DYNAMIC MODULUS OF SYNTACTIC FOAM AND PARTICULATE COMPOSITE CORE - A NON DESTRUCTIVE APPROACH

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ABSTRACT

Developments in aviation posed requirement of lightweight, high strength and highly damage tolerant materials. Sandwich structured composites, fulfilling these requirements became the first choice for many applications including structural components for ground transport and marine vessels. Sandwich composites are a special class of composite materials which are widely used because of their high specific strength and bending stiffness. Lower density of these materials makes them well suited for marine and aerospace applications. Syntactic foams are hollow particle filled core materials that have recently emerged as attractive material for use in applications requiring low weight, low moisture absorption and high insulation properties. Quasi-static and dynamic properties of these syntactic foams are very important as these materials are used in various important structural applications in aerospace and marine industry. Even though various destructive techniques such as quasi-static compression and split Hopkinson pressure bar apparatus are used as tools to find quasi-static and dynamic modulus, there is a need for characterizing these materials non-destructively.

Several Non Destructive techniques such as Ultrasound are used in the industry to check for quality in these materials. Although Ultrasonic imaging can be used for a variety of materials, it is difficult to use this technique for porous materials and foams due to high attenuation of ultrasonic waves in air. The present study focuses on the prediction of dynamic modulus using ultrasonic testing in various types of solid particulate and hollow particle reinforced syntactic foam composites. Solid particulate and hollow particle filled syntactic foams are fabricated with three different volume fractions of 10, 30 and 60%. Longitudinal and shear wave velocities are used for calculating the dynamic modulus using the time of arrival of reflections from the sample. Effect of longitudinal attenuation behavior along with longitudinal and shear wave velocities on the varying density and volume fraction of syntactic foams is also discussed. The Pulse Echo method is used for the ultrasonic characterization of these materials.

Introduction

Syntactic foams are light-weight particulate composites made from a mixture of a polymer resin and hollow particles [1]. 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.

Syntactic foams possess attractive mechanical and physical properties such as high compressive strength [2,3], low moisture absorption [4,5] and low coefficient of thermal expansion, making them an attractive material for use in aerospace and marine applications. With increasing use of these materials in aerospace and marine applications, there is a need to optimize existing non-destructive 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 [6]. Ultrasound can be used for determining the size, shape and location of defects in a structure.

Computation of ultrasonic velocity and attenuation (loss of energy 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.
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 [7]. Longitudinal ($V_L$) and shear ($V_s$) wave velocities are used to compute the longitudinal ($L$) and shear ($G$) moduli, respectively, where,

$$L = \rho \cdot V_L^2$$  \hspace{1cm} [1]$$
$$G = \rho \cdot V_s^2$$  \hspace{1cm} [2]$$

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.

$$k = L - \frac{4}{3} G$$  \hspace{1cm} [3]$$
$$E = \frac{G(3L - 4G)}{L - G}$$  \hspace{1cm} [4]$$
$$\nu = \frac{L - 2G}{2(L - G)}$$  \hspace{1cm} [5]$$

Neither $V_L$ 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.

Attenuation

According to American Society for Nondestructive 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 [8]. 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:

$$A = A_0 \cdot e^{-\alpha X}$$  \hspace{1cm} [6]$$

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.

In this research pulse echo ultrasonic imaging is used for characterization of syntactic foams and solid particulate composites, which are used as the core materials in a sandwich structure. Hollow glass particles with varying internal radius are used to fabricate syntactic foams. Three different volume fractions of 10, 30 and 60% are used to fabricate slabs. Also, solid glass particles are used to fabricate solid particulate composites with varying volume fractions of 10, 30 and 60%. Attenuation and
velocity study is performed for characterizing foams and solid particulate composites. Further, more dynamic mechanical properties are predicted from the longitudinal and shear wave velocities computed from hollow particulate composites and solid glass particulate composites.

Experimental

Materials

Fifteen different types of syntactic foam samples and solid particulate composites are fabricated in this study. The samples are fabricated by changing the volume fraction of four different types of microballoons and one type of solid glass particle to 10, 30 and 60% respectively. The glass microballoons are supplied by 3M and have a trade name of Scotchlite. The sample nomenclature indicates the microballon type and the volume fraction. In S2230 type of sample, S22 is the microballon 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 30 type of sample, solid denotes the glass sphere used and 30 denotes the volume fraction of particles.

All types of microballoons have particle size distribution in the same range as given in Table 1. These values are provided by the manufacturer. Microballoons of type S32, S38 and K46 have the same mean particle size (outside diameter) of 40 µm, whereas S22 microballoon and solid glass particle have the same particle size of 35 µm.

