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Characterization of advanced composites - a nondestructive approach

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CHARACTERIZATION OF ADVANCED COMPOSITES- A NONDESTRUCTIVE APPROACH

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

by

Phani Surya Kiran Mylavarapu
M.S., University of Missouri Kansas City, 2003.

December 2007
This dissertation is dedicated

to my parents Shri Mylavarapu Siva Rama Krishna

and Smt. Late Mrs. Gayatri

for their endless love, cooperation and encouragement throughout my life.
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ABSTRACT

Adhesively bonded sandwich structures comprising of particulate composites as core and graphite epoxy skins as stiffeners are widely used for various applications in the marine and aerospace industry. The core material and the stiffener are held together by an adhesive bond. Particulate composites are made from a mixture of a polymer resin and hollow or solid particles. Hollow particulate composites are known as syntactic foams. Particulate composites possess attractive mechanical and physical properties such as high compressive strength etc, making them attractive materials for use in structural applications.

Characterization of the adhesive bondline and core material in sandwich structures is important for ensuring structural stability and reliability. Nondestructive evaluation [NDE] techniques such as ultrasound are used for better evaluation of these sandwich structured materials.

The present study addresses the problems of detection of disbonds, bond surface characteristics and porosity in the adhesive panels along with characterization of particulate composites separately using NDE. The importance of the attenuation coefficient in computing the longitudinal velocities of the ultrasonic wave in particulate composite samples is also discussed. Five sets of adhesively bonded carbon epoxy composite specimens with varying bond surface preparation, twenty four different types of hollow syntactic foams and six different types of solid particulate composites, are fabricated. The adhesively bonded panels are made by including known defects in the bond layer of the samples. The particulate composites (syntactic foams and solid particulates) are fabricated by varying the volume fraction of each of the four types of microballoons and solid particle from 10% to 60%. Pulse echo UI method is selected for use in the present work. The results of this research provides a better understanding of
adhesive joints and particulate composites and thus help in characterizing structures composed of these constituents.

One of the major findings in this research is the discovery of a nondestructive method to determine the dynamic modulus of particulate composites. In addition, a constitutive model explaining the effect of particle size, porosity, radius ratio on the ultrasonic attenuation coefficient in particulate composites is developed.
1. INTRODUCTION

Modern day structural applications demand materials with a specific set of properties. It is usually impossible to achieve all the required properties using a single material. Hence new materials are fabricated by combining two or three different types of materials. Composite materials are developed by the combination of two or more materials which have superior properties than the individual constituents. According to ASM hand book, a composite is a “macroscopic combination of two or more distinct materials, having a recognizable interface between them” [ASM Handbook, 2003]. Other definitions include “custom blending of materials with distinct characteristics lead to composites with tailor-made properties” [Composites, 2004b].

Some of the primary advantages of composite materials are high strength to weight ratio, high bending stiffness, corrosion resistance, excellent fatigue characteristics (comparable to metals) and good thermal insulation properties. The distinct advantage of composite materials is that the properties can be tailored according to the application requirements in the form of directional and spatial properties. Currently, the primary areas of application of composite materials are aerospace industry, automobile industry, ship building industry and sports equipment. The primary reason for this wide range of applications is the requirement of high strength to weight ratio for these industries. Composite structures are widely used in the industry in forms such as truss, bridge, sandwich structures etc. Even though this research does not deal with the adhesive layer and foam (core) together as a sandwich structure, an introduction of sandwich composites is necessary in order to understand the importance of the constituents. Moreover, an understanding of nondestructive characterization of the constituents will lead to a nondestructive testing of sandwich structures.
1.1 Sandwich Composites

Sandwich composites are a special class of composite materials which are widely used because of their high specific strength and bending stiffness. Low density of these materials makes them well suited for marine and aerospace applications. The concept of sandwich structure composite materials can be traced back to as early as the year 1849 AD [1] but the potential of such a construction was realized only during the Second World War. Developments in aviation posed the need for lightweight, high strength and highly damage tolerant materials. Sandwich structured composites, fulfilling these requirements became the first choice for many applications including structural components. Now their structural applications spread even to the ground transport and marine vessels.

Adhesively bonded sandwich structures are primarily comprised of a low density core along with two thin but stiff face sheets attached on either side using an adhesive. The thickness of the adhesive layer is generally neglected as it is much smaller than the thickness of the skins or the core. Various other types of sandwich structures can also be fabricated by varying the designs but the key factor in these materials is the low density core and thin stiffeners. The properties of sandwich composites depend upon properties of the core and skins, their relative thickness and the bonding characteristics between them.

1.1.1 Core

In sandwich structures, core materials are selected depending upon the performance requirements [2]. Core materials can be broadly divided into three classes as described below:

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

High-density materials used for the purpose of making expanded core include aluminum, titanium and various polymers. The interfacial contact area between skins and core is mainly affected by the structure of the core material. Expanded high density materials normally provide much smaller contact area compared to the solid low density materials. The choice of appropriate structure for core provides additional parameter to design a sandwich composite as per given specifications or service conditions. The use of cores like closed cell structured foam gives some distinct advantages over open cell structured foams and cores. Some of the advantages are:

1. The specific compressive strength of close cell structured foams is much higher than the open cell structured foams, and
2. Closed cell structural foams absorb less moisture than open cell structured foam.

1.1.2 Skins

Metals such as aluminum, titanium, steel and fiber reinforced plastics are some of the common examples of skin materials. In case of fiber reinforced skins, the material properties can be controlled directionally in order to tailor the properties of the sandwich composite. Fiber reinforced polymers are widely used as skins due to their low density and high specific strength. Another advantage offered by the use of polymer composite skins is that the same polymer can be used to make the skin and the core. Cross-linking of polymers between core and skin would provide adhesion strength level equal to the strength of the polymer. This provides possibility of making the skin an integral part of the structure eliminating the requirement of the adhesive. When an adhesive is used to bond the skin and the core together, selection of adhesives becomes important to account for its compatibility with both skin and the core materials. The adhesion must also have the desired strength level and should remain unaffected by the working environment.
Choice of skins is important from the point of view of the work environment as this part of the structure comes in direct contact with the environment. Corrosion, heat transfer characteristics, thermal expansion characteristics, moisture absorption and other properties of the whole sandwich composite can be controlled by proper choice of skin material. In most cases, even though both skins of the sandwich are of the same type, there could be differences such as materials, thickness, fiber orientation, fiber volume fraction or in any other possible form depending upon specific requirements.

1.1.3 Properties of Sandwich Composites

The main advantage of any type of composite material is the possibility of tailoring its properties in accordance with the application. The same advantage also applies to sandwich composites. Proper choice of core and skins makes sandwich composites adaptive to a large number of applications and environmental conditions. Some general characteristics of sandwich composites are described below:

1. Low density: Choice of lightweight core or expanded structures of high-density materials decrease the overall density of the sandwich composite. Volume of core is considerably higher in the sandwich composite compared to the volume of skins so any decrease in the density of the core material has significant effect on the overall sandwich density.

2. Bending stiffness: This property comes from the skin part of the sandwich. Due to a higher specific stiffness, sandwich composites result in lower lateral deformation, higher buckling resistance and higher natural frequencies compared to other structures.

3. Damage tolerance: Use of flexible foam or crushable material as core makes sandwich materials highly damage tolerant. For this reason foam core or
corrugated core sandwich structure materials are popular materials in packaging applications.

1.1.4 Advantages of Sandwich Composites

The following are some of the advantages of sandwich composites:

1. Tailoring of properties according to requirements,
2. Large available choice of constituents for core and skins,
3. Low density leading to saving of weight,
4. High bending stiffness,
5. Higher damage tolerance,
6. In-situ fabrication, and
7. Good vibration damping capacity.

1.1.5 Limitations of Sandwich Composites

Some of the limitations of sandwich structures are given below. These limitations can be overcome by developing new materials and manufacturing methods.

1. Higher thickness of the sandwich composites,
2. Higher cost of sandwich composites compared to conventional materials,
3. Processing is expensive,
4. Difficult to join, and
5. Difficult to repair, if damaged.

1.1.6 Applications of Sandwich Composites

Sandwich structures are widely used in various applications that require materials of low density, high strength and high damage tolerance. Some of the main areas of applications of sandwich composites are listed below:

1. In structural applications of aircrafts, spacecrafts, submarines, ships and boats,
surface transport vehicles, building materials, etc.,

2. Packaging materials,

3. Thermal and electrical insulation, and

4. Storage tanks.

1.2 Particulate Composites

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

1.2.1 Filler Materials

A variety of particles are used as fillers in composites [3]. Usage of fillers in particulate composites vary depending upon the advantages expected such as reducing the cost of expensive polymeric components, modification in strength, magnetic, electrical or fire retardant properties and change in density. Several materials such as particles of minerals, metals, ceramics, polymers and also some industrial wastes can be selected as fillers for the polymers [4]. Some common examples of filler materials are particles of alumina, silica, hollow and solid particles of glass, wood chips, fly-ash and carbon black. Selection of materials is mainly based on the desired properties of the composite.

Properties of particulate composites are primarily dependent upon the shape of the filler particles. Particles are normally classified based on their shapes into spherical,
cubical, blocks, flaky and fibrous types. The surface area of particles for the same volume differs from shape to shape, thus affecting the size of the interfacial region between the particle and the matrix resin. For each of these shapes the stress concentration factor would be different from particle to particle due to their different corner radius of curvatures and aspect ratios. Spherical particulate fillers are more popular compared to the other types.

Use of hollow particles, known as microballoons, has increased considerably in recent years in the production of core materials of low density and high damage tolerance. Such low density materials are classified as close cell structured foams and are known as—“syntactic foams”. Density of syntactic foams can be modified either by changing the wall thickness of the microballoons or by changing the volume fraction of the hollow microballoons in the matrix.

### 1.2.2 Syntactic Foams

Syntactic foams are known for their high specific compressive strength, low moisture absorption and excellent damping properties. They are used as core materials in sandwich composites for weight sensitive structural applications. Syntactic foams are multi-functional composite materials due to their broad range of mechanical properties coupled with vibration damping characteristics, and ability to be fabricated in functionally graded configurations. These materials were developed in the 1960s as buoyancy aid materials for deep sea applications [5]. Presently they are used in aircraft, spacecraft and ship structures [6].

One of the major advantages of syntactic foams is their ability to be designed and fabricated according to the physical and mechanical property requirements of the application. Depending upon the service conditions, the matrix resin can be chosen from a wide range of thermosetting and thermoplastic resins. Similarly, microballoons of
polymer, ceramic or metal can be chosen [7-8]. Another parameter that can be adjusted is the density of syntactic foams.

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

1.2.3 Structure of Syntactic Foams

Syntactic foams have two phases in their structure, namely matrix resin and microballoons. Structure of syntactic foam can be observed in the scanning electron micrograph presented in Figure 1. The micrograph shown in this figure is taken from the as-cut surface of syntactic foam specimen. Microballoons embedded in the matrix resin are visible in the structure. During the fabrication of syntactic foams some air is inevitably trapped in the structure and is present as open cell structured porosity. This entrapped air, termed as “voids“, makes syntactic foams three phase materials. In the present work the fabricated syntactic foams have three phase structures.

Several studies have been conducted on the use of microballoons made from different materials such as steel, aluminum and glass. However, glass microballoons have emerged as the most attractive alternative for use as fillers in particulate composites because of their high strength and low density.
Figure 1: Surface of syntactic foam specimen.

Syntactic foams containing glass microballoons possess attractive mechanical and physical properties such as high compressive strength, low moisture absorption and low coefficient of thermal expansion, making them an attractive material for use in aerospace and marine applications. Various types of destructive and nondestructive techniques are used for quality control of these structures. A brief introduction of these nondestructive techniques is given below.

1.3 Nondestructive Testing [NDT]

Nondestructive testing is exclusively used in the industry for characterizing different materials such as metals, nonmetals, ceramics, etc. Some of the nondestructive techniques which are used in the industry are Ultrasonic Testing, Acoustic Emission, X-ray Radiation, Thermography, Vibration Testing, Liquid Penetrant, Magnetic Particle and Eddy Current. Liquid Penetrant and Magnetic Particle testing account for about one-half of all NDT, Ultrasonics and X-ray methods about another third, Eddy Current testing about 10%, and all other methods for only about 2% of the many different NDT techniques used in industry [9]. Ultrasonic inspection is the main method used in this study.
1.3.1 Ultrasonic Inspection

Ultrasonic Inspection uses high frequency elastic waves to nondestructively inspect manufactured materials [10]. This technique is used for discontinuity detection, thickness measurement, determination of elastic moduli, study of metallurgical structure, and evaluation of the effect of processing variables on the component. The following are the advantages and disadvantages of ultrasonic testing.

