Ultrasonic immersion techniques for the measurement of elastic constants in fiber-reinforced composites.

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Abstract. *Fibrous composites materials pose a significant challenge to the ultrasonic nondestructive evaluation process due to the high attenuation as well as the influence of material anisotropy. Two ultrasonic immersion techniques to determine the elastic constants of glass-epoxy composites samples were implemented. The first is the through-transmission technique implemented with a pair of 1 MHz ultrasonic transducer in a water tank. The second is the back-reflection technique that uses a single transducer of 1 MHz in pulse-eco mode. The wave mode conversion phenomenon at the specimen-liquid interface with oblique incidence of the ultrasonic wave in a water tank provides the information to calculate the elastic constants of the specimen by measuring longitudinal and shear wave speeds and critical angles. For any given direction of propagation in an anisotropic medium, there are three different modes of waves, namely, quasi-longitudinal, quasi-shear and pure shear. If the elastic constants of a medium are know, it is possible to obtain all the three wave speeds in particular propagations directions by solving the Christoffel equation. Inverting the Christoffel equation, it's possible to obtain the elastic constants from the measured wave speed in several specific directions of the anisotropic material. Measurements were carried out on transversely isotropic (five independent elastic constants) unidirectional glass-epoxy composite material. The experimental results obtained with both techniques are compared with literature and with a simple model based on the Rule of Mixture estimation.*

Keywords: *ultrasonic; nondestructive testing; Material characterization; Elastic constants; Fiber reinforced composites.*

1. Introduction

In the last two decades the technologic of composite materials supported an incredible development, although its concept is older. As anisotropic materials such as fiber-reinforced composites are widely used for many structural applications, the determination of mechanical properties is critical for ensuring reliable performance. The determination of mechanical properties is a critical point to ensure optimum performance of anisotropic materials such as fibrous composites. The knowledge of complete elastic stiffness matrix is essential for modeling and evaluating the mechanical behavior of composite materials under severe loading conditions. The disadvantages of conventionally used techniques are: (a) some engineering constants of anisotropic materials are difficult to measure, (b) they are destructive in nature, (c) high costs involved in producing samples of desired shape and size and (d) in situ measurements are difficult. Ultrasonic techniques are however uniquely qualified for nondestructive measurement of all of the elastic constants of such materials. Elastic constants are determined by measurement of ultrasonic velocities, usually phase or group velocities, which are related to the material properties. There are several methods to measure ultrasonic velocity in the material. As the ultrasonic wave travels through the test sample, the wave is transmitted or reflected in part as it encounters materials of different acoustic impedance. The reflected signal is captured by the receiving transducer and displayed or digitally stored for further examination. To different extent, the elastic moduli, the material microstructure, the morphological conditions and associated mechanical properties can be characterized using ultrasonic testing.

An anisotropic material in general is described by 21 independent elastic constants. The unidirectional lamina, which is the basic building block of composite laminates and structures, is treated as a quasi-homogeneous orthotropic material. Determination of material properties of the unidirectional lamina is especially important because this characterization allows prediction of the properties of any multidirectional laminate. In general, the unidirectional lamina is characterized by nine independent elastic constants. However, many composite materials have the additional property of transverse isotropy. In such cases the number of independent constants is reduced to five. It is often desirable to characterize a composite material by measuring these mechanical properties nondestructively.

The reconstruction of the five elastic constants of a transversely isotropic composite material from experimentally measured ultrasonic velocities measured at different orientations of propagation are mathematically related to elastic constants and density, through the Christoffel's equation (Rosenbaum, 1988), (Auld, 1990). Thus, if experimentally measured ultrasonic data are available, computing the required stiffness involves solving the inverse problem. Generally, the inverse problem in wave propagation are often highly nonlinear and hence, non-unique in nature. Moreover, practical difficulties and the constraint of limited data sets (due to the experimental technique used) further tend to increase the degree of difficulty in the inversion.

The first experimental study about the measurement of ultrasonic wave velocities to determine all the elastic constants of a composite material was done by Zimmer and Cost (1970). These authors have done ultrasonic velocity measurements using through-transmission contact technique to obtain dynamic elastic stiffness necessary to determine fully the elastic properties of unidirectional glass-epoxy composite materials. They demonstrated that the measurement of the ultrasonic wave velocities is a valid method for obtaining the elastic stiffness constants of a unidirectional fiber composite.

