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Determination of the elastic properties in CFRP composites: comparison of different approaches based on tensile tests and ultrasonic characterization

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Abstract. The mechanical characterization of composite materials is nowadays a major interest due to their increasing use in the aeronautic industry. The design of most of these materials is based on their stiffness, which is mainly obtained by means of tensile tests with strain gauge measurement. For thin laminated composites, this classical method requires adequate samples with specific orientation and does not provide all the independent elastic constants. Regarding ultrasonic characterization, especially immersion technique, only one specimen is needed and the entire determination of the stiffness tensor is possible. This paper presents a study of different methods to determine the mechanical properties of transversely isotropic carbon fibre composite materials (gauge and correlation strain measurement during tensile tests, ultrasonic immersion technique). Results are compared to ISO standards and manufacturer data to evaluate the accuracy of these techniques.

Keywords: carbon fibre composite; elastic constants; immersion ultrasonic characterization; tensile test; stiffness tensor

1. Introduction

Fibre-reinforced composites are widely used for many structural applications. The primary benefits of the composite components are the reduction of weight and the simplification of assembly (Soutis 2005). The determination of the mechanical properties, specially the stiffness, is essential for ensuring performance to the composite structures. In addition, the knowledge of complete elastic stiffness matrix is important for modeling and evaluating the mechanical behavior of composite materials under loading conditions (El Bouazzaoui *et al.* 1996).

For now, tensile test with strain gauge measurements is the technique normalized by ISO standards to identify composites elastic properties. Such conventional method is destructive and provides only a part of elastic constants when thin plates like laminated composite structures are considered. Accordingly, ultrasonic techniques based on the measurement of ultrasonic wave

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velocities provide an interesting and non destructive way to address such issue.

First experimental studies based on ultrasonic technique were done by Zimmer and Cost (1970). They have used transmission contact technique to obtain dynamic elastic stiffness necessary to derive the unidirectional composite elastic behavior. Several ultrasonic bulk wave methods have also been developed to obtain phase velocities in anisotropic plates, especially when only one or two sides of the sample are accessible (Chu *et al.* 1994, Vishnuvardhan *et al.* 2007).

Immersion ultrasonic technique represents a particularly suitable method in the case of composite materials with small thickness (Munoz *et al.* 2014, Reddy *et al.* 2005). For a transversely isotropic composite, measurements in symmetry planes are sufficient to entirely determine the stiffness tensor (that is the five independents constants) (Baste and Hosten 1990). Various numerical methods and/or technical devices have then been implemented to interpret experimental results (Balasubramaniam and Whitney 1996, Kawashima *et al.* 1998).

Generally speaking, two experimental protocols can be used for such immersion technique to get velocities measurements, namely through-transmission and back-reflection techniques (Reddy *et al.* 2005). Through-transmission method requires two transducers, one to send the wave through the sample and the second one to receive the transmitted wave (Margueres 2000, Franco *et al.* 2010). As the ultrasonic wave travels through the test sample, the wave is reflected in part as it encounters a medium of different acoustic impedance. Then the transmitted wave is received by the transducer and displayed or stored for analyses. The difficulty of this method is that it requires a tracking of the arrival wave and therefore difficult to implement in the case of immersion.

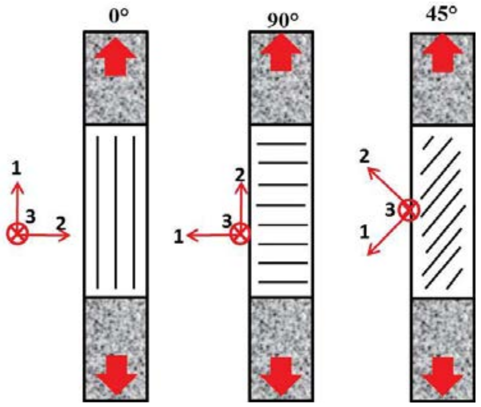
In the back-reflection technique, a transducer working in pulse/echo mode is associated to a large flat reflector which is positioned parallel to the transmitter. The back-reflected wave travels exactly along the same path as the incident wave in the opposite direction (Rokhlin and Wang 1992). When the sample angle is changed, the position of the incident wave on the back reflector is modified. Compared to through-transmission, such method appears then much relevant in the immersion case since it is not necessary to move the reflector or the transmitter/receiver transducer due to the large dimensions of the reflector.