The advantages of Ultrasonic Inspection are:

1. High sensitivity, permitting detection of minute discontinuities,
2. Good penetrating power, allowing examination of extremely thick sections,
3. Accuracy in the measurement of discontinuity position and estimation of discontinuity size,
4. Fast response, permitting rapid and automated testing, and
5. Need for access to only one surface of the test object.

The disadvantages of Ultrasonic Inspection are:

1. Unfavorable test object geometry (size, contour, surface roughness, complexity, and discontinuity orientation), and
2. Undesirable internal structure (grain size, structure porosity, inclusion content or fine, and dispersed precipitates).

In an ultrasonic nondestructive examination of any material, the ultrasonic wave propagates through the entire body. Due to this reason, a qualitative analysis of volume of the object, further followed by calculation of material properties using ultrasonic imaging parameters is possible using ultrasonic NDT. Ultrasonic test is typically performed in two ways. A beam of ultrasonic energy is directed into the test object and one of the following processes happen,

1. The energy transmitted through the object is measured, or
2. The energy reflected from discontinuities in the object is measured. The first process is the basis of the technique known as through-transmission technique while the second process is the method used in pulse-echo method.

1.3.4 Ultrasonic Scanning Techniques

Immersion systems can be used with three forms of ultrasonic scanning: the A-scan, B-scan or C-scan.

A-Scan: The ultrasonic A-scan presents one-dimensional data showing the response along the beam path at a specific location of the test object. These scans produce detailed information about discontinuities in the scanned material. The size of discontinuities can be estimated from the amplitude of the reflected signal.

B-Scan: In the ultrasonic B-scan, the test object is scanned along one axis to produce a presentation of its cross section. The location along the scanning path is shown on the X axis and time of flight values is shown along the Y axis. Because a cross section is produced, the B-scan is less practical for nondestructive testing where large volumes of material must be inspected.

C-Scan: The ultrasonic C-scan is applied to the test object in a raster pattern and presents a view of the discontinuity’s area as seen from above. Discontinuity location and size data are available from changes in amplitude as a function of position. Modern C-scan systems use computers to control the transducer position and to acquire, display, document and store the test results. The computer synchronously acquires the digitized position of the transducer and the associated value of a specific ultrasonic parameter.

Because of memory limits, most computerized C-scan systems acquire only one or two ultrasonic parameters as a function of position. In most cases, the parameter is time of flight or the amplitude of reflection, or transmission at a certain time range. The parameter is digitized with the aid of an analog to digital converter.
1.3.6 Ultrasonic Measurement Parameters

Computation of ultrasonic velocity and attenuation (loss of intensity due to interactions with material microstructure) are the key factors in ultrasonic determination of material properties. These ultrasonic measurements are computed using RF wave form signals rather than the actual mechanical waves in the material. RF wave form signals are obtained using probes or transducers coupled to the material sample surface.

1.3.6.1 Ultrasonic Velocity

The most frequent application of ultrasonics to material property measurement involves the study of elastic constants and related strength properties. According to physical acoustics theory, the elastic behavior of solids can be determined by measurements of ultrasonic wave velocity [10]. Longitudinal \(V_L\) and shear \(V_s\) wave velocities are used to compute the longitudinal \(L\) and shear \(G\) moduli, respectively, where,

\[
L = \rho * V_i^2 \quad [1]
\]

\[
G = \rho * V_s^2 \quad [2]
\]

For linear elastic, isotropic solids computation of longitudinal and shear moduli is sufficient for defining the complete elastic behavior, using interconnecting relations with other moduli, such as bulk modulus\(k\), Young’s modulus\(E\) and Poisson’s ratio\(\nu\) as shown in Equations 3-5.

\[
k = L - \frac{4}{3} * G \quad [3]
\]

\[
E = \frac{G(3L - 4G)}{L - G} \quad [4]
\]

\[
\nu = \frac{L - 2G}{2(L - G)} \quad [5]
\]
Defining the complete elastic behavior in anisotropic solids is complicated due to the reason that the principal moduli $L$ and $G$ assume different values according to the direction of ultrasound propagation. Therefore, the elastic characterization of an anisotropic material will depend upon nine independent longitudinal and shear wave velocity measurements, in three mutually perpendicular directions. For the special case of transversely isotropic solids, e.g. unidirectional fiber composites, five independent velocity measurements will be sufficient [11].

Neither $V_\ell$ nor $V_s$ can be measured unambiguously as a unique quantity except in the case of a “nondispersive” material. A medium can be dispersive or attenuative because of its geometric boundaries or internal constituents or both. Thus, a proper understanding of attenuation in the material under study is also important for material characterization.

1.3.6.2 Attenuation

According to American Society for Non Destructive Testing (ASNT), attenuation is defined as a “loss or decrease in energy or signal amplitude in transmission from one point to another”. Attenuation is caused by scattering, reflection and true absorption of ultrasonic waves by the interfaces in the material [12]. The scattering is due to the inhomogeneity of the material, i.e. the acoustic impedance mismatch between two interfaces having different sound velocities or densities. Absorption is due to the conversion of sound energy into heat. The absorption factor in attenuation increases with frequency of the transducer. Reflection of ultrasonic signals is caused by the discontinuities in the material as well as by the couplant used in contact and non contact testing. For example, water as a coupling medium in immersion testing distorts transmitted signals at high frequencies. Due to the distortion of transmitted signals, ultrasonic wave is significantly attenuated and the peak frequency of a broad band signal is downshifted.
Hence, attenuation coefficient should be calculated by considering the effects of reflection, scattering and absorption in the material. By neglecting losses due to reflection, attenuation can be expressed as shown in Equation 6:

\[ A = A_0 \cdot e^{-\alpha \cdot X} \]  

where:

\( X \) = propagation distance [meters],

\( \alpha \) = frequency dependent amplitude attenuation coefficient of the medium [neper.m\(^{-1}\)],

\( A_0 \) = unattenuated amplitude, and

\( A \) = attenuated amplitude.

Accounting for ultrasonic attenuation effects in materials is important due to the reason that the signal amplitude reduced by attenuation can affect the quality of the image produced and thereby affecting the quality of results. By knowing the attenuation that an ultrasound beam experiences traveling through a medium, one can either adjust the input signal amplitude to compensate for any loss of energy at the desired imaging depth or perform necessary corrections in calculations. Corrections in calculations can be in the form of picking up the right front and back wall reflections for calculating longitudinal velocity. For example, as shown in Figure 2, the locations of back wall reflections move back and forth in the time domain due to attenuation in the material. This alteration in time domain of the waveforms will affect the velocity values and thus finally affecting the quality of the results. In order to further understand the importance of attenuation, an example can be that due to attenuation, the C-scan obtained at a higher frequency can display an image showing severe porosity in the sample, whereas C-scan obtained at a lower frequency on the same sample show an image free of porosity. This discrepancy in C-scan display is due to an attenuation increasing with an increase in frequency of the
transducer. This could lead to rejecting a structurally sound material or accepting a deficient material.

As shown in Figure 3 [13], proper selection of transducer, frequency, and focus are required for composite inspection for studying various flaws that are expected during manufacture. Even though a higher frequency transducer can give better resolution, the quality of the results have to be sacrificed due to higher attenuation compared to a lower frequency transducer.

Figure 2: Shift of peaks due to attenuation in particulate composites.

With increasing use of these composite structures in aerospace and marine applications, there is a need to optimize existing nondestructive evaluation [NDE] techniques such as ultrasound, for better evaluation of these materials. Ultrasonic imaging has emerged as the most promising of all NDE technologies because of its high sensitivity and accuracy in determining cracks, defects and physical properties in a structure, and its simplicity of use, ease of application, and cost effectiveness.

As in this study, knowledge of attenuation coefficient is required for characterizing syntactic foams and other porous materials using Ultrasonic Imaging [UI] technique.
Hence, understanding the relation between porosity content of the material and attenuation coefficient will help in characterizing these materials. Porosity content in composite materials can be calculated using attenuation values of ultrasonic signals in a specimen.

In this research pulse echo ultrasonic imaging is used for characterization of syntactic foams and adhesive bonds separately, which are the primary constituents of a sandwich structure. Hollow glass particles with varying internal radius are used to fabricate syntactic foams. The volume fractions in these slabs are varied from 0-60%. Attenuation and velocity study is performed for characterizing foams. Further, more dynamic mechanical properties are predicted from the longitudinal and shear wave velocities computed from hollow particulate composites. Also the bond lines in CFRP are analyzed using ultrasonic imaging.

This report will start with a review of relevant literature that provide background and support to the research project. Thereafter, materials, specimen preparation, and test methods/equipment will be discussed. Finally, test results and conclusions will be presented. The chapter layout is as follows:

Chapter 1 gives a brief introduction to the topic of this research as well as background information on the use of particulate composites and quality control of particulate composites using nondestructive testing.

Chapter 2 reviews literature relevant to the topics of this research including: the different NDT methods available to perform ultrasonic testing on adhesively bonded structures or metal-metal and metal- composite type, followed by methods available to perform ultrasonic testing on particulate composites.

Chapter 3 focuses on the research objective used for accomplishing the research project, including fabrication and nondestructive characterization of adhesive bonds and particulate composites.
<table>
<thead>
<tr>
<th>Broad Areas of Use</th>
<th>Freq. (MHz)</th>
<th>Broad Areas of Use</th>
<th>Specific Applications</th>
</tr>
</thead>
<tbody>
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<td>Medical mammography, heart, fetal scans</td>
<td>1.0</td>
<td></td>
<td>Large forgings, welds and castings, nuclear pipe welds, pressure vessels</td>
</tr>
<tr>
<td>Medical: fragments in eyes, eye dimensions, blood vessel walls, burns</td>
<td>2.25</td>
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</tr>
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<td>Typical PAM SEAM, and SLAM operating ranges</td>
<td>3.5</td>
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<td>Industrial NDE: flaw detection, quality control, process evaluation and control, materials characterization of 3.0 mm to 0.1 mm resolution images</td>
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<td>Traffic: 4000.0</td>
<td></td>
<td></td>
<td>Porosity, cracks, structural ceramics diamond compacts, ceramic capacitors, ceramic IC packages semiconductor heat-sink bonds, laser welds IC die attachments</td>
</tr>
</tbody>
</table>

Figure 3: Description of frequencies of transducers and their applications.
Chapter 4 covers the interpretation and analysis of experimental results and discussions as well as the relevant insight and trends and phenomena observed from tests conducted on adhesively bonded specimens and particulate composites.

Chapter 5 includes the mathematical model derived for computation of ultrasonic attenuation coefficient in particulate composites.

Chapter 6 includes a summary of the project as well as relevant conclusions.
2. LITERATURE SURVEY

Adhesively bonded sandwich structures are widely used in aerospace and marine industry. Quality assurance of adhesive bonds and particulate composites that are used as the skin and core materials of adhesively bonded sandwich structures respectively is important for the structural stability of composite structures. Earlier, quality assurance techniques such as tapping method are used by Cawley and Adams [14-15] for characterizing adhesive joints. By examining the force input by the tapper to the adhesive joint, Cawley and Adams have found that the force input to a structure by a tap is affected by the quality of the bond at the joint. Even though tapping method resulted in a substantial improvement in the reliability of the technique, it is still limited to thin layers (less than or equal to 1mm) and the smallest detectable unbond is 10 mm in diameter in a typical test specimen. The quality of adhesion is critical to the performance of the adhesive as a bond between the components of an assembly [16]. Also, as the interface layer is often a fraction of micrometer thick, methods such as tapping are not useful for characterizing the adhesive bond.

