DAMAGE CHARACTERISATION OF CERAMIC MATRIX COMPOSITES

E. Grippon[1,2*], S. Baste[2], E. Martin[1] , C. Aristégui[2] , G. Couégnat[1]

[1]LCTS, Univ. Bordeaux, 3 allée de la Boétie, 33600 Pessac, FRANCE
[2]I2M, Acoustique, Univ. Bordeaux, 351 cours de la libération, 33405 Talence Cedex, FRANCE
**edith.grippon@u-bordeaux1.fr*

Keywords: ultrasonic characterisation, ceramic matrix composites, multi-scale modeling.

Abstract

The CMC's damage is due to the growth of micro-crack. It was shown that this one has the effect of the non-linearity in the stress-strain relationship and the degradation of the stiffness tensor. Thus, the measurement of the stiffness tensor, during a tensile test, provides access to the damage evolution.
The use of an ultrasonic device gives access to all the components of the stiffness tensor of the material. Tensile tests under different material axes were planned to separate all the cracks orientation. Characterisation of CMC samples, with different symmetries material (stiffness tensor with 6 to 13 non-zero and independent components) can be done using a genetic algorithm. The tensor measurement will be compared with numerical predictions provided by multi-scale modeling.

1 Introduction

Due to their good thermo-mechanical properties, ceramic matrix composites (CMC) are attractive materials for high temperature applications. Snecma Propulsion Solide (SAFRAN group) has developed a woven SiC/SiC with a self-healing matrix, for a turbine part of the next generation of engine of civil plane.
The heterogeneities of this material result from the presence of four phases: fibre, matrix, interphase and porosity. Each part plays a special role in the anisotropic behaviour of this material. The fibers acting as reinforcement are immersed in a matrix whose mechanical strength is lower. The first signs of damage appear in the matrix, where the propagation of microcracks is then stopped by the fibres (Figure 1). Two modes of fracture have been identified [1]: matrix cracks oriented perpendicularly to the load direction and interface of debonding. Additional information may be obtained during off-axis tests.
Understanding and monitoring the damage of strongly anisotropic CMC is essential to the formulation of constitutive equations [2]. In order to consider the tridimensional non-linear mechanical behaviour of these materials, the stiffness tensor is chosen as the damage variable [3]. The effect of micro-cracks growth is the degradation of the stiffnesses (Figure 2). By using an ultrasonic device coupled to a tensile machine [4], all the components of the stiffness tensor are identified during a load test. These measurements can thus be compared with numerical predictions provided by a constitutive law in order to identify the damage kinetics [5].

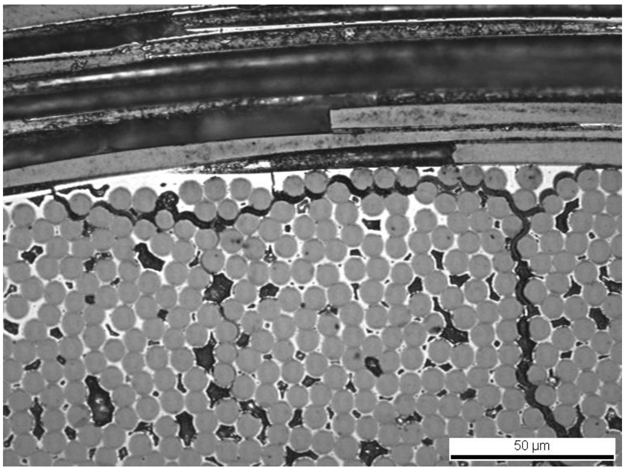

Figure 1. Deviation of a transverse crack at the interface between a longitudinal yarn and a transverse yarn [5].

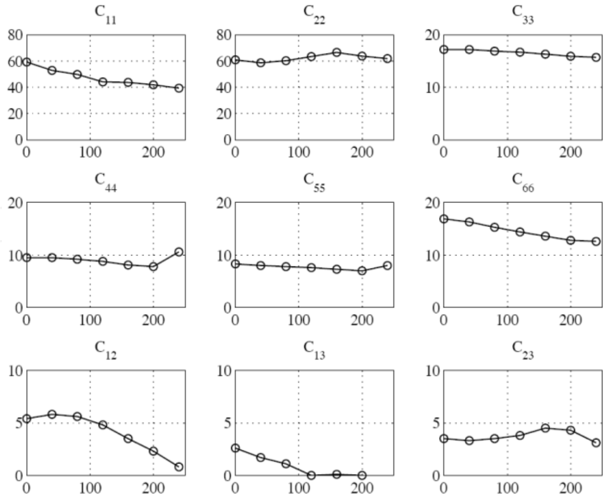

Figure 2. Change of the stiffness tensor components (GPa) depending of the applied stress (MPa) for a monotonic tensile test at 0° [5].

