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Two data reduction methods for evaluation of thermal diffusivity from step-heating measurements

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Abstract—In this paper the principle and theoretical basis of the step-heating technique for thermal diffusivity measurement is summarized. Application of two numerical fitting methods for data reduction—least-squares and Levenberg–Marquardt methods—is described. Results of thermal diffusivity estimation from both simulated and experimental data are presented and discussed. Copyright © 1996 Elsevier Science Ltd.

1. INTRODUCTION

The step-heating method is a promising photothermal technique for measurement of thermal diffusivity of solids [1, 2]. In this method the thermal diffusivity of a material is measured by subjecting the front face of a small, usually disk-shaped sample to a constant heat flux condition. From the resultant temperature rise at the opposite (rear) face of the sample the thermal diffusivity can be obtained. The step-heating method can be viewed as an extension of the well known flash method, based on measurement and analyzing the temperature response at the rear face after application of an instantaneous heat pulse [3]. Although the flash technique was primarily proposed for measurement of homogeneous and isotropic materials, it has also been successfully applied for measurement of heterogeneous and anisotropic materials including layered, dispersed and fiber-reinforced composites. However, extensions of its use for some types of insulators and explosive materials is limited due to the relatively large front face temperature rise caused by the heat pulse. There are also difficulties involved in measuring the thermal diffusivity of large-grained heterogeneous materials, especially oriented fiber-reinforced composites, where the scale of the microstructure is comparable with the sample thicknesses usually used [4]. Substituting step-heating for the laser pulse tends to overcome these difficulties, the possibility of using samples of relatively large dimensions in comparison to those used in the flash method allows the extension to cases where the material can be considered to behave as a homogeneous medium [5]. Another advantage of the step-heating method is the relatively low intensity of the imposed heat flux in comparison with that necessary for the pulse-heating techniques. The sample is therefore less likely to exhibit a phase transition or decompose as a result of a sudden large temperature increase at the front face.

2. MATHEMATICAL BACKGROUND OF THE STEP-HEATING METHOD

The ideal model is based on the behavior of a homogeneous, thermally insulated, infinite slab with uniform and constant thermal properties and density, subjected to a constant heat flux, uniformly applied since the time origin, over its front face ($x = 0$). The transient temperature $T = T(e, t)$ at the rear face ($x = e$) of the sample can be obtained by solving the one-dimensional heat conduction equation

$$\frac{1}{a}\frac{\partial T}{\partial t} = \frac{\partial^2 T}{\partial x^2}, \quad 0 \leqslant x \leqslant e; \quad t > 0 \tag{1}$$

with the initial and boundary conditions

$$T(x, 0) = 0, \quad 0 \leqslant x \leqslant e \tag{2}$$

$$\frac{\partial T(0, t)}{\partial x} = -\frac{q}{k}, \quad t > 0 \tag{3}$$

$$\frac{\partial T(e, t)}{\partial x} = 0, \quad t > 0 \tag{4}$$

where a is the thermal diffusivity, k is the thermal

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NOMENCLATURE

a	thermal diffusivity	q	heat flux per unit area
a^*	estimated value of thermal diffusivity	r_s	sample radius
B	heat flux parameter	S_β	normalized sensitivity coefficient
C_p	specific heat	t	time
e	sample thickness	T	temperature.
h_0, h_e, h_r	heat transfer coefficients		
H, H_0, H_e, H_a, H_r	Biot numbers		
H^*	estimated Biot number	Greek symbols	
k	thermal conductivity	θ	function
O	computational complexity	ρ	density.

conductivity and q is the heat flux per unit area. The general expression for the sample temperature as a function of position x and time t is [6]

$$T(x,t)=\frac{q}{\rho ce}\left[t+\frac{e^2}{3a}-\frac{ex}{a}+\frac{x^2}{2a}-2\sum_{n=1}^{\infty}\cos\left(\frac{n\pi x}{e}\right)\frac{\exp\left(\left(-\frac{n^2\pi^2}{e^2}\right)at\right)}{\left(\frac{n^2\pi^2}{e^2}\right)a}\right]. \quad (5)$$

The temperature $T(t) = T(e,t)$ at the rear face ($x = e$) can be written in the form

$$T(e,t)=\frac{q}{\rho ce}\left[t-\frac{e^2}{6a}-2\sum_{n=1}^{\infty}(-1)^n\frac{\exp\left(\left(-\frac{n^2\pi^2}{e^2}\right)at\right)}{\left(\frac{n^2\pi^2}{e^2}\right)a}\right] \quad (6)$$

where ρ is the density and c is the specific heat.