Because of the above disadvantages of tapping method, quality assurance methods such as acoustics and ultrasonics are currently used as NDE methods for characterizing unbonds in adhesive joints. Of these methods, ultrasonic imaging is the most widely used method for characterizing adhesively bonded joints as it not only offers a means of defect detection, but also used to measure the physical properties of the material under inspection [17-19]. Several researchers used ultrasonic imaging as a tool for the quality assurance in adhesive bonds and particulate composites. Some of the important contributions are given next.
Alers and Elsley [20] have performed ultrasonic signal analysis on aluminum adherends bonded with a 6 mm layer adhesive and were able to deduce the physical properties of the adhesive and the adherend/adhesive interface. Yi et al. [21] have performed pulse echo ultrasonic method on Al6061-T6 specimens bonded with Cemedine 1500 adhesive. In their study, ultrasonic signals obtained from DCB specimens (of Al6061-T6 bonded with the adhesive) under stress with mode I fracture are used to evaluate parameters related to attenuation and amplitude variations. From the study of Yi et al., it was concluded that the thickness of the adhesively bonded layer should be considered for the strength evaluation of adhesively bonded material, as it greatly influences the ultrasonic wave evaluation.

Rokhlin and co-workers [22] have investigated the environmental degradation of metal-metal adhesive joints using leaky guided modes of the adhesive layer. They found correlations between certain features in the frequency spectrum and joint strength as measured by the lap shear test. Lavrentyev et al. [23] has also studied environmental damage initiation and evolution in metal-metal adhesive joints using ultrasonic angle beam technique. They found that the joint degradation is accompanied by a shift of the ultrasonic reflection spectrum minimum to a lower frequency. Weiss et al. [24] have as well performed ultrasonic time domain techniques such as normal incidence and oblique incidence along with spectral analysis on aluminum-aluminum adhesive joints and have found out that ultrasonics can be used for distinguishing different aluminum surface preparations along with the environmental degradation.

Moidu et al. [25] have investigated the durability of two commercial epoxy adhesives on metal adherends nondestructively using ultrasonic reflection measurements from the interfacial region. Freemantle and Challis [26] have subjected steel and aluminum substrates bonded with structural adhesives to high frequency ultrasonic longitudinal wave testing and have concluded that detection of front and back face
disbonds along with quantifying the substrate and bond-line thickness is possible. Borum [27] has performed ultrasonic testing of bonds on aluminum extruded profiles and has concluded that the evaluation of adhesion in these profiles is possible using ultrasonic pulse-echo technique.

Hanneman et al. [28] have proposed a novel technique of characterizing metal-metal adhesive joints. In this method, an exact solution was given for the problem of reflection and transmission of a plane, time-harmonic longitudinal wave through a layered medium. They have performed testing on 7475 aluminum adherends of equal thickness bonded by an aerospace film adhesive AF-163-2 and have concluded that there is an excellent agreement between their theoretical and experimental results [29]. Qu [30] has performed through transmission tests on Al 2024 bonded with FM-300 sheet adhesive to correlate the aging time of the bond joint with the generation of higher harmonics. He concluded that aging increases the magnitude of higher order harmonics.

Rose and Ditri have performed pulse echo [31] and through transmission lamb wave techniques on aluminum-to-aluminum adhesively bonded lap-joint and have concluded the feasibility of using lamb waves compared to the traditional ultrasonic technique. However, the knowledge of dispersion curves of the structure to be evaluated is essential in order to obtain proper accuracy in the lamb wave inspection. There is also a considerable difference in the signal quality between the good and bad bond in their study [32]. Dewen and Cawley [33] have developed a technique for the quantitative determination of the cohesive properties of adhesive joints based on the measurement of the reflection coefficient from the top adhesive/adherend interface and the bond line transit time. They performed the testing on aluminum adherends joined by an epoxy resin and have concluded that the method provides a reliable, nondestructive means of measuring the cohesive properties of a bonded joint. However, the frequencies involved in this process are considerably higher, thereby restricting them to the metal-metal joints.
Brotherhood et al. [34] have studied the detectability of dry contact kissing bonds in aluminum-aluminum adhesive joints using three ultrasonic imaging techniques namely the conventional longitudinal wave inspection, shear wave inspection and high power electro-magnetic acoustic transducer [EMAT] inspection. It was found that the high power technique showed the greatest sensitivity to kissing bonds at low contact pressure, however at high contact pressures, conventional longitudinal and shear wave testing are more sensitive.

Recently, continuous carbon fiber reinforced polymer (CFRP) composites form an important class of materials for aerospace structural applications. Joining CFRP composites using conventional techniques such as nuts and bolts or clamps and rivets is not feasible, because of the loss of strength of composite due to the damage on the main load-bearing component, i.e. the fiber. However, the use of adhesives does not cause fiber damage and thus adhesives are extensively used to bond composite materials to each other or with metallic parts. Voids, cracks, porosity also occur in CFRP composites, thus making their NDE examination difficult compared to metal-metal adhesive joints. Liu et al. [35] have performed ultrasonic NDE of CFRP composites using normal incident ultrasound waves.

Porosity estimation in composite materials using ultrasonic methods is also performed by Shark et al. [36] and Daniel et al. [37] using attenuation measurements. Hale et al. [38] and Martin [39] have developed models to study the ultrasonic attenuation due to voids in fiber reinforced composites. Even though several researchers have performed ultrasonic characterization of metal-metal adhesive bonds using different techniques, limited information is present on the ultrasonic characterization of composite-composite adhesive joints, thus making it an important problem in quality assurance of composite structures. Thus, in this research ultrasonic characterization of composite-
composite adhesive joints is performed and various problems associated with the characterization are discussed.

Particulate composites are used as core materials in composite structures for various applications and thus the quality assurance of these materials is also important. A number of theories currently existing in the available literature predict the ultrasonic behavior of random particulate composites [40]. Ying and Truell [41] have studied the scattering of a plane longitudinal wave by a spherical obstacle in an unbounded elastic matrix. In their model, they have given expressions for the scattered wave and the total scattering energy by an isotropically elastic sphere, a spherical cavity and a rigid sphere respectively. Sabina and Willis [42] used self-consistent scheme to develop the approximate analysis of waves in a matrix containing inclusions. In their approach, they have developed simple explicit equations for scattering from a single inclusion. The major disadvantage with the self consistent scheme is its difficulty in quantifying the results.

Datta [43] used the self-consistent scheme to analyze multiple scattering by elastic ellipsoidal inclusions. Kanuan, Levin and Sabina [44] have used various versions of effective medium method [EMM] to the solution of propagation of plane monochromatic wave through matrix composite materials with a random set of spherical inclusions. The major drawback of the EMM method is its failure in detecting foreign inclusions in the matrix. Multiple scattering of a plane wave by a random spherical distribution of particles is studied by Watermann et al. [45], and Lax [46]. Datta et al. [47] considered effective longitudinal and shear wave propagation through a medium containing a random distribution of spherical inclusions. They have assumed that the particles and matrix are separated by a thin layer of elastic material with different properties. Different models of Pb-epoxy and SiC-Al are tested using the iterative model. Their iterative solution underestimated attenuation in the composite and over estimated phase velocity at low frequencies.
Kinra and Ker [48] have performed through transmission ultrasonic scans on periodic particulate composites and have concluded that the dispersion curve is characterized by pass bands and stop bands. Kinra, Petraitis and Datta [49] have performed through transmission ultrasonic scans on randomly distributed spherical glass particles in an epoxy matrix in a frequency range of 0.3-5 MHz. They have found that the phenomenon of cut-off frequencies which is a characteristic of periodic particulate composites is not observed in random particulates. They have also found that the theoretical model [43] does not predict the actual phase velocities. Kinra et al [50], have subjected a particulate composite containing a layer of lead spherical inclusions with random or periodic arrangement embedded in a polyester matrix to normally incident plane longitudinal wave. They found that the most dominant feature of the transmission spectrum is the excitation of the rigid-body translation resonance for both the random and the periodic composites. In the case of periodic composites, the rigid body resonance is even more significant as all the spheres go in and out of resonance at the same frequency.

Kinra and Anand [51] have performed ultrasonic characterization of random particulate composites at both the long and short wavelengths and have concluded that the wave propagation is fairly non-dispersive at long and short wavelengths. Kinra, Ker and Datta [52] have performed ultrasonic characterization of random particulate composites having glass spheres in the intermediate wave length range between long and short wavelengths and have concluded that the wave propagation behavior is influenced by the particle excitations of the particle resonance. Beltzer, Bert and Striz [53] have analyzed wave propagation in random particulate viscoelastic composite. Their method incorporates both the scattering effect and viscoelastic losses as well as the Kramers-Kronig relationships.

Explicit expressions for the attenuation and dispersion are derived and compared with results from Kinra et al [49] and Kinra and Anand [51]. Szabo and Wu [54] have also modeled the longitudinal and shear wave propagation in viscoelastic media. Layman et al.
measured longitudinal, shear wave phase velocities and attenuation as a function of frequency for random particulate composites, consisting of spherical glass spheres embedded in an epoxy matrix. They compared experimental results with Watermann and Truell method [WT] [45] and dynamic generalized self-consistent method [DGSCM] [56]. They have found that the models match with the experimental results at low concentrations. However, at high concentrations, the results matched with the phase velocity computed from models. Gubernatis and Domany [57] have investigated the effects of microstructure statistics on the speed and attenuation of an elastic wave propagating through a porous material. In their research, Gubernatis and Domany found that the effective wave number is most sensitive to the most probable pore radius. Therefore, the research performed so far in the field of random particulate composites is more focused on solid particulate composites rather than syntactic foams and thus making the present study relevant.

Thus in this research, ultrasonic characterization of random particulate composites having either glass microballoons or solid glass spheres immersed in an elastic or visco-elastic matrix is performed. Effect of radius ratio on the ultrasonic longitudinal wave velocity, shear wave velocity and longitudinal attenuation through the particulate composites are also studied. A constitutive model showing the effect of radius ratio, particle size, volume fraction of particles on the attenuation coefficient of particulate composites is developed. Dynamic modulus and Poisson’s ratio are predicted on the particulate composite materials using longitudinal and shear wave velocities in the samples. In addition, ultrasonic characterization of composite-composite adhesive joints is performed in this research and various problems associated with the characterization are discussed.
3. RESEARCH OBJECTIVE

The objective of the present study is to characterize adhesive joints and random particulate composites separately using ultrasonic techniques. Adhesive joints are fabricated by joining two carbon fiber reinforced polymer (CFRP) laminates using adhesive. Solid glass spheres and hollow glass microballoons are used to fabricate random particulate composites by mixing in either an elastic or visco-elastic epoxy matrix.

3.1 Characterization of Adhesive Joints

Various problems associated with detection of disbonds and porosity in adhesively bonded carbon fiber reinforced composite panels is studied. Five sets of adhesively joined carbon-epoxy composites with different adherend surface preparations are fabricated and subjected to ultrasonic imaging. The panels contained known defects in the bondline of the samples. Normal incident ultrasonic scans in pulse-echo mode are used to characterize the adhesively bonded samples. Ultrasonic imaging (UI) results are interpreted to identify various existing defects such as voids, cracks and disbonds in the joints. Attenuation coefficient values for all types of composites are utilized to ascertain the validity of the image analysis.

3.2 Characterization of Random Particulate Syntactic Foams

Syntactic foams are characterized for studying the effect of radius ratio, density of microballoons, volume fraction of particles and particle size on the longitudinal and shear wave propagation. Hollow particulate syntactic foams are fabricated using four types of microballoons. Out of the four types of microballoons used, three of them have the same average outer diameter, but different inner diameters whereas the fourth microballoon has a slightly smaller outer diameter. In all the slabs, the volume fraction is varied from 10%
to 60% for each type of particles. Attenuation coefficient values are calculated using ASTM standard E664-93 for all the twenty four types of foams to check the wave dispersion in the composite. Longitudinal and shear wave velocities of hollow microballoon dispersed random particulate composites are used for computing the Poisson’s ratio and dynamic modulus using ultrasonic testing techniques. It is discovered that the modulus computed using ultrasonic longitudinal and shear wave velocity values in samples correspond to the dynamic modulus rather than the quasi-static modulus.

