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Flash Method for the Remote Sensing of the Thermal Diffusivity and the Absorption Coefficient at the Excitation Wavelength of Thin Film Materials

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Abstract A novel front-face flash-monitoring algorithm for remote sensing of the thermal diffusivity and the absorption coefficient at the excitation wavelength of thin film materials is presented. The method relies on the surface transient temperature vs. time curve initiated by a short-duration heating pulse that is followed by time intervals during which the temperature response depends sensitively on the absorption coefficient and thermal diffusivity. An analytical solution of the problem has been derived to solve the inverse heat-transfer problem and, by comparison with a numerical experiment, to check the accuracy of the proposed algorithm for obtaining the absorption coefficient at the excitation wavelength and thermal diffusivity. The uncertainty of the proposed method is better than 0.1%.

Keywords Absorption coefficient · Criterion of thermal homogeneity · Flash method · Remote sensing · Thermal diffusivity · Thin films

1 Introduction

The laser-flash technique relies on the use of a short-duration heat pulse to produce an initial thermal gradient in a sample. Subsequently the time development of the

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temperature can be measured at the back or at the front surface of the sample. The first option is called "double-ended," i.e., the excitation source and the detection system are on opposite sides of the sample. This conventional technique is widely used in thermo-analytical laboratories because of its rapidity and simplicity of implementation. It is, however, limited to thin opaque samples. If we try to apply this technique also to absorbing media, we accordingly must take into account absorption of the heat pulse since the penetration depth of the radiation changes the temperature distribution in the investigated material. This results in an effective (not the real) thermal diffusivity.

This conventional technique is, strictly speaking, of little efficiency. It allows measurement of only the *average* values of physical parameters such as thermal diffusivity or thermal conductivity. With the conventional method, we cannot investigate real, frequently layered systems especially if we do not know the thermal properties of at least one of the layers.

Accordingly, if the laser-flash technique shall be used to monitor also quality and process control on the production line, more flexible methods must be developed that can be applied to bulk materials of arbitrary thickness and internal structure and that should require access to one surface only. We will call these more advanced methods "front-face flash-monitoring methods (FFFMM)," in the following. In particular, single-ended FFFMM (with the complete apparatus only on one side of the investigated object) can provide a reliable tool to measure the properties also of layered and laminated samples, even if there is only imperfect contact at the corresponding interfaces. Moreover, using FFFMM we obtain at once, from *one* experiment, several physical characteristics and possible relations among themselves. The purpose of this paper is to describe a FFFMM to satisfy such requirements.

2 Description of the Method

In the following discussion we assume a one-dimensional heat flow model. This assumption is valid when the thickness of the sample is much smaller than the lateral dimensions of the area that is uniformly illuminated by the optical excitation flash. To further simplify the mathematical analysis, we assume the heat loss due to convection and radiation at the sample surfaces and due to lateral conduction to be negligible.

The evolution of the position (x)- and time (t)-dependent "added" temperature (above the ambient temperature, T_a), $\Theta(x,t)$ inside the sample, generated by the absorption of the flash pulse, is described by the one-dimensional Fourier equation,

$$\frac{\partial\Theta(x,t)}{\partial t} = a\frac{\partial^2\Theta(x,t)}{\partial x^2} \tag{1}$$

with the adiabatic boundary condition,

$$\frac{\partial\Theta(x,t)}{\partial x} = 0 \tag{2}$$

at the sample surfaces after the excitation flash. As usual, a denotes the thermal diffusivity of the sample and x is the distance into the sample (with $x = 0$ indicating the position of the irradiated surface). We shall further assume that the excitation flash, at $t = 0$, is of infinitesimal duration.

The heat generated inside the sample due to the absorption of the flash energy is proportional to the term $\alpha e^{-\alpha x}$ at a distance x from the irradiated surface; see Refs. [1–6]. In this expression, α denotes the absorption coefficient at the excitation wavelength. Hence, the initial temperature distribution inside a thin solid slab of thickness L is

$$\Theta(x, 0) = A\alpha e^{-\alpha x}, \tag{3}$$

where $x \le L$ and A is a constant depending on the flash pulse energy and the heat capacity of the sample.

