Nanoclay syntactic foam composites

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NANOCLAY SYNTACTIC FOAM COMPOSITES

A Thesis

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
Master of Science in Mechanical Engineering

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by
Sameer Leo Peter
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ABSTRACT

Syntactic foams are composite materials in which the matrix phase is reinforced with hollow particles called microballoons. They possess low moisture absorption, low thermal conductivity and high damage tolerance because of their compositions. Traditionally, syntactic foams are used in many high strength applications such as in aerospace and marine industries, thus there is a need to achieve both high compressive strength and high fracture strain with minimal increase in density. This research studies the effect of nanoclay on the high strain rate mechanical properties of syntactic foams. Nanoclay reinforced syntactic foams are fabricated by adding 1, 2 and 5% volume fraction of Nanomer I.30E nanoclay in syntactic foams having 10, 30 and 60% microballoon volume fraction. Transmission electron microscopy is performed to determine the dispersion of nanoclay in matrix. To compare the effect of nanoclay, plain syntactic foams without nanoclay are fabricated with same microballoon volume fraction. Two types of glass microballoons, S22 and K46, having different wall thickness are used in plain and nanoclay syntactic foams. High strain rate tests using split Hopkinson pressure bar (SHPB) apparatus are conducted on all types of plain and nanoclay syntactic foams and dynamic strength and modulus values are calculated. Also, quasi-static tests are conducted using MTS-810 machine and results are compared with dynamic SHPB results. The results demonstrated the importance of strain rate, nanoclay volume fraction and microballoon wall thickness in determination of syntactic foam properties. It is found that inclusion of 1% nanoclay gives the optimum enhancement in strength and modulus of nanoclay syntactic foams at all three microballoon volume fractions. The behavior of strength and modulus dependence on nanoclay volume fraction is found to be similar in both composite foams having S22 and K46 microballoons. Specimens exhibited higher strength and modulus at high strain rate than at lower strain rates. Based on stress-strain behavior of composite foams, energy absorption is also
calculated. It is found that thicker walled microballoons (K46) composite foams showed higher strength, modulus and energy absorption than those with thin walled (S22) microballoons. Scanning electron microscopy is performed to study the fracture behavior under different loading rates.
CHAPTER 1. INTRODUCTION

1.1 Composite Materials

1.1.1 Definition

In modern day applications use of high performance materials is the key for efficient functioning of the system. It is difficult to achieve such high level of performance from any single material. Thus new materials, which are combination of two or three different materials, are fabricated to satisfy the diverse performance needs in various applications. Such combinations of different materials are called composite materials.

Composite materials are engineered materials made from two or more constituent materials with significantly different physical or chemical properties and which remain separate and distinct on a macroscopic level within the finished structure. The properties of composite materials are different and improved as compared to constituent materials.

1.1.2 Classification of Composites

The constituent of composites includes a matrix and a reinforcement phase. The matrix can be either polymer matrix, metal matrix or ceramic matrix. Polymer matrix composites are more widely used because of several advantages such as resistance to chemicals, resistance to corrosion, high strength to weight ratio, low thermal and electrical conductivity and low moisture absorption. Further discussion in this research work deals with polymer based matrix composites. The reinforcement can be either single phase or multi phase. A multi phase reinforcement is achieved when number of materials are used together. Based on the reinforcement, composites are classified as fibrous composites or particulate composites [1].

1.1.2.1 Fibrous Composites

Fiber reinforced polymer (FRP) composites consist of fiber as filler material. The arrangement or orientation of the fibers relative to one another, the fiber concentration and the
fiber distribution have a significant influence on the strength and other properties of fiber-reinforced composites. FRPs have one or more discontinuous reinforcing phase in continuous matrix phase. These composites are widely used in many structural applications where their mechanical performances are of primary importance. Examples of fiber reinforced polymer composites include, vinyl ester resin matrix reinforced with glass fibers, epoxy matrix reinforced with carbon fibers, etc.

1.1.2.2 Particulate Composites

Particulate composites are made of one or more particulate phase reinforced in a continuous matrix phase. The reinforcing particles can be metallic, ceramic or polymeric [1]. The shape of the filler particles play important role in determining the properties of the composite. Some of the common particle shapes are spherical, flaky and cubical. Due to the difference in particle shape, the surface area is different for each particle at same volume fraction. Variation in size of particles affects the interfacial area between the particle and the matrix. Also, the stress concentration factor is different due to particle size and aspect ratio. Out of all the particle shapes available, spherical particles are more popular. Appropriate choice of particles can produce composites having superior strength, high damage tolerance, excellent wear and chemical resistance.

1.2 Syntactic Foams

The composites fabricated using hollow particles are called as syntactic foams. The hollow particles used are called as microballoons. Syntactic foams are classified as closed pore foams since the porosity exists inside the microballoons. Syntactic foams consist of a microballoons phase and a resin phase. Figure 1 shows a scanning electron microscopic image of syntactic foam having microballoons embedded in resin. During fabrication of syntactic foams, voids are formed in the matrix due to entrapped air. Because of the presence of these
voids, syntactic foams become three phase structures. A schematic representation of two and three phase structure is shown in Figure 2.

Figure 1. Scanning electron microscopic image showing syntactic foam

Figure 2. (a) Schematic of two phase syntactic foam and (b) Schematic of three phase syntactic foam

Syntactic foams were developed in the 1960s as buoyancy aid materials for deep sea applications [2]. Syntactic foams fabricated with microballoons possess lower density as compared to solid particulate composites and fiber reinforced composites. Microballoons used can be of glass, ceramic, steel and aluminum [3, 4] and are available in various sizes [5]. Closed
pore structures are considerably stiffer and stronger than open cell structured foams [6]. Syntactic foam composites usually possess low moisture absorption [7] and low thermal conductivity. They have high compressive strength and damage tolerance as compared to other closed cell structured materials, making them very attractive for structural applications [8]. Syntactic foams fabricated with higher volume fraction of microballoons can be used in various low density applications, such as naval, aeronautical and aerospace applications [9-13].

The physical and mechanical properties of syntactic foam composites can be varied by changing their density. Density of these composites is a function of size and volume fraction of microballoons. Low wall thickness and high volume fraction of microballoons gives lower density syntactic foams [7, 14]. This type of syntactic foams possesses relatively low compressive strength and high fracture strain. On the other hand, higher density microballoons impart higher compressive strength and lower fracture strain to syntactic foams [15]. However, these syntactic foams find limited use in various marine and aerospace applications where weight is an important factor. Thus there is a need to achieve both high strength and high fracture strain with minimal change in density. Published studies have concluded that addition of nanoclay have improved the mechanical properties of syntactic foams [16-21]. Thus, in this research work, nanoclay is chosen as reinforcement material in syntactic foam. Also, nanoclay possesses a unique ability of increasing impact strength and modulus of syntactic foams [22]. Since syntactic foams are used in various aeronautical and marine applications, study of high strain rate properties is important. Thus current research deals with the fabrication and characterization of nanoclay incorporated syntactic foams and plain syntactic foams. Nanoclay incorporated syntactic foams are fabricated with 1, 2 and 5% volume fraction of Nanomer I. 30E nanoclay and two types of microballoons, S22 and K46, having different wall thickness. The volume fraction of microballoons is 10, 30 and 60% in composite foams. Plain syntactic foams are fabricated
with same volume fractions of microballoons, but without nanoclay. Dynamic and quasi-static tests are performed on composite foams using split Hopkinson pressure bar apparatus and Instron MTS-810 machine, respectively. Dynamic test conducted on SHPB is termed as high strain rate test. Strength and modulus values are calculated using the SHPB data. Also, strength and modulus are calculated in quasi-static test using load-elongation curve obtained during testing of these materials. The strength and modulus values of plain and nanoclay incorporated syntactic foam composites obtained in these tests are compared to study the dependence of strain rate. Also, the combined effect of nanoclay volume fraction and microballoon wall thickness is studied.

1.3 Thesis Organization

Chapter 1 gives a brief introduction to syntactic foams and its advantages and applications.

Chapter 2 includes the previous work performed on syntactic foams to study its properties under different test parameters. It also includes the advantages of reinforcing syntactic foams with various fillers and selection of nanoclay for current research work.

Chapter 3 includes the description of various raw materials used, composition of materials in plain and nanoclay syntactic foams and fabrication procedure.

Chapter 4 includes the testing parameters considered in this research work.

Chapter 5 deals with the equations used for calculation of strength, modulus and energy absorbed.

The effect of strain rate, microballoon wall thickness and nanoclay volume fraction on strength and modulus of plain and nanoclay syntactic foams is discussed in Chapter 6.

Chapter 7 presents the conclusions and future work.
CHAPTER 2. LITERATURE REVIEW

Syntactic foams are engineered composite materials having a matrix phase and reinforcing microballoons phase. Bunn and Mottram studied the compressive properties of syntactic foams fabricated with phenolic microballoons at different volume fractions [23]. A decrease in compressive strength was observed by Bunn et al. [23] as the microballoon volume fraction increased. Gupta et al. conducted compressive tests on syntactic foams fabricated with glass microballoons and concluded that compressive strength is found to be higher than syntactic foams having phenolic microballoons [24]. Lin et al. concluded that as the volume fraction of microballoons increased the compressive strength decreased [25]. Leidner and Woodhams studied the strength of polymeric composites containing spherical filler materials [26].

Gupta et al. studied the mechanical properties of syntactic foams by taking radius ratio of microballoons into consideration [15]. Radius ratio is defined as the ratio between inner to outer radius of microballoons. Difference in radius ratio of microballoons causes a change in density of syntactic foams. The lower the radius ratio, the higher is the density of microballoons [15]. Gupta et al. conducted compressive tests on syntactic foams fabricated with five different types of microballoons having different radius ratio [15]. It was observed by Gupta et al. [15] that compressive strength and modulus of syntactic foams are dependent on microballoon radius ratio and specimen aspect ratio. It was also found that the compressive strength and modulus for syntactic foams incorporated with lower radius ratio microballoons was higher when compared to the syntactic foams with higher radius ratio microballoons.

The mechanical properties of syntactic foams can be altered by changing its density. Density of syntactic foams depends upon wall thickness and volume fraction of microballoons. Gupta and Woldesenbet performed compression tests on syntactic foams fabricated with different wall thickness microballoons [27]. It was found by Gupta and Woldesenbet that the
compressive strength and modulus were higher for syntactic foams having thicker walled microballoons. Also the stiffness of syntactic foams increases due to thicker walled microballoons [28]. d’Almeida studied the effect of microballoon size on compressive properties of syntactic foams and concluded that syntactic foams fabricated with smaller size microballoons showed lower stress and modulus than larger microballoons syntactic foam [5]. Gupta et al. conducted flat-wise and edge-wise compression test on syntactic foams fabricated with different radius ratio microballoons [15]. They concluded that the edge-wise compression test yielded better compressive modulus and compressive strength than flat-wise compression tests. Kim et al. studied the compressive failure mechanism of syntactic foam having varying concentration of resin [29]. They concluded that longitudinal splitting and layer crushing of specimen takes place under compression.

The quasi-static tensile properties of syntactic foams were studied by Gupta and Nagorny [30]. It was concluded by Gupta and Nagorny that the tensile strength increased with an increase in microballoon density and decreased with an increase in the volume fraction of microballoons having the same density. Also, the tensile modulus was found to increase with the microballoon density.

