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On the use of periodic photothermal methods for materials diagnosis

Laurent Autrique[a,d,*,1], Laetitia Perez[b], Emmanuel Scheer[c]

[a] LAMI, IUT Montluçon, avenue Aristide Briand, BP 2235, 03101 Montluçon cedex, France
[b] LTN, BP 50609, rue Christian Pauc, BP 50609, 44306 Nantes cedex 3, France
[c] DGA-CEP-EHF, 10 rue des fours solaires, BP 59, 66121 Font Romeu, France
[d] ISTIA – LISA, 62 avenue Notre Dame du Lac, 49000 Angers, France

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ABSTRACT

This work aims the analysis of valuation methods devoted to materials diagnosis in order to provide an efficient estimation in practical operational conditions and environment (by the observation of a thermal tracer representative of a damage). The followed methodology consists in implementing observation techniques based on a periodic photo-thermal excitation so that the observation of the heated structure response allows to identify characteristic parameters of the studied materials. In most cases, simplistic hypotheses required for analytical model validation are not satisfied. Thus, analysis in the frequency domain requires the computing of a specific finite elements method.

1. Introduction

The development of proper diagnoses strategy dedicated to the characterisation of innovating materials is a crucial requirement in many emergent technological fields. Once the material is elaborated with an eye to future applications, it is essential to test if the desired properties are not altered in using conditions. In most cases, industrial requirements are, on one hand, to improve the knowledge of materials properties (multi-scale analysis in order to take into account structural heterogeneities, relations between parameters of the elaboration process and specific properties) and on the other hand to ensure that functionality is preserved in using conditions.

In such a framework, results are expected in order to answer to industrial concrete problems such as for example: what is the effect of a thermo mechanical cycling on a multi-layered material? How to quantify efficiency loss of a stuck structure? What are the relations between global properties of a reinforced composite and local properties of its fibres and matrix? How to detect the emergence of a fouling likely to induce a defect in a reactor?

Considering several spatial scales, multi-scale analyses can link a local behaviour (some micrometers) with a global behaviour (some millimetres). Thus, the understanding of the local damage mechanisms will be improved, and this study could contribute to provide technological solutions for structures life increase. In order to investigate these various objectives, the proposed diagnoses must be quite general.

In the following, diagnosis is defined as a material state estimation deduced from observations in order to detect a possible alteration in comparison with a previously estimated reference state. Then, a comprehensive methodology based on numerous advantages of periodic methods [1] providing such a diagnosis has to be developed and has to be validated for several classes of materials and various specific applications. Generally, dynamic methods dedicated to materials thermal properties identification are based on the behaviour's 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 [2].

Each of these methods includes advantages and drawbacks that make that either can be more relevant in a given configuration. The investigated methodology is based on the analysis of thermal waves induced by a photo-thermal periodic excitation which allows material characterisation at several geometrical scales. Previous studies, [3–6], have allowed to develop analytical models in simplified geometrical configurations (semi-infinite wall, parallel multi-layered system). In most cases, these simplistic hypotheses are not assumed to be satisfied and all parametric identification results ensuing from an erroneous forward problem have to be considered with great circumspection. So, it becomes essential to numerically solve the forward problem with an adapted method

* Corresponding author. Tel.: +33 470 022 010; fax: +33 470 022 078.
E-mail addresses: autrique@moniut.univ-bpclermont.fr (L. Autrique), laetitia.perez@univ-nantes.fr (L. Perez).
[1] Tel.: +33 241 226 560; fax: +33 241 226 561.

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Nomenclature

$\Omega_i \in \mathbb{R}^3$	space domain
T	time domain
$X=(x,y,z)\in\Omega$	space variable
$t\in T$	time variable (s)
$\Gamma \in \mathbb{R}^2$	surface of Ω
ω	pulsation (rad s^{-1})
$\phi(X,t)$	heat flux (W m^{-2})
λ	thermal conductivity (W m^{-1} K^{-1})
$\theta(X,t)$	temperature (K)
C	volumetric heat (J m^{-3} K^{-1})
h	convective heat transfer coefficient (W m^{-2} K^{-1})
$\vec{n}$	normal vector exterior to Γ
α	thermal diffusivity (m^2 s^{-1})
μ	diffusion length (m)
f	frequency excitation (Hz)
φ	phase lag (rad)
$\lvert\theta_\omega\rvert$	modulus (rad)
e	sample thickness (m)
R	heating source radius (uniform distribution) (m)
r	dimensionless ratio e/μ
L	square side length (m)
r_0	heating source radius (Gaussian distribution) (m)
S	observable
β	parameter
F^*	reduced sensitivity function

avoiding the hypothesis indispensable to the establishment of an analytical solution (dimensions, linearity, and homogenisation).

This study in the frequency domain requires the computing of a specific finite elements method. The partial differential equations system allowing to obtain the temperature modulus and phase lag will be taken into account in three dimensional geometries representative of using conditions. The parametric identification can be performed by minimisation of a quadratic criterion with the help of iterative methods. The whole approach requires the study of the following aspects: boundaries conditions formulation considering material heterogeneities, parameters identifiability, optimal design of experiment (periodic excitation type, frequency, amplitude, and optimal data acquisition strategy), and inversion in the frequency domain [7].

These problems, taken as a whole, aim at solving in good conditions the inverse problem leading to the thermophysical parametric identification for heterogeneous and anisotropic materials [8]. Such an inverse problem resolution can also be helpful in order to test the sample state versus its nominal state and to answer to the following questions: for heterogeneous material, at which scale the material can be considered as homogeneous? Considering a previously estimated nominal state, has the chosen tracer (for example the thermal diffusivity) been modified (which can be representative of its structure modification)? Has the contact thermal resistances inherent in a multi-layered material been modified (which can be representative of high internal constraints)? Is the interfaces size unchanged (which can detect cracks evolution)?

Finally, this study will allow disposing of a global and robust method for the monitoring of a key parameter contributing towards the materials diagnosis. This paper is organised as follows. The next section deals with the proposed periodic method and is focused on the required hypothesis for the validation of an analytical solution of the forward problem. A more global context is investigated using a finite element numerical approach. In Section 3, limitation and invalidation of the analytical approach models are pointed out in several usual configurations. Considering the numerical solution (based on finite element discretisation), sensitivity analysis is performed as millimetric and micrometric scales in Section 4. Finally, in the last section, several experimental devices developed in the DGA-CEP-EHF institute are briefly exposed.

2. Periodic photothermal method

In the following, periodic methods are implemented to observe a tracer representative either of an expected efficiency (aim: characterisation and elaboration) or of an efficiency loss (aim: damage). When signal/noise ratio is low, periodic methods are quite relevant since material observations are analysed using excitation sources with a small energy level. Thus, characteristics of structure or modifications can be identified in most cases according to non-destructive methods.

This work concerns the analysis of the response of the material submitted to a periodic input located on the sample surface. Adapted to each configuration, material valuation can be performed from millimetric scale (frequency excitation can be inferior to 0.01 Hz) to micrometric scale (frequency excitation can be greater than 500 kHz). The output 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).

Thermal waves produced by a periodic heat generation in homogeneous and inhomogeneous solids are examined from the theoretical point of view in [1]. When a solid sample submitted to a sinusoidal excitation reaches a thermal steady-state, the temperature in all points is the sum of a continuous component and a periodic one (of same period that excitation) characterised by its amplitude and phase lag versus thermal excitation. The temperature amplitude and temperature phase lag analysis in the material allow identifying some key parameters of the heat transfer.

