COMPARATIVE STUDY ON THE DETERMINATION OF THE ELASTIC PROPERTIES OF COMPOSITE MATERIALS BY TENSILE TESTS AND ULTRASOUND MEASUREMENT

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SUMMARY : This paper presents a comparative study on different ways to determine the mechanical properties of glass fibre composite materials. This work underlines the accuracy of the results obtained with a dynamic characterisation method which is developed as part of non destructive evaluation by ultrasonic waves, compared to a static characterisation method by tensile tests.

KEYWORDS : mechanical properties, characterisation, non-destructive evaluation, tensile tests, ultrasound measurement, glass fibres.

INTRODUCTION

The mechanical characterisation of composite materials is a research axis of scientific and economic importance. In fact, it is i.e. essential to measure the elastic constants of a material with accuracy in order to realize structural analysis and optimize the design. Among the developed methods, the static methods based on strain measurement present some disadvantages, that is destructive evaluation and the difficulties indeed impossibilities to measure the out-of-plane elastic modulus because of the thin thickness of the plates even if in-plane ones could be determined by only two tests [1].

These disadvantages could be avoided by the use of non-destructive dynamic methods such as modal analysis [2-3] or ultrasonic wave evaluation [4-5]. Among the non-destructive methods, ultrasounds are efficient and easy to use. Besides, because of the accuracy of the results as well as the repetitivity of the measures, scientists and industrialists are generalizing this method.

In this paper, the results obtained on the one hand by tensile tests and on the other hand by ultrasound tests are compared. The ultrasound tests are realized on two systems, the first one is a characterisation with direct contact and the second one is a characterisation in immersion. The tests are applied on a glass fibre-polyester composite material, which is a unidirectional fibre (Roving).

CONSTITUTIVE EQUATIONS

Dynamic Case

The solid is submitted to waves. The local displacement u_i in each location x_k of this solid varies with time : $u_i = u_i(x_k, t)$.

$$\rho \frac{\partial^2 u_i}{\partial t^2} = C_{ijkl} \frac{\partial^2 u_l}{\partial x_j \partial x_k} \tag{1}$$

The solution is known as a progressive wave which propagates in the direction defined by the unitary vector $\vec{n}(n_1, n_2, n_3)$ perpendicular to the wave planes $\vec{n}.\vec{x} = cst$:

$$u_i = u_i^0 F(t - \frac{\vec{n}.\vec{x}}{V}) \tag{2}$$

From Eqn 1 and 2, V denotes the phase velocity and u_i^0 the polarization of the wave, the Christoffel equation is :

$$\rho V^2 u_i^o = C_{ijkl} n_j n_k u_l^0 \tag{3}$$

Then introducing the second order tensor $\Gamma_{il} = C_{ijkl} n_j n_k$, Eqn 3 becomes :

$$\Gamma_{il} u_l^0 = \rho V^2 u_i^0 \tag{4}$$

So the phase velocities and the polarization of the plane waves propagate following the direction $\vec{n}$ in a crystal of stiffness C_{ijkl} are the eigenvalues and the eigenvectors of the tensor $\Gamma_{il} = C_{ijkl} n_j n_k$.

The components of the propagation tensor Γ_{il} from the general equation 4 are [6] :

$$\begin{gathered}
\Gamma_{11} = C_{11}n_1^2 + C_{66}n_2^2 + C_{55}n_3^2 + 2C_{16}n_1n_2 + 2C_{15}n_1n_3 + 2C_{56}n_2n_3 \\
\Gamma_{12} = C_{16}n_1^2 + C_{26}n_2^2 + C_{45}n_3^2 + (C_{12} + C_{66})n_1n_2 + (C_{14} + C_{56})n_1n_3 + (C_{46} + C_{25})n_2n_3 \\
\Gamma_{13} = C_{15}n_1^2 + C_{46}n_2^2 + C_{35}n_3^2 + (C_{14} + C_{56})n_1n_2 + (C_{13} + C_{55})n_1n_3 + (C_{36} + C_{45})n_2n_3 \\
\Gamma_{22} = C_{66}n_1^2 + C_{22}n_2^2 + C_{44}n_3^2 + 2C_{26}n_1n_2 + 2C_{46}n_1n_3 + 2C_{24}n_2n_3 \\
\Gamma_{23} = C_{56}n_1^2 + C_{24}n_2^2 + C_{34}n_3^2 + (C_{46} + C_{25})n_1n_2 + (C_{36} + C_{45})n_1n_3 + (C_{23} + C_{44})n_2n_3 \\
\Gamma_{33} = C_{55}n_1^2 + C_{44}n_2^2 + C_{33}n_3^2 + 2C_{45}n_1n_2 + 2C_{35}n_1n_3 + 2C_{34}n_2n_3 \\
\Gamma_{21} = \Gamma_{12} \quad \Gamma_{31} = \Gamma_{13} \quad \Gamma_{32} = \Gamma_{23}
\end{gathered} \tag{5}$$

