MICROSCALE THERMAL CHARACTERIZATION BY INVERSE METHOD IN THE FREQUENCY DOMAIN

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Abstract: In this communication, several aspects of the implementation of a finite element method for the resolution of inverse problems (occurring in parameter identification) are exposed. Identification of a thermal parameter (diffusivity) at micrometric scale is obtained from observations of output signal due to a periodic input. Resolution of this identification problem is facilitated by the formulation of a complex temperature in the Fourier space and a finite element analysis.

Keywords: Parameter identification, Thermal diffusivity, Frequency-response methods, Phase lag, Partial differential equations, Finite element method.

1. INTRODUCTION

The development of new methods capable of characterizing the micro scale thermal behavior of heterogeneous complex materials is a crucial step for optimizing elaboration process. In such a way, a thermal diffusivity measuring device has been developed. The experimental bench is based upon a periodic method dedicated to microscopic study. The principle consists in heating a sample by a modulated laser beam on one face and to measure a temperature dependent coefficient (reflectance) near the heating source. The temperature evolution (induced by the periodic heating input) is the sum of a continuous component and a periodic one characterised by its amplitude and its phase lag versus thermal excitation. Reflectance being temperature dependent, the phase lag between reflectance variations and heating input depends on the thermal properties of the heated volume. Thus, it leads to the identification of the thermal diffusivity of the material in a microscopic area ($\leq 20\mu m$). This experimental bench is developed in order to study at a microscopic scale :

- thermal characteristics of materials (thermal property distribution, anisotropy, …),
- thermal interfaces between multi-component materials,
- thermal discontinuities (cracks, …) .

An inverse problem has to be solved in order to identify the thermal diffusivity of the studied material. Minimization of difference between observed phase lag and simulated phase lag is performed. Simulation values are deduced from mathematical model describing heat transfer induced by periodic excitation. Semi analytical solutions are proposed in (Gervaise *et al.*, 1997) for homogeneous samples and in (Pruja *et al.*, 2004) for several types of discontinuities. In both previous communications, partial differential equation system is solved using a space Fourier transform; calculation of the inverse

Fourier transform is carried out numerically and temperature amplitude and phase lag values are compared to the incident heat flux. However, semi analytical solution validity sharply depends on strong assumptions which can be quite difficult to verify for heterogeneous materials. In fact, the distance on which the heat has propagated during a period is called thermal diffusion length $\delta = \sqrt{a/\pi f}$ where a is the thermal diffusivity and f is the modulation frequency.

Table 1. Diffusion length estimation

frequency $[Hz]$	diffusivity $[m^2.s^{-1}]$	diffusion length $[\mu m]$
$f = 10^4$	$a = 10^{-5}$	$\delta \approx 20$
$f = 10^6$	$a = 10^{-6}$	$\delta \approx 0.5$

In the studied configurations, the thermally excited volume does not exceed some μm^3. When spatial heterogenities dimension is about the thermal diffusion length, semi analytical solution are not valid. For example, identification of thermal diffusivity in micro-metric fibers whom diameter is inferior to $10\mu m$, can not be performed using inverse Fourier transform. In such a framework, a numerical solution based on a finite element method is proposed in the following (Autrique *et al.*, 2003). Then, measuring bench is exposed and experimental results are shown.

2. MODELING IN THE FREQUENCY DOMAIN

Thermal waves produced by a periodic heat generation in homogeneous and inhomogeneous solids are studied from the theoretical point of view in (Gurevich *et al.*, 2003). Application to thermal diffusivity measurement using harmonic and one-dimensional propagation of thermal waves is proposed in (Muscio *et al.*, 2004). Let us consider the following notations : $\Omega_i \in \mathbb{R}^3$ is the space domain corresponding to the component i, $X = (x, y, z) \in \bigcup \Omega_i$ is the space variable, $t \in T$ is the time variable. In (Pruja *et al.*, 2004), the periodic heating flux focused at the surface Γ on point I is expressed in the form :

$$\phi(r_X, t) = \phi_0 e^{-\frac{r_X^2}{r_0^2}} e^{j\omega t} \quad (1)$$

where ϕ_0 is the heating flux amplitude $[W.m^{-2}]$, r_X is the distance XI in $[m]$, r_0 is characteristic of the heating flux spatial distribution $[m]$, ω is the pulsation $[rad.s^{-1}]$.