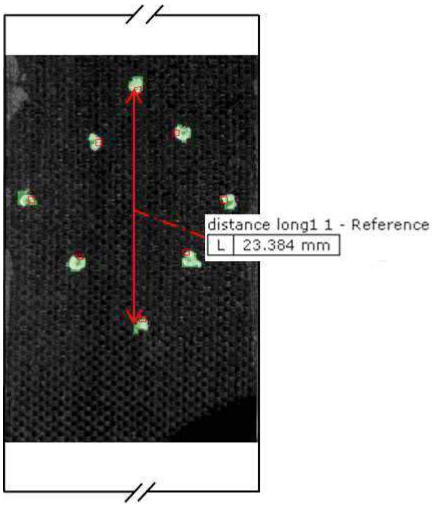
The aim of this paper is to compare the back-reflection immersion ultrasonic method with classical mechanical characterizations based on tensile tests. Strain gauges are used to measure the axial and lateral strains and stiffness constants are then estimated from the elastic parts of the stress-strain response. At the same time, a digital image correlation system is also implemented to corroborate the gauge data. Regarding immersion technique, a specific device including a rotation system has been set up to study the response of the material under various incident waves. These three results of stiffness tensor measurement (strain gauges, digital image correlation during tensile tests and ultrasonic characterization) are finally compared and discussed.

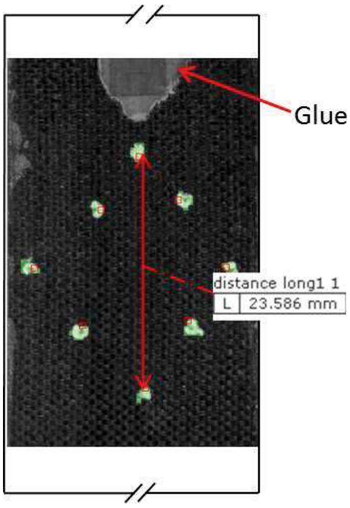
2. Specimen and experimental procedure

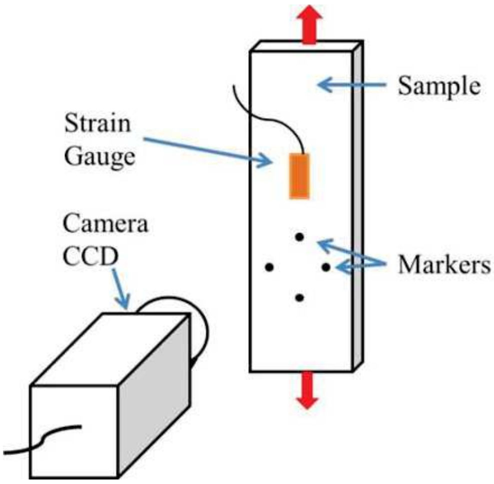
2.1 Specimen

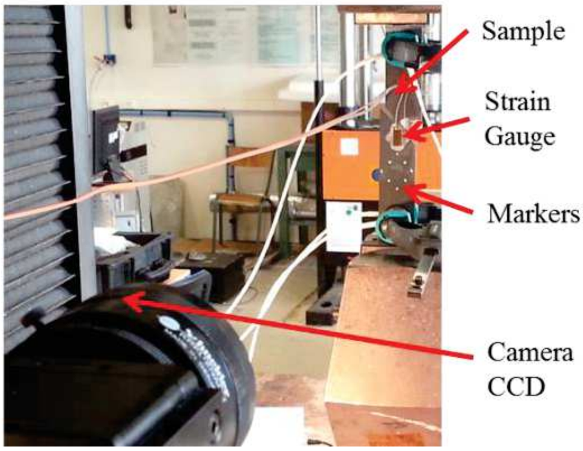
In view of its increasing use in aeronautics, a carbon fibre reinforced laminate is considered for this study. The M10R/38%/UD150/CHS composite is made of 14 unidirectional plies of prepreg leading to a thickness h=2 mm. Sample fabrication is carried out using the manual lay up technique. Then the sample is cured at 125°C during 90 minutes at a pressure of 2 bars as recommended by the material manufacturer Hexcel®. The plate was checked using ultrasonic C-


