International Journal of Thermal Sciences 129 (2018) 385–395

Contents lists available at ScienceDirect

International Journal of Thermal Sciences

journal homepage: www.elsevier.com/locate/ijts

Signal noise ratio improvement technique for bulk thermal diffusivity measurement

Emmanuel Ruffio, Christophe Pradere, Alain Sommier, Jean-Christophe Batsale, Andrzej Kusiak, Jean-Luc Battaglia*

Institute of Mechanical Engineering (I_2M), UMR CNRS 5295, University of Bordeaux, 351 cours de la Libération, 33405, Talence Cedex, France

ABSTRACT

This paper presents an improvement of the classical flash technique that allows measuring the thermal diffusivity of highly diffusive materials without the need of blackbody coatings. The method consists in heating the front face of the sample using a periodic pulses sequence with suitable period and pulse duration. The rear face temperature is recorded simultaneously. An inverse approach is then used to estimate the thermal diffusivity. The underlying model is completely analytical and includes heat transfer and analog signal processing which has been specifically designed for this experiment. A sensitivity analysis and an experiment optimization are performed. Applied to an uncoated copper sample, this method appears to be reliable even in the case of very low optical emission/absorption. Theoretical expectations have been confirmed from experimental data obtained considering copper. Thermal diffusivity has been estimated with less than 4% on both the standard and absolute deviation.

1. Introduction

Measuring the thermal diffusivity of highly conductive materials remains challenging and large deviations are still observed according to the experimental operating conditions. The classical photothermal methods are based on the thermal response of the sample to a heat pulse, considered as a Dirac function from the mathematical point of view. The temperature increase on the opposite face (rear face) of the excitation is classically measured using an infrared (IR) detector. This so-called flash experimental technique that has been developed first by Parker [1] and that has been improved along time by several contributions [2–5]. An exhaustive list of the developments can be found in Ref. [6]. However, highly diffusive samples are generally of metallic nature with very low optical emission/absorption in the visible and infrared wavelengths. Thereby, if no optical transceiver is present on both faces of the sample, the measured signal is very noisy and this leads to poor confidence interval of the identified property. Therefore, the classical approach requires depositing thin blackbody coatings on both faces, the one on the front face assuring the maximum energy absorption of the photothermal source whereas the other one on the rear face is assumed to make the emissivity close to one, thus enhancing the signal noise ratio of the IR detector. Those coatings are generally deposited from spray constituted by graphite particles within an epoxy resin [7,8]. Nevertheless, they have a significant influence on the measured signal according to their thermal properties. This can be easily demonstrated from a model of the one-dimensional heat conduction through a three-layers system [9]. This model can be simulated using the classical quadrupoles method based on integral transform technique [10]. Let us assume for instance a cylindrical sample made of copper (thermal conductivity $k_{Cu} = 400$ W. m^{-1}. K^{-1}, specific heat per unit volume $(\rho\ C_p)_{Cu} = 3.5$ MJ. K^{-1}. m^{-3} and thickness 3 mm) and whose both faces are coated with a graphite-based spray (thermal conductivity $k_d = 1$ W. m^{-1}. K^{-1}, specific heat per unit volume $(\rho\ C_p)_d = 2$ MJ. K^{-1}. m^{-3}). The apparent thermal diffusivity of the one-layer sample is reported in Table 1 according to the thickness e_d of the coating. It is clear that a significant difference in the thermal diffusivity of copper (114.3 mm^2. s^{-1}) occurs when the coating thickness is larger than 10 µm. In addition it is also remarkable that the thermal resistance of one coating is comparable or larger than that of the copper layer ($R_{Cu} = e_{Cu}/k_{Cu} = 7.5 \times 10^{-6}$ K. m^2. W^{-1}) when $e_d \geq 10$ µm. It is thus recommended to control accurately the coating thickness and the contact resistance when using a 3-layers model or to find highly diffusive blackbody coatings. Both previous recommendations are not easy to respect given the inherent composition of the spray and the deposition process that is random by nature. In addition, deposition of other coatings using chemical vapor deposition or pressure vapor deposition processes for instance, makes the experiment more complex.