Use of this simple adiabatic model is limited due to the difficulty of creating the ideal conditions considered here. In a real experiment heat transfer between the sample and its environment is often unavoidable. Although one can reduce heat losses by using radiation shields, vacuum chambers etc., in some cases (high temperatures, poorly thermal conductive materials) it is necessary to take heat exchange into consideration in the working equation.

Let us consider a disk-shaped sample with thickness e and radius r_s (Fig. 1). If we take into account heat losses from the sample governed by Biot numbers related to each face $H_0 = h_0 e/k$, $H_e = h_e e/k$ and

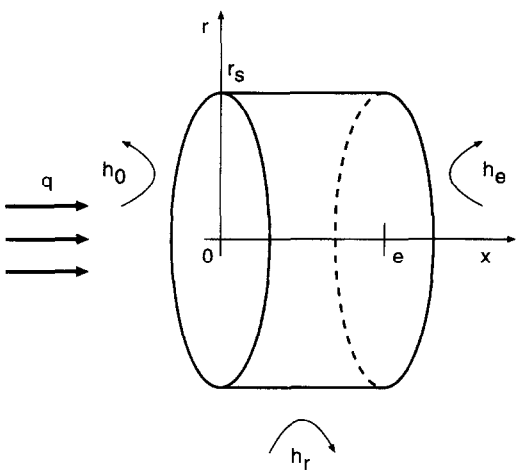

Fig. 1. Model with heat losses.

$H_r = h_r r_s/k$ (h_0, h_e, h_r being the axial and radial heat transfer coefficients), the equation to be solved is

$$\frac{1}{a}\frac{\partial T}{\partial t}=\frac{\partial^2 T}{\partial x^2}+\frac{\partial^2 T}{\partial r^2}+\frac{1}{r}\frac{\partial T}{\partial r}, \quad 0\leqslant x\leqslant e;\quad 0\leqslant r\leqslant r_s;\quad t>0 \quad (7)$$

with the boundary conditions

$$T(x,r,0)=0,\quad 0\leqslant x\leqslant e;\quad 0\leqslant r\leqslant r_s \quad (8)$$

$$\frac{\partial T(0,r,t)}{\partial x}=-\frac{q}{k}+\frac{H_0}{e}T(0,r,t),\quad t>0 \quad (9)$$

$$\frac{\partial T(e,r,t)}{\partial x}=-\frac{H_e}{e}T(e,r,t),\quad t>0 \quad (10)$$

$$\frac{\partial T(x,r_s,t)}{\partial r}=-\frac{H_r}{r_s}T(x,r_s,t),\quad t>0 \quad (11)$$

where $T = T(x,r,t)$ is the temperature at the point (x,r) and the time t. The general solution of this problem can be obtained in the form [7]

$$T(x,r,t)=\frac{q}{\rho ce}\sum_{n=1}^{\infty}A_n\left(H_0,H_e,\frac{x}{e}\right)$$

$$\times \sum_{m=1}^{\infty} B_m\left(H_r, \frac{r}{r_s}\right) \frac{1-\exp\left(-\left(\frac{u_n^2}{e^2}+\frac{w_m^2}{r_s^2}\right)at\right)}{a\left(\frac{u_n^2}{e^2}+\frac{w_m^2}{r_s^2}\right)} \quad (12)$$

where

$$A_n\left(H_0, H_e, \frac{x}{e}\right) = a_n\left(\cos\left(u_n\frac{x}{e}\right)+\frac{H_0}{u_n}\sin\left(u_n\frac{x}{e}\right)\right) \quad (13)$$

$$a_n = \frac{2u_n^2(u_n^2+H_e^2)}{(u_n^2+H_0^2)(u_n^2+H_e^2)+(H_0+H_e)(u_n^2+H_0H_e)} \quad (14)$$

$$B_m\left(H_r, \frac{r}{r_s}\right) = \frac{2H_rJ_0\left(w_m\frac{r}{r_s}\right)}{J_0(w_m)(w_m^2+H_r^2)} \quad (15)$$

and u_n and w_m are the positive roots of the equations

$$(u^2 - H_0H_e)\tan(u) = (H_0+H_e)u \quad (16)$$

$$wJ_1(w) = H_rJ_0(w) \quad (17)$$

and J_0 and J_1 are Bessel functions of the first kind, order 0 and 1. The temperature $T(t) = T(e, 0, t)$ in the center of the rear face can be written as:

$$T(e,0,t) = \frac{q}{\rho ce}\sum_{n=1}^{\infty} A_n(H_0, H_e, 1)$$

$$\times \sum_{m=1}^{\infty} B_m(H_r, 0)\frac{1-\exp\left(-\left(\frac{u_n^2}{e^2}+\frac{w_m^2}{r_s^2}\right)at\right)}{a\left(\frac{u_n^2}{e^2}+\frac{w_m^2}{r_s^2}\right)}. \quad (18)$$

