AN ORIGINAL NON DESTRUCTIVE TECHNIQUE FOR FIBRE RATE MEASUREMENT

LAURENT AUTRIQUE[1], MIGUEL CANO[2], LAETITIA PEREZ[3]
[1]*Laboratoire d'Ingénierie des Systèmes Automatisés EA4094, 62 Avenue Notre Dame du Lac - 49000 Angers – France; autrique@moniut.univ-bpclermont.fr*
[2]*Institut Français de Mécanique Avancée Campus de Clermont-Ferrand les Cézeaux - BP 265 63175 Aubiere Cedex – France; miguel.cano@ifma.fr*
[3]*Laboratoire de Thermocinétique de Nantes, UMR CNRS 6607, BP 50609, 44306 Nantes cedex 3 – France; laetitia.perez@univ-nantes.fr*

ABSTRACT

This study presents an original method for estimating the rate of resin content in orthotropic layered composite materials while avoiding their destruction. The composite materials considered, commonly used in aeronautic, result of the impregnation of fibre reinforced by epoxy resin. Thermal or mechanical analyses using numerical tools are essential to optimize their implementation, and to better understand their behaviour. The prediction quality of these tools requires the knowledge of input parameters with enough accuracy. Usually dedicated to the identification of the thermal diffusivity, the proposed approach consists in comparing the responses obtained on thermal resin and these obtained on pre-impregnated fibres under periodic thermal waves. An experimental device has been developed and a test campaign has been conducted. The modelling of heat transfer through the complex temperatures method has also been implemented.

KEYWORDS

Characterization; time frequency analysis; composite materials.

INTRODUCTION

Nowadays, composite materials, resulting from the combination of a reinforced carbon fibre and epoxy matrix, meet a great success to major industrial sectors (automobile, aerospace, rail…). Their assets lie in, for example, the formatting of complex parts while reducing the number of interfaces (bolting, riveting or other weld metallic structures). These composites also have a better resistance to mechanical stress and increase resistance to fatigue. Their low density is also an advantage when the structure lightning is a priority. However, as any innovative material dedicated to a specific application, the determination of their properties is essential to ensure their quality. At the present time, the determination of the rate of resin in pre-impregnated fibres is generally realised with heavy chemical treatments. Indeed, a solution of concentrated sulphuric acid and hydrogen peroxide has to be used. This method involves measuring the difference in mass by weighing before and after the extraction of resin by acid attack until a constant mass (Verdu, 1999). One consequence of this process is, of course, the complete destruction of the material studied. To overcome this shortcoming, a non-destructive method has been developed. This method is based on an analysis of the heat wave propagation in a sample submitted to a thermal periodic excitation. Different applications are set for example in (Garrido et al., 2005) (Salazar et al., 1996). Although the key parameter affecting the propagation of heat waves is the thermal diffusivity of the studied material, the use of periodic methods for measuring the rate of fibre is particularly attractive in avoiding the destruction of material to examine. In this communication, the principle of periodic methods is exposed. The experimental bench and results on

isotropic and anisotropic materials are detailed. Finally, the results obtained from the resin and the pre-impregnated fibre are presented.

Implementation of periodic methods.

Generally, dynamic methods dedicated to materials thermal properties identification are based on the behaviour observation of samples submitted to calibrated excitation. Usually, these dynamic methods are classified according to the thermal excitation type. The more usual being the heat step function, the Dirac pulse, the sine-wave modulation and, more recently, the pseudo random sequences. Each of these methods includes advantages and drawbacks that make that either can be more applicable in a given configuration. The approaches based on the implementation of periodic solicitations present the advantage of supplying low energy (compared to flash methods for example) to the studied material as allowing a signal processing informative enough to perform parameter identification without damaging the composite.

Principle

The principle of periodic methods is described on the figure 1. While one face of the material is submitted to a periodic heat flux; observations of the evolution of the system's state (the temperature will be noted θ) are carried out on the opposite face.