The stiffness tensor takes particular forms depending on the symmetry of the studied material. The considered CMC is initially an orthotropic material with 9 non-zero components. During the off-axis solicitation, due to the growth of micro-cracks perpendicularly to the loading, the symmetry of the sample becomes monoclinic and the stiffness tensor has 13 non-zero components [6].

The aim of this paper is to propose a new procedure for identifying the stiffness tensor of a CMC using ultrasonic measurements. In a first part, the ultrasonic measurement method used is described. Then, in order to determine these 13 components and because of the specific acoustic properties of the CMC investigated, a new method of identification is presented.

2 Ultrasonic determination of stiffness tensor of the studied CMC

The determination of stiffness components from measurement of ultrasonic wave speed is known for several years. Many authors have exploited the properties of bulk waves propagating in anisotropic solids in order to characterise them. From the early 80s, the Laboratoire de Mécanique Physique (LMP) (renamed Institut de Mécanique et Ingénierie (I2M)), developed a characterisation technique, called ultrasonic spectro-interferometry [7]. The evaluation of material stiffnesses by ultrasonic method consists of collecting speeds of waves propagating in different planes of this solid. The acquisitions are made in four planes (Figure 6) corresponding to the plane (**1**,**2**), (**1**,**3**), (**1**,Ψ) and (**1**,-Ψ), where R = (**1**, **2**, **3**) is the coordinate system and Ψ is the azimuthal angle equal to 45°. Wave speed measurements are performed by comparing ultrasonic pulses propagating in water with pulses which are refracted by a plate-like sample immersed in water (Figure 3).

Under small strains, the Christoffel equation, relating the mechanical properties of homogeneous media to the propagation properties of elastic bulk waves, is written as:

$$\left|\mathbf{\Gamma} - \rho V^2 \mathbf{I}\right| = 0, \qquad \textbf{(1)}$$

with $\Gamma_{ij} = C_{ijkl} n_k n_l \left(i, j, k, l = 1,2,3\right)$ and where ρ is the mass density, $\mathbf{n}$ is the unit vector along the wave propagation direction, V is the wave speed (phase velocity) of ultrasonic waves, C_{ijkl} are the stiffness components and $\mathbf{I}$ is the identity tensor. Generally, three modes can propagate along the direction **n** with different speeds and polarisations: one quasi-longitudinal mode (QL) and two quasi-transverse modes (QT1) and (QT2). If the incident plane coincides with a plane of symmetry, only two waves are generated (one quasi-longitudinal and one quasi-transverse).