0°
90°
45°
1
2
3
2
3
1
2
3
1


distance long1 1 - Reference
L
23.384 mm


Glue
distance long1 1
L
23.586 mm


Sample
Strain Gauge
Camera CCD
Markers


Sample
Strain Gauge
Markers
Camera CCD

According to standards, elastic constants are calculated for strain states between $P_R/10$ and $P_R/2$ where P_R is the yield strength of the specimen. With the 0° tensile test, Young modulus E_1 and Poisson ratio ν_{12} are classically deduced from the ratios of axial stress and axial and lateral strains. Young modulus $E_2=E_3$ is calculated in the same way from 90° tensile test. Finally, Eq. (1) provides the shear modulus G_{12} by calculating Young modulus E_{45° obtained with the 45° tensile test

$$\frac{1}{G_{12}} = \frac{4}{E_{45^\circ}} - \frac{1}{E_1} - \frac{1}{E_2} + 2\frac{\nu_{12}}{E_1} \tag{1}$$

As said before, this procedure does not allow to obtain the last elastic property, namely the Poisson ratio ν_{23}, that would entirely determine the elastic behavior of the laminated composite.

2.3 Experimental procedure for ultrasonic characterization

2.3.1 General principle

The elastic stiffness matrix for a transversely isotropic material is given by the following tensor written in the coordinate axes described in Fig. 1

$$[C_{ijkl}] = \begin{bmatrix} C_{1111} & C_{1122} & C_{1122} & 0 & 0 & 0 \\ C_{1122} & C_{2222} & C_{2233} & 0 & 0 & 0 \\ C_{1122} & C_{2233} & C_{2222} & 0 & 0 & 0 \\ 0 & 0 & 0 & C_{2323} & 0 & 0 \\ 0 & 0 & 0 & 0 & C_{1212} & 0 \\ 0 & 0 & 0 & 0 & 0 & C_{1212} \end{bmatrix} \tag{2}$$

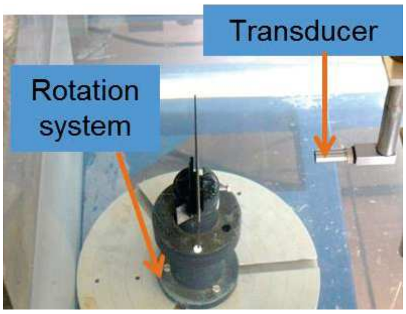
Ultrasonic characterization aims at providing five components representative of the elastic behavior of such material, namely C_{1111}, C_{2222}, C_{1122}, C_{2323} and C_{1212} (C_{2233} can be derived from C_{2222} and C_{2323} based on the elastic relations). This determination relies on the resolution of the Christoffel equation (Rose 1999)

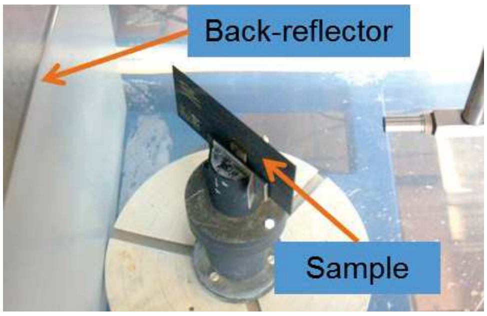
$$|n \cdot C \cdot n - \rho V(n) I| = 0 \tag{3}$$

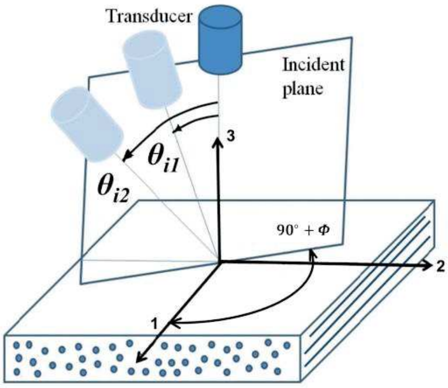
where $V(n)$ is the wave velocity related to the propagation direction of unit vector n, ρ is the material density and I is identity second-order tensor. Accordingly, this requires the measurement of ultrasonic velocities for different orientations of wave propagation in the sample. For a transversely isotropic composite, measurements in planes of symmetry are sufficient to determine all five independent elastic constants (Aristégui and Baste 1997, Chu *et al.* 1994).

