FPCM-9 (2008)
The 9th International Conference on Flow Processes in Composite Materials
Montréal (Québec), Canada
8 ~ 10 July 2008

A NEW EXPERIMENTAL DEVICE FOR GLOBAL THERMAL CHARACTERIZATION OF ORTHOTROPIC COMPOSITES

M. Thomas [1], N. Boyard [1, 2], Y. Jarny[1] and D. Delaunay[1]

[1] *CNRS, Université de Nantes, Nantes Atlantique Universités, Laboratoire de Thermocinétique, UMR 6607, rue Christian Pauc, BP 50609, F- 44306 Nantes cedex 3, France*
[2] *Corresponding author's Email: nicolas.boyard@univ-nantes.fr*

SUMMARY: This paper deals with the development of a new experimental measurement. Based on an inverse method, an original design of a heating device allows the estimation of the whole thermal conductivity tensor and the specific heat of orthotropic composite material. The heater design development, the sensitivity analysis, the available estimation strategies, as well as the experimental validation of the method are presented.

KEYWORDS: thermal conductivities, experimental device, inverse method

INTRODUCTION

Nowadays, a large range of processes is available for the manufacturing of composite materials parts. Before their assembling and their use, their properties must be accurately determined. More specifically, the knowledge of thermal properties of orthotropic layered Carbon Fibre Reinforced Polymer (CFRP) composite materials and the control of uncertainties associated to their experimental measurements are among the essential points to perform accurate and reliable numerical computations for aircraft structures. The prediction of temperature maps of a part enables to assess to the repartition of thermal stresses.

Even if no standard currently exists to measure these thermal properties, a lot of techniques [1-3] already exist to estimate them. However, they have to be combined or repeated several times for an extensive and full thermal characterisation. This paper aims the design a new experimental device to estimate simultaneously the thermal conductivities in the main directions (diagonal terms of the thermal conductivity tensor) and the specific heat (C_P) of orthotropic composite materials. The identification of the parameters is performed by solving a 3-D inverse heat conduction problem. The conception of the device is focused on fast characterization, i.e. few experiments and no tedious intrusive instrumentation. Experimental measurements are made on PMMA samples to validate the methodology and unidirectional CFRP samples.

GENERAL PRINCIPLE OF THE DEVICE

The experimental device (Fig. 1) consists of a thin electrical heater, sandwiched between two similar composite samples. The symmetrical assembly (heater and samples) is located in a thermo-regulated vacuum chamber. The vacuum chamber is equipped with a ZnSn window through which an IR pyrometer measures the surface temperature of the sample.

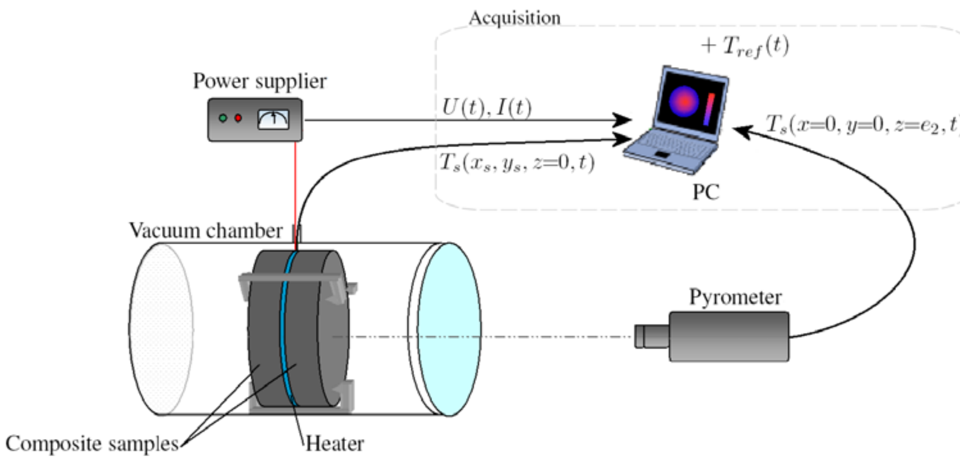

Fig.1 Schematic presentation of the experimental device.

