Single-sided determination of elastic constants of thick composites using acoustoultrasonic technique

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Abstract
The determination of elastic constants is vital for any in-depth study of material performances. One of the more frequently used methods for elastic constant determination involves ultrasonic velocity measurements. Although this method is convenient in isotropic materials, it involves more complicated procedures for anisotropic materials. In this study, a measurement method is introduced that does not require cutting samples for velocity measurements in different directions. This method utilizes the acoustoultrasonic technique and deduces the elastic constants of transversely isotropic materials from the time-of-flight of obliquely reflected echoes which are received by another transducer placed on the same surface. Analytical and numerical analyses are described which reveal the sensitivity of the results to different kinds of measurement errors. It is reported that systematic errors are most detrimental to the extraction of elastic constants, and appropriate steps are demonstrated which reduce this kind of error. This method is experimentally tested on three unidirectional graphite/epoxy composite plates. Three of the elastic constants were found using pulse-echo velocity measurements normal to the top surface of the sample plate. The other two elastic constants were computed using acoustoultrasonic technique. The results show good agreement with nominal values of elastic constants obtained by cutting one of the tested samples.

Keywords
elastic constants, errors, composite materials, plates, anisotropy, ultrasonic testing, graphite, epoxides

Disciplines
Aerospace Engineering | Materials Science and Engineering | Structures and Materials

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INTRODUCTION

The extensive use of composites as structural materials in today’s industries has demanded a greater understanding of their properties. The anisotropic elastic constants of composites are among the most important. Knowledge of these constants is essential for static stress analysis in design and for predicting structural vibration. In addition, the elastic constants play an important role in understanding the results of ultrasonic nondestructive inspection. A case of particular interest in this paper is that of thick composites, with thicknesses ranging from 20 to 30 mm. These types of composites are being considered for hulls of submerged vehicles.1

Several methods are available for determining the elastic constants of thick composites. Ultrasonic wave speed measurement is one of the most commonly used methods for the determination of elastic constants of anisotropic materials.2 The conventional way of measuring elastic constants using ultrasound usually requires cutting specimens from the structure and finding the ultrasonic wave speed in different directions.3 However, other than being a tedious task, the cutting of a sample from the whole structure may be undesirable or impossible. There have been several studies having as an objective the nondestructive determination of elastic constants. Pearson and Muri4 first found the elastic constants of a composite by cutting small samples at different angles. They then performed an immersion test by sending a nonnormal incident signal through the sample and received the signal on the other side. They found disagreement between theoretical and experimental results due to difficulty in locating where the signal exited the sample. Many researchers have worked on related techniques for finding the elastic constants nondestructively.5–8 A common problem found in all the studies is the sensitivity of particular elastic constants to small errors in the measurements.9

In most techniques used for determination of elastic constants, through transmission measurements are used. This requires access to both side of the samples. Also, in some cases the immersion of the sample in a fluid is required. Doyle and Scala10 proposed a nondestructive scheme for measuring the elastic constants, while having access to only one side of the sample and using laser ultrasonic generation and interferometric detection. Also, Rose et al.11 attempted to determine the elastic constants of an orthotropic graphite/epoxy using one sided ultrasonic technique. He assumed the composite material to be homogeneous with depth and used guided surface and sub-surface waves to measure the elastic constants. Two of the elastic constants that he found were not possible since they caused the stiffness matrix not to be positive definite.

In the acoustoultrasonic technique, two transducers are placed on one side of a sample and the time-of-flight is measured as a function of the separation distance. Hsu and Margetan12 used this technique and showed that the experimentally observed times-of-flight were in agreement with theoretical predictions using elastic constants measured separately. In the present study, the acoustoultrasonic technique was used in the “inverse” manner. A method was developed to infer the elastic constants of thick composites nondestructively from the time of arrival of the obliquely reflected, acoustoultrasonic signals.

The paper starts with a theoretical justification of the use of the acoustoultrasonic method for the extraction of elastic constants for an orthotropic system whose surfaces correspond to one of the mirror planes of the material. Then, the numerical procedures for implementing this method on a
FIG. 1. Orientation of coordinate system with respect to the sample. Plane perpendicular to 3 axis is the accessible surface.

unidirectional composite (transversely isotropic system) are presented. After that some theoretical and numerical analysis of the sensitivity of the procedure to experimental errors are discussed. Finally, the elastic constant results obtained from acoustoultrasonic measurements in three unidirectional samples are shown.

I. GENERAL THEORY

A. Variation of wave velocity with respect to direction

1. Determination of phase velocity

The rectangular coordinate system used in this study has the 3-axis perpendicular to the sample and 1 and 2 axes are along the principle axes of the material (see Fig. 1). The general Christoffel equation (13) is given as

\[ \sum_{ijkl} C_{ijkl} k_i k_j U_k = 0, \quad i,j,k,l = 1,2,3, \]

where \( C_{ijkl} \) are the elastic constants; \( k_1, k_2, k_3 \) are the wave vector components in 1, 2, and 3 directions; \( \rho \) is the density; \( \omega \) is the angular frequency; and \( U \) is the displacement vector.