3. DATA REDUCTION

3.1. *Use of the least-squares fitting procedure*

Estimation of the thermal diffusivity can be performed by comparing the experimental data and the temperature vs time curve computed from equation (6) or (18). An iterative algorithm has appeared in the literature based on comparing the ratio of temperatures $V = T(t_1)/T(t_2)$ in various times t_1 and t_2 for the ideal adiabatic model (equation (6)). In order to increase the precision and to ensure the validity of the considered model, thermal diffusivity was proposed to be calculated at several different times [1, 2].

Use of computers in experimental data analysis allows one to utilize more powerful data reduction methods. The objective of this section is to show how a least-squares fitting can be used for data reduction in the step-heating method.

Least-squares fitting can be applied, when we use the correct model and the experimental data are corrupted by a statistically uncorrelated error with the normal (Gaussian) distribution [8]. The least-squares method applied to step-heating experimental data is based on minimization of the merit function

$$R = \sum_{j=1}^{N} [T_j - T(t_j)]^2 \quad (19)$$

where T_j are the experimental data at the time points t_j, $T(t_j)$ are the corresponding temperatures calculated from the exact solution and N is the number of data points. We assume that the least-squares merit function R has one unique minimum, which corresponds to the true values of the parameters being estimated.

Let us consider the ideal adiabatic model: then we have two parameters to be estimated—thermal diffusivity a and the term $B = q/\rho ce$. The necessary conditions for the extreme of $R = R(a, B)$ are given by

$$\frac{\partial R(a,B)}{\partial a} = 0 \quad (20)$$

$$\frac{\partial R(a,B)}{\partial B} = 0. \quad (21)$$

A good deal of information is given by a normalized form of sensitivity coefficients, defined as

$$S_\beta = \beta\frac{\partial T}{\partial \beta} \quad (22)$$

which may be viewed as a change δT in the temperature T given by a small relative variation in the parameter $\beta[\delta T \cong S_\beta(\delta\beta/\beta)]$ [8, 9]. Figure 2 presents sensitivity coefficients S_a and S_B vs time computed from equation (6). The calculations were done for the sample parameters $a = 5\cdot10^{-5}$ m^2 s^{-1}, thickness $e = 30$ mm and $B = 0.01$ K s^{-1}. The shape of the sensitivity coefficients curves indicates that they are linearly independent. Thus a and B can be simultaneously estimated. Performing the operators indicated in equations (20) and (21) and after some manipulations, we obtain a set of algebraic equations for a and B in the form

$$\sum_{j=1}^{N} T_j\Theta_j(a)\sum_{j=1}^{N}\Theta_j(a)\frac{\partial\Theta_j(a)}{\partial a} - \sum_{j=1}^{N} T_j\frac{\partial\Theta_j(a)}{\partial a}\sum_{j=1}^{N}\Theta_j^2(a) = 0 \quad (23)$$

$$B = \frac{\sum_{j=1}^{N} T_j\Theta_j(a)}{\sum_{j=1}^{N}\Theta_j^2(a)} \quad (24)$$

where $\Theta_j(a)$ is defined as

$$\Theta_j(a) = t_j - \frac{e^2}{6a} - 2\sum_{n=1}^{\infty}(-1)^n\frac{\exp\left(-\left(\frac{n^2\pi^2}{e^2}\right)at_j\right)}{\left(\frac{n^2\pi^2}{e^2}\right)a}. \quad (25)$$

