Thermal diffusivity measurement by photothermal radiometry under random excitation and parametric analysis

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Abstract. The aim of this work is to approach in an experimental way, the possibilities of diffusivity thermal measurement, under less energy constraints, offered by front face random photothermal radiometry associated to a parametric analysis. First, we present the principle of the random method. Then, we present the experimental device SAMMIR used in our study. In a third stage, we present the studied sample, the experimental conditions selected and the model developed for the study. We show finally, using the experimental study of a sample of nylon 6.6 that the photothermal method allows, in a particular case, a good approximation of the thermal diffusivity parameter.

1. Introduction
Infra-red photothermal radiometry is a non destructive method operating at a distance and without contact. Its principle consists in subjecting the sample to be analyzed with a luminous flow which absorption produces a local rise in temperature close to the laser impact, then to observe the variations of spectral lighting of material using a chain of infra-red optical detection. The photothermal signal thus obtained depends on the optical and thermal properties of analyzed material, which then makes it possible to characterize them. Traditionally, to take measurements of thermal diffusivity, an impulse excitation combined with a continuous detection is implemented (flash method, [1]). This method, very powerful, presents however the obligation, to obtain an excitation close to a Dirac function, to deposit an important energy in a very short time, which does not authorize, for example, the analysis of delicate material (biological materials, works of art). To allow the study of these last types of materials and thus to extend the field of investigation of the photothermal radiometry, new studies and new types of analysis are still to develop. For a few years, thanks to the computerization of the instrumentations and with the development of the signal treatment methods, have appeared in many fields of physics, new methods of complementary analyzes of those traditionally used (Fourier analysis and Flash analysis). One of them consists in associating a random excitation and a parametric analysis to the mode of control used [2]. The principal advantage of this association is to give access to the impulse response of materials, while implementing a lesser density of excitation and thus, to allow the material analysis more delicate than with the flash method. The idea to associate a random excitation and a parametric analysis to the photothermal radiometry front face is then attractive, since being able to offer new possibilities as regards thermal metrology. For this purpose, we undertook a study aiming at approaching the possibilities of the method as regards measurement of thermal diffusivity. The results obtained during this study are presented here. Initially, we present the principle
of random photothermal radiometry associated with a parametric analysis. Then, we present the studied samples, the experimental conditions and the experimental device implemented during this study. In a third stage, we present the method of evaluation of the thermal parameter diffusivity. Finally, we show the random photothermal method allows a good estimation of the thermal diffusivity of a sample of nylon 6.6.

2. Principle of front face random photothermal radiometry
The front face random photothermal radiometry consists initially, to subject the studied sample to a modulated luminous flow in a random way. Then, it consists in starting from the thermal response given by the sample, to build a behaviour parametric model of the experiment. For that, we adjust, using the recursive least squares method, the parameters of the behaviour models in order to equalize the physical photothermal response to those calculated by the model. It finally consists in calculating the theoretical response of this model for excitation equal to a delta Dirac function to lead to the required pulse response [2]. The excitation sequence, near to a white noise (the more the excitation sequence will contain frequencies, the more the sample answer will contain information and thus the more the behaviour model will be fine), implemented for our study is a pseudo random binary sequence (PRBS); that primarily for its simplicity of implementation. The behaviour model used for the study is an ARMA Model (Auto Regressive Average Model, 1). It considers that the output signal of the electronic filter, to which is comparable the physical system to identify, is related in a linear way to the various states of the input signal of the filter \( \{ e(n) \} \), with the various intrinsic parameters of the filter \( \{ a_n, b_n \} \), but also with the various former states of the output signal itself \( \{ s(n-m) \} \). It is thus very complete, because it is a classical convolution model \( \hat{s}(n) = \sum_{m=0}^{q} b_m e(n-m) \) with taking into account of a possible memory effect \( \hat{s}(n) = \sum_{m=1}^{p} a_m \hat{s}(n-m) \). That explains its implementation.

\[
\hat{s}(n) = \sum_{m=1}^{p} a_m \hat{s}(n-m) + \sum_{m=0}^{q} b_m e(n-m) \quad (1)
\]