To avoid a trivial solution, the characteristic determinant of Eq. (1) is set to zero:

\[ \Omega(k,\omega) = \det[ \sum_{ijkl} C_{ijkl} k_i k_j ] = 0. \]

Then, to define the velocity in terms of direction (\( \theta \) and \( \phi \)), \( k_1 \) and \( k_2 \) can be written as (see Fig. 1):

\[ k_1 = k_3 \tan \theta \cos \phi, \]
\[ k_2 = k_3 \tan \theta \sin \phi. \]

By substituting Eqs. (3) and (4) into (2) and expanding the determinant, a sixth degree polynomial can be obtained.

\[ A_1 (\frac{k_3}{\omega})^6 + A_2 (\frac{k_3}{\omega})^4 + A_3 (\frac{k_3}{\omega})^2 + A_4 = 0. \]

The solutions to the polynomial (5) are the components of the slowness in the 3 direction, for a given set of elastic constants, direction (\( \theta \) and \( \phi \)), and density. By substituting these roots into Eqs. (3) and (4), the other two components of the slowness vector can be determined. For an orthotropic system, the A’s in Eq. (5) are functions of \( C_{11}, C_{22}, C_{33}, C_{44}, C_{55}, C_{66}, C_{12}, C_{13}, C_{23}, \theta, \phi, \) and \( \rho \).

If the wave is propagated along the three direction \( (k_1 = k_2 = \theta = 0) \), then the constant coefficients (A’s) in Eq. (5) are only functions of \( C_{33}, C_{44}, C_{55}, \) and \( \rho \). Also, the roots of the polynomial in this case will be

\[ \left( \frac{k_3}{\omega} \right)^2 = \frac{\rho}{C_{33}}, \]
\[ \left( \frac{k_3}{\omega} \right)^2 = \frac{\rho}{C_{44}}, \]
\[ \left( \frac{k_3}{\omega} \right)^2 = \frac{\rho}{C_{55}}, \]

where Eq. (6) corresponds to the L-wave velocity along the 3 direction, and Eqs. (7) and (8) correspond to the shear velocity along the 3 direction polarized along the 2 and 1 direction, respectively.

Now, consider launching plane waves in the 1–3 plane \( (k_2 = \phi = 0) \). In this case the constant coefficients (A’s) in Eqs. (5) will be functions of \( C_{11}, C_{33}, C_{44}, C_{55}, C_{66}, C_{13}, \theta, \) and \( \rho \). In the same way, if waves are propagated in the 2–3 plane \( (k_1 = 0, \phi = \pi) \), then the constant coefficients (A’s) in Eq. (5) will be functions of \( C_{22}, C_{33}, C_{44}, C_{55}, C_{66}, C_{23}, \theta, \) and \( \rho \).

2. Determination of group velocity

In a lossless anisotropic materials, the energy propagates along the group velocity direction. The group velocity vector\(^{13}\) is written as

\[ \mathbf{v}_g = - \frac{\partial \mathbf{\Omega}}{\partial k}, \]

where

\[ \mathbf{v}_g = \frac{\partial x}{\partial k_1} \mathbf{e}_1 + \frac{\partial x}{\partial k_2} \mathbf{e}_2 + \frac{\partial x}{\partial k_3} \mathbf{e}_3. \]

Here \( \mathbf{v}_g \) is the group velocity vector and \( \mathbf{\Omega} \), given by Eq. (2), defines the slowness surface. It is important to notice that, in calculating the group velocity, the gradient of \( \mathbf{\Omega} \) is a vector normal to the slowness surface; therefore, the group velocity is along the normal vector to the slowness surface.

B. Inverse problem for extraction of elastic constants

1. Orthotropic case

The experimental technique used in this study measures the group velocity vector \( \mathbf{v}_g \) (see Fig. 2). Therefore, the above equations must be solved for the elastic constants in terms of the group velocity vector. First consider the general case of orthotropic symmetry with known principle axes along 1, 2, and 3 directions (see Fig. 1). In cases in which the principle axes are not known, there are experimental methods that can be used to determine these axes.\(^{14}\) Three of the elastic constants can easily be found by simple measurements. If an L wave and two shear waves polarized in the 2 and 1 directions are propagated in the 3 direction, the elastic constants \( C_{33}, C_{44}, \) and \( C_{55} \) can be found using Eqs. (6) to (8). To find the six remaining elastic constants, first oblique
FIG. 2. Phase and group velocity propagation for a typical anisotropic material. The receiving transducer detects the signal along the group velocity direction.

$L$ waves are launched in 1–$3$ plane. The elastic constants affecting the $L$-wave velocity in this plane are $C_{11}$, $C_{33}$, $C_{55}$, and $C_{13}$. Two of these elastic constants have been already found using Eqs. (6) to (8). Therefore the remaining two elastic constants effecting the $L$-wave velocity in this plane, $C_{11}$ and $C_{13}$, can be found using Eqs. (5) and (9).

Similarly, oblique $L$ waves could be propagated in the 2–$3$ plane. The elastic constants affecting the $L$-wave velocity in this plane are $C_{22}$, $C_{33}$, $C_{44}$, and $C_{23}$, of which $C_{33}$ and $C_{44}$ have been found from the through thickness measurements. Thus, Eqs. (5) and (9) could be solved for $C_{22}$ and $C_{23}$.

