IDENTIFICATION OF THE MECHANICAL PROPERTIES OF THE THICK INTERPHASE IN A COMPOSITE MATERIAL BY MEASUREMENTS OF KINEMATICS FIELDS.

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Abstract

The aim of the study is the in situ identification of the mechanical properties of the fibre -matrix interphase in a composite. Chemical reactions occur between the coating of fibre and the matrix during the elaboration of the composite. The mechanical properties of the interphase influence the properties of the composite. The knowledge of these mechanical properties is thus very significant. We propose a methodology to identify these properties in situ based on measurements of kinematics fields by digital images correlation. The virtual fields method based on the virtual works principle has been used for the local identification the elastic properties of a thick interphase.

1.Introduction

The interphase of a composite material with a polymeric matrix reinforced by fibres is a zone of transfer of three-dimensional stresses whose local phenomena have consequences on the macroscopic behaviour of material. Its intrinsic properties result from the physicochemical interactions between fibres and the matrix. The interphase does not exist as a material itself but is created within the composite during its manufacture. It is thus necessary to be able to in-situ determine its properties.

The purpose of this study [1] is to develop a characterization of the mechanical properties of an interphase of a micro composite. The strain field obtained by numerical derivation of the experimental displacements field is treated by an inverse approach, such as the method of the virtual fields, to allow the identification of the parameters of the behaviour law of the interphase.

2. Determination of the strain fields in the interphase.

To set up a method of displacements measurement by digital images correlation, it was decided to realize a specific specimen. The interphase is mainly regarded as a reaction between the sizing treatment on the glass fibres and the resin of the matrix. This component which is usually formed during the processing of the composite will have, here, being artificially created to obtain necessary dimensions.

The geometry of the specimen is fixed to allow sufficient measures into the interphase (Fig1). We wished to have ten points of measurements to correctly estimate the strain gradient in this zone.

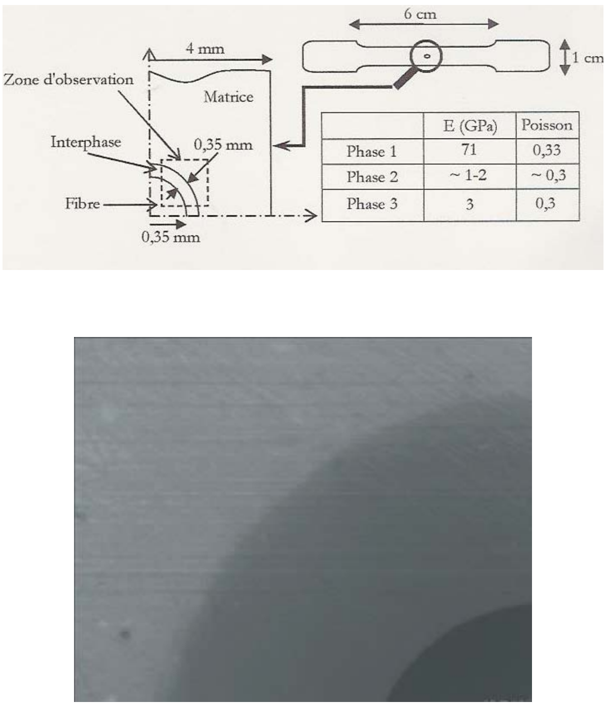


Fig 1: Specimen geometry

This specimen is geometrically representative of a composite material with a long fibre, drowned in a matrix. The phase corresponding to fibre is made of aluminium and has a diameter of 700 µm and that related to the matrix is made of epoxy resin (DGEBA-DDS). The interphase is carried out by a mixture of epoxy resin, 70% identical to the matrix and 30% of industrial sizing without lubricant. The thickness of the interphase is of 350 µm.

A tensile test is carried out on the specimen with interphase. The fibre is placed transversely compared to the applied load.

The system of observation consists of a microscope with long frontal distance associated to a movie camera CCD 12 bits. The loading is stopped at different levels in order to take digital pictures. It is vertically moved thanks to the micrometric table at each stage in order to take the images necessary to the treatment by correlation. The displacements field is obtained with the Correli software developed by F Hild [2]. The aluminium fibre disturbs the field of displacement. The observation during this type of test allows seeing the evolution of the displacements fields during the test, (Fig.2).