Moreover, by introducing the complex temperature concept, the phase lag can be obtained in steady state without taking into account the time evolution. Let us consider the following notations: $\Omega_i \in \mathbb{R}^3$ is the space domain corresponding to each material i, $X=(x,y,z)\in\Omega=\cup\Omega_i$ is the space variable, $t\in T$ is the time variable. The periodic heat flux focused on the surface Γ of Ω can be expressed in the form:

$$\phi(X,t) = \phi_0(X)\,e^{j\omega t} \tag{1}$$

where $\phi_0(X)$ is the heat flux amplitude (W m^{-2}) at point X, and ω is the pulsation (rad s^{-1}). Let us consider that initially the material temperature is uniform θ_i (however, it is obvious that steady periodic solution reached after a sufficiently long time does not depend on the initial condition). Let us assume that the uniform temperature of the fluid in contact with the material surface during the experimentation is θ_i. Temperature is denoted by $\Theta(X,t)$ and the evolution of reduced temperature $\theta(X,t)=\Theta(X,t)-\theta_i$ in $\cup\Omega_i$ is described by the following equations:

$$\forall(X,t)\in\cup\Omega_i\times T \qquad div\left(\vec{\vec{\lambda}}_i\overrightarrow{grad\,(\theta(X,t))}\right) - C_i\frac{\partial\theta(X,t)}{\partial t} = 0 \tag{2}$$

where $\vec{\vec{\lambda}}_i$ is the thermal conductivity tensor (W m^{-1} K^{-1}) and C_i is the volumetric heat (J m^{-3} K^{-1}) of material i. Boundaries conditions are described by

$$\forall(X,t)\in\Gamma\times T \qquad -\lambda_i\frac{\partial\theta(X,t)}{\partial\vec{n}} = \Re_e(\phi(X,t)) - h\theta(X,t) \tag{3}$$

where $\vec{n}$ is the normal vector exterior to Γ, $\Re_e(\phi(X,t))$ is the real part of the complex term $\phi(X,t)$, h is the convective heat transfer coefficient (W m^{-2} K^{-1}). h is considered uniform over all the body surface.

Since the heat flux is periodic on Γ, temperature variations in $\cup\Omega_i$ will be periodic as well. When the steady-state is established, a continuous component and a periodic one are considered:

$$\tilde{\theta}(X,t) = \theta_c(X) + \theta_\omega(X)\,e^{j\omega t} \quad (4)$$

where $\tilde{\theta}(X,t)$ is the complex temperature. In the following, the study is devoted to the periodic component i.e. computation of its amplitude and phase lag with respect to the incident flux. From (2) and (3), we obtain:

$$\forall X \in \cup\,\Omega_i \qquad div\left(\vec{\lambda_i}\overrightarrow{\mathrm{grad}\,(\theta_\omega(X))}\right) - j\omega C_i\theta_\omega(X) = 0 \quad (5)$$

$$\forall X \in \Gamma \qquad -\lambda_i\frac{\partial\theta_\omega(X)}{\partial\vec{n}} = \phi_0(X) - h\theta_\omega(X) \quad (6)$$

In specific configurations such as homogeneous solid, semi-infinite geometries, temperature independent parameters, particular multi-component configurations (for which thermal interfaces are well identified), calculation of the inverse Fourier transform (or Laplace inverse transform) leads to a semi-analytical solution, see theoretical aspects and applications in [8–12].

From the experimental point of view, for heterogeneous materials which do not verify previous assumptions, thermal diffusivity identification according to semi-analytical solution can lead to erroneous estimation. In order to provide a general alternative for the resolution of Eqs. (5) and (6), the finite element method is implemented (using Comsol® software).

3. Analytical approach limitations

In the following, the limitations of geometrical hypothesis are investigated. Let us denote by $\alpha_i = \lambda_i/C_i$ in $(m^2\,s^{-1})$ the thermal diffusivity and by $\mu_i = \sqrt{\lambda_i/\pi C_i f} = \sqrt{\alpha_i/\pi f}$ the diffusion length in (m) where $f = \omega/2\pi$ represents the frequency excitation in (Hz).

3.1. 1D hypothesis

The studied configuration has been devoted to thermal diffusivity identification of homogeneous semi-infinite plane samples. An experimental bench has been developed (see Section 5.1). The sample is periodically heated on the upper face ($x = 0$) and temperature evolution is measured on the lower face ($x = e$), see Fig. 1. This configuration is also called transmission. The heat flux spatial distribution is assumed to be uniform on a disk (radius $R \geq e$). Since the main characteristic of thermal wave spatial attenuation is the diffusion length, we are mainly interested in the relation between R and the ratio $r = e/\mu$ where e is the plane thickness. Three material types are considered in order to investigate several thermal behaviours: ($\alpha_{silver} \approx 170 \times 10^{-6}\,m^2\,s^{-1}$;

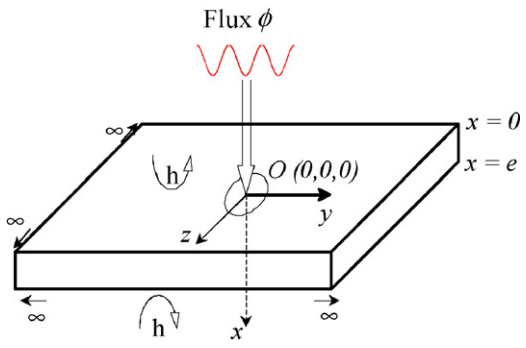

Fig. 1. Studied configuration for 1D hypothesis.