Evolution of temperature $\theta(X,t)$ in $\bigcup \Omega_i$ is described by, the following equations :

$$\begin{aligned} &\forall (X,t) \in \bigcup \Omega_i \times T \\ &\Delta\theta(X,t) - \frac{1}{\alpha_i}\frac{\partial \theta(X,t)}{\partial t} = 0 \end{aligned} \quad (2)$$

where α_i is the unknown diffusivity,

$$\begin{aligned} &\forall (X,t) \in \Gamma \times T \\ &-\lambda_i \frac{\partial \theta(X,t)}{\partial \vec{n}} = \phi(r_X, t) - h\theta(X,t) \end{aligned} \quad (3)$$

where λ_i is the thermal conductivity, $\vec{n}$ is the normal vector exterior to Γ, h is the convective exchange coefficient,

$$\forall X \in \bigcup \Omega_i \qquad \theta(X,0) = 0 \quad (4)$$

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

$$\theta(X,t) = \theta_c(X) + \theta_\omega(X) e^{j\omega t} \quad (5)$$

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. Deduced from the non linear partial differential equations system (2,3,4), the following equations are considered :

$$\forall X \in \bigcup \Omega_i \qquad \Delta\theta_\omega(X) - \frac{j\omega}{\alpha_i}\theta_\omega(X) = 0 \quad (6)$$

$$\forall X \in \Gamma \qquad -\lambda_i \frac{\partial \theta_\omega(X)}{\partial \vec{n}} = \phi(r_X) - h\theta_\omega(X) \quad (7)$$

In specific configurations such as homogeneous solid, semi infinite geometry, temperature nondependent parameters, particular multi-component configurations (for which thermal interface are well identified), calculation of the inverse Fourier transform leads to semi analytical solution, see theoretical aspects and applications in (Gervaise *et al.*, 1997, 2001), (Milcent *et al.*, 1998), (Gagliano *et al.*, 2001), (Pruja *et al.*, 2004). From the experimental point of view, for heterogeneous materials which 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 equations $(6,7)$, finite element method is implemented. Complex temperature $\theta_\omega(X)$ is separated in real part and imaginary part :

$\theta_\omega(X)=\theta_{\mathrm{Re}}(\omega,X)+j\theta_{\mathrm{Im}}(\omega,X)$. Then, the following coupled systems have to be solved :

$$\begin{cases} \Delta\theta_{\mathrm{Re}}(\omega,X) = -\dfrac{\omega}{\alpha_i}\theta_{\mathrm{Im}}(\omega,X) & \forall X \in \bigcup\Omega_i \\ -\lambda_i \dfrac{\partial\theta_{\mathrm{Re}}(\omega,X)}{\partial\vec{n}} = \phi(r_X) - h\theta_{\mathrm{Re}}(\omega,X) & \forall X \in \Gamma \end{cases}$$

$$\begin{cases} \Delta\theta_{\mathrm{Im}}(\omega,X) = \dfrac{\omega}{\alpha_i}\theta_{\mathrm{Re}}(\omega,X) & \forall X \in \bigcup\Omega_i \\ -\lambda_i \dfrac{\partial\theta_{\mathrm{Im}}(\omega,X)}{\partial\vec{n}} = -h\theta_{\mathrm{Im}}(\omega,X) & \forall X \in \Gamma \end{cases}$$

An iterative procedure is carried out in order to solve previous systems.

3. SIMULATION AND NUMERICAL RESULT

In the following, micrometer fibers are investigated; transversal and radial thermal diffusivity have to be identified. Several geometrical configurations are considered (see figures 1 and 2).