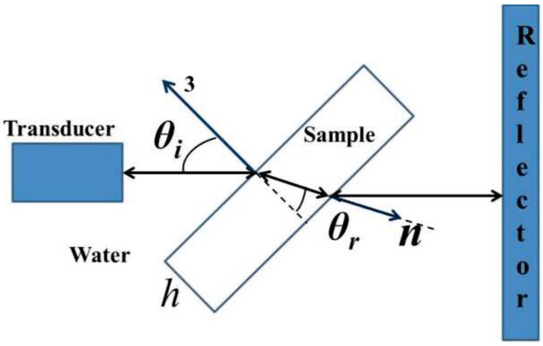
For the immersion ultrasonic technique, water acts as a couplant that transfers the wave from the transducer to the sample under inspection (Castagnède *et al.* 1990, Rokhlin and Wang 1992). The transducer is not directly connected with the sample and hence consistent coupling is ensured. In this way, it is possible to measure the wave velocities at different angles of propagation either by adjusting transducer orientation or by rotating the sample (Reddy *et al.* 2005). Here the sample is held and moved using a rotation system (see Fig. 4). As ultrasonic generator, an Omniscan 32: 128 PR is used with a mono-element transducer connected to it. The transducer acts at the same time as ultrasonic source and receiver (reflection mode). Classical value of frequency is used, namely 5 MHz, which allows mainly to avoid inside reflection; back-reflector and specimen were checked for alignment at normal incidence.

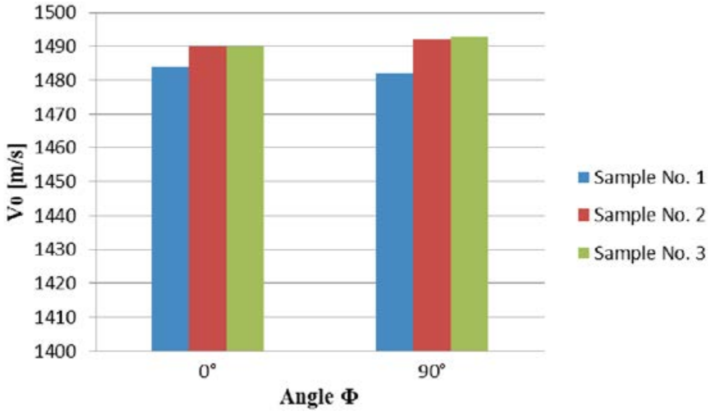
The measurements are performed for different angles Φ between plane 2-3 and incident plane (see Fig. 5). For a given Φ, the sample is then rotated in the incident plane for different incident angles θ_i between the transducer and axis 3 of the sample. The wave passes through the sample in