The heater (Fig. 2) is the most important element of the method since it serves as both heat source and temperature measurement device. Its development has required several tests and experiments. The final version presented here, is composed of a stack of Kapton3 discs on which are set two distinct circular heating tracks: a central heating disc of radius r_{int}, and a peripheral heating corona between the interior radius r_{int} and the exterior radius r_{ext}. Fourteen *T*-type micro-thermocouples (30 µm diameter) are located inside the heater (seven per face – red dots-, as displayed in Fig. 2). They ensure the measurement of temperature evolutions for different directions (for different angles) and at different distances (different radii) from the centre.

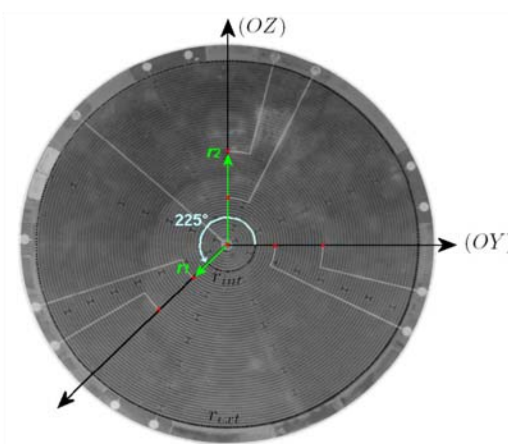

Fig. 2 Upper face of the heater composed of two heating tracks.

An IR pyrometer focused on the centre of the upper face of one sample ensures the measurement of the transverse thermal gradient. At least, thin rubber sheets are placed between the composite samples and the heater to limit the roughness effect, and thus to ensure a good interface thermal contact. Two thermo-regulated plates are located on both sides of the assembly. They ensure the measurement temperature of the assembly by radiation.

Two samples of the same material are required for tests with this device. Their shape is circular with a radius of 60mm and their maximum thickness is 50mm in the actual design. The two samples have to be oriented along the same direction when they sandwich the heater. The experiment consists of dissipating a known heat flux in the heater and then measuring the resulting temperature increase. The analysis of the temperature evolution in the heater as well as on the sample upper face using the 3D inverse heat conduction problem permits to characterize the thermal properties of the tested composite material. The measurement temperature range starts from the ambient up to 200°C (maximum temperature for the heater).

IDENTIFICATION PROCESS OF THE THERMAL PROPERTIES

The aim of the developed identification process is to determine the thermal properties of an orthotropic composite material. In this case, the thermal conductivity tensor in the coordinate system of the main directions (Oxyz) is diagonal. The main directions are supposed to be orthogonal. We naturally consider the transverse direction (Oz), which is the sample thick-direction, as one of the main direction.

Except in some peculiar case (e.g., unidirectional composites), the composite thermal principal directions are generally unknown. Let us consider the coordinate system of the heater (OXYz). In this coordinate system, the composite thermal conductivity tensor can be written as:

$$\Lambda^c_{OXYz} = \begin{bmatrix} \lambda^c_{XX} & \lambda^c_{XY} & 0 \\ \lambda^c_{XY} & \lambda^c_{YY} & 0 \\ 0 & 0 & \lambda^c_{zz} \end{bmatrix} \quad (1)$$

The objective of the measurement method aim is thus to estimate these five parameters as well as the specific heat. Then, from Λ_{OXYz} it is possible to find a basis in which this tensor is diagonal [4]. This basis composed by the main directions, is found determining eigenvector whereas the components of Λ_{Oxyz} are the eigenvalues. (Oxyz) and (OXYz) are rotated about the (Oz) axis through an angle θ.

An inverse method is used for estimating these parameters. The direct problem is solved using a 3D finite element solver. Identification of the unknown composite thermal properties is based on the minimisation of a function representing the sum of the differences between the observations (measured temperatures) and the calculated temperatures (direct problem) at the same locations.

