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The least square method in the determination of thermal diffusivity using a flash method

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Résumé. — L'article décrit une méthode d'ajustement des données expérimentales au modèle de Parker *et al.* [1] utilisé dans les mesures de diffusivité thermique par méthode flash laser. La méthode présentée est adaptée au cas d'ajustement où les fréquences de bruit sont dans la bande spectrale du signal transitoire, ce qui rend difficile voire impossible le filtrage par filtre passe-bas. La méthode de minimisation numérique de la fonction des moindres carrés, proposée par Booth et Booth [2], est utilisée et un exemple de son application est présenté.

Abstract. — This paper is devoted to the description of a fitting method of experimental data for the model of Parker *et al.* [1] used in thermal diffusivity measurements by a laser flash method. The presented method is adapted to the fitting case when the noise frequencies are in the spectral band of the transient signal, thus making the filtering by a low-pass band filter difficult if not impossible. The method of numerical minimization of the least square function shown by Booth and Booth [2] is used and an example of its application is presented.

1. Introduction.

The laser flash method is actually one of the most popular to measure thermal diffusivity of solids. The oldest and simplest model on which the diffusivity measurements is based, relates the temperature evolution with time of the sample rear face to its thickness L (the sample is supposed to be an infinite slab) and to its diffusivity a by

$$T(L, t) = T_{\max} \left[1 + 2 \sum_{n=1}^{\infty} (-1)^n \exp\left(-\frac{an^2 \pi^2 t}{L^2}\right) \right] \quad (1)$$

where $T_{\max}$ is the sample back face maximum temperature. Of course the front face of the sample ($x = 0$) is supposed to be submitted to an instantaneous pulse of energy.

The Fourier transform of such a signal, depicted in figure 1, shows that the frequency spectrum is in

the low frequencies range. Of course the highest frequency values of the Fourier transform depends on the tested sample parameters such as its thickness (thinner is the sample higher are the frequencies for the same value of the transform) and its diffusivity (an increase of the diffusivity corresponds to a shift of the spectrum to higher frequencies, see e.g. [3, 4]).

Fig. 1. — Digital Fourier transform of the transient signal given by equation (1) calculated up to the time $5 \cdot t_{0.5}$ ($t_{0.5}$ is the time corresponding to half-maximum signal) for the following parameters : $a = 0.0493 \text{ cm}^2/\text{s}$ and $L = 0.1362 \text{ cm}$, the sampling period being $5 t_{0.5}/100$.

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Any diffusivity experiment which uses an infrared detector is accompanied by noises perturbing the transient signal. These noises are due to the statistical nature of the radiation detection but sometimes also to the electromagnetic perturbation of the high resistivity i.r. detector by external radiation source such as laser charge, electrical supply of the furnace etc. Surely the electrical grounding should limit or even eliminate such a perturbation however it is not always possible especially when measurements are performed at low temperature. The second type of noises is shown in figure 2 where the Fourier transform of the transient signal exhibits perturbations at 50 Hz and harmonics. Such perturbations make impossible the use of a low-pass band filter, such as the one discussed recently because the noises are in the spectral range of the transient signal containing the requested information. That is why we have tried to use a least squares method (LS) to fit the noisy experimental data to the model described by equation (1).

Fig. 2. — Digital Fourier transform of the transient signal obtained during the measurements of the diffusivity of a plasma sprayed NiAl sample having a thickness $L = 0.136$ cm and a mean temperature of 612 K (the sampling period is of about 0.6 ms).

2. Data fitting by least squares method.

Formally the LS fitting method can be applied when three conditions are fulfilled [5] :

- 1° The model has a correct form,
- 2° The data are typical,
- 3° The data are statistically uncorrelated i.e. the perturbations have a Gaussian distribution.

The condition 1° is fulfilled : equation (1) is valid i.e. without any heat losses from the sample and when no finite pulse correction is needed. As the discussed low-frequencies perturbations are more important at low temperature the heat losses are not a problem. The finite pulse correction might be needed, but in such a case equation (1) should be transformed into the adequate form for a finite pulse duration with a given shape. The 2° condition is easily fulfilled if the expe-

periment is made correctly. The third one is violated. The noises are not statistically uncorrelated and the perturbation distribution, as far as low frequency noise is concerned, is not Gaussian. Having no formal background about this point we will show through our experimental data that the LS function has a minimum and that this minimum corresponds to the true diffusivity.

The LS function has the following expression :

$$R(A, B) = \sum_{i=1}^N \left\{ U_i - A \left[1 + 2 \sum_{n=1}^K (-1)^n \times \exp(-B t_i n^2) \right] \right\}^2 \quad (2)$$

where U_i are the voltage points as given by the experimental set (i.r. detector, preamplifier, amplifier) and corresponding to the time point t_i . A is an adjustable parameter corresponding to the maximum voltage, B is a second experimental parameter such as :

$$B \equiv \frac{a\pi^2}{L^2} \quad (3)$$

N is the number of experimental points and K is the higher limit of the sum given by equation (1). For the calculations $K = 10$ was taken.