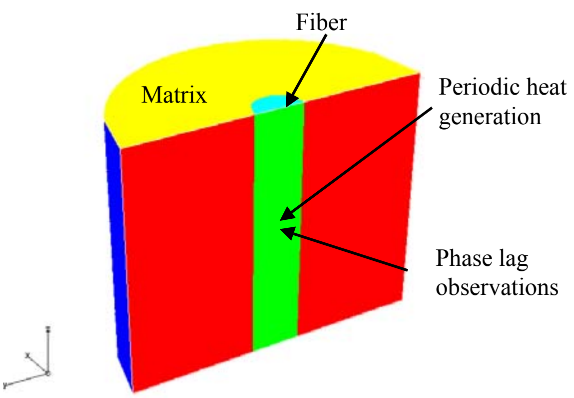

Fig. 1. Geometrical configuration for transversal identification

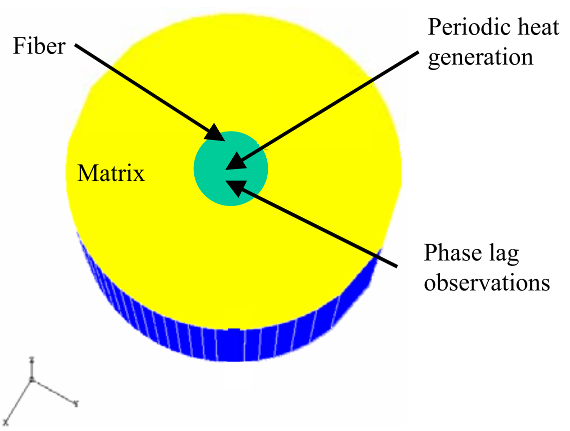

Fig. 2. Geometrical configuration for radial identification

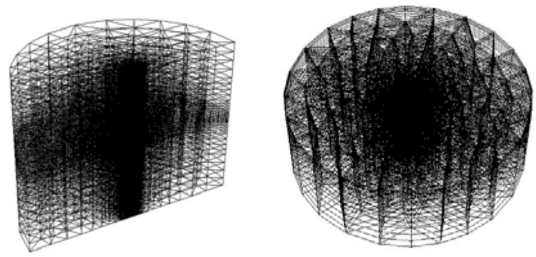

Fig. 3. Finite element meshes.

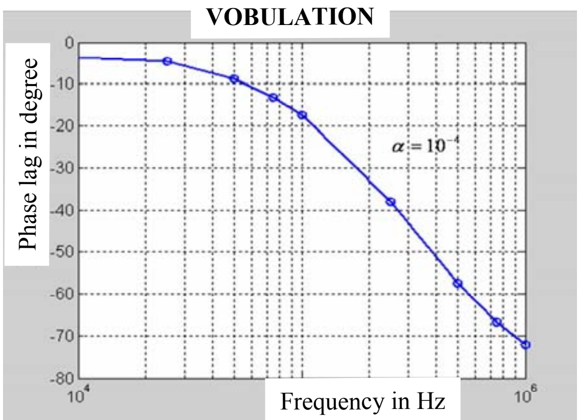

Fig. 4. Example of simulation results obtained according to the finite element method

Corresponding meshes are shown on figure 3. Fibers diameter is about $8\mu m$. Meshes are adapted in the neighborhood of point I where the periodic heating flux is focused at the surface Γ and near the phase lag observation point.

Systems are solved according to the iterative procedure exposed in paragraph 2. According to the studied configuration, phase lag is determined and an example of relevant result is shown on figure 4 for a given fiber diffusivity.

In the following paragraph, microscopic measurement bench is exposed.

4. MEASURING BENCH

The experimental device used for obtaining measurements able to characterize the micro-scale thermal behavior of heterogeneous materials is a versatile photo-thermal microscope. Although the principle of such device is well-known since Rosencwaig's works (Rosencwaig *et al.*, 1986) it will be reminded in order to point out its mains advantages and drawbacks.

4.1 Description

The measurement technique is based on the sample thermal response when it is submitted to a micro-scale periodic thermal excitation. A modulated laser beam (pump), focused by a microscope objective onto the sample surface, produces a local thermal excitation (≈ 1 µm diameter spot). At a given distance (≈ 4-5 µm), a continuous laser beam (probe) is used to detect the thermal wave diffusion by observing the variations of the surface reflectance that depends on temperature; (Milcent *et al.*, 1998) (Gervaise *et al.*, 2001), (Dilhaire *et al.*, 2004).