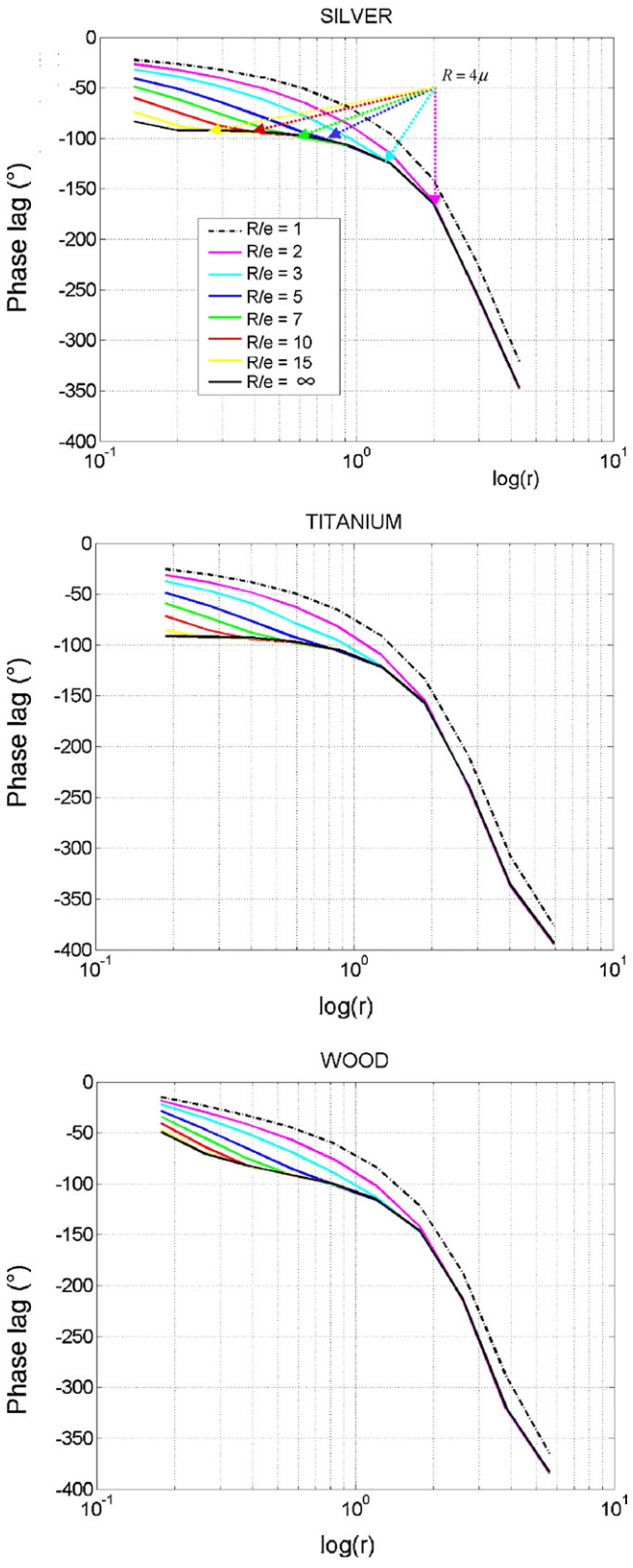

Fig. 2. 1D hypothesis limitation.

$\alpha_{titanium} \approx 9 \times 10^{-6}\,m^2\,s^{-1}$; $\alpha_{wood} \approx 0.1 \times 10^{-6}\,m^2\,s^{-1}$). Let us consider a given thickness $e = 10^{-3}$ m and according to each material, the excitation frequency is scanning such that $0.5 \leq r = e/\mu \leq 5$. Then, phase lag is computed in the middle of the lower face (0,0,e) for several heat source radius R. Geometrical hypothesis required

for 1D model validation is satisfied while $R \to \infty$. As it is shown on Fig. 2, for all studied materials a 1D model is relevant only for high value of $r = e/\mu$ (i.e. small thermal diffusion length μ, high frequency f). It is quite important to notice that if $R \approx e$ or $R \leq 4\mu$, analytical 1D solution is erroneous and can not be considered for thermal diffusivity identification. In fact, for $R \geq 4\mu$, relative error is inferior to 2% while for $R \ll 4\mu$, relative error can be greater than 65%.

3.2. 2D axis symmetry hypothesis

The studied configuration has been devoted to thermal diffusivity identification of multi-layered material. An experimental bench has been developed (see Section 5.2) for the identification of a thick carbonaceous foam located on a thin steel plate. In this section, we are mainly focused on the boundaries effect that can affect the thermal diffusivity computation based on a 2D axis symmetry hypothesis. In such an aim, the geometry is similar to that exposed in Fig. 1. Let us consider a square steel plate (thickness $e = 2 \times 10^{-3}$ m, square side length is denoted by L). The heat flux spatial distribution on the upper face ($x = 0$) is assumed to be described by: $\phi_0(X) = \phi_{\max} e^{-\left((y^2+z^2)/r_0^2\right)}$ where $r_0 = 10^{-2}$ m. It is obvious that if $r_0 \ll L$, the 2D axis symmetry hypothesis is satisfied. In Fig. 3, phase lag are computed on the lower face for two sensor locations: $y = 0$ and $y = 10^{-2}$ m. Frequency excitation is 0.01 Hz, thermal diffusion length is about 2.65×10^{-2} m. As it is shown on Fig. 3, in the specific studied configuration, for $L/2 < 3r_0$, phase lag deduced from a mathematical model based on 2D axis symmetry hypothesis are erroneous. In fact, for $L > 6r_0$, relative error is inferior to 2% while for example, for $L > 2r_0$, relative error is greater than 68%. Boundaries effects are shown in a 3D geometry (square side length $L = 2 \times 10^{-2}$ m) in Fig. 4.

In the following section, sensitivity analysis is performed for several usual situations encountered in the context of thermal diffusivity identification based on photothermal periodic method.

4. Sensitivity analysis

A sensitivity study of an observable S on the model parameters $\beta = [\beta_1, \beta_2, \ldots, \beta_n]$ allows either to reduce the forward model or to discuss the possibility of accurate physical parameters identification [13]. Sensitivity functions are defined as the absolute variation of the observable induced by an absolute variation of the considered parameter. In order to compare these coefficients with each others, the reduced sensitivity functions of S versus parameter β are defined by the following relation: $F^*_{\beta_i} = \beta_i(\partial S(\beta)/\partial\beta_i)$. It is obvious that for parameters β_j which have to be identified, $|F^*_{\beta_j}|$ have to be greater than $|F^*_{\beta_k}|$ where β_k are nuisance parameters (unknown or non-controlled parameters). In the studied framework, a reduced sensitivity study of the modulus ($S = |\theta_\omega|$) and of the phase lag ($S = \varphi = \arg(\theta_\omega)$) on model parameters is investigated in situation. This sensitivity study has been performed for the unknown parameters but also for the known parameters (which are *a priori* known with given uncertainties). We are mainly interested in situations where analytical approaches cannot be implemented. Experimental situations encountered in our institutes are exposed in the following.

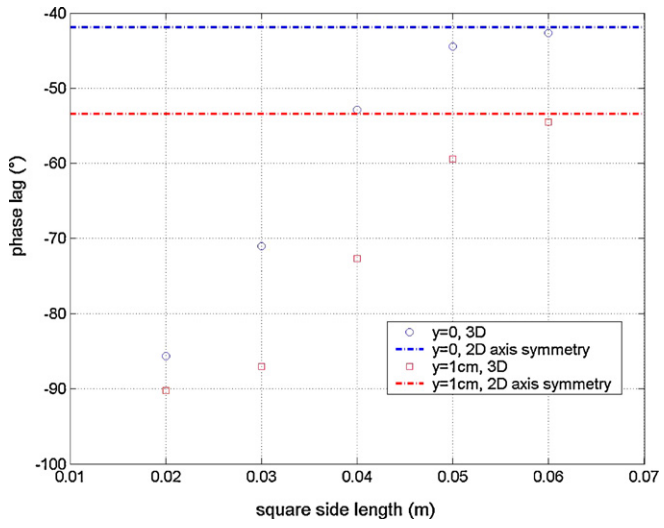

Fig. 3. 2D axis symmetry hypothesis limitation.