Finally, oblique $L$ waves are propagating in a direction which has components in all three directions ($\theta \neq 0$, $\phi \neq 0$). In this case the $L$-wave velocity depends on all the elastic constants. Hence, the two remaining elastic constants, $C_{66}$ and $C_{12}$, could be computed. It must be noted for this case, in reference to Fig. 2, that the group velocity will be in the plane of the figure as shown. However, the phase velocity will in general have components out of the plane. This will slightly complicate the computational procedure due to the difference in plane of group velocity and phase velocity but adds no fundamental difficulty.

2. Transversely isotropic case

In transversely isotropic materials, there are only five independent elastic constants, and significant simplification occurs. Assuming that the plane of isotropy is the 2–$3$ plane, then the elastic constants are $C_{11}$, $C_{22}$=$C_{33}$, $C_{44}$, $C_{55}$=$C_{66}$, $C_{12}$=$C_{13}$, $C_{23}$=$C_{22}-2C_{44}$. If an $L$ wave and two shear waves, polarized in the 2 and 1 direction, are propagated in the 3 direction, then the elastic constants $C_{33}$, $C_{44}$, and $C_{55}$ can be found using Eqs. (6) to (8). Thus the only remaining elastic constants to be found will be $C_{11}$ and $C_{13}$. These two elastic constants could be found by measuring the group velocity vector of oblique $L$ waves propagating in the 1–$3$ plane.

Therefore, the elastic constants in transversely isotropic materials could be found in the same procedure as in orthotropic materials, except the oblique $L$-wave arrival times need only be measured in one plane instead of in three planes.

A detailed analysis of this procedure follows, preceeded by a discussion of the experimental techniques used in obtaining the data on the arrival times of the obliquely propagating waves.

II. EXPERIMENTAL PROCEDURES

In this work the elastic constants of unidirectional graphite/epoxy composites were found in accordance with the general procedures just described. First, three unidirectional graphite/epoxy composite samples with thicknesses close to 1 in. (25.4 mm) were chosen. Two of the samples were fabricated by LTV Aerospace using AS4/3502 prepregs. The third sample was fabricated by Hercules. Then a 1-MHz, 0.5-in. (12.7 mm)-diam $L$-wave transducer was used to find the velocity in the normal direction (3 direction), employing a pulse-echo overlap technique. This measurement was used to calculate $C_{33}$ using Eq. (6). Also, a 1-MHz, 0.5-in. (12.7 mm)-diam shear transducer was used to find the shear velocities propagating along the normal direction (3 direction) and polarized in 1 and 2 directions. Again, the pulse-echo overlap technique was used for these measurements. These two shear velocities determined $C_{44}$ and $C_{55}$ according to Eqs. (7) and (8).

To determine the other two elastic constants ($C_{11}$ and $C_{13}$), oblique quasi-$L$ waves had to be generated. This was done by using a 1-MHz, 0.5-in. (12.7 mm)-diam $L$-wave transducer as the transmitter, and a similar $L$-wave transducer as the receiver (see Fig. 3). This experimental setup is referred to as the "acoustoultrasonic" configuration, where one normal incidence contact transducer was used for generating the waves and another normal incidence contact transducer was used to receive the waves downstream. As seen in Fig. 3, some of the signals from the transmitter are obliquely propagated due to the diffraction of the beam. The reflected signal from these oblique rays is detected by the receiver. Of course, there are mode conversions when the oblique signals are reflected; however, the $L$ wave will be detected first by the receiver due to its higher speed with respect to shear waves for these kinds of materials. There are unusual materials that could have faster shear wave speeds than $L$-wave speed in some directions; thus, care must be taken to deter-
A typical signal detected by receiving transducer in acoustoultrasonic measurement technique.

By changing the separation distance \( D \) between the transmitter and the receiver, signals with higher incident angles are detected by the receiver. At each separation distance, the time-of-flight \( t \) of the quasilongitudinal mode echo is recorded. The separation distance \( D \) was measured using a ruler with estimated error of \( \pm 1 \) mm. The time-of-flight was taken to be the delay between the trigger time and the main central peak of received signal (see Fig. 4). A peak was chosen because of uncertainties in locating the beginning of the received signal. Therefore, there was an unknown over estimation in time-of-flight measurements. This over estimation in time-of-flight measurements changed at different separation distances due to the change in pulse shape. This was a systematic error. Also, there was error in reading the time from the oscilloscope. The smallest increment in time measurements was \( \pm 40 \times 10^{-9} \) s. This was a random error in the time-of-flight measurements. The other measurements used in this study, which include \( C_{33}, C_{44}, C_{55} \) and thickness \( h \), possess very small random errors compared to the separation distance and time-of-flight measurement uncertainties in the acoustoultrasonic technique. They are thus ignored. Figure 5 shows the variation of the time-of-flight versus the separation distance for an experiment performed on one of the unidirectional graphite/epoxy composite samples.