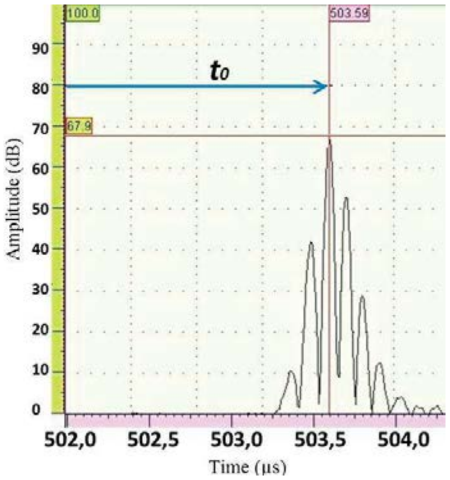

Transducer
Rotation system

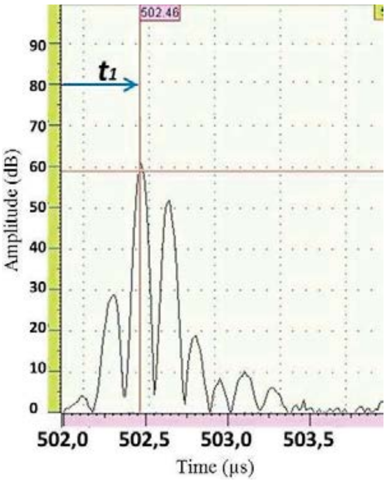

Back-reflector
Sample

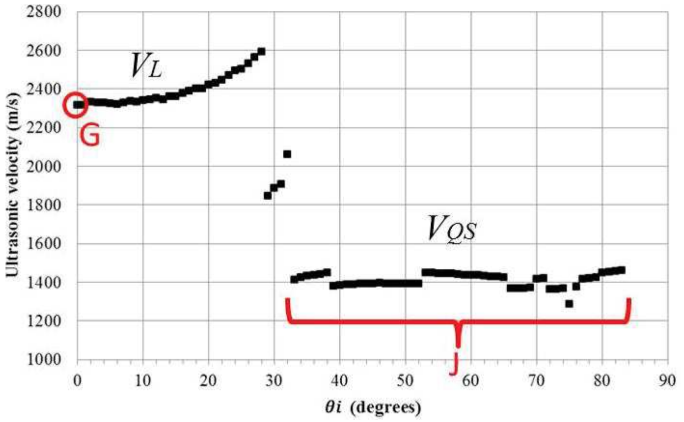

Transducer
Incident plane
θ_{i1}
θ_{i2}
3
$90^\circ + \Phi$
2
1


3
Transducer
θ_i
Sample
θ_r
$\vec{n}$
Water
h
Reflector

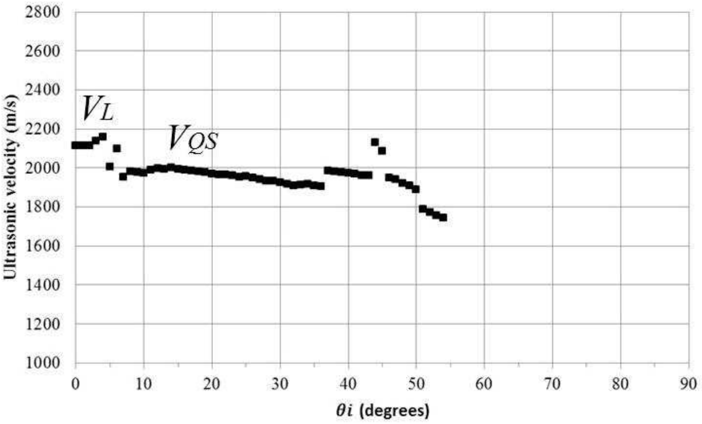

1500
1490
1480
1470
1460
1450
1440
1430
1420
1410
1400
V0 [m/s]
0°
90°
Angle Φ
Sample No. 1
Sample No. 2
Sample No. 3


100.0
503.59
67.9
t0
90
80
70
60
50
40
30
20
10
0
Amplitude (dB)
502,0
502,5
503,0
503,5
504,0
Time (µs)


502.46
t1
90
80
70
60
50
40
30
20
10
0
Amplitude (dB)
502,0
502,5
503,0
503,5
Time (µs)



2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
Ultrasonic velocity (m/s)
0
10
20
30
40
50
60
70
80
90
θi (degrees)
V_L
G
V_{QS}
J



2800
2600
2400
2200
2000
1800
1600
1400
1200
1000
Ultrasonic velocity (m/s)
0
10
20
30
40
50
60
70
80
90
θi (degrees)
V_L
V_{QS}

$$C_{2323} = \rho\left(V_{QS}^{mean}(\Phi = 0°)\right)^2 \tag{6}$$

C_{1111}, C_{1122} and C_{1212} are obtained from both longitudinal and shear velocity data measured in 1-3 plane. Experimental velocity data is fitted in the following two relations obtained from the Christoffel equation's solution (Eq. (3)). This allows then to determine the three unknown parameters.

