# CHARACTERIZATION AND DAMAGE EVOLUTION OF S.M.C COMPOSITES MATERIALS BY ULTRASONIC METHOD

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## **SUMMARY:**

An experimental investigation coupled to a micromechanical approach are described in this paper in order to study the damage behaviour of a randomly short-fiber SMC composite subjected to a tensile test. The experimental investigation is based on the ultrasonic technique which allow us to determine the nine elastic constant of an orthotropic material. The damage mechanisms of this kind of composite are identified and related to the anisotropic degradation of the material. A micro-macro relation based on Mori and Tanaka's model is developed to evaluate the overall stiffness tensor of the composite and its anisotropic degradation due to fiber/matrix interface damage. Moreover, we propose an inverse method using the ultrasonic measurement which is able to evaluate the fiber/matrix failure properties.

**KEYWORDS**: ultrasound, elastic constants, anisotropic damage, fiber/matrix interface, multi-scale model.

## I - INTRODUCTION

Organic matrix composites reinforced by randomly oriented short glass-fibers are widely used because of their interests like a high strength/density ratio, a high modulus, a capacity of energy absorption, etc. It is thus essential that the characteristics composite material can be measured via a simple and reliable method, prior to the design office stage, for structure calculation. Among these methods, the quasi-static ones, based on strain and stress measurements (tensile, shear tests, etc.) present some major drawbacks such as destructiveness and difficulties in measuring shear constants as well as Young's moduli along directions other than the main axes.

These difficulties can be overcome using non-destructive methods such as ultrasonic evaluation. These methods are based on the propagation of stress waves within the medium to be characterized. The wave propagation velocity depends on the material density, elastic constants and on the direction of propagation. Each constant is solely determined on the basis of the measurement of a known wave velocity. Thus, the full characterization of a composite with a given symmetry requires as many propagation velocity measurements in adequate directions as independent elastic constants (five for a transverse isotropic material, nine for an orthotropic one, etc.). This approach requires several orientated sections for a full evaluation. But, some propagation directions are not always accessible, especially in most very thin composite (few millimeters).

In this study, we put forwards an iterative elastic constant identification approach, which consists in minimizing the square deviation between calculated velocities and the experimental ones measured under variable incidence in immersion conditions. Then, a micromechanical model is presented to estimate the mechanical behaviour and damage evolution of an SMC composite (Sheet Molding Compound), based on Mori et Tanaka's model. In this paper, we show how the ultrasonic measurement can be a efficient tool for the identification of a fiber-matrix interface failure criterion and it's introduction in a multi-scale predictive model.

## II - IMMERSION TECHNIQUE

Markham [1], using an immersion technique demonstrated the capability to recover the elastic moduli for transversally isotropic media via multiple velocity measurements at oblique incidence. This method was improved by Roux et al. [2], Hosten et al. [3], Rokhlin and Wang [4] [5] and [6], who automated the experimental procedure and proposed an optimization process for the mechanical characterization of anisotropic materials. The immersion technique uses refraction for mode conversion at a liquid-solid interface to generate the desired mode at the desired angle in the material. The method consists in placing a sample of the material to characterize, a plate of uniform thickness, in a water bath between two transducers, a transmitter and a receiver, adjusted initially perpendicular to the sample. Wave velocities are determined by measuring the change in time of flight between the transmitter and the receiver of a pulse, with and without the sample. The wanted time is measured accurately using an appropriate signal processing.

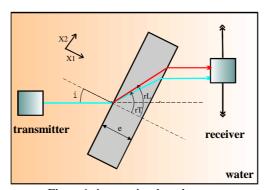


Figure 1: immersion bench test

## **III - RECOVERING OF ELASTIC CONSTANTS**

For a homogenous elastic and anisotropic solid of density  $\rho$ , the resolution of the wave equation considering plane was gives the so-called Christoffel equation :

 $|\Gamma_{ij} - \rho V^2 \delta_{ij}| = 0$ , where  $\Gamma_{ij} = C_{ijkl} n_k n_l$  is the Christoffel tensor and  $C_{ijkl}$  is the second order elastic constant tensor of the solid ( $C_{IJ}$  in contracted notation) [7]. The first purpose of this study is to resolve the inverse problem, in other words, to recover the second order elastic constant from the velocity measurements.