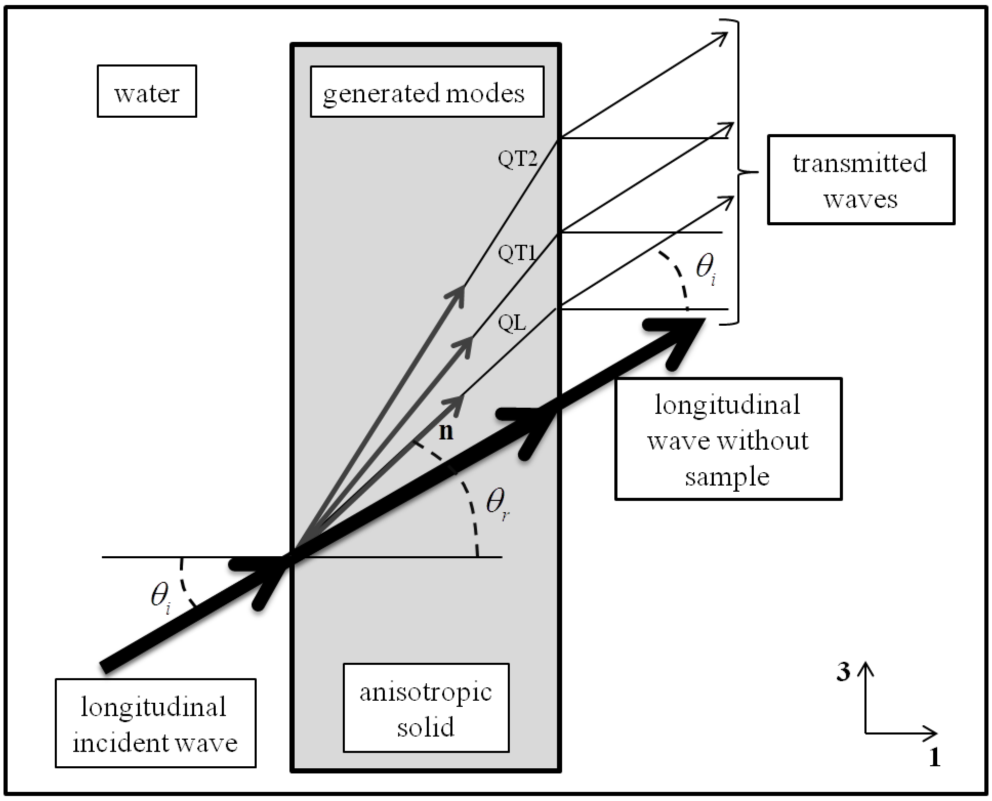

Figure 3. generation of three modes at solid/liquid interface.

By inverting equation (1), the stiffness components can be determined from a suitable set of experimental speeds collected for various directions. Usually, the stiffness tensor is determined by a Newton-Raphson algorithm [8]. Due to the need to characterise monoclinic materials and the specific properties of the considered samples of SiC/SiC, such as the low values of the angles of transition between quasi-longitudinal and quasi-transverse modes, a more robust optimisation method was developed.

The material studied is a three-directional SiC/SiC composite with a self-healing matrix. The yarn cross section has an elliptical geometry about 1 mm in diameter along the major axes. These yarns are woven to obtain, after densification, a composite of a thickness around 3.7 mm. The porosity varies from 10 % to 14 % and the phase velocity of the longitudinal bulk wave is about 3.4 $km.s^{-1}$ at normal incidence.

In general, four phenomena make the acoustic measurements tricky (Figure 4): *i.* the echoes overlapping, *ii.* The mixed mode occurring when the longitudinal and the transverse modes are difficult to be identified, *iii.* the dispersion linked to the properties and heterogeneities of the material and *iv.* the presence of noise. The choice of wavelength of the pulse propagating in the material depends on the combination of these characteristics. For these CMC, the chosen central frequencies of the transducers are 1 and 2.25 MHz. The lowest frequency limits the dispersion phenomenon while the highest frequency allows a better separation of the modes.

In Figure 5, two signals processing methods are represented: one using the cross-correlation processing, the other the Hilbert transform [9].

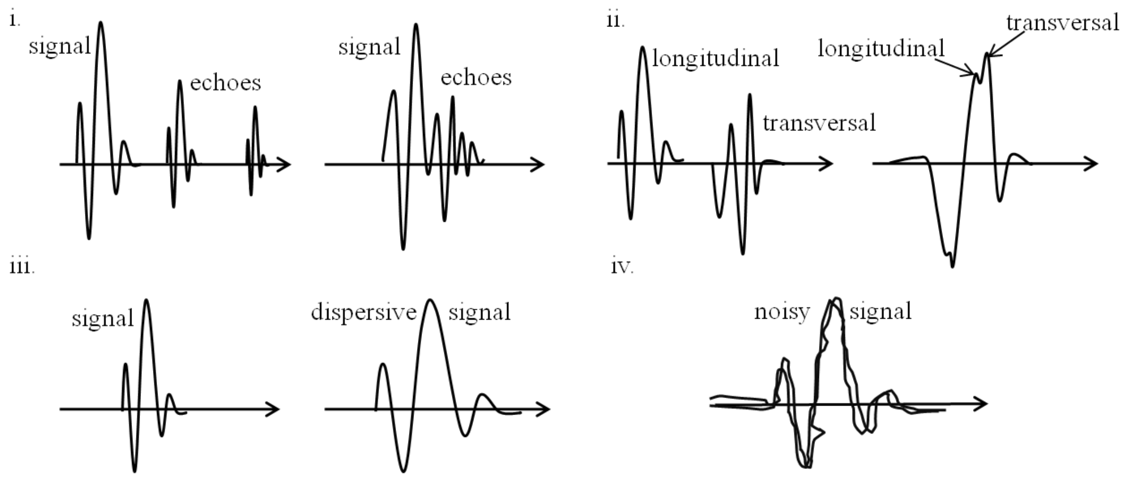

Figure 4. Representation of perturbation during acoustic propagation: *i.* echoes overlapping, *ii.* mixed mode, *iii.* dispersion and *iv.* noise.