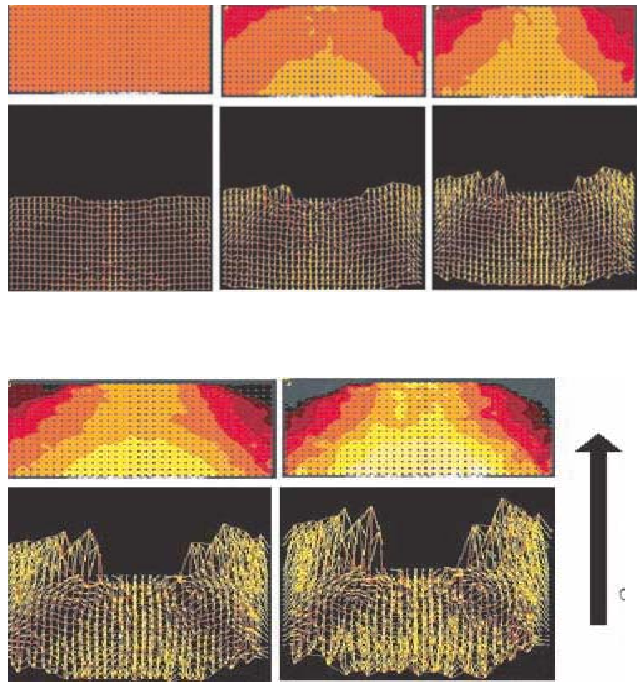
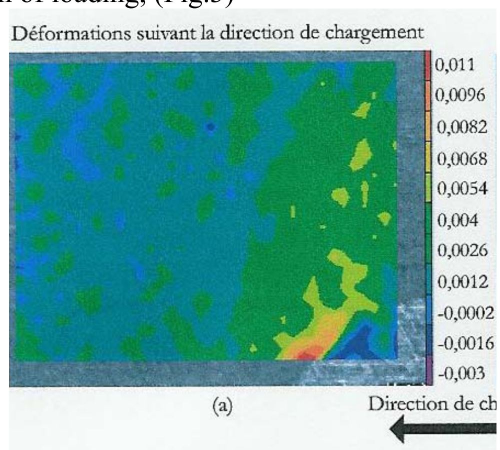


Fig. 2 Evolution of displacements according to the direction of loading during the experiment.

From the obtained displacements fields, a numerical algorithm of derivation is applied, [3], in order to obtain the strain fields for the various directions. Those are illustrated according to the direction of loading, (Fig.3)



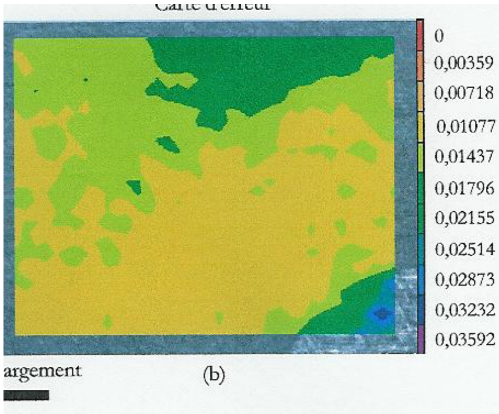
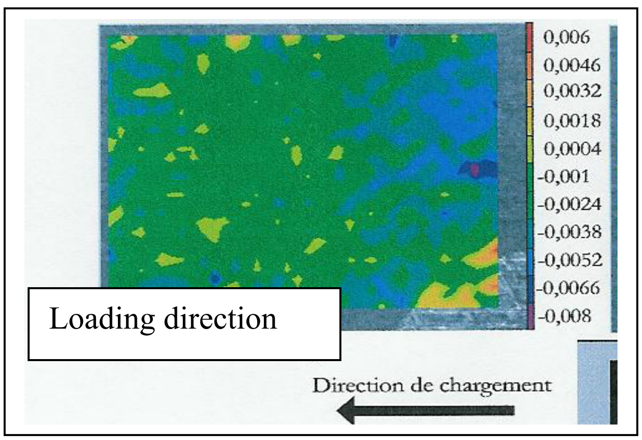


Fig. 3: Map of the strains:
(a) longitudinal strain,
(b) map of error



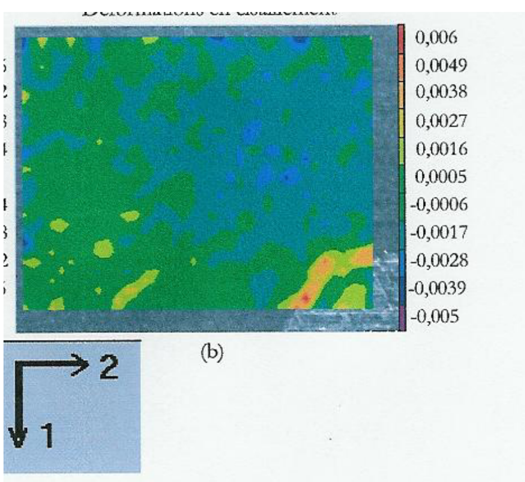


Figure 4: Map of the strain :
(a) perpendicular to the loading
(b) shear strain

The maps of the three strains show concentrations of deformations around heterogeneity. More particularly, the strain fields according to the direction of loading reveals a concentration of deformation at the interface between fibre and the interphase with a gradient of strain within the interphase.