Rokhlin and Wang (1989), show an application of critical angle measurement for determination of elastic constants of carbon-epoxy composite materials. Utilizing a simple type of goniometer with immersion techniques in water, they show how to its possible to utilize one transducer of longitudinal waves for excite shear waves in material. With this, given a direction of propagation relatives of plane of incidence of material, exist three critical angles, one for longitudinal wave e the others for shear waves. Wooh and Issac (1991) have been measure all the elastic constants of carbon-epoxy materials utilizing the technique of trough-transmission. They describe also a method based on resonance frequency for measure the phase velocity from the group velocity.

The determination of elastic constants from ultrasonic bulk wave velocity data along planes of symmetry has been studied by Chu and Rokhlin (1994). Using displacement polarization factors, the authors have defined the degree of material anisotropy and performed sensitivity analysis of ultrasonic velocities to elastic constants. They stated that using quasi-longitudinal and quasi-shear velocity data in two accessible planes of symmetry one can obtain a complete set of five independent elastic constants for a transversely isotropic material and seven of the nine independent elastic constants for an orthotropic material. Chu et al. (1994) reported the analysis of the reconstruction of elastic constants from ultrasonic velocity measurements in nonsymmetry planes of unidirectional composite materials. The authors identified the fact that, when all nine elastic constants of orthotropic material were reconstructed from velocity data in nonsymmetry planes, the inversion is highly dependent on the initial guesses and susceptible to random data scatter. When seven elastic constants are found from velocity data in planes of symmetry, the remaining two can be found from nonsymmetry plane data independently of initial guesses and scatter levels. Balasubramaniam and Whitney (1996) determined a method to experimentally obtain the stiffness constants of thick unidirectional (transversely isotropic) and cross ply thick glass-epoxy composites using a conventional ultrasonic immersion system and commercially available inversion software.

In this paper, immersion techniques have been studied for the measurement of ultrasonic velocities in transversely isotropic materials. The ultrasonic velocities were measured using two immersion techniques described in the literature: back-reflection and through-transmission techniques. From the measured velocity data, the elastic constants were determined through a nonlinear least-square algorithm (Rokhlin and Wang, 1989). The inversion method has been verified by using the Rule of Mixtures estimation and data provided by literature. The accuracy and precision of these two techniques were determined and compared on two glass-epoxy transversely isotropic composite materials.

2. Theorical Background

Assuming small displacements, the relationship between the stress (σ_{ij}) and strain (ε_{ij}) tensors for a generally anisotropic material is expressed in the form

$$\sigma_{ij} = C_{ijkl}\varepsilon_{kl}, \tag{1}$$

where C_{ijkl} is the stiffness tensor of the material. For a plane wave with no external boundaries, the displacements are given as

$$u_m = U_m = \exp[kj(n_i x_i - ct)], \tag{2}$$

where k is the wave number, j is the unit imaginary number, n_i is the direction cosines of the normal to the wave front, U_m is the displacement amplitude and c the wave speed. Substituting Eqs. (1) and (2) into the equation of motion

$$\frac{\partial \sigma_{ij}}{\partial x_j} = \rho \frac{\partial^2 u_i}{\partial t^2}, \tag{3}$$

yields the following eigenvalue equation:

$$\left(C_{ijkl} n_l n_j - \rho c^2 \delta_{ik}\right) U_k = 0, \tag{4}$$

where δ_{ik} is the Kronecker delta and ρ the density of the material. The 21 material constants of generally anisotropic materials are reduced to nine for orthotropic materials that are described by the following stress-strain relationship:

$$\begin{Bmatrix} \sigma_{11} \\ \sigma_{22} \\ \sigma_{33} \\ \sigma_{23} \\ \sigma_{31} \\ \sigma_{12} \end{Bmatrix} = \begin{pmatrix} C_{11} & C_{12} & C_{13} & 0 & 0 & 0 \\ C_{12} & C_{22} & C_{23} & 0 & 0 & 0 \\ C_{13} & C_{23} & C_{33} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{44} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{55} & 0 \\ 0 & 0 & 0 & 0 & 0 & C_{66} \end{pmatrix} \begin{Bmatrix} \varepsilon_{11} \\ \varepsilon_{22} \\ \varepsilon_{33} \\ \gamma_{23} \\ \gamma_{31} \\ \gamma_{12} \end{Bmatrix}, \tag{5}$$