A theoretical solution has been proposed by Vozär et al. [11] that

* Corresponding author.
E-mail address: jean-luc.battaglia@u-bordeaux.fr (J.-L. Battaglia).

https://doi.org/10.1016/j.ijthermalsci.2018.03.011
Received 19 August 2017; Received in revised form 18 January 2018; Accepted 12 March 2018
Available online 30 March 2018

Table 1
Apparent thermal diffusivity a_{app} of a copper sample coated on both faces with graphite-based spray, with respect to the coating thickness e_d. The thermal resistance of the coating $R_d = e_d/k_d$ is also given. First line refers to the diffusivity estimated by an inverse approach (the sample is assumed homogeneous but a 3-layers model is used to simulate experimental data). Second line is the apparent diffusivity given by Ref. [10] (Eq. (10)). Contact resistance is omitted.

e_d μm	0	1	10	20	50
a_{app} mm^2. s^{-1}	114.3	114.3	113.4	109.4	85.4
a_{app} mm^2. s^{-1}[10]	114.3	114.3	113.2	108.9	83.4
$R_d \times 10^{-6}$ K. m^2. W^{-1}	0	1	10	20	50

consists in heating the sample using severals pulses in order to increase the amount of energy entering the sample while minimizing temperature gradient. In this paper, a variant of this idea is proposed with the following changes. The photothermal source is now continuously emitting heat pulses at low frequency f_{exc}. As a consequence, the mean temperature of the sample increases slowly. Once the steady periodic regime is reached, the rear face temperature is recorded after every pulses. Thus, N_s pulses lead to N_s columns vectors $T_i(t)$ of temperature values, with $1 \le i \le N_s$. Since the temperature evolution is periodic, differences between these N_s vectors are due to the measurement noise only. By computing the average temperature over index i, the resulting vector $\overline{T}(t) = \frac{1}{N_s}\sum_{i=1}^{N_s} T_i(t)$ shows an improved signal noise ratio. Assuming vectors are statistically independent, the noise standard deviation of temperature values $\overline{T}(t)$ is theoretically reduced by $\sqrt{N_s}$ compared to $T_i(t)$. As high as N_s could be ($N_s = 2000$ in this study), it has no effect on the temperature amplitude and low voltage signals have still to be recorded in the case of low optical emission/absorption sample. A specific signal amplifier based on a high-pass filter has been developed.

The experimental setup is presented is section 2. In section 3, the corresponding heat transfer and electrical models are presented and an analytical solution is derived. The photothermal pulse width and the contribution of the filter are considered. A sensitivity analysis is performed in section 4 in order to optimize the experimental operating conditions. In section 5, the method is applied to a copper sample.

2. Experimental setup

The experimental setup is represented schematically in Fig. 1. It is composed of a Coherent Matrix Q-switch Nd:YAG diode-pumped laser (1064 nm wavelength) delivering pulses in a continuous or burst mode. The pulse width is lower than 40 nsec and the maximum pulse frequency is $f_p = 100$ kHz. The maximum rms power (10 W) is reached with a pulse frequency of 30 kHz. The laser beam radius is 0.55 mm (<3 mrad divergence) and it is directed at the front face of the sample. There is no way to produce a pure continuous wave with this laser. Therefore, a finite pulse width excitation is produced by emitting a N_p-pulse train with the laser burst mode. It consists in emitting a quick succession of N_p pulses at frequency f_p. The pulse width being thereby $\Delta t_{burst} = N_p/f_p$. The N_p-pulse train is repeated with frequency f_{exc} using a function generator (Agilent 33320A). This repetition frequency dictates the heat transfer dynamics in the sample. A fast photodiode with 0.5 nsec rise time (Thorlabs DET 10A/M) is used to trigger the acquisition device (a Lecroy Waverunner LT 364 scope). The temperature variations of the rear face are low so that there is a linear relationship between the temperature change and the emitted infrared radiation which is monitored by a HgCdTe photoconductive infrared detector (Judson J15D12) working in the (2–14) μm range with the maximum sensitivity located at 11 μm. The location and solid angle (45°) of the detector are such that the sample is fully contained in the field of view. As a result, the detector output (a variation of electrical resistance R_{det}) is related to the average surface temperature. This configuration allows using non-uniform distribution of the deposited heat flux at the front face as well as wavelength-dependency of optical coefficient. A germanium window (high band-pass optical filter with 1.4 μm cut-off wavelength) is put in front of the detector to reject all laser diffuse reflection that could affect temperature measurements.