Heat diffusion under adiabatic boundary conditions, Eq. (2), for a thin solid slab of thickness L was discussed in Carslaw and Jaeger [7]. If in the present case the initial temperature distribution inside the slab, $f(x)$, is given by Eq. (3), the temperature at a distance $x > 0$ from the irradiated surface at a later time accordingly reads

$$\Theta(x, t) = \frac{1}{L}\int_0^L \Theta(x, 0)\mathrm{d}x + \frac{2}{L}\sum_{n=1}^{\infty} e^{-n^2\pi^2\frac{at}{L^2}} \cos\left(\frac{n\pi x}{L}\right)\int_0^L \Theta(x, 0)\cos\frac{n\pi x}{L}\mathrm{d}x.$$

From this equation, with $\Theta(x, 0)$ from Eq. (3), the temperature vs. time curve at the irradiated surface after the excitation can be represented as

$$\Theta(0, t) = \frac{A}{L}\left[(1 - e^{-\alpha L}) + 2\sum_{n=1}^{\infty} e^{-n^2\pi^2 Fo}\left(\frac{1 - (-1)^n e^{-\alpha L}}{1 + \frac{n^2\pi^2}{\alpha^2 L^2}}\right)\right], \tag{4}$$

where $Fo = \frac{at}{L^2}$ is the Fourier number.

If the absorption coefficient, α, is very large such that $\alpha L \to \infty$, Eq. (4) reduces to the corresponding expression given by Parker et al. [8]. If, on the other hand, the sample is transparent at the excitation wavelength ($\alpha = 0$), no excitation and hence temperature excursion will be observed.

The surface temperature excursion depends on α and on *Fo*. To determine from measured surface temperature excursions the thermal diffusivity and the absorption coefficient, we have to look for the inverse of the solution of the direct heat-transfer problem. For this purpose, one should preferentially use the simplest solution of Eq. (4). A considerable reduction of the complexity of Eq. (4) can be obtained if we take into account only the first term of the series expansion

$$\Theta \approx \frac{A}{L}\left[(1 - e^{-\alpha L}) + \frac{2\mathrm{e}^{-\pi^2 Fo}(1 + \mathrm{e}^{-\alpha L})}{1 + \frac{\pi^2}{\alpha^2 L^2}}\right]. \tag{5}$$

To justify this approach, it is necessary to identify an interval of *Fo* in which this approximation is valid. This check could be done, in principle, by means of a regularity coefficient, K, defined in the following equation that is determined by the ratio of the temperatures calculated using Eqs. (4) and (5),

$$K = 1 - \frac{(1 - e^{-\alpha L}) + 2\sum_{n=1}^{\infty}\left[\frac{1-(-1)^n e^{-\alpha L}}{1+n^2\pi^2/\alpha^2 L^2}\right] e^{-n^2\pi^2 Fo}}{(1 - e^{-\alpha L}) + 2\left(\frac{1+e^{-\alpha L}}{1+\pi^2/\alpha^2 L^2}\right) e^{-\pi^2 Fo}}. \tag{6}$$

For $K \to 0$, the interval for *Fo* can be determined if K is known.

However, in the present case, the regularity coefficient, K, cannot be identified directly from a real physical experiment. Instead, we refer to the previously [9] introduced dimensionless criterion of thermal homogeneity, *To*, which can be obtained directly from experiment

$$To = \frac{\Theta}{t\Theta'}, \tag{7}$$

where Θ and Θ' are the temperature and its derivative with respect to time, respectively, measured as a function of time.

The procedure is as follows: first, we calculate the exact temperature curve (full solution) from Eq. (4), the truncated solution of the approximate Eq. (5) and the coefficient K from Eq. (6), in order to check under which *Fo* the results would coincide. This identifies an effective Fourier number, Fo^*, for the conditions under which the reduced Eq. (5) may be applied, while the coefficient K indicates the accuracy of the process. Next, we use Eq. (8) (see below) to calculate the inverse of the criterion for thermal homogeneity, which shows that the minimum of this equation corresponds to Fo^* (for which Eq. (5) is applicable). We accordingly can use the criterion, *To*, instead of the regularity coefficient, K, to identify the interval in which our approximation is valid.

Using Eqs. (4) and (7), the inverse value of *To* reads

$$\frac{1}{To} = -\frac{2\sum_{n=1}^{\infty} n^2\pi^2 Fo\left[\frac{1-(-1)^n e^{-\alpha L}}{1+n^2\pi^2/\alpha^2 L^2}\right] e^{-n^2\pi^2 Fo}}{(1 - e^{-\alpha L}) + 2\sum_{n=1}^{\infty}\left[\frac{1-(-1)^n e^{-\alpha L}}{1+\frac{n^2\pi^2}{\alpha^2 L^2}}\right] e^{-n^2\pi^2 Fo}}. \tag{8}$$

Numerical results for K and $1/To$ using Eqs. (6) and (8) are presented in Fig. 1. They show that the minimum of $(1/To)$ practically corresponds to Fo^*, where Eq. (5) is valid. The coefficient K defined in Eq. (6) at this *moment* amounts to $K = -0.00061$. We have $Fo^* = 0.108$, i.e., the error due to the use of Eq. (5) instead of Eq. (4) is 0.061%. For $Fo > Fo^*$ (i.e., for times later than that of the minimum of $1/To$), the error is negligible. This is also obvious from Fig. 2.