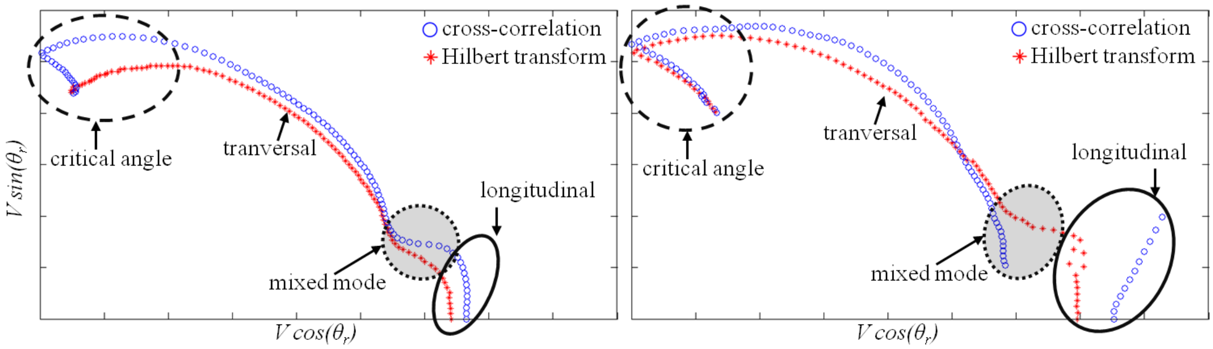

Figure 5. Example of phase velocity measurements: on left at 1MHz and on right at 2.25MHz.

The cross-correlation processing gives poor results when the signals are dispersive [9]. We can observe a larger gap for the longitudinal speeds between the two methods when the frequency of the reference signal is 2.25 MHz. It results from the signal dispersion. However, this working frequency facilitates the separation of the two modes. Thus, considering the low number of longitudinal points for the studied CMC, when it is possible, we choose to work at 2.25 MHz to promote the mode separation in spite of the dispersion.

From Figure 5, it can also be observed the encircled hook occurring for large incident angles. This corresponds to the critical angle defined by Snell-Descartes law. After all, for the studied CMC, 5 to 10 velocities measurement correspond to the longitudinal mode and around 20 to 30 points correspond to the velocities of the transverse mode.

The number of measured velocities is not sufficient to permit an easy convergence of the Newton-Raphson algorithm generally used to invert equation (1). In addition, the characterisation of monoclinic material requires a new formulation of the minimisation problem.

3 The genetic algorithm

A genetic algorithm [10], is used and adapted to our problem. This identification method is enough robust to identify the overall stiffness tensor by operating in all acquisition planes.

The algorithm seeks the components C_{ij} (in reduced notation) which minimise the error between the model responses $V_t(C_{ij},\mathbf{n})$ and the experimental values $V_e(\mathbf{n}(\theta_r))$. The model responses are calculated using the Christoffel equation (1). The sought components are the solutions of the following minimisation problem:

$$\left(C_{ij}\right)=\min F_e\left(C_{ij}\right) \quad \text{with} \quad u<C_{ij}<v$$

$$\text{with} \tag{2}$$

$$F_e\left(C_{ij}\right)=\sum_{\mathrm{m}}\sum_{r=1}^{N}\left|V_t^{(\mathbf{1},m)}(C_{ij},\mathbf{n}_r)-V_e^{(\mathbf{1},m)}(\mathbf{n}_r(\theta_r))\right|^2,$$

where u and v are the limits imposed to the components of the stiffness tensor. N is the number of points measured in each plane, $\mathbf{n}_r$ is the unit vector in the wave propagation direction and θ_r is the refracted angle. $m=\mathbf{1},\mathbf{2},45°$ *or* $-45°$ represents the different acquisition planes. The coordinate system R for ultrasonic measurement is chosen in such a way that the axis **1** corresponds to the normal to the sample.