In our experiment (see figure 5), the thermal excitation is delivered 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 (x50, MITUTUYO) on a gaussian micro-scale spot at the sample surface. The 632 nm measurement beam, originated from a He-Ne laser (ORIEL 79200) crosses a polarization 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 polarization beamsplitter which reflects it towards a fast response photodiode. The photodiode AC component signal is amplified and analyzed 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.

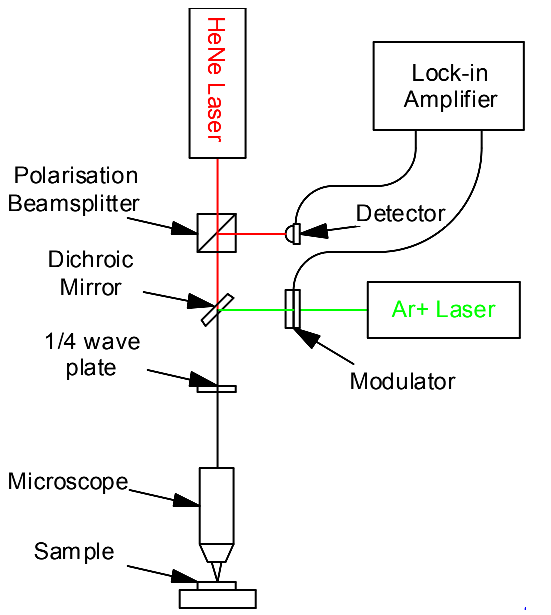

Fig. 5. Schematic drawing of the photothermal microscope.

The unknown thermal properties are thus micro-scale 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.

4.2 Calibration procedure

The experimental system (optical and electronic devices) introduces an additional phase shift that should be subtracted from the measured value to keep only the thermal contribution. For this, a calibration procedure consists in picking up with an optical fiber a small part of the pump beam and sending it directly to the detector. The resulting phase, measured on the whole frequency range, is stored in a table that will be used to correct the set of experimental values obtained on the samples.

Because of the short distance between pump and probe spots, their shape and size are to be taken into account. Although commercially available beam analyzers did allow measurements of micro-scale beams, they are not adapted for analyzing beams focused by such high numerical aperture objectives. The spots characteristics are investigated by a two-step procedure (Gagliano *et al.*, 2001). A scanning slit beam profiler (DATA RAY Beamscope P5) capable of measuring focused beam profiles of some tens of microns is used to analyze the beam shape in several locations upstream and downstream the waist. This step allows to verify that the beams are gaussians and to determine the beam quality coefficient (M^2). It become then possible to extrapolate the value of the waist diameter by applying the gaussian beam propagation law.

Analysis performed on the system equipped with the specified objective (x50, MITUTUYO) give the following results :

- pump spot diameter: 1.00 ± 0.04 µm,
- probe spot diameter: 1.24 ± 0.04 µm.

4.3 Sample holder and positioning

The sample is held by a two-stage micro-positioning system (0.1 µm resolution) driven by the computer. This allows 1-D scanning of the sample surface that will be used for thermal parameter estimation, or 2-D scanning for imaging the map of surface thermal transfers. Heterogeneous samples often comprises materials of different hardness and so the polishing results in surface altitude variations of some microns. Because of the low depth-of-field, these variations have to be corrected. The sample holder involves a third movement in z-direction so that to maximize the probe laser reflected beam.

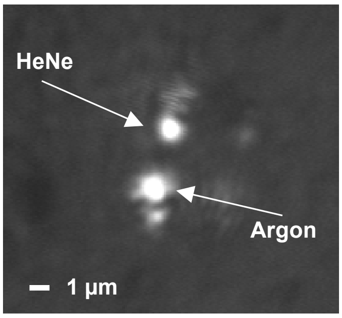

Fig. 6. Example of surface image.