Gupta and Woldesenbet performed hygrothermal studies on syntactic foams and found that the compressive modulus decreased with an increase in moisture content [6]. It was also found by Gupta and Woldesenbet [6] that the peak compressive strength decreased with an increase in temperature from 25ºC to 70ºC. Also, the specimens showed a significant decrease in peak compressive strength compared to dry specimens at temperature of 70ºC.

Syntactic foams are used as core materials in sandwich structures because they possess good amount of favorable properties. Sandwich composites are fabricated by attaching two thin but stiff skins to light weight core material. Gupta et al. conducted both edge-wise and flat-wise
compression test on syntactic foam core sandwich composites [31]. It was concluded that the nature of the reinforcing skin has an affect on the test results in edge-wise compression test. Uniaxial tension and compression test was performed on syntactic foam core sandwich composites by Rizzi et al. [32]. It was concluded that sandwich structures have higher Young’s modulus in tension than in compression. Gupta and Woldesenbet conducted three and four point bending and short beam shear test on syntactic foam core sandwich structures [33]. Inorder to understand the affect of mode of loading on the flexural properties, the volume fraction of microballoons in syntactic foams used as core materials was maintained constant. It was concluded that the core shear stress and facing bending stress obtained in three-point and four-point tests were independent of wall thickness of microballoons. In short beam shear test, it was concluded that core shear stress and facing bending stress decreased with an increase in wall thickness of microballoons. Gupta et al. conducted three-point bending test on syntactic foam core sandwich structure having different specimen aspect ratio [8]. It was observed that the magnitude of bending stress in larger aspect ratio specimens was 30% more than lower aspect ratio specimens.

Earl and Shenoi studied the water uptake in closed cell polymeric structural foam and concluded that mass of composite foam increased with square root of time [34]. Hobaica and Cook studied the characteristics of syntactic foams used for buoyancy [35]. They concluded that as the density of syntactic foams decreased, the percentage weight change due to water absorption also decreased. Pal studied the electrical conductivity of particulate composite having varying volume fraction of particles and concluded that electrical conductivity increased as the volume fraction of particles increased [36].

Bardella and Genna constructed a numerical model to study the elastic behavior of syntactic foams [37]. They later compared the numerical prediction with analytical results and
concluded that analytical bulk modulus is found to be higher than numerical bulk modulus. Marur presented an analytical approach to compute the effective elastic moduli of syntactic foams [38]. It was concluded that the computed Young’s modulus values agree with the measured values for an epoxy filled with glass microspheres.

The quasi-static as well as dynamic properties of composite foams should be studied since the composite foams are increasingly substituted for metallic materials in various mechanical structures. The study of impact behavior of composites is important since they are employed in automobiles [39, 40]. Woldesenbet concluded that maximum load values of syntactic foams in impact increase for an increase in the wall thickness of microballoons [41]. Also, syntactic foams having thinner wall microballoons were found to have lower initiation energy but higher propagation energy compared with those having thicker wall microballoons at high impact velocity. In addition, the maximum load was found to higher for thinner wall microballoons. Li and Muthyala conducted impact test on hybrid grid stiffened structure having syntactic foam as core material [42]. Kim and Khamis also conducted impact tests on microsphere epoxy resin composites and concluded that the impact performance of composites used as protective materials can be enhanced by increasing the content of micro-spheres [43]. Wouterson et al. concluded that the fracture toughness of syntactic foams increased with increasing volume fraction of glass microballoons [44].

The analysis and design of composite materials or structures subjected to dynamic loading requires the input of high strain rate properties [45, 46]. Also, it is important to study the energy absorption of syntactic foams based on stress-strain behavior. Woldesenbet et al studied the dependence of strain rate on syntactic foams fabricated with different wall thickness microballoons [47]. Results demonstrate considerable increase in peak strength of syntactic foams for higher strain rates and increasing wall thickness of microballoons. It was also
observed that the elastic modulus increased with an increase in the strain rate and density of microballoons. Song et al. examined the behavior of epoxy syntactic foams at strain rate ranging from 550/s to 1030/s [48]. It was observed that the dynamic compressive strength of composite foam increased with strain rate up to 1030/s. Another study by Song et al. concluded that the failure strength of the epoxy syntactic foam increased due to an increase in strain rate [49]. Thiruppukuzhi and Sun also concluded that variation in strain rate could cause appreciable change in stress-strain curves of composite foams [50]. Yen et al. studied the effect of strain rate on crack tip behavior of particulate composite [51]. It was also concluded by Yen et al. that the fracture resistance of particulate composite increase with an increase in strain rate. Mae et al. studied the affect of strain rate on the tensile behavior of polypropylene syntactic foams having polymer microballoons [52]. They concluded that yield stress, modulus and rupture strain of syntactic foams are strongly dependent on strain rate. Song et al. used split Hopkinson pressure bar apparatus to study the affect of temperature on the dynamic compressive properties of syntactic foams [53]. It was concluded that for temperatures above a threshold value, the stress-strain curves exhibited increasing initial modulus. Also, at a fixed strain rate, when environmental temperature was above threshold temperature thermal softening dominated stress-strain behavior.

The properties of syntactic foams can be altered by varying the volume fraction and density of microballoons. Syntactic foams having high compressive strength and modulus can be fabricated by using high density microballoons (350-460 kg/m$^3$). However, such foams suffer from disadvantages of high density and low fracture strain of about 8-10% [17]. Also, this limits the use of syntactic foams in light weight applications. It is observed that syntactic foams using low density microballoons (200-350 kg/m$^3$) have lower strength but their fracture strain is higher than high density microballoons [15, 17]. Thus, there arises a need to achieve combination of...
high strength and high fracture strain in syntactic foams with minimal increase in density. One of the methods to achieve this improvement is to reinforce syntactic foams with filler materials. Some useful properties such as impact strength and modulus, damage tolerance and fracture strain can be modified by the addition of filler materials in matrix.

Wouterson et al. studied the effect of short carbon fiber reinforcement in syntactic foams [54]. It was concluded that inclusion of 3 wt% of short carbon fiber increased the tensile strength, Young’s modulus and fracture toughness of plain syntactic foams. The reinforcement effect of long Kevlar and carbon fiber was investigated by Huang et al. [55]. The results showed substantial increase in strength and modulus compared with the unreinforced syntactic foam when fibers were oriented in longitudinal direction. In transverse direction, the effect of addition of fibers in syntactic foams was not significant. On the other hand, particulate reinforcement materials don’t follow any orientation in the matrix. Gupta et al. studied the effect of rubber reinforcement on compressive properties of syntactic foams and reported an increase in fracture strain. However, the compressive modulus and strength were reduced [56]. Azimi et al. reported that the impact properties and fracture toughness of syntactic foams were improved by adding crumb rubber particles in the syntactic foam matrix [57]. However, a decrease in brittleness of the composite was found due to addition of crumb rubber leading to a reduction in the modulus values making the composite soft.

Nanoclay particles, on the other hand, have a unique ability of simultaneously increasing impact strength and modulus of composite foams [22]. Published studies have concluded that addition of nanoclay have improved the mechanical properties of syntactic foams. Gupta and Maharsia studied the compressive properties of nanoclay reinforced syntactic foams for sandwich core applications [17]. They concluded that with an addition of nanoclay in syntactic foams matrix, energy absorption increased by 80-200%. Maharsia and Jerro conducted tensile
tests on syntactic foams reinforced with nanoclay and concluded that tensile strength increased with the addition of nanoclay particles [16]. Maharsia et al. studied the flexural properties of nanoclay reinforced syntactic foam and concluded that flexural properties decreased for higher volume fraction of nanoclay [20]. Zhai et al. concluded that addition of nanoparticles increased the adhesion strength of an epoxy adhesive [19]. Timmerman et al. conducted the effect of nanoclay reinforcement on cryogenic micro-cracking of carbon fiber epoxy foams [18]. It was concluded that transverse cracking reduced significantly due to addition of very low concentration of nanoclay. Yu et al. investigated the friction and wear behavior of Polyoxymethylene composite reinforced with nanometer sized copper particle [21]. They concluded that reinforcement increased friction coefficient and decreased wear-weight loss compared to unreinforced composite.

The dynamic properties nanoclay reinforced syntactic foams under impact was studied by Woldesenbet [41]. It was concluded that addition of 1% volume fraction of nanoclay (3.59% by weight) is the optimum concentration required to achieve high impact resistance in syntactic foams having 60% volume fraction of microballoons. Dear et al. studied the impact velocity effect on nanoclay reinforced syntactic foam core sandwich structure [58]. It was concluded that higher impact produced through-the-thickness fracture resulting in the penetration of sandwich structures.

Various researchers have also studied the mixing behavior and dispersion aspect of nanoclay in the matrix. In conventional syntactic foam composites, the phase mixing occurs at micro-scale, whereas in nanoclay reinforced foam composites, mixing takes place at nano-scale [17, 59]. Nanocomposites involve mixing of nano or micro sized particles into the resin system. Recent studies have shown that small amount (up to 5 wt%) of ceramic nanoclay in polymer improves mechanical and wear properties [21]. The main reason for the increase in strength of
the composite matrix is the fact that large numbers of interfaces are created in the matrix upon dispersion of nanoclay [17, 60-62]. However, the degree of dispersion of nanoclay in epoxy resin is reported to have a significant impact on the mechanical properties of nanocomposites [22, 63]. The specific characteristics of nanocomposites such as strength, modulus and wear resistance can only be effective if it is provided that the nanoclay is well dispersed in the surrounding polymer [64-66]. Nanoclay has a strong tendency to agglomerate, and achieving homogenous dispersion is difficult. This is due to the reason that the free volume which allows nanoclay to move around decreases for relatively high nanoclay content. The higher the volume fraction of nanoclay, the lesser is the free volume for each nanoclay particle to move around in the epoxy resin [67]. Therefore, mechanical stirring, shear mixing and ultrasonic separation techniques only, cannot be effectively used to create good nanoclay dispersion when high percentage of nanoclay is included in polymers. In general, however, addition of nanoclay as reinforcing material in polymer matrix using these existing methods has produced encouraging results.

Even though several studies are performed to study the quasi-static and impact behavior of nanoclay reinforced syntactic foams, no information is available on the high strain rate properties of nanoclay syntactic foams. Moreover, since these syntactic foams are used in various high strength applications such as in aerospace and marine industries, studying their high strain rate behavior becomes significant.