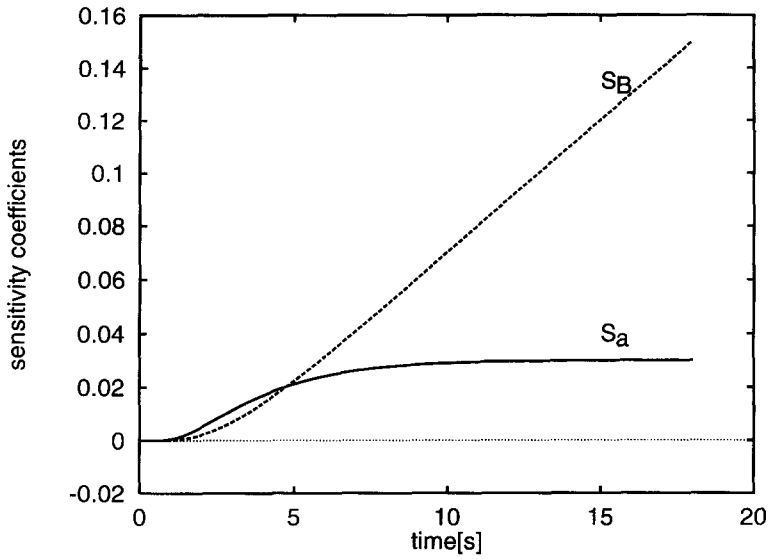

Fig. 2. Sensitivity coefficient curves for the adiabatic model (equation (6)).

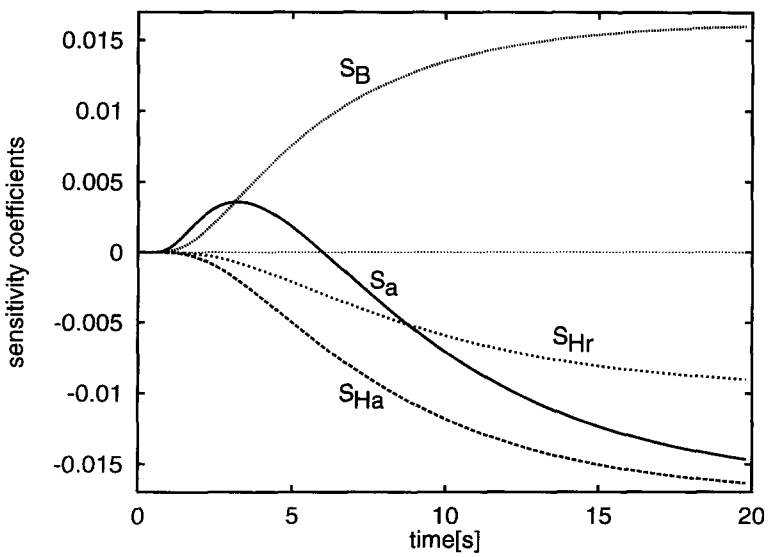

Fig. 3. Sensitivity coefficient curves for the model with heat losses (equation (18)).

The problem of finding the optimal value of thermal diffusivity is due to the linear dependence of temperature rise $T(e, t)$ on parameter B reduced to solving one algebraic equation (23). This equation has only one non-trivial root a^*, which corresponds to the desired optimal value of the thermal diffusivity. To search for this value we used an iterative algorithm, based on the standard numerical bisection method. The main advantage of this algorithm is that the calculation of thermal diffusivity is independent of knowledge of the parameter B, which is then calculated from equation (24).

Let us consider the model described by equation (18) if we assume the same heat losses from the front and rear surface, then we have only one axial Biot number $H_a = H_0 = H_e$. Figure 3 presents the sensitivity coefficients s_a, S_B, S_{H_a} and S_{H_r} vs time for this two-dimensional (2D) model. The curves were computed for $a = 5 \times 10^{-5}$ m^2 s^{-1}, $B = 0.01$ K s^{-1}, $H_a = 1.5$, $H_r = 0.1$, $e = 30$ mm and $r_s = 10$ mm. Similar shapes of the sensitivity curves for axial and radial Biot numbers H_a and H_r indicate that these curves are close to being linearly dependent. This means that the parameters H_a and H_r cannot be determined independently in the least-squares fitting process, therefore we assume that there are equal axial and radial heat transfer coefficients $h_a = h_r$ and radial and axial Biot numbers fulfil the condition

$$H_r = H_a \frac{r_s}{e}. \tag{26}$$

Thus data reduction consists of estimation of three unknown parameters, a, B and H_a. From the

conditions for a minimum of the function $R = R(a, B, H_a)$, we obtain the following algebraic equations

