with Master of Engineering degree in metallurgical engineering in 1998. His work in masters thesis was on advanced particulate composites known as Syntactic Foams. The research paper published in the Journal of Non-Destructive Evaluation from his master’s thesis, was awarded the Best Paper Award from Indian Society for Non-Destructive Testing.

Inspired by the family background of teaching, Nikhil joined Punjab Engineering College (PEC), Chandigarh, India, as Lecturer (permanent) in 1998. His field of research was particulate composites at PEC. Within a brief period of one year he successfully developed a laboratory for the fabrication of advanced particulate composites and guided a graduate student for his master’s thesis in the field of glass fiber reinforced particulate composites.

Nikhil’s aspiration to solve the mysteries of the intriguing world of materials and build a career in teaching and research led him to join Louisiana State University, Baton Rouge, for a doctoral program, in 1999. He joined Dr. Eyassu Woldesenbet, Assistant Professor in Mechanical Engineering department for his studies. Nikhil’s field of research for doctoral degree was syntactic foams and their sandwich composites for weight sensitive applications. The research work carried out during his doctoral studies gained nationwide recognition when he was selected for American Society for Composites Ph.D. Research Scholarship Award and his research featured on the cover page of Journal of Materials Science in the year 2002. Nikhil’s research for his doctorate has yielded over two-dozen refereed journal papers, conference proceeding papers and presentations.