EVALUATION OF POROSITY IN GRAPHITE-EPOXY COMPOSITE BY FREQUENCY DEPENDENCE OF ULTRASONIC ATTENUATION

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INTRODUCTION

The ultrasonic attenuation of a solid containing a distribution of voids depends on the number of voids per unit volume and the ultrasonic scattering cross-section of the voids. This physical picture has been shown to provide an accurate description of the attenuation behavior of ultrasound in metals containing low level porosity. For example, methods developed from this model for evaluating the average size and the volume fraction of porosity in cast aluminum have yielded good results [1].

Fiber reinforced composite laminates, such as the graphite-epoxy system, are structurally more complex than a homogeneous metal and consequently the morphology of the porosity in composites is also characteristically different from, say, gas porosity in cast aluminum. Pores in metals are generally more spherical whereas voids in composite laminates tend to occur at the interface between the plies and are generally flattened and elongated along the axial direction of adjacent fibers [2]. In composites the distribution of the geometric dimensions of the pores spans a much wider range than pores in metals. The frequency dependence of the ultrasonic attenuation also appears to be different in the two cases. The attenuation due to porosity in cast aluminum displays a power law behavior in the Rayleigh regime, then rises monotonically and reaches a saturation plateau at higher frequencies. In graphite-epoxy laminates, most experimentally obtained attenuation may be regarded as approximately linear with frequency up to 10MHz or so. Investigation at higher frequencies are difficult due to the high attenuation and experimental data are generally unavailable. In any case, the presence of porosity in composite laminates drastically increases the attenuation level to more than 100 dB/cm at a few percent voids by volume. Several studies relating the ultrasonic attenuation to porosity content may be found in the literature [3-5].

In this work we explore the correlation between the porosity volume fraction and the frequency dependence of the attenuation in graphite-epoxy composites. Based on a study of the morphology of the pores in graphite-epoxy laminates, the pores are approximated by long cylindrical voids with elliptical cross-section. The ratio of the pore length to its cross-sectional dimensions is usually quite large. A model is therefore developed
for the frequency dependent attenuation due to the scattering by long cylindrical pores with an elliptical cross-section. The slope of the attenuation \( \frac{da}{df} \) of the approximately linear section of the \( a \) versus \( f \) curve is found to be proportional to the volume fraction of the pores and the proportionality constants are material property dependent. Using this theoretical result, the porosity volume fractions of a set of graphite-epoxy laminates containing increasing amounts of porosity are computed from the measured attenuation slope. The predicted volume fractions are in reasonably good agreement with the volume fractions obtained by destructive means, i.e., acid digestion and microscopy/image analysis.

In this work the attenuation as a function of frequency is measured with a broadband technique. Energy loss mechanisms not associated with porosity, such as beam spreading, interface losses and frequency response of the broadband transducer, are accounted for by measurement-model based algorithms and by using appropriate measurement configurations. Correlation between the porosity volume fraction and spectral features of the ultrasonic pulse, namely, the spectral peak amplitude and the centroid frequency shift, is also observed. Since the origin of the centroid frequency shift is the frequency dependence of the attenuation, the frequency shift is related to the attenuation slope via some constants. This relationship is also verified experimentally.

A Simple Model of Longitudinal Wave Attenuation Due to Cylindrical Pores

In this section the essence of the simple model developed for the attenuation of longitudinal waves propagating perpendicular to the composite laminate (and hence normal to the plies and fibers in the laminate) is described. The pores are assumed to be long cylindrical voids with an elliptical cross-section. Because of the configuration of the pores, the incident longitudinal wave is normal to the length of the pore and also to the major axis of its cross-section. We follow the attenuation model for porosities in an isotropic medium and write

\[
\alpha = \frac{1}{2} n \gamma
\]  

where \( n \) is the number of pores per unit volume and \( \gamma \) is the total scattering cross-section of a pore. Several comments should be made here: 1) for a distribution of pores sizes, the attenuation should be \( \alpha = \frac{1}{2} \sum n_i \gamma_i \) where the summation is for the different sizes. However, in our subsequent development of the model, we shall use the average pore features such as the cross-sectional aspect ratio in computing the scattering cross-section and drop the summation notation; 2) Since we approximate the pores as infinitely long cylindrical voids, the problem is reduced to a two-dimensional situation. The number density \( n \) then becomes the number of pores per unit area in the cross-sectional plane and \( \gamma \) is then the scattering cross-section per unit length; 3) Although the model is for the 2-D situation; but, to make the physical meaning more transparent, we shall carry the pore length \( \ell \) in the following equations to show the origin of the volume fraction term.