In non-destructive evaluation, transmission and reflection methods are developed in order to characterize materials. In these two cases the wave amplitude and the time of crossing enables the crossed environment to be defined. Ultrasound measurements of longitudinal and transverse velocities makes it possible to characterize composite materials. Then the elastic mechanical properties are obtained : elastic modulus, shear modulus and Poisson coefficient.

Wave propagation in particular directions

For an orthotropic composite material, the relation between elastic modulus and propagation velocity of ultrasound waves is obtained by the propagation of plane waves following appropriate directions in the material [7]. In the equation below C_{ij} represents the component of the orthotropic stiffness matrix and, V_{ij} , V_{Si} the propagation velocities defined by Eqn 5 (ρ is the density of the material) :

$$\begin{aligned}
&C_{11} = \rho.V_{11}^2 \quad , \quad C_{44} = \rho.V_{23}^2 = \rho.V_{32}^2 \\
&C_{22} = \rho.V_{22}^2 \quad , \quad C_{55} = \rho.V_{13}^2 = \rho.V_{31}^2 \\
&C_{33} = \rho.V_{33}^2 \quad , \quad C_{66} = \rho.V_{12}^2 = \rho.V_{21}^2 \\
&C_{23} = \sqrt{(C_{22} + C_{44} - 2\rho.V_{S1}^2).(C_{33} + C_{44} - 2\rho.V_{S1}^2)} - C_{44} \\
&C_{13} = \sqrt{(C_{11} + C_{55} - 2\rho.V_{S2}^2).(C_{33} + C_{55} - 2\rho.V_{S2}^2)} - C_{55} \\
&C_{12} = \sqrt{(C_{11} + C_{66} - 2\rho.V_{S3}^2).(C_{22} + C_{66} - 2\rho.V_{S3}^2)} - C_{66}
\end{aligned} \tag{7}$$

The inversion of the stiffness matrix gives :

$$[S] = \frac{1}{D} \cdot \begin{bmatrix}
C_{22} \cdot C_{33} - C_{23}{}^2 & C_{13} \cdot C_{23} - C_{12} \cdot C_{33} & C_{12} \cdot C_{23} - C_{13} \cdot C_{22} & 0 & 0 & 0 \\
C_{13} \cdot C_{23} - C_{12} \cdot C_{33} & C_{11} \cdot C_{33} - C_{13}{}^2 & C_{12} \cdot C_{13} - C_{11} \cdot C_{23} & 0 & 0 & 0 \\
C_{12} \cdot C_{23} - C_{13} \cdot C_{22} & C_{12} \cdot C_{13} - C_{11} \cdot C_{23} & C_{11} \cdot C_{22} - C_{12}{}^2 & 0 & 0 & 0 \\
0 & 0 & 0 & \frac{D}{C_{44}} & 0 & 0 \\
0 & 0 & 0 & 0 & \frac{D}{C_{55}} & 0 \\
0 & 0 & 0 & 0 & 0 & \frac{D}{C_{66}}
\end{bmatrix} \tag{8}$$

with, $D = C_{11}.C_{22}.C_{33} - C_{11}.C_{23}^2 - C_{33}.C_{12}^2 - C_{22}.C_{13}^2 + 2.C_{12}.C_{13}.C_{23}$.

From Eqn 8 the mechanical properties are deduced :

$$\begin{aligned}
&E_1 = \frac{D}{(C_{22}.C_{33} - C_{23}^2)} \quad , \quad E_2 = \frac{D}{(C_{11}.C_{33} - C_{13}^2)} \quad , \quad E_3 = \frac{D}{(C_{11}.C_{22} - C_{12}^2)} \\
&G_{23} = C_{44} \quad , \quad G_{13} = C_{55} \quad , \quad G_{12} = C_{66} \\
&\nu_{23} = \frac{-E_2(C_{12}.C_{13} - C_{23}.C_{11})}{D} \quad , \quad \nu_{32} = \frac{-E_3(C_{12}.C_{13} - C_{23}.C_{11})}{D} \\
&\nu_{13} = \frac{-E_1(C_{12}.C_{23} - C_{13}.C_{22})}{D} \quad , \quad \nu_{31} = \frac{-E_3(C_{12}.C_{23} - C_{13}.C_{22})}{D} \\
&\nu_{12} = \frac{-E_1(C_{13}.C_{23} - C_{12}.C_{33})}{D} \quad , \quad \nu_{21} = \frac{-E_2(C_{13}.C_{23} - C_{12}.C_{33})}{D}
\end{aligned} \tag{9}$$