Hence, the current study deals with the fabrication and characterization of plain and nanoclay syntactic foam composites in high strain rate conditions. As syntactic foams are used in various high strength and high modulus applications, high strain rate tests are conducted on nanoclay reinforced syntactic foams using split Hopkinson pressure bar apparatus. Quasi-static compression tests are also conducted on these foam materials using Instron MTS-810 machine.
Strength and modulus values are calculated using the SHPB data. Also, strength and modulus are calculated in quasi-static test using load elongation curve obtained during testing of these materials. The effect of microballoon density, strain rate and nanoclay volume fraction on the dynamic strength and modulus of plain and nanoclay syntactic foam composites is studied. Stress vs. strain graphs also give an indication of energy absorbed by these materials.
CHAPTER 3. FABRICATION AND NOMENCLATURE

3.1 Raw Materials

3.1.1 Nanoclay

The nanoclay used is a surface modified montmorillonite mineral having a trade name of ‘Nanomer I.30E Nanoclay’. It is manufactured by Nanocor Inc. and supplied by Sigma Aldrich Company. Nanoclay contains 25-30 wt. % octadecylamine. The general formula for class of montmorillonite is \((\text{Na,Ca})_{0.33}(\text{Al}_{2+y}\text{Mg}_y)\text{Si}_4\text{O}_{10}(\text{OH})_2\cdot n\text{H}_2\text{O}\) [68]. Figure 3 shows the crystal lattice structure of montmorillonite nanoclay. Nanoclay has a sheet type or a plately structure. A schematic arrangement of nanoclay platelets is as shown in Figure 4. From Figure 4 it can be observed that nanoclay occurs in stack of platelets where each platelet has a thickness of 1 nm. Average length and width of each platelet is approximately 1 µm. Each platelet is separated by a 2 nm space called ‘gallery’ due to surface modification. The bulk modulus of a single platelet is predicted to be 270 GPa by Manevitch et al. using simulation technique and different averaging models [68]. The physical properties of nanoclay are mentioned in Table 1 [69].

![Figure 3. Crystal lattice structure of montmorillonite nanoclay](image-url)
Figure 4. Schematic arrangement of nanoclay platelets

Table 1. Physical properties of Nanomer I.30E nanoclay

<table>
<thead>
<tr>
<th>Properties</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Appearance</td>
<td>White powder</td>
</tr>
<tr>
<td>Mean dry nanoclay stack size</td>
<td>8-10 µm</td>
</tr>
<tr>
<td>Density</td>
<td>1710 kg/m³</td>
</tr>
<tr>
<td>Moisture</td>
<td>3% max</td>
</tr>
<tr>
<td>Mineral purity</td>
<td>98.5% min</td>
</tr>
</tbody>
</table>

3.1.2 Microballoons

Two types of microballoons are used in this research work. These microballoons are identified as ‘S22’ and ‘K46’ which are hollow spherical particles of chemically-stable soda-lime-borosilicate glass. Microballoons are non-porous in nature and are manufactured and supplied by 3M Company under the trade name ‘Scotchlite’. The average outer diameter of each microballoon is 35 µm. These microballoons have different wall thickness leading to difference in density. K46 type of microballoons has a higher wall thickness than S22 microballoons, thus
having a lower radius ratio. Radius ratio is defined as the ratio of inner radius to outer radius. Equation 1 gives the radius ratio of microballoons.

\[ \eta = \frac{r_i}{r_o} \]  

Equation 1 gives the radius ratio of microballoons.

Figure 5 shows a schematic representation of microballoon radii. The other properties of these microballoons are mentioned in Table 2.

![Schematic representation of a microballoon](image)

Table 2. Properties of microballoons

<table>
<thead>
<tr>
<th>Properties</th>
<th>Microballoon Type</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>Average particle density [70]</td>
<td>S22</td>
<td>220 kg/m³</td>
</tr>
<tr>
<td>Radius ratio [27]</td>
<td></td>
<td>0.922</td>
</tr>
<tr>
<td>Average wall thickness [27]</td>
<td></td>
<td>1.26 μm</td>
</tr>
<tr>
<td>Isostatic crush strength [70]</td>
<td></td>
<td>2.8 MPa</td>
</tr>
<tr>
<td>Ultimate compressive load [71]</td>
<td></td>
<td>12 mN</td>
</tr>
<tr>
<td>Fracture energy [71]</td>
<td></td>
<td>25 nJ</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Properties</th>
<th>Microballoon Type</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>K46</td>
<td>460 kg/m³</td>
</tr>
<tr>
<td>Radius ratio [27]</td>
<td></td>
<td>0.863</td>
</tr>
<tr>
<td>Average wall thickness [27]</td>
<td></td>
<td>2.74 μm</td>
</tr>
<tr>
<td>Isostatic crush strength [70]</td>
<td></td>
<td>41 MPa</td>
</tr>
<tr>
<td>Ultimate compressive load [71]</td>
<td></td>
<td>15 mN</td>
</tr>
<tr>
<td>Fracture energy [71]</td>
<td></td>
<td>35 nJ</td>
</tr>
</tbody>
</table>

3.1.3 Resin

The resin used is a high purity diglycidylether of bisphenol-A (DGEBA) having a trade name of D.E.R.332. It is manufactured and supplied by DOW Chemical Company. D.E.R.332 has a tendency to crystallize if stored at a temperature below 25°C [72]. Crystallization may be induced by chilling, seeding by dust particles or incorporation of filler materials. However, heating the resin at 50-55°C restores it to liquid state. Long-term warm storage may result in
slight discoloration. This reversible process does not affect the performance of resin [73]. Typical properties of this resin are mentioned in Table 3.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density at 25 °C</td>
<td>1160 kg/m³</td>
</tr>
<tr>
<td>Flash point</td>
<td>252 °C</td>
</tr>
<tr>
<td>Viscosity at 25 °C</td>
<td>4000-6000 mPas</td>
</tr>
<tr>
<td>Appearance</td>
<td>Clear liquid</td>
</tr>
<tr>
<td>Weight at 25 °C</td>
<td>9.7 lbs/Gal</td>
</tr>
<tr>
<td>Shelf life</td>
<td>24 months</td>
</tr>
</tbody>
</table>

3.1.4 Diluent

C_{12-14} aliphatic mono-glycidyl ether is used as diluent for reducing the viscosity of resin. It has a trade name of Erisys GE-8 and is manufactured and supplied by CVC Specialty Chemicals. It is found to be compatible with epoxy resin at all concentrations [74]. The amount of diluent used in the resin system, affects the gel time and curing properties [74]. Table 4 displays typical properties of Erisys GE-8 diluent.

<table>
<thead>
<tr>
<th>Properties</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>890 kg/m³</td>
</tr>
<tr>
<td>Flash point</td>
<td>95 °C</td>
</tr>
<tr>
<td>Viscosity at 25 °C</td>
<td>5-10 mPas</td>
</tr>
<tr>
<td>Appearance</td>
<td>Clear liquid, clean</td>
</tr>
<tr>
<td>Weight at 25 °C</td>
<td>7.4 lbs/Gal</td>
</tr>
</tbody>
</table>

3.1.5 Curing Agent (Hardener)

Hardener D.E.H 24 is used for curing of matrix material. D.E.H 24 is liquid aliphatic polyamine curing agent called triethylene-tetra-amine (TETA). Due to its hygroscopic nature
[75], D.E.H.24 is stored in its original closed packaging. Table 5 displays typical properties of curing agent.

Table 5. Typical properties of D.E.H.24 epoxy curing agent

<table>
<thead>
<tr>
<th>Properties</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density</td>
<td>981 kg/m³</td>
</tr>
<tr>
<td>Flash point</td>
<td>118 °C</td>
</tr>
<tr>
<td>Viscosity at 25 °C</td>
<td>27 mPas</td>
</tr>
<tr>
<td>Appearance</td>
<td>Clear liquid</td>
</tr>
<tr>
<td>Shelf life</td>
<td>24 months</td>
</tr>
</tbody>
</table>

3.2 Fabrication Procedure

For fabrication of nanoclay incorporated syntactic foams, resin is preheated in an oven at 50-55 °C for 24 hours to restore it to liquid state. This reduces the viscosity of resin and helps in better wetting of microballoons and nanoclay during mixing. Resin is then removed from the oven and diluent is added to further reduce its viscosity. Nanoclay is then added in required quantity and mixture is mixed conventionally. Conventional mixing involves the use of hands to break the lumps visible under naked eye until they disappear. The mixture is then treated to ultrasonication process for 30 minutes under ultrasonic mixer, Figure 6, which is set at 30 seconds ON and 5 seconds OFF cycle. The ultrasonic mixer has the advantages of multi-effect of ultrasound such as crushing, activation and dispersion of particles [76]. In this research work, ultrasonic mixer is used for mixing of nanoclay in the matrix.

When ultrasonic waves pass through liquid medium (mixture of resin and diluent), a large number of microbubbles form, grow and collapse in very short time. This process is termed as ultrasonic cavitation [76]. During this process, nanoclay platelets are separated from each other forming nanoclay particles. These nanoclay particles can become energetic and can settle into formed bubbles. This mixture is removed after ultrasonication process and allowed to cool to ambient temperature as temperature rise takes place under ultrasonic mixer. Microballoons are
added and the mixture is mixed conventionally. Care should be taken so as not to damage the microballoons during mixing procedure. The mixture is again let cool to ambient temperature before hardener is added as rise in temperature could lead to exothermic reaction damaging the composite.

Once the temperature of mixture is equal to ambient temperature, hardener is added and the mixture is again mixed conventionally. The entire mixture is then poured into mold cavity. The mold cavity used is an aluminum tray which is pre-coated with a mold release agent. This mold release agent is a low surface energy liquid which forms a semi-permanent release barrier between the mold cavity and composite foam [77] contributing to the easy release of composite foam from the mold cavity. After the mixture is poured in the pre-coated mold cavity, it is uniformly compacted. It is then left for curing at ambient temperature for 36 hours. Cast foam
slabs are then removed from mold cavity and placed in an oven for post curing process for 3 hours at 100ºC.

Figure 7 shows fabricated composite foam slab after post curing process is completed. Plain syntactic foams are also fabricated using the same method but without the addition of nanoclay in the matrix.

3.3 Specimen Nomenclature

Nanoclay incorporated syntactic foams are termed as Nanoclay Syntactic Foams, thus having the acronym NSF. On the other hand, plain syntactic foams have acronym SF. The nomenclature of nanoclay syntactic foams and plain syntactic foams consists of an alphanumeric code indicating volume fraction and type of microballoons used. For example NSF2210-5 stands for nanoclay syntactic foam fabricated with S22 type of microballoons having 10% volume fraction and 5% volume fraction of nanoclay. Similarly, the alphanumeric code SF4660 stands for syntactic foam having K46 microballoons with 60% volume fraction.
3.4 Composition and Density of Composite Foams

In this research, eighteen different types of nanoclay syntactic foams and six different of plain syntactic foams (without nanoclay) are fabricated by varying the volume fraction of microballoons and nanoclay in the matrix. Table 6 shows the amount of nanoclay in composite foam and in matrix. From Table 6, it can be observed that nanoclay weight fraction in composite is different for composite foams having S22 and K46 microballoons. This is due to the reason that the density of K46 microballoons is more than S22 microballoons leading to an increase in weight of composite foams. Density and void content of plain and nanoclay reinforced syntactic foams are mentioned in Table 7.