where $[C_{ij}]$ is the stiffness matrix, subscripts 1,2,3 denote the principal material axis, and γ_{ij} are the engineering shear strains. Then, Eq. (4) takes the form:

$$\begin{pmatrix} \Gamma_{11}-\rho c^2 & \Gamma_{12} & \Gamma_{13} \\ \Gamma_{12} & \Gamma_{22}-\rho c^2 & \Gamma_{23} \\ \Gamma_{13} & \Gamma_{23} & \Gamma_{33}-\rho c^2 \end{pmatrix} \begin{Bmatrix} U_1 \\ U_2 \\ U_3 \end{Bmatrix} = \begin{Bmatrix} 0 \\ 0 \\ 0 \end{Bmatrix}, \tag{6}$$

where the so-called Christoffel stiffnesses Γ_{ij} are given by

$$\begin{aligned} \Gamma_{11} &= n_1^2 C_{11} + n_2^2 C_{66} + n_3^2 C_{55}, \\ \Gamma_{22} &= n_1^2 C_{66} + n_2^2 C_{22} + n_3^2 C_{44}, \\ \Gamma_{33} &= n_1^2 C_{55} + n_2^2 C_{44} + n_3^2 C_{33}, \\ \Gamma_{12} &= n_1 n_2 (C_{12} + C_{66}), \\ \Gamma_{23} &= n_2 n_3 (C_{23} + C_{44}), \\ \Gamma_{13} &= n_1 n_3 (C_{13} + C_{55}). \end{aligned} \tag{7}$$

A nontrivial solution of the homogeneous system of equations (6) exists when the determinant of the square matrix vanishes, and it takes the form of the following equation:

$$\det \begin{vmatrix} \Gamma_{11}-\rho c^2 & \Gamma_{12} & \Gamma_{13} \\ \Gamma_{12} & \Gamma_{22}-\rho c^2 & \Gamma_{23} \\ \Gamma_{13} & \Gamma_{23} & \Gamma_{33}-\rho c^2 \end{vmatrix} = 0. \tag{8}$$

For a material with transversal isotropy, which is a reasonable assumption for glass-epoxy, the following relations are true:

$$\begin{aligned} C_{22} &= C_{11}, \\ C_{23} &= C_{13}, \\ C_{55} &= C_{44}, \\ C_{66} &= (C_{11} - C_{12})/2. \end{aligned} \tag{9}$$

Then, the engineering constants can be expressed in terms of the five components of the stiffness matrix for a three-dimensional state of stress:

$$\begin{aligned} & E_1 = \frac{C}{C_{22}^2 - C_{23}^2}, \qquad E_2 = \frac{C}{C_{11}C_{22} - C_{12}^2}, \\ & G_{12} = C_{66}, \qquad G_{23} = C_{44}, \\ & \nu_{12} = \frac{C_{12}}{C_{22} + C_{23}}, \qquad \nu_{23} = \frac{C_{11}C_{23} - C_{12}^2}{C_{11}C_{22} - C_{12}^2}, \end{aligned} \tag{10}$$

Where:

$$C = \left(C_{22} - C_{23}\right)\left[C_{11}\left(C_{22} + C_{23}\right) - 2C_{12}^{2}\right]. \quad (11)$$

3. Experimental Setup

In this work, the ultrasonic velocities were measured in different angles of propagation in the sample to obtain the elastic constants of the material. In immersion techniques, the sample is immersed in water during the test. The water acts as a couplant to transfer the mechanical energy from the transducer to the sample. As the transducer is not in direct contact with the sample, we can guarantee a good coupling with the immersion technique. In addition, it is possible to measure the velocities in different angles by simply rotating the sample. The assembly of the experiment was done in a way that the sample is imprisoned to a rotating device that has a goniometer, which allows turning the sample in different angles. There were implemented the through-transmission and back-reflection techniques.

In the through-transmission, one transducer is the transmitter and the other is the receiver. The sample is fixed between them, having one degree of freedom of rotation, thus allowing different orientations for the propagation of waves, as shown in Fig. 1. In the back-reflection, a single transducer acts as emitter and receiver, as shown in the Fig. 2.