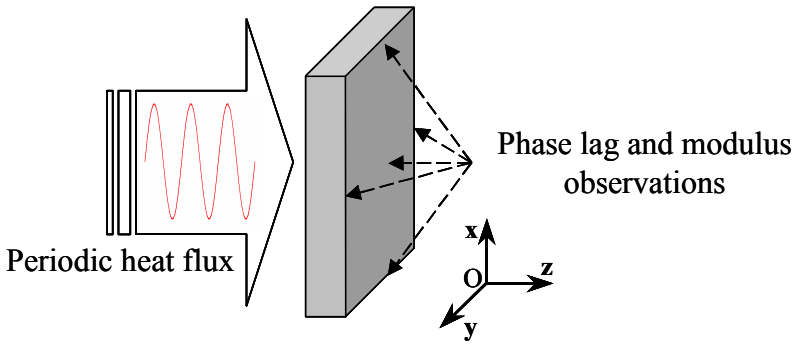


Figure 5 – Principle of periodic methods

Considering the system of equations as linear and considering that the steady state is reached, the system state $\theta(X;t)$ at instant $t \in T$ for each point $X=(x,y,z)\in \Omega \subset \mathbb{R}^3$ of the sample periodically excited can be written as a sum of a steady component and a periodic component which period is the same as excitation (Gurevich *et al.* 2003, Muscio *et al.* 2004).

Thus:

$$\theta(X;t)=\theta_{steady}(X)+\theta_{periodic}(X;t) \qquad [1]$$

with $\theta_{periodic}(X;t)$ a periodic function written as:

$$\theta_{periodic}(X;t)=M(X)e^{j\omega t}e^{j\varphi} \qquad [2]$$

where M [K] is the modulus (amplitude of the oscillations), φ [rad] the phase lag and $f=\frac{\omega}{2\pi}$ [Hz] the frequency of heating input solicitation. $\mu=\sqrt{\frac{\max \vec{\alpha}}{\pi f}}$ in [m] is called diffusion length where $\vec{\alpha}$ is the thermal diffusivity tensor $\left[m^2.s^{-1}\right]$.It is usual to consider that at a distance up to 3μ, at least 95% of the thermal wave is attenuated. The phase lag φ of the thermal wave depends on the thermal diffusivity tensor of the studied

material so observations of φ lead to the estimation of $\vec{\alpha}$. The periodic methods are based on the analysis of thermal waves induced by a photo-thermal periodic excitation which allows material characterization at several geometrical scales. Thus, according to considered spatial scale and according to studied materials, the frequencies domain can change from 0.01 Hz (for insulating materials which have a thermal diffusion length of a few millimeters) to 500 kHz (for thermal conductor materials which have a diffusion length of a few micrometers). These methods can be involved for the characterization of laminated materials (coatings) or, orthotropic materials (such as long fibres composites for example). The measurement can be the temperature, if the material characteristic size and the response time of the data acquisition line allow it, or a temperature dependant parameter (surface reflectance coefficient for example (Autrique *et al*., 2007)). In order to submit the sample of pre-impregnated fibres to a periodic heat source, an experimental bench has been developed and described in the following paragraph.

Experimental bench

An experimental device (Figure 2) has been developed in order to record heat wave propagation using an infra-red camera on the front face of the sample heated from a periodic excitation on its back face. The experimental bench comprises three main parts:

(1) The sample part where the tested material is located in the focal plane of a Köhler optical device (Figure 3). This Köhler optical device is composed of two sets of lenses designed to spatially homogenize the heat flux.

(2) The excitation part constituted by a halogen lamp (36 V - 400 W) under this assembly associated with a process control to generate a periodic input (square, sinusoidal…) on the back face of the sample.

(3) The measurement part made up by an Infra Red camera located on the front face of the sample linked to a signal processing.

