THERMAL DIFFUSIVITY MEASUREMENTS BY THE LASER PULSE METHOD USING GAUSS ESTIMATION PROCEDURE WITH EXTENDED NUMBER OF PARAMETERS

by

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Paper presents application of the Gauss estimation procedure in measuring thermal diffusivity using the laser pulse method. Unlike the well-known direct procedure originally established by Parker et al. and its variations, this one belongs to the so-called inverse technique, which makes use of the complete measured signal. Paper describes how to achieve minimum deviation between estimated and experimental curves and to obtain more accurate results proposing an estimation procedure with extended number of parameters in three steps.

Introduction

The original laser pulse method of measuring thermal diffusivity [1] assumes ideal boundary and initial conditions, *i.e.* zero heat loss, infinitely short laser pulse, and uniform heating of the sample face. Simplicity of the method is marred in practice by difficulties of realising these idealised conditions. Thanks to theoretical and experimental work of many researchers, the original concept has been gradually improved to take into account real experimental conditions. Review of these contributions is given in [2].

Corrections following publication of the original work [1], were virtually all directed toward correcting measured half-rise time of the transient. In mathematical sense these procedures are known as direct approaches to thermal diffusivity determination. Possibilities of modern data acquisition and data reduction systems, however, offer much more than procedures limited to the analysis of the half-rise time, or few points more. Distinct advantages offered by the inverse method should be used instead, as for thermal diffusivity identification it relies on the complete transient response. Due to minimum deviation between theoretical and experimental curve achieved in this approach it results in a better reliability and efficacy of thermal diffusivity measurement.

For the flash method it is convenient to use the parameter estimation procedure, which enables simultaneous determination of more than one parameter from the same temperature response. Possibilities of such determinations are influenced by the sensitivity coefficient of each single parameter. Sequential analysis estimating beside thermal

diffusivity the maximum temperature rise has been applied [3]. The Gauss estimation procedure, however, could extend estimation to additional parameters, like the Biot number, laser pulse-width, and the onset of the temperature response.

Gauss estimation procedure

Generally, all approaches are based on minimising the difference between the measured values and corresponding values obtained by the mathematical model. Among several of them the Gauss parameter estimation procedure [4, 5] has been preferred. It is used in a non-linear case of estimation, *i. e.* when sensitivity coefficients are parameter dependent. For example, sensitivity coefficient of thermal diffusivity in the laser pulse method is strongly non-linear dependent on thermal diffusivity. This approach is also attractive because it is relatively simple and because it specifies direction and size of the parameter vector corrections.

Some parameters of the applied model can be estimated easier, some not. There are criteria that one must comply with if reliable results for a given parameter [6] are wished. In that sense, sensitivity coefficients play the most important role, giving an information about estimation possibilities of desired parameter. They are defined by

$$\mathbf{X} = \begin{bmatrix} \partial T_1/\partial b_1 & \cdots & \partial T_1/\partial b_p \\ \vdots & & \vdots \\ \partial T_n/\partial b_1 & \cdots & \partial T_n/\partial b_p \end{bmatrix} \tag{1}$$

where T_j is a value calculated from the model at the time τ_j ($j = 1, ..., n$), n is the number of measured values, b_i is the i^{th} parameter ($i = 1, ..., p$), and p is the number of parameters for estimation. Sensitivity coefficients are analysed for each model in particular, and also for different parameter values of the same model. Usually their normalised values are analysed, defined as

$$X^*_{ji} = b_i \frac{\partial T_j}{\partial b_i} \tag{2}$$

Sensitivity coefficients given in Eq. (2) are compared to indicate parameters which might be simultaneously or separately estimated with desired accuracy. The general criterion is that these coefficients should be linearly independent as much as possible, and having high and mutually comparable values.

In practice, sensitivity coefficients are usually very complex functions of parameters. Therefore, their values may be numerically calculated using an approximate formula

$$X^*_{ji} = b_i \frac{\partial T_j}{\partial b_i} \approx b_i \frac{T_j(b_1, b_2 ... b_i + \delta b_i ... b_p) - T_j(b_1, b_2 ... b_i - \delta b_i ... b_p)}{2\delta b_i} \tag{3}$$

where δb_i is of the order of $10^{-3} b_i$ or $10^{-4} b_i$.