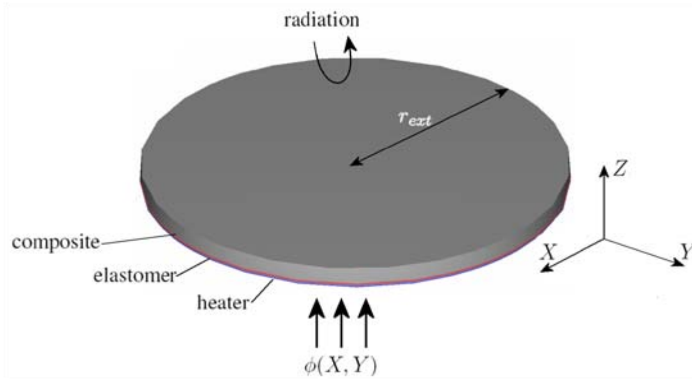

Fig. 3 Finite element geometry scheme containing heater, elastomer and composite fields.

Modelling of Spatial Temperature Fields with Finite Elements

The assembly (composite sample/ elastomer sheet / heater / elastomer sheet / composite sample) being symmetrical, the thermal problem is reduced to the geometry presented in Fig. 3. The spatial domain of the model equations involves three sub-fields: Ω_1 for the heater, Ω_2 for the elastomer and Ω_3 for the composite sample.

At the initial time of the experiment the heater is off and the temperature of both composite samples and heater are uniform and equal to that of the vacuum chamber. For $t \in]0, t_f]$, a uniform heat flux is dissipated by the heater. It is modelled with a heat flux density $\phi(X, Y)$ applied on the mid-plane. Then, the temperature fields $T_i(x,y,z,t)$, *i=1,2,3,* are obtained by solving the heat conduction equation in Ω_i :

$$(x,y,z) \in \Omega_i \,,\, 0 < t < t_f :\ \rho_i Cp_i \frac{\partial T_i}{\partial t} = \nabla \cdot \left(\Lambda_i \nabla T_i \right) \tag{2}$$

The following boundary and interface conditions are considered for computing the solution:

- on the lower face of the heater, dissipation of a heat source;
- on the lateral face of the heater, on the lateral and upper faces of the composite: radiative condition;
- on the internal boundary between the heater and the composite: spatially uniform heat flux condition where a thermal contact resistance is introduced to account for the non-perfect contact between the heater and the samples;
- on the other boundaries, adiabatic conditions are considered to account for the symmetry configuration (note that *n* is the normal vector to the surface).

Sensitivity Fields

Computation of sensitivities to the unknown parameters is of prime importance. On one hand, they provide information on the identification feasibility and enable determining the temporal window for which a variation of each unknown parameter β_j is independent of the others and leads to a significant influence on the system. On the other hand, they directly play a role in the inverse algorithm. The sensitivity field to the parameter vector $\beta = [\beta_j]_{j=1:m}$ in the sub-field Ω_i is defined by:

$$X_{ij}(x,y,z,t;\beta)=\frac{\partial T_i(x,y,z,t)}{\partial \beta_j},\ j=1:m;\ i=1:3 \qquad (3)$$

In practice, the thermal conductivity tensor, specific heat, thermal contact resistance between the heater and the samples, and the emissivity of the external surfaces must be determined. This defines an unknown parameter vector. The sensitivity fields are obtained by differentiating the model equations used in the previous sub-section with respect to each component of the parameter vector.

It is observed that the sensitivity equations are similar to the heat equation, but they involve additional terms, coupled to the model equations. Thus, as the temperature fields $T_i(x, y, z, t)$, the sensitivity fields are calculated with finite elements. To avoid storing the coupled terms, it is judicious to calculate simultaneously the whole fields T_i, X_{ij} (I = 1 to 3; j = 1 to m) by solving a global set of equations.

Optimal Heater Design for Estimation Strategy

Special care has been given to the design of the heater. Numerical tests have been performed to determine the power required, the dimensions of the central heating part and the location of the thermocouples. In the latter case, sensitivity fields were therefore of paramount importance.