For computational convenience we shall use the reduced scattering cross-section \( \Gamma(k_b) \), defined by

\[
\Gamma(k_b) = \frac{\gamma(k_b)}{4b\ell}
\]  

1186
where $b$ is the semi-major axis of the pore cross-section and $\ell$ is the pore length. The reduced cross-section (per unit length) of a long cylindrical cavity with different cross-sectional aspect ratio $(b/a)$ are then computed for normal incident P waves using a boundary integral method [6]. Figure 1 shows the computed results for three aspect ratios, including the circular cylinder case for comparison.

Since the experimentally measured attenuation in graphite-epoxy laminates containing porosity is approximately linear, we expand the reduced scattering cross-section about its inflection point (the most linear section) and proceed to calculate the slope of the attenuation with respect to frequency.

$$\Gamma(kb) = \text{const.} + kb \Gamma'$$

(3)

where $\Gamma'$ is the slope of the curves in Fig. 1, or $d\Gamma(kb)/dkb$, at the inflection point. The value of $\Gamma'$ depends on the aspect ratio $b/a$ and the velocity ratio $V_S/V_L$. Substituting (3) and (2) into (1), we have

$$a = \text{const.} + \frac{4}{V_L} \left[ n\pi ab\ell \right] (b/a) \Gamma'$$

(4)

Since the terms in the brackets are equal to the volume fraction $c$, we obtain, upon differentiating Eq. (4), the attenuation slope:

$$\frac{da}{df} = \left( \frac{4c}{V_L} \right) (b/a) \Gamma'$$

(5)
The porosity volume fraction is thus related to the attenuation slope via material and pore characteristics dependent constants $V_L$, $b/a$ and $\Gamma'$:

$$c = \left(\frac{V_L}{4\Gamma'}\right)(a/b) \frac{da}{df} \quad (6)$$

**EXPERIMENTAL METHOD**

**Samples**

The set of samples used in this investigation are eight 16-ply graphite-epoxy laminates fabricated by Rohr Industries. They were fabricated specifically to contain various amounts of porosity. Four of the laminates are unidirectional and the other four have a quasi-isotropic [±45/0/90]$_{2S}$ layup. The porosity volume fractions determined by the manufacturer using acid digestion method are given in Table 1. The coupons are 4"x4" and typically 0.1" thick.

<table>
<thead>
<tr>
<th>Sample No.</th>
<th>Layup</th>
<th>Porosity Volume Fraction (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A5</td>
<td>unidirectional</td>
<td>0.2</td>
</tr>
<tr>
<td>A4</td>
<td>unidirectional</td>
<td>1.14</td>
</tr>
<tr>
<td>A2</td>
<td>unidirectional</td>
<td>2.04</td>
</tr>
<tr>
<td>A1</td>
<td>unidirectional</td>
<td>6.51</td>
</tr>
<tr>
<td>B5</td>
<td>quasi-isotropic</td>
<td>0.34</td>
</tr>
<tr>
<td>B4</td>
<td>quasi-isotropic</td>
<td>1.25</td>
</tr>
<tr>
<td>B2</td>
<td>quasi-isotropic</td>
<td>2.82</td>
</tr>
<tr>
<td>B1</td>
<td>quasi-isotropic</td>
<td>4.05</td>
</tr>
</tbody>
</table>

Most of the attenuation measurements for developing and verifying the porosity volume fraction evaluation method in this work are made on this set of eight samples. The method is also tried on another set of graphite-epoxy laminates from a different source. These are typically 4"x9" 64-ply panels with porous regions containing <1% void by volume.