Starting from the *maximum a posteriori* (MAP) criteria [5] and linearising the temperature **T** using the Taylor series, the Gauss iterative equation has the following form:

$$\mathbf{b}^{(k+1)} = \mathbf{b}^{(k)} + \left[\mathbf{X}^{T(k)}\mathbf{W}\mathbf{X}^{(k)} + \mathbf{U}\right]^{-1} \cdot \left\{\mathbf{X}^{T(k)}\mathbf{W}\left[\mathbf{Y} - \mathbf{T}^{(k)}(\mathbf{b}^{(k)})\right] + \mathbf{U}\left[\mu - \mathbf{b}^{(k)}\right]\right\} \tag{4}$$

where **Y** is the matrix of measured values [$n\times1$], **T** is the matrix of calculated values from the model [$n\times1$], **b** is the matrix of parameters [$p\times1$], μ is the matrix of *a priori* parametric values [$p\times1$], **W** is the variance-covariance matrix of measured values [$n\times n$], and **U** is the variance-covariance matrix of parameters *a priori* [$p\times p$]. The variance-covariance matrix has a form Ψ^{-1}, where diagonal elements of matrix Ψ are variances of each measured value, since other elements represent a degree of correlation among measured values. When the correlation is absent, the matrix Ψ is diagonal. The iterative procedure (4) should be ceased when the following condition is satisfied:

$$\frac{b_i^{(k+1)} - b_i^{(k)}}{\left|b_i^{(k)}\right| + \xi} < \vartheta \tag{5}$$

i being 1 to p, where ϑ is a number of order 10^{-4}, and $\xi \leq 10^{-10}$ to avoid dividing by zero.

Standard deviation of estimated parameters **b**, could be calculated from the approximated expression:

$$\sigma^2_{\bar{\mathbf{b}}} \approx \frac{1}{np}\left[\mathbf{Y} - \mathbf{T}(\bar{\mathbf{b}})\right]^T\left[\mathbf{Y} - \mathbf{T}(\bar{\mathbf{b}})\right] \cdot \mathrm{diag}(\mathbf{X}^T\mathbf{X})^{-1} \tag{6}$$

Beside this standard deviation value that represents a criterion of estimation accuracy, one could define the normalised sum of relative differences between estimated theoretical curve and the experimental response as another convenient criterion for the reliability of estimated parameters. This sum can be expressed as:

$$s(T) = \frac{1}{n}\sum_{j=1}^{n}\bar{\varepsilon}_j^2(T) \tag{7}$$

with

$$\bar{\varepsilon}_j(T) = \frac{T_j(\bar{\mathbf{b}}) - Y_j}{\max(Y)} \tag{8}$$

Estimated parameters are more reliable if the sum $s(T)$ has a smaller value. For better insight into exactness of estimation, it is convenient to compare the sum (7) with a new sum $s(P)$ calculated with differences $\bar{\varepsilon}(P)$ instead of $\bar{\varepsilon}(T)$. **P** is a [$n\times1$] matrix of values which represent a polynomial curve of a high degree (8^{th} or 9^{th}) fitted through the experimental signal in the estimation range. Supposing that polynomial P is the best

fit, sum $s(P)$ is the minimum possible, so previous criterion could be rather a ratio between sums $s(T)$ and $s(P)$, *i.e.*

$$\eta = \frac{s(T)}{s(P)} \tag{9}$$

It follows that agreement between theoretical and experimental curves is better and estimated values are more reliable when the ratio η is closer to 1. In practice both described criteria, (6) and (9), should to be used.

Estimation procedure applied to the laser pulse method

Theoretical model

In the laser pulse method the model is represented by the temperature response of the sample rear side. Let the laser pulse with a short duration τ_p be absorbed uniformly in a very thin layer of the front side of the specimen. If one measures transient temperature of the whole specimen rear side, mathematical expression of the temperature response is [7, 8]:

$$T(\tau) = 2T_m\left[\int_0^{\tau_p} f(\tau,\tau_p)d\tau\right]^{-1} \sum_{n=1}^{+\infty}\beta_n \frac{\beta_n\cos\beta_n + \mathrm{Bi}_L\sin\beta_n}{\beta_n^2 + \mathrm{Bi}_L^2 + 2\mathrm{Bi}_L} \sum_{n=1}^{+\infty}\frac{4\mathrm{Bi}_R^2}{Z_i^2(Z_i^2+\mathrm{Bi}_r^2)} \cdot$$
$$\cdot\exp\left[-a\tau\left(\frac{Z_i^2}{R^2}+\frac{\beta_n^2}{L^2}\right)\right]\int_0^{\tau_p}\exp\left[a\tau\left(\frac{Z_i^2}{R^2}+\frac{\beta_n^2}{L^2}\right)\right]f(\tau,\tau_p)\mathrm{d}\tau \tag{10}$$

where τ is time ($\tau > \tau_p$), $\mathrm{Bi}_L = h_L L/\lambda$ and $\mathrm{Bi}_R = h_R R/\lambda$ are the Biot numbers for base and lateral sample sides respectively, h_L and h_R are the radiative heat transfer coefficients (axial and lateral heat losses), L and R are the sample thickness and radius respectively, λ is thermal conductivity, a is thermal diffusivity, and $f(\tau, \tau_p)$ is a dimensionless function describing the laser pulse as a function of time. Because experiments are usually performed under vacuum conditions, convective and conductive heat losses from the sample are neglected, and the only important mode of heat exchange is radiative heat transfer. T_m is equal to $Q/(L\rho C_p)$, and represents the maximum temperature rise when $h_L = h_R = 0$. Q is absorbed laser energy per square meter, C_p is specific heat, and ρ is density. Coefficients β_n and Z_i ($n, i = 1, 2, 3\ldots$) are positive roots of corresponding transcendental equations [7].