Although the velocity measurements are not very difficult, obtaining the precise elastic constants from the experimental data can be a tedious task, particularly for highly anisotropic materials. In such materials the propagated modes are generally neither longitudinal nor transverse. In general, in anisotropic materials, the velocity measurement in any arbitrary direction can depend on 21 elastic constants, as is the case for the triclinic symmetry. By considering the wave propagation in particular directions, some elastic constants could be directly determined, but this method needs several orientated cuts of the material. Beside its destructive character, as it is necessary to have more than one oriented cut, the full characterization of the majority of composites is impossible by this means, because most of them are very often elaborated in the form of thin plates.

To overcome these difficulties, iterative numerical methods associated to multiple velocity measurements were widely used [8]. These methods of minimization are generally based on Newton's algorithm. It is well known that this algorithm requires a good initialization to converge towards the solution of the problem. Indeed, if the initialization of the process is not close enough to the exact solution, the algorithm sometimes converges towards relative minima which gives elastic constants which do not correspond to the physical reality of the materials[9]. The methodology to recover the elastic constants, consists in minimizing the square deviation between calculated velocities and the experimental ones measured under variable incidence from the immersion device. The minimizing algorithm used is the Levenberg-Marquard one, which combines the Newton method and the gradient one in a single algorithm. As in other iterative method, the minimizing process has to be initialized by introducing some initial elastic constants. Before starting the iterative optimization process, the initial values are estimated by superimposing at best the calculated and experimental velocities curves.

The developed algorithm is valid for any kind of symmetry weaker than the orthotropic one. The symmetry that we consider in this study is the orthotropic one, sufficiently general to describe the mechanical behaviour of most composites. These materials are represented by their matrix of elasticity containing nine second order independent elastic constants [5]. The auscultation of a principal plane allows us to obtain at most four elastic constants. In this study, index 1 refers to the normal to the plate.

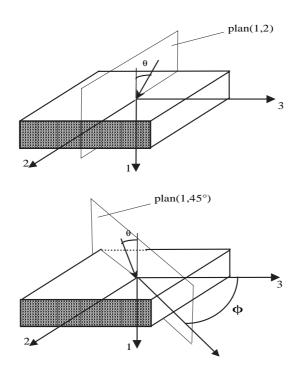


Figure 2: geometric reference

The exploration of two principal planes (12) and (13) allows to recover seven of the nine elastic constants (  $C_{11}$ ,  $C_{22}$ ,  $C_{33}$ ,  $C_{55}$ ,  $C_{66}$ ,  $C_{12}$ ,  $C_{13}$ ).  $C_{11}$  can be measured at normal incidences. For a full identification of the whole matrix of elasticity, the auscultation of the third plane of symmetry is needed. Because of the small thickness of these samples due to their elaboration process, we can not investigate this plane. The propagation in a non-principal plane is then required [3]. The remaining elastic constants are  $C_{23}$  and  $C_{44}$ . the non-principal plane in which these constants have the greatest influence on ultrasonic propagation is the one defined by axis 1 and the axis which is contained in plane (23) and makes an angle of  $45^{\circ}$  with axis 2. Thus, in this plane the velocities of the three excited modes can be measured for different incidences. The optimization described above is applied to these data for a precise identification of these remaining constants. So, the complete evaluation of this material is done.

Stiffness tensor of SMC is:

$$\mathbf{C} = \begin{bmatrix} 17.00 & 7.06 & 6.14 & 0 & 0 & 0 \\ 7.06 & 22.48 & 10.00 & 0 & 0 & 0 \\ 6.14 & 10.00 & 22.95 & 0 & 0 & 0 \\ 0 & 0 & 0 & 6.00 & 0 & 0 \\ 0 & 0 & 0 & 0 & 5.13 & 0 \\ 0 & 0 & 0 & 0 & 0 & 4.51 \end{bmatrix} . (\mathbf{GPa})$$

# IV - IDENTIFICATION OF MACROSCOPIC DAMAGE EVOLUTION

So as to quantify the effect of damage on elastic properties of the composite, we have developed a tensile machine adapted to the ultrasonic bench test.

This has allowed us to put in obviousness the anisotropic evolution of the damage through the diminution of wave velocity depending on the applied load[10].