3.1 Numerical simulation of the convergence of the genetic algorithm for two particular material symmetries

The studied CMC could be considered as a quasi-quadratic material, which explains that only one transverse mode is measurable in planes $(\mathbf{1},45°)$ and $(\mathbf{1},-45°)$. For quadratic material, the minimisation problem using Newton-Raphson method does not allow to determine independently the constants C_{23} and C_{44} (only the combination $C^*=C_{23}+2C_{44}$ can be measured). The formulation of the minimisation problem by the genetic method takes off this indeterminacy.

Data type	C_{11}	C_{22}	C_{33}	C_{44}	C_{55}	C_{66}	C_{12}	C_{13}	C_{23}
Original data	12.9	100	100	35.5	9.7	9.7	3.2	3.2	16.1
Results with a large number of longitudinal velocities	12.9 (0)	99.2 (0.9)	99.7 (1.2)	34.7 (3.2)	9.7 (0)	9.7 (0)	3.2 (0)	3.1 (0.1)	18 (21.9)
Results corresponding to experimental configuration	12.6 (0)	105 (19.5)	102.9 (22.8)	34.9 (3)	9.7 (0.1)	9.5 (0.1)	3.4 (1.4)	3.4 (1.6)	14.5 (22.8)

Table 1. Results ($*10^{-2}$ normed) of stiffness components using ultrasonic velocity data simulated for a material with quadratic symmetry. The simulations have been made by adding noise around 1% of the normal velocity value. The initial constraint is equal to more or less 30 % of the original data for diagonal components. For the initial constraints of the off-diagonal stiffnesses, the stiffness tensor is sought to be positive definite.

Table 1 shows simulation results for a quadratic material. Each optimised value is associated with a confidence interval in parentheses. The results obtained with a large number of longitudinal velocities are close to the original data. Removing longitudinal velocities, to approximate the experimental configuration, degrades the results and increases the associated confidence interval, while the errors remain low.

One of the objectives set during the revisiting of the minimisation problem is to be able to characterise monoclinic material. Table 2 shows simulation results for a monoclinic material. The first line shows the original data used for the simulation. The next one presents the results for twenty longitudinal points. We can see that the results are almost similar to the original data for the diagonal components, C_{12} and C_{13}. On all other off-diagonal components, the C_{23} is the value with the most important error. Indeed the sensitivity of the functional (2) to this parameter is particularly low. Ultrasonic measurement in contact [11] will help to the experimental determination of this parameter. The results corresponding to the experimental configuration remains correct. The largest difference between the result and the original data is also observed for the C_{23}.

Data type	C_{11}	C_{22}	C_{33}	C_{44}	C_{55}	C_{66}	C_{12}	C_{13}	C_{14}	C_{23}	C_{24}	C_{34}	C_{56}
Original data	12.9	93.5	100	41.9	10.3	9.7	3.2	3.9	0.6	21.9	-0.6	6.5	-0.8
Results with a large number of longitudinal velocities	12.9 (0.1)	95.9 (3.5)	100.3 (18.8)	41.7 (11.8)	10.3 (0.9)	9.5 (0.2)	3.3 (0.5)	3.0 (0.8)	0.2 (4.6)	5.9 (29.5)	1.6 (1.4)	6.5 (12.9)	-0.1 (4.2)
Results corresponding to experimental configuration	12.9 (0.1)	93 (26.1)	102.3 (26.2)	38.5 (23.7)	10.4 (0.8)	10.1 (1.5)	2.4 (2.6)	3.5 (2.7)	-0.6 (9.3)	2.1 (67.7)	1.7 (20.8)	0.2 (17.8)	-0 (3.6)

Table 2. Results ($*10^{-2}$ normed) of stiffness components using ultrasonic velocity data simulated for a material with monoclinic symmetry. The simulations have been made by adding noise around 1% of the normal velocity value. The initial constraint is equal to more or less 30 % of the original data for diagonal components. For the initial constraints of the off-diagonal stiffnesses, the stiffness tensor is sought to be positive definite.

