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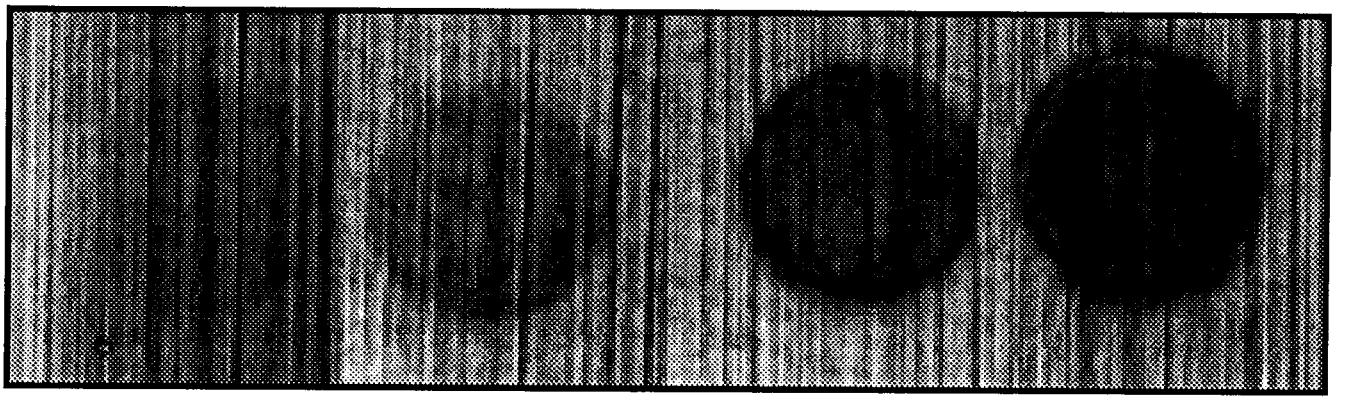
This report covers the work done on NASA grant NAG-1-431 for the period from November 1, 1987 to October 31, 1988. The major work completed during this period was an investigation of the temperature dependence of the ultrasonic properties of AS4/lexan and XAS/lexan composites. The results of this research was presented at the Fifteenth Annual Review of Progress in Quantitative Nondestructive Evaluation held in San Diego, California. The manuscript that will be published in the proceeding is appended to this report. Also in this time period work was begun on the characterization of wave propagation in composites. A preliminary report is included here.

ULTRASONIC WAVE PROPAGATION IN COMPOSITES

To make quantitative measurements on composite systems using ultrasonic waves, the interaction of the wave with the material must be understood. The objective is the determination of material properties of graphite fiber/polymer matrix composites using analysis of ultrasonic wave propagation. This will also allow identification of parameters such as porosity , fiber volume fraction, resin rich areas, and damage areas. Toward this aim we began investigation of samples with manufactured defects. The samples are T300/5208, 16 ply uniaxial samples. To mimic porosity, hollow carbon spheres ranging in size from 10 microns to 200 microns were introduced between each ply during the lay-up process. The amount was varied to give porosity values of 1%, 2%, 4%, and 8%. This work is only preliminary and shown in figure 1 is the C-scan views of the four samples. This investigation will be the focus of the 1988-89 renewal grant.

1%, 2%, 4%, AND 8%
POROSITY SAMPLES



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TEMPERATURE DEPENDENCE OF THE ULTRASONIC PROPERTIES OF AS4/LEXAN AND XAS/LEXAN COMPOSITE*

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INTRODUCTION

The integration of composites into critical components of aerospace structures requires that their integrity can be nondestructively evaluated. Conventional ultrasonic techniques are sufficient for gross flaw detection but nondestructively measuring matrix consolidation, fiber matrix adhesion and fiber breakage calls for the development of new techniques. Presented here is an approach which will aid in the understanding of the interaction of ultrasonic waves with composite materials.

