ULTRASONIC CHARACTERIZATION OF POROSITY IN COMPOSITE
MATERIALS BY TIME DELAY SPECTROMETRY

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INTRODUCTION

The presence of porosity in a wide range of materials, whether ceramics, steel or fiber reinforced composites, has a dramatic effect on the strength and mechanical properties of that material. Therefore, the presence of any porosity in a composite laminate during the manufacture of aerospace components is a basis for component rejection.

This paper describes research being conducted to evaluate the effects of porosity on an ultrasonic field so that an accurate quantitative assessment of porosity can be made.

Porous study specimens with controlled void volume percent, bubble size and distribution have been produced using careful moisture and thermal management techniques. Measurements of ultrasonic attenuation have been made at multiple frequency values utilizing Time Delay Spectrometry (TDS).

Existing models have been used to calculate apparent porosity volume percentages and pore sizes from the ultrasonic data. Suitability of the models for NDT purposes is discussed.

APPROACH

To extract porosity values from absolute ultrasonic measurements, it is necessary to define the multiple and complex interaction of ultrasound with voids, matrix material, and fibers. Since the pore size can easily be on the order of the wave length, scattering becomes the dominant mechanism. Exploiting this behavior, ultrasonic attenuation is the measurement of choice and is the focus of this experimental work. While it has been recognized that velocity measurements could also be useful for porosity characterization, this research was restricted in scope to measurements of attenuation.

Attenuation has several sources: Reflection factor losses, diffraction losses, hysteresis losses, mechanical damping losses and scattering losses. Clearly, a model was needed to accurately predict these interactions. As yet, no model exists to deal with the most complex cases. However, an excellent analytical formulations of attenuation have been suggested for scatterers placed in a homogeneous medium[1, 2, 3, 4].

These works suggests that by extracting multiple measurements of attenuation at discrete wavelength values in the Rayleigh regime, a value of the characteristic porosity parameters can be deduced. Utilizing measurements in the Rayleigh region marks the uniqueness of this approach. Estimating the porosity by calculating the slope of the Rayleigh scattering behavior allows the discountance of extraneous sources of attenuation not within control of the experimenter. This includes, for example, reflection factor losses and nonoptimum coupling. The concept is illustrated in Figure 1, where poor coupling, for example, may lead to excessive estimates of porosity content when using the short wavelength approximator.

In this paper experiments are discussed in which neat resins were used as host media for porosity ranging from 0 to 22 percent in volume. Measurements of attenuation were made at discrete frequencies, and pore diameter and porosity volume calculated.

SPECIMEN PREPARATION

Although porosity cannotes a very specific defect, there are, in fact a wide variety of porosity formations which may occur in a material, and which require variations in the approach to ultrasonic evaluation. A total discussion of porosity classifications and its impact on materials is contained in Reference 5. The defect of interest in this research is that encountered most commonly in production graphite epoxy laminates. This can be described as a continuous solid phase, isolated pore phase, structure with nearly spherical interlaminar pores, which are considerably larger than other system constituents (fibers).
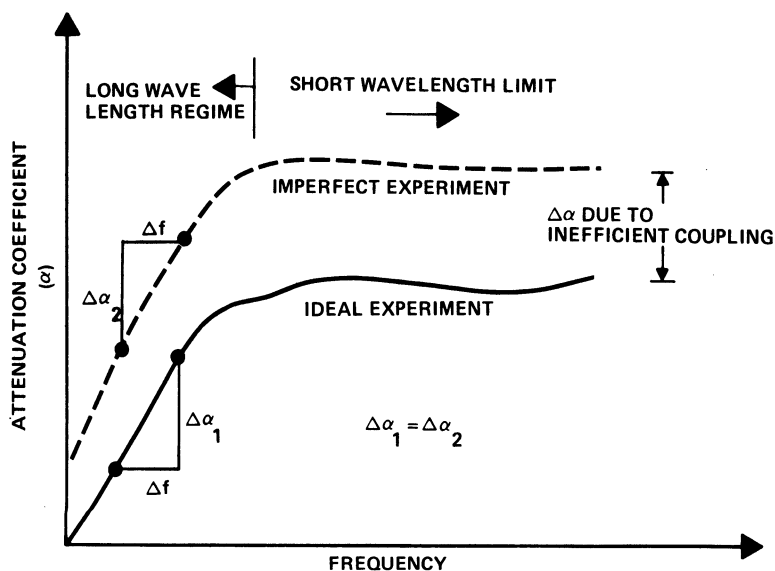