3.2 Numerical comparison between the genetic algorithm and a classical Newton-Raphson method

The validation of the determination of the stiffness tensor by genetic algorithm, is made by comparing, for the same simulation, the results obtained by this methode with those obtained using the Newton-Raphson algorithm. The comparison is possible only for orthotropic symmetry.

Data type	C_{11}	C_{22}	C_{33}	C_{44}	C_{55}	C_{66}	C_{12}	C_{13}	C_{23}
Original data	24.2	92.4	100	34.4	10.2	8.9	5.1	6.4	19.1
Newton-Raphson algorithm	23.9	54.3	49.3	25.4	9.7	8.6	0.9	0.3	12.4
Genetic algorithm	24.1	98.1	99.5	35.7	9.9	8.9	5.8	6.2	26.1

Table 3. Results ($*10^{-2}$ normed) of stiffness components using ultrasonic velocity data simulated for a material with orthotropic symmetry. The simulations have been made by adding noise around 1% of the normal velocity value. The number of longitudinal velocities corresponds to experimental configuration (around 10). For the Newton-Raphson algorithm, the initial values are the original data. For the genetic algorithm, the initial constraint is equal to more or less 30 % of the original data for diagonal components. For the initial constraints of the off-diagonal stiffnesses, the stiffness tensor is sought to be positive definite.

Numerous advantages of the genetic algorithm were highlighed in this comparaison (Table 3). Indeed, when the longitudinal points (experimental or simulate) are less than 10 (corresponding to experimental configuration), the Newton-Raphson method convergence is espacially difficult and its sensitivity to initialisation values greatly increases. However, in this configuration, the genetic algorthm is much more stable and the initialisation problem is replaced by the easier definition of a constraint.

The use of the genetic algorithm for determining of the stiffness tensor satifies our expectations. It allows to characterise the CMC for all studied symmetry: quadratic, orthotropic and monoclinic. Furthermore, this algorithm gives accurate results despite of the low number of velocities data of longitudinal mode, which reveals a typical acoustic property of the studied CMC.

3.3 Experimental test

The determination of the components of the stiffness tensor is now tested with experimental data. Figure 6 shows all the measured speeds of wave propagation in the four acquisition planes. As in Figure 5, we can pick out the longitudinal, the transversal, the mixed mode and the critical angle.

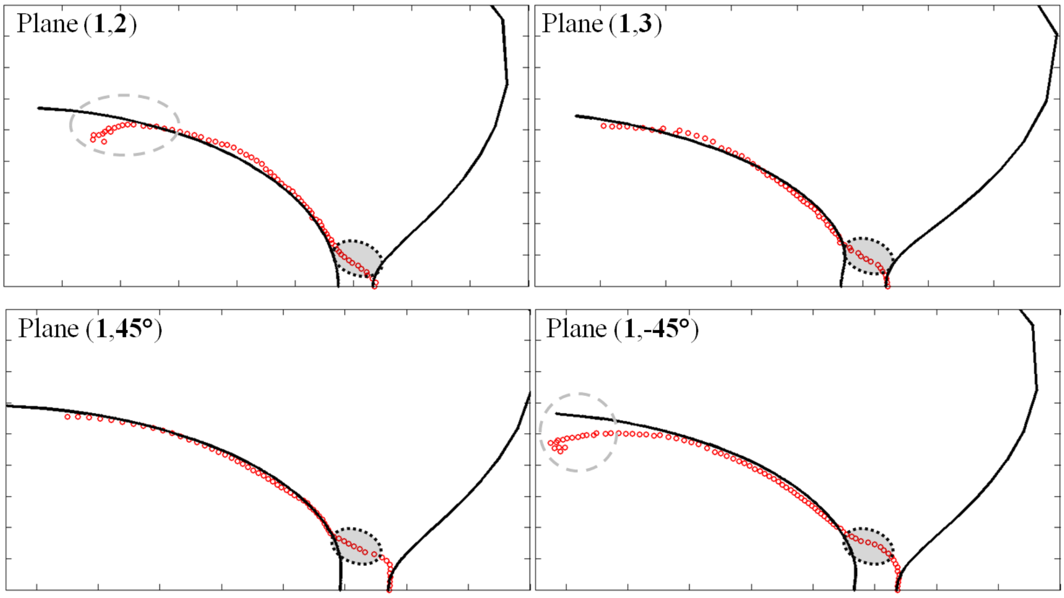

Figure 6. Wave speed measurement in the planes (**1**,**2**), (**1**,**3**), (**1**,45°) and (**1**,-45°). The points represent the measuring speeds. The lines represent the calculated speeds obtained from the optimised stiffness tensor. The velocities are projected along the direction **n** of wave propagation in material. The circled areas represent the mixed mode and the critical angles.