Fig. 3. — Sketch of the position of the points used in the iterative procedure to minimize the LS function.

The minimum of the LS function has been searched using the iterative method proposed by Booth and Booth [2]. Starting from the point (A_0, B_0) , a better approximation of the coordinates of the minimum is obtained from the following equations :

$$A_1 = \frac{[R_4 - R_1] \delta^2 y - [R_5 - R_3] \delta xy}{\Delta} \cdot h$$

$$B_1 = \frac{[R_5 - R_3] \delta^2 x - [R_4 - R_1] \delta xy}{\Delta} \cdot k$$

where h and k are the steps shown in figure 3,

$$\delta^2x = R_1 - 2 R_0 + R_4 ,$$

$$\delta^2y = R_3 - 2 R_0 + R_5 ,$$

$$\delta xy = R_0 - R_1 + R_2 - R_3 ,$$

$$\Delta = 2[\delta^2x.\delta^2y - (\delta xy)^2] .$$

To demonstrate the use of this LS method for a very noisy signal we develop in the following the results obtained for the diffusivity measurements with the signal corresponding to the evolution with time of the rear face temperature of a plasma sprayed sample of

Fig. 4. — Experimental evolution of the back face temperature of a NiAl sprayed sample which thickness is $L = 0.136$ cm and mean temperature 612 K.

NiAl [6] which thickness is $L = 0.136$ cm and which mean temperature in the furnace is 612 K. The first part of the curve up to the laser flash is obviously to be eliminated. In the second part we have choosen only the points distant from each other by a sampling period $T_s = 1/\nu_{0.1}$ ($\nu_{0.1}$ being the frequency corres-

Fig. 5. — Evolution on the LS function vs. adjusted parameters.

a unit in $\text{cm}^2/\text{s} = 0.03A$ in $V = 1.5DA$ in $V = 0.05Da$ in $\text{cm}^2/\text{s} = 0.01$		
No of iteration = 1a in $\text{cm}^2/\text{s} = 0.0422918495$	Maximal voltage in $V = 2.8061829$	Least square function = 177.898097
No of iteration = 2a in $\text{cm}^2/\text{s} = 0.0497094234$	Maximal voltage in $V = 2.44684993$	Least square function = 33.8261335
No of iteration = 3a in $\text{cm}^2/\text{s} = 0.0494328681$	Maximal voltage in $V = 2.471596$	Least square function = 27.2313216
No of iteration = 4a in $\text{cm}^2/\text{s} = 0.0493243879$	Maximal voltage in $V = 2.47290541$	Least square function = 27.1460148
No of iteration = 5a in $\text{cm}^2/\text{s} = 0.0493256006$	Maximal voltage in $V = 2.47300565$	Least square function = 27.1339802
No of iteration = 6a in $\text{cm}^2/\text{s} = 0.0493250539$	Maximal voltage in $V = 2.47300939$	Least square function = 27.1341105
No of iteration = 7a in $\text{cm}^2/\text{s} = 0.049325071$	Maximal voltage in $V = 2.47300979$	Least square function = 27.1340512
X-axis division in s = 0.0290859375	Y-axis division in $V = 2$	
DATE = 12/04/84 10 H 51 SAMPLE = NIALA2	Sampling period in s = 1.9921875E-03

Fig. 6. — Iterative procedure and final fitting of the experimental data to the model curve.

ponding to one tenth of maximum value of the DFT cf. Fig. 1). Finally only the heating part of the curve (see Δt in Fig. 4) has been analysed because it allows to fit the data to the model.

For these experimental points we have drawn in figure 5 the least squares function (R) vs. the adjusted parameters : diffusivity (a) and maximum amplitude of the signal (A) expressed in volts. The least square function found for the 100 points, corresponding to a diffusivity in the range 0.01 to 0.1 cm²/s and an amplitude in the range 1.5 to 3.5 volts, is represented as bars orthogonal to the surface of adjusted parameters. The function has a clearly pronounced minimum. This graphical estimation permits to find, using the minimization procedure described above, the diffusivity after seven successive iterations (Fig. 6).

To verify the fitting precision, the above procedure was used to analyse non-perturbed data. The diffu-

sivity in this case was determined using the well known formula :

$$a = \frac{1.38 L^2}{\pi^2 t_{0.5}}, \quad (4)$$

$t_{0.5}$ being the time corresponding to the half of the maximum temperature of the sample back face. The obtained diffusivity was within 5 % in good agreement with the value found by the LS method.

For practical use this LS procedure was developed with a small microcomputer (16 bits, 64 kbytes). The time necessary to fit one transient signal to the diffusivity by this LS method varies from half an hour to two hours and depends on the number of experimental points (typically 100 to 200) and on the precision of first approximation.

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