3.3 Characterization of Random Particulate Composites

Solid particulate composites are characterized for studying the effect of density of glass sphere and particle size on the longitudinal and shear wave propagation. Solid particulate composites are fabricated using one type of glass sphere, but different volume fractions. In all the slabs, the volume fraction is varied from 10% to 60%. Attenuation coefficient value are calculated using ASTM standard E664-93 for all the six type of solid particulate to check the wave dispersion in the composite. Longitudinal and shear wave velocities of solid particles dispersed random particulate composites are used for computing the Poisson’s ratio and dynamic modulus. It is shown that the modulus computed using ultrasonic longitudinal and shear wave velocity values correspond to the dynamic modulus rather than the quasi-static modulus.

3.4 Mathematical Modeling for Predicting Attenuation Coefficient in Particulates

A mathematical model for predicting the longitudinal attenuation coefficient in particulate composites is developed. This mathematical model shows the effect of radius ratio, void fraction, particle size and volume fraction of particles on the longitudinal attenuation coefficient of particulate composites. Loss of ultrasonic energy due to absorption and scattering by the particles are considered in the model. Effect of
interaction between particles on the ultrasonic signal distortion due to scattering and absorption is neglected in developing this model. It is shown that the mathematical model predicts the attenuation coefficient in particulates and syntactic foams of volume fractions ranging from 10 to 30%.
4. RESULTS AND DISCUSSIONS

Ultrasonic characterization of adhesive bonds and particulate composites is performed in order to understand the feasibility of detecting defects such as porosity in adhesive joints and core materials of particulate composites. Challenges in acquiring the ultrasonic signal from adhesive joints and particulates along with computation of ultrasonic velocities are discussed. Further, computation of dynamic modulus and Poisson’s ratio in particulate composites is performed using ultrasonic velocity values.

4.1 Ultrasonic Characterization of Adhesive Bonds

Characterization of adhesive bonds in composite materials using UI technique has deficiencies due to problems such as high acoustic attenuation and high signal-to-noise ratio. These problems can be attributed to the inhomogeneity in composite structures. The pulse-echo immersion ultrasonic technique has been used for characterization of adhesive joints in the present study. In the Ultrasonic Pulse-Echo technique, a wave traveling from one material to another will be partially reflected and partially transmitted through the interface as shown in Figure 4. The amplitude of each of the two generated components of the wave is determined by the degree of mismatch in the acoustic impedance between the two materials.

The reflection, $r$ and transmission, $t$ coefficients for the intensity of a wave incident normal to an interface between two half-spaces [17, 58-59] are given by

$$ r = \left( \frac{z_1 - z_2}{z_1 + z_2} \right)^2 $$

[7]

and,

$$ t = 1 - r $$

[8]

where, $z_1$ and $z_2$ are the acoustic impedances of the two materials, defined by the relation,

$$ z_j = \rho \cdot v $$

[9]
\( \rho \) is the density of the materials, and

\( v \) is the velocity of ultrasound in the materials. \( t \) and \( r \) represent the amount of transmission and reflection of the ultrasonic signal at the interface.

The larger the impedance mismatch between two materials, the higher will be the reflected amplitude, which is proportional to the reflection coefficient. Depending on the value of the reflection coefficient, it is easier to distinguish between a good and a poor bond. A weak bond will have a reflection coefficient of 1 and a relatively good bond will have a value between 0 and 1 [14]. Thus, in order to determine the amount of reflection and transmission of the ultrasonic signal at each interface and thereby to characterize the bondline, details about the velocity of ultrasonic wave and density of the interfacial layers are required.

\[
Z_1 = \rho_1 \times v_1 \\
Z_2 = \rho_2 \times v_2
\]

Figure 4: Pictorial depiction of reflected and transmitted waves from the interface.
4.1.1 Experimental: Materials and Methods

4.1.1.1 Materials

Adhesively joined composite specimens used in this study were supplied by Bell Helicopter, Fort Worth, TX, U.S.A. The samples were fabricated by joining two carbon fiber laminates with an epoxy-based adhesive. Each adhesively joined specimen had two CFRP laminates of equal thickness, 4.63 mm, joined by an epoxy-based adhesive. These specimens were approximately 10.65 mm thick and 254 mm long. The adhesive layers in these samples were fabricated with different surface properties in the bond layer to change the bond properties. Two samples of each of the five different types of panels, numbered 1 through 5, were used in this study. Two samples of adhesive joints are shown in Figure 5 in which the top panel was fabricated with a relatively good bondline and the bottom panel with a clearly visible defective bondline. Further investigation shows that the samples used in this study had varying amount of porosity.

![Figure 5: Two composite specimens showing good [top] and poor [bottom] bondlines.](image)

4.1.1.2 Ultrasonic Testing

A Pulse-Echo Ultrasound technique [31] was utilized to determine the bondline response of the bonded composite specimens to an ultrasonic wave. As shown in Figure 6
[60], radio frequency [RF] signal from the bondline will have a clear spike whereas the RF signal from the disbond will flatten out. A Physical Acoustic system [Physical Acoustics Corporation, U.S.A] of water immersion type, shown in Figure 7, was used for the ultrasonic testing. After the initial study of the signal, the data acquisition channels [gates] were set at the reflections corresponding to the adhesive layer as shown in Figure 8. The RF waveform from the bondline in Figure 8 appears to be saturated because of the high gain in the system. Initial tests utilizing 5 MHz frequency were carried out on the adhesively bonded samples. However, the strength of the signal reflected from the specimen back wall was not strong enough to obtain meaningful and reliable data due to the attenuation in the carbon fiber laminates. Hence, further testing was carried out using a 2.25 MHz frequency transducer.

![Figure 6: Behavior of ultrasonic pulse from a good [left] and a poor [right] adhesive bondlines.](image-url)
Figure 7: Ultrasonic Equipment.

Figure 8: Amplitude versus time plot showing focused area from adhesive layer.

4.1.2. Results and Discussion

Figures 9-13 reveal the RF waveforms of Panels 1-5. All the waveforms were acquired at the same gain in the instrument in order to show the comparative attenuation behavior in the samples. Due to the increasing attenuation in the bond-lines from Panels 1-5, the signal strength from the back wall decreases. In particular for Panel 5, the back
wall reflection is very weak. This should be taken in consideration in the characterization of adhesive bond lines.

Figure 9: Amplitude versus time plot of Panel 1, showing front, back and bond layer reflections.

Figure 10: Amplitude versus time plot of Panel 2, showing front, back and bond layer reflections.
Figure 11: Amplitude versus time plot of Panel 3, showing front, back and bond layer reflections.

Figure 12: Amplitude versus time plot of Panel 4, showing front, back and bond layer reflections.

Adhesive bondline characteristics were obtained in the form of C-scan images in this study. Figures 14-18 show the C-scan images of the bond-lines for all types of samples. The difference in percentage of attenuation in the C-scan images is represented in Figure 19. In this figure, reduction in signal strength due to variation in bond strength is
indicated as a shift in color pattern from red to green [or darker to lighter region on gray scale].

Figure 13: Amplitude versus time plot of Panel 5, showing front, back and bond layer reflections.

Figure 14: C-Scan Image of Panel 1, showing distribution of few white spots along the specimen dimensions.

Figure 15: C-Scan Image of Panel 2, showing distribution of porosity along the specimen dimensions.
Figure 16: C-Scan Image of Panel 3, showing distribution of porosity along the specimen dimensions.

Figure 17: C-Scan Image of Panel 4, showing distribution of porosity along the specimen dimensions.

Figure 18: C-Scan Image of Panel 5, showing distribution of porosity along the specimen dimensions.

Figure 19: Relationship between colors in C-scan Images and the percentage of signal strength.

A closer look at Figures 14 through 18 reveals that the color pattern is very consistent in Figure 14 showing less variation compared to other figures. This indicates that the bond quality of the specimen shown in Figure 14 is better than the other samples. It can be observed that white spots follow an increasing trend from Figure 14 through 18, which correspond to Samples 1 through 5, respectively. White spots represent voids where complete attenuation of ultrasonic signal occurred and no reflected signal was observed. These observations mean that the amount of porosity increases from Sample 1
through Sample 5. The increase in porosity leads to an increase in the attenuation coefficient value.

Thus, in order to look into the effect of porosity on the attenuation coefficient, attenuation coefficient values were calculated for each of the specimens tested in this study. Obtaining successive back wall echoes for attenuation coefficient calculation is difficult due to high attenuation in these composite materials. Thus, the attenuation coefficient was calculated by taking the front and back wall echoes into consideration and was calculated using Equation 10.

\[
\frac{A}{A_o} = e^{-\alpha x}
\]  

[10]

where, \( A \) and \( A_o \) are the amplitudes of the back wall and front wall echoes, respectively [61]. The other parameters \( \alpha \) and \( x \) are the attenuation coefficient and the distance between the front wall and back wall echoes, respectively. Readings were taken at two different locations on the adhesive bond to check the variation in attenuation with changes in porosity in the adhesive layer. The specimen thickness is related to the time interval between the front and back wall echoes and was used to calculate the velocity of ultrasonic waves in each of these specimens.

Longitudinal velocity for all the five specimens is plotted in Figure 20. It is found that the ultrasonic wave velocity is approximately 2858 m/s. The amplitude values from the panels were used to calculate the attenuation coefficients of the specimens and are presented in Table 1. The value reported in Table 1 is the average value of attenuation coefficient and longitudinal velocity in the panels.
Table 1: Values of attenuation in various adhesively bonded panels.

<table>
<thead>
<tr>
<th>Panel Number</th>
<th>Thickness of Adhesive Panel, mm</th>
<th>Velocity of Ultrasonic Waves, m/s</th>
<th>Attenuation $10^{-3}$ db/mm</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>10.54</td>
<td>3085</td>
<td>127</td>
</tr>
<tr>
<td>2</td>
<td>10.62</td>
<td>2686</td>
<td>131</td>
</tr>
<tr>
<td>3</td>
<td>10.44</td>
<td>2806</td>
<td>179</td>
</tr>
<tr>
<td>4</td>
<td>10.54</td>
<td>2518</td>
<td>161</td>
</tr>
<tr>
<td>5</td>
<td>12.6</td>
<td>2986</td>
<td>234</td>
</tr>
</tbody>
</table>

In order to compute the attenuation coefficient value in the adhesive layer, first a CFRP laminate (the same material as the adherend in the specimen) of 10.48mm thickness was subjected to ultrasonic immersion testing. The attenuation coefficient value obtained from this laminate was multiplied by the adherend thickness in the specimen. This value was subtracted from the attenuation coefficient value obtained from the samples to determine the attenuation coefficient value in the adhesive layer. However, before proceeding to the determination of the attenuation coefficient, the porosity in the adhesive layer has to be quantified. Therefore, in order to obtain a relationship between porosity
and the attenuation coefficient value, C-scan images (Fig 14-18) are subjected to image analysis.

In the image analysis of the C-scans, a ratio is obtained between the number of white pixels and the total number of pixels in the image. Porosity and attenuation coefficient values in the adhesive layer are shown in Figure 21. It is observed from these values that attenuation in these samples shows an increase from Panel 1 to Panel 5. Similarly, increase in porosity in the samples is observed. The bondline in Panel 1 has the lowest porosity compared to other samples and Panel 5 has the highest porosity.

![Graph showing porosity and attenuation coefficient in the adhesive layer.](image)

Figure 21: Porosity and attenuation coefficient in the adhesive layer.

Historically, an accurate determination of the quality of an adhesive bond has been possible by an estimation of shear strength of the adhesive joint which is adversely affected by the porosity in the bondlines [58]. The shear strength of the joint is calculated by taking the ratio between the applied load and the contact area between the adherends. As the porosity is increased the contact area between the adherends decreases and so does the shear strength. Hence, a correct estimate of porosity by means of nondestructive testing must be obtained. The present effort has successfully carried out this task.