Table 6. Composition of nanoclay and microballoons

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Nanoclay vol. frac. in composite (%)</th>
<th>Nanoclay wt. frac. in composite (%)</th>
<th>Nanoclay vol. frac. in matrix (%)</th>
<th>Nanoclay wt. frac. in matrix (%)</th>
<th>Microballoon vol. frac. in composite (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NSF2210-1</td>
<td>1</td>
<td>1.65</td>
<td>1.11</td>
<td>1.69</td>
<td>10</td>
</tr>
<tr>
<td>NSF4610-1</td>
<td>1</td>
<td>1.61</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NSF2210-2</td>
<td>2</td>
<td>3.28</td>
<td>2.22</td>
<td>3.35</td>
<td></td>
</tr>
<tr>
<td>NSF4610-2</td>
<td>2</td>
<td>3.21</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NSF2210-5</td>
<td>5</td>
<td>8.07</td>
<td>5.55</td>
<td>8.24</td>
<td></td>
</tr>
<tr>
<td>NSF4610-5</td>
<td>5</td>
<td>7.89</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NSF2230-1</td>
<td>1</td>
<td>1.99</td>
<td>1.43</td>
<td>2.16</td>
<td></td>
</tr>
<tr>
<td>NSF4630-1</td>
<td>1</td>
<td>1.84</td>
<td></td>
<td></td>
<td></td>
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<tr>
<td>NSF2230-2</td>
<td>2</td>
<td>3.96</td>
<td>2.85</td>
<td>4.29</td>
<td></td>
</tr>
<tr>
<td>NSF4630-2</td>
<td>2</td>
<td>3.66</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NSF2230-5</td>
<td>5</td>
<td>9.72</td>
<td>7.14</td>
<td>10.51</td>
<td></td>
</tr>
<tr>
<td>NSF4630-5</td>
<td>5</td>
<td>8.98</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NSF2260-1</td>
<td>1</td>
<td>2.92</td>
<td>2.5</td>
<td>3.76</td>
<td></td>
</tr>
<tr>
<td>NSF4660-1</td>
<td>1</td>
<td>2.34</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NSF2260-2</td>
<td>2</td>
<td>5.78</td>
<td>5</td>
<td>7.43</td>
<td></td>
</tr>
<tr>
<td>NSF4660-2</td>
<td>2</td>
<td>4.65</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NSF2260-5</td>
<td>5</td>
<td>14.03</td>
<td>12.5</td>
<td>17.91</td>
<td></td>
</tr>
<tr>
<td>NSF4660-5</td>
<td>5</td>
<td>11.35</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 7. Density and void content of plain and nanoclay reinforced syntactic foams having S22 and K46 microballoons

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Density</th>
<th>Void Content (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Theoretical (kg/m^3)</td>
<td>Actual (kg/m^3)</td>
</tr>
<tr>
<td>S2210-0</td>
<td>1030.31</td>
<td>1004.91</td>
</tr>
<tr>
<td>S2210-1</td>
<td>1036.21</td>
<td>1011.75</td>
</tr>
<tr>
<td>S2210-2</td>
<td>1042.11</td>
<td>1017.54</td>
</tr>
<tr>
<td>S2210-5</td>
<td>1059.79</td>
<td>1035.02</td>
</tr>
<tr>
<td>S2230-0</td>
<td>850.24</td>
<td>807.8</td>
</tr>
<tr>
<td>S2230-1</td>
<td>856.14</td>
<td>816.54</td>
</tr>
<tr>
<td>S2230-2</td>
<td>862.04</td>
<td>829.74</td>
</tr>
<tr>
<td>S2230-5</td>
<td>879.73</td>
<td>848.57</td>
</tr>
<tr>
<td>S2260-0</td>
<td>580.14</td>
<td>547.34</td>
</tr>
<tr>
<td>S2260-1</td>
<td>586.03</td>
<td>556.72</td>
</tr>
<tr>
<td>S2260-2</td>
<td>591.93</td>
<td>563.59</td>
</tr>
<tr>
<td>S2260-5</td>
<td>609.62</td>
<td>580.24</td>
</tr>
<tr>
<td>K4610-0</td>
<td>1054.31</td>
<td>1043.12</td>
</tr>
<tr>
<td>K4610-1</td>
<td>1060.21</td>
<td>1050.96</td>
</tr>
<tr>
<td>K4610-2</td>
<td>1066.1</td>
<td>1057.43</td>
</tr>
<tr>
<td>K4610-5</td>
<td>1083.8</td>
<td>1073.37</td>
</tr>
<tr>
<td>K4630-0</td>
<td>922.24</td>
<td>902.3</td>
</tr>
<tr>
<td>K4630-1</td>
<td>928.14</td>
<td>909.5</td>
</tr>
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<td>K4630-2</td>
<td>934.04</td>
<td>917.03</td>
</tr>
<tr>
<td>K4630-5</td>
<td>951.73</td>
<td>940.1</td>
</tr>
<tr>
<td>K4660-0</td>
<td>724.14</td>
<td>685.26</td>
</tr>
<tr>
<td>K4660-1</td>
<td>730.03</td>
<td>695.05</td>
</tr>
<tr>
<td>K4660-2</td>
<td>735.93</td>
<td>700.86</td>
</tr>
<tr>
<td>K4660-5</td>
<td>753.62</td>
<td>720.71</td>
</tr>
</tbody>
</table>

3.5 Specimen Preparation

Specimens required for high strain rate and compression tests are cylindrical and rectangular in shape, respectively. For high strain rate test, nine cylindrical samples are core drilled from the composite foam slab using a core-drill. The end surfaces of the specimen are ground flat so as to fit between the flat ends of SHPB bars. The specimen obtained is as shown in Figure 8. The average length and diameter of each specimen is 12 mm and 9.5 mm, respectively.
Specimens required for quasi-static compressive test are cut in 25.4 mm × 12.7 mm and height of 25.4 mm dimension using the ASTM standard D695-02a [78]. Figure 9 shows a sample for quasi-static compression test.

Figure 8. Specimen for high strain rate test

Figure 9. Specimen for quasi-static compression test
3.6 Exfoliation and Intercalation

The complete dispersion of nanoclay in the matrix is called exfoliation [79] and improper dispersion is termed as intercalation [80]. The enhancement of mechanical properties of composite foams depends upon the extent of intercalation and/or exfoliation of nanoclay in the matrix [80, 81]. Transmission electron microscopy (TEM) is performed on two specimens having different volume fraction of microballoons. Figure 10 shows the TEM image of NSF2210-5. It can be observed that nanoclay stacks are separated into nanoclay platelets (particles), thus dispersing efficiently into the matrix. This phenomenon is called exfoliation.

Figure 10. TEM image of nanoclay dispersion in NSF2210-5

Figure 11 shows the TEM image of NSF2260-5. Here it can be observed that nanoclay stacks are not separated into nanoclay platelets (particles), leading to agglomeration. This phenomenon is termed as intercalation. The importance of nanoclay dispersion is discussed in Chapter 6.
Figure 11. TEM image of nanoclay dispersion in NSF2260-5
CHAPTER 4. TESTING

4.1 Split Hopkinson Pressure Bar Apparatus

A split Hopkinson pressure bar apparatus is used to conduct dynamic compression tests on specimens for strain rates exceeding several hundred per second, Figure 12. Hopkinson pressure bar apparatus consists of three axial bars, pressure gun assembly and a pressure release valve. The three bars are striker, incident and transmitter bar, of which striker bar is positioned inside the pressure gun assembly. All three bars are made of maraging steel having an elastic modulus of 200 GPa. They are axially mounted on blocks which provide support. These bars are carefully aligned so as to produce one-dimensional wave front. If these bars are not aligned, then a non-uniform stress wave distribution will exist across the bar cross-section resulting into a multi dimensional strain field. The faces of all bars are flat and parallel. The diameter of the bars is 9.5 mm and length of striker, incident and transmitter bars are 152, 1220 and 610 mm, respectively. Thus the length to diameter ratio is more than 10 as suggested by Kaiser [82].

The specimen is sandwiched between the incident and transmitter bar as shown in Figure 13. The striker bar impacts the incident bar when accelerated by the pressure gun on the actuation of pressure release valve. The velocity of striker bar is controlled by the pressure inside the pressure gun assembly. At the impact, an elastic compressive wave is generated at the striker-incident bar interface and travels towards the specimen. This wave is shaped using a pulse-shaper so that the specimens are in dynamic stress equilibrium [83]. The pulse shaper used is made of mild steel with 6.73 mm diameter and 3.29 mm length [14]. When this wave reaches the specimen, a part of it gets reflected from the specimen-incident bar interface and remaining wave transmits to the transmitter bar through the specimen. The reflected wave is tensile in nature.
Figure 12. Split Hopkinson pressure bar apparatus

Figure 13. Specimen sandwiched between incident and transmitter bar
Large numbers of internal wave reflections are experienced in the short specimen during the transition of compressive wave through the specimen. It is assumed that the specimen deforms uniformly throughout its length. During compression the specimen shortens in length and expands radially. A frictional constraint exists at the pressure bar-specimen interface due to this radial expansion. This constraint is highest when the specimen is at rest and reduces once the specimen starts sliding. Due to this initial constraint, the ends of specimen are restrained and it expands at the middle section resulting in a barrel shaped deformation. Such kind of deformation is clearly not uniform in nature. By applying thin films of lubricant at the pressure bar-specimen interfaces, this frictional constraint can be greatly reduced [84]. In this research work, a thin film of molybdenum disulphide lubricant is applied at the pressure bar-specimen interface in order to reduce friction.

Two strain gages with 350 Ω and 2.105 gage factor, each, are mounted on incident and transmitter bar, respectively, at an equal distance of 185 mm from the incident-transmitter bar interface. These strain gages are connected to signal bridge conditioner where the signal from
strain gages is amplified and is then displayed in an oscilloscope. The data from the oscilloscope is then transferred to a computer for further data reduction. Figure 14 shows the signal bridge conditioner and the oscilloscope. Figure 15 shows the block diagram of the arrangement of Hopkinson bar apparatus.

In this research work, the specimens are subjected to three different strain rates. The strain rates obtained are dependent on the velocity of the striker bar which is dependent on the pressure inside the pressure gun assembly. Three different pressures are used to produce three different strain rates ranging from 360/s to 1100/s. To obtain the repeatability, three replicate specimens are subjected to same strain rate.

4.2 Material Test System-810

A MTS-810 material test system is used to conduct quasi-static compression testing on the specimens. It is a hydraulic machine which can be used to conduct compression test, tensile test, fatigue test, flexural test on materials such as aluminum, composites, plastics, elastomers, steel [85]. In this research work, the machine is used to conduct quasi-static compression test on composite foam specimens according to ASTM standard D695-02a [78]. Figure 16 shows a MTS-810 machine. A microprocessor controlled data acquisition system is used to collect data.
obtained during the tests. During the test, the crosshead speed is maintained at 1.3 mm/min. For repeatability, five specimens of same type are compressed edgewise along the specimen height of 25.4 mm between two hardened steel plates as shown in Figure 17.

Figure 16. MTS-810 machine

Figure 17. Edgewise compression testing of composite foam specimen
CHAPTER 5. DETERMINATION OF MECHANICAL PROPERTIES

5.1 Dynamic Properties

The raw data obtained from the SHPB apparatus is in the form of voltage vs. time graph as shown in Figure 18. Figure 18 shows reading obtained from two strain gages mounted on incident and transmitter bar, respectively. Strain gage mounted on incident bar is termed as channel 1 and that on transmitter bar is termed as channel 2.