The temperature response at short times ($\tau < \tau_p$) could be calculated from the same expression (10), but with uppers limits τ instead of τ_p in both integrals.

Estimation possibilities

Theoretically all constants in (10) could be treated as parameters for estimation. However, by increasing number of parameters for estimation accuracy is reduced and vice-versa. In practice, the sample thickness L and radius R can be measured with high accuracy, so there is no need for their estimation. Likewise, for a given laser type the laser pulse-width τ_p could be also known accurately, or determined in advance. When, however, the laser pulse width varies from pulse to pulse, this parameter should be also estimated.

In practice both base and lateral heat losses can be considered to be the same, so $\mathrm{Bi}_L = \mathrm{Bi}_R \cdot R/L = \mathrm{Bi}$. It can be shown that sensitivity coefficient of Bi is greater than sensitivity coefficients of separate Biot numbers, giving thus better possibility for the estimation of Bi.

Figure 1 shows selected values of sensitivity coefficients X^* of four parameters figuring in Eq. (10). Sensitivity coefficients were calculated using Eq. (3), for $a = 6.5 \cdot 10^{-5}$ m^2/s and rectangular pulse function $f(\tau, \tau_p)$. It shows that absolute values of sensitivity coefficients $X^*_{\tau_p}$ and X^*_{Bi} are one or two orders less than these of a and T_m. This difference

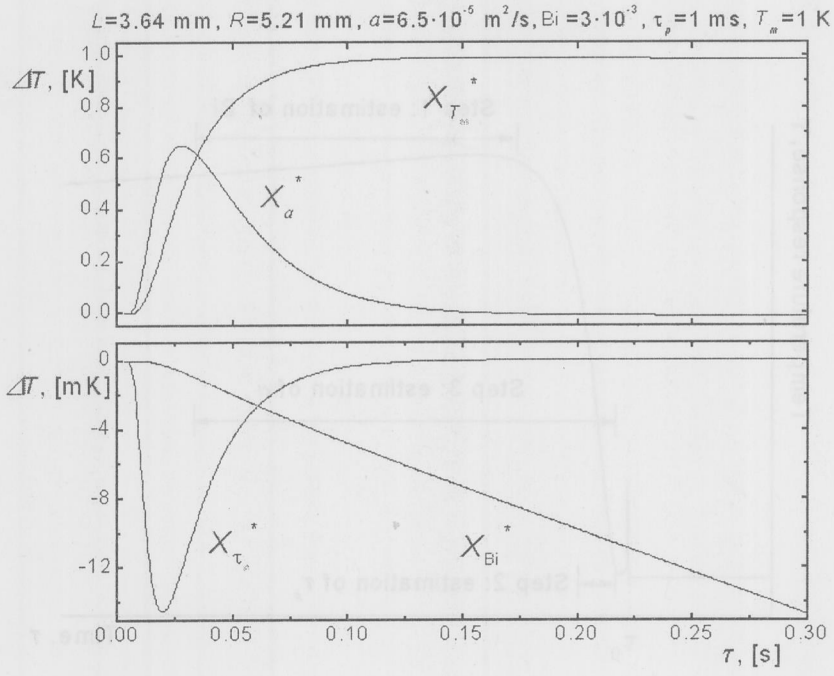

Figure1. Normalized sensitivity coefficients of parameters a, T_m, Bi, and τ_p

complicates simultaneous estimation of these four parameters. However, sensitivity coefficients of all four parameters are linearly independent within certain time range, especially in the period of temperature rise. This allows their simultaneous estimation despite considerable differences in absolute values, particularly when high accuracy of parameters τ_p and Bi is not required.

Since parameter T_m affects the model only as a multiple factor its sensitivity coefficient has the same form and values as the temperature response. Therefore, the parameter T_m is best estimated around the maximum of the signal. Further, Fig. 1 shows that the most preferable estimation range is the descending part of the signal for Bi, the initial portion of the signal for parameter τ_p, and the ascending part for parameter a. According to this, one could establish the following procedure for accurate thermal diffusivity measurement simultaneously with other parameters.