**Attenuation Measurement**

Ultrasonic attenuation is measured using 10MHz 1/4" diameter unfocussed immersion transducers in both pulse-echo and through-transmission modes. In pulse-echo measurements the transducer is typically 7.5cm from the sample, and in through-transmission measurements the transducers are separated by a distance of 15cm. The transducers are driven with a broadband spike voltage and the sound propagation direction is perpendicular to the laminate. The attenuation is obtained by comparing the front and back surface echoes in the pulse-echo mode and by a substitution method in the through-transmission mode where the pulse through water only and the pulse through the sample are compared. In both configurations the signals are processed in such a way that signal reduction mechanisms not associated with the presence of porosity are accounted for [7]. These include beam spreading effects, interface transmission/reflection losses, transducer bandwidth effects and background attenuation in porosity free laminates. Since most of these corrections are frequency dependent,
they are performed in the frequency domain. As a result, the ultrasonic attenuation is obtained as a function of frequency over the entire bandwidth of the transducer in a single pulsed measurement. Special attention is given to the slope of the attenuation with respect to frequency \((\text{df/d}a)\). Due to the spatial inhomogeneities in the porosity distribution, the \(a\) versus \(f\) curves obtained at different locations of the laminate often show considerable fluctuation. To obtain an average value of \(\text{df/d}a\) for correlation with the acid digestion determined porosity content, spatial averages over five to ten locations of the test piece are usually made.

To verify the accuracy of the measurement technique, we first measured the attenuation of a thin slab (0.1") of polyethylene—a material with established attenuation value and a linear frequency dependence. Excellent agreements with published results were obtained.

RESULTS

The measured attenuation slope is plotted as a function of the void content (based on acid digestion) in Fig. 2. Results are presented for seven samples since the 6.5% sample was highly attenuative and no reliable data were obtained from it. Both the unidirectional and the quasi-isotropic samples seem to behave similarly and the data points define an approximate straight line. These data were obtained with a substitution method in the through-transmission mode using the pulse passed through only water as the reference signal. The intercept on the ordinate \((0.5\text{Np/cmMHz})\) may be regarded as the background attenuation slope of a void-free sample and the dashed line in Fig. 2 is therefore the porosity-induced attenuation slope. To compute the porosity volume fraction from the measured attenuation slope, we use Eq. (6) and \(V_L = 0.32/\text{cm/\mu s}, a/b = 0.5,\) and \(\Gamma' = 2.45.\)

![Fig. 2. Correlation between void content and the total attenuation slope (solid line, including the background of void-free material) and the porosity-induced attenuation slope (dashed line) in graphite-epoxy laminates.](image-url)
Table 2. Porosity volume fraction deduced from attenuation slope.

<table>
<thead>
<tr>
<th>Sample</th>
<th>(da/dv)\textsubscript{porosity} (Np/cm\textsubscript{MHz})</th>
<th>Volume Fraction Computed from Eq. (6)</th>
<th>Volume Fraction by Acid Digestion</th>
</tr>
</thead>
<tbody>
<tr>
<td>B1</td>
<td>2.07</td>
<td>3.4%</td>
<td>4.05%</td>
</tr>
<tr>
<td>B2</td>
<td>1.52</td>
<td>2.5</td>
<td>2.82</td>
</tr>
<tr>
<td>A2</td>
<td>1.12</td>
<td>1.8</td>
<td>2.04</td>
</tr>
<tr>
<td>B4</td>
<td>0.68</td>
<td>1.1</td>
<td>1.25</td>
</tr>
<tr>
<td>A4</td>
<td>0.49</td>
<td>0.8</td>
<td>1.14</td>
</tr>
<tr>
<td>B5</td>
<td>0.12</td>
<td>0.2</td>
<td>0.32</td>
</tr>
<tr>
<td>A5</td>
<td>0.28</td>
<td>0.5</td>
<td>0.2</td>
</tr>
</tbody>
</table>

The results are listed in Table 2 and compared to the volume fraction from acid digestion. We found that a value of 0.5 for the a/b ratio (somewhat greater than that obtained from image analysis) provided a reasonably good agreement between the last two columns in Table 2. The value of \( \Gamma' \) is the slope of the reduced scattering cross-section at its inflection point for a/b = 0.5 and \( V_S/V_L = 0.56 \) (curve B, Fig. 1).