$$V_L(\Phi = 90°, \theta_i) = \sqrt{\frac{A+\sqrt{A^2-4B}}{2\rho}} \tag{7}$$

$$V_{QS}(\Phi = 90°, \theta_i) = \sqrt{\frac{A-\sqrt{A^2-4B}}{2\rho}} \tag{8}$$

where

$$A = (C_{1122}\cos^2\theta_i + C_{1111}\sin^2\theta_i + C_{1212}) \tag{9}$$

$$B = C_{2222}C_{1212}\, cos^4\,\theta_i + C_{1111}C_{1212}\, sin^4\,\theta_i + \frac{sin^2\,2\theta_i}{4}[C_{2222}C_{1111} + C_{1212}^2 - (C_{1122} + C_{1212})^2] \tag{10}$$

To solve undetermined Eqs. (7)-(8), one can use the nonlinear least-square optimization technique which minimizes the deviations between the experimental and theoretical velocities for the considered angles of propagation (Reddy *et al.* 2005)

$$\min_{C_{ijkl}\in R^n} \frac{1}{2}\sum_{i=1}^{m}\left(V_i^e - V_i^t\right)^2 \tag{11}$$

In Eq. (11), *n* is the number of independent parameters to be extracted (here 5 elastic constants) and *m* is the number of measurements of velocities in different directions (here 137 experimental data). V^e and V^t are the experimental and theoretical phase velocities, respectively.

In a last step, component C_{2233} is calculated using this relation between elastic stiffness components

$$C_{2233} = C_{2222} - 2C_{2323} \tag{12}$$

Engineering moduli in the coordinate system (E_1, E_2=E_3, ν_{12}, ν_{23} and G_{12}) can be deduced by inverting the stiffness tensor.

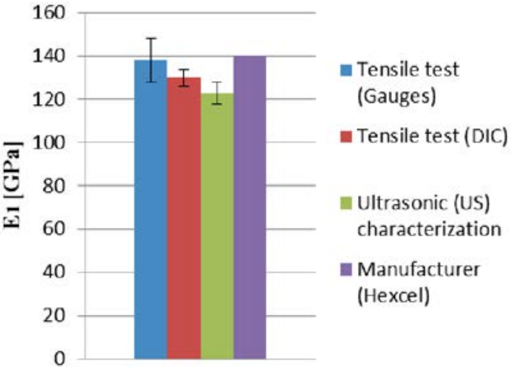
3. Results and discussions

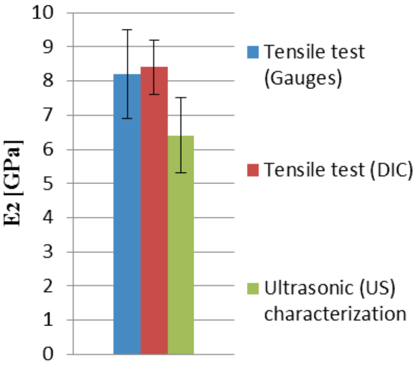
Fig. 10 summarizes the elastic constants estimation obtained by means of the three different methods used to characterize the carbon fibre/epoxy composite. The arithmetic mean $\overline{x}$ is calculated as the sum of the sampled values divided by the number *N* of tests (*N*=3 for each method)

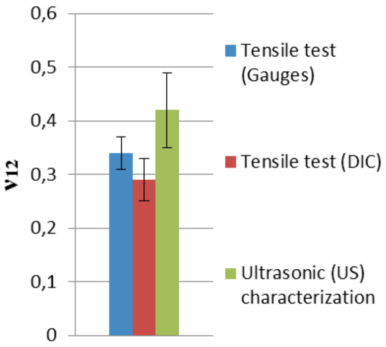
$$\overline{x} = \frac{1}{N}\sum_{i=1}^{N} x_i \tag{13}$$