Experiments were carried out in a water tank. The experimental setup consists of a Pulser/Receiver (PANAMETRICS 5072PR), a 12bits, 62.5 Msamples/s A/D acquisition board, a PC and a pair of 1.0 MHz, 19 mm diameter ultrasonic transducers (PANAMETRICS A314S). The experimental setup was aligned prior to each experiment. The ultrasonic wave signal was acquired with 4096 points to guarantee the record of the total eco length (Fig. 3). The sample was turned until seventy degrees in steps of five degrees. For each angle of incidence, the obtained signal was the average of 64 acquisitions, to reduce noise. For each sample, different readings were made to check the repeatability of the experiment.

In all the experiments the ultrasound signal in the time domain was stored, for each angle of incidence. For each experiment, the signal without the composite sample (only through the water), it was acquired as a reference signal. To calculate the ultrasonic velocity in the composite sample it is necessary to accurately measure the difference of time of propagation of the wave with and without the sample for each angle. This time difference was determined using a cross-correlation technique (Adamowski et al., 1995) of the echoes in Fig. 3.

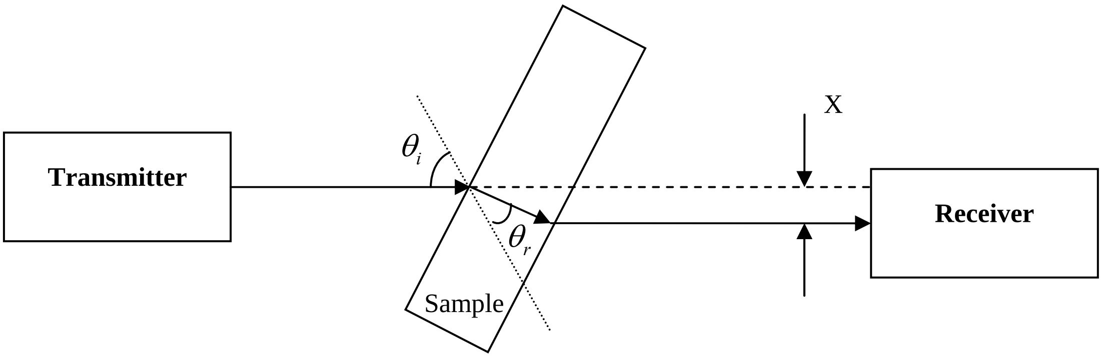


Fig. 1: Schematic of through-transmission immersion technique.

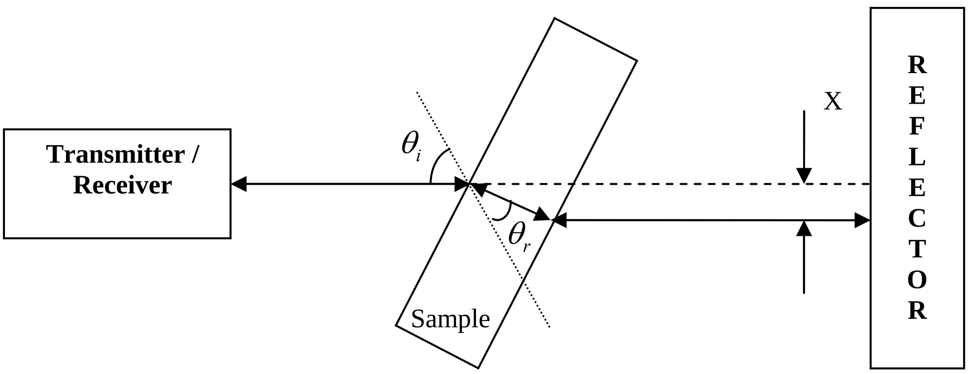


Fig. 2: Schematic of back-reflection technique.