The heater design offers two distinct heating configurations. The configuration ① consists in powering the central part of the heater. For orthotropic composite materials, the isotherm lines are distorted in ellipses. Thanks to the micro thermocouples located in the heater, the analysis of the different temperature raises for different locations in the heater plane leads to the determination of the planar thermal conductivities: λ_{2xx}, λ_{2yy} and λ_{2xy}, as well as the specific heat. Moreover, thanks to the pyrometer, the temperature evolution measurement of the external surface of the sample, allows us to estimate the transverse thermal conductivity λ_{2ZZ}. In the configuration ②, both central part and peripheral corona are powered to dissipate a known uniform heat flux on the entire samples faces that are in contact. In that case, the mid-plane ($O,x,y,z = 0$) is isothermal, so that $X(\lambda_{2xx})$, $X(\lambda_{2yy})$ and $X(\lambda_{2xy})$ are null.

Inverse Method

The method we use to estimate the thermal properties of composite samples is based on the Ordinary Least Square (O.L.S.) estimation technique. Considering ns the number of sensors and nt the number of sampling times, the output model vector at the sensor locations can be defined by:

$$Y(\beta)=[C]\cdot T(\beta) \text{ with } \dim(Y)=ns\times nt \qquad (4)$$

where $T(\beta)$ is the finite element approximation of the solution of the model equations, and $[C]$ the sensor location matrix. Measurements are assumed to be corrupted only by an uncorrelated, zero mean, Gaussian, additive noise ε and: $\tilde{Y} = [C] \cdot T + \varepsilon$. The estimation of $\beta = [\beta_j]_{j=1:m}$ consists of minimising the O.L.S. criterion:

$$S(\beta) = \sum_{k=1}^{ns \times nt} \left(\tilde{Y} - Y(\beta) \right)^2 \tag{5}$$

The model solution $Y(\beta)$ being not linear with respect to β, the minimum $\hat{\beta} = \arg\min S(\beta)$ is computed according to the Gauss Newton algorithm. For each iteration, the new estimate $\beta^{(k+1)}$ is computed by adding the parameter variation $d\beta$ to the estimate $\beta^{(k)}$

$$d\beta^{(k)} = (X_s^{(k)t} X_s^{(k)})^{-1} X_s^{(k)t} \left(\tilde{Y}_s - Y_s\left(\beta^{(k)}\right) \right) \tag{6}$$

The subscript "s" indicates the variable is considered at the temperature sensor locations. To obtain an idea on the uncertainty of the results, it is common to introduce the final relative estimation errors on each component

$$re(\beta_j) = \frac{\sigma_N}{\beta_j} \sqrt{diag_j (X_s^t X_s)^{-1}} \tag{7}$$

where σ_N is the standard deviation of the measurement noise. It is evaluated during a short period prior to heating. (The notation *diag* indicates that the only diagonal terms are considered here.)

Estimation Strategy

It has been demonstrated that two strategies can be employed for the parameters estimation:
• Firstly, the use of configuration ① only permits to estimate all the parameters. Nevertheless, we have noticed that some parameters are measured with a rough accuracy.
• Secondly, another strategy would consist in using the two configurations for the estimation of the whole properties. In fact, employing first configuration ②, one can estimate the parameters λ_{zz}, *Cp*, and *Rtc*. It is important to notice that this estimation is not dependent of the in-plane thermal conductivities (the associated sensitivities are zero since *T(X, Y)* is isothermal). Once these parameters estimated, the use of configuration ① permits to estimate the three other parameters λ_{XX}, λ_{YY}, and λ_{XY}. Thus, all the parameters are estimated in two steps. Dividing the estimation in two experiments, relative errors are lower. Consequently, the estimation is then more accurate. Note nevertheless that the knowledge of parameters λ_{zz}, *Cp*, and *Rtc* is required for the configuration ①, It is then not possible to inverse the order of the estimations.

EXPERIMENTAL RESULTS

Validation for PMMA

In order to validate the proposed measurement method, tests on isotropic material with well-known thermal properties will be carried out. PMMA is an isotropic homogeneous material, whose thermal conductivity and specific heat have already been characterized.