The interaction of ultrasonic energy with composite material is complicated by the inherent anisotropy of the structure. The matrix is a viscoelastic material with appreciable acoustic absorption which also supports low acoustic velocity. The fiber which is crystalline provides little acoustic absorption, has a high ultrasonic wave speed and will have negligible temperature dependence for interaction with ultrasonic waves. The properties of the material at the matrix-fiber interface are unknown. Taking advantage of the different temperature dependence of the constituent parts the contribution of the matrix and the graphite fiber to the propagation and total scattering of the ultrasonic wave can be evaluated. Thus a comparison of the pure matrix and the composite samples over a wide temperature range will allow separation of contributions of the matrix and the fiber to interaction with an ultrasonic wave.

The temperature dependence of the ultrasonic properties of polymeric materials has been previously investigated by Hartmann and Jarzynski [1], who determined the temperature dependent values of the ultrasonic wave speed and elastic constants. Sutherland and Lingle [2] also examined polymeric materials and determined the master equation as well as the temperature dependent ultrasonic velocity. Smith and Winfree [3] have reported the temperature dependent acoustic properties of polysulfone and polysulfone/graphite fiber composite. In that study [3] the approximate contribution of the fiber and matrix to the total attenuation was determined. In continuation of that earlier work the

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temperature dependence of the velocity and attenuation for both the matrix and composite materials is presented here. This introduction is followed by a description of the experimental technique, a section on data reduction and a concluding section on the results.

EXPERIMENTAL TECHNIQUE

The experiment was performed in an insulated bath of 70% solution ethylene glycol with mechanical refrigerant cooling to vary the temperature from -40C to 20C. A broadband immersion transducer with a center frequency of 5 MHz was used in a pulse echo mode with pulse excitation. The front surface and subsequent round trip echoes where digitized at 100 megasamples/sec, signal averaged 100 times, and stored for later analysis. An air dam behind the specimens provided total reflection at the back surface. A chromel-alumel thermocouple with an electronic zero point reference was used to record the sample temperature. The system was cooled to -40C and allowed to warm to room temperature over the course of tens of hours assuring thermal equilibrium of the sample and liquid. The digitized waveforms where recorded for every half degree variation. The samples included a 100% lexan, 8 and 16 ply uniaxial XAS/lexan specimens with differing fiber volume fractions (V_f= 0.58, 0.61, 0.36, and 0.35), and a 16 ply uniaxial AS4/lexan sample with a fiber volume fraction of 0.55. All of the specimens had undergone the same processing history. The resin content and corresponding fiber volume fraction were determined by measuring pre- and post-processing parameters and are accurate to a few percent.

DATA REDUCTION

A representative ultrasonic waveform is shown in figure 1. The front surface echo is evident as well as the first and second round trips through the sample. To solve for the ultrasonic velocity, attenuation, and reflection coefficient, the front surface reflection at each temperature is convolved with a plane wave model of the round trip waveforms. This is a variation of the technique suggested by Papadakis [4]. The model used is shown here:

$$\frac{(1-R^2)e^{-2d(K_1+K_2\omega)-2i\omega d/c}}{R} + \frac{R(1-R^2)e^{-4d(K_1+K_2\omega)-4i\omega d/c}}{R}$$

where R is the reflection coefficient, c is the wave speed, ω is the radial frequency and K_1 and K_2 give the frequency dependence of the attenuation, which is assumed to be a constant plus a frequency dependent term. This model calculation is compared to the data using a Levenburg-Marquardt non-linear fitting routine [5] for the variables R, c, K_1 and K_2.