### 4.2 Ultrasonic Characterization of Random Particulate Composites

Density and strength of microballoons having the same outer diameter is affected
by the change in wall thickness. Wall thickness of microballoons is defined by a parameter called Radius Ratio, \( \eta \), [62-63] given by Equation 11.

\[
\eta = \frac{r_i}{r_o} \quad [11]
\]

In the above equation \( r_i \) and \( r_o \) are the inner and the outer radii of microballoons, respectively. By increasing the value of \( \eta \), microballoon wall thickness decreases thereby decreasing microballoon density and strength. If the matrix and particle volume fractions are kept constant in syntactic foams, any change in mechanical properties of syntactic foams can be associated with the change in microballoon \( \eta \) alone. The glass microballoons are brittle and give rise to small fragments upon fracture. These fragments occupy more volume than the volume of the material composing the microballoon due to spatial mismatch between them. It is known that if microballoons have \( \eta \) lower than the critical value, 0.71, the wall thickness of the microballoons is too high and the resulting debris after fracture will occupy more volume than the microballoon before fracture [64]. Hence it is important to use microballoons of \( \eta \) greater than the critical value; so that the resulting debris occupies lesser volume after fracture and the stress concentration is reduced. This helps in further relieving the stress and in absorbing more energy by the foam specimen [62]. In the present study four types of microballoons with different \( \eta \) values, all greater than the critical value of 0.71, are selected for the fabrication of syntactic foams. Of these different types of microballoons, three of them have the same average outer diameter of 40 \( \mu \)m, whereas the fourth microballoon has an outer diameter of 35 \( \mu \)m.
4.2.1 Experimental: Materials and Methods

4.2.1.1 Fabrication

4.2.1.1.1 Glass Microballoons

The glass microballoons are manufactured and supplied by 3M under the trade name of Scotchlite. Physical properties of selected microballoons, supplied by the manufacturer, are presented in Table 2. Microballoon types S32, S38 and K46 selected for this research have the same mean outer diameter of 40 µm whereas microballoon type S22 has a mean outer diameter of 35 µm. The mean inner diameter has been calculated by taking the difference in the average true particle density of solid and hollow particles made of same material. Subsequently, the average wall thickness of microballoons is calculated. The difference in wall thickness of different types of microballoons causes the difference in their density. The calculated $\eta$ for all types of microballoons is also given in Table 2. The microballoon type in Table 2 is the manufacturer’s code for the identification of selected microballoons.

Table 2: Microballoon size distribution and radius ratio.

<table>
<thead>
<tr>
<th>Microballoon Type</th>
<th>Microballoon Size Distribution (µm)</th>
<th>Average True Particle Density (kg/m³)</th>
<th>Average Wall Thickness (µm)</th>
<th>Pressure for Min. 80% Fractional Survival (MPa)</th>
<th>$\eta$</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10&lt;sup&gt;th&lt;/sup&gt; percentile</td>
<td>50&lt;sup&gt;th&lt;/sup&gt; percentile</td>
<td>90&lt;sup&gt;th&lt;/sup&gt; percentile</td>
<td></td>
<td></td>
</tr>
<tr>
<td>S22</td>
<td>20</td>
<td>35</td>
<td>60</td>
<td>220</td>
<td>0.52</td>
</tr>
<tr>
<td>S32</td>
<td>20</td>
<td>40</td>
<td>75</td>
<td>320</td>
<td>0.88</td>
</tr>
<tr>
<td>S38</td>
<td>15</td>
<td>40</td>
<td>75</td>
<td>380</td>
<td>1.05</td>
</tr>
<tr>
<td>K46</td>
<td>15</td>
<td>40</td>
<td>70</td>
<td>460</td>
<td>1.29</td>
</tr>
</tbody>
</table>

4.2.1.1.2 Solid Glass Spheres

The solid glass spheres are manufactured by Potters Industries Inc under the trade name SPHERIGLASS® Solid Glass Spheres: E Glass. These glass particles are manufactured from a borosilicate glass composition similar to fiberglass. Solid particles
selected in this research have a density of 2.5g/cc and a mean diameter of 35µm similar to the diameter of S22 type hollow glass sphere.

4.2.1.1.3 Resin System

Epoxy resin D.E.R. 332, manufactured by DOW Chemical Company along with an amine based hardener D.E.H. 24 and a C₁₂-C₁₄ aliphaticglycidylether diluent is used as the matrix material. The diluent is mixed with the epoxy resin in 5% by weight quantity to reduce its viscosity. Reduction in viscosity makes it easier to mix higher volume fraction of particles in the matrix resin. The volume fraction of microballoons is varied from 10-60%, in the syntactic foam samples. Similarly, the volume fraction of glass spheres is varied from 10-60% in the solid particulate samples.

The microballoons and glass spheres were mechanically mixed in the matrix resin to make syntactic foam and solid particulates composition, respectively, and cast in stainless steel molds. Cast slabs were cured at room temperature for 24 hrs and post cured at 100±3°C for 3 hours. In the case of syntactic foams, the fabricated specimens have porosity within the microballoons, known as closed cell porosity and also in the matrix material due to mechanical mixing process, known as open cell porosity. However, in the case of solid particulates, porosity only exists in the matrix material due to the mechanical mixing process. Void content with the change in volume fraction in these syntactic foams is shown in Figure 22.

4.2.2 Specimen Nomenclature

The syntactic foam samples containing only glass microballoons in epoxy matrix have a three digit alphanumeric code such as S22. Here, the first letter represents Syntactic Foam and the next two digits are related to the true particle density of microballoons. The sample nomenclature indicates the microballoon type and the volume
fraction. In S2230 type of sample, S22 is the microballoon type and 30 is the volume fraction of microballoons. Similarly, solid particulate composites are also denoted by the particle type and the volume fraction in the composite. In Solid 10 type of sample, solid denotes the glass sphere used and 10 denotes the volume fraction of particles.

![Figure 22: Total void content in hollow particle syntactic foams with varying volume fraction.](image)

**4.2.3 Density Measurement**

To measure the density of the fabricated syntactic foam material standard ASTM C 271-94 [65] is followed. This standard is for measuring the density of sandwich core materials. This standard is selected considering the intended use of the fabricated syntactic foam slabs as core material in sandwich composites. The density values are obtained by measuring dimensions and weight of at least 5 pieces of $25 \times 25 \times 12.5 \text{ mm}^3$ dimensions. Results of density calculation of fabricated syntactic foam and solid particulates specimens are shown in Figure 23 and Figure 24. As shown in Figure 23 and Figure 24, the measured density decreases with an increase in volume fraction in the case of syntactic foams, whereas the measured density increases with an increase in volume.
fraction in the case of solid particulate composites.

![Graph of measured densities of syntactic foams with varying volume fraction.](image1)

Figure 23: Measured densities of syntactic foams with varying volume fraction.

![Graph of measured density of solid particulates with varying volume fraction.](image2)

Figure 24: Measured density of solid particulates with varying volume fraction.

4.2.4 Ultrasonic Characterization

Ultrasonic imaging [UI] of syntactic foam and solid particulate composite specimens are carried out using Physical Acoustic Corporation’s water immersion type system UltraPAC™ with Ultrawin™ software. Samples are subjected to pulse echo immersion ultrasound technique to determine the response of the material to ultrasonic
waves. The samples used for tests are approximately 12.5 mm thick and 25 mm in length. Five samples of each syntactic foam and solid particulate composite respectively with a specific volume fraction are tested. Frequencies of 1 MHz and 2.25 MHz are used for longitudinal wave characterization and a frequency of 2.25 MHz is used for the shear wave characterization of all the samples. Tests utilizing 2.25 MHz in pulse echo method were not effective for syntactic foams due to the high material porosity resulting in grass effect [66]. Therefore a 1 MHz transducer is used for testing all the syntactic foam samples. In the case of solid particulates, frequencies of 1 and 2.25 MHz are used for longitudinal wave characterization in pulse echo mode and a frequency of 2.25 MHz for shear wave characterization. The ultrasonic transducers have diameter and focal length of 0.5 and 1.5 inch respectively. All the UI scans are carried out at a sampling rate of 15.625 MHz and 31.25 MHz for frequencies of 1 MHz and 2.25 MHz respectively. The waveforms are acquired for each of the sample. The gain in the equipment was set such that the signal does not saturate.

4.2.5 Results and Discussions

Computation of apparent attenuation coefficient along with longitudinal and shear wave velocities are very important for characterization of particulate composites using ultrasonic testing. Apparent attenuation, longitudinal and shear wave velocities are calculated using RF waveforms obtained from the particulate composite samples. Figure 25 to Figure 28 represent RF waveforms obtained from ultrasonic testing of syntactic foam and particulate composite samples. In each of the waveforms, the front wall followed by first, second and third back wall reflections are clearly observed. ASTM Standard E664-93 [67] is used for computing the apparent attenuation in these foam and particulate composite samples. The apparent attenuation is computed by the Equation 12.
Apparent attenuation = \[20\log_{10} \frac{A_m}{A_n} - \frac{1}{2(n-m)*T}\] \[12\]

where,

\[A_m\] and \[A_n\] = amplitudes of the \(m\)th and \(n\)th back reflections (\(n>m\)), and

\[T\] = specimen thickness.

The apparent attenuation is calculated using the first and third back wall reflections for all the samples (syntactic foams and solid particulates) in this research. Calculation of apparent attenuation is performed using the waveforms acquired with 1 MHz transducer. The calculation of apparent attenuation using waveforms acquired with 2.25 and 5 MHz was not possible as the feasibility of obtaining two successive back wall echoes is limited due to the high porosity in the structure of syntactic foams [68-69]. Therefore, the attenuation coefficient is calculated by taking the ratio of first and third back wall echoes using a 1 MHz transducer.

Figure 25: Amplitude vs. time plot of SF2230 sample showing clear signal in time domain.
Figure 26: Amplitude vs. time plot of SF3240 sample.

Figure 27: Amplitude vs. time plot of SF3830 sample.
Figure 28: Amplitude vs time plot of Solid particulate at 20% volume fraction sample.

Figure 29: Attenuation Coefficient for S32, S38 and K46 foam samples at varying volume fractions.
Figure 30: Attenuation coefficient of S22 and solid particulate composites at varying volume fractions.

Attenuation coefficient values computed from four different types of syntactic foam and solid particulate composite samples are given in Figure 29 and Figure 30. Syntactic foam type S32, S38 and K46 are compared as they have the same outer diameter of 40 µm whereas syntactic foam of S22 type and solid particulate composite having solid glass spheres have the same outer diameter of 35 µm. From Figure 29 and Figure 30, it is evident that the attenuation coefficient values increases from 10% volume fraction to 30% volume fraction and then decrease from 30% volume fraction to 60% volume fraction. This trend of increase and decrease in attenuation coefficient of particulate composites can be attributed to the following reasons. In particulate composites, with an increase in volume fraction from 10% to 30%, the number of particles in the composite increases, thus increasing the scattering of ultrasonic energy. In addition to the scattering of ultrasonic energy by particles, energy is lost by absorption in epoxy matrix as well. Hence, in particulate composites of volume fractions from 10-30%, the percentage of epoxy is more than the percentage of particles in the matrix and thus increasing the attenuation coefficient from 10% to 30%. Due to the above reasons, the attenuation
coefficient increases from a volume fraction of 10% to 30%. Volume fractions of 10-30% are termed as dilute suspensions as the particles are widely dispersed in the epoxy matrix [40]. However, after 30% volume fraction, with an increase in volume fraction, the number of particles increases, and thus particles start getting close to each other increasing the probability of contact between particles. Another consequence that occurs in the volume fraction range of 30-60% is the decrease of amount of epoxy in the matrix and thus decreasing the absorption of ultrasonic signal. Therefore, the ultrasonic signal has a higher probability of traveling through the particles rather than interacting with the epoxy matrix lying around. Thus, from 30% volume fraction, the attenuation coefficient decreases in all the types of syntactic foams and particulate composite.

From Figure 29, it is also evident that the attenuation coefficient value decreases from S32 to K46 for almost all the volume fractions used in this study. This is due to the reason that the radius ratio decreases from S32 to K46 thereby decreasing the void content enclosed in the microballoons. This in return causes the attenuation coefficient values to decrease with a decrease in the void content in the composite as less ultrasonic energy is absorbed in the smaller voids. In order to understand the absorption of ultrasonic energy by pure epoxy, longitudinal ultrasonic characterization is performed on pure epoxy samples at 1 MHz and the attenuation coefficient is calculated as $222 \times 10^{-3}$ db/mm.

Figure 30 shows the comparison of attenuation coefficient between S22 type syntactic foam and solid particulate composite. As the outer diameter is the same in both the S22 type and solid particulate composite, the attenuation coefficient is expected to be higher in S22 type of syntactic foam rather than solid particulate composite due to the presence of voids in S22 type of foam. But, the trend obtained experimentally is reverse to the expected trend. This reversal in attenuation coefficient trend is attributed to the fact of resonance in solid particulate composites. According to Kinra and Ker [48], the frequency above which resonance effects become important in glass-epoxy particulates is
0.5 MHz (approx). Thus, due to the resonance of the solid glass spheres, some ultrasonic energy is absorbed because of the vibration of glass spheres and thus increasing the attenuation coefficient.