Two PMMA discs, 5 mm thick, were instrumented with four thermocouples so has to measure the temperature rise inside the PMMA. This precaution permits to know if the F.E. model is able to predict the temperature raise inside the tested samples and to detect if the heat flux dissipated by the heater is well shared in two equal parts in both samples. Since the PMMA material is isotropic, only three parameters need to be estimated. Then, the parameter vector to estimate is

$$\beta = [\lambda^{PMMA} \quad C_P^{PMMA} \quad Rtc] \tag{8}$$

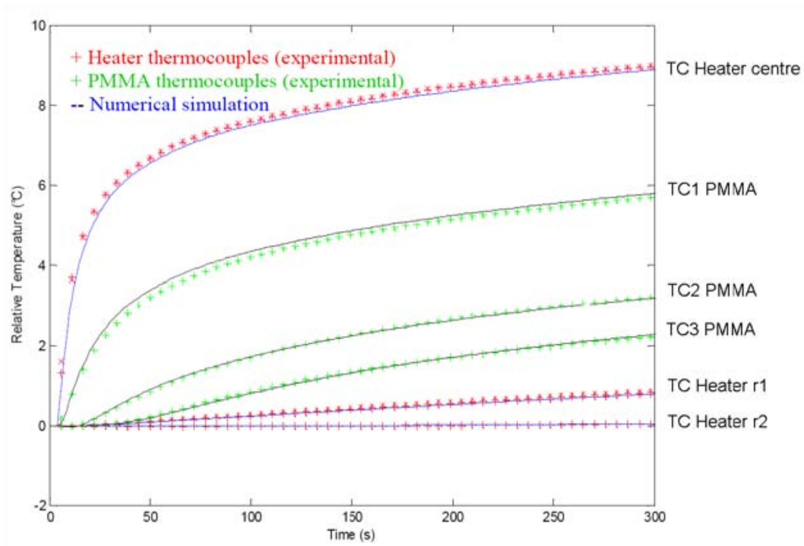

Fig. 4 Experimental and numerical temperature variations in PMMA sample - Configuration ①.

Two PMMA discs, 5 mm thick, were instrumented with four thermocouples so has to measure the temperature rise inside the PMMA. This precaution permits to know if the finite element model is able to predict the temperature raise inside the tested samples and to detect if the heat flux dissipated by the heater is well shared in two equal parts in both samples. Since the PMMA material is isotropic, only three parameters need to be estimated. Then, the parameter vector to estimate is

$$\beta = [\lambda^{PMMA} \quad C_P^{PMMA} \quad Rtc] \tag{9}$$

Both configurations (① and ②) can be used for estimating these parameters at 45°C. Fig. 4 shows the temperature evolution inside heater and PMMA samples when configuration ① is employed. A radial thermal gradient is observed. Estimated parameters are carried forward Table 1. Although a slight difference is observed between the results of both configurations, results are in agreement with dedicated measurements, and literature data. This validates the experimental measurement method in the case of an isotropic material.

Validation for a Unidirectional Composite

The tested material is pure unidirectional composite is an AS4/8552 RC34 AW196 UD tape from Hexcel Composites. The resin content in weight is about 34%, and its density is 1590 kg/m^3. In order to estimate the three components of the thermal conductivity tensor Λ_{3OXYz}, the angle θ and the specific heat, we used both heating configurations. For the results presented, unidirectional composite samples are oriented so that the fibres direction makes an angle of 20° with the *Ox* direction of the heater.

Table 1 Estimated thermal properties at 45°C of PMMA using both heating configurations, and comparison with a dedicated guarded hot plate (GHP) and DSC measurements and a data from literature [5]

Parameter		Conf. A 45°C	Conf. B 45°C	GHP / DSC 45°C	Literature 20°C
λ^{PMMA}	(W/m.K)	0.202 ± 1%	0.197 ± 0.5%	0.20	0.197
C_P^{PMMA}	(J/kg.K)	1508 ± 1%	1491 ± 0.5%	1532	1380
Rtc	(m².K/W)	$4\ 10^{-3}$ ± 3%	$3\ 10^{-3}$ ± 2%		