$$\sum_{j=1}^{N} T_j\Theta_j(a,H_a)\sum_{j=1}^{N}\Theta_j(a,H_a)\frac{\partial\Theta_j(a,H_a)}{\partial a} - \sum_{j=1}^{N} T_j\frac{\partial\Theta_j(a,H_a)}{\partial a}\sum_{j=1}^{N}\Theta_j^2(a,H_a)=0 \quad (27)$$

$$\sum_{j=1}^{N} T_j\Theta_j(a,H_a)\sum_{j=1}^{N}\Theta_j(a,H_a)\frac{\partial\Theta_j(a,H_a)}{\partial H_a} - \sum_{j=1}^{N} T_j\frac{\partial\Theta_j(a,H_a)}{\partial H_a}\sum_{j=1}^{N}\Theta_j^2(a,H_a)=0 \quad (28)$$

$$B = \frac{\sum_{j=1}^{N} T_j\Theta_j(a,H_a)}{\sum_{j=1}^{N}\Theta_j^2(a,H_a)} \quad (29)$$

with

$$\Theta_j(a,H_a) = \sum_{n=1}^{\infty} A_n(H_a,H_a,1)\sum_{m=1}^{\infty} B_m(H_r,0) \times \frac{1-\exp\left(-\left(\frac{u_n^2}{e^2}+\frac{w_m^2}{r_s^2}\right)at_j\right)}{a\left(\frac{u_n^2}{e^2}+\frac{w_m^2}{r_s^2}\right)}. \quad (30)$$

Because of the linear dependence of the temperature rise on the heat-flux term parameter B for these models, the problem of finding the desired optimal value of thermal diffusivity a and Biot number H_a is reduced to solving the two algebraic equations (27) and (28). Both these equations have only the non-trivial roots a^* and H_a^*, which correspond to the desired optimal values of thermal diffusivity and Biot number. These values can be found by an iterative root-finding technique. Parameter B is then calculated from equation (29).

3.2. *Levenberg–Marquardt method*

As the second method for data reduction we used the Levenberg–Marquardt (LM) χ^2 based procedure. Detailed description of this widely used technique, together with the source code is given in ref. [10]. We have already successfully used the method for processing data gained by the flash method [11]. Here we give only a brief description of the fitted equation and concentrate on the conditions, which must be fulfilled for successful use LM in the case of the step-heating method.

Fitting to the adiabatic model described by equation (6) is straightforward and therefore we do not pay further attention to it here. However, fitting in the case of heat losses from the sample surface governed by equation (18) is more complicated. We assume the same heat transfer coefficient from the front and rear surface $h_a = h_0 = h_e$. The fitted equation then has the form

$$T(a,B,H_a,H_r,T_{zl},t) = T_{zl} + B\sum_{n=1}^{r} A_n\sum_{m=1}^{s} B_m\frac{1-\exp\left(-\left(\frac{u_n^2}{e^2}+\frac{w_m^2}{r_s^2}\right)at\right)}{a\left(\frac{u_n^2}{e^2}+\frac{w_m^2}{r_s^2}\right)} \quad (31)$$

where

$$B = \frac{q}{\rho ce} \quad (32)$$

$$A_n = a_n(\cos(u_n)+\sin(u_n))$$

$$a_n = \frac{2u_n^2}{u_n^2+H_a^2+2H_a} \quad (33)$$

$$B_m = \frac{2H_r}{J_0(w_m)(w_m^2+H_r^2)} \quad (34)$$

and r and s are the number of summation terms. Parameters to be fitted are B, thermal diffusivity a, Biot numbers $H_a = H_0 = H_e$, H_r and a zero level correction term T_{zl}. The last parameter makes the method insensitive to a possible shift of the baseline.

In order to independently estimate all five parameters, they must be uncorrelated and their sensitivity coefficients linearly independent. The sensitivity coefficients for the case of heat losses are shown in Fig. 3. It is evident that a certain correlation between S_B, S_{H_a} and S_{H_r} occurs. To estimate the measure of the parameter correlation we used Pearson's linear correlation coefficient PLC [10], which lies between -1 and 1. Correlated (anticorrelated) distributions yield values close to 1 (-1), while uncorrelated distributions give PLC close to 0. Figure 4 shows the time dependence of PLC for all combinations of parameters. From the figure we can see that the parameters B, H_a and H_r are very strongly correlated for the extent of the measurement time, while the parameter a shows low correlation with the rest of the parameters in a certain time interval. For this particular case the optimal interval for determination of a is 6–7 s (Fig. 4).