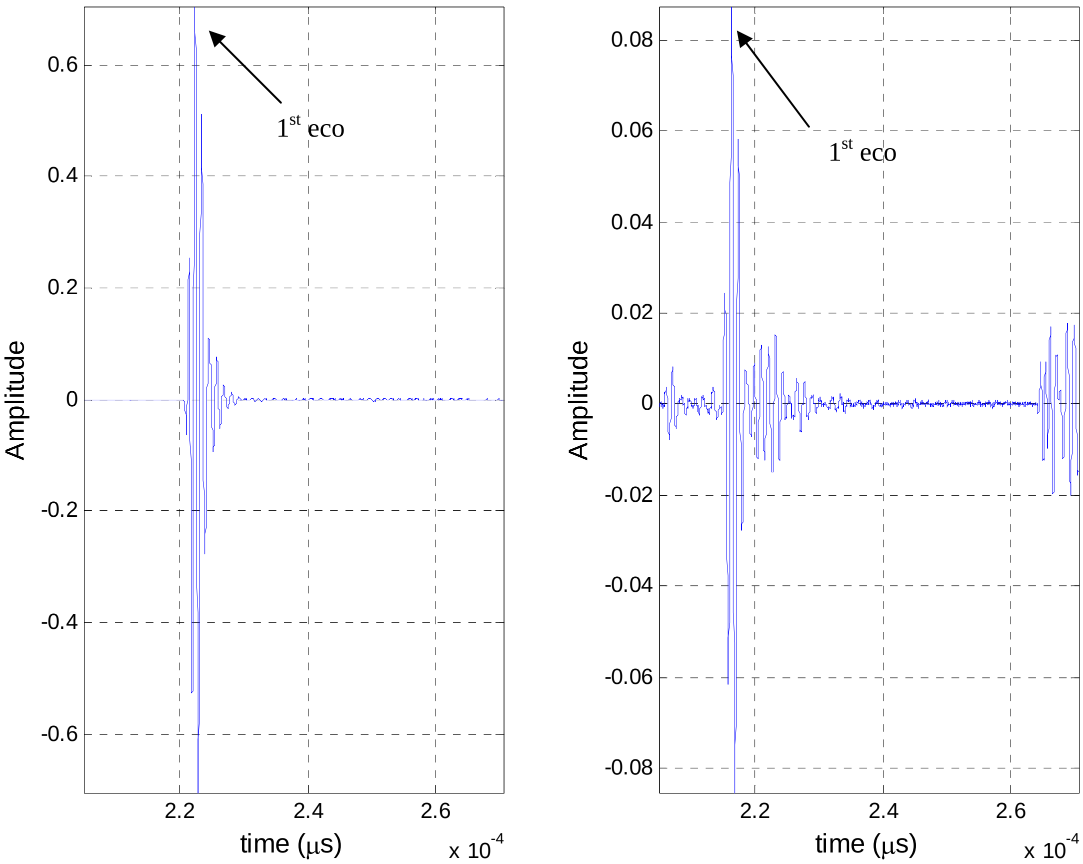


Fig. 3: Time of flight using the through-transmission technique. The time difference was calculated using cross-correlation of the 1st echo in (a) Reference signal (without sample) and (b) Signal with sample.

3.1. Velocity calculation

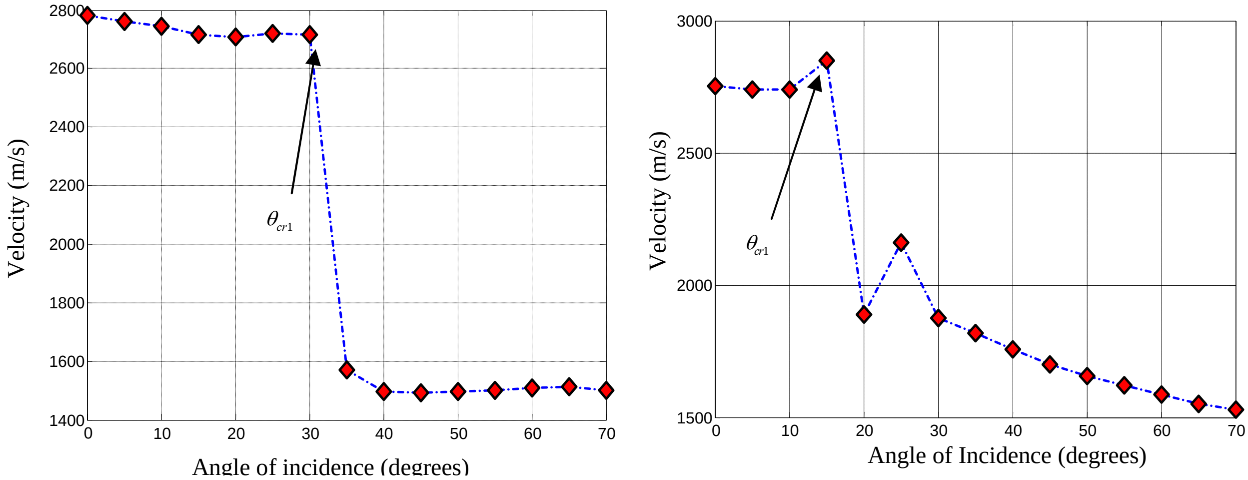
From the measured time-of-flight data, the phase velocity in the material for each incident angle with back-reflection technique is calculated using Eq. (12). In the case of through-transmission the phase velocity is calculated using the Eq. (13) (Rokhlin and Wang, 1992):