The map of error, Fig.3. (b), enables us to validate the use of measurements of the interphase and the matrix for which the levels of errors are low and homogeneous. The performances of the measurement by digital images correlation are given in table 1 below for a zone of interest (ZOI) and a shift of 16 pixels.

	(px)	(μm)	$(-)$
Displ.spat.resolution	16	11,5	–
Displ. resolution	$2,8 \times 10^{-4}$	2×10^{-4}	–
Displ. uncertainty	8×10^{-3}	$5,7 \times 10^{-3}$	–
Strain spat. resolution	50,6	36,3	–
Strain resolution	–	–	$5,6 \times 10^{-6}$
Strain uncertainty	–	–	$1,8 \times 10^{-4}$

Table 1: performance of the Digital Image Correlation

One can thus estimate that the taken measurements are reliable.

3. Identification of the mechanical properties of the interphase.

The mesurement of fields are effective experimental tools to feed the methods of identification. A specific mathematical procedure is necessary to correctly treat the significant number of data available. The virtual fields method (VFM) [4] is one of these procedures which allows the identification of the parameters of a preset law of behaviour, directly starting from the heterogeneous fields of deformations measured on the surface of the specimen.

The application of the virtual fields method is detailed for a specific geometry corresponding to the study of a thick interphase, illustrated Fig.5. Each geometry requires a particular strategy for a good determination of the parameters which control the behaviour law of the material. Three elements guide the choice of this strategy.

1. The solid is composed of several materials.
2. The mechanical properties of 2 of the 3 materials are known.
3. The analysed zone of the specimen has a cylindrical geometry.

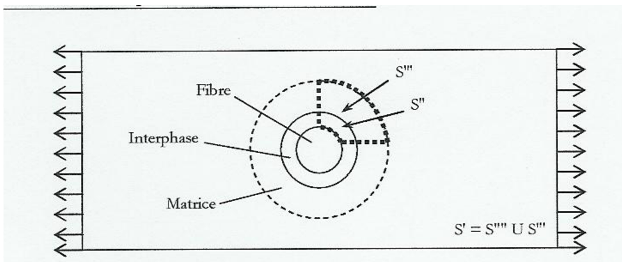


Fig. 5: Geometry of the section of the specimen analyzed by the VFM

Because of the cylindrical geometry of fibre, the polar coordinates will be used associated to a construction of the special virtual fields developed in sub-domains by E. Toussaint, [5] , specifically for this study. One considers an orthotropic law of the linear elastic behaviour for the interphase and the matrix, (Eq.1) .

$$\begin{pmatrix} \sigma_r \\ \sigma_\theta \\ \sigma_s \end{pmatrix} = \begin{pmatrix} Q_{rr_{int}} & Q_{r\theta_{int}} & 0 \\ Q_{r\theta_{int}} & Q_{rr_{int}} & 0 \\ 0 & 0 & Q_{ss_{int}} \end{pmatrix} \begin{pmatrix} \epsilon_r \\ \epsilon_\theta \\ \epsilon_s \end{pmatrix} \quad (1)$$

S'' and S''' are divided respectively into p and q sub-domains. The writing of the virtual works principle by using four principal virtual fields can be discretized as a sum on the various sub-domains:

$$\sum_{i=1}^{p}\int_{S_i} \epsilon_r\tilde{\epsilon}_r^1 dS\cdot Q_{rr_{int}} + \sum_{i=1}^{p}\int_{S_i} \epsilon_\theta\tilde{\epsilon}_\theta^1 dS\cdot Q_{\theta\theta_{int}} + \sum_{i=1}^{p}\int_{S_i} (\epsilon_\theta\tilde{\epsilon}_r^1 + \epsilon_r\tilde{\epsilon}_\theta^1) dS\cdot Q_{r\theta_{int}} + \sum_{i=1}^{p}\int_{S_i} \epsilon_s\tilde{\epsilon}_s^1 dS\cdot Q_{ss_{int}} +$$
$$\sum_{i=1}^{q}\int_{S_i} \epsilon_r\tilde{\epsilon}_r^1 dS\cdot Q_{rr_{mat}} + \sum_{i=1}^{q}\int_{S_i} \epsilon_\theta\tilde{\epsilon}_\theta^1 dS\cdot Q_{\theta\theta_{mat}} + \sum_{i=1}^{q}\int_{S_i} (\epsilon_\theta\tilde{\epsilon}_r^1 + \epsilon_r\tilde{\epsilon}_\theta^1) dS\cdot Q_{r\theta_{mat}} + \sum_{i=1}^{q}\int_{S_i} \epsilon_s\tilde{\epsilon}_s^1 dS\cdot Q_{ss_{mat}}$$
$$= \int_{\delta\Omega} f\cdot\tilde{u}^1 dS \quad (2)$$