![Figure 18. Graph obtained in oscilloscope during SHPB test](image)

5.1.1 Calculation of Strength

From the data obtained, stress is calculated using Equation (2)

\[
\sigma = E \times \varepsilon_T \times \frac{D_B^2}{D_S^2}
\]  

(2)

where, \(\sigma = \text{stress}\)
\(E\) = Elastic modulus of bar,

\(\varepsilon_T\) = Transmitted strain,

\(D_B\) = Diameter of bar,

\(D_S\) = Diameter of specimen.

The highest value of stress obtained is termed as strength of the specimen.

Considering that the specimen deforms uniformly, the strain in the incident bar is equal to strain in transmitted bar. The strain in the incident bar is a sum of incident strain and reflected strain, due to incident and reflected waves passing through channel 1, respectively. The equality is seen in Equation (3).

\[\varepsilon_T = \varepsilon_i + \varepsilon_R \tag{3}\]

where, \(\varepsilon_i\) = Incident strain,

\(\varepsilon_R\) = Reflected strain.

### 5.1.2 Calculation of Strain Rate

Strain rate is calculated using Equation 4.

\[
\dot{\varepsilon} = \frac{2 \times C \times \varepsilon_R}{L_S} \tag{4}
\]

where, \(\dot{\varepsilon}\) = Strain rate

\(L_S\) = Length of specimen,

\(C\) = Velocity of wave in the bar.

This velocity of wave in the bar is calculated using Equation 5.

\[
C = \frac{\sqrt{E}}{\sqrt{\rho}} \tag{5}
\]

where, \(\rho\) = Density of bar.
5.1.3 Calculation of Strain

This strain is the strain generated in specimen. It is calculated using Equation 6.

\[ \varepsilon = \dot{\varepsilon}_n \times \Delta t + \dot{\varepsilon}_{n-1} \]  \hspace{1cm} (6)

where, \( \dot{\varepsilon}_n = \) Strain rate at an instance,

\( \dot{\varepsilon}_{n-1} = \) Strain rate at previous instance,

\( n = \) Natural number,

\( \Delta t = \) Difference in sampling rate.

5.1.4 Stress vs. Strain Graph and Modulus

Stress vs. strain graph is plotted to calculate the modulus of specimen. Figure 19 shows a typical stress vs. strain graph at lower strain rate. Modulus is further calculated from stress vs. strain graph. The linear part of this graph is considered and its slope represents the modulus value for the specimen.

![Figure 19. Stress vs. strain graph for dynamic test](image-url)
5.1.5 Energy Absorbed

The area under the stress vs. strain curve is termed as energy absorbed per unit volume. It is calculated using the trapezoidal rule as shown in Figure 20.

![Figure 20. Trapezoidal rule for calculation of energy absorbed](image)

5.2 Quasi-Static Properties

Data obtained in quasi-static test is in the form of load vs. elongation curve. Stress is calculated using Equation 7. The highest value of stress obtained is the strength of the specimen.

\[
\sigma_{QS} = \frac{\text{Load}}{C_S}
\]  

(7)

where, \( \sigma_{QS} = \) Quasi-static stress,

\[
\text{Load} = \text{Load applied on the specimen},
\]

\[
C_S = \text{Cross-section area of the specimen} = 25.4 \times 12.7 \text{ mm}^2.
\]
Figure 21 shows the stress vs. strain graph. The linear part of this curve is considered for obtaining the modulus value of specimen.

Strain is calculated using Equation 8.

\[ \varepsilon = \frac{\text{Extension}}{H_s} \]  

(8)

where, \( \varepsilon = \) Strain,

Extension = Extension of specimen,

\( H_s = \) Specimen height.

Figure 21. Stress vs. strain graph for quasi-static test
CHAPTER 6. RESULTS AND DISCUSSION

Dynamic compression test results of nanoclay syntactic foams are discussed and further compared with plain syntactic foams. In addition, quasi-static tests are also conducted in order to compare the dynamic results with the quasi-static values.

6.1 Strength

The strength results obtained from dynamic and quasi-static compressive tests for composite foams having S22 microballoons are mentioned in Tables 8-11. These tables show the average values of dynamic strength at three different strain rates along with quasi-static strength.

Particular results of dynamic and quasi-static strength for some of the specimens having S22 microballoons are plotted in Figures 22-24 as a function of nanoclay volume fraction at three different strain rates. These strain rates are termed as high (approximately 800/s), medium (approximately 450/s) and low (quasi-static).

Table 8. Strength of S22 composite foams at 0% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s⁻¹)</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF2210</td>
<td>Quasi-static</td>
<td></td>
</tr>
<tr>
<td></td>
<td>409.09</td>
<td>92±4</td>
</tr>
<tr>
<td></td>
<td>508.78</td>
<td>71±4</td>
</tr>
<tr>
<td></td>
<td>591.34</td>
<td>118±12</td>
</tr>
<tr>
<td></td>
<td>135±10</td>
<td></td>
</tr>
<tr>
<td>SF2230</td>
<td>Quasi-static</td>
<td></td>
</tr>
<tr>
<td></td>
<td>524.78</td>
<td>64±2</td>
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<tr>
<td></td>
<td>708.47</td>
<td>54±0</td>
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<tr>
<td></td>
<td>820.3</td>
<td>103±7</td>
</tr>
<tr>
<td></td>
<td>121±12</td>
<td></td>
</tr>
<tr>
<td>SF2260</td>
<td>Quasi-static</td>
<td></td>
</tr>
<tr>
<td></td>
<td>518.4</td>
<td>31±3</td>
</tr>
<tr>
<td></td>
<td>729.13</td>
<td>60±3</td>
</tr>
<tr>
<td></td>
<td>1067.98</td>
<td>76±4</td>
</tr>
<tr>
<td></td>
<td>104±7</td>
<td></td>
</tr>
</tbody>
</table>
Table 9. Strength of S22 composite foams at 1% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s⁻¹)</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Quasi-static</td>
<td></td>
</tr>
<tr>
<td>NSF2210-1</td>
<td>431.22</td>
<td>100±4</td>
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<td></td>
<td>572.82</td>
<td>88±10</td>
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<td></td>
<td>653.96</td>
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<td>827.946</td>
<td>65±6</td>
</tr>
<tr>
<td></td>
<td>143±10</td>
<td></td>
</tr>
<tr>
<td>NSF2260-1</td>
<td>507.8</td>
<td>40±3</td>
</tr>
<tr>
<td></td>
<td>791.23</td>
<td>58±5</td>
</tr>
<tr>
<td></td>
<td>966.17</td>
<td>77±4</td>
</tr>
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<td></td>
<td>64±9</td>
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</table>

Table 10. Strength of S22 composite foams at 2% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s⁻¹)</th>
<th>Strength (MPa)</th>
</tr>
</thead>
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<tr>
<td></td>
<td>Quasi-static</td>
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</tr>
<tr>
<td>NSF2210-2</td>
<td>473.43</td>
<td>88±3</td>
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<td>585.85</td>
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<td></td>
<td>606.38</td>
<td>163±3</td>
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<tr>
<td></td>
<td>183±10</td>
<td></td>
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<tr>
<td>NSF2230-2</td>
<td>476.12</td>
<td>75±2</td>
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<tr>
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<td>713.83</td>
<td>72±6</td>
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<td></td>
<td>837.67</td>
<td>120±7</td>
</tr>
<tr>
<td></td>
<td>123±2</td>
<td></td>
</tr>
<tr>
<td>NSF2260-2</td>
<td>562.6</td>
<td>33±3</td>
</tr>
<tr>
<td></td>
<td>600.64</td>
<td>45±2</td>
</tr>
<tr>
<td></td>
<td>690.39</td>
<td>71±3</td>
</tr>
<tr>
<td></td>
<td>78±7</td>
<td></td>
</tr>
</tbody>
</table>

Figure 22 shows strength vs. nanoclay volume fraction for nanoclay and plain syntactic foams fabricated with 10% volume fraction of S22 microballoons. It can be observed that the strength at high strain rate test is higher compared to medium and low strain rate tests demonstrating the effect of strain rate on the materials properties. This behavior is due to the propagation of cracks through the specimen at different speeds during mechanical test
Table 11. Strength of S22 composite foams at 5% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s⁻¹)</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Quasi-static</td>
<td></td>
</tr>
<tr>
<td>NSF2210-5</td>
<td>450.29</td>
<td>82±2</td>
</tr>
<tr>
<td></td>
<td>548.11</td>
<td>95±4</td>
</tr>
<tr>
<td></td>
<td>563.23</td>
<td>170±3</td>
</tr>
<tr>
<td></td>
<td>562.6</td>
<td>56±2</td>
</tr>
<tr>
<td></td>
<td>600.64</td>
<td>75±4</td>
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<tr>
<td></td>
<td>690.39</td>
<td>119±8</td>
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<tr>
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<td>21±2</td>
<td>135±6</td>
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<tr>
<td></td>
<td>55±3</td>
<td></td>
</tr>
<tr>
<td>NSF2230-5</td>
<td>450.29</td>
<td>56±2</td>
</tr>
<tr>
<td></td>
<td>548.11</td>
<td>75±4</td>
</tr>
<tr>
<td></td>
<td>563.23</td>
<td>119±8</td>
</tr>
<tr>
<td></td>
<td>55±3</td>
<td></td>
</tr>
<tr>
<td>NSF2260-5</td>
<td>450.29</td>
<td>21±2</td>
</tr>
<tr>
<td></td>
<td>548.11</td>
<td>45±3</td>
</tr>
<tr>
<td></td>
<td>563.23</td>
<td>48±4</td>
</tr>
<tr>
<td></td>
<td>55±3</td>
<td></td>
</tr>
</tbody>
</table>

Figure 22. Strength vs. nanoclay volume fraction for 10% S22 microballoons composite foams performed at different strain rates. At low strain rate, the speed of cracks is low and they travel preferentially through the matrix avoiding most of the microballoons and nanoclay in their path.
Figure 23. Strength vs. nanoclay volume fraction for 30% S22 microballoons composite foams

Figure 24. Strength vs. nanoclay volume fraction for 60% S22 microballoons composite foams
However, at high strain rate cracks propagate faster and do not follow a preferential direction or path [47]. Figure 25 and Figure 26 shows crack propagation in nanoclay syntactic foam specimens at medium and high strain rates, respectively. At high strain rate, these cracks encounter microballoons and nanoclay particles which are stronger than the matrix. These particles can sustain more force and thus causing the strength of the specimen to increase. The more the amount of nanoclay encountered by cracks, the higher is the strength. Thus, there is an increase in strength at high and medium strain rates when nanoclay upto 5% by volume is added to pure syntactic foams. At low strain rate (quasi-static), there is no significant variation in strength when nanoclay is incorporated in the specimen due to the preferential path followed by cracks.

![Figure 25. SEM image showing crack passing around microballoon in NSF2210-2 specimen at medium strain rate](image)

However, comparing the strength for pure syntactic foam and 1% nanoclay volume fraction nanoclay syntactic foam, the strength at quasi-static strain rate is found to be higher than that at medium strain rate. This unexpected result can be explained by analyzing the SEM
images of the fractured surfaces of the two samples, Figure 27 and Figure 28. In quasi-static case, the crack propagation is mainly through the matrix, while in medium strain rate (450/s), the crack has a tendency to go through the weaker interface between the matrix and the microballoon. This phenomenon in addition to crushing of microballoons is common to the fracture behavior of plain syntactic foams at medium and low strain rates.