Determination of effective elastic constants from ultrasonic velocity measurements varying the incidence in immersion

Wave propagation in anisotropic media

The ultrasonic characterization of materials is based on the relationship between the elastic constants and the phase velocities of progressive plane waves. If a plane displacement wave is substituted into the displacement equation of motion, the characteristic equation for plane waves propagating in anisotropic media is given by [6-8] :

$$\det\left[\Gamma_{ij} - \rho V^2 \delta_{ij}\right] = 0 \tag{10}$$

where $\Gamma_{ij} = C_{ijkl}\, n_j n_k$, ρ is the mass density, V is the phase velocity and δ_{ij} is the Kronecker delta. In the definition of Γ_{ij}, known as the Christoffel tensor, C_{ijkl} are the elastic constants, components of the fourth rank stiffness tensor, and n is the unit vector normal to the plane wave.

Instead of the tensor components C_{ijkl} it is convenient to use the matrix components C_{ij} with the contracted subscript notation [9-10]. The number of independent elastic constants characterizing the material depends on its structural anisotropy i.e. the arrangement of the material constituants (atoms for single crystals, grains for polycrystals, fibres for fibrous composites...). For orthotropic material considered in this work (laminated aluminium alloys), there are 9 independent elastic constants which characterize the elastic behaviour. Then, the associated matrix expressed in the principal axis 1,2,3 is:

$$[C_{ij}] = \begin{bmatrix} C_{11} & C_{12} & C_{13} & 0 & 0 & 0 \\ & C_{22} & C_{23} & 0 & 0 & 0 \\ & & C_{33} & 0 & 0 & 0 \\ & & & C_{44} & 0 & 0 \\ & \text{Sym} & & & C_{55} & 0 \\ & & & & & C_{66} \end{bmatrix} \tag{11}$$

Since Eqn 10 is cubic in ρV^2, three modes may be propagated along the direction n with different velocities and polarisation. Except along particular directions, these modes are not pure ones i.e. longitudinal or transversal. They are most often denoted QL : quasi-longitudinal and QT1 and QT2 : quasi-transversal. Their velocities are the solution of Eqn 10. In our case, it is convenient to use a numerical resolution of the Christoffel equation which relates the phase velocities to the propagation direction and the elastic constants. So, knowing the elastic constants of a material, it is possible to calculate any velocity for a given direction of propagation.

The inverse problem resolution

As we have seen by knowing the elastic constants of a material, it is possible to calculate the velocity for any direction of propagation. Determining the elastic constants from velocity measurements is the inverse problem. These elastic constants have to be recovered from the analytical expressions of the velocities derived from Eqn 10 and from suitable experimental

values of velocities. The most suitable method to solve this problem is based on a nonlinear optimization technique.

Thus, the experimental device allows multiple velocity measurements (varying the direction of propagation), for principal and non principal planes of propagation. The problem is then overprescripted, there being more data (measured velocities) than independent parameters to determine (elastic constants). We then use a least square fit of the calculated to the measured velocities and, through this optimization process optimal values of elastic constants that minimize the velocity deviation are obtained. So in practice, the unknown material properties are found by minimizing the sum of the squares of deviations between the experimental and calculated velocities, considering the elastic constants as unknown. The least square algorithm we use is the Levenberg Marquardt one [11].

This two-step procedure (velocity measurements in different directions - elastic constants recovering) has been used to characterize the sample in its initial state, and then for the same sample, at different levels of the applied static load.