Finally, one of the samples (sample No. 1) was used to measure the elastic constants in the conventional way. That is, small specimens were cut from the sample, and the velocities in different directions were found. These values of elastic constants \( (C_{ij}) \) are referred to as the nominal values, and they were found to be (in GPa)

\[
\begin{align*}
C_{11} &= 128.0, \quad C_{22} = C_{33} = 14.0, \\
C_{44} &= 3.4, \quad C_{55} = C_{66} = 6.2, \\
C_{12} &= C_{13} = 7.0, \quad C_{23} = 7.2, \quad \rho = 1586.0 \ \text{kg/m}^3.
\end{align*}
\]

III. DATA ANALYSIS FOR EXTRACTION OF ELASTIC CONSTANTS

A. Analysis based on two observation points

1. Solution based on perturbation approach

The theoretical discussion indicates that it is possible to determine three of the five elastic constants of a transversely isotropic, unidirectional composite from through thickness velocity measurements. The remaining two, \( C_{11} \) and \( C_{13} \), must be inferred from measurements of the angular dependence of the velocity in the 1–3 plane. Such information can be found from the acoustoultrasonic measurement procedure discussed in the previous section.

The simplest procedure would be to measure the arrival time at two different transducer separations. This would provide the minimum information necessary to infer \( C_{11} \) and \( C_{13} \). In the remainder of this subsection, an analysis of the accuracy of such an approach will be presented. To gain insight into the importance of various physical parameters, this will be based on a perturbation theory which is valid when the elastic constants of the composite are not too different from their nominal values. This assumption will allow an analytical sensitivity analysis which will reveal a number of important factors governing the ability to extract elastic constants by this approach.

The elastic constants of individual samples of graphite/epoxy composite material with the same layup are usually somewhat different from each other. In fact, the existence of these differences, mainly due to manufacturing and curing processes, are the motivation of this work. For example, the three unidirectional composite samples used in this study were manufactured at two different companies; thus, the values of their elastic constants were not expected to be identical.

Consider elastic constants \( (C_{ij}) \) for a unidirectional composite material with thickness \( h \), and density \( \rho \). To be perturbed from their nominal values \( (C_{ij}^0) \) the unknown elastic constant of a real sample can be written as:
\begin{align}
C_{11} &= C_{11}^0 + \Delta C_{11}, \\
C_{13} &= C_{13}^0 + \Delta C_{13}.
\end{align}

The values of these unperturbed elastic constants (in GPa) were chosen to be

\begin{align*}
C_{11}^0 &= 140.0, \\
C_{12}^0 &= C_{33}^0 = 16.0, \\
C_{44}^0 &= 4.0, \\
C_{55}^0 &= C_{66}^0 = 7.0, \\
C_{15}^0 &= C_{16}^0 = 7.0, \\
C_{23}^0 &= 8.0.
\end{align*}

These values are close to the elastic constants of a unidirectional graphite/epoxy composite.

Now consider two given separation distances \(D_1\) and \(D_2\), for which the times-of-flight are \(t_1\) and \(t_2\), respectively. For each separation, the measured group velocity \(V_g\) is calculated by

\[ V_g = \frac{\sqrt{(2h)^2 + D^2}}{t}. \tag{12} \]

The differences in group velocities at separation distances \(D_1\) and \(D_2\) between the real sample and those which would be expected for the unperturbed medium are because of deviations from \(C_{11}^0\) and \(C_{13}^0\), \(\Delta C_{11}\), and \(\Delta C_{13}\). A procedure is described below to find those elastic constant differences from the measured velocities, as given by Eq. (12).

The analytical formula for group velocity \(V_g\) as a function of the group velocity direction \(\theta_g\) is very complicated, and in this study a numerical scheme was used to find the group velocity in terms of the group velocity direction. However, it can be represented in functional form as

\[ V_g = f(C_{11}, C_{13}, \theta_g). \tag{13} \]

Of course, the group velocity is also a function of the other material properties; however, it is assumed that the values of these parameters are known and can be considered as constants in the function \(f\). Applying a Taylor series expansion of Eq. (13) about the nominal elastic constants, and using Eqs. (10) and (11), the group velocity \(V_g\) can be approximated as

\[ V_g \approx f(C_{11}^0, C_{13}^0, \theta_g) + \frac{\partial f}{\partial C_{11}} \Delta C_{11} + \frac{\partial f}{\partial C_{13}} \Delta C_{13}. \tag{14} \]

The measured group velocities in the real sample at separation distances \(D_1\) and \(D_2\) are denoted as \(V_{g1}\) and \(V_{g2}\), respectively. The group velocity directions are \(\theta_{g1}\) and \(\theta_{g2}\), and can be calculated by (see Fig. 2)

\[ \theta_{gi} = \tan^{-1} \left( \frac{D_i/2}{h} \right), \quad i = 1, 2. \tag{15} \]

By applying Eq. (14) to the two separation distances, one finds

\[ V_{g1} \approx f(C_{11}^0, C_{13}^0, \theta_{g1}) + \frac{\partial f}{\partial C_{11}} \Delta C_{11} + \frac{\partial f}{\partial C_{13}} \Delta C_{13}. \tag{16} \]

\[ V_{g2} \approx f(C_{11}^0, C_{13}^0, \theta_{g2}) + \frac{\partial f}{\partial C_{11}} \Delta C_{11} + \frac{\partial f}{\partial C_{13}} \Delta C_{13}. \tag{17} \]