Figure 1. Porosity Characterization by Measurements in the Rayleigh Region Eliminates Effects of Imperfect Experiments

The porosity samples were manufactured using polyetherimide, a translusent thermoplastic resin supplied by General Electric (Ultem 1000 research grade). Although this work utilizes a thermoplastic resin system, the results are general and apply to all standard epoxy systems currently in use. It was also desirable to select a material that could dissolve and maintain lighter volatiles, and then, by virtue of its thermoplastic properties, allow controlled bubble formation at elevated temperatures. A translucent material was required so that porosity distribution and overall uniformity could be visually confirmed without requiring destructive sectioning. The details of specimen manufacture are interesting and are the subject of reference 6. They will not be discussed further here.

DETERMINATION OF POROSITY CHARACTER

The independent (nonultrasonic) characterization of porosity was an important consideration. The total porosity content was determined by comparison of the bulk density to the true density of the material.

Density measurements were obtained by determining their weight in water in conjunction with the Archimedes method. The true density was determined for porosity free samples in the same manner as porous samples. The average measured value for pore free material was 1.286 g/cm^3.

Pore size was obtained optically with a calibrated gradicule. Forty measurements were obtained for each sample, averaged and the deviations obtained (accuracy 1/10,000 inch). Table I describes the samples and their porosity values and pore size.

DATA ACQUISITION AND ANALYSIS

The parameter of intrest in these studies is ultrasonic attenuation. The parameter (attenuation coefficient) must be obtained indirectly by the comparison of two ultrasonic amplitudes, each corresponding to a specific time of flight in a material. If a measurement of the difference in signal amplitude A(f) and A_o(f) can be made, then α(f) can be calculated directly. Time Delay spectrometry was the technique used in this work. TDS is a multifrequency analog technique capable of accurate measurements in near real time. It is currently under development at the Naval Surface Weapons Center, White Oak, Maryland.[7] With this technique, only a single pair of transducers is required, and an exceptionally large band width is obtainable (20 MHz).

Time delay spectrometer primarily consists of a swept frequency source and a tracking filter. The tracking filter is comprised of a mixer and a filter which typically has a variable band width and fixed frequency. Signals arrive at the receiver with a time delay that depends on the pathlength and the propagation velocity through a line of sight. Since the transmitter frequency is swept, the time delay is equivalent to a frequency offset. Now, the analytic signal and the energy spectrum can be obtained easily.

The TDS system provides a direct measure of energy vs. frequency without numerical transform technique. The TDS has several advantages over traditional pulsed methods for this work. They are, for example, the very large dynamic range (120 dB), the great increases in noise tolerance, and the signal-to-noise ratio.

A through transmission approach was coupled with this technique to calculate attenuation. Waterpath spectra and sample spectra were

Table I. Nonultrasonic Characterization of Porosity Samples

Sample Designation	Porosity (%)	Mean Void Dia. x (in.)	T (in.)	Fabrication* Designation	Width (cm)
A	11.350	0.033	0.031	T4	1.30
B	0.311	0.000	0.000	DE7	1.08
C	5.365	0.021	0.005	T_2	1.30
D	8.243	0.017	0.007	DE3	1.30
E	5.000	0.025	0.023	DE5	1.24
F	0.000	0.000	0.000	SD1	0.30
G	0.000	0.000	0.000	SD2	0.49
H	0.311	---	---	SD3	1.29
I	0.311	0.009	0.002	SD4	1.21
J	0.777	0.022	0.008	SD5	1.30
K	1.400	0.015	0.006	SD6	1.30
L	0.622	0.013	0.003	SD7	1.20
M	3.810	0.011	0.003	SD8	1.35
N	0.000	0.000	0.000	SD9	1.28
O	22.110	0.026	0.021	DE2	1.25
P	0.311	0.005	0.002	SD10	1.28
Q	3.344	0.015	0.013	DE6	1.31
R	18.420	0.017	0.007	DE9	1.44
S	8.130	0.012	0.005	UNKNOWN	---
T	3.610	0.029	0.004	NT1	---
U	6.610	0.016	0.004	NT2	---
V	3.110	0.017	---	T_1	1.51
W	7.510	0.027	---	T_3	1.26