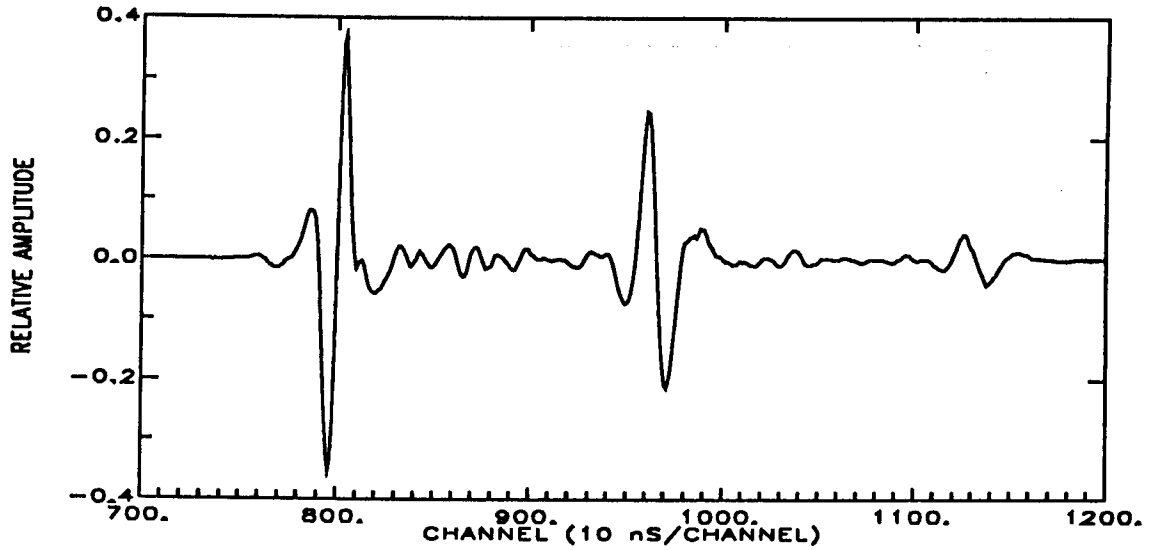

Fig. 1. Acoustic response of the AS4/lexan (V_f = 0.55) sample at -4C.

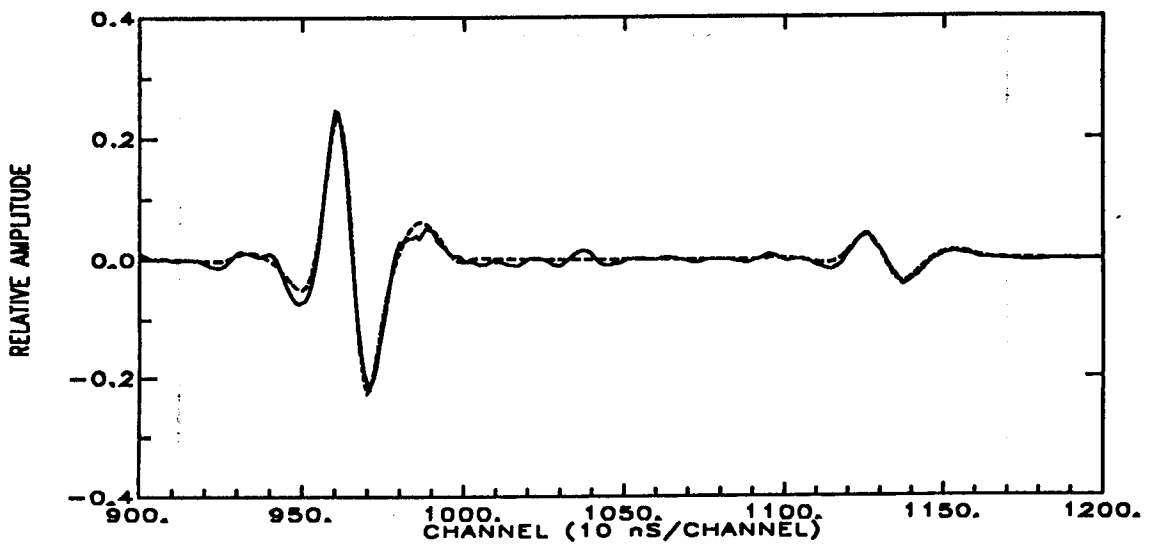

Fig. 2. Experimental data (-) and fit (---) for the AS4/lexan sample -4C.

RESULTS

An example of the first and second round trip response and the fit to the data is shown in figure 2. The fit is quite good and the same procedure is applied to all the data.

The attenuation for the AS4/lexan and the 100% lexan sample is shown in figure 3. The total attenuation is calculated by adding the K_1 and 5 MHz times the K_2 value ($K_1 + 5*K_2$) at each temperature. Although the transducer used is broadband the choice of 5 MHz locates the approximate centroid of the energy distribution in the ultrasonic wave. The absorption attenuation of the 100% lexan is above that of the composite. The composite is a stiffer structure and losses are due to absorption and scattering of the ultrasonic wave. The relative temperature response of the composite is the same viscoelastic response of the pure matrix and a linear function could be used to fit the lexan data to the composite data.