The derivatives of the function \(f\) with respect to the elastic constants \(C_{11}\) and \(C_{13}\) are dependent on the group velocity direction \(\theta_g\). The following notations are used to identify each term of Eqs. (16) and (17):

\[ \alpha_1 = \frac{\partial f}{\partial C_{11}} \theta_g = \theta_{g1}, \quad \alpha_2 = \frac{\partial f}{\partial C_{11}} \theta_g = \theta_{g2}, \quad \beta_1 = \frac{\partial f}{\partial C_{13}} \theta_g = \theta_{g1}, \quad \beta_2 = \frac{\partial f}{\partial C_{13}} \theta_g = \theta_{g2}. \]

Thus the \(\alpha\)'s and \(\beta\)'s represent the sensitivity of the group velocity to the elastic moduli of interest and the \(\Lambda\)'s represent the deviations of the measured velocities from those expected in the unperturbed medium. Equations (16) and (17) can be written as

\[ \alpha_1 \Delta C_{11} + \beta_1 \Delta C_{13} = \Lambda_1, \tag{18} \]

\[ \alpha_2 \Delta C_{11} + \beta_2 \Delta C_{13} = \Lambda_2, \tag{19} \]

where \(V_{g1}^1\) and \(V_{g2}^2\) can be measured experimentally using the acoustic-ultrasonic configuration. Also, the function \(f\) can be obtained numerically for the two group velocity directions \(\theta_{g1}\) and \(\theta_{g2}\) using the unperturbed elastic constants. Therefore, the terms \(\Lambda_1\) and \(\Lambda_2\) can be computed. The terms \(\alpha_1, \alpha_2, \beta_1,\) and \(\beta_2\) can also be computed numerically for the two group velocity directions \(\theta_{g1}\) and \(\theta_{g2}\). This is done by changing the unperturbed elastic constants \(C_{11}^0\) and \(C_{13}^0\) by small amount and finding the new value of function \(f\). Finally, by knowing \(\alpha_1, \alpha_2, \beta_1, \beta_2, \Lambda_1,\) and \(\Lambda_2,\) the two simultaneous Eqs. (18) and (19) can be solved for \(\Delta C_{11}\) and \(\Delta C_{13}\):

\[ \Delta C_{11} = \frac{\Lambda_1 \beta_2 - \Lambda_2 \beta_1}{\alpha_1 \beta_2 - \alpha_2 \beta_1}, \tag{20} \]

\[ \Delta C_{13} = \frac{\alpha_1 \Lambda_2 - \alpha_2 \Lambda_1}{\alpha_1 \beta_2 - \alpha_2 \beta_1}. \tag{21} \]

Equations (20) and (21) were derived by assuming a linear relation between group velocity \(V_g\) and \(\Delta C_{11}\) and \(\Delta C_{13}\). To examine the accuracy of using such an analysis based on the first term in a Taylor series approximation, an example problem using extreme values for \(\Delta C_{11}\) and \(\Delta C_{13}\) was considered. First, the values of unperturbed elastic constants \((C_{ij}^0)\) were used to compute the function \(f\) and its derivatives with respect to \(C_{11}\) and \(C_{13}\) at two group velocity directions \(\theta_{g1}=35^\circ\) and \(\theta_{g2}=55^\circ\). Then, by choosing some
extreme values for $\Delta C_{11}$ and $\Delta C_{13}$ ($\Delta C_{11} = -30$ GPa, $\Delta C_{13} = 3$ GPa) the expected group velocities $V_{g1}^2$ and $V_{g2}^2$ were computed based on the full equations. By using these values, $\alpha_1$, $\alpha_2$, $\beta_1$, $\beta_2$, $\Lambda_1$, and $\Lambda_2$ were calculated, and Eqs. (20) and (21) were solved for $\Delta C_{11}$ and $\Delta C_{13}$. They were found to be $\Delta C_{11} = -32$ GPa, $\Delta C_{13} = 3.1$ GPa. There are 4% to 7% differences between the computed values of $\Delta C_{11}$ and $\Delta C_{13}$ and the values chosen. This showed that the formulation is sufficiently accurate to provide a good basis for an error analysis when the changes are of this order.

2. Use of perturbation solution to analyze error sensitivity

In the above analysis, use of computed values essentially assumed that the group velocities $V_{g1}^2$ and $V_{g2}^2$ could be measured exactly. However, in any experimental measurement there will be some uncertainties in the data. In this case, the measured values are separation distances and the time-of-flight. The errors in the other measured values, such as thickness of the sample, are considered to be negligible. The uncertainties in separation distances and times-of-flight are denoted as $\delta D$ and $\delta t$, respectively. In turn, these uncertainties in measurements cause uncertainties in the final prediction of $\Delta C_{11}$ and $\Delta C_{13}$, $\delta(\Delta C_{11})$ and $\delta(\Delta C_{13})$. Assuming the uncertainties in the four experimental variables to be independent, the uncertainties in final results can be computed by

$$
\delta(\Delta C_{11})^2 = \left( \frac{\partial(\Delta C_{11})}{\partial D_1} \right)^2 (\delta D_1)^2 + \left( \frac{\partial(\Delta C_{11})}{\partial D_2} \right)^2 (\delta D_2)^2 + \left( \frac{\partial(\Delta C_{11})}{\partial t_1} \right)^2 (\delta t_1)^2 + \left( \frac{\partial(\Delta C_{11})}{\partial t_2} \right)^2 (\delta t_2)^2, \quad j = 1, 3.
$$