Fitting to a model with nearly correlated parameters is not a trivial problem, since the minimum has shape of a flat valley, as we show further. Let us construct an error function D:

$$D(a,B,H_a,H_r) = \sum_{t_i}[T(a,B,H_a,H_r,t_i) - T(a^*,B^*,H_a^*,H_r^*,t_i)]^2 \quad (35)$$

which reflects a deviation of the response with parameters a, B, H_a and H_r from the curve with optimal parameters a^*, B^*, H_a^* and H_r^*. Two plots of this function are shown in Fig. 5. Figure 5(a) shows $D(a, B, H_a^*, H_r^*)$ for a and B varying in a close neighbourhood

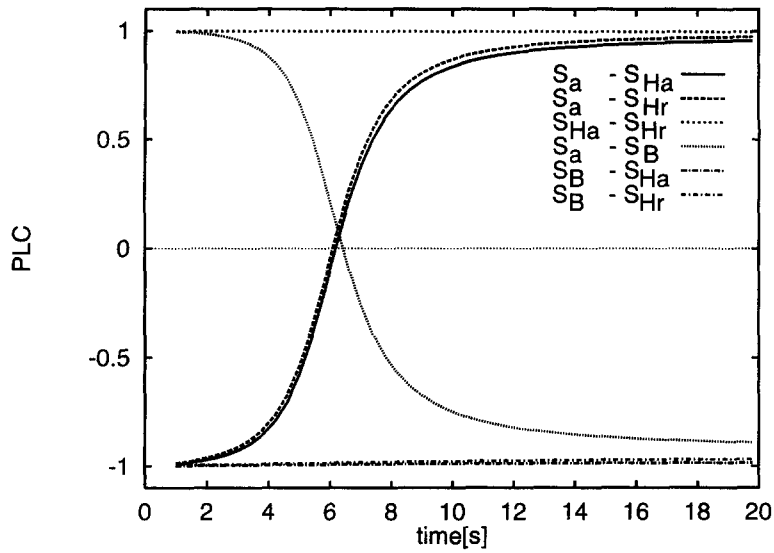

Fig. 4. Pearson's correlation coefficient as a function of measurement time for all the combinations of fitted parameters.

of the point $[a^*, B^*]$. The closed contours show that D has the unambiguous minimum in $[a^*, B^*]$. The case of $D(a^*, B^*, H_a, H_r)$ near the point $[H_a^*, H_r^*]$ is different. The parallel contours of Fig. 5(b) show that there is a continuous line of minimum points instead of a single one. Since the iteration process can stop at each of these points, depending on the initial guess, only the ratio H_a/H_r can be estimated, i.e. various combinations of H_a and H_r with the same ratio lead to the same value of the thermal diffusivity. The advantage of the LM method is its good behaviour in such near-degeneracy cases (where other methods may fail), but at the cost of a tendency to wander around the minimum and thus finding the optimal parameters is time consuming [10].

3.3. *Optimization of the LM fitting process*

Computational complexity of the LM method can be expressed as $O(r \cdot s \cdot i \cdot p)$, where $r \cdot s$ is number of sum terms in equation (31) taken into account, i is number of iterations and p is number of fitted parameters. Proper choice of r and s can significantly speed up the iterative process.

For the purpose of a time analysis of the problem let us express the nmth exponential of equation (31) as

$$T_{nm} = \frac{q}{\rho c e} k_{nm}\left(1 - \exp\left(-\frac{t}{\tau_{nm}}\right)\right), \qquad (36)$$

where $\tau_{nm} = 1/\{a[(u_n^2/e^2) + (w_{ml}^2/r_s^2)]\}$ is its time constant and $k_{nm} = A_n B_m \tau_{nm}$. Table 1 shows k_{nm} and τ_{nm} for some selected values of n and m. We see that k_{nm} is falling much faster with growing m than with n (e.g. $k_{1,5} < k_{100,1}$) and therefore it is possible to take fewer m-members into account.