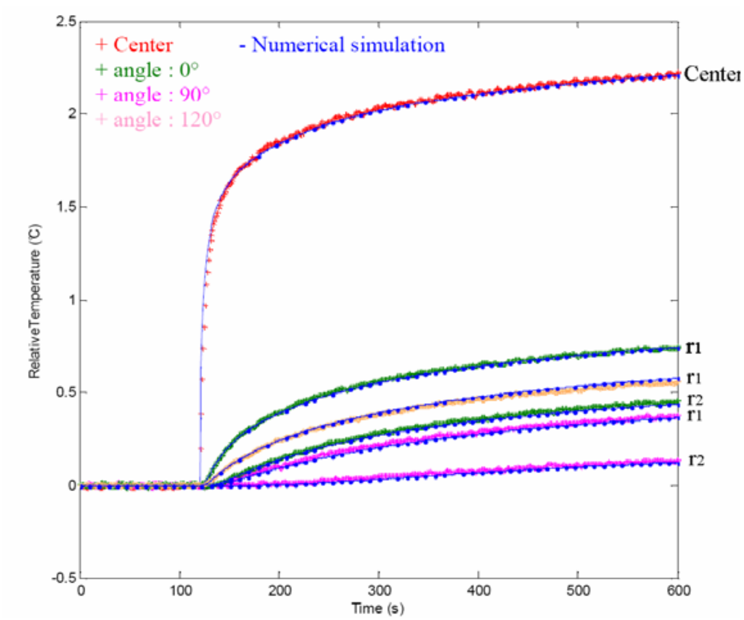

Fig. 5 Heating configuration ①: Experimental and numerical temperature rises for a unidirectional composite sample.

Heating configuration ① is then used to estimate λ_{3XX}, λ_{3YY} and λ_{3XY} whereas configuration ② is devoted the determination of λ_{3zz}, Cp_3 and *Rtc*. Fig. 5 illustrates the concordance between experimental results and numerical computation once inverse program has converged. Results are given in Table 2. Thermal conductivity tensor components are expressed in the composite main thermal direction coordinate system.

The angle value estimated between the two coordinate systems is θ = 23°. This result is close to the orientation we imposed (θ = 20°). The estimation of the main thermal directions of the composite seems thereby to be efficient. Note however, that this composite presents a strong anisotropy: $\lambda_{3xx} / \lambda_{3yy} \approx 8$. One can notice that the thermal contact resistance estimated is very low. This is due to the use of rubber sheets that ensure a good thermal contact between the samples and the heater.

Table 2 Estimated thermal properties at 45°C of a unidirectional composite using both heating configurations

Parameter	Estimated value	Relative error
λ_{3xx}	5.35 W/m.K	
λ_{3yy}	0.662 W/m.K	
λ_{3zz}	0.62 W/m.K	2.23%
Cp_3	1050 J/kg.K	4.06%
Rtc	$5\ 10^{-4}$ m².K/W	8.09%
θ	23°	

CONCLUSION

A new experimental device based on a non-stationary method is developed to identify simultaneously the specific heat and the three components of the thermal conductivity tensor of orthotropic composite materials. Thanks to the 3D heat conduction model and to the sensitivity analysis, an optimal design of the heater has been achieved and the feasibility of identification is checked. The parameters identification method is based on the numerical resolution of an inverse heat conduction problem. For that purpose, the solution of the mathematical equations of the model is computed by a finite elements solver. We also clearly show that two strategies of estimation can be proposed to the experimenter. Conclusive tests have been performed on a PMMA sample with a well-known thermal conductivity. Experimental measurements on a pure unidirectional CFRP composite have been then realized, leading to thermal properties in good agreement with other previous results.

ACKNOWLEDGEMENTS

The authors thank Airbus® industry for the financial support of this work.

REFERENCES

1. U.Grigull and H. Sandner, "Heat Conduction". *Springer Verlag*, 1984.

2. Parker, Jenkins, Butler, Abbot, "Flash Methods of Determining Thermal Diffusivity, Heat Capacity and Thermal Conductivity", *Journal of Applied Physics*, Vol. 23, 1961, pp. 1679-l684.

3. A. Degiovanni, J.C. Batsale, D. Maillet, "Mesure de la diffusivité longitudinale de matériaux anisotropes", *Revue générale de thermique*, Vol. 35, 1996, pp. 141-147.

4. N. Ozisik, "Heat conduction". *John Wiley Sons inc.*, 1993.

5. D.W. Van Krevelen, "Properties of Polymers", *Elsevier*, Amsterdam, 1990.