Equation (22) involves derivatives of $\Delta C_{11}$ and $\Delta C_{13}$ with respect to the transducer separation distance $D$, and time-of-flight $t$. Both of these variables directly affect $\Lambda$ [see Eq. (12)]. Moreover, any change in separation distance also causes a change in group velocity direction $\theta_g$, and consequently affects $\alpha$ and $\beta$. By using Eqs. (20) and (21), the derivatives of $\Delta C_{11}$ and $\Delta C_{13}$ with respect to $D$ and $t$ will be

$$
\frac{\partial(\Delta C_{11})}{\partial D_1} = \frac{\partial(\Delta C_{11})}{\partial \alpha_1} \frac{\partial \alpha_1}{\partial D_1} + \frac{\partial(\Delta C_{11})}{\partial \beta_1} \frac{\partial \beta_1}{\partial D_1} + \frac{\partial(\Delta C_{11})}{\partial \Lambda_1} \frac{\partial \Lambda_1}{\partial D_1},
$$

$$
\frac{\partial(\Delta C_{11})}{\partial D_2} = \frac{\partial(\Delta C_{11})}{\partial \alpha_2} \frac{\partial \alpha_2}{\partial D_2} + \frac{\partial(\Delta C_{11})}{\partial \beta_2} \frac{\partial \beta_2}{\partial D_2} + \frac{\partial(\Delta C_{11})}{\partial \Lambda_2} \frac{\partial \Lambda_2}{\partial D_2},
$$

$$
\frac{\partial(\Delta C_{11})}{\partial t_1} = \frac{\partial(\Delta C_{11})}{\partial \alpha_1} \frac{\partial \alpha_1}{\partial t_1} + \frac{\partial(\Delta C_{11})}{\partial \beta_1} \frac{\partial \beta_1}{\partial t_1} + \frac{\partial(\Delta C_{11})}{\partial \Lambda_1} \frac{\partial \Lambda_1}{\partial t_1},
$$

$$
\frac{\partial(\Delta C_{11})}{\partial t_2} = \frac{\partial(\Delta C_{11})}{\partial \alpha_2} \frac{\partial \alpha_2}{\partial t_2} + \frac{\partial(\Delta C_{11})}{\partial \beta_2} \frac{\partial \beta_2}{\partial t_2} + \frac{\partial(\Delta C_{11})}{\partial \Lambda_2} \frac{\partial \Lambda_2}{\partial t_2},
$$

where $i = 1, 2$ and $j = 1, 3$. The derivatives of $\Delta C_{11}$ and $\Delta C_{13}$ with respect to $\alpha$, $\beta$, and $\Lambda$ can be derived from Eqs. (20) and (21). Also, the derivative of $\theta_g$ with respect to $D$ can be found from Eq. (15). Finally, derivatives of $\alpha$, $\beta$, and $\Lambda$ with respect to $\theta_g$ are computed numerically.

As was mentioned before in the discussion of experimental procedures, it was found that the random uncertain-
computed in step 3 will produce the same separation distance as in the experimental measurement, but the time-of-flight will in general be different.

d. Step 4—Calculation of time-of-flight. This calculated time-of-flight \( t_c \) for the computed phase velocity is recorded.

e. Step 5—Calculation of time-of-flight deviations. The difference between the measured time-of-flight \( t \) and the computed time-of-flight \( t_c \) is mainly due to the fact that the estimated elastic constants, \( C_{11} \) and \( C_{13} \), are not quite correct. This difference in time-of-flight, \( d_i \), is recorded for the \( i \)th separation distance

\[
d_i = t - t_c.
\]

f. Step 6—Calculation of \( Q \) parameter. The above calculations were only for one of the experimental separation distances in the measurements. Therefore, steps 2, 3, 4, and 5 are repeated for all the experimental measurements, and the squares of the differences in time-of-flight for all the points are added

\[
Q = \sum_{i=1}^{n} d_i^2,
\]

where \( n \) is the number of data points (separation distances) in the experiment.

g. Step 7—Minimization of \( Q \). The \( Q \) parameter indicates how good the initial guesses of the elastic constants were. Now, the value of unknown elastic constants are changed in a way to minimize the value of the \( Q \) parameter. The minimization routine is stopped when either of two criteria is met: (1) the deviation of two consecutive values of \( Q \) must be less than \( 1.0 \times 10^{-15} \); (2) the number of iterations must not exceed 200. In the second case a warning message is also displayed. However, in all the calculations in this study, the first criteria determined the termination of the minimization program. The termination usually occurred after 50 to 70 iterations.

If we had chosen the exactly correct elastic constants and performed the experiments without any error, then \( Q \) would have been zero. However, due to experimental errors, \( Q \) does not minimize to zero, but converges to some small minimum value (it is denoted as "converged \( Q \)"") for a set of elastic constants.

Due to the presence of errors in the data, the elastic constants found for the converged \( Q \) may not be the correct values. Experimental errors and the sensitivity of the elastic constants to these errors may cause these elastic constants to be considerably different from the correct answers.