Fabrication

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 volume fraction of microballoons and solid particulate composites are varied to 10, 30 and 60%, to fabricate syntactic foams and solid particulate composites respectively. The microballoons and solid glass particles were mechanically mixed individually in the matrix resin to make syntactic foam and solid particulate 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 hollow glass particulate syntactic foams, the fabricated specimens have porosity within the microballoons, called as closed cell porosity and in the matrix material due to mechanical mixing process, called open cell porosity. However, in the solid glass particulate composites, fabricated specimens have porosity only within the matrix material due to mechanical mixing process.

Density Measurement

To measure the density of the fabricated syntactic foam material standard ASTM C 271-94 [9] 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 × 25 × 12.5 mm³ dimensions. Results of density calculation of fabricated syntactic foam and solid particulates specimens are shown in Figure 1 and Figure 2. As shown in Figure 1 and Figure 2, 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.

Table 1: Microballoon size distribution and radius ratio

<table>
<thead>
<tr>
<th>Microballoon Type</th>
<th>10th percentile</th>
<th>50th percentile</th>
<th>90th percentile</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>η</th>
</tr>
</thead>
<tbody>
<tr>
<td>S22</td>
<td>20</td>
<td>35</td>
<td>60</td>
<td>220</td>
<td>0.52</td>
<td>2.76</td>
<td>0.9703</td>
</tr>
<tr>
<td>S32</td>
<td>20</td>
<td>40</td>
<td>75</td>
<td>320</td>
<td>0.88</td>
<td>13.79</td>
<td>0.9561</td>
</tr>
<tr>
<td>S38</td>
<td>15</td>
<td>40</td>
<td>75</td>
<td>380</td>
<td>1.05</td>
<td>27.58</td>
<td>0.9474</td>
</tr>
<tr>
<td>K46</td>
<td>15</td>
<td>40</td>
<td>70</td>
<td>460</td>
<td>1.29</td>
<td>41.37</td>
<td>0.9356</td>
</tr>
</tbody>
</table>
Ultrasonic Testing

Ultrasonic imaging [UI] of syntactic foam and solid particulate composite specimens are carried out using Physical Acoustic Corporation’s water immersion type system UltraPACTM with UltrawinTM 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. Frequency of 1 MHz is used for longitudinal wave characterization and a frequency of 2.25 MHz is used for the shear wave characterization of all the samples. 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.

Results

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 3 and Figure 4 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 [10] is used for computing the apparent attenuation in these foam and particulate composite samples. The apparent attenuation is computed by the Equation 7.

\[
\text{Apparent attenuation} = 20 \log_{10} \frac{A_m}{A_n} \times \frac{1}{2(n-m)T} \quad [7]
\]

where,

\[A_m\] and \[A_n\] = amplitudes of the \(m^{th}\) and \(n^{th}\) back reflections \((n>m)\), and \(T = \text{specimen thickness.}\)

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 calculation 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 [11, 12]. Therefore, the attenuation coefficient is calculated by taking the ratio of first and third back wall echoes using a 1 MHz transducer.

Attenuation coefficient values computed from four different types of syntactic foam and solid particulate composite samples are given in Table 2. From Table 2, it is evident that the attenuation coefficient values increases from 10% to 30% and decreases from 30%-60% volume fraction. This increase in attenuation coefficient is due to the reason of increased number of particles from 10-30% volume fraction and thus scattering of ultrasonic energy increases. Due to more scattering of ultrasonic energy, the attenuation increases. But, from 30-60% volume fraction, the wave propagation characteristics change, thereby decreasing the attenuation coefficient. After computation of attenuation coefficient in syntactic foam and solid particulate composites, the longitudinal and shear wave velocities are calculated for predicting dynamic modulus and Poisson’s ratio.

![Figure 3: Amplitude vs. time plot of SF2230 sample showing clear signal in time domain.](image)

![Figure 4: Amplitude vs time plot of Solid particulate at 30% volume fraction sample.](image)
Table 2: Attenuation Coefficients in different particulates with varying volume fractions at 1MHz frequency

<table>
<thead>
<tr>
<th>Volume Fraction</th>
<th>Attenuation Coefficient, 10^3 db/mm</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>S22</td>
</tr>
<tr>
<td>10%</td>
<td>245.7±18.6</td>
</tr>
<tr>
<td>30%</td>
<td>291.9±53.5</td>
</tr>
<tr>
<td>60%</td>
<td>201.8±10.1</td>
</tr>
<tr>
<td>Pure Epoxy</td>
<td>222.3±11.1</td>
</tr>
</tbody>
</table>

According to ASTM E 494-95 [14], 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 5. In Figure 5, 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. 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.