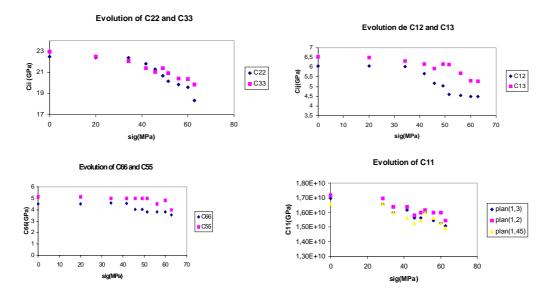


Figure 3: Evolution of elastic constants

The evolution of the different elastic constants according to the applied stress (Figure 3) shows the anisotropic character of the damage. The  $C_{IJ}$  are not affected by the same manner. The decrease of elasticity constants in the direction of loading is the most important. This proves that fissures are mainly opened perpendicularly to this direction. The loading induces a supplementary anisotropy as compared to the virgin material.

# V - MICROMECHANICAL MODEL

## V-1 Mori and Tanaka's model for the undamaged material

To estimate of the mechanical behaviour of composite materials, we must take into account specific characteristics of the material: heterogeneity and anisotropy. Homogeneisation methods allow to define the macroscopic global behaviour of a heterogeneous material from mechanical and geometrical characteristic of the different phases of the material. The chosen model comes from works of T.Mori and K.Tanaka [11][12] where the authors calculate the average strain in the matrix of a material containing inclusions presenting eigenstrain, the extension to a material containing the heterogeneity being obtained by applying the theory of the equivalent inclusion of Eshelby [13]. The reinforcement is drowned in an infinite middle having properties of the matrix of the composite. We obtain thus an expression of the elasticity tensor of the composite:

$$C^{comp} = C^m \Big[ I + f < Q > (I + f < (S - I)Q >)^{-1} \Big]^{-1}$$

where <Q> is the average of the "pseudo - tensors" of localisation,  $Q^{ri}$ , define for each family of reinforcements by :

$$Q^{ri} = ((C^m - C^{ri})(S^{ri} - I) - C^{ri})^{-1}(C^{ri} - C^m)$$

where  $S^{ri}$  is the tensor of Eshelby which is function of the mechanical characteristics of the matrix and the geometry of reinforcements.  $C^{ri}$  is the rigidity tensor of each family of reinforcement i and  $C^{m}$  that of the matrix. All tensors are expressed here in the macroscopic scale of the plate. In the case of the SMC, reinforcements are assimilated to ellipsoïds of a infinite aspect ratio and randomly distributed in the plan of the plate. The matrix present an elastic behaviour.

#### V - 2 local strain calculation

The use of the Mori and Tanaka's model allows us to access to the local stress state in reinforcements under a given macroscopic loading.

In the case of SMC, the main source of damage is fiber - matrix interface failure is necessary to calculate the interfacial stresses.

The average stress tensor inside a fiber oriented in the direction  $\theta$  is given by :

$$\sigma^{i} = C^{m}(I + (S^{i} - I)Q^{i})(I + f < (S - I)Q >)C^{m-1}\Sigma$$

The stress tensor in the interface can be calculated by using the condition of normal stress continuity across the interface. Tangential and normal stresses in a point of the interface of normal n are obtained by simple projection of the stress tensor in the fiber.

$$\sigma = \sigma^{i} \overline{n.n} = \overline{T.n}$$
 and  $\tau = \sqrt{\|\overline{T}\|^2 + \sigma^2}$ 

## V-3 Statistical fracture criterion

## *V-3-1 Fiber – matrix interface failure criterion*

## a - Determinist Criterion

The interfacial failure being the fruit of the combination of a local normal stress and a local shear stress, one introduces this coupling in all point of the interface under the form of an elliptic criterion:

$$(\sigma/\sigma_0)^2 + (\tau/\tau_0)^2 = 1$$

where  $\sigma$  and  $\tau$  are respectively normal and tangential stress to the considered point of the interface.

the handwriting of a such criterion necessitates the identification of parameters  $\sigma_0$  and  $\tau_0$ .

#### b - Statistical Criterion

several studies [14][15] have shown that SMC microstructure presents two types of dispersion. One linked to the presence of rich matrix zone in and others rich in fibers. So we have a distribution of fiber volume fraction. One can deduce that, at reinforcements scale, the distribution stress in fibers, on interfaces and in the matrix will be very sensitive to the distribution of fiber volume fraction. The kinetics of damage will be strongly modified.