When tested at the same strain rate, the volume fraction of the nanoclay is found to affect the strength values. At low volume fraction of microballoons, the amount of free volume of matrix is more. Therefore, nanoclay gets dispersed more efficiently at lower microballoon volume fraction than at higher microballoon volume fraction as can be observed in the TEM image of NSF2210-5 (Figure 10). Due to good dispersion, cracks tend to encounter the nanoclay platelets. Figure 29 demonstrates this reasoning by showing the fractured surfaces of NSF specimen with 5% nanoclay tested at high strain rate.

As shown in Figure 22, the effect of nanoclay volume fraction is shown to be even more significant for higher strain rate. The higher the strain rate, the higher is the chance for the
cracks to directly encounter nanoclay than at low strain rate because of indiscriminate crack propagation as mentioned earlier.

Figure 27. Crack passing through matrix in NSF2210-1 specimen at low strain rate (quasi-static)

Figure 28. SEM image showing crack passing around microballoons in NSF2210-1 specimen at medium strain rate

Figure 23 shows strength vs. nanoclay volume fraction for nanoclay and plain syntactic foams having 30% volume fraction of S22 microballoons. It can be observed that the strength at
high strain rate is higher than that observed at medium and low strain rate. This is again due to the speed of crack propagation at varying strain rates. Figure 30 shows the SEM image indicating the preferential path followed by the crack in NSF2230-1 specimen at medium strain rate. On the other hand, at high strain rate (800/s), crack follows an indiscriminate path as shown
rate. On the other hand, at high strain rate (800/s), crack follows an indiscriminate path as shown in SEM image of NSF2230-2 specimen in Figure 31.

![Crack through microballoon in NSF2230-2 specimen at high strain rate](image)

**Figure 31.** Crack through microballoon in NSF2230-2 specimen at high strain rate

The strength at medium and low strain rate follows similar trend as in 10% microballoon volume fraction composite foams. In addition, as in the case of 10% microballoon volume fraction nanoclay syntactic foams, the quasi-static strength is greater than the strength value at medium strain rates with nanoclay at 0 and 1% volume fractions, respectively. However, the difference is smaller at 30% microballoon volume fraction because of the higher influence of microballoons on the fracture strength.

The strength at high strain rate for composite foams having 30% volume fraction of S22 microballoons is found to be much higher than both medium and low strain rate as compared to that found in composite foams having 10% volume fraction of S22 microballoons. While the influence of nanoclay on the property of the composite foam at medium and low strain rate is not significant, the influence at high strain rate is clearly evident as shown in Figure 23. At high strain rate, the strength increases with 1% nanoclay addition and decreases on further addition of
nanoclay. More specifically, the high strain rate strength at 2 and 5% nanoclay is found to be similar to plain syntactic foam. One reason for the reduction in strength for 2 and 5% nanoclay syntactic foam is the non-uniform dispersion of the nanoclay caused by their incomplete wetting which is due to the reduction of the matrix phase in the composite foams. Thus, nanoclay agglomerations are formed leading to stress concentrations and voids in the matrix due to increased bare surface of nanoclay platelets without any matrix coverage in the composite foam. Another reason for reduction of strength is that the flake shape of the nanoclay platelet enhances the inhomogeneous stress distribution in itself [86]. The strength similarity of plain syntactic foam and 2 and 5% volume fraction nanoclay syntactic foam at 30% microballoon volume fraction can be explained by the balance between the increase in strength due to proper dispersion and decrease due to agglomeration and flake shape of the nanoclay platelets.

Figure 32. SEM image showing crack propagation in NSF2260-1 in quasi-static test

Figure 24 shows strength vs. nanoclay volume fraction for nanoclay and plain syntactic foams having 60% volume fraction of S22 microballoons. It can be observed that the strength at medium strain rate is higher than low strain rate unlike the previous cases at 10 and 30% volume
fraction composite foams. Here also, the strength at high strain rate is higher than at medium
and low strain rate tests clearly indicating the effect of high strain rate on nanoclay syntactic
foams. At low strain rate (quasi-static) the crack propagates slowly in a preferential path
avoiding most microballoons and nanoclay as observed in Figure 32. Moreover, at 60%
microballoon volume fraction, the chance for the cracks to propagate through the microballoons
is higher than at the lower microballoon volume fractions as can be observed in Figure 33.
Therefore, the strength at high and medium strain rates is higher than at low strain rate as the
cracks encounter microballoons that are stronger materials than the matrix.

![SEM image showing crack propagation in NSF2260-1 in high strain rate test](image)

On the other hand, the effect of nanoclay is more damaging at 60% microballoon volume
fraction specimens. It can be observed that at high strain rate, the strength slightly increases with
a 1% addition of nanoclay to pure SF. For further increase of nanoclay to 2 and 5%, strength
reduces and goes below the value for plain syntactic foams. This unusual observation is directly
related to the amount of microballoons. At 60% microballoon volume fraction the amount of
matrix present in the composite is less and is further reduced by the addition of nanoclay. This leads to the reduction in the load transfer between the reinforcing and matrix phases of the composite. In addition, nanoclay has less free volume to disperse in the matrix at 60% microballoon volume fraction. Figure 34 shows the TEM image of dispersion of nanoclay in NSF2260-5. It can be observed that at 60% microballoon volume fraction, equivalent to 12.5% nanoclay volume fraction in the matrix, nanoclay agglomerations are higher as compared to 10% and 30% microballoon volume fraction syntactic foams. These nanoclay agglomerations in the vicinity of the microballoons, creates point load conditions at the outer surface of the microballoons causing premature fracture of the microballoons, Figure 35.

![Figure 34. TEM image showing nanoclay agglomeration in NSF2260-5 specimen](image)

These nanoclay agglomerations also lead to creation of voids and thus stress concentration due to increased bare surfaces of nanoclay platelets [14]. Nanoclay interfacial area has been known to affect the characteristics of materials. Wetzel et al. concluded that high nanoclay concentration showed higher interfacial area [87]. However, this interfacial area contributes to higher inter-particle interaction which makes dispersion difficult beyond a small
amount of nanoclay. Also, the flake shape of nanoclay causes inhomogeneous stress distribution in itself leading to reduction in strength of composite foams [86]. Moreover, the ultrasonic dispersion technique cannot be used effectively at higher nanoclay volume fraction [67]. Thus, more defects in the form of nanoclay agglomerations and voids area are created.

Figure 36 shows the SEM image of nanoclay syntactic foam having 60% microballoon and 5% nanoclay volume fraction indicating voids in the matrix because of the inefficient percolation. During tests, the generated cracks travel faster through these voids as they face no resistance, eventually leading to composite foam failure as shown in SEM image in Figure 37. This phenomenon is partially responsible for reduction of strength in higher microballoon volume fraction at all strain rates. Also, it is mentioned that the characteristic properties of nanoclay syntactic foams are only observed when nanoclay is well dispersed in the matrix [62]. However, at 1% nanoclay volume fraction where there is no significant change in strength values, there exists a balance between the effect of fast crack propagation through microballoons and nanoclay increasing the strength and the crack propagation through voids and weakened matrix due to various defects mentioned.

Figure 35. Nanoclay agglomeration in the vicinity of microballoons
Figure 36. SEM image showing voids in matrix of NSF2260-5 specimen

Figure 37. SEM image showing crack propagation through voids in NSF2260-5

The results obtained from dynamic and quasi-static compressive strength for composite foams having K46 microballoons are mentioned in Tables 12-15.

Figure 38 shows strength vs. nanoclay volume fraction for nanoclay and plain syntactic foams having 10% volume fraction of K46 microballoons. It can be observed that the strength at
Table 12. Strength of K46 composite foams at 0% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s(^{-1}))</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>SF4610</strong></td>
<td>Quasi-static 454.23</td>
<td>105±5</td>
</tr>
<tr>
<td></td>
<td>529.52</td>
<td>78±5</td>
</tr>
<tr>
<td></td>
<td>679.55</td>
<td>113±3</td>
</tr>
<tr>
<td></td>
<td>Quasi-static 441.54</td>
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<td>605.03</td>
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<td>694.37</td>
<td>132±9</td>
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<td><strong>SF4630</strong></td>
<td>Quasi-static 483.52</td>
<td>59±6</td>
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<td>624.67</td>
<td>62±6</td>
</tr>
<tr>
<td></td>
<td>722.56</td>
<td>59±2</td>
</tr>
</tbody>
</table>

Table 13. Strength of K46 composite foams at 1% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s(^{-1}))</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
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<tr>
<td></td>
<td>Quasi-static 418.43</td>
<td>100±2</td>
</tr>
<tr>
<td></td>
<td>534.97</td>
<td>91±2</td>
</tr>
<tr>
<td></td>
<td>547.59</td>
<td>163±10</td>
</tr>
<tr>
<td><strong>NSF4630-1</strong></td>
<td>Quasi-static 573.71</td>
<td>70±4</td>
</tr>
<tr>
<td></td>
<td>656.35</td>
<td>85±9</td>
</tr>
<tr>
<td></td>
<td>766.55</td>
<td>140±14</td>
</tr>
</tbody>
</table>

high strain rate is higher than at medium and low strain rate. This result demonstrates the effect of strain rate on the strength of composite foams which is found to be irrespective of the type of microballoons. Thus the speed and path of crack is determined by the strain rate to which the to which the specimens are subjected.
Table 14. Strength of K46 composite foams at 2% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s(^{-1}))</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Quasi-static</td>
<td></td>
</tr>
<tr>
<td></td>
<td>522.87</td>
<td>101±2</td>
</tr>
<tr>
<td>NSF4610-2</td>
<td>559.66</td>
<td>107±5</td>
</tr>
<tr>
<td></td>
<td>655.2</td>
<td>159±6</td>
</tr>
<tr>
<td></td>
<td>Quasi-static</td>
<td>208±22</td>
</tr>
<tr>
<td>NSF4630-2</td>
<td>478.12</td>
<td>95±6</td>
</tr>
<tr>
<td></td>
<td>573.85</td>
<td>81±3</td>
</tr>
<tr>
<td></td>
<td>712.9</td>
<td>175±9</td>
</tr>
<tr>
<td></td>
<td>Quasi-static</td>
<td>201±15</td>
</tr>
<tr>
<td>NSF4660-2</td>
<td>591.52</td>
<td>68±2</td>
</tr>
<tr>
<td></td>
<td>644.21</td>
<td>78±2</td>
</tr>
<tr>
<td></td>
<td>793.96</td>
<td>133±2</td>
</tr>
<tr>
<td></td>
<td></td>
<td>143±6</td>
</tr>
</tbody>
</table>

As mentioned earlier, cracks encounter more amounts of microballoons and nanoclay at high strain rate increasing the strength of composite foams. This effect of strain rate is more significant at 5% volume fraction of nanoclay. Thus, the dependency of strength on strain rate is similar to as observed in composite foams having 10% volume fraction of S22 microballoons.