Procedure for thermal diffusivity measurement

According to the above analysis, derivation of thermal diffusivity from re-scaled experimental signal should be effected in the following three steps (Fig. 2):

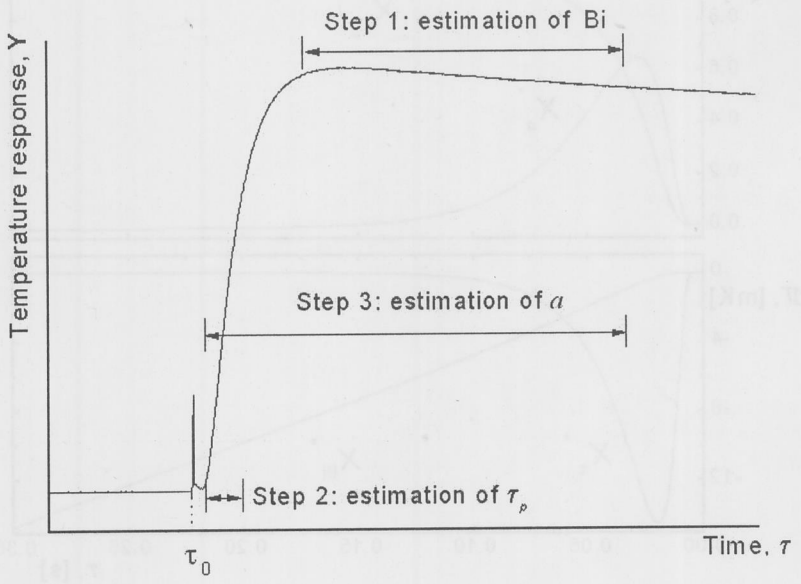

Figure 2. Three-step parameter estimation procedure applied to temperature response

(1) The first involves estimation of Bi, from the time just before its maximum to a certain value along its descent. This should be effected simultaneously with parameters a, T_m, and τ_p, with their *a priori* starting values. Estimation of these three parameters is only auxiliary, so in this step their estimated values may not be exact. This "auxiliary estimation" improves fitting between theoretical and experimental curves. Estimation of Bi alone would not give a good fit due to its small influence on the temperature response, even in the range after the maximum.

(2) The second step refers to estimation of the parameter τ_p in the initial portion of the signal rise, simultaneously with a and T_m as auxiliary parameters. Due to small influence of the parameter Bi on that portion of the signal, value Bi is fixed at the value estimated in the first step.

(3) The third step is estimation of thermal diffusivity, a, with auxiliary parameter T_m. Parameters Bi and τ_p are fixed at the values estimated in previous steps. Estimation should be effected in the time scale that corresponds to both ascending and descending portions of the temperature response.

Comparison of typical experimental and theoretical curves calculated from Eq. (10) and their differences in the process of estimation procedure is shown on Figs. 3, 4, and 5. After several iterations good agreement between the curves in all 3 steps could be seen, suggesting that estimated values (Bi, τ_p or a) should be very close to the real ones.

In this estimation procedure T_m always serves as an auxiliary parameter. It improves the fitting process leading to lesser deviation between theoretical and experimental curves, *i.e.* to the lesser value of η. Its accurate value should be rather calculated directly from the temperature response itself.

Uncertainty of estimated parameters

The parameter estimation procedure in three steps helps to obtain the highest accuracy and reliability of thermal diffusivity. While in the first two steps one obtains accurate values of Bi and τ_p, in step 3 one gets the final value of a.

The highest measured uncertainty Δ of each parameter is three times its standard deviation calculated from Eq. (6). In most cases Δ of estimated thermal diffusivity, Δ_a, obtained in step 3, amount to about 0.1%. To this value one has to add the uncertainty L of parameter Δ_L. Uncertainty Δ_R of the sample radius R can be neglected due to its small contribution to overall accuracy.

Uncertainty of other parameters depends on the portion of the signal that is used in estimation. In step 1 parameter Bi is estimated with the high accuracy and its value is closest to that in reality so it is used as fixed in steps 2 and 3. While some methods in the calculation of Bi deal with limited number of signal points, for example [9], this procedure suggests using of complete descending part of the experimental signal ensuring thus high accuracy of the obtained value of Bi. In all examined cases uncertainty Δ_{Bi} fell within the range from 0.4% to the maximum of 5%, depending on noise level and deviations of the experimental response.