![Figure 5: Shift of peaks due to attenuation in particulate composites](image)

Table 3: Longitudinal Velocities in different particulates with varying volume fractions at 1 MHz frequency

<table>
<thead>
<tr>
<th>Volume Fraction</th>
<th>Longitudinal Velocity, m/s</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>S22</td>
</tr>
<tr>
<td>10%</td>
<td>2489±32</td>
</tr>
<tr>
<td>30%</td>
<td>2421±46</td>
</tr>
<tr>
<td>60%</td>
<td>2296±10</td>
</tr>
<tr>
<td>Pure Epoxy</td>
<td>2838±51</td>
</tr>
</tbody>
</table>

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. Table 3 shows the longitudinal wave velocities in different types of syntactic foam samples and particulate composites subjected to longitudinal ultrasonic waves of 1 MHz frequency. Immersion type of testing is used for the longitudinal scans using 1 MHz transducer frequency. The ultrasonic velocities in syntactic foams are found to be in the range of 2200-2900 m/s as shown in Table 3. From Table 3, 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 1. Thus, longitudinal velocity in syntactic foams decreases with an increase in volume fraction.
From Table 3, 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.

Also, from Table 3 it is evident that the ultrasonic longitudinal velocity in solid particulate composites increases with the volume fraction at frequencies of 1 MHz. This increase in velocity is due to the reason that with an increase in volume fraction, the density of these solid particulate composites increases as shown in Figure 2 and thus increasing the longitudinal velocity. Shear wave velocities computed from syntactic foams and particulates composites with an increase in volume fraction is shown in Table 4. 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.

Table 4: Shear Velocities in different particulates with varying volume fractions at 2.25 MHz frequency

<table>
<thead>
<tr>
<th>Volume Fraction</th>
<th>Shear Velocity, m/s</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>S22</td>
</tr>
<tr>
<td>10%</td>
<td>1256±12</td>
</tr>
<tr>
<td>30%</td>
<td>1274±6</td>
</tr>
<tr>
<td>60%</td>
<td>1283±22</td>
</tr>
<tr>
<td>Pure Epoxy</td>
<td>1240±33</td>
</tr>
</tbody>
</table>

**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 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 [15-18].

Longitudinal Velocity,

$$V_L = \sqrt{\frac{\lambda + 2\mu}{\rho}} \quad [8]$$

Shear Wave Velocity,

$$V_S = \sqrt{\frac{\mu}{\rho}} \quad [9]$$

Poisson’s ratio,

$$\nu = \frac{\lambda}{2(\lambda + \mu)} \quad [10]$$

Finally, modulus can be computed using,

$$E = \frac{V_L^2 \rho (1 + \nu) (1 - 2\nu)}{1 - \nu} \times \frac{0.733}{6.89 \times 10^4} \times 0.00689 \text{ Mpa} \quad [11]$$

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 and quasi-static testing 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 8-11, dynamic modulus in syntactic foams is predicted as shown in Table 5.
Table 5: Dynamic modulus values of syntactic foams and solid particulate composites predicted using UI

<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>2156</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>S3210</td>
<td>0.32±0.00</td>
<td>4071 ±45</td>
<td>3048-4099</td>
<td>2310</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>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>2318</td>
</tr>
<tr>
<td>S3830</td>
<td>0.32±0.00</td>
<td>3718 ±73</td>
<td>3008-3661</td>
<td>2326</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 ±45</td>
<td>3698-4722</td>
<td>2386</td>
</tr>
<tr>
<td>K4630</td>
<td>0.32±0.01</td>
<td>3953 ±71</td>
<td>3590-4182</td>
<td>2508</td>
</tr>
<tr>
<td>Solid 10</td>
<td>0.33±0.00</td>
<td>5372±76</td>
<td>5285-5866</td>
<td>2718</td>
</tr>
<tr>
<td>Solid 30</td>
<td>0.30±0.00</td>
<td>7480±113</td>
<td>6250-8067</td>
<td>3514</td>
</tr>
<tr>
<td>Solid 60</td>
<td>0.27±0.00</td>
<td>14016±183</td>
<td>11394-14431</td>
<td>4708</td>
</tr>
</tbody>
</table>

Conclusions

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 fractions of 10, 30 and 60%. Particulate composites are fabricated using one type of solid glass sphere with varying volume fraction from 10, 30 and 60%. Attenuation coefficient and longitudinal velocities are calculated for varying volume fractions for each of the particle (microballoon/solid particulate) type. It was found that that attenuation coefficient increase from 10-30% and decrease from 30%-60% in each of the types of syntactic foams and particulate composite. Increase and decrease in attenuation coefficient is due to the reason that the scattering of ultrasonic energy increases from 10-30% volume fraction, whereas the wave propagation characteristics change 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 is calculated using UI. This is the first time that a relationship is established between the modulus obtained from UI and material modulus.

Acknowledgments

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