Table 15. Strength of K46 composite foams at 5% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s(^{-1}))</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Quasi-static</td>
<td></td>
</tr>
<tr>
<td></td>
<td>547.09</td>
<td>85±2</td>
</tr>
<tr>
<td>NSF4610-5</td>
<td>589.84</td>
<td>101±3</td>
</tr>
<tr>
<td></td>
<td>650.58</td>
<td>172±10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>217±3</td>
</tr>
<tr>
<td></td>
<td>Quasi-static</td>
<td></td>
</tr>
<tr>
<td>NSF4630-5</td>
<td>504.53</td>
<td>64±1</td>
</tr>
<tr>
<td></td>
<td>635.13</td>
<td>87±10</td>
</tr>
<tr>
<td></td>
<td>721.82</td>
<td>162±5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>197±7</td>
</tr>
<tr>
<td></td>
<td>Quasi-static</td>
<td></td>
</tr>
<tr>
<td>NSF4660-5</td>
<td>436.12</td>
<td>55±2</td>
</tr>
<tr>
<td></td>
<td>767.11</td>
<td>65±12</td>
</tr>
<tr>
<td></td>
<td>949.61</td>
<td>71±5</td>
</tr>
<tr>
<td></td>
<td></td>
<td>84±7</td>
</tr>
</tbody>
</table>
Figure 38. Strength vs. nanoclay volume fraction for 10% K46 microballoons composite foams

Figure 39 shows strength vs. nanoclay volume fraction for nanoclay and plain syntactic foams having 30% volume fraction of K46 microballoons. It can also be observed that strength follows similar trend at different strain rates as in S22 composite foams having 30% volume fraction of microballoons. Similar to S22, the strength values at low strain rate (quasi-static) are higher than strength values at medium strain rate for plain syntactic foams and nanoclay syntactic foams having 1% volume fraction of nanoclay. However, the difference in strength values between medium and low strain rate is similar because of the presence of more amounts of microballoons in matrix.

The influence of nanoclay volume fraction also affects the strength values when specimens are subjected to same strain rate. However, it is the strain rate effect on strength values which is more evident. At high strain rate, strength increases due to addition of 1%
volume fraction of nanoclay. However, it decreases for further addition of nanoclay to 2 and 5% volume fraction. At 30% volume fraction of microballoons, the amount of matrix available for complete dispersion of nanoclay in matrix is reduced and it reduces further due to addition of 2 and 5% volume fraction of nanoclay. As mentioned earlier, higher nanoclay content causes inconsistencies in the matrix leading to reduction in strength at high strain rate. Thus the increase and decrease of strength at high strain rate is attributed to the crack propagation behavior and dispersion of nanoclay in matrix. At medium and low strain rates, the effect of nanoclay on strength of composite foams is not significant. However, there is a steady increase in strength values at medium strain rate. On the other hand, the strength at low strain rate increases for 1% addition of nanoclay and decreases for further addition of 2 and 5% nanoclay. This decrease in strength values compared to values at medium strain rate is expected.
Figure 40 shows strength vs. nanoclay volume fraction for nanoclay and plain syntactic foams having 60% volume fraction of K46 microballoons. From Figure 40 it can be observed that strength value is lower for low strain rate as compared to medium strain rate, which is an expected result. At high strain rate, the strength values are higher than at medium and low strain rate test. This is related to crack propagation in specimen as observed in SEM image in Figure 41.

![Graph showing strength vs. nanoclay volume fraction for 60% K46 microballoons composite foams](image)

Figure 40. Strength vs. nanoclay volume fraction for 60% K46 microballoons composite foams

Nanoclay volume fraction also affects the strength of composite foams when subjected to the same strain rate. It can be observed that strength of composite foam increases with an addition of 1% volume fraction of nanoclay. However, it reduces for addition of 2 and 5% volume fraction of nanoclay. This behavior depends on the dispersion of nanoclay in the matrix. Due to availability of less free volume of matrix, improper wetting of nanoclay takes place,
leading to agglomerations as observed in Figure 42. As mentioned earlier, these nanoclay agglomerations are responsible for reduction of strength of composite foams at 2 and 5% volume fraction.

Figure 41. SEM image showing crack propagation in NSF4660-1 at high strain rate test

Figure 42. TEM image showing nanoclay agglomeration

Figures 43-45 indicates the dependency of strength on the theoretical density of composite foams. As compared to S22 composite foams, strength values of composite foams
Figure 43. Density effect on strength for 10% microballoons volume fraction composite foams

Figure 44. Density effect on strength for 30% microballoons volume fraction composite foams
Figure 45. Density effect on strength for 60% microballoons volume fraction composite foams having K46 microballoons are higher at all three strain rates and nanoclay volume fraction. This is because of the density of K46 microballoons. K46 microballoons have higher wall thickness as compared to S22 microballoons. Due to higher wall thickness, the crush strength and ultimate compressive load of K46 microballoons is higher. Thus the composite foams having K46 microballoons can sustain more loads. The effect of wall thickness is more prominent at high strain rate test where cracks propagate through microballoons. Cracks certainly require more force to damage K46 microballoons, thus increasing the strength of composite foams.

6.2 Modulus

The modulus values of composite foams having S22 microballoons obtained from dynamic and compressive tests are displayed in Tables 16-19. These tables show the average values of dynamic strength at three different strain rates along with quasi-static strength.
Table 16. Modulus of S22 composite foams at 0% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s(^{-1}))</th>
<th>Modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF2210</td>
<td>Quasi-static 409.09</td>
<td>2339±45</td>
</tr>
<tr>
<td></td>
<td>508.78</td>
<td>2864±23</td>
</tr>
<tr>
<td></td>
<td>591.34</td>
<td>3284±34</td>
</tr>
<tr>
<td></td>
<td></td>
<td>4205±46</td>
</tr>
<tr>
<td>SF2230</td>
<td>Quasi-static 524.78</td>
<td>1966±20</td>
</tr>
<tr>
<td></td>
<td>708.47</td>
<td>1803±34</td>
</tr>
<tr>
<td></td>
<td>820.3</td>
<td>2920±44</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3480±29</td>
</tr>
<tr>
<td>SF2260</td>
<td>Quasi-static 518.4</td>
<td>1429±33</td>
</tr>
<tr>
<td></td>
<td>729.13</td>
<td>1583±47</td>
</tr>
<tr>
<td></td>
<td>1067.98</td>
<td>2508±50</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3464±32</td>
</tr>
</tbody>
</table>

Table 17. Modulus of S22 composite foams at 1% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s(^{-1}))</th>
<th>Modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NSF2210-1</td>
<td>Quasi-static 431.22</td>
<td>3661±36</td>
</tr>
<tr>
<td></td>
<td>572.82</td>
<td>3012±56</td>
</tr>
<tr>
<td></td>
<td>653.96</td>
<td>3498±67</td>
</tr>
<tr>
<td></td>
<td></td>
<td>8919±89</td>
</tr>
<tr>
<td>NSF2230-1</td>
<td>Quasi-static 536.64</td>
<td>3002±80</td>
</tr>
<tr>
<td></td>
<td>726.43</td>
<td>2214±46</td>
</tr>
<tr>
<td></td>
<td>827.946</td>
<td>3162±54</td>
</tr>
<tr>
<td></td>
<td></td>
<td>8323±39</td>
</tr>
<tr>
<td>NSF2260-1</td>
<td>Quasi-static 507.8</td>
<td>2227±78</td>
</tr>
<tr>
<td></td>
<td>791.23</td>
<td>2135±35</td>
</tr>
<tr>
<td></td>
<td>966.17</td>
<td>2603.5±78</td>
</tr>
<tr>
<td></td>
<td></td>
<td>6359±48</td>
</tr>
</tbody>
</table>

Particular results of dynamic and quasi-static modulus for specimens having S22 microballoons are plotted in Figures 46-48 as a function of nanoclay volume fraction at high (approximately 800/s), medium (approximately 450/s) and low (quasi-static) strain rates.

From Figures 46-48, it can be observed that modulus values are higher in tests conducted at high strain rate than at medium and low strain rate tests. At high strain rate, cracks
Table 18. Modulus of S22 composite foams at 2% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s(^{-1}))</th>
<th>Modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NSF2210-2</td>
<td>Quasi-static 473.43</td>
<td>3532±90</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3335±96</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3933±45</td>
</tr>
<tr>
<td></td>
<td>585.85</td>
<td>9516±86</td>
</tr>
<tr>
<td></td>
<td></td>
<td>8782±83</td>
</tr>
<tr>
<td>NSF2230-2</td>
<td>Quasi-static 476.12</td>
<td>3106±21</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2715±45</td>
</tr>
<tr>
<td></td>
<td>713.83</td>
<td>3467±79</td>
</tr>
<tr>
<td></td>
<td>837.67</td>
<td>8782±83</td>
</tr>
<tr>
<td>NSF2260-2</td>
<td>Quasi-static 562.6</td>
<td>2032±54</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3470±39</td>
</tr>
<tr>
<td></td>
<td>600.64</td>
<td>3031±50</td>
</tr>
<tr>
<td></td>
<td>690.39</td>
<td>7908±81</td>
</tr>
</tbody>
</table>

Table 19. Modulus of S22 composite foams at 5% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s(^{-1}))</th>
<th>Modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NSF2210-5</td>
<td>Quasi-static 450.29</td>
<td>3345±65</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3678±10</td>
</tr>
<tr>
<td></td>
<td>548.11</td>
<td>5548±53</td>
</tr>
<tr>
<td></td>
<td>563.23</td>
<td>12817±70</td>
</tr>
<tr>
<td>NSF2230-5</td>
<td>Quasi-static 562.6</td>
<td>2391±59</td>
</tr>
<tr>
<td></td>
<td></td>
<td>3054±80</td>
</tr>
<tr>
<td></td>
<td>600.64</td>
<td>5329±80</td>
</tr>
<tr>
<td></td>
<td>690.39</td>
<td>10696±80</td>
</tr>
<tr>
<td>NSF2260-5</td>
<td>Quasi-static 555.14</td>
<td>1556±45</td>
</tr>
<tr>
<td></td>
<td></td>
<td>2567±90</td>
</tr>
<tr>
<td></td>
<td>576.93</td>
<td>4067±60</td>
</tr>
<tr>
<td></td>
<td>749.14</td>
<td>9154±70</td>
</tr>
</tbody>
</table>

propagating indiscriminately encounter stiffer microballoons and nanoclay, resulting in an increase in the modulus of the composite foams. This effect is more clearly observed when 5% volume fraction of nanoclay is added in the composite foams. However, at medium and low strain rate tests, the modulus values do not increase significantly. It is expected that modulus of composite foam specimen subjected to medium strain rate test should be higher than at low strain
Figure 46. Modulus vs. nanoclay volume fraction for 10% S22 microballoons composite foams

Figure 47. Modulus vs. nanoclay volume fraction for 30% S22 microballoons composite foams
Figure 48. Modulus vs. nanoclay volume fraction for 60% S22 microballoons composite foams rate test. However, at 1% nanoclay volume fraction, modulus of composite foams at low strain rate is higher than medium strain rate. This is attributed to the fact that while cracks go through microballoons and nanoclay during medium strain rate tests, some cracks propagate along the weak interface between the matrix and microballoons or nanoclay. These cracks propagating through the interface weaken the composite foam. This weakening effect is balanced by the cracks propagating through stiffer microballoons and nanoclay. Therefore, the average stiffness at 1% nanoclay volume fraction and medium strain rate ends up similar or higher to quasi-static value where cracks tend to go only through the matrix.