*Fabrication Designation:
DE - Extended dry series
SD - Standard dry 168 hours series
T - Time variation at nucleation temp. series
NT - Nucleation in temperature variation series

obtained, and the suitable deconvolutions performed. Use of logarithmic amplifiers and displays simplified this calculation greatly. Data were acquired using 2.25 and 10 MHz transducers for each of 23 samples. First data were taken through a sample-free water path. The amplitude vs. frequency data were displayed in a logarithmic fashion referenced to 1 mw terminated into 50 ohms. Next, samples of varying thickness and porosity level were placed between the transducers and data acquired. Subtraction of the sample spectra from the water path spectra yields insertion loss, which is total attenuation in addition to reflection losses at the surface. The attenuation coefficient for each porous sample was calculated by correcting for surface losses.

In addition to sample attenuation, the attenuation attributed solely to the presence of porosity was calculated from these experimental data, and is the variable of interest.

CALCULATION OF POROSITY PARAMETERS FROM DATA

To extract actual values of pore size and volume fraction, the treatment of Gubernatis and Domany [8] was used. This treatment allows the enrichment of the original semiempirical model to accommodate

pore size and the volume fraction of pores. The attenuation can be expressed in terms of scattering only as

$$\alpha(f) = 3\pi \, C\left(\frac{D}{2}\right)^3 \beta \, f^4 \, (8.68).$$

(1)

where β is a constant which depends strictly on the moduli of the material, C is the volume fraction and D is the pore diameter. 8.68 is a constant conversion factor which allows $\alpha(f)$ to be expressed in dB instead of nepers.

$$\alpha(f) = 7.42 \, \frac{C}{D}$$

(2)

as $\beta \, f^4 \left(\frac{D}{2}\right)^4$ converges to a constant value.

It was possible to make several estimations of pore size and volume fraction using these enhanced expressions. An ultrasonic estimate of pore size was possible by noting the frequency of the characteristic bend in attenuation behavior. The frequency of this transition can be theoretically calculated for the general case of gas filled pores.[9]

The transition frequency is located at the maximum reduced cross section or $\frac{KD}{2} = 1.45$ for this material where k is the wave number. Pore size can be deduced from an individual experiment by noting the frequency of the transition point and solving the above equation for D.

The volume fraction of porosity can now be calculated from either Equation 1 or Equation 2.

Pore size has been calculated using this technique for all suitable test samples. In considering the result all ultrasonic estimates are well within the deviation of the optically determined diameter data. For the most part, pore sizes are overestimated as would be expected. Errors in size estimates can be attributed to several sources, primarily the distribution of actual pore diameters. Large dispersion in actual pore size tends to make the transition point at $\frac{KD}{2} = 1.45$ less distinct and therefore quite difficult to nonarbitrarily assign a frequency value.

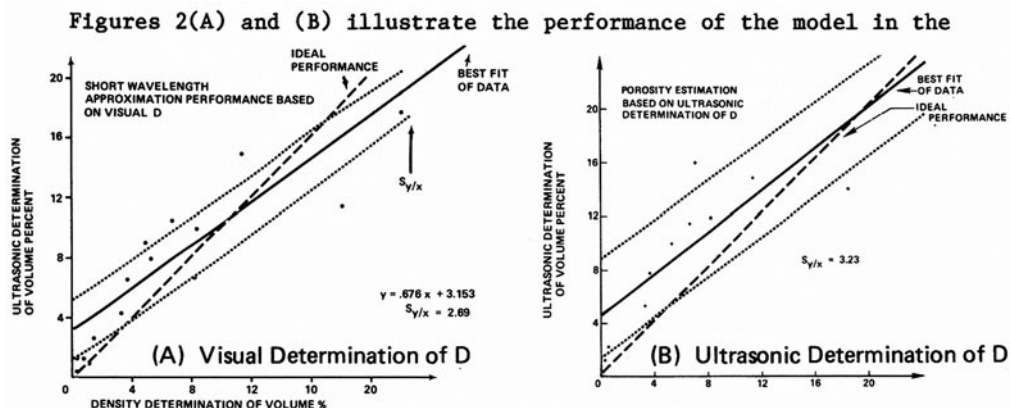Figures 2(A) and (B) illustrate the performance of the model in the

Figure 2. Performance of Short Wavelength Approximation

short wavelength regime, assuming that volume fractions obtained by density measurement are accurate. The ultrasonic estimates are generally higher with a deviation of 2.7 percent for optically based calculations and 3.2 for ultrasonically based calculation.