3. The experimental system implemented for our study
The experimental system implemented for our study, is the analysis system of thin materials by infrared (SAMMIR) of the Laboratory (figure 1). The excitation optics of SAMMIR, is first made up, of a laser diode being able to deliver a maximum power of 0.55 Watts to a wavelength of 0.81 µm. This light source is then associated to a collimation optics and a focusing optics. The laser beam, once formatted, has a characteristic diameter of about 500 µm. The laser diode is controlled electronically, in order to be able to deliver pseudo random binary sequences (figure 2) length going from 64 terms to 4096 terms by geometrical ratios of 2. The sampling rate of these signals can go from some tenth of hertz to 500 Khz. An oversampling, a repetition and a numerical signals filtering are possible to limit aliasing spectrum influence and to increase the signal/noise ratio. The detection optics of SAMMIR is composed of an association of off axis parabolic mirrors and of a quantum detector HgCdTe mono element, cooled with nitrogen liquid. This last has a peak of sensitivity to 10.6 µm and a significant surface of 100µm * 100µm. SAMMIR includes finally an electronic acquisition and a data processing of piloting and signals treatment. The piloting electronics allows an amplification, with almost null phase, of the signal issued from the infrared detector and a digitalization of this last until a frequency of 500 Khz. Amplification is adaptive according to the level of received signal, which makes it possible to digitize with the maximum of dynamics and for each study the photothermal signal collected. The data processing of treatment of the signals allows parametric analyses, and a post processing of the data.
4. The analyzed sample and experimental conditions retained for the study

The sample analyzed in this study is a cylindrical sample of Nylon 6.6. It is 1.75mm thick. Its diameter is 30mm. Its thermophysical properties are a thermal conductivity of 0.26 W/mK, a density of 1150kg/m³, a heat-storage capacity of 1700 J/kg K and thus a thermal diffusivity of $1.33 \times 10^{-7}$ m²/s. The experimental conditions implemented during the study, are the use of a pseudo random binary sequence of 1024 terms, of a sampling rate of 5 bits/s, with an over sampling of 10 and a parameters number $(a_n, b_n)$ of the ARMA behaviour model equal to 40.

5. The theoretical model developed for the study

The theoretical model developed for the theory/experiment adjustment is a 1D model of the photothermal flash method. The sample is assimilated to a wall of infinite surface and with thickness $e$. Initially, the temperature of this wall is equal to 0 °c. Heat exchanges with the surrounding medium are considered on the front and back faces of this last. The excitation is comparable with a Dirac function and is supposed to be deposited in a very thin surface section material. Lastly, an assumption of small variations in temperature due to the thermal impulse makes it possible to regard the thermal properties as independent of the temperature. The mode of resolution of the heat equation used is the thermal quadruples method [3]. It leads to an expression of the front face temperature of the analyzed sample, expressed in the space of Laplace, following:

$$
\theta_e(p) = \frac{Q(Cosh(e\sqrt{\frac{p}{d}}) + hS)}{2S\sqrt{\frac{p}{d}} - \frac{p}{d} Sinh(e\sqrt{\frac{p}{d}})}
$$

(2)

The return in temporal space is obtained numerically using the method of Stephest.
6. The experimental results obtained
On figure 3, we first present an example of photothermal response obtained at the time of our studies. It acts, as we can see it, of a random answer rather difficult to interpret just as it is. On figure 4 (red curve), we then present an example of pulse response rebuilt by parametric analysis. We find the traditional form of the pulse responses obtained by front face flash method. In order to lead to a measurement of the thermal diffusivity parameter, the theoretical model of the photothermal experiment flash developed for the study was then adjusted with this rebuilt pulse response. The method of minimization used is the Box-Kanemasu method [4]. The result of this adjustment is presented on figure 4 (black curve). It shows that it is for a thermal diffusivity value of $1.30 \times 10^{-7}$ m$^2$/s, that theoretical (thick black curve) and experimental (fine black curve) curves are close to each other. This value of diffusivity thus estimated, close to the value is given by the literature ($1.33 \times 10^{-7}$ m$^2$/s), which shows the possibility of the photothermal method as regards thermal diffusivity measurement.

![Figure 3. Example of photothermal signal collected](image1)

![Figure 4. Example of thermal diffusivity measurement of a nylon 6.6 sample](image2)

7. Conclusion
In this work, we tried to approach the possibilities of thermal measurement of diffusivity, under less energy constraints; by front face random photothermal radiometry associated with a parametric analysis. First, we present the principle of the front face random photothermal method. Then, we present the experimental device SAMMIR used in our study. In a third stage, we present the studied sample, the experimental conditions selected and the model developed for the study. We finally showed the photothermal method implemented allowed a suitable measurement of the thermal diffusivity of a sample of nylon 6.6. This experimental result obtained on a particular sample, is encouraging. It can open the way with the photothermal sensitive material characterization. Now, it has to be confirmed and generalized. Studies are in progress.

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