2. Constraints on elastic constants

Particular constraints have to be imposed in the iteration of the unknown elastic constants in step 7 of the numerical procedure. The values of \( C_{11} \) and \( C_{13} \) are expected to be positive for this class of materials, and the stiffness matrix has to remain positive definite. The minimization routine used in this study (Nelder and Mead method) did not support enforcement of the constraints. Therefore, the two design variables were defined in a way to insure all the necessary constraints. Reference 17 has a detailed definition of the design variables.

3. Sensitivity of numerical procedure to experimental error

The sensitivity analysis performed earlier using the perturbation method considered only two experimental data points. However, in this study more than two data points were taken in each experiment. To study the sensitivity of the numerical procedure to errors, a series of experiments were simulated by adding random errors to the "correct" measurements. First, the set of unperturbed (nominal) elastic constants chosen earlier was used to simulate ten experimental data points (ten sets of separation distances versus times-of-flight). When these ten simulated experimental data were fed to the numerical procedure, the unperturbed elastic constants were found by the program. This was done mainly as a check of the numerical procedure.

As mentioned before, during the course of experiments it was realized that there were two types of errors involved with the experimental data. First, there were random errors caused by difficulties in achieving accurate measurements in the experiments. The second type of error was a systematic error in the time-of-flight measurements. The latter error was mainly due to the difficulty in specifying a precise origin of time for delay measurements, and the time that the signal actually starts. Each type of error was analyzed separately.

4. Random errors

As was mentioned before, it was found that the uncertainties in time-of-flight and separation distance measurements were \( \pm 40 \times 10^{-9} \) s and \( \pm 1 \) mm, respectively. To study the effect of random errors, a normally distributed random number generator program was used. This program generated ten sets of random numbers with standard deviations equal to the uncertainties in time-of-flight and separation distance measurements \( \pm 40 \times 10^{-9} \) s and \( \pm 1 \) mm. Then, one of these normally distributed random numbers was added to each of the simulated experimental data points to introduce the effects of measurement noise. Next, the simulated experimental data with noise were fed to the program for extraction of elastic constants. This addition of random numbers to the simulated experimental data was repeated fifteen times to ensure unbiased results. Finally, the standard deviation of the computed elastic constants from nominal values \( (\sigma_{C_{11}} \) and \( \sigma_{C_{13}} \) ) were computed.

This procedure was then repeated for different numbers of simulated experimental data points (ten to two data points), and the deviation of computed elastic constants from nominal values was found for each case. Figure 6 shows the standard deviations of the elastic constants, \( \sigma_{C_{11}} \) and \( \sigma_{C_{13}} \), from their nominal values versus the number of simulated experimental points. As can be seen from the graphs, the accuracy of the computed results is greater when more experimental data points are used. Also, it is interesting to note that the deviations of elastic constants when using two data points are
FIG. 6. Deviation of computed elastic constants from their nominal values due to the random errors. The nominal values are $C_{11}=140.0$, $C_{13}=7.0$ GPa.

$\sigma_{C_{11}}=13.94$ GPa, $\sigma_{C_{13}}=1.16$ GPa,

which are very close to the values found when using the perturbation method.

5. Systematic errors

As it was mentioned before, one major source of inaccuracy in this experimental technique is the measurement of the time-of-flight. This error in time stems from the uncertainty in the beginning of the trigger (trigger starts after the zero time) and in the time that the received signal actually starts (received signal starts before main central peak where the measurement is taken). Therefore, the time-of-flight measured in these experiments is usually overestimated, and this overestimation changes at different separation distances due to change in pulse shape. In this study, however, it was assumed that this overestimation in time was constant for all the separation distances. Thus by subtracting a constant $\Delta t$ from all the simulated times-of-flight, the error in extracted elastic constants $C_{11}$ and $C_{13}$ was found. Figure 7 shows the variation of computed elastic constants against time shift $\Delta t$ for a set of noise free simulated data (ten data points). Notice that $C_{11}$ and $C_{13}$ have their respective correct values (140.0 and 7.0 GPa) at zero time shift. These graphs clearly demonstrate the greater effect of systematic error than random errors in the computed elastic constants. Also shown in Fig. 7 is the variation of converged $Q$ with the time shift $\Delta t$. As it was stated before, $Q$ is an indication of how well the computed elastic constants agree with actual elastic constants. It is important to note that when there are no systematic errors in the time-of-flight, the value of converged $Q$ is a minimum (zero with no noise) and the extracted $C_{11}$ and $C_{13}$ have their correct values. Thus the variation of converged $Q$ as a function of time shift can be used as a guide to determine the amount of time shift correction that must be applied to the experimental data.
To find how much shift is required, the value of converged Q versus separation distance by the small amount of time $\Delta t$. The value of the converged Q is found using steps 1 to 7. As was attracted from all the time-of-flight measurements. Again the time shift is used. First, the value of converged Q is found according to how much shift has been applied to the time-of-flight measurements. Again the variation of the value of converged Q is the smallest. The elastic constants found at this minimum converged Q are considered to be the best estimate of the elastic constants of the material. The agreement between the measured and computed time-of-flight is better with the proper time shift $\Delta t$. This difference between time shifted results and that without time shift is strikingly obvious. However, the effect on the accuracy of the extracted elastic constants is quite large. Table I lists the numerical results that show the effect of time shift $\Delta t$ on the extracted elastic constants for sample no. 1. The $C_{11}$ and $C_{13}$ given in the heading of Table I are the nominal values of the elastic constants of sample no. 1 which had been measured independently.