The attenuation data for representative experiments on XAS/lexan is shown in figure 4 along with the 100% lexan data. The data represents different composite panels that were measured in a different series of experiments. The top attenuation curve is for an 8 ply sample with V_f=0.58 and the other is a 16 ply sample with V_f=0.61. For this case the attenuation is both greater than and less than that of the pure matrix material. This is also the case for the other composite samples when the experiments are repeated at different locations on the sample. The attenuation data can be linearly fit with the 100% lexan attenuation data and the magnitude differences are most likely due to local scatterers in the material. These scatterers are visible as small

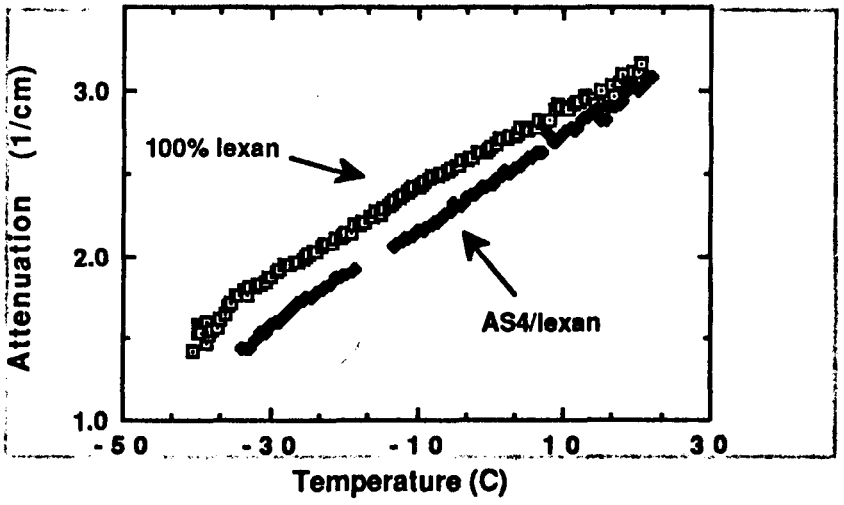

Fig. 3. Attenuation as a function of temperature for AS4/lexan and 100% lexan.

amplitude returns between the surface echoes shown in fig. 1. The data is not corrected for this internal backscatter and only the surface reflections are used in the calculation

The velocity variation with temperature is shown in figure 5. The result is typical of viscoelastic materials where the stiffness increase at lower temperature supports higher wave speeds. The increase with fiber content is representative of the contribution of the fiber

to the increased stiffness of the samples. The progression with fiber content is regular for the XAS fiber samples. For the subset of fiber volumes the difference in the specimen velocity values is the likely result of the variance in the fiber volume fraction which is greater than the accuracy of the known values. The reproducibility of the velocity data on an individual samples is excellent and is independent of the relative attenuation value at the measurement location. Other experimental results for the same samples measured at later times at different locations on the sample are shown on fig. 5 and the data points overlap well.

The results for the AS4/lexan velocity calculations seem out of place on this graph. In fact since the AS4 and XAS fibers have similar properties the AS4/lexan data should lie between the two sets of XAS/lexan data due to its measured value of fiber volume fraction. The offset of the AS4/lexan is greater than the uncertainty in the fiber volume fraction values and is due to a difference in the physical makeup of the AS4/lexan specimen as compared to the XAS/lexan specimen. This difference was noted by other researchers examining the same composite systems. Concurrently with the work reported here the interlaminar fracture toughness of samples identical to the ones studied here was being performed [6]. The properties of the fiber are nearly identical but work by Hinkley [6] revealed that a proprietary surface coating applied to the XAS fiber lead to a probable reaction with the matrix during processing of the samples. He found evidence of a higher porosity for the XAS as compared to the AS4. A post-processing examination of the material at the fiber matrix interface revealed that the molecular weight of the matrix had degraded from initial values. Also, the interlaminar fracture toughness of the XAS/lexan was 40% below that of the AS4/lexan. Therefore, the XAS/lexan velocity data maybe lower than that of the AS4/lexan because of the