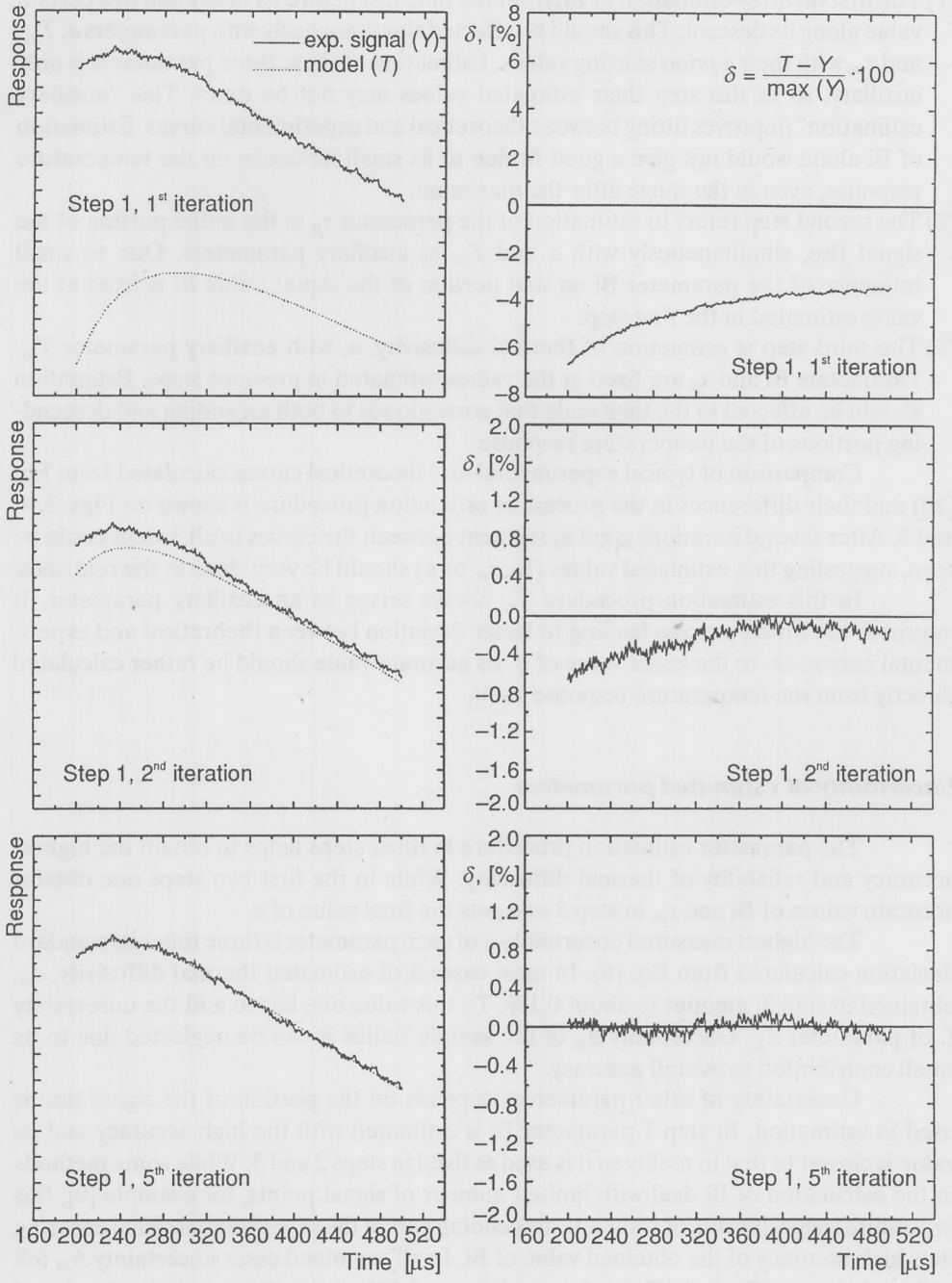

Figure 3. Difference between theoretical and experimental curves in the process of the estimation procedure in step 1