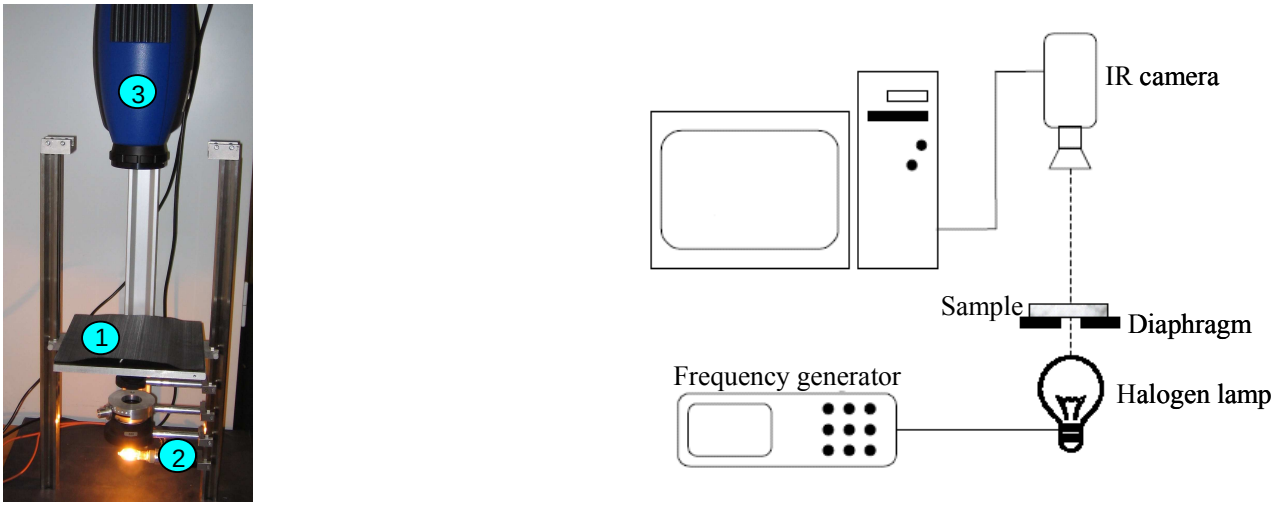


Figure 6 – Experimental bench

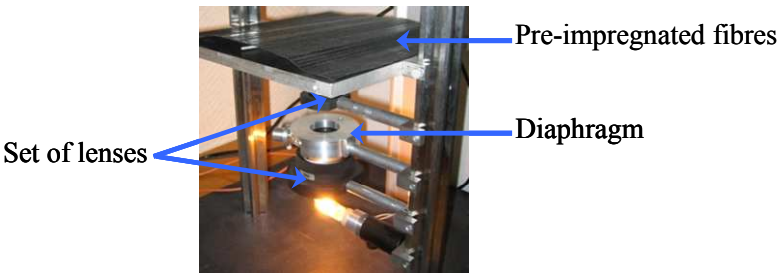


Figure 7 – Optical device

The spatial distribution of the temperature on the sample recorded by the IR camera is representative of the material's nature (isotropic for circular distribution, orthotropic for ellipsoidal distribution …). The heating source power is determined to avoid boundaries effects (the direct problem resolution supposed a given thickness semi infinite plane material). The signal processing allows extracting the amplitude of the signal and the phase lag between periodic input and thermal response of the material, even if the noise level is high. Spatial distributions analysis is performed in order to define the sample geometry, the maximum temperature, the excitation frequency and the distance between IR camera and the observed face. 500 pictures are recorded in order to measure more than 10 periods once the steady state reached.
The main advantage of periodic methods is to allow the periodic signal repetition a lot of times. Thus, they can be used when the signal to noise ratio on observable output is low. A lock-in algorithm is implemented to determine at each point of the front face the phase lag with temperature evolution in the middle of this non heated face. This technique is achieved by software (Max, 1972). In the following of this communication, the whole methodology is validated for reference materials.

Example for insulating isotropic material

The validity of the minimization algorithm has been previously tested for a reference material: a glass plate covered with a thin layer of black paint whose optical behaviour is well known (in order to control the incident flux). The thickness of the plate is $e = 3.85 \quad 10^{-3} \quad \mathrm{m}$. The thermal diffusivity of this insulating isotropic sample is well known: $\alpha_{\mathrm{glass}} = 6 \quad 10^{-7} \quad \mathrm{m}^2.\mathrm{s}^{-1}$. A frequency scanning analysis is carried out to identify, from the phase lag along the thickness (along the Oz-axis), the thermal diffusivity of the material. The results are presented in table 1.

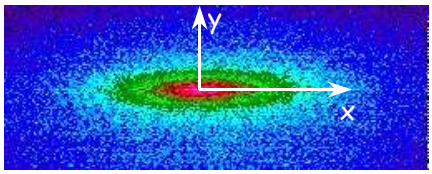
Excitation frequency [Hz]	0.07	0.08	0.09	0.1
Phase lag observed [°]	149	158	172	184
Phase lag after identification [°]	150.8	161.3	171.1	180.4

Table 4 – Validation on isotropic reference material

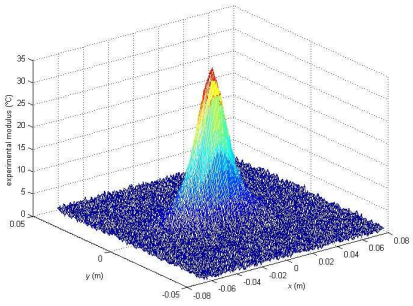
The value of the thermal diffusivity at the end of iterative process of minimization is $\alpha_{\mathrm{glass}} = 5.99 \quad 10^{-7} \quad \mathrm{m}^2.\mathrm{s}^{-1}$ for an initial value of the minimization algorithm of $\alpha_{\mathrm{glass\_0}} = 2 \quad 10^{-6} \quad \mathrm{m}^2.\mathrm{s}^{-1}$. The spatial distribution of the temperature on the sample recorded by the IR camera is representative of the material's nature. Indeed, the glass is an isotropic material; their spatial distributions of temperature are circular.