Accordingly, the point on the surface temperature vs. time curve where the inverse value of the dimensionless criterion of thermal homogeneity has its minimum, can be used as a reference for determination of the thermal diffusivity from Fo^*. Furthermore,

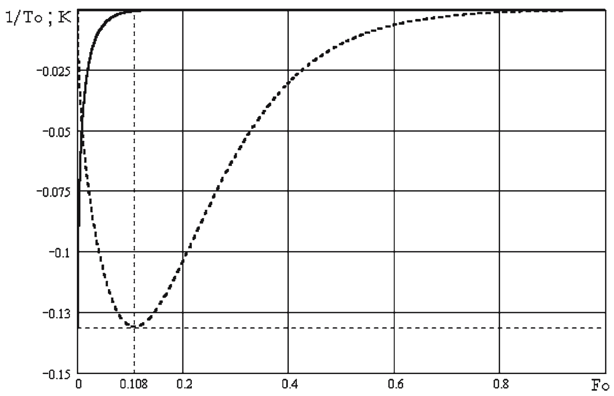

Fig. 1 Inverse value of thermal homogeneity, $1/To$ (dashed line), and coefficient K (solid line) as functions of Fourier number, *Fo*, for $\alpha L = 1$

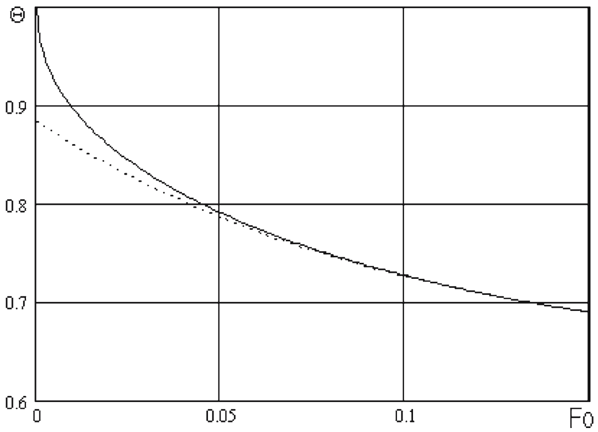

Fig. 2 Surface temperature evolution as a function of Fourier number, *Fo*, for $\alpha L = 1$; solid line for Eq. (4), dashed line for Eq. (5)

after elementary transformations of Eq. (5), it follows that

$$a = \frac{L^2}{\pi^2 \Delta t} \ell n \left(\frac{\Theta_1 - \Theta_2}{\Theta_2 - \Theta_3} \right), \tag{9}$$

where Θ_1, Θ_2, and Θ_3 are a sequence of temperatures measured with time steps of $\Delta t = 0.1 t_{\mathrm{m}}$. The time t_{m} corresponds to the minimum of $1/To$.

When $Fo > Fo^*$, i.e., for $t > t_{\mathrm{m}}$, the surface temperature approaches the steady-state value, Θ_{k}. It is practically equal to the steady-state value within 0.06% at $Fo = 6Fo^*$, i.e., at $t = 6t_{\mathrm{m}}$.

Beginning with the *moment*

$$\Theta = \Theta_\kappa = \frac{A}{L}(1 - e^{-\alpha L}) \tag{10}$$

and using the initial value of the surface temperature (compare Eq. (3)) for $x = 0$

$$\Theta_o = A\alpha, \tag{11}$$

one obtains

$$\frac{\Theta_\kappa}{\Theta_o} = \frac{1 - e^{-\alpha L}}{\alpha L}. \tag{12}$$

Then the absorption coefficient, α, can be derived iteratively from the transcendental Eq. (12); the temperature Θ_o must be measured just after the end of the heat pulse.

3 Numerical Test of Proposed Method

A numerical experiment has been performed to test the proposed method. In the first step, we solved the direct heat-transfer problem using Eq. (4) for a given αL, i.e., we found $\Theta = \Theta(Fo)$, the surface temperature vs. time excursion. In the second step, we solved the inverse heat-transfer problem as described in the previous section and then used Eqs. (9) and (12). As a result, the uncertainty of the proposed method is below 0.1%.

4 Conclusion

A novel method to obtain the absorption coefficient at the excitation wavelength and the thermal diffusivity of thin film materials has been presented. A numerical simulation was used to validate the applicability of the method for absorbing media. The agreement between theory and numerical experiment is very good. Like the "diverging thermal wave technique" introduced in Ref. [10], this method is another step to facilitate determination and process control of thermophysical properties of films and coatings under industrial working conditions.

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