Contribution of each exponential T_{nm} is after some time ($\tau \approx 3\tau_{nm}$) constant and equal to $A_n B_m \tau_{nm}$. If we neglect all terms with $\tau < t/s$, where t is measurement duration and s is number of samples, the error is constant over the whole time interval and results in a shift of the curve. However, this approximation also results in an error in the determination of the curve parameters. Since the deviation is constant, we can improve the precision by fitting the zero level term T_{zl} of the working equation (equation (31)). Table 2 shows results of fitting the models with different numbers of r and s terms to a simulated noiseless response

Table 1. τ_{nm} and k_{nm} for selected n and m (see equation (36))

n	τ_{n1} [s]	k_{n1} [s]	τ_{n2} [s]	k_{n2} [s]	τ_{n5} [s]	k_{n5} [s]
1	4.352319	2.781931	0.132045	−0.002748	0.011237	0.000036
2	1.071038	−1.627136	0.120815	0.005976	0.011149	−0.000085
5	0.108704	0.215835	0.060449	−0.003908	0.010208	0.000102
10	0.022300	−0.045400	0.019162	0.001270	0.007485	−0.000077
20	0.005041	−0.010315	0.004861	0.000324	0.003483	−0.000036
50	0.000759	−0.001556	0.000755	0.000050	0.000711	−0.000007
100	0.000186	−0.000381	0.000186	0.000012	0.000183	−0.000002

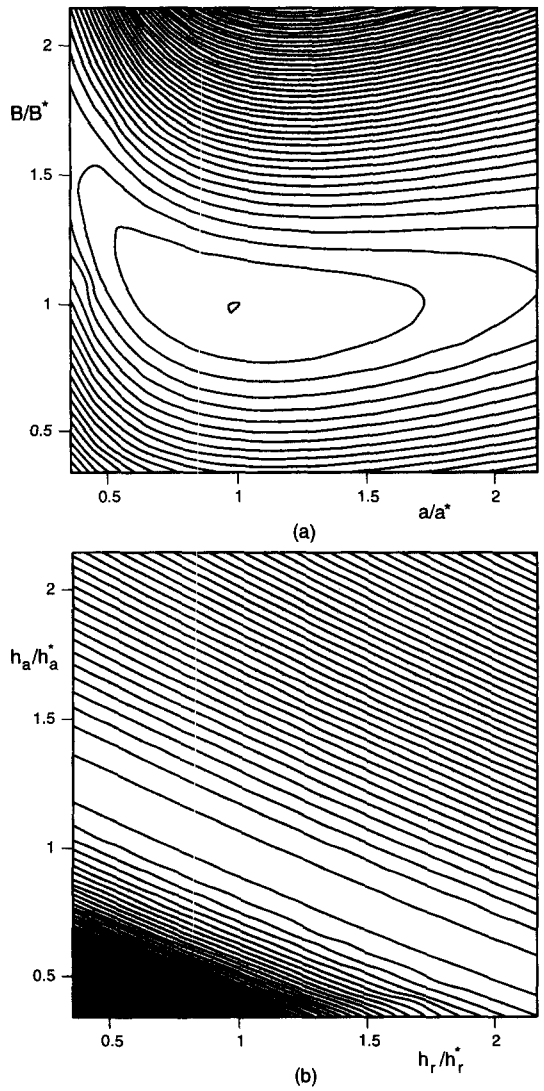

Fig. 5. Isocontours of the D function near point $[a^*, B^*, H_a^*, H_r^*]$ (see equation (35)).

with $r = 100$ and $s = 10$. In the third column are values of a obtained without fitting the T_{zl} term, while in the fourth column are results obtained with this term. We see that in the worst case, for $r = 30$ and $s = 1$, the error of the result is nearly eight times lower with fitting of the T_{zl} term. However, errors increase with further decrease of r, since the condition $\tau < t/s$ is no longer fulfilled for all discarded exponentials.

The last column of Table 2 shows duration of the fitting procedure. We can see that even a 15-fold speed up can be reached (30×1 vs 100×10 terms) without a significant lose of precision.

4. RESULTS AND DISCUSSION

In the previous sections we have described the models used for determination of the thermal diffusivity by the step-heating method. Three possible cases are discussed. The case when no heat losses from the sample surface occur (equation (6)) is straightforward. From Fig. 2 we can see that sensitivities for both fitted parameters a and B are linearly independent in the whole time region. However, the conditions of this model are rarely fulfilled in a real experiment.

The most common case is that of equal axial and radial heat transfer coefficients. The third possibility, with different axial and radial heat losses, may occur when axial and radial surfaces of a sample are not of the same roughness.

Sensitivity analysis of equation (18) depicted in Fig. 3 shows that there is a certain linear dependence of the sensitivity coefficients S_B, S_{H_a} and S_{H_r}. Pearson's test [10] confirms a strong correlation of these parameters. However, the thermal diffusivity is not correlated with other parameters in a certain time region (Fig. 4).