Si is the surface of each sub-domain. The form of the sub-domain is selected in such a way that virtual displacements $\tilde{\mathbf{u}}$ are written like the expansion of the shape functions Ni multiplied by the virtual displacements at the nodes $\tilde{\mathbf{u}}_i$:

$$\tilde{\mathbf{u}} = \sum_{i=1}^{n_{nodes}} N_i\cdot\tilde{u}_i \quad (3)$$

where n_{nodes} is the number of nodes by sub-domain. In polar coordinates, equation 3 is written:

$$\tilde{\mathbf{u}} = \tilde{u}_r.\mathbf{e_r} + \tilde{u}_\theta.\mathbf{e_\theta}$$
$$\begin{cases} \tilde{u}_r(r,\theta) = \sum_{i=1}^{p} \tilde{u}_{r_i}(r_i,\theta_i)\cdot N_i(r,\theta) \\ \tilde{u}_\theta(r,\theta) = \sum_{i=1}^{p} \tilde{u}_{\theta_i}(r_i,\theta_i)\cdot N_i(r,\theta) \end{cases} \quad (4)$$

Where $\tilde{\mathbf{u}}_{ri}$ $(ri,\ \theta i)$ and $\tilde{\mathbf{u}}_{\theta i}$ $(ri,\ \theta i)$ respectively represents the displacements according to directions e_r and e_θ at the nodes i of the considered subdomain.

The shape functions are bilinear functions which can be written in the form:

$$Ni(r,\theta) = \frac{(r - rj)(\theta - \theta j)}{(ri - rj)(\theta i - \theta j)} \quad (5)$$

The virtual strains are calculated by derivation of displacements $\tilde{\mathbf{u}}_r$, $\tilde{\mathbf{u}}_\theta$ in polar coordinates. In the present case, we take $p = q = 3$.

The application of the virtual fields method to the strain fields obtained by the technique of digital images correlation, (Fig.3 and 4), allows the evaluation of rigidities of the interphase. Within the framework of this study, the data detailed below are used.

- The stiffness of the matrix corresponds to an isotropic material of epoxy resin type with a Young modulus E = 3 GPa and Poisson's ratio $\nu = 0,3$.
- The mesh has 250 points of measurement within the interphase and 320 on the matrix. The distribution of the number of points of measure to each part is relatively equitable.

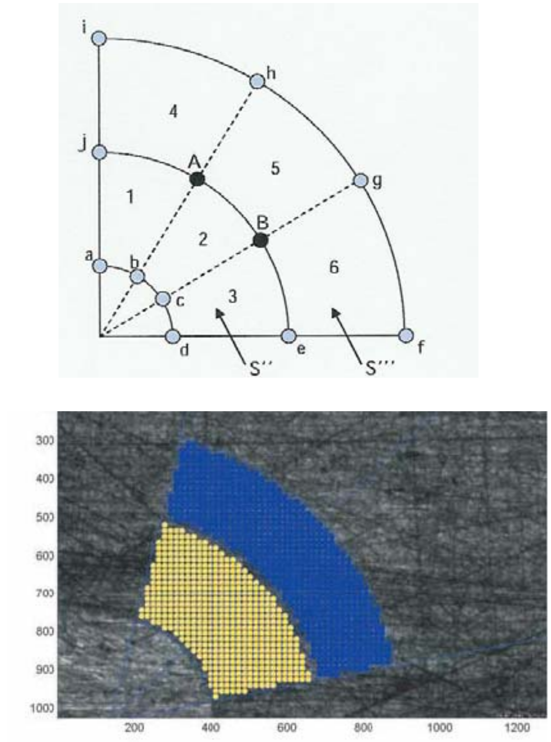


Fig. 6: Mesh of the analyzed zone.

A condition for the application of the virtual work principle is that the virtual fields must be admissible kinematically to zero. One defines the space of the fields kinematically acceptable to zero as being the displacement fields on Ω satisfying boundary conditions equal to zero. Thus, virtual displacement must be zero according to the border S'= S'' *U S'''*, i.e. virtual displacements of the nodes a, b, c, d, e, f, g, h, i, j (fig 6) must be zero. The consequence is that only two nodes A, B have not their displacements equal to zero, *i.e.* $\tilde{u}_{rA}$, $\tilde{u}_{\theta A}$, $\tilde{u}_{rB}$, $\tilde{u}_{\theta B}$.