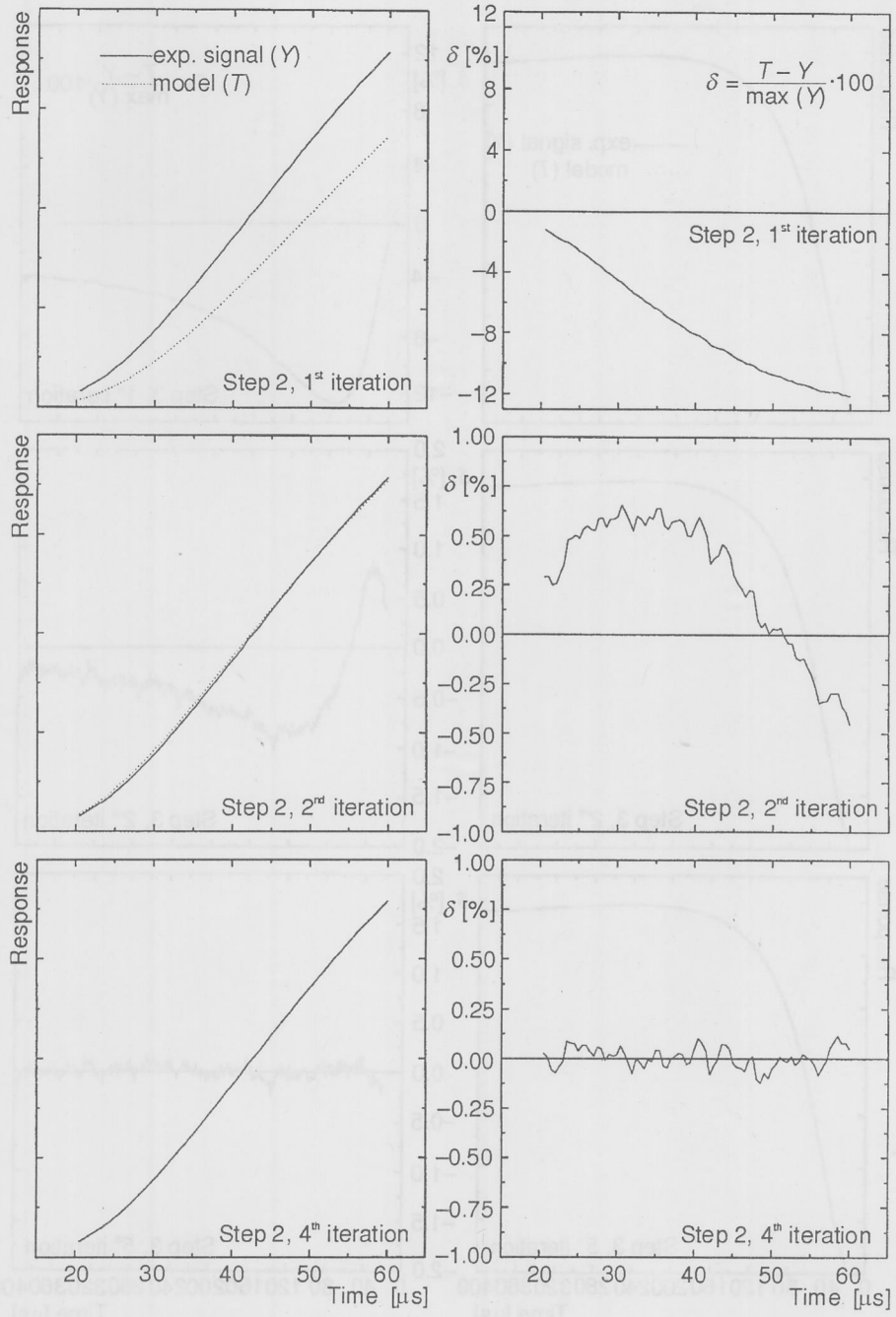

Figure 4. Difference between theoretical and experimental curves in the process of the estimation procedure in step 2