A CCD camera sights the surface by means of a beamsplitter. Its aim is an accurate positioning of both spots on the sample surface as well as a measurement of distance between spots. (Note: disturbing reflections observed on figure 6 come from color filters and not from the surface). The various illumination levels (pump and probe spots, surface lighting) are very different and so a raw image would be saturated and unusable. In order to balance them, the surface is lightened by a pulsed 932 nm diode and the composite beam reflected by the surface passes trough color filters attenuating the 514 nm and 632 nm wavebands.

5. EXPERIMENTAL RESULTS

The measuring device has been used for several fiber configurations (see figures 7 and 8).

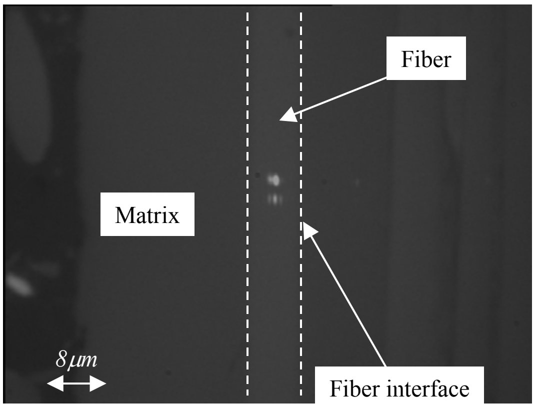

Fig. 7. Measurements for transversal identification

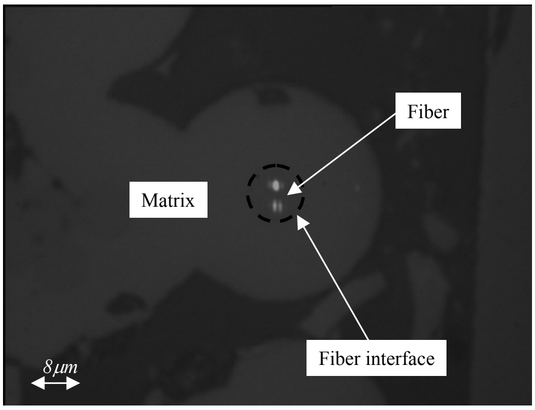

Fig. 8. Measurements for radial identification

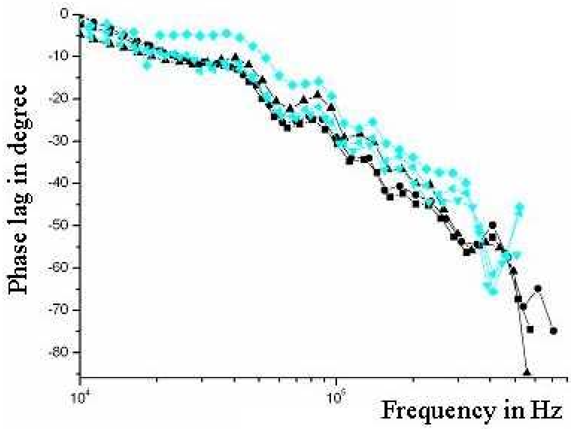

Fig. 9. Example of phase lag measurements

Examples of phase lag measurements are shown on figure 9. Comparisons between simulation on figure 4 and observations on figure 9 seems to ensure that :

- thermal model described by coupled systems in order to determine real part and imaginary part is well adapted to phase lag estimation,
- experimental bench leads to phase lag measurements in micro scale heterogeneous materials.

An identification procedure has to be developed in order to estimate thermal diffusivity by minimizing the difference between simulated phase lag (figure 4) and measured phase lag (figure 9). For the resolution of such an inverse problem, one can refer to (Beck *et al.*, 1977), (Alifanov, 1994) and (Walter *et al.*, 1997).

6. CONCLUDING REMARKS

In this communication the interest of a finite element approach in parametric identification is presented. An experimental bench (dedicated to microscopic scale study) is developed for the identification of thermal diffusivity in heterogeneous material. To estimate this property a model is presented and resolution by a finite element method is proposed in order to improve the identification when semi analytical solutions can not be considered.

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