2. Uncertainties in time shift $\Delta t$

To minimize the value of the converged Q, a time shift was applied to the experimental data. However, for very small time shifts, the sensitivity of the converged Q is limited because of minor oscillation in its value for very small incremental changes in time shift. Hence the time shift increment had to be bounded below. In this study it was found that the time shifts of less than 0.05 $\mu$s did not produce a more accurate minimum converged Q. Thus the uncertainty in the time shift was 0.05 $\mu$s.

To investigate the errors in extracted elastic constants $C_{11}$ and $C_{13}$, due to the bound in time shift, the results from the systematic error analysis based on multiple observation points were used. By using the graphs from Fig. 7 it was found that the errors in extracted elastic constants are $\pm 3.5$ GPa or $2.5\%$ for $C_{11}$ and $\pm 0.8$ GPa or $11.5\%$ for $C_{13}$ when $\Delta t$ is systematically changed by 0.05 $\mu$s.

IV. RESULTS

In this study, three unidirectional graphite/epoxy composite samples were used for the experiments. The thicknesses of all three samples were about 25 mm. The experiments were performed using 1 MHz, 0.5-in. (12.7 mm) diam $L$-wave contact transducers. Separate data sets were taken on each sample by two different people to ensure the repeatability of the measurements. The minimum separation distance was 20 mm and the maximum separation was limited by the sample size. In sample no. 1 the maximum separation distance was 120 mm and in samples no. 2 and no. 3 it was 100 mm. The number of data points in each experimental set varied between 15 to 20 points. Then the measurement results were fed into the computer program to find the unknown elastic constants $C_{11}$ and $C_{13}$.

Table II shows the results obtained from the experiments on the three composite samples. To verify our results, the nominal values of the unknown elastic constants for sample no. 1 were measured. These values were measured by cutting a small part of one of the samples and measuring the velocities in the various directions.

If it is assumed that the minimization of the converged Q eliminates the systematic errors, then the only type of error affecting the final results will be random error. The sensitivity analysis in the previous section predicted that for uncertainties of $\pm 40 \times 10^{-9}$ s in time-of-flight and $\pm 1$ mm in separation distance measurements, the random errors for $C_{11}$ and $C_{13}$ are $\pm 1.0$ and $\pm 0.1$ GPa, respectively. However, Table II shows that the deviation of unknown elastic con-
TABLE II. The unknown elastic constants computed from experimental data (units in GPa). Independently measured values of the unknown elastic constants of sample No. 1 are $C_{11}=128.0$ and $C_{13}=7.0$ GPa.

<table>
<thead>
<tr>
<th>Experiment No.</th>
<th>Sample No.</th>
<th>1</th>
<th>2</th>
<th>3</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>$C_{11}$</td>
<td>125.0</td>
<td>142.0</td>
<td>126.0</td>
</tr>
<tr>
<td></td>
<td>$C_{13}$</td>
<td>7.9</td>
<td>8.3</td>
<td>6.4</td>
</tr>
<tr>
<td>2</td>
<td>$C_{11}$</td>
<td>121.0</td>
<td>141.0</td>
<td>122.0</td>
</tr>
<tr>
<td></td>
<td>$C_{13}$</td>
<td>7.6</td>
<td>8.8</td>
<td>7.4</td>
</tr>
</tbody>
</table>

Constants from the nominal values for sample no. 1 are $-3.0$ and $-7.0$ GPa for $C_{11}$ and $0.9$ and $0.6$ GPa for $C_{13}$. It is believed that these are the result of the lower bound on the time shift, which produced an uncertainty in the extracted elastic constants of $\pm 3$ GPa for $C_{11}$ and $\pm 0.8$ GPa for $C_{13}$, which are on the order of the deviations in Table II.

It must be mentioned that there are other sources of the error that were not considered in this study. Some of these sources of error are (1) the systematic errors were not constant for all separation distances (as originally assumed), (2) the finite size of the transducers used in the experiments (the formulation was based on point transmitter and receiver), (3) nonuniform thickness of the samples, which in turn implies inhomogeneity of the material.

V. CONCLUSION

All the values of elastic constants of three unidirectional graphite/epoxy composites laminates were found nondestructively and by having access to only one side of the samples. Three of the elastic constants were determined by using longitudinal and shear waves propagated in the normal direction of the laminate. The remaining two elastic constants were found using the acoustoutrasonic technique. In this technique the oblique signals and their reflections traveled in the 1–3 plane. For a unidirectional composite, the measurement in the 1–3 plane was adequate to find the remaining two elastic constants. The values obtained by the experiments were close to independently measured values of the elastic constants. It was also shown numerically that the accuracy of the results would be inadequate if an insufficient number of experimental data points were used. Sensitivity analysis indicated that systematic errors affect the final results more than random errors.

This method of elastic constant measurement will be extended to other anisotropic composites with lower symmetry than the unidirectional case.

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