$$V_p = \left[\left(\frac{\Delta t}{2h} \right)^2 + \frac{\Delta t \cos \theta_i}{h V_w} + \left(\frac{1}{V_w} \right)^2 \right]^{-1/2} \tag{12}$$

$$V_p = \left[\left(\frac{\Delta t}{h} \right)^2 + \frac{2 \Delta t}{h V_w} \cos \theta_i + \left(\frac{1}{V_w} \right)^2 \right]^{-1/2} \tag{13}$$

Where V_p = phase velocity (m/s), V_w = velocity in water (m/s), h = thickness of material (mm), θ_i = incident angle (in radians), $\Delta t = t_2 - t_1$, time difference between the signal with (t_2) and without (t_1) sample (s).

The velocity plots of 9.60 mm thick unidirectional glass-epoxy composite plate are shown in Fig. 4. The longitudinal wave propagating in 1–2 plane shows slight anisotropy of the material since the velocity is slightly decreasing with the incident angle. The shear velocity in 1–2 plane is almost constant for all angles of propagation after the first (θ_{cr1}) critical angle, exhibiting transversely isotropy of the material. Because of the anisotropy of the medium in 1–3 plane, both longitudinal and shear velocities are changing with the angle. The longitudinal wave velocities in 1–3 plane is increasing with the increase in incident angle whereas the shear wave velocity is decreasing with angle.



(a) (b)

Fig. 4: Velocity vs. incident angle plot for sample 1 with data taken using back-reflection technique. (a) For wave propagating in 1-2 plane (isotropic plane). (b) For wave propagating in 1-3 plane (anisotropic plane).

3.2. Propagation angle

The calculated velocities were used in Snell's law to find the propagation angles in the composite samples. Snell's is given by

$$\theta_r = \sin^{-1}\left(\frac{V_p}{V_w}\sin\theta_i\right) \tag{14}$$

where θ_i = incidence angle (radians), θ_r = refracted angle (radians), V_w = velocity in water (m/s), V_p = velocity in the specimen (m/s).

3.3. Composite samples

Two unidirectional glass-epoxy composite samples were chosen as representative of transversely isotropic symmetric material and were prepared using manual lay-up technique. The samples were properly machined to give smooth surface finish on both sides. Tab. 1 gives the properties of each sample.

Tab. 1: Properties of the samples.

	Thickness (mm)	**Density (Kg/m^3)**	**Fiber volume fraction (%)**
Sample 1	9.60	1637	0.33
Sample 2	7.01	1901	0.52

4. Elastic constant estimation

The elastic constants were estimated using both back-reflection and through-transmission techniques on two unidirectional glass epoxy composite plates as representative of transversely isotropic material. The elastic constant C11 is calculated using the normal incidence velocity by solving the Eq. (8). The elastic constant C66 is calculated using average shear velocity measured in 1–2 plane (transversely isotropic plane), using the same equation. The other three unknown elastic constants are calculated from the measured velocity in 1–3 an isotropic plane using a nonlinear least-square optimization fitting. This technique minimizes the deviations between the measure and calculated ultrasonic velocities at different angles of propagation by determining the set of elastic constants. The algorithm is valid for materials of arbitrary anisotropy and for arbitrary directions.

To extract elastic constants of composites from the experimental angle-dependent phase velocity data by the non-linear least squares optimization technique, it is convenient to use analytical solutions of the Christoffel equation, which relates the propagation directions, the phase velocities and the elastic constants. This is done using Cardan's solution of the cubic equation (Rokhlin and Wang, 1992). The unknown material properties can be found by minimizing the sum of the squares of the deviations between the experimental and calculated velocities considering the elastic constants as variables in a multi-dimensional space *n* (Rokhlin and Wang, 1992):

$$\min_{C_{ijlm}\in\Re^n} \frac{1}{2}\sum_{i=1}^{m}\left(V_i^e - V_i^c\right)^2, \tag{15}$$

where n is the number of independent parameters to be extracted (elastic constants and density) and m is the number of measurements of velocities in different directions; V^e and V^c are the experimental and calculated phase velocities, respectively. For transversely isotropic materials, the number of independent elastic constants n should be 5. This method has the same advantages as the conventional least-squares method. For example, it minimizes the effect of random deviations of experimental points on the reconstruction of the results.