Static Case

The roving, which is an unidirectionnal composite material, has a transversely isotropic behaviour. Five independant elastic coefficients take place in the constitutive law [9]. These five coefficients are : the longitudinal elastic modulus E_L, the transverse elastic modulus E_T, the shear in-plane modulus G_{LT}, and the Poisson coefficient ν_{LT} and ν_{TL}.
The stiffness matrix is :

$$\begin{bmatrix} C_{11} & C_{12} & C_{13}=C_{12} & 0 & 0 & 0 \\ & C_{22} & C_{23} & 0 & 0 & 0 \\ & & C_{33}=C_{22} & 0 & 0 & 0 \\ & & & C_{44}=\dfrac{C_{22}-C_{23}}{2} & 0 & 0 \\ & \text{Sym} & & & C_{55}=C_{66} & 0 \\ & & & & & C_{66} \end{bmatrix} \tag{12}$$

The compliance matrix is :

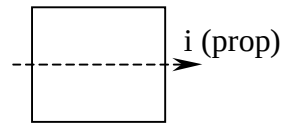
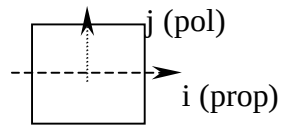
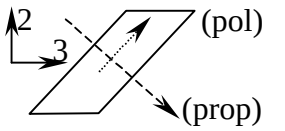
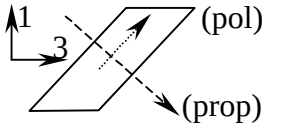
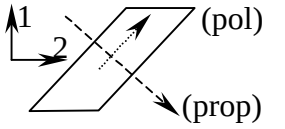
$$\begin{bmatrix} \dfrac{1}{E_L} & -\dfrac{\nu_{LT}}{E_L} & -\dfrac{\nu_{LT}}{E_L} & 0 & 0 & 0 \\ & \dfrac{1}{E_T} & -\dfrac{\nu_{TL}}{E_T} & 0 & 0 & 0 \\ & & \dfrac{1}{E_T} & 0 & 0 & 0 \\ & & & \dfrac{2(1+\nu_{LT})}{E_T} & 0 & 0 \\ & \text{Sym} & & & \dfrac{1}{G_{LT}} & 0 \\ & & & & & \dfrac{1}{G_{LT}} \end{bmatrix} \tag{13}$$

EXPERIMENTATION

Direct Contact Device

The contact ultrasound characterization is a destructive evaluation as regards the samples which are cut out using the diamond wire saw ESCIL. This method requires four types of samples (Table 1) : A-type in order to measure V_{ii} and V_{ij}, B-C-D-type to measure V_{Si}.

Table 1 : Velocities measurements and samples (pol : polarization, prop : propagation)

Velocity	Sample type / Wave type	Geometry of samples
$V_{ii}\begin{cases}V_{11}\\V_{22}\\V_{33}\end{cases}$	A / Longitudinale	
$V_{ij}\begin{cases}V_{12}\\V_{13}\\V_{23}\end{cases}$	A / Transversale	
V_{S1}	B / Transversale	
V_{S2}	C / Transversale	
V_{S3}	D / Transversale	

The ultrasound characterisation device with direct contact is the same as the system for velocity of ultrasound measurement [12]. The device (Fig. 1) used for the ultrasonic characterization of the materials consists of the following : a pulse generator Panametrics Sofranel (200MHz), a numerical oscilloscope Hewlett Packard 5420A (500MHz), transducers in the range 1 to 20 Mhz. Measurement is done by transparency, the wave transmitted by the transmitter crosses the sample to be picked up by the receiver. The direct contact technique is used to transmit the ultrasonic wave.

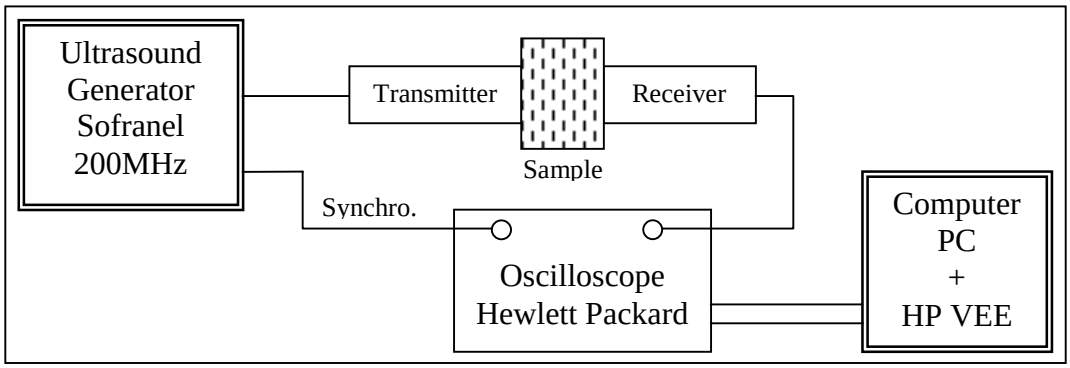


Fig. 1 : Contact ultrasound device

Immersion Experimental Device

To determine the velocities of propagation as a function of the incidence angle, a complex device has been elaborated. It consists of an immersion ultrasonic tank and its data collection [13]. In this study, we use the simple through transmission technique. The sample being placed between two transducers (Fig. 2).