The presence of porosity not only changes the attenuation slope, but also affects the spectral content of the broadband pulse. An inspection of the frequency spectra of the ultrasonic pulses transmitted through sample containing increasing amount of porosity (shown in Fig. 3) reveals that the centroid frequency decreases monotonically. The centroid frequency \( \langle f \rangle \) is defined as

\[
\langle f \rangle = \frac{\int f |A(f, c)|^2 \, df}{\int |A(f, c)|^2 \, df}
\]

(7)

The shift of the centroid frequency, \( \Delta \langle f \rangle \), when normalized by the centroid frequency of the void-free specimen \( \langle f_0 \rangle \), shows a linear correlation with the void content. The correlation between \( \Delta \langle f \rangle / \langle f_0 \rangle \) does not depend on the particular transducer frequency bandwidth, as shown by the results in Fig. 4 obtained with two transducers of center frequencies of 5MHz and 10MHz, respectively. The physical mechanism for the centroid frequency shift is, of course, the frequency dependence of the attenuation and that the higher frequencies suffer a greater attenuation than the lower frequencies. The centroid frequency shift and the attenuation slope are related. By assuming a linear frequency dependence of the attenuation and Gaussian spectrum of the pulse, one can invoke the theory of spectral moments \([7]\) and show that

\[
\frac{d}{dc} \left[ \frac{\Delta \langle f \rangle}{2z\sigma^2} \right] = \frac{d}{dc} \left[ \frac{da}{dv} \right]
\]

(8)

where \( z \) is the sample thickness and \( \sigma \) is the spectral variance. In Fig. 5, \( \Delta \langle f \rangle /2z\sigma^2 \) of the same seven samples is shown against the void content. A comparison of dashed lines in Figs. 2 and 5 shows that Eq. (8) indeed holds for the experimental results. The shift of the centroid frequency can therefore serve as an alternative method for estimating the porosity volume fraction.
Fig. 3. Frequency spectra of the ultrasonic pulse transmitted through water only (no sample) and graphite-epoxy laminates containing increasing amounts of porosity. The void-free specimen was from a different source and its spectrum is plotted as a dashed line.

Fig. 4. Correlation between void content and the normalized centroid frequency shift. $<f_0>$ is the unshifted centroid frequency of a void-free specimen. Notice the similarity of the data obtained with a 5MHz transducer and a 10MHz transducer.
Fig. 5. Correlation between void content and the shift in centroid frequency $\Delta<\nu>/2z\sigma^2$. The dashed line (parallel to the solid line and goes through the origin) is the porosity contribution.

DISCUSSION

Using a set of graphite-epoxy laminates containing various amounts of porosity, we have demonstrated the correlation between the attenuation slope, the centroid frequency shift, and the void content. Furthermore, we have interpreted the experimental results quantitatively using the model of cylindrical pores. The results indicate that the relationship between the void content and the attenuation slope is basically correct. However, being a simplified model and neglecting the statistical aspects of the pore parameters, Eq. (6) has its limitations in accurately predicting the void content. For example, it contains constants $(a/b$ and $\Gamma')$ pertaining to averaged pore characteristics. On the other hand, from an applications viewpoint, the interesting question is how well does Fig. 2 (and Fig. 5) hold as calibration curves for graphite-epoxy laminates from different sources and manufactured under different conditions.

We have so far tested our methods on one other set of specimens that are 64-ply and contain different numbers of $0, \pm 45^\circ$, and $90^\circ$ plies. By measuring the attenuation slope and the frequency shift of a porous region on one panel and using a nonporous region as the reference signal, void contents of 0.8% and 0.9% were predicted by the dashed lines of Figs. 2 and 5, respectively. Using the pulse passed through water as the reference signal, Fig. 5 (solid line) predicted 0.7% for the same region. The porous region was subsequently cut out and its void content determined by density measurement, the result was 0.9-1%. On another panel ultrasonic measurements yielded a void content in the range of 0.4 - 0.7%, followed by a density determination that showed 0.5% void. Although the limited testing made to date proved successful, more extensive tests are clearly needed.
ACKNOWLEDGEMENT

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REFERENCES