4.1. Rule of mixtures

If the volumes of fiber v_f and matrix v_m, the elastic constants of fiber E_f and matrix E_m are known, the longitudinal elastic constant E_L of the composite can be calculated, through the rule of mixtures formula given by (Jones, 1999):

$$E_L = v_f E_f + v_m E_m \tag{16}$$

5. Experimental results

Three trial sets of data were taken for each composite sample using the two experimental techniques. Based on these data sets, the elastic constants were obtained using a nonlinear least-square optimization algorithm. The results for both samples using immersion techniques are show in Tab. 2. Note that sample 1 has the lower fiber volume fraction and consequently the lower density. Table 3 show the engineering constants of both samples compared with literature and rule of mixtures.

Table 2: Results for both samples compared with literature. Elastic constants in GPa and density in kg/m^3.

		C11	C13	C33	C44	C66	ρ
Sample 1	Through-Transmission	12.22	10.98	29.27	2.6	3.43	1637
	Back-reflection	12.82	8.06	32.50	3.25	3.54	
Sample 2	Through-Transmission	16.15	6.95	42.50	3.64	4.68	1901
Literature	Ref. Balasubramaniam (1996)	15.48	7.18	38.51	4.54	4.77	1700
	Ref. Reddy et al. (2005)	15.04	11.70	49.60	3.90	4.63	1798

Table 3: Engineering constants of both samples compared with works in literature.

		E_3(GPa)	E_1(GPa)	σ_{12}	G12(GPa)	G23(GPa)
Sample 1	Back-reflection	25.50	9.53	0.34	3.54	3.25
	Rule of mixtures	26.77	*N/A*	*N/A*	*N/A*	*N/A*
Sample 2	Through-Transmission	38.29	12.89	0.30	4.68	3.64
	Rule of mixtures	39.98	*N/A*	*N/A*	*N/A*	*N/A*
Literature	Ref. Reddy et al. (2005)	36.46	11.54	*N/A*	4.63	3.90
	Ref. Jones (1999)	39.00	8.60	0.28	*N/A*	3.60

The measurements have been done using a goniometer device, which allows measurements with through-transmission or back-reflection technique. All experiments are made in-plane of symmetry of composites.

6. Conclusions

Two critical angles have been observed during the angle beam immersion evaluation of the two composite materials, one for quasi-longitudinal and other for quasi-transversal waves. It is shown that, for composite materials at the critical angle, both phase and group velocities are oriented parallel to the sample surface and, therefore, Snell's law can be used for phase velocity calculation from the critical angle measurements. By measuring phase velocities from the critical angles at different angles between the incident plane and the fiber direction, two phase velocities (quasi-longitudinal and quasi-shear) could be determined experimentally.

The elastic constants of two samples of composites are obtained by using a nonlinear least-squares optimization technique for the best fitting of experimental data. This fitting method minimizes the squares of the deviation between the theoretical and experimental velocities. Thus, more experimental data implies in better results.

The results of the two samples are in good agreement with the Rule of Mixtures. On the other hand, those experimental results are not in perfect agreement with the literature due to the difficulty of obtaining the same values for the densities. Density is a very important parameter in the determination of constants C11 and C66. Lower density implies lower longitudinal velocity and consequently lowers elastic constants of the composite sample.

The setup of the through-transmission technique showed a hard work to implement due to the following reasons: difficulty in the alignment of the two transducers and sample; and the position of the receiver has to be shifted according the sample thickness as shown in Fig. 1. In this figure, X is the horizontal displacement of the transmitted beam with respect to the incidence ray. At normal incidence X is zero and increases as the incident angle θ_i increases. In other hand, the back-reflection technique has an easier setup because it doesn't have those problems.

Both techniques can be used to measure all elastic constant of a cross-ply composite by adding a sample tilting device.

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9. Responsibility notice

The authors, Daniel Verga Boeri and Julio Cezar Adamowski, are the only responsible for the printed material included in this paper.