We have proposed the use of two data reduction methods for evaluation of the thermal diffusivity. The first is the least-squares fitting algorithm, which can be used in the case where heat losses are negligible (equation (6)) or for samples with equal axial and radial heat transfer coefficients. In this case only two linearly independent parameters a and H_a are fitted. Parameter B is then calculated from equation (24) and parameter H_r from the condition of equal axial and radial heat transfer coefficients. The main advantage of this technique is that the calculation of the thermal diffusivity is independent of knowledge of the parameter B.

As the second method we used the Levenberg–Marquardt method. Fitted parameters are a, B, H_a, H_r and a zero level correction term T_{zl}, which makes the method insensitive to a possible shift of the baseline.

Table 2. Fitting noiseless simulated response with different r and s with (a_{fitzl}) and without (a_{fit}) the zero level correction term T_{zl}. True thermal diffusivity $a = 5 \times 10^{-5}$ m^2 s^{-1}

r	s	a_{fit} [10^{-5} m^2 s^{-1}]	a_{fitzl} [10^{-5} m^2 s^{-1}]	Fit time [s]
100	10	5.0	5.0	115
100	2	4.999	4.999	26
50	10	4.961	4.997	65
30	1	5.089	5.012	8.5

Table 3. Thermal diffusivity and its deviation gained from simulated data by LM method. The simulated noise was 0.5%, 1% and 5%, respectively

Time [s]	$a_{0.5\%}$ [10^{-5} m^2 s^{-1}]	$a_{1\%}$ [10^{-5} m^2 s^{-1}]	$a_{5\%}$ [10^{-5} m^2 s^{-1}]
4	5.133±0.312	5.327±0.680	5.362±0.680
7	5.028±0.160	5.066±0.220	5.160±0.390
14	5.082±0.103	5.115±0.173	5.250±0.377
20	5.081±0.105	5.125±0.173	5.244±0.326

Table 4. Thermal diffusivity a of stainless steel and graphite using both LS and LM method and compared with literature data

Sample	a_{recomm} [10^{-5} m^2 s^{-1}]	a_{LS} [10^{-5} m^2 s^{-1}]	a_{LM} [10^{-5} m^2 s^{-1}]
Graphite	76.6	72.47±0.11	73.4±0.04
Steel	3.77	3.56±0.18	3.66±0.10

The main advantage of this procedure is the possibility of fitting to equation (18) with different axial and radial heat transfer coefficients. However, processing of such a near-degenerated case with nearly correlated parameters B, H_a and H_r (as was shown above) results in wandering around the minimum performing tenths of iterations and is very time consuming. The time analysis in the previous sections shows how we can optimize the number of sum terms in equation (31) and thus reduce the fitting time significantly.

To test the reliability of the LM method applied to the case with different axial and radial heat losses we generated a set of 50 Monte-Carlo curves with three levels of additive noise and four different measurement times. Table 3 shows the obtained mean thermal diffusivity and its deviation by fitting to the complex model (equation (31)). All the simulations were done for the sample parameters mentioned in the previous section. From Table 3 we see that we get the most precise value of thermal diffusivity by fitting the response with a duration of 7 s, i.e. in the region where a is not correlated with other parameters. Thus it is evident that the precision of the fitting process depends on the duration of the measurement. With increasing noise the precision of estimating the thermal diffusivity decreases. Its standard deviation increases even more rapidly as a result of the flat valley of minimum points depicted in Fig. 5(b).

The two data reduction algorithms were also tested on experimental data obtained from graphite (diameter 12.8 mm, length 27 mm) and stainless steel (diameter 12 mm, length 6 mm) samples. The results gained from both methods together with the values given by NIST [12, 13] are given in Table 4. Although the measurements were done at room temperature, the graphite sample exhibits heat losses and the difference between the values gained by using the correct model and adiabatic model was approximately 20%. Thus for processing the measured data we use the model with equal axial and radial heat losses.

5. CONCLUSION

The step-heating technique is a suitable tool for determining the thermal diffusivity of materials. It is an extension of the well-known flash method made by exchanging the source of the light pulse (laser, flash lamp) for a source of constant heat flux (halogen lamp). In this paper we have outlined use of two data reduction methods for this technique. Both are based on a non-linear fitting to the equation which involves heat loss terms. The sensitivity analysis shows that the duration of measurement can increase the precision of the results of data reduction. The proposed fitting algorithms were tested on both simulated and experimental data and the analysis above shows that they can be successfully used to process data gained by the step-heating technique.

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