Table 4 presents the results obtained with experimental data keeping the mixed mode and the results obtained from selected data, *i.e.* by removing the points measured in presence of mixed mode. The correlation between these results shows the low sensibility of the genetic algorithm in the presence of mixed mode. This property is particularly interesting for the characterisation of the CMC studied.

Data type	C_{11}	C_{22}	C_{33}	C_{44}	C_{55}	C_{66}	C_{12}	C_{13}	C_{23}
Experimental results with all data	13.5	94	97.9	32.8	11.1	11.2	1.6	1.6	24.6
Experimental results with select data	13.5	91.9	100	30.0	10.8	11	3	1	18.9

Table 4. Results (*10^{-2} normed) of stiffness constant using experimental ultrasonic velocity data for an orthotropic material at 2.25 MHz.

4 Conclusion

The non linear mechanical behaviour of CMC involves the initialisation and the growth of microcracks. At a macroscopic scale, mechanics of damage can be associated to change of the

stiffness tensor components. By using an ultrasonic device couple to a tensile machine, the method of spectro-interferometry allows characterisation of materials during its damaging.
Combining this method with the identification of this stiffness tensor by a genetic algorithm allows to characterise the CMC for different symmetries: quadratic, orthotropic and monoclinic. Considering the acoustic properties of the CMC studied, this algorithm has several qualities: robustness despite the low number of measuring points, low sensibility to mixed mode and to initial values.

Acknowledgments
The authors are indebted to the Region Aquitaine and the CNRS for its financial support. S.P.S. (SAFRAN group) is also gratefully acknowledged.

References

[1] El Bouazzaoui R., Baste S., *Anisotropic damage of Carbon-SiC composite under off-axis loading* in Proceeding of *Ninth International Conference on Composite Materials*, Madrid, Spain, (1993).
[2] Chaboche J.L., *Le concept de contrainte effective appliqué à l'élasticité et à la viscoplasticité en présence d'un endommagement anisotrope* in "Comportement mécanique des solides anisotrope", edited by EuroMECH 115, Villard de lens, **1**, pp. 738-760, (1979).
[3] Baste S., Audoin B., On internal variables in anisotropic damage. *Eur. J. Mec.*, Ser. A, **10**, 587-606 (1991).
[4] Audoin B., Baste S., Ultrasonic evaluation of stiffness tensor changes and associated anisotropic damage in a Ceramic Matrice Composite, *J. Appl. Mech*, **61**, pp. 309-316, (1994).
[5] Couegnat G., Approche multiéchelle du comportement mécanique de matériaux composites à renfort tissé, Thèse de doctorat, Université Bordeaux 1, 2008.
[6] Baste S., Aristégui C., Induced anisotropy and crack systems orientations of a ceramic matrix composite under off-principal axis loading, *Mech. Mater.*, **29**, pp. 19-41, (1998).
[7] Hosten B., Barrot A., Roux J., Interférométrie numérique ultrasonore pour la détermination de la matrice de raideur des matériaux composites. *Acustica*, **53**, pp. 212-217, (1983).
[8] Castagnède B., Jenkins J.T., Sashse W., Baste S., Optimal determination of elastic constants of composite materials from ultrasonic wave speed measurements., *J. Appl. Phys.*, **67 (6)**, pp. 2753-2761, (1990).
[9] Audoin B., Roux J., An innovative application of Hilbert transform to time delay estimation of overlapped utrasonic echoes, *Ultrasonics*, **34**, 25-33, (1996).
[10] Couegnat G., Identification numérique et expérimentale d'un modèle de comportement appliqué à un composite à matrice céramique, Rapport de Travail de Fin d'étude, Ecole des Mines d'Albi-Carmaux, 2004.
[11] Aristégui C., Baste S., Optimal recovery of the elasticity tensor of general anisotropic materials from ultrasonic velocity data, *J. Acoust. Soc. Am.*, **101**, pp. 813-833, (1997).