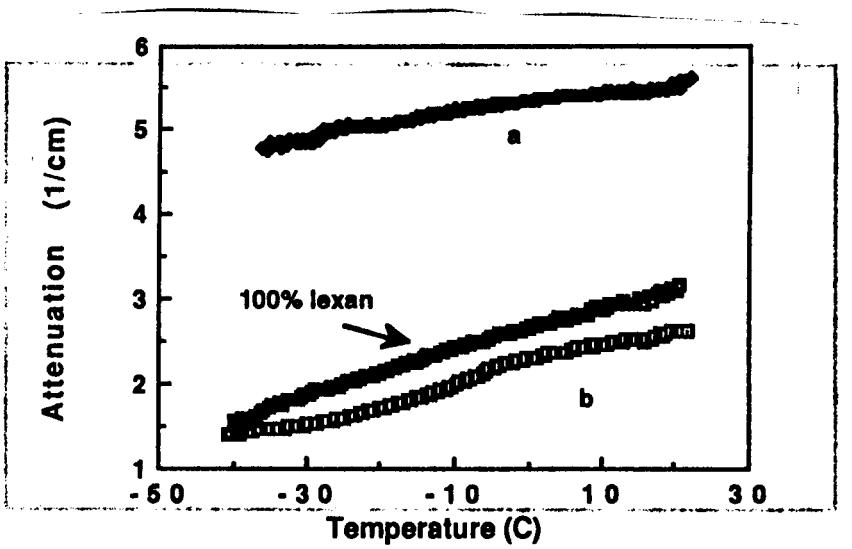

Fig. 4. Attenuation as a function of temperature for XAS/lexan (a) 8-ply: V_f=0.58, (b) 16-ply: V_f=0.61, and 100% lexan.

presence of porosity. The effect of porosity on decreasing the ultrasonic wave velocity has been noted by Reynolds and Wilkinson [7]. The other possibility is that the local variation in molecular weight near the fiber-matrix interface changes the elastic constants sufficiently to dramatically alter the transverse velocity.

CONCLUSION

We have presented here a technique for the measurement of the ultrasonic properties of composite and matrix materials as a function of temperature. This method illustrates the first step towards separating the contributions of the constituent parts to interaction with an ultrasonic wave.

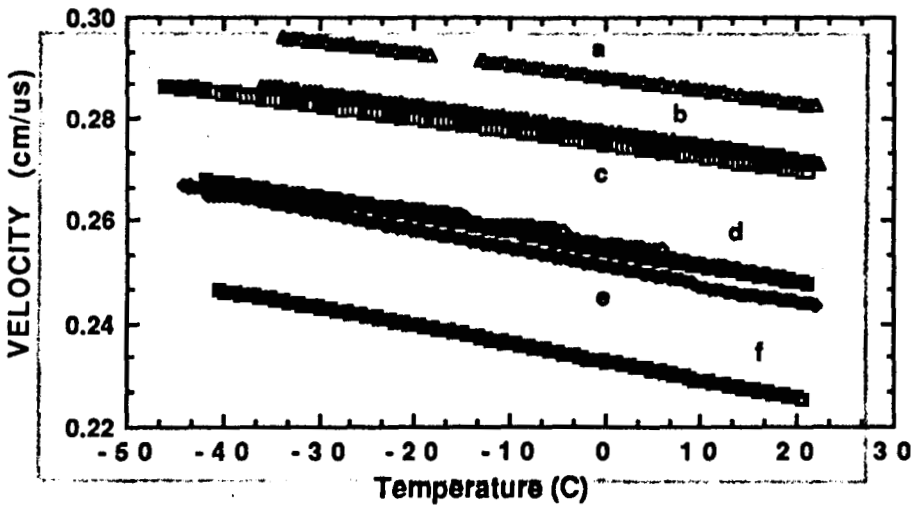

Fig. 5. Velocity as a function of temperature for: (a) AS4/lexan (0.55), (b) XAS/lexan (0.58), (c) XAS/lexan (0.61), (d) XAS/lexan (0.35), (e) XAS/lexan (0.36), and (f) 100% lexan.

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