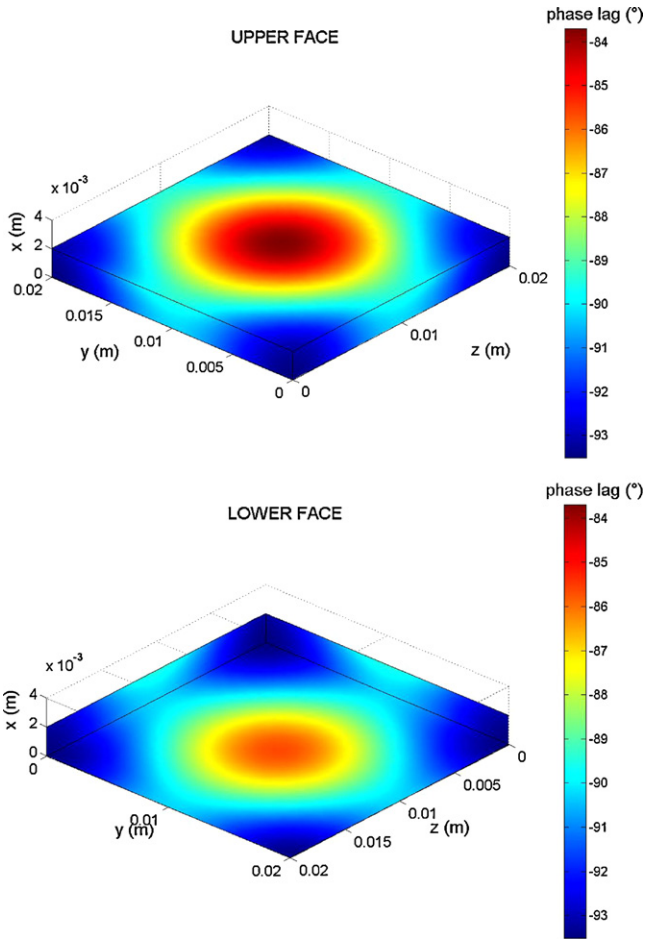

Fig. 4. Phase lag distribution on the thin steel plate.

4.1. Transmission

Let us consider a multi-layered system which consists in two glass square plates which are glued by an adhesive layer. The studied situation aims to detect the modification of the glue properties due to a thermal cycle (for optical assembly in spatial conditions, for example). Let us consider that each of the three layers is $r_0 = 10^{-3}$ m thick and that the square size is $L = 2 \times 10^{-2}$ m. The studied configuration is similar to that represented in Fig. 1. The heat flux spatial distribution is assumed to be uniform $\phi_0 = 10^3$ W m^{-2} on a disk (radius $R = 2 \times 10^{-3}$ m). Then, for a given excitation frequency $f = 0.01$ Hz, spatial distribution of both observables ($S = |\theta_\omega|$) and of the phase lag ($S = \varphi = \arg(\theta_\omega)$) on the non-heated face are considered

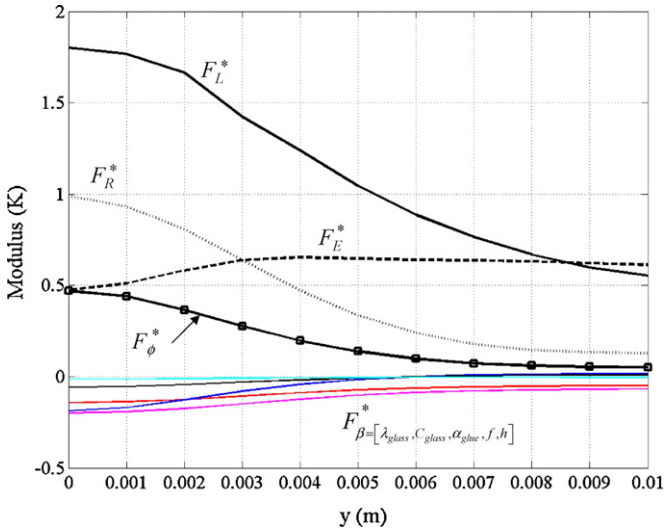

Fig. 5. Reduced sensitivity functions for modulus in transmission.

in order to identify α_{glue}. Model parameters are

$$\beta = [\lambda_{glass}, C_{glass}, \alpha_{glue}, f, \phi_0, h, R, e, L] \quad (8)$$

In order to perform sensitivity analysis the following parameters are considered:

$$\lambda_{glass} = 1\,W\,m^{-1}\,K^{-1}; \quad C_{glass} = 2.3 \times 10^6\,J\,m^{-3}\,K^{-1}$$

$$\alpha_{glue} = 5.55 \times 10^{-7}\,m^2\,s^{-1}; \quad h = 10\,W\,m^{-2}\,K^{-1}$$

Reduced sensitivity functions are evaluated as the ratio between a finite variation of S and a finite variation of beta (10%):

$$F^*_{\beta_i} = \beta_i \frac{\partial S(\beta)}{\partial \beta_i} = \beta_i \frac{S(1.1\beta_i) - S(\beta_i)}{0.1\beta_i} = 10(S(1.1\beta_i) - S(\beta_i))$$

Sensitivity functions for modulus are presented in Fig. 5 and for phase lag in Fig. 6. It is shown (Fig. 5) that the thermal wave amplitude mainly depends on the sample geometry (square size L, thickness $3e$), on the heating source radius (R) and on the amplitude of the harmonic heat flux fluctuation ϕ_0. For practical reasons, it can be quite difficult to accurately know ϕ_0. Thus, thermal diffusivity (α_{glue}) identification can not be performed considering modulus observations. On Fig. 6, sensitivity on geometrical parameters (square size L, thickness $3e$) is quite important and it is essential to accurately known these parameters. Excitation frequency (f) is also a key parameter which has to be carefully controlled. It is shown that:

- phase lag does not depend on amplitude of the harmonic heat flux fluctuation ϕ_0,
- uncertainty on heating source radius R is not influent for $y > R$,
- glass properties λ_{glass} and C_{glass} have to be accurately known (in fact, only the ratio $\lambda_{glass}/C_{glass} = \alpha_{glass}$ is influent),
- convective exchange (parameter h) can be neglected. However for lower frequency, this parameter is influent and can be considered as a nuisance parameter.

4.2. Reflection

In this section, thermal diffusivity in a micrometric domain is investigated considering reflection (both excitation and observation are performed on the same surface). Let us consider a micrometric cylindrical fibre (radius $r = 10^{-5}$ m) in a matrix (cubic

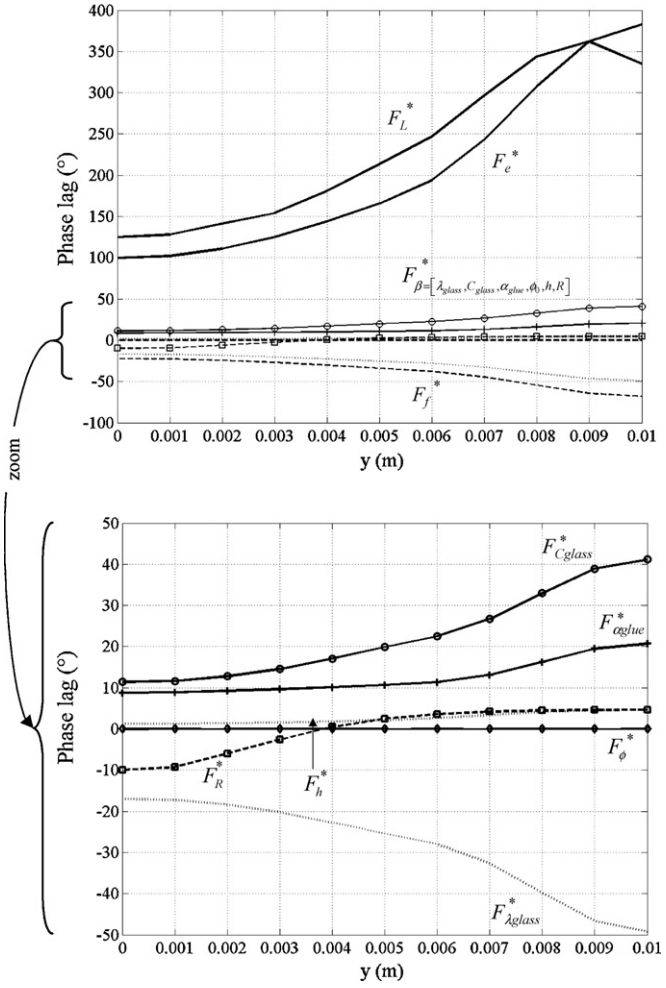

Fig. 6. Reduced sensitivity functions for phase lag in transmission.