As presented in Fig. 2, the temperature dependent resistance R_{det} of the photoconductive detector is part of a Wheatstone bridge-based circuit leading to a first stage amplification of gain$K_1 = 10$. A 2nd-order high-pass filter of Sallen-Key type is then used in order to remove the time average temperature, i.e. the DC signal, as well as all potential temperature drifts and signal shifts. Thanks to this, the signal variations stay centered on 0 V even with the lowest scope sensitivity. The gain $K_2 = 1 + R_1/R_2$ is related to the damping factor of the filter. Finally, the signal is amplified by a factor $K_3 = 30$ and recorded using the scope (input impedance $R_{load} = 1$ MΩ, 8 bits vertical resolution, 11 bits resolution for math tools). The signal average is performed over N_s sweeps or sets (up to $N_s = 4000$ with this scope) of N_{data} (up to 50 000) temperature samples.

The overall analog signal processing is described by a 2nd-order transfer function H_f. Some test was carried out with a function generator as input to check the consistency of this model. Results were in good agreement with theoretical expectations [12]. $H_f(j\,\omega)$ is:

$$H_f(j\,\omega) = G\frac{-\omega^2/\omega_c^2}{1 + j\,2\,m\,\omega/\omega_c - \omega^2/\omega_c^2}\ ,\ j^2 = -1 \tag{1}$$

With f be the signal frequency under consideration and $\omega = 2\,\pi\,f$ the corresponding angular frequency. G is the gain. $\omega_c = 2\,\pi\,f_c$ with f_c is the high-pass cut-off frequency. m is the damping factor. The high pass filter is built with potentiometers allowing f_c to vary from 0.1 to 30 Hz. Based on Fig. 2, it comes: $\omega_c = 1/R\,C$ and $m = \frac{1}{2}(3 - K_2)$. Components were chosen in such a way as to get $f_c \approx 0.8$ Hz and $m \in [0.5 - 0.7]$ range. Using a square wave generator as input signal and an inverse approach, it was found $f_c = 0.85 \pm 0.09$ Hz and $m = 0.7 \pm 0.07$. There is no use in estimating the amplifier gain G since the signal processing model will be combined with the heat transfer model so that the overall gain will include the optical part as well (emissivity of the sample, solid angle, efficiency and bias current of the detector …).

3. Mathematical model

Thermal diffusivity and conductivity of the material are denoted a and k respectively. The sample is assumed opaque within the visible and infrared wavelength range. Temperature variations are small

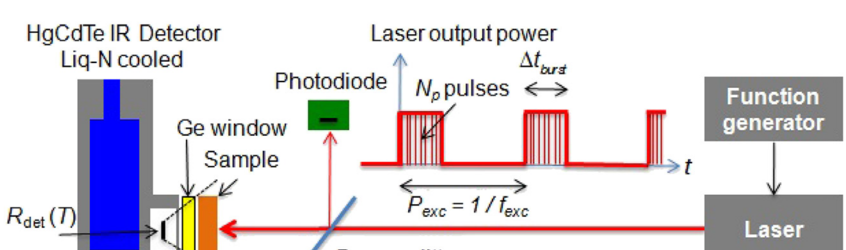

Fig. 1. Experimental setup - the photothermal excitation $\phi(r,\ \theta,\ t)$ is an N_p-pulse train whose equivalent width is Δt_{burst}, periodically repeated with frequency f_{exc}.