According to ASTM E 494-95 [70], the time lag between the front and back wall reflections is taken into account for calculating the longitudinal ultrasonic velocities in the samples. Thus by taking the peak-to-peak distances in materials, one could easily compute the ultrasonic velocity. However, caution need to be exercised when measuring velocity of porous materials. Due to the higher attenuation of ultrasonic signals in porous materials such as particulate composites, the back wall reflection shifts back and forth as shown in Figure 31. In Figure 31, theoretical first back wall reflection is the back wall reflection in the particulate according to ASTM E 494-95, whereas actual first back wall reflection is the back wall reflection corresponding to the particulate. Therefore computing the longitudinal velocity by taking peak-to-peak time lag according to ASTM E494-95 is error prone.

Figure 31: Shift of peaks due to attenuation in particulate composites
In order to adjust for the error in peak shift due to attenuation, back wall reflection is selected by tracking the distance between front and back wall in time domain using data acquisition software.

After properly selecting the front and back wall reflections in particulates, the longitudinal velocity is computed by taking the ratio between twice the thickness of sample to the time taken between front and back wall reflections. For computing the longitudinal ultrasonic velocity, each of the samples of a particular particle size and volume fraction, are subjected to longitudinal scans. Figure 32 show the longitudinal wave velocities in S22, S32, S38 and K46 types of syntactic foam samples subjected to longitudinal ultrasonic waves of 1 MHz frequency. Immersion type of testing is used for the longitudinal scans using 1 MHz transducer frequency. Immersion testing is also performed on syntactic foam samples using longitudinal frequency of 2.25 and 5 MHz, but due to the high attenuation in the samples, no consistent signal was obtained. Therefore, the results obtained with 2.25 and 5 MHz frequency transducers in immersion type are not reported.
The ultrasonic velocities in syntactic foams are found to be in the range of 2200-2900 m/s as shown in Figure 32. From Figure 32, it is also evident that the longitudinal velocity in foam composites decreased, with an increase in volume fraction. This can be attributed to the fact that, for each type of microballoon, the measured densities in these composites decreases with an increase in the volume fractions as shown in Figure 23. Thus, longitudinal velocity in syntactic foams decreases with an increase in volume fraction.

From Figure 32, it can also be observed that at a particular volume fraction, the longitudinal wave velocities increase from S22 to K46 with a corresponding increase in density from S22 to K46 types of syntactic foams. In order to understand the shift in longitudinal velocity due to addition of particulates into epoxy matrix, pure epoxy samples are also tested at 1 MHz and the longitudinal velocity in pure epoxy is computed as 2838 m/s.

![Figure 33: Longitudinal wave velocity of solid particulates at varying volume fractions.](image-url)
Figure 33 shows the ultrasonic longitudinal velocity in solid particulate composites with varying volume fraction at frequencies of 1 and 2.25 MHz. The longitudinal velocity in solid particulates is lower when test was performed at 2.25 MHz frequency compared to the value computed at 1 MHz frequency. This can be attributed to the fact that the wavelength of ultrasonic waves decreases with an increase in frequency. As the wavelength decreases, wave starts interacting with the particles more extensively and results in increasing scattering. Due to the reason given earlier, the wave takes longer time to traverse through the composite sample and thus decreasing the ultrasonic velocity.

At each of the particular frequency, the longitudinal velocity increases with the volume fraction. This is due to the reason that with an increase in volume fraction, the density of these solid particulate composites increases as shown in Figure 24 and thus increasing the longitudinal velocity. Figure 34 and Figure 35 show the shear wave velocity trend in syntactic foams and particulate composite with volume fraction. The shear wave velocity calculations are performed using a 2.25 MHz frequency contact type transducer. The shear wave velocities in syntactic foams and particulate composites increase with an increase in volume fraction.

![Shear wave velocities of syntactic foam samples at varying volume fractions.](image)
4.2.6 Prediction of Dynamic Modulus Using UI

A relationship is established between the modulus obtained from UI and material modulus. This relationship provides for the first time the means to characterize the dynamic mechanical properties of material using UI without mechanical testing in particulate composites and syntactic foams. Dynamic modulus can be predicted using ultrasonic technique with the aid of the following equation. Using longitudinal velocity and shear wave velocity from the foam samples, Lame’s parameters, $\lambda$ and $\mu$ are calculated using fundamental theory of elasticity [71-74].

Longitudinal Velocity,\[ V_l = \sqrt{\frac{\lambda + 2\mu}{\rho}} \] \hspace{1cm} [13]

Shear Wave Velocity,\[ V_s = \sqrt{\frac{\mu}{\rho}} \] \hspace{1cm} [14]

Poisson’s ratio,\[ \nu = \frac{\lambda}{2(\lambda + \mu)} \] \hspace{1cm} [15]

Finally, modulus can be computed using,
\[ E = \frac{V_l^2 \rho (1+\nu)(1-2\nu)}{1-\nu} \times 0.733 \times 6.89 \times 10^4 \times 0.00689 \text{ Mpa} \] \hspace{1cm} [16]
where, $V_l$, $V_s$, are in cm/s and $\rho$ is in g/cm³

Dynamic modulus values predicted from ultrasonic characterization of the syntactic foams for varying volume fractions are compared with modulus values obtained from high strain rate using split Hopkinson pressure bar (SHPB) and quasi-static testing [75] in Table 3. The values reported in Table 3 are calculated using the longitudinal velocity values obtained at frequency of 1 MHz. The shear wave velocity was only tested at 2.25 MHz as the signal was not clear below and above the frequency of 2.25 MHz. Using the longitudinal and shear wave velocities along with Equations 13-16, dynamic modulus in syntactic foams is predicted.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Poisson’s Ratio</th>
<th>UI Modulus MPA</th>
<th>SHPB Modulus MPA</th>
<th>Static Modulus MPA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Epoxy</td>
<td>0.34±0.00</td>
<td>4476±183</td>
<td>4272-5084</td>
<td>2320±40</td>
</tr>
<tr>
<td>S2210</td>
<td>0.30±0.00</td>
<td>3667 ± 89</td>
<td>3310-3903</td>
<td>2192</td>
</tr>
<tr>
<td>S2220</td>
<td>0.30±0.01</td>
<td>3539 ± 30</td>
<td>3438-3897</td>
<td>2151</td>
</tr>
<tr>
<td>S2230</td>
<td>0.28±0.01</td>
<td>2807 ± 54</td>
<td>2092-2944</td>
<td>1966</td>
</tr>
<tr>
<td>S2240</td>
<td>0.28±0.00</td>
<td>2393 ± 20</td>
<td>2194-2525</td>
<td>1803</td>
</tr>
<tr>
<td>S2250</td>
<td>0.25±0.01</td>
<td>2173 ± 41</td>
<td>1701-2244</td>
<td>1512</td>
</tr>
<tr>
<td>S3210</td>
<td>0.32±0.00</td>
<td>4071 ± 45</td>
<td>3048-4099</td>
<td>2375</td>
</tr>
<tr>
<td>S3220</td>
<td>0.32±0.01</td>
<td>3772 ± 114</td>
<td>3601-3897</td>
<td>2370</td>
</tr>
<tr>
<td>S3230</td>
<td>0.31±0.01</td>
<td>3457 ± 62</td>
<td>2991-3371</td>
<td>2282</td>
</tr>
<tr>
<td>S3240</td>
<td>0.29±0.01</td>
<td>3174 ± 86</td>
<td>2829-3524</td>
<td>2252</td>
</tr>
<tr>
<td>S3250</td>
<td>0.28±0.00</td>
<td>2800 ± 88</td>
<td>2146-2808</td>
<td>2052</td>
</tr>
<tr>
<td>S3260</td>
<td>0.27±0.01</td>
<td>2132 ± 42</td>
<td>1926-2033</td>
<td>1878</td>
</tr>
<tr>
<td>S3810</td>
<td>0.33±0.00</td>
<td>4309 ± 77</td>
<td>4162-4882</td>
<td>2593</td>
</tr>
<tr>
<td>S3820</td>
<td>0.32±0.01</td>
<td>3912 ± 92</td>
<td>3698-4277</td>
<td>2466</td>
</tr>
<tr>
<td>S3830</td>
<td>0.3±0.01</td>
<td>3718 ± 73</td>
<td>3008-3661</td>
<td>2326</td>
</tr>
<tr>
<td>S3840</td>
<td>0.29±0.00</td>
<td>3481 ± 25</td>
<td>3244-3664</td>
<td>2351</td>
</tr>
<tr>
<td>S3850</td>
<td>0.25±0.01</td>
<td>3069 ± 51</td>
<td>2469-3078</td>
<td>2087</td>
</tr>
<tr>
<td>S3860</td>
<td>0.23±0.01</td>
<td>2549 ± 47</td>
<td>2132-2774</td>
<td>2099</td>
</tr>
<tr>
<td>K4610</td>
<td>0.34±0.00</td>
<td>4375 ± 56</td>
<td>3698-4722</td>
<td>2576</td>
</tr>
<tr>
<td>K4620</td>
<td>0.32±0.00</td>
<td>4150 ± 55</td>
<td>3872-4282</td>
<td>2470</td>
</tr>
<tr>
<td>K4630</td>
<td>0.3±0.01</td>
<td>3953 ± 71</td>
<td>3590-4182</td>
<td>2508</td>
</tr>
<tr>
<td>K4640</td>
<td>0.29±0.01</td>
<td>3643 ± 121</td>
<td>3474-3846</td>
<td>2414</td>
</tr>
<tr>
<td>K4650</td>
<td>0.25±0.01</td>
<td>3385 ± 109</td>
<td>2901-3446</td>
<td>2473</td>
</tr>
</tbody>
</table>
Table 4: Comparison of Dynamic and Quasi-Static Modulus for solid particulates.

<table>
<thead>
<tr>
<th>Sample Name</th>
<th>Poisson’s Ratio (1 MHz)</th>
<th>Poisson’s Ratio (2.25 MHz)</th>
<th>UI Modulus MPA (1 MHz)</th>
<th>UI Modulus MPA (2.25 MHz)</th>
<th>SHPB Modulus MPA</th>
<th>Static Modulus MPA</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pure Epoxy</td>
<td>0.34±0.0</td>
<td>0.33±0.00</td>
<td>4476±183</td>
<td>4385±154</td>
<td>4272-5084</td>
<td>2320±40</td>
</tr>
<tr>
<td>Solid 10</td>
<td>0.33±0.0</td>
<td>0.32±0.00</td>
<td>5372±76</td>
<td>5245±61</td>
<td>5285-5866</td>
<td>2718</td>
</tr>
<tr>
<td>Solid 20</td>
<td>0.31±0.0</td>
<td>0.31±0.01</td>
<td>6360±70</td>
<td>6505±93</td>
<td>5859-7169</td>
<td>3116</td>
</tr>
<tr>
<td>Solid 30</td>
<td>0.30±0.0</td>
<td>0.28±0.00</td>
<td>7480±113</td>
<td>6951±145</td>
<td>6250-8067</td>
<td>3514</td>
</tr>
<tr>
<td>Solid 40</td>
<td>0.29±0.02</td>
<td>0.28±0.00</td>
<td>11129±600</td>
<td>10305±122</td>
<td>10174-11538</td>
<td>3912</td>
</tr>
<tr>
<td>Solid 50</td>
<td>0.28±0.0</td>
<td>0.27±0.01</td>
<td>12303±264</td>
<td>11798±792</td>
<td>12082-13000</td>
<td>4310</td>
</tr>
<tr>
<td>Solid 60</td>
<td>0.27±0.0</td>
<td>0.24±0.00</td>
<td>14016±183</td>
<td>12908±154</td>
<td>11394-14431</td>
<td>4708</td>
</tr>
</tbody>
</table>

Table 4 shows the predicted dynamic modulus and Poisson’s ratio values in solid particulates using 1 and 2.25 MHz longitudinal frequency transducers. From Table 4 it is evident that the dynamic modulus predicted from both the frequencies is almost the same. The Poisson’s ratio predicted from this study in all the syntactic foams and solid particulates is in the range of 0.24-0.33. This prediction of Poisson’s ratio satisfies the theoretical prediction of Bardella et.al. [76].