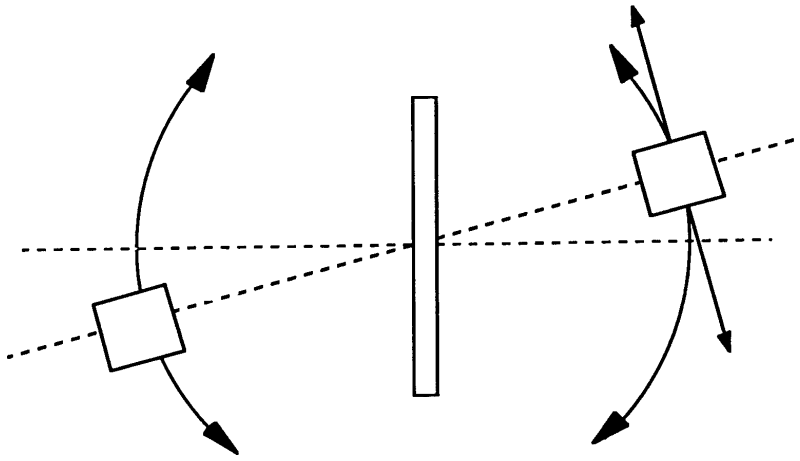


Fig. 2 : Immersion experimental device

So, varying the angle of incidence provides numerous directions and modes of propagation in the sample, according to Snell-Descartes's laws. To compute the transit time inside the sample, a reference signal, corresponding to the propagation in water without the sample is needed. An appropriate signal processing of the received pulses using the cross-correlation function enables us to measure the velocities for each mode along its own direction of propagation. For this determination, in addition to the transit time at the incidence angle, the thickness of the sample has also to be accurately known. As the thickness varies with the static load, we have developed a particular procedure enabling its automatic measurement from ultrasonic signals obtained at normal incidence.All the controls (temperaure, rotations), the thickness and the velocity measurements are computer assisted. With this device and when the temperature is controlled to 2/100 of °C, we reach an uncertainty on the velocity measurement of less than 2m/s [14-15].

Tensile Tests

Tensile tests are the direct way to characterize the mechanical elastic properties of materials. This method consist of applying a regular increasing load on a sample so that it can be considered as uniaxial loading in a uniform manner. On a normal cross-sectional area, the stress statement then comes in a uniform distribution of tensile stresses. These tensile tests follow the EN61 norm and are performed on a INSTRON 50kN tensile testing machine. Measurement of strains is accomplished by the use of two perpendicular strain transducers.
In longitudinal tensile tests (0°), a load F_1 is applied on the sample cross-sectional area S_1 of the composite material. The stress is given by the following equation :

$$\sigma_{11} = \frac{F_1}{S_1} \tag{14}$$

The experimental characterization consist of the measurement of three values : the load F_1, the stretching ΔL_1 along the length L_1, the stretching ΔL_2 along the transversal dimension L_2.
The longitudinal and transverse strain are given respectively by :

$$\varepsilon_{11} = \frac{\Delta L_1}{L_1} \text{ and } \varepsilon_{22} = \frac{\Delta L_2}{L_2} \tag{15}$$

The longitudinal modulus E_L and the poisson coefficient ν_{LT} are deduced from the following expressions :

$$E_L = \frac{\sigma_{11}}{\varepsilon_{11}} \text{ and } \nu_{LT} = -\frac{\varepsilon_{22}}{\varepsilon_{11}} \tag{16}$$

In the case of a transverse tensile test, a load F_2 is applied in the transverse direction (90°) :

$$E_T = \frac{\sigma_{22}}{\varepsilon_{22}} \text{ and } \nu_{TL} = -\frac{\varepsilon_{11}}{\varepsilon_{22}} \tag{17}$$