Furthermore, we can note the presence more or less marked of porosity. They are distributed in all the volume of the composite. The presence of porosities more or less voluminous and more or less neared of fibers ( some are even localized on the interface) causes an important variations of local stresses.

Classical homogeneisation model assume a homogeneous repartition of the different components. The microstructure is described in average and local variation of stress aren't considered. Thus, we introduce a statistical form of the criterion:

$$Pr(R) = 1 - \exp(-((\sigma/\sigma_0)^2 + (\tau/\tau_0)^2)^m)$$

# c – Identification of the criterion by using the ultrasonic measurement

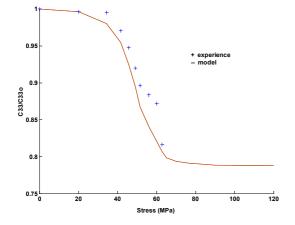
Multiple local damage mechanisms have been observed in the case of the SMC: matrix fissuration (linked or not to the presence of porosity), the interfaces debonding of very large particles of chalk, but especially the of fiber/matrix interfaces debonding [14]. It has been also shown that the first interface failure occurs for the fibers oriented at  $90^{\circ}$ . So a first evaluation of  $\sigma$ 0 can be obtained by calculated (using the model) the maximum interfacial normal stress for the  $90^{\circ}$  oriented fibers. For this calculation, we apply a tensile macroscopic stress corresponding to the first non-linearity (40 MPa).

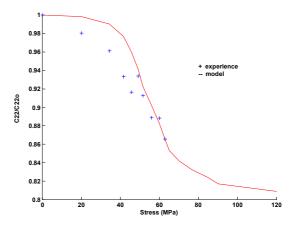
Then the other two parameters ( $\tau$ 0 and m) are identified by an optimization process which minimize the difference between the evolution of experimental  $C_{IJ}$  and those given by the model [16].

Finally, after several iterations, we obtain:

$$\sigma_0 = 62 \text{ MPa}, \quad \tau_0 = 49 \text{ MPa}, \quad m=1.4$$

the result shows a good correlation between the evolution of elastic constant given by model and experience (Figure 3). One note that the fibers oriented around 90° (Figure 4) are firstly damaged since the less misoriented fiber interfaces breaks later. This scheme of damage has been observed by in-situ tensile tests [14].





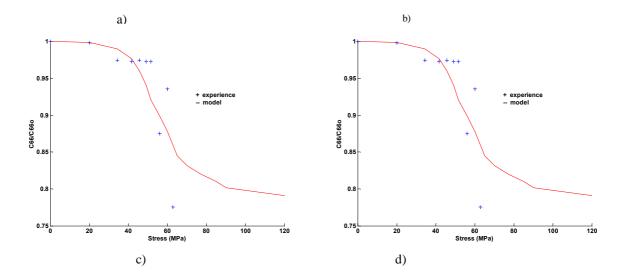


Figure 3: relative evolution of experimental and simulated stiffness: a) C33, b) C22, c) C13, d) C66

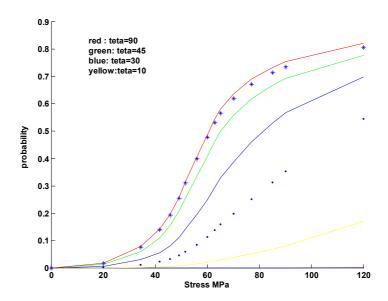


Figure 4: Probability of failure of each family vs applied stress

## VI - CONCLUSION

The full mechanical and structural characterization of this material was done non destructively using the ultrasonic immersion device associated to an optimization process. The performance and the reliability of this method were demonstrated for these kinds of composites and for other anisotropic materials like ceramic and metal matrix ones.

The anisotropic damage is observed and quantified in SMC R42 composites under tensile loading. The macroscopic properties of this composite was illustrated by continuous stiffness degradation.

A new method to evaluate the statistical local fiber matrix interface properties is presented. It corresponds to an inverse method using as entry data the anisotropic loss of stiffness of the composite under tensile loading. The used model is based on a micromechanical approach which integrates the interfacial failure at the local scale. A quiet good correlation between experimental and simulated results is demonstrated.

The next step of our work will consist in the validation of the identified micromechenical model by developing a biaxial tensile machine coupled with the ultrasonic measurement.

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