Example for anisotropic material

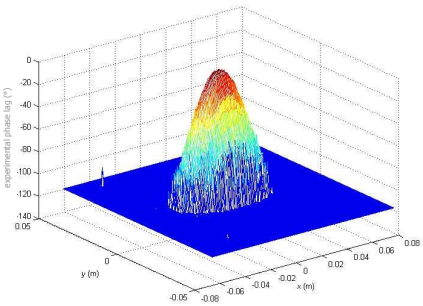
An orthotropic material (fibre-reinforced or woven composites) has been studied using the experimental device presented figure 2. As for the reference material, spatiotemporal observations of temperature distributions allowed to extract modulus and the phase lag of the output signal. Ellipsoidal distributions observed are characteristics of an orthotropic material and highlight the preferential direction of the propagation of the heat.



An infra-red picture : temperature distribution



Modulus



Phase lag

Phase lag distribution of thermal waves

1.1.1.1 Modulus distribution of thermal waves

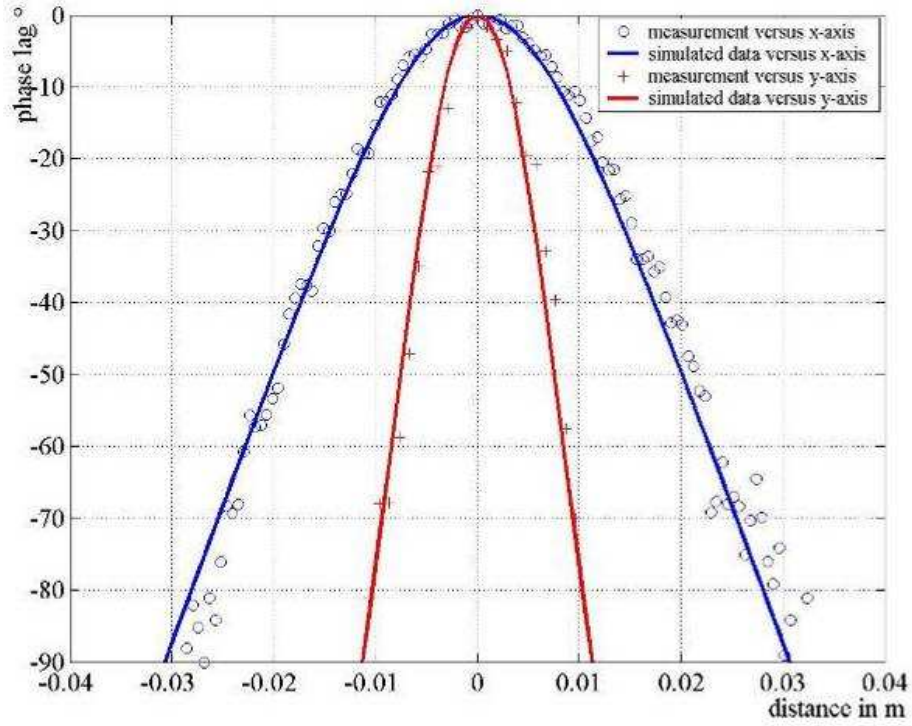


Figure 8 – Validation for orthotropic material

Then phase lag cartography can be considered (figure 4) in order to carry out the identification versus *x*-axis and *y*-axis. Resolution has been performed according to the phase lag observed on each axis. It has been estimated that $\alpha_x \approx 10\alpha_y$ and that $\alpha_x \approx \alpha_z$. This preliminary result is characteristic of fibre stack in a reinforced matrix.