size $L = 5r$). Geometrical domain is presented in Fig. 7. Periodic excitation on the upper face is located on the centre of the fibre: heat flux spatial distribution is assumed to be described by: $\phi_0(X) = \phi_{max}\, e^{-\left((y^2+z^2)/r_0^2\right)}$ where $r_0 = 2 \times 10^{-6}$ m. Let us consider that phase lag is measured at a fixed position in the fibre: $y_{obs} = 5 \times 10^{-6}$ m (distance between the fibre centre and the sensor location) for a given frequency scanning $f \in [10^3; 10^5]$ Hz. Thermal wave modulus

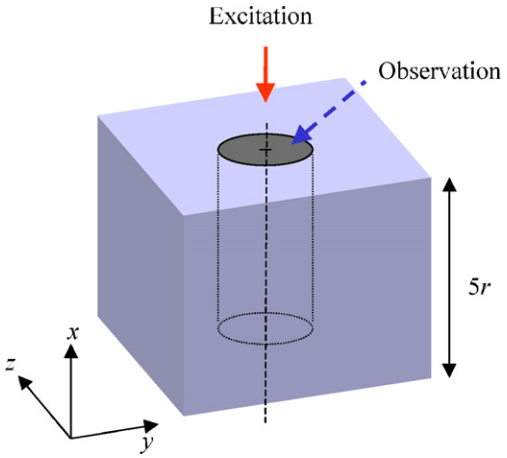

Fig. 7. Studied micrometric geometry for sensitivity in reflection configuration.

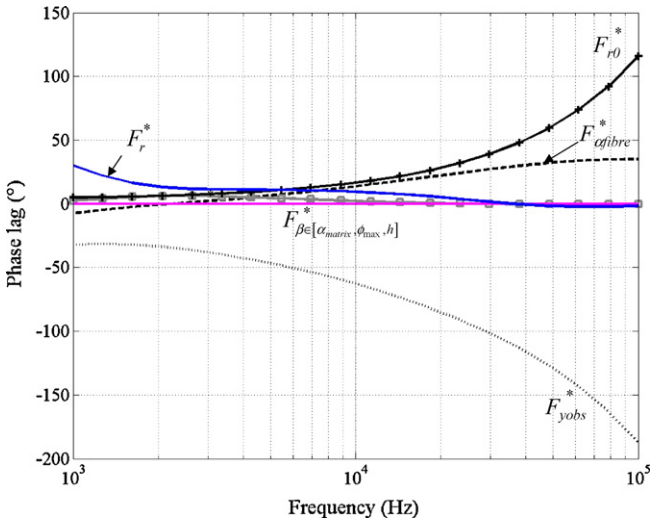

Fig. 8. Reduced sensitivity functions for phase lag in reflection.

is not considered since it depends on ϕ_{max} which is quite difficult to accurately characterise. We are mainly interested in fibre thermal diffusivity identification. Model parameters are

$$\beta = [\alpha_{fibre}, \alpha_{matrix}, \phi_{max}, h, r_0, r, y_{obs}] \quad (9)$$

$$\alpha_{fibre} = 10^{-6}\,m^2\,s^{-1}; \quad \alpha_{matrix} = 20 \times 10^{-6}\,m^2\,s^{-1};$$
$$\phi_{max} = 10^6\,W\,m^{-2}; \quad h = 10\,W\,m^{-2}\,K^{-1}$$

Sensitivity functions for phase lag observations are presented in Fig. 8. It is shown that:

- phase lag does not depend on heating flux ϕ_{max}, on convective exchanges h (for high frequency, h is not a nuisance parameter), and on matrix thermal diffusivity α_{matrix},
- for $f < 10^4$ Hz, α_{fibre} can not be identified: phase lag depends on numerous parameters and reduced sensitivity function $F^*_{\alpha_{fibre}}$ is not significant enough,
- for $f > 10^4$ Hz, α_{fibre} can be identified from phase lag observations if both the heating flux spatial distribution (r_0) and the measurement location (y_{obs}) are accurately known,
- fibre radius (r) is not an influent parameter for $f > 10^4$ Hz.

Such a brief sensitivity analysis (Sections 4.1 and 4.2) is a crucial preliminary step and can provide a relevant observation strategy: which observable has to be chosen? which key parameter has to be accurately known? which nuisance parameters avoid the identification procedure? which experimental parameters have to be controlled? Considering periodic analysis, finite element method for complex temperature calculation and sensitivity analysis, several experimental devices are developed in the DGA-CEP-EHF institute.

5. Experimental devices for diagnosis and application examples

In the following, several experimental benches are detailed in order to present configurations in transmission and reflection and to investigate micrometric and millimetric scales.

5.1. Characterisation of orthotropic materials

This experimental bench is mainly devoted to transmission analysis for millimetric samples [3]; it comprises three main parts (see Fig. 9):

- The sample part where the tested material is located in the focal plane of a Köhler optical device.
- 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 upper face of the sample.
- The measurement part made up by an Infra Red camera located on the lower face of the sample linked to a signal processing.

The spatial distribution of the temperature on the sample recorded by the IR camera is representative of the material's structure (isotropic for circular distribution, orthotropic for ellipsoidal distribution...). 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. 512 pictures are recorded in order to measure more than 10 periods. A lock-in algorithm is implemented to determine at each point of the front face the phase lag with temperature evolution in the

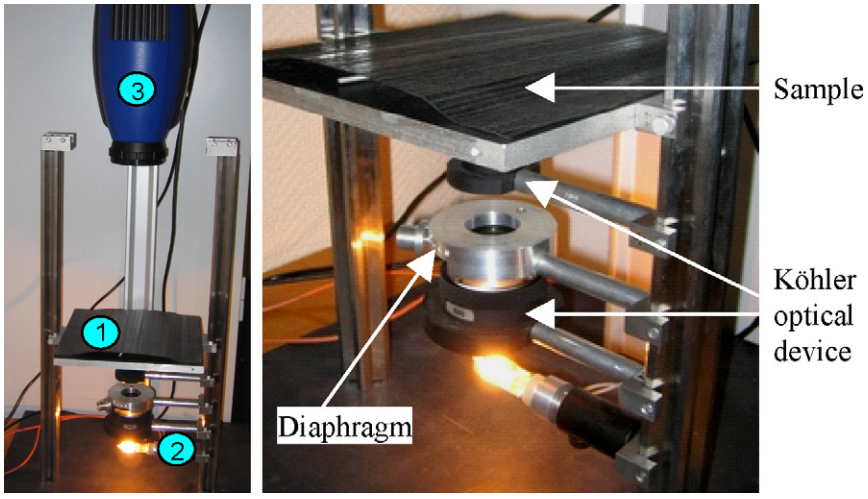

Fig. 9. Experimental device for millimetric analysis (transmission). (1) sample, (2) photo-thermal excitation, and (3) infrared camera.