The modulus of composite foams increase and decrease due to addition of 2% volume fraction of nanoclay at medium and low strain rate, respectively, at all three volume fractions of microballoons. The effect of strain rate on the modulus is obvious from the results of quasi-static and medium strain rate tests. The modulus values at medium strain rate are higher due to
the fact that cracks encounter more nanoclay than at 1% volume fraction. On the other hand, the quasi-static modulus reduces due to propagation of cracks through the increased number of weak interfaces between nanoclay and resin.

For addition of 5% volume fraction of nanoclay, modulus further increase at medium strain rate and decrease at low strain rate, for 10 and 30% volume fraction of microballoons. However, for 60% volume fraction of microballoons, the modulus decreases even at medium strain rate test. This is due to the increasing amount of nanoclay in the matrix. As mentioned earlier, the amount of matrix is low at 60% volume fraction of microballoons and it reduces further due to addition of nanoclay. Shortage of matrix leads to improper wetting of nanoclay causing agglomerations. Also, higher nanoclay content increases the stress concentration areas in the matrix. Cracks propagating through these areas ultimately damage the nanoclay syntactic foams resulting in reduced modulus values.

Tables 20-23 display the dynamic and quasi-static compressive modulus for composite foams having K46 microballoons.

Table 20. Modulus of K46 composite foams at 0% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s(^{-1}))</th>
<th>Modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SF4610</td>
<td>Quasi-static 454.23</td>
<td>3719±80</td>
</tr>
<tr>
<td></td>
<td>519.56</td>
<td>2668±39</td>
</tr>
<tr>
<td></td>
<td>601.34</td>
<td>4545±69</td>
</tr>
<tr>
<td>SF4630</td>
<td>Quasi-static 441.54</td>
<td>2508±45</td>
</tr>
<tr>
<td></td>
<td>605.03</td>
<td>2856±57</td>
</tr>
<tr>
<td></td>
<td>694.37</td>
<td>4552±31</td>
</tr>
<tr>
<td>SF4660</td>
<td>Quasi-static 483.52</td>
<td>2260±33</td>
</tr>
<tr>
<td></td>
<td>624.67</td>
<td>2196±21</td>
</tr>
<tr>
<td></td>
<td>722.56</td>
<td>3000±33</td>
</tr>
</tbody>
</table>
Figures 49-51 show modulus vs. nanoclay volume fraction for nanoclay and plain syntactic foams having 10, 30 and 60% volume fraction of K46 microballoons, respectively. It can be observed that the behavior of K46 composite foams for modulus values under various strain rates is similar to composite foams having S22 microballoons. Thus, similar reasons apply for modulus values of composite foams having K46 microballoons for all three strain rates and nanoclay and microballoon volume fractions.

Table 21. Modulus of K46 composite foams at 1% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s⁻¹)</th>
<th>Modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NSF4610-1</td>
<td>Quasi-static 500.86 552.2 574.28</td>
<td>3848±90 3336±90 6292±66 9590±55</td>
</tr>
<tr>
<td>NSF4630-1</td>
<td>Quasi-static 418.43 534.97 547.59</td>
<td>3807±40 2974±33 6050±45 9305±44</td>
</tr>
<tr>
<td>NSF4660-1</td>
<td>Quasi-static 573.71 656.35 766.55</td>
<td>3272±56 2637±49 5453±76 8386±77</td>
</tr>
</tbody>
</table>

Table 22. Modulus of K46 composite foams at 2% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s⁻¹)</th>
<th>Modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>NSF4610-2</td>
<td>Quasi-static 522.87 559.66 655.2</td>
<td>3626±39 3880±66 6955±60 10105±88</td>
</tr>
<tr>
<td>NSF4630-2</td>
<td>Quasi-static 478.12 573.85 712.9</td>
<td>3490±29 3288±6 7261±50 11369±21</td>
</tr>
<tr>
<td>NSF4660-2</td>
<td>Quasi-static 591.52 644.21 793.96</td>
<td>3117±36 2910±54 6827±54 9465±70</td>
</tr>
</tbody>
</table>
Table 23. Modulus of K46 composite foams at 5% nanoclay volume fraction

<table>
<thead>
<tr>
<th>Foam Type</th>
<th>Strain Rate (s⁻¹)</th>
<th>Modulus (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Quasi-static</td>
<td></td>
</tr>
<tr>
<td>NSF4610-5</td>
<td>547.09</td>
<td>3539±49</td>
</tr>
<tr>
<td></td>
<td>589.84</td>
<td>6606.5</td>
</tr>
<tr>
<td></td>
<td>650.58</td>
<td>9008±25</td>
</tr>
<tr>
<td></td>
<td></td>
<td>12998±90</td>
</tr>
<tr>
<td>NSF4630-5</td>
<td>504.53</td>
<td>2984±66</td>
</tr>
<tr>
<td></td>
<td>635.13</td>
<td>3779±38</td>
</tr>
<tr>
<td></td>
<td>721.82</td>
<td>8317±77</td>
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<tr>
<td></td>
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<td>12156±28</td>
</tr>
<tr>
<td>NSF4660-5</td>
<td>436.12</td>
<td>2712±38</td>
</tr>
<tr>
<td></td>
<td>767.11</td>
<td>3350±50</td>
</tr>
<tr>
<td></td>
<td>949.61</td>
<td>6959±78</td>
</tr>
<tr>
<td></td>
<td></td>
<td>10011±89</td>
</tr>
</tbody>
</table>

Figure 49. Modulus vs. nanoclay volume fraction for 10% K46 microballoons composite foams

Figures 52-54 indicates the dependency of modulus on density of composite foams. It can be observed that modulus of composite foams having K46 microballoons is higher than
Figure 50. Modulus vs. nanoclay volume fraction for 30% K46 microballoons composite foams

Figure 51. Modulus vs. nanoclay volume fraction for 60% K46 microballoons composite foams
Figure 52. Density effect on modulus for 10% microballoons volume fraction composite foams

Figure 53. Density effect on modulus for 30% microballoons volume fraction composite foams
Figure 54. Density effect on modulus for 60% microballoons volume fraction composite foams composite foams having S22 microballoons for at all three strain rates and for different volume fractions of nanoclay and microballoon. The reason for this behavior is the density of microballoons. K46 microballoons have higher density due to higher wall thickness as compared to S22 microballoons. As wall thickness of microballoons increases, the stiffness also increases [28] leading to an increase in modulus values for thicker walled K46 microballoons.

6.3 Energy Absorbed

Figures 55-57 show the stress vs. strain behavior at high strain rate for composite foams having 10, 30 and 60% volume fraction of S22 microballoons, respectively. The area under the stress-strain curve represents the energy absorbed per unit volume. The energy absorbed is the energy required for initiation and propagation of cracks in the specimen.

From Figure 55 and Figure 56, it can be observed that the energy absorption increases with an addition of 5% volume fraction of nanoclay to plain syntactic foam having 10 and 30%
Figure 55. Stress vs. strain behavior for 10% S22 microballoons composite foams at 800/s

Figure 56. Stress vs. strain behavior for 30% S22 microballoons composite foams at 800/s
volume fraction of microballoons. However, the trend reverses for 60% microballoon volume fraction as shown in Figure 57. At 10 and 30% microballoon volume fraction composite foams, nanoclay is relatively well dispersed in the matrix and propagating crack encounters more amounts of stiffer nanoclay. At 60% microballoon volume fraction, achieving uniform dispersion of nanoclay is difficult due to less amount of the matrix, leading to agglomeration of nanoclay bringing about the weakness in the composite as mentioned earlier. Therefore, cracks require less energy for propagation.

From Figure 58 it can be observed that the energy absorption behavior of composite foams having K46 microballoons is similar to composite foams having S22 microballoons. Thus similar reasons can be stated for energy absorption behavior of K46 composite foams.

The microballoon wall thickness effect on energy absorption is also clearly visible from Figure 59. Figure 59 displays a particular stress vs. strain plot for S22 and K46 types of
Figure 58. Stress vs. strain behavior for 10% K46 microballoons composite foams at 800/s

Figure 59. Effect of microballoon wall thickness on dynamic properties
composite foams having 60% and 1% volume fraction of microballoons and nanoclay, respectively. The effect of microballoon wall thickness is significant. When K46 microballoons are used instead of S22 microballoons, a doubling effect on the strain energy is observed. This is an indication of the significant energy required to break the thicker microballoons. Thus it can be concluded that the energy absorption in K46 microballoons reinforced composite foams is higher as compared to composite foams having S22 microballoons.
CHAPTER 7. CONCLUSIONS

7.1 Conclusions

In this research, high strain rate properties of nanoclay syntactic foams are studied. Nanoclay syntactic foams are fabricated using the ultrasonic mixing technique. For comparison purpose, plain syntactic foams are also fabricated without the addition of nanoclay. Transmission electron microscopy is performed to observe the dispersion of nanoclay in nanoclay syntactic foams. High strain rate test and quasi-static compressive test are performed on plain and nanoclay syntactic foams using SHPB and MTS-810 machine, respectively. Scanning electron microscopy is performed to study the crack propagation behavior under different strain rates.

Strength and modulus of both types of composite foams is calculated using SHPB data and the load-deflection curves obtained during high strain rate and quasi-static tests. It is found that strength and modulus of all types of syntactic foam composites is found to be directly affected by strain rate. Composite foams subjected to high strain rate showed higher strength and modulus as compared to those subjected to lower strain rates. SEM image showed that at lower strain rate test, the speed of crack is slow and follow a preferential path. However, in high strain rate test, cracks indiscriminately propagate faster in the specimen encountering a relatively large amount of microballoons and nanoclay. The volume fraction of the nanoclay is also found to affect the composite foam strength and modulus when subjected to same strain rate. It is found that with an addition of 1% volume fraction of nanoclay, optimum enhancement in strength and modulus of composite foams is obtained. However this effect depends on the amount of microballoons present in the matrix. The nanoclay is found to disperse more efficiently at lower microballoon volume fraction than at higher microballoon volume fraction. This led to higher probability of nanoclay encounter by cracks propagating through the
specimen, thus increasing the mechanical properties. On the other hand, at higher microballoon volume fractions, stress concentrations and voids formed due to less amount of matrix and lack of uniform dispersion of nanoclay reduced the strength of the composites at all strain rates. In addition, the interaction between agglomerated nanoclay and microballoons has led to premature failure of the microballoons due to point load conditions at the outer surface of the microballoons.

The wall thickness of microballoons also affected strength, modulus and energy absorbed values of composite foams. Composites foams fabricated with K46 microballoons showed higher strength, modulus and energy absorbed as compared to composite foams having S22 microballoons at strain rates.

7.2 Future Work

In this research, nanoclay could not be dispersed efficiently using ultrasonic mixing technique alone at higher microballoon volume fraction due to less free volume of matrix available. In future, a better method should be adopted to efficiently disperse nanoclay in the matrix. Acetone could be used to disperse nanoclay under ultrasonic mixer and 3-roll mixing could also be performed. Affect of hygrothermal degradation on high strain rate properties can also be studied.
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VITA

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