It is possible to check the reliability and consistency of the resulting elastic modulus with the hypothesis of symetry of the compliance matrix :

$$\frac{\nu_{LT}}{E_L} = \frac{\nu_{TL}}{E_T} \tag{18}$$

For 45° tensile tests, the load F is applied on the cross-sectional area S. The measurement of the stretching in this direction allows to deduce the elastic modulus ($E_{45°}$).
Then from these three tests the shear modulus G_{LT} could be obtained [10]:

$$\frac{1}{E_{45°}} = \frac{1}{E_L} \cdot \cos^4\theta + \frac{1}{E_T} \cdot \sin^4\theta + \left(\frac{1}{G_{LT}} - 2 \cdot \frac{\nu_{LT}}{E_L} \right) \cdot \sin^2\theta \cdot \cos^2\theta \tag{19}$$

$$\frac{1}{G_{LT}} = \frac{4}{E_{45°}} - \frac{1}{E_L} - \frac{1}{E_T} + 2\frac{\nu_{LT}}{E_L} \tag{20}$$

RESULTS

Each sample possesses a different fibre rate (V_{fibre}) contained between 38% and 53% in volume (Table 2).

Table 2 : Results from samples of different fibre rate [GPa]

	E_1	E_2	E_3	ν_{12}	ν_{13}	ν_{23}	G_{12}	G_{13}	G_{23}
Tensile (V_{fibre}=52,2%)	44,49	13,25	13,25	0,28	0,28	-	5,27	5,27	-
Contact (V_{fibre}=38,7%)	31,85	10,51	10,29	0,35	0,38	0,40	4,23	4,29	3,86
Immers (V_{fibre}=52,7%)	45,50	14,70	14,90	0,31	0,27	0,40	6,00	6,50	4,70

With the aim of comparing the results obtained on several samples by the three methods, the fibre rate has to come down to an identical rate of 40%. For this composite material (Glass fibre-Polyester) the engineering constants are quite proportionnal to the fibre rate between 40

to 50%. From the results obtained for each method, we could determine the nine coefficient of the orthotropic behaviour, and then the five independant coefficients (Table 3).

Table 3 : Comparative results ultrasound-tensile tests (volumic fibre rate : 40%)[GPa]

	$E_1=E_L$	$E_2=E_T$	$E_3=E_T$	$\nu_{12}=\nu_{LT}$	$\nu_{13}=\nu_{LT}$	$\nu_{23}=\nu_{TT}$	$G_{12}=G_{LT}$	$G_{13}=G_{LT}$	$G_{23}=G_{TT}$
Tensile	34,07	10,15	10,15	0,35	0,35	-	4,04	4,04	-
Contact	32,96	10,88	10,65	0,34	0,37	0,40	4,38	4,44	4,00
Immers	34,52	11,15	11,31	0,39	0,34	0,50	4,55	4,93	3,57

The results obtained by tensile tests and immersion ultrasound are quite close. Contact ultrasound results are in the same order but there is a small difference which is certainly due to our approximation of proportionnality of the engineering constants to the fibre rate. However the results are included in an interval of 5% for E_L, 10% for E_T,ν_{LT}, and 15% for G_{LT}, G_{TT}.

CONCLUSION

In this study, the elastic properties of an unidirectionnal glass fiber polyester composite have been studied using quasi-statique tests and two ultrasonic methods, a destrutive one using many samples in direct contact and a non destructive one using one sample varying the direction of propagation in an immersion test. The elastic constants obtained from all used methods show a good agreement. The quasi-statique method based on tensile tests did not allow to determine all elastic constants of the material assumed orthotropic. The choice of the coupling milieu between transducers and sample is important and much more in composite materials. In a manual contact non-destructive testing, the thickness of the coupling liquid is not constant which involves important variation of the transmitted energy and so quite different results on velocities and mechanical properties. The immersion testing gives much more reliable results. However, contact testing is easy to use compared to the immersion technique which demands much more preparation.

The use of ultrasonic waves for the determination of the mechanical properties of the composite materials allows reliable results to be obtained. The comparison of the elastic constants issued from the ultrasound method with those issued from the tensile tests makes it possible to certify that the ultrasonic waves determination is efficient and accurate, and besides it is easy to make use of. In fact, for a common fibre rate we obtain a difference lower than 10-15% compared with the tensile tests. This method possesses real advantages in comparison with the static one. The strong point of the characterisation in immersion is the ability, thanks to optimization technique, to determine the orthotropic elastic behavior of a material from only one plate, contrary to the characterisation with contact which necessitates four samples. So this method is well adapted to this kind of composite material elabotated in the form of thin plates. Because of the good accuracy of the method it could be succesfuly used to characterise woven composites. Endeed, for such composites It could be interseting to study the effects of the wavy architecture of the material resulting from the difference between the warp an welft directions in such composites. This is the next step of our work.

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