Measurement of elastic moduli from ultrasonic measurements yields static modulus in the case of metals, metal matrix and FRP composites [74]. However, in the case of particulate composites, the modulus predicted is comparable to the high strain rate testing modulus or the dynamic modulus rather than the quasi-static modulus. This is due to the reason that, metals and metal matrix composites are ultrasonically inspected at frequencies ranging from 10 MHz and above [77-78]. In the frequency range of 10 MHz and above, ultrasonic wave propagation in metals and metal matrix composites is non dispersive. Also, as the stress applied by testing metals and metal matrix composites at higher frequencies is very small, the strains produced also are relatively low [78]. Thus the modulus obtained is of static type.
In the case of FRP composites that are tested in the range of 1-10MHz, at low frequencies, the observed dispersion and attenuation effects obtained from ultrasonic testing are weak or non existent [74]. In such cases composite behaves as a homogeneous but anisotropic material. This generally happens when the frequency is well below any of the internal resonance frequencies associated with the microstructure of the composite. The wavelength of the waves is also large compared with the characteristic dimensions such as lamina thickness, fiber diameter, and inter-diameter spacing. In this case it is not the separate fiber and matrix properties that govern the propagation of waves but rather some weighted average of both fiber and matrix properties. Thus the modulus that is computed using ultrasonic testing is static modulus rather than a dynamic modulus. Similar is the case with metals [74].

However in the case of particulate composites, even though the wavelengths are large compared to the characteristic dimensions of the composite such as particle diameter, and inter-particle distance the attenuation in the composite is prominent. In addition, as the frequencies of inspection are close to the internal resonant frequency associated with microstructure of the particulate composite, this composite behaves as an inhomogeneous and anisotropic composite. Attenuation of ultrasonic signals in these particulate composites is caused by wave absorption in epoxy matrix at low volume fractions of 10-30% and by scattering between particles at high volume fractions of 30-60%. Due to the above reasons, individual inclusion properties govern the wave propagation behavior rather than the weighted average of both matrix and inclusion properties as in the case of FRP composites.

Thus, in particulate composites, wave propagation behavior such as scattering at individual inclusions governs the elastic properties obtained by ultrasonic testing. Scattering of ultrasonic energy at inclusions is dependent upon the ratio between the wavelength and the particle size. As shown in Table 5, at 1MHz longitudinal frequency,
the ratio of wavelength to the particle size in particulate composites is in the range of 65-100. Thus there is more probability for ultrasonic wave to look at the composite as clusters rather than a uniform solid. Each of the clusters contains about 20,000 particles that are placed together. However, the ultrasonic wave will just have to pass through 65-100 clusters rather than millions of particles that are present in the composite. Therefore, scattering of ultrasonic wave does not occur at each and every particle-particle interface rather than between clusters of particles. Due to the above said reasons, the longitudinal velocity of the composite computed at lower frequencies will be more than the longitudinal velocity computed at higher frequencies, i.e. where the wavelength is close to the particle size. Therefore, as the longitudinal velocity is directly proportional to the modulus, at lower frequencies with an increase in velocity, the ultrasonic modulus of particulate composites increases and thus approach the dynamic modulus value.

Table 5: Ratio of wavelength to the particle size at 1MHz frequency in particulate composites.

<table>
<thead>
<tr>
<th>Volume Fraction</th>
<th>S22</th>
<th>S32</th>
<th>S38</th>
<th>K46</th>
<th>Solid</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.1</td>
<td>71.1</td>
<td>69.4</td>
<td>71.5</td>
<td>72.5</td>
<td>81.9</td>
</tr>
<tr>
<td>0.2</td>
<td>70.2</td>
<td>68.5</td>
<td>70.4</td>
<td>70.8</td>
<td>82.3</td>
</tr>
<tr>
<td>0.3</td>
<td>69.2</td>
<td>68.4</td>
<td>69.1</td>
<td>70.6</td>
<td>85.6</td>
</tr>
<tr>
<td>0.4</td>
<td>68.7</td>
<td>67.3</td>
<td>68.9</td>
<td>69.9</td>
<td>96.9</td>
</tr>
<tr>
<td>0.5</td>
<td>67.7</td>
<td>66.5</td>
<td>66.8</td>
<td>67.8</td>
<td>98.8</td>
</tr>
<tr>
<td>0.6</td>
<td>65.6</td>
<td>64.3</td>
<td>64.9</td>
<td>66.7</td>
<td>103</td>
</tr>
</tbody>
</table>
5. MODELLING OF ATTENUATION COEFFICIENT

Pulse echo ultrasonic attenuation will be derived in terms of volume fraction of glass microballoons/spheres, outer radius of microballoon/spheres, along with scattering and absorption cross sections of microballoons/spheres. An input ultrasonic transducer is assumed to apply a uniform pressure of amplitude $P_o$ that is sinusoidal in time on the surface of a specimen. It is also assumed that the planar area of the ultrasonic beam propagates and comes back through the specimen of thickness $\ell$ without alteration. Portions of the input ultrasonic energy are scattered and absorbed by the $n$ randomly distributed glass spheres in particulate composites. Absorption of ultrasonic energy in particulates composites is primarily due to the resonance of solid glass spheres. Similarly, in the case of syntactic foams, portions of input ultrasonic energy is scattered as well as absorbed by the ‘n’ randomly distributed hollow microballoons in the ultrasonic beam path.

The output transmitted pressure is measured by the same transducer through which the input energy was sent into the specimen. The output pressure is given by $P_o e^{-\alpha \ell}$ where $\alpha$ is the attenuation which is a function of ultrasonic frequency in the material. It is assumed that energy carried in the scattered and absorbed waves is lost and scattering and absorption are the dominant energy dissipating mechanisms considered here.

The intensity $I$ of a plane longitudinal traveling wave can be related to the pressure $P$ as given in Equation 17 [61].

$$I = \frac{P^2}{\rho V_l} \quad [17]$$

where, $\rho$ is the mass density of the material and $V_l$ is the longitudinal wave speed. Thus, the incident ultrasonic intensity $I_o$ is given by
\[ I_0 = \frac{P_0^2}{\rho V_i} \]  

The power lost due to scattering from a single microballoon or a glass sphere is given by \( \gamma_i I_0 \), where \( \gamma_i \) is the scattering cross section of a microballoon or a glass sphere.

According to Ying and Truell [41], the scattering cross section of a spherical cavity is given by the equation:

\[ \gamma = \frac{4\pi}{9} k^4 r_o^6 g \]  

where,

\[ k = \frac{2\pi}{\lambda_L} = \frac{2\pi f}{V_L} \]

\( r_o \) is the outer radius of glass particles

\[ g = \frac{4}{3} + 40 \left( \frac{2 + 3(V_r^5)}{4 - 9(V_r^2)} \right) - \frac{3}{2} V_r^2 + \frac{2}{3} V_r^3 + \frac{9}{16} V_r^4 \]

and,

\[ V_r = \frac{V_i}{V_s} \]

Assuming no interaction between glass microballoons/spheres, the total power lost due to scattering \( P_{\text{scattered}} \) by \( n \) glass microballoons/spheres is

\[ P_{\text{scattered}} = I_0 \sum_{i=1}^{n} \gamma_i \]

where,

\( n \) is the total number of “scatterers” (microballoons/glass spheres) in the ultrasonic beam path and where the \( i^{th} \) void has scattering cross section \( \gamma_i \) [41].

Therefore,

\[ P_{\text{Scattered}} = \frac{P_0^2}{\rho V_i} \sum_{i=1}^{n} \gamma_i \]
In addition to the energy lost due to scattering by microballoons/solid glass spheres in syntactic foams as well as solid particulate composites, energy is also lost due to different phenomena in syntactic foams and solid particulate composites. In the case of syntactic foams, part of the energy is also absorbed which is given by $\gamma_a I_o$.

The absorption cross section is directly proportional to the ratio between the volume of hollow cavity and the wavelength of longitudinal wave [79-80].

$$\gamma_a = \frac{4 \times 4 \times \pi \times r_i^3}{3 \times \lambda} = \frac{4 \times 4 \times \pi \times r_o^3 \times \eta^3}{3 \times \lambda}$$ \hspace{1cm} [25]

where,

$\gamma_a$ is the absorption cross section of microballoons

$r_o$ is the outside radius of microballoon

$r_i$ is the inside radius of microballoon

$\lambda$ is the wavelength of longitudinal wave in the specimen, and

$\eta$ is the radius ratio of inside to outside radius of hollow microballoon.

Thus, the total power lost due to absorption is given by Equation 26.

$$P_{absorbed} = I_o \sum_{i=1}^{n} \gamma_{a,i} = \frac{P_o^2}{\rho V} \sum_{i=1}^{n} \gamma_{a,i}$$ \hspace{1cm} [26]

In the case of particulate composites, portion of the energy is absorbed due to resonance of solid glass spheres. As shown in Equation 27, the resonance cross section is directly proportional to the volume of the glass sphere and inversely proportional to the wavelength.

$$\gamma_r = \frac{4 \times 4 \times \pi \times r^3}{3 \times \lambda}$$ \hspace{1cm} [27]

where, $\gamma_r$ is the resonance cross section of glass particles

$r$ is the radius of glass sphere, and

$\lambda$ is the wavelength of longitudinal wave in the specimen
Therefore, the ultrasonic energy lost by resonance of glass spheres in solid particulates is given by,

\[ P_{\text{resonance}} = I_o \sum_{i=1}^{n} \gamma_{r,i} = \frac{P_o^2}{\rho V_i} \sum_{i=1}^{n} \gamma_{r,i} \]  

[28]

The input ultrasonic power can be obtained by

\[ P_{\text{in}} = \frac{P_o^2}{\rho V_i} A \]  

[29]

where, \( A \) is the planar beam area of the ultrasonic beam.

Similarly, the output ultrasonic power can be obtained as

\[ P_{\text{out}} = \left( \frac{P_o e^{-2d \alpha}}{\rho V_i} \right)^2 A \]  

[30]

It is assumed that the output power is obtained after some of the power from the input power is lost by scattering, absorption and resonance by microballoon/solid glass spheres.

Thus, the equation for energy balance is given by

\[ P_{\text{in}} - P_{\text{scattered}} - P_{\text{absorbed}} - P_{\text{resonance}} = P_{\text{out}} \]  

[31]

Substituting equations for \( P_{\text{in}} \), \( P_{\text{scattered}} \), \( P_{\text{absorbed}} \), \( P_{\text{resonance}} \) and \( P_{\text{out}} \) in Equation 31,

\[ \frac{P_o^2}{\rho V_i} A - \frac{P_o^2}{\rho V_i} n \gamma_s - \frac{P_o^2}{\rho V_i} n \gamma_a - \frac{P_o^2}{\rho V_i} n \gamma_r = \frac{P_o^2 e^{-2d \alpha}}{\rho V_i} A \]  

[32]

By cancelling \( \frac{P_o^2}{\rho V_i} \) in the Equation 32, the equation is reduced to,

\[ A - n \gamma_s - n \gamma_a - n \gamma_r = e^{-4d \alpha} A \]  

[33]

Taking terms having \( n \) as common, Equation 33 is reduced to

\[ A(1 - e^{-4d \alpha}) = n(\gamma_s + \gamma_a + \gamma_r) \]  

[34]

From Equation 34, \( \alpha \) can be simplified as,

\[ \alpha = -\frac{1}{4d} \ln \left( 1 - \frac{n}{A} \left( \gamma_s + \gamma_a + \gamma_r \right) \right) \]  

[35]
However, \( n \) is the number of particles covered by the ultrasonic beam of area \( A \).