RESULTS

Spatial temperature distributions being representative of the material's nature, it is guaranteed to get circular distribution for isotropic materials like pure resin and ellipsoidal distribution for anisotropic materials such as pre-impregnated fibres (resin & reinforced fibres). The analysis of the geometric ratio between circular distribution and ellipsoidal distribution makes possible the determination of the resin rate contained in a pre-impregnated fibres sample. Indeed, the more the pre-impregnated fibres sample will contain pure resin, the

more the spatial temperature distribution will tend toward a circular distribution. The results presented figure 5 and figure 6 have been recorded after the steady state has been reached (excitation frequency: $f = 0.025$ Hz).

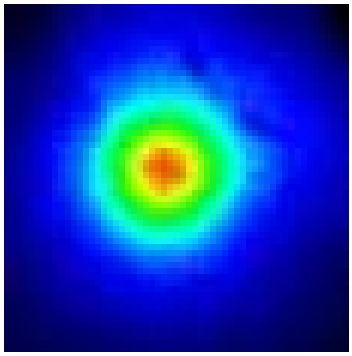


Figure 9 – Experimental spatial temperature distribution of pure resin

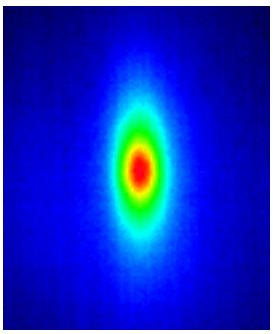


Figure 10 – Experimental spatial temperature distribution of pre-impregnated fibres

Pure resin: low heating powers have been implemented at the beginning of the test in order to not deteriorate the resin (10 cm square plate). Indeed, its lack of rigidity and its sensitivity to temperature rises could warp the sample. Thereafter, in order to obtain an output signal of satisfactory amplitude, the power was increased carefully. Figure 5 shows the circular spatial distribution of the temperatures which characterise an isotropic material. The maximum temperature reached was around 38 °C in the front face of the sample.

Pre-impregnated fibres: A 20 cm square sample of composite material has been used for the realization of this experiment. Their fibre reinforcements are positioned in a single direction. The difference between the thermal diffusivity of fibre reinforcement and its of pure resin reveals a ellipsoidal spatial distribution (Fig. 6) of temperatures. The temperature in the front face of the composite material was close to 38 °C.

The results of the Figures 6 and 7 put in evidence the large difference of thermal behaviour and therefore the properties of two samples: pure resin (epoxy matrix) and pre-impregnated fibres (resin & fibre reinforced).

CONCLUSION

In this communication, the feasibility of a non-destructive technique for the determination of the rate of fibre on pre-impregnated fibres was studied. The analysis of the heat waves propagation generated by a periodic solicitation can characterize the “rate of isotropy ” of an anisotropic material. Indeed, in the studied configuration, as the resin is an isotropic material (characterized by circular thermal waves) and pre-impregnated is a material strongly orthotropic (characterized by ellipsoidal waves), geometrical considerations will quantify the fibres rate in charge of the preferential direction of heat waves. Even if the presented methodology and device has not been developed in the specific framework of in-organic binder systems, this new and attractive non destructive approach has to be extend and characterisations performed on inorganic-bonded fibres composites are planed.

REFERENCES

Autrique L., Perez L., Serra J.J. 2007. « Finite element modelling for micro scale thermal investigations using photo thermal microscopy data inversion », Measurement Science and technology, Institute of physic journal, 18(1) 1-11.
Garrido F., Salazar A., Alonso F., Saez-Ocariz I. 2005. « Characterization of buried cylinders and spheres by pulsed infrared thermography », Journal of applied physics 98.
Gurevich Y.G., Logvinov G.N., de la Cruz G.G., Lopez G.E. 2003. « Physics of thermal waves in homogeneous and inhomogeneous (two-layer) samples », International Journal of Thermal Sciences 42 63-69.
Max J. 1972. Méthodes et techniques de traitement du signal et applications aux mesures physiques, T.1, Masson.
Muscio A., Bison P.G., Marinetti S., Grinzato E. 2004. « Thermal diffusivity measurement in slabs using harmonic and one-dimensional propagation of thermal waves », International Journal of Thermal Sciences, 43 453-463.
Salazar A., Sanchez-Lavega A, Ocariz A., Guitonny J., Pandey C., Fournier D., Boccara A.C. 1996.« Thermal diffusivity of anisotropic materials by photothermal methods », J. Appl. Phys. 79(8) 3984-3993.
Verdu J. 1999. « Introduction à l'analyse physico-chimique des polymères » Techniques de l'ingénieur, AM 3270.