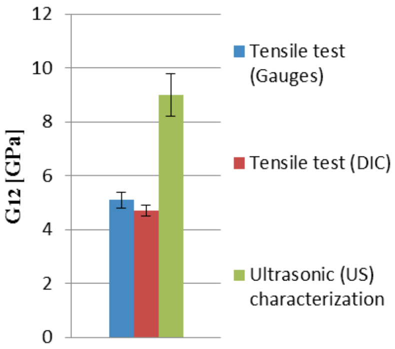
The standard deviation σ is found by taking the square root of the average of the squared differences of the values x_i from their average value

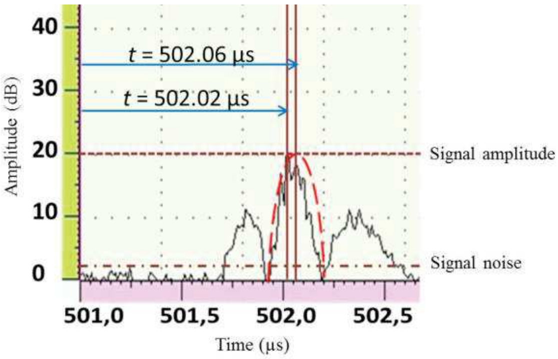
$$\sigma = \sqrt{\frac{1}{N}\sum_{i=1}^{N}(x_i - \overline{x})^2} \tag{14}$$

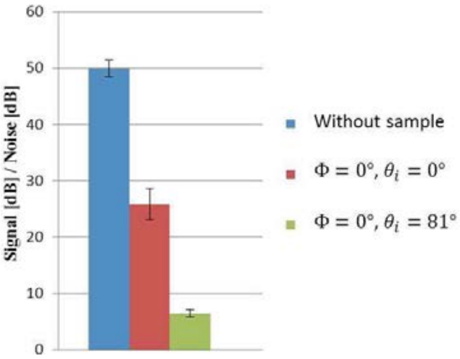

E1 [GPa]
160
140
120
100
80
60
40
20
0
Tensile test (Gauges)
Tensile test (DIC)
Ultrasonic (US) characterization
Manufacturer (Hexcel)


E2 [GPa]
10
9
8
7
6
5
4
3
2
1
0
Tensile test (Gauges)
Tensile test (DIC)
Ultrasonic (US) characterization


ν12
0,6
0,5
0,4
0,3
0,2
0,1
0
Tensile test (Gauges)
Tensile test (DIC)
Ultrasonic (US) characterization


G12 [GPa]
12
10
8
6
4
2
0
Tensile test (Gauges)
Tensile test (DIC)
Ultrasonic (US) characterization


40
30
20
10
0
Amplitude (dB)
t = 502.06 µs
t = 502.02 µs
Signal amplitude
Signal noise
501,0
501,5
502,0
502,5
Time (µs)


60
50
40
30
20
10
0
Signal [dB] / Noise [dB]
Without sample
$\Phi = 0°, \theta_i = 0°$
$\Phi = 0°, \theta_i = 81°$

unidirectional carbon fibre/epoxy laminates. Two strain measurement techniques (gauge as standard, Digital Image Correlation) during tensile tests and the immersion ultrasonic method were analyzed.

Results highlight first the interest of DIC to determine the strain response and its good accuracy on the final determination of elastic constants. Yet, tensile tests do not allow to entirely provide the elastic stiffness and some indetermination remains for structures calculation.

This study has shown also the ability of the immersion ultrasonic technique to derive all elastic constants of a UD laminated composite through the measurement of time of flight inside a sample. The main advantage of this technique is that only one specimen is needed instead of cutting many samples with desired size and shape for a classical characterization tensile test. If elastic properties estimations are quite encouraging, some limitations on the precision have been noted that would require further investigations on the influence of the time of flight. This could be improved by adjusting the experimental set-up in such a way that the distance between transducer and reflector is fixed during all the acquisition or by using two transducers (through-transmission). Also, several signal data processing will be tested for weak amplitude signals in order to estimate the influence of the definition of the TOF on the elastic properties.

Given that, this immersion ultrasonic method could then be extended with the same principle to more complex anisotropic materials including orthotropic symmetry. This would need another degree of freedom to rotate the sample that will provide the possibility to propagate ultrasonic waves along more azimuthal angles/planes with respect to the fibre axis.

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