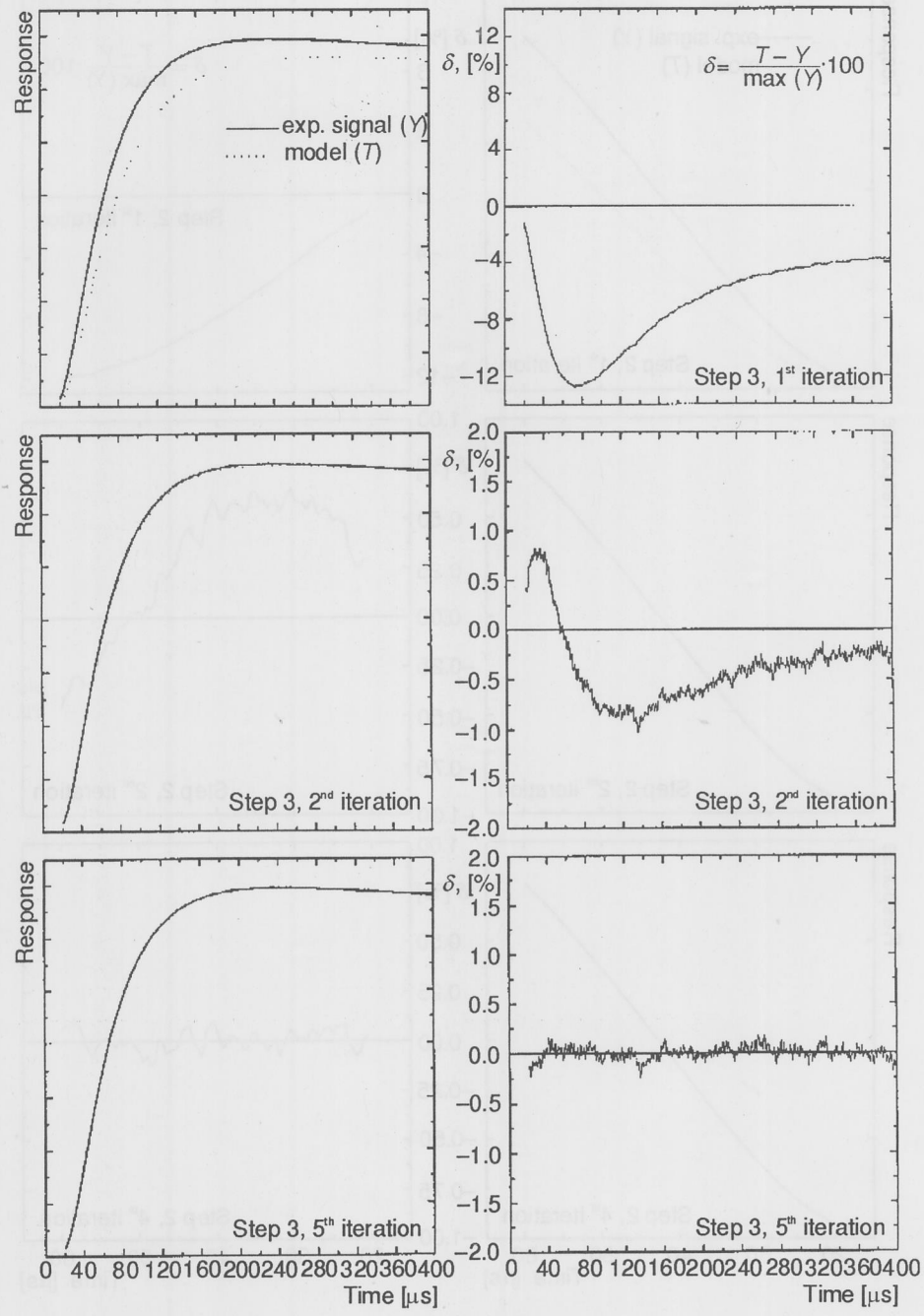

Figure 5. Difference between theoretical and experimental curves in the process of the estimation procedure in step 3

All methods for finite pulse width correction assume that the value of τ_p is already known. In that sense, if τ_p is previously known, step 2 could be omitted in estimation procedure described in this paper. On the other hand, if pulse duration τ_p is non-stable for a given laser one should estimate this parameter. The greatest accuracy of τ_p is then obtainable in step 2, where one uses the initial portion of the signal. In all examined cases uncertainty $\Delta_{\tau p}$ was between 2% and 15%, and due to its small influence on overall accuracy such estimated value τ_p was used as fixed in step 3.

Experimental testing

In order to test the foregoing, Gauss estimation procedure was applied to signals obtained with samples of tungsten and alumina (Al_2O_3). Tungsten has been selected for its relatively high thermal diffusivity and good stability of its thermophysical properties over a wide temperature range, while alumina was a choice for its relatively low thermal diffusivity. Translucence and porosity of alumina could contribute however to the inaccuracy of obtained values. Tungsten sample was NBS SRM-1468 thermal conductivity reference material, and alumina was studied within the NPL organised inter-laboratory comparisons of thermal diffusivity measurement technique [10]. Both samples were 10 mm in diameter. The samples of tungsten and alumina were 3.64 mm and 1 mm thick, respectively.

Temperature responses were analysed using the described inverse technique. In both cases parameters L and R were considered to be invariant. *A priori* value for τ_p was 1 ms. Other *a priori* values were taken from each particular temperature response and the referent signal level. Correction for thermal expansion was not applied to either of materials.

For comparison sake, a direct procedure according to Heckman [11] was also applied. This method, based on the original Parker analysis was chosen because it uses corrections for the finite laser pulse and the heat loss effects, which were both present in the above measurements.

Present results for tungsten are shown in Fig. 6, together with the tungsten thermal diffusivity values calculated from the NBS tungsten thermal conductivity reference data [12] and the literature data for specific heat [13] and density [14], and the CINDAS recommended thermal diffusivity values of tungsten [15].

For Heckman and estimation data reduction procedures, experimental data were fitted with respective polynomials of the 4^{th} degree, and in Fig. 6 data points were taken from these interpolated functions. Deviation of individual values from their corresponding polynomials never exceeded 1.5%. It can be seen that thermal diffusivity values obtained with two data reduction procedures lie within 2.6% limits in the whole measurement range. They are also in good agreement with the NBS calculated values. Results of the direct approach and those determined by estimation procedure lie relatively close to each other.

Present results for alumina are shown in Fig. 7, together with thermal difussivity literature data of Bertier, Chang *et al.*, Plummer *et al.* and Rudkin et al. taken from the TPRL Data Series [16]. For both reduction procedures applied experimental data were fitted with respective polynomials of the 5^{th} degree, and data points in Fig. 7 were taken