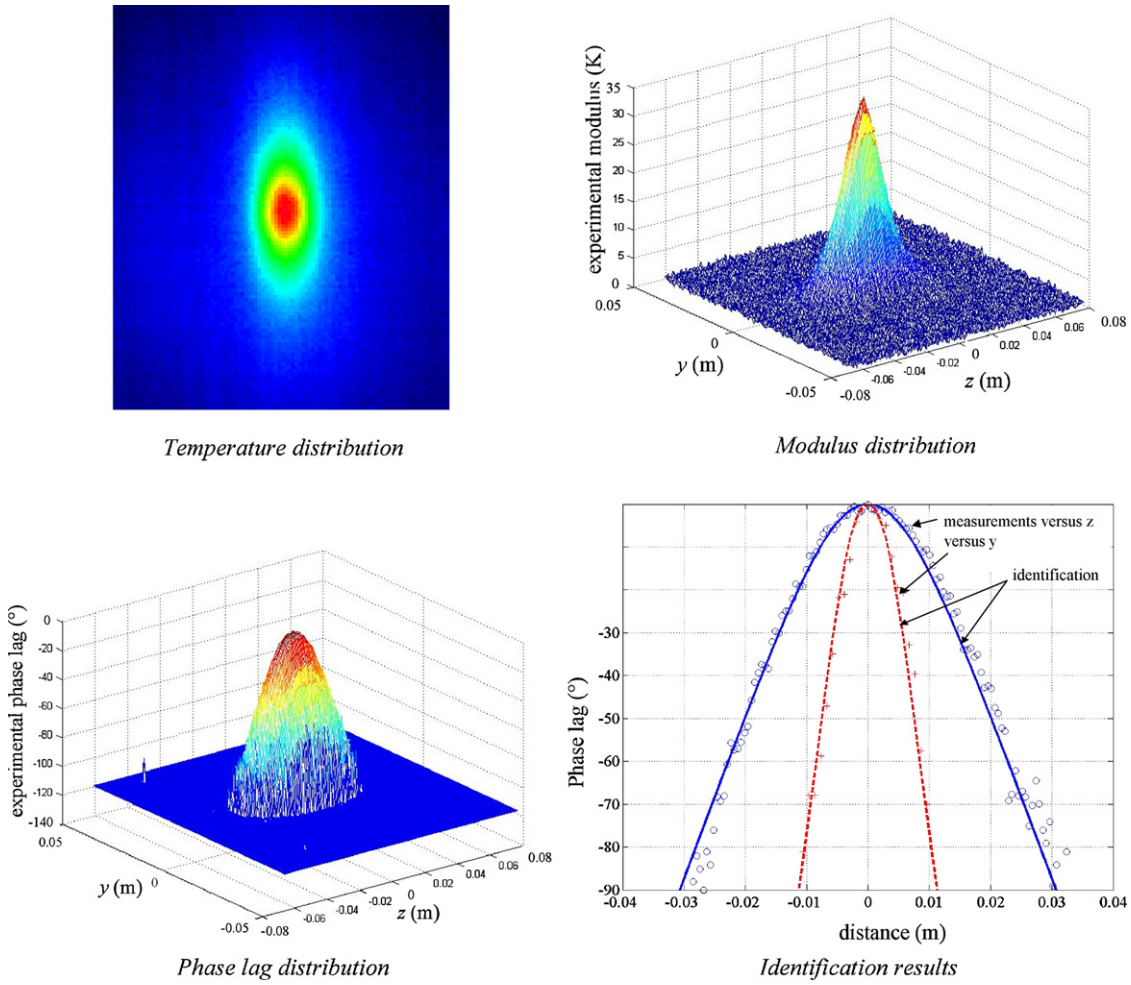

Fig. 10. Experimental results (millimetric analysis – transmission).

middle of this non-heated face. A previous sensitivity analysis (see Section 4.1) has shown that even if phase lag and modulus are measured, only phase lag observations lead to a correct identification (since diffusivity obtained from modulus is correlated to nuisance parameters). However, modulus cartographies are taken into account in order to determine the low level of attenuation for which output signal is not significant enough (see Fig. 10 for an example of temperature, modulus and phase lag distributions obtained for orthotropic materials). The correct convergence of the algorithm is presented on Fig. 10 and it has been estimated that $\alpha_z \approx 10\alpha_y$ and $\alpha_x \approx \alpha_z$. This result is characteristic of fibre stack in a reinforced matrix. Thus, this device is well adapted to the characterisation of orthotropic materials global behaviour (fibre-reinforced or woven composites).

5.2. Main solar furnace for insulating carbonaceous layer characterisation

In this section, periodic excitations are generated using the main solar furnace built in DGA-CEP-EHF institute [4]. This original device is composed with three main elements (see Fig. 11):

- a 230-m^2 plane reflector (heliostat) composed with 638 reflecting mirrors whose function is to reflect solar radiation in a horizontal north–south direction (to the attenuator and the concentrator),
- a solar radiation modulator mainly based on a flux attenuator consisting of a group of fast-moving 20 shutters. Depending on their orientation, processed by a microcomputer, it determines the solar flux which can reach the sample placed in the experiment chamber.
- a 10.75-m focal length concentrator covering an area of 100 m^2 which gives a focal zone energy distribution that is quasi constant over a diameter of 50 mm.

Sunbeams have to be directed towards the concentrator. In such an aim, mirrors are positioned in order to obtain the sun image accurately located in the heliostat centre. This crucial requirement is performed thanks to the observation of the solar distribution with a 4-quadrant measurement device pointed at the heliostat centre. Then, in order to ensure that the same flux is obtained in each quadrant, a PID (Proportional Integral Derivative) controller is implemented in order to shift the mirror according to the sun trajectory. A fine control is essential to ensure the radiative flux quality especially for long duration experiments (several hours). In order to obtain time dependent heat flux $\phi(t)$, the attenuator is used as a fast shutter. The concentrated flux generated on the target is measured, then the shutter opening (see Fig. 11) is adjusted in order to satisfy the desired heating flux $\phi(t)$. Opening angle is adjusted in the range [0,90°] in order to deliver up to 100% of the concentrated flux. Due to the weak mass of each shutter, the flux can be delivered from 0 to 100% in a very small time: 0.01 s. Then, quite accurate