Finally, the deformations depend only on the displacements of two nodes and all the integrals can be written like a function depending on $\tilde{u}_{rA}$, $\tilde{u}_{\theta A}$, $\tilde{u}_{rB}$, $\tilde{u}_{\theta B}$.

The system is then solved linearly by the use of the special virtual fields :

$$\begin{bmatrix} \int_S \epsilon_x \tilde{\epsilon}_x^1 dS & \int_S \epsilon_y \tilde{\epsilon}_y^1 dS & \int_S (\epsilon_y \tilde{\epsilon}_x^1 + \epsilon_x \tilde{\epsilon}_y^1) dS & \int_S \epsilon_s \tilde{\epsilon}_s^1 dS \\ \int_S \epsilon_x \tilde{\epsilon}_x^2 dS & \int_S \epsilon_y \tilde{\epsilon}_y^2 dS & \int_S (\epsilon_y \tilde{\epsilon}_x^2 + \epsilon_x \tilde{\epsilon}_y^2) dS & \int_S \epsilon_s \tilde{\epsilon}_s^2 dS \\ \int_S \epsilon_x \tilde{\epsilon}_x^3 dS & \int_S \epsilon_y \tilde{\epsilon}_y^3 dS & \int_S (\epsilon_y \tilde{\epsilon}_x^3 + \epsilon_x \tilde{\epsilon}_y^3) dS & \int_S \epsilon_s \tilde{\epsilon}_s^3 dS \\ \int_S \epsilon_x \tilde{\epsilon}_x^4 dS & \int_S \epsilon_y \tilde{\epsilon}_y^4 dS & \int_S (\epsilon_y \tilde{\epsilon}_x^4 + \epsilon_x \tilde{\epsilon}_y^4) dS & \int_S \epsilon_s \tilde{\epsilon}_s^4 dS \end{bmatrix} = \begin{bmatrix} 1 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 \\ 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 1 \end{bmatrix} \quad (6)$$

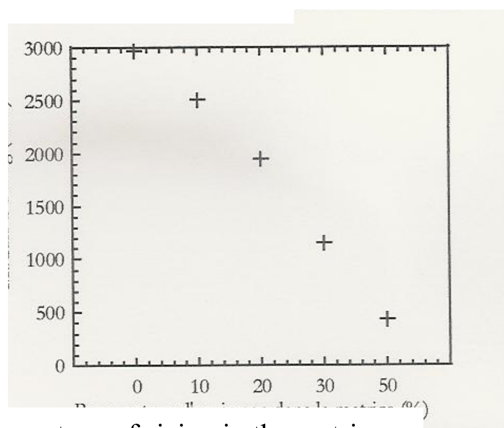
This leads to the identification of the 16 values corresponding to the vectors representing each virtual field. One thus obtains the four coefficients of the orthotropic behaviour law of the interphase: Q_{rr}, $Q_{\theta\theta}$, $Q_{R\theta}$, and Q_{ss}.

Rigidities of the interphase, obtained by the application of the Virtual Field Method, are:

Q_{rrint}	$Q_{\theta\theta int}$	$Q_{r\theta int}$	Q_{ssint}
1,49 *GPa*	1,58 *GPa*	0,15 *GPa*	0,94 *GPa*

The Young modulus of the interphase thus determined is 1,47 Gpa. Different specimens made with various rates of sizing were tested. That made it possible to identify the evolution of the Young modulus with the percentage of sizing in the matrix (Fig 7).

Young modulus (Mpa)



Percentage of sizing in the matrix

Fig. 7: Evolution of the Young modulus of the interphase according to the percentage of sizing.

The value of the Young modulus obtained by the virtual fields method and that directly obtained by experiments for 30% of sizing are in agreement. The values of rigidities for Q_{rrint} and $Q_{\theta\theta int}$ are relatively equivalent, synonymous with isotropy. On the other hand, the Poisson's ratio found ν= 0.1 is far from that hoped: ν = 0.4. That comes from the difficulty in obtaining a stable value of $Q_{r\theta}$.

4. Conclusion

The application of the method of the virtual fields allows determining the mechanical properties of the interphase. The results of the Young modulus seem relatively coherent. For the Poisson's ratio, the method has to be improved. The instability of $Q_{r\theta int}$ rigidity may be the cause of this uncertainty. This value is very sensitive. The use of data with noise induces errors.

5. References

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