The performance in fact is probably much better. By restricting interest to volume fractions less than 6 percent, deviations less then 2 percent can be realized. In addition, much error can be associated with the inability to accurately determine the true volume fraction by density measurement. This also holds for long wavelength estimates.

The source of the error is as follows: First some samples are not uniformly porous; instead, a large portion of the sample is virgin resin surrounding a portion containing porosity from which data is acquired. Initial calculations indicate that some values may change by as much as 50 percent. Selective trimming will provide a better estimate of performance. Second, variations in the density of the virgin material itself restrict the accuracy of density derived volume fractions. Lastly, absorbed moisture has been shown to change overall weight of the sample thereby casting an additional uncertainty on the measured volume fraction. Alternate methods of volume fraction determination will help clear the performance picture.

Figures 3(A) and (B) display the results of estimating the volume fraction of the samples in the Rayleigh regime. Since the attenuation varies greatly in this region (equation 1) it is possible to obtain a better estimate of the volume fraction than in the short wavelength limit. This has been done by obtaining the slope of the attenuation behavior in the Rayleigh region. An estimate of the volume fraction was obtained by using the following expression.

$$C = \frac{D(\alpha_1 - \alpha_2)}{5.13(\Gamma_1 - \Gamma_2)} \tag{3}$$

where Γ represents the reduced cross section of a pore and is given by

$$\text{is} \quad \Gamma = \beta K^4 \left(\frac{D}{2}\right)^4 \tag{4}$$
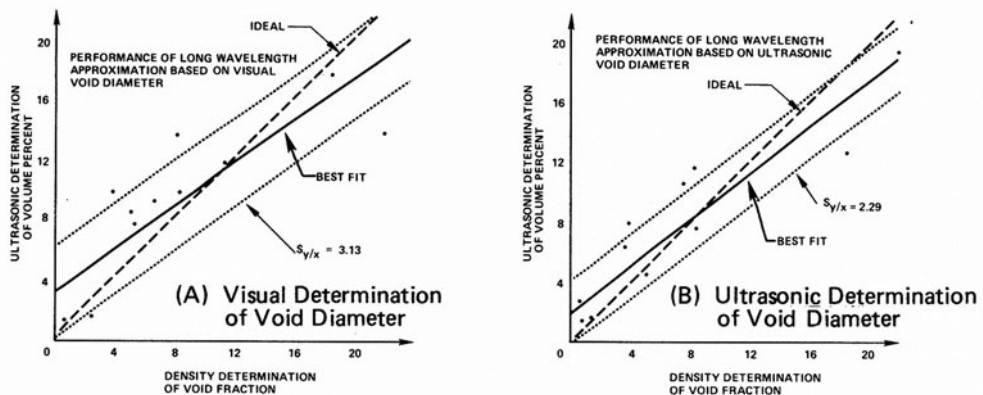
in this frequency range.

Figure 3. Long Wavelength Approximation Performance Based on Visual and Ultrasonic Void Diameters

This method is desirable over the short wavelength approximation as all physical conditions at the time of the measurement may not be at the disposal of the analyst.

As can be seen, the performance is enhanced when considering porosity values less than 6 percent. A deviation of 1.6 percent can be obtained which is well within the confidence limits estimated on the density determined volume fraction. The same sample errors exist, as already discussed.

SUMMARY

In summary, ultrasonic quantification of porosity by ultrasonic means has been shown possible. In some instances, the ultrasonic estimates are probably more accurate than those obtained by density determination. Restricting interest to less than 6 percent improves performance and is reasonable considering the application is to NDE of composites. Best results were obtained by estimating porosity from the Rayleigh scattering with an ultrasonically determined pore diameter. Large variations from the mean in pore diameter complicate the accurate sizing of pores and requires a more sophisticated treatment than used here. Too, the technique is quite sensitive to the void diameter, considering the absolute size of the pore, factors of 2 in the attenuation can result from a few thousandths change in void diameter.

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