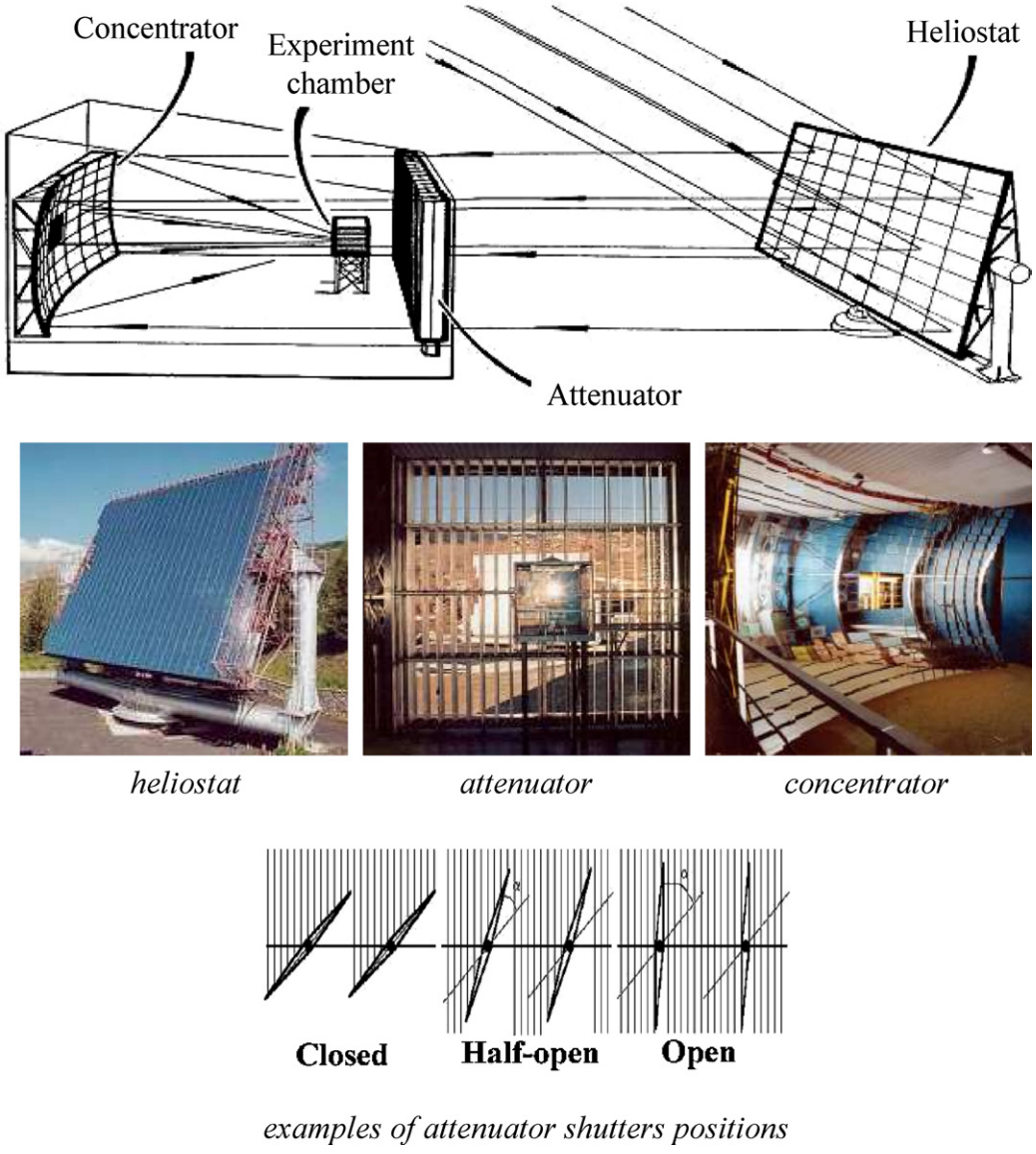

Fig. 11. Main solar furnace.

thermal aggressions can be provided by the main solar furnace: periodic excitation (tests have been performed for heating cycle with 1 Hz frequency), heating pulse of short duration (for nuclear heating waves).

In this section, results are shown for thermal diffusivity identification of a thick porous layer developed on a thin steel plate. Thus, only two layers are considered: the steel substrate (which thermal properties are well known) and the unknown layer. For the identification of the insulate layer thermal diffusivity, since this insulating layer is thick, excitation frequency has to be small enough in order to obtain a relevant thermal diffusion length. Thus, period is greater than 40 s in order to obtain a reliable ratio signal/noise on the external substrate face (steel plate). However, it is quite difficult to consider period greater than 360 s due to the solar flux available during experimentation. In fact, it is usual to take into account up to 50 periods for the steady state and experiments based on solar flux cannot exceed 5 h. Results have been obtained for a 2-mm thick steel plate coated by a 20-mm thick layer. Then, periodic excitation is performed using the main solar furnace and phase lag between heating flux and thermocouple responses (on the steel

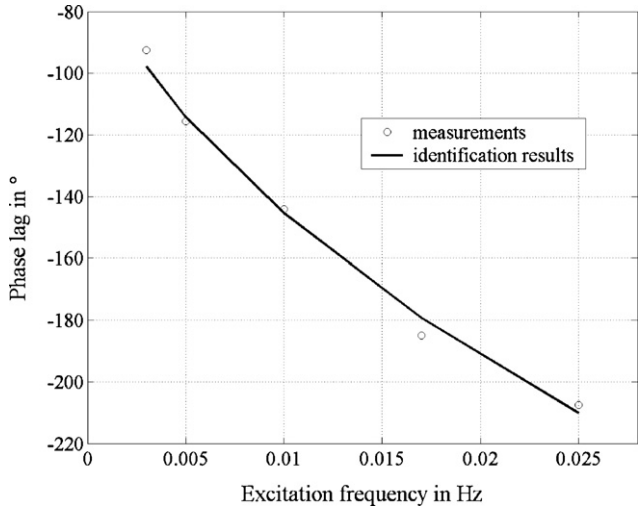

Fig. 12. Identification results for carbonaceous layer.

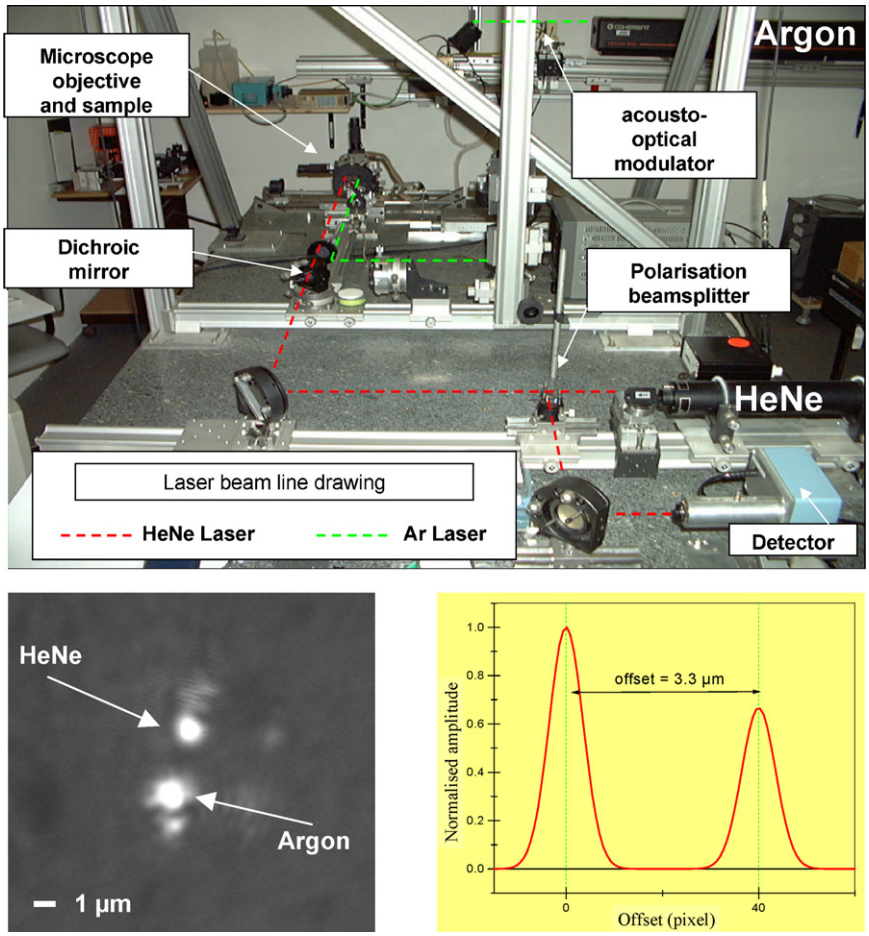

Fig. 13. Photothermal microscope.

plate) are measured. Results are given in Fig. 12. The minimisation algorithm implemented for thermal diffusivity identification leads to the following value: $3.9 \times 10^{-6}\ m^2\ s^{-1}$.