Thus, \( n \) can be defined by the relation,

\[
n = \frac{V_f \cdot 1 \text{mm}^3 \cdot A \cdot l}{\text{vol.of.one.particle}} \quad [36]
\]

where,

- \( V_f \) is the volume fraction of particles
- \( 1 \text{mm}^3 \) is the unit volume of sample
- \( A \) is the Beam Area
- \( l \) is the sample dimension through which the beam passes through

Substituting equation for volume of one particle in Equation 36,

\[
n = \frac{V_f \cdot 1 \text{mm}^3 \cdot A \cdot l}{(4\pi r^3 / 3)} \quad [37]
\]

Simplifying Equation 37,

\[
n = \frac{3V_f \cdot A \cdot l}{4\pi r^3} \quad [38]
\]

By substituting equation for \( n \) from Equation 38 in Equation 35, attenuation coefficient is given by,

\[
\alpha = \frac{-1}{4l} \ln \left( 1 - \frac{3V_f \cdot A \cdot l}{A \cdot 4\pi r^3} \cdot (\gamma_s + \gamma_a + \gamma_r) \right) \quad [39]
\]

Canceling \( A \) from numerator and denominator, Equation 39 is reduced to,

\[
\alpha = \frac{-1}{4l} \ln \left( 1 - \frac{3V_f \cdot l}{4\pi r^3} \cdot (\gamma_s + \gamma_a + \gamma_r) \right) \quad [40]
\]

Using Equation 40, ultrasonic longitudinal attenuation in particulate composites due to both scattering and absorption can be calculated. The equation is equally applicable for hollow and solid particulate composites as the absorption term in solid particulate
composites will vanish due to the zero value of radius ratio and the resonance term will vanish in the case of syntactic foams. However, absorption of ultrasonic energy due to the epoxy matrix needs to be added to the attenuation coefficient value obtained above to compute the total attenuation in the composite.

Therefore, the attenuation in the composite is

\[ \alpha_{\text{Comp}} = \alpha_{\text{Epoxy}} + \alpha \]  \[41\]

where, \( \alpha_{\text{Comp}} \) is the ultrasonic attenuation in the composite as a whole, and \( \alpha_{\text{Epoxy}} \) is the ultrasonic attenuation due to epoxy in the composite.

Experimental and modeling results obtained from this study are compared with the results of Yamakawa [81] for a dilute suspension which is given by

\[ \alpha_{\text{Comp}} = \alpha_{\text{Epoxy}} + \frac{3V}{8\pi r_o^\gamma} \]  \[42\]

Where, \( \gamma \) is the scattering cross section of the microballoons/glass spheres given by Equation 19. Figure 36-Figure 40 show the comparison of experimentally calculated attenuation coefficient with Yamakawa’s model and our model. A clear correlation between the experimental and theoretical results are found for all the types of syntactic foams and particulate composites till 30% volume fraction as this model is only valid for dilute suspensions considering the fact that the inter-particle interactions are neglected. Yamakawa’s model under predicted the attenuation coefficient values in all these hollow and solid particulate foams.
Figure 36: Comparison of experimental and theoretical attenuation coefficients in S22 type particulates.

Figure 37: Comparison of experimental and theoretical attenuation coefficients in S32 type particulates.
Figure 38: Comparison of experimental and theoretical attenuation coefficients in S38 type particulates.

Figure 39: Comparison of experimental and theoretical attenuation coefficients in K46 type particulates.
Figure 40: Comparison of experimental and theoretical attenuation coefficients in solid particulates.
6. CONCLUSIONS

Ultrasonic imaging is used to characterize adhesive bonds and particulate composites. Adhesively bonded panels fabricated with varying amounts of porosity in the bondlines were tested using the pulse echo method of ultrasonic imaging. These panels also had different thicknesses. The C-Scan images of the panels obtained using ultrasonic imaging showed variation in porosity in the bondlines of five types of samples. Image analysis was performed on the C-scans to obtain the amount of porosity in the adhesive bondlines. The image analysis of the C-scans depicts variation in the dependency of attenuation coefficient on the extent of porosity. Attenuation coefficient values were found to increase due to the increase in the porosity of the samples. Additionally, in order to analyze the effect of laminate thickness on attenuation, samples of similar as well as different thicknesses were considered. The attenuation coefficient values were also found to be dependent on the thickness variation in the panels. In general, the attenuation coefficient increases as the thickness increases. This behavior is a characteristic resemblance of highly attenuative materials such as composites.

Further, ultrasonic characterization of syntactic foams and solid particulates dispersed within an epoxy matrix is performed. Syntactic foams are fabricated using four types of microballoons with same outer diameter but varying internal diameter and varying volume fraction from 10-60%. Particulate composites are fabricated using one type of solid glass sphere with varying volume fraction from 10-60%. Attenuation coefficient and longitudinal velocities are calculated for varying volume fractions for each of the particle (microballoon/solid particulate) type. It is shown that that attenuation coefficient values increase with volume fraction from 10-30% in each of the types of syntactic foams and particulate composite and decrease from 30%-60%. The increasing and decreasing trend of attenuation coefficient is due to scattering of ultrasound by the
glass particles and absorption of ultrasound by epoxy matrix. With an increase in volume fraction from 10-30%, the number of microballoons/glass particles increases, thus increasing the scattering of ultrasonic energy. Also, in the range of volume fractions from 10-30%, the percentage of epoxy in the matrix is much more than the percentage of particles. Therefore, ultrasonic energy is also absorbed by the epoxy in the matrix. Furthermore, in the range of 30%-60%, the ultrasonic absorption caused by the epoxy decreases, as the percentage of epoxy in the composite decreases. Also, in the volume fraction range of 30-60%, the particles are closer to each other and thus decreasing the distance between particles. Due to this proximity between particles, particles tend to act as clusters rather than individual particles. Thus, scattering of ultrasonic energy occurs between cluster to cluster rather than between particle to particle. Therefore, there is a decrease in the scattering of ultrasound in the composite. Hence, the attenuation coefficient value increases from 10-30% and then decreases from 30-60% volume fraction. Longitudinal velocities are calculated for each of the composite samples and it was found that the longitudinal velocities decreased with an increase in volume fraction for each type of microballoon due to the subsequent decrease in density of syntactic foam composite. However, the longitudinal velocities increased with an increase in volume fraction in solid glass particles. This increase in velocity with an increase in volume fraction is due to the increase in density of solid glass particulates. Also, dynamic modulus and Poisson’s ratio are calculated using UI. This is the first time that a relationship is established between the modulus obtained from UI and material modulus. In addition, a constitutive model explaining the effect of particle size, porosity, radius ratio on the ultrasonic attenuation coefficient in particulate composites is developed.
REFERENCES


67. ASTM E664-93 Standard practice for the measurement of the apparent attenuation of longitudinal ultrasonic waves by immersion method, ASTM International, PA


70. ASTM E494-95 Standard practice for measuring velocity in materials. ASTM International, PA, USA


## APPENDIX-SUPPLEMENTAL ULTRASONIC DATA FOR PARTICULATES

**Table A1: Attenuation Coefficients in different particulates with varying volume fractions at 1 MHz frequency.**

<table>
<thead>
<tr>
<th>Volume Fraction</th>
<th>S22</th>
<th>S32</th>
<th>S38</th>
<th>K46</th>
<th>Solid</th>
</tr>
</thead>
<tbody>
<tr>
<td>10%</td>
<td>245.7±18.6</td>
<td>272.0±36.9</td>
<td>265.5±38.3</td>
<td>238.6±32.9</td>
<td>276.9±34.7</td>
</tr>
<tr>
<td>20%</td>
<td>272.1±62.6</td>
<td>287.0±35.0</td>
<td>270.6±40.5</td>
<td>254.1±42.6</td>
<td>284.2±17.7</td>
</tr>
<tr>
<td>30%</td>
<td>291.9±53.5</td>
<td>291.0±50.8</td>
<td>285.0±71.9</td>
<td>263.4±31.3</td>
<td>300.9±12.2</td>
</tr>
<tr>
<td>40%</td>
<td>250.9±49.2</td>
<td>264.0±88.3</td>
<td>252.2±25.1</td>
<td>252.7±12.0</td>
<td>289.1±23.5</td>
</tr>
<tr>
<td>50%</td>
<td>218.2±10.3</td>
<td>247.4±45.0</td>
<td>207.1±11.7</td>
<td>201.6±18.4</td>
<td>261.8±20.3</td>
</tr>
<tr>
<td>60%</td>
<td>201.8±10.1</td>
<td>225.4±12.4</td>
<td>205.2±7.5</td>
<td>201.5±15.0</td>
<td>261.4±11.6</td>
</tr>
</tbody>
</table>
| Pure Epoxy      | 222.3±11.1|          |          |          |\

**Table A2: Longitudinal Velocities in different particulates with varying volume fractions at 1 MHz frequency.**

<table>
<thead>
<tr>
<th>Volume Fraction</th>
<th>S22</th>
<th>S32</th>
<th>S38</th>
<th>K46</th>
<th>Solid</th>
</tr>
</thead>
<tbody>
<tr>
<td>10%</td>
<td>2489±32</td>
<td>2776±27</td>
<td>2859±35</td>
<td>2902±51</td>
<td>2868±19</td>
</tr>
<tr>
<td>20%</td>
<td>2458±13</td>
<td>2741±63</td>
<td>2817±48</td>
<td>2831±26</td>
<td>2882±32</td>
</tr>
<tr>
<td>30%</td>
<td>2421±46</td>
<td>2737±48</td>
<td>2763±59</td>
<td>2824±47</td>
<td>2994±26</td>
</tr>
<tr>
<td>40%</td>
<td>2403±7</td>
<td>2691±35</td>
<td>2758±24</td>
<td>2795±91</td>
<td>3393±16</td>
</tr>
<tr>
<td>50%</td>
<td>2368±19</td>
<td>2661±44</td>
<td>2672±49</td>
<td>2710±62</td>
<td>3459±6</td>
</tr>
<tr>
<td>60%</td>
<td>2296±10</td>
<td>2574±35</td>
<td>2595±46</td>
<td>2666±19</td>
<td>3595±12</td>
</tr>
</tbody>
</table>
| Pure Epoxy      | 2838±51   |          |          |          |\

**Table A3: Shear Velocities in different particulates with varying volume fractions at 2.25 MHz frequency.**

<table>
<thead>
<tr>
<th>Volume Fraction</th>
<th>S22</th>
<th>S32</th>
<th>S38</th>
<th>K46</th>
<th>Solid</th>
</tr>
</thead>
<tbody>
<tr>
<td>10%</td>
<td>1256±12</td>
<td>1290±16</td>
<td>1275±5</td>
<td>1274±5</td>
<td>1315±16</td>
</tr>
<tr>
<td>20%</td>
<td>1241±16</td>
<td>1283±5</td>
<td>1306±12</td>
<td>1325±11</td>
<td>1409±13</td>
</tr>
<tr>
<td>30%</td>
<td>1274±6</td>
<td>1344±23</td>
<td>1386±8</td>
<td>1419±15</td>
<td>1487±4</td>
</tr>
<tr>
<td>40%</td>
<td>1270±9</td>
<td>1391±31</td>
<td>1436±5</td>
<td>1454±6</td>
<td>1711±11</td>
</tr>
<tr>
<td>50%</td>
<td>1379±37</td>
<td>1416±16</td>
<td>1548±19</td>
<td>1548±39</td>
<td>1833±12</td>
</tr>
</tbody>
</table>
| 60%             | 1283±22   | 1407±15   | 1550±41   | 1992±23   |\
| Pure Epoxy      | 1240±33   |          |          |          |
VITA

Phani Surya Kiran Mylavarapu was born in 1980, in Kakinada, Andhra Pradesh, India. He completed his schooling from Railway Mixed High School, Hyderabad, and intermediate education from Aditya Junior College, Kakinada, India. He joined the Department of Mechanical Engineering at Andhra University, India, in 1997 and earned a bachelor of engineering degree in May 2001. After his graduation he decided to pursue higher education in United States and enrolled in the master’s program in the Department of Mechanical Engineering at University of Missouri- Kansas City and graduated in Aug 2003.

His interest in the field of composite materials and an interest in building a career in research led him to pursue a doctoral program in the Department of Mechanical Engineering, at Louisiana State University, Baton Rouge, in August 2003. He joined Dr. Eyassu Woldesenbet, Associate Professor in the Department of Mechanical Engineering at LSU for his doctoral study. His primary area of interest for research was characterization of advanced composites using nondestructive approach in weight sensitive aerospace and marine sandwich structural applications. Phani’s research for his doctorate has resulted in over 15 publications including journal papers, conference proceedings and presentations. He realized his ambition by earning a Doctor of Philosophy degree in mechanical engineering in the Fall 2007. Phani intends to pursue his career in the research environment.