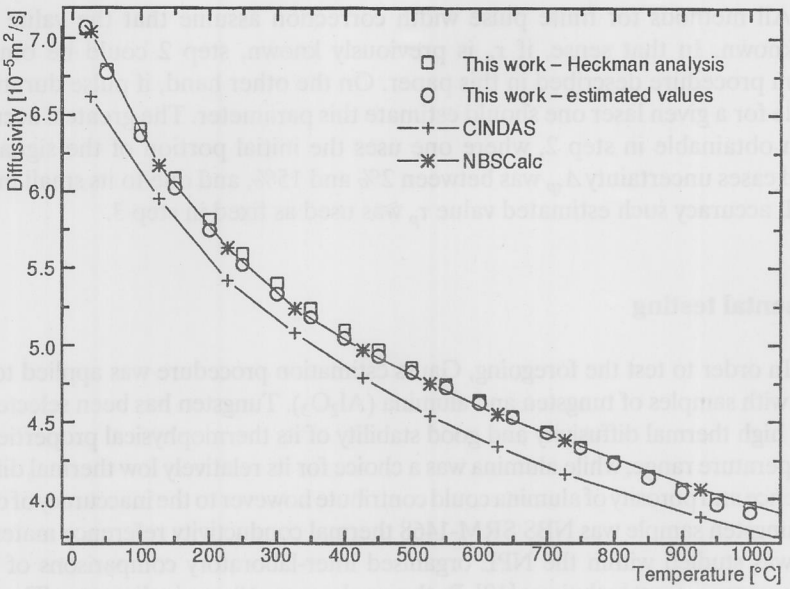

Figure 6. Thermal fusivity of tungsten

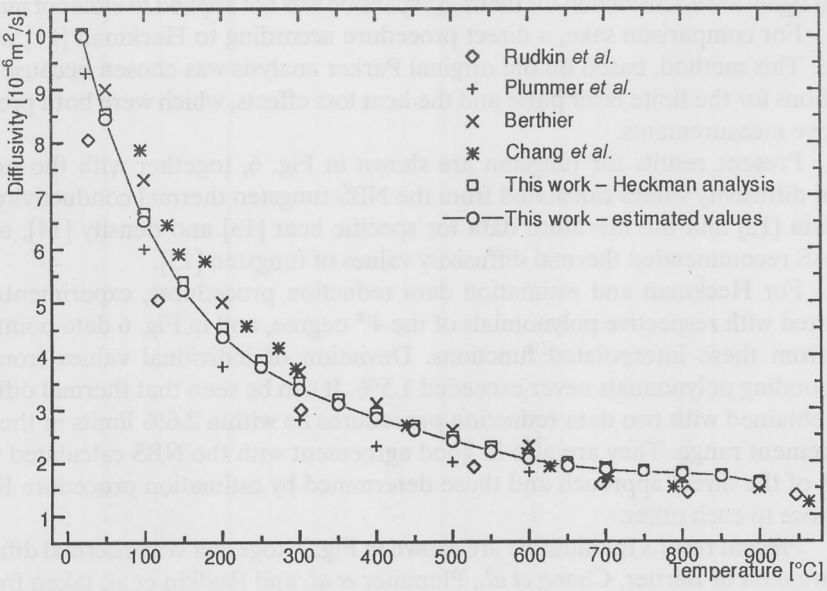

Figure 7. Thermal diffusivity of alumina

from these interpolated functions. Deviation of individual values from their corresponding polynomials never exceeded 1%. Differences between results obtained using different procedures are similar to those obtained for the tungsten sample.

Regarding measuring uncertainty of final results, there are no significant differences between two applied procedures. However, because of large number of applied signal points for the estimation of all parameters, results obtained by Gauss estimation procedure are more reliable, particularly when one uses noisy and/or deformed signals. That explains small differences among thermal diffusivity values obtained in this experimental testing shown on Figs. 6 and 7.

Conclusion

The laser pulse method, the most versatile and the most popular method of measuring thermal diffusivity of solid materials in a wide temperature range has been subject of continuing improvements since its appearance in early sixties. Most of them refer to improvements of the original direct approach. The last decade, however, has evidenced appearance of contributions applying inverse approach to this purpose.

The Gauss estimation procedure used in this work belongs to the latter. It uses the whole transient response for obtaining information about thermal diffusivity and other parameters such as the Biot number and the laser pulse-width. Proposed three-step procedure, a separate estimation of different parameters in different parts of the temperature response, might be a contribution to the reliability and accuracy of thermal diffusivity measurement, particularly with noisy and/or deformed signals.

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Nomenclature

a – thermal diffusivity
$\underline{\mathbf{b}}$ – matrix of parameters in estimation procedure
$\overline{\mathbf{b}}$ – matrix of estimated parameters
b_i – single parameter in estimation procedure
Bi – total Biot number
C_p – specific heat
f – dimensionless laser pulse function

h – radiative heat transfer coefficient
i – index
k – index of iteration
L – sample thickness; axial (as index)
n – total number of signal points
p – number of parameters in estimation procedure
$\mathbf{P}$ – matrix of polynomial values
Q – absorbed laser energy per square meter
R – sample radius; radial (as index)
s – normalized sum of differences between experimental and theoretical curves
$\mathbf{T}$ – matrix of calculated values from the model
T_m – maximum temperature rise
$\mathbf{U}$ – variance-covariance matrix of parameters *a priori*
$\mathbf{W}$ – variance-covariance matrix of measured values
$\mathbf{X}$ – matrix of sensitivity coefficients
X^* – normalized sensitivity coefficient
$\mathbf{Y}$ – matrix of measured values
Z_i – roots of corresponding transcendental equation

Greek symbols

β_n – roots of corresponding transcendental equation
δ – difference between experimental and theoretical curves
Δ – measured uncertainty of estimated parameter
δ_{bi} – increment of b_i
$\bar{\varepsilon}$ – relative difference between experimental and theoretical curves
η – ratio between sums as criterion of reliability of estimated parameters
ϑ – condition for iterative procedure
λ – thermal conductivity
μ – matrix of parameters *a priori*
ρ – density
$\sigma^2_{\bar{b}}$ – standard deviation of estimated parameters
τ – time
τ_p – laser pulse-width
ξ – arbitrarily small number
Ψ – variance matrix of measured values

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