5.3. Photothermal microscope

The experimental device used for obtaining measurements able to characterise the microscale thermal behaviour of heterogeneous materials is a versatile photo-thermal microscope [5]. The measurement technique is based on the sample thermal response when it is submitted to a microscale periodic thermal excitation (see Fig. 13). A modulated laser beam (pump), focused by a microscope objective onto the sample surface, produces a periodic local thermal excitation (≈1 μm diameter spot). At a given distance (≈5 μm), a continuous laser beam (probe) is used to detect the thermal wave propagation by observing the variations of the surface reflectance that depends on temperature. The heat flux provided by the pump is chosen such as the relation between the reflectance and the temperature is linear. The thermal excitation is achieved by an ion-argon laser (COHERENT, Innova 305) the 514 nm waveband of which being selected. An acousto-optic modulator (ISOMET 1211) driven by a computer-programmable function generator and a RF amplifier modulates the beam at the desired frequency. After shape setting, the beam is reflected by a dichroic plate and focused by a microscope objective (50×, MITUTUYO) on a gaussian microscale spot at the sample surface. The 632 nm measurement beam, originated from a He-Ne laser (ORIEL 79200) crosses a polarisation beamsplitter and the dichroic plate, then is directed to the same objective which focuses it close to the heating spot. The distance between spots (called offset) is accurately adjusted by means of wedge prisms rotation. The reflected part is sent back to the polarisation beamsplitter which reflects it towards a fast response photodiode. The photodiode component signal is amplified and analysed by a wide bandwidth lock-in device (EGG 5302), the reference signal of which comes from the acousto-optic controller. The lock-in amplifier output (amplitude and phase lag) is finally recorded by the control computer. The phase lag between reflectance variations and heating laser modulation corresponds to the thermal diffusion process between excitation (pump) and observation (probe) spots. The unknown thermophysical properties are thus microscale characteristics of the investigated zone. These properties are then identified by analyzing the evolution of the phase lag versus an adjustable parameter (independent variable) such as excitation frequency, distance between spots or distance from a thermal discontinuity. This device is well adapted to the characterisation of heterogeneous materials in a micrometric scale and provides valuation for local behaviour. In Table 1, results of the validation campaign are presented for several metallic samples.

Table 1
Errors for reference samples.

Metal (purity > 99.9%)	α_{hit} ($mm^2\ s^{-1}$) [literature]	α_{meas} ($mm^2\ s^{-1}$) [measured]	$\left\lvert\frac{1-\alpha_{hit}}{\alpha_{meas}}\right\rvert$ (%)
Titanium	9.09	9	1
Rhenium	16.43	14	15
Platinum	25.46	29	14
Gold	125.75	112	11
Silver	172.98	191	10

6. Conclusions

In this study, the use of periodic thermal excitation of material samples to evaluate specific properties like thermal diffusivity, etc. has been investigated. A numerical approach is used to find the limit of some analytical solutions and to perform a sensitivity analysis of the method. This numerical approach is based on a finite element method in order to compute the complex temperature distribution in the material exposed to a periodic radiative excitation. Thus, in several situation, results are presented for both modulus and phase lag computation. It is shown that modulus analysis cannot be performed for thermal diffusivity identification if the heat source amplitude is not accurately known. Then, for phase lag analysis, the limit of some analytical solutions or semi-analytical solution are investigated in order to provide relevant informations for experiment design. Several experimental devices which have been developed according to this experiment design have been presented and some experimental application of the method are shown.

Several outlooks can be considered:

- An important aspect of the presented methodology lies in the multi-scale analysis allowed by the use of periodic methods for diagnosis. In fact, various spatial scales occur either considering the materials, or considering the resources implemented for the thermal characterisation: they extend from micrometric scale to millimetric scale. According to the studied materials, several geometrical scales can be studied (adjusting excitation frequencies) what naturally leads to take an interest in relations between the local scale behaviour (a few micrometers) and the global scale behaviour (a few millimetres). So, this approach allows on one hand to better understand the homogenisation by quantifying at best the microstructures morphology and, on the other hand, to improve material alteration understanding. The presented benches will also contribute to investigate structures lifetime optimisation. Multi-scale diagnosis development will improve the comprehension about the relation between local properties (for composite materials or multi-layered structures) and global behaviour (properties or damages).
- The determination of reliable confidence or uncertainty intervals for the parameters identified via periodic methods is a further essential stage to bring a guarantee to the diagnoses. It seems to be attractive to investigate the use of new interval complex arithmetic within the set inversion and set projection algorithms, in order to derive guaranteed diagnosis results in an efficient way. We also plan to carry on methodological developments in order to solve set membership identification in the time domain when the model involves non-linear differential equations.

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Biographies

Laurent Autrique received a postgraduate degree (DEA) in process control from the Ecole Centrale (Nantes, France) in 1992, and a PhD degree from the same institute in 1995. He was a research scientist for the PROMES Research Institute (C.N.R.S. Perpignan, France) until 2002. Then he was from 2002 with 2007 in the E.H.F. Department (Expertise Hauts Flux), D.G.A. (Weaponry Department of French Ministry of Defence) Font Romeu, (France). He is currently professor in LISA (ISTIA, University of Angers, France). His research works are devoted to parametric identification, inverse problems, and process analysis. Main publications are focused on non linear partial differential equations system describing state evolution of complex thermal processes.

Laetitia Perez received her postgraduate degree (DEA) in process engineering from the University of Perpignan (France) in 2000 and her PhD degree from the Ecole Nationale Supérieure des Arts et Métiers (ENSAM – France) in 2003. She has from 2004 with 2006 a temporary research monitoring position in the E.H.F Department (Expertise Hauts Flux), D.G.A. (Weaponry Department of French Ministry of Defence) Font Romeu (France). In 2006, she joined the Thermocinetic Laboratory of Nantes where she is currently an associate professor. Her research interests are in the modelling of thermal process, the experimental benches development and the resolution of inverse heat conduction problems.

Emmanuel Scheer is 38 years old. He is employed by the DGA (Délégation Générale pour l'Armement), French Ministry of Defense for 15 years as a superior technician. Also he is incharge of tests and measurements at the solar furnace facility and on thermal diffusivity test benches in Font-Romeu. He has got superior technician degree (Optical Instruments and Precision) from Victor Bérard High School (Morez) and one year specialization in opto-electronics from Fresnel High School (Paris). He has completed production engineering and laser metallurgy training from IREPA Laser (Strasbourg). He has masters in industrial systems engineering from the University of Perpignan.