

# A PARAMETER ESTIMATION PROCEDURE IN THERMAL DIFFUSIVITY MEASUREMENTS USING THE LASER FLASH METHOD

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**Abstract** – Paper presents the application of Gauss estimation procedure in measuring thermal diffusivity of single materials using the laser pulse method. Unlike procedures, based on that originally established by Parker et al., where thermal diffusivity is derived using one or more characteristic points of experimental signal, this one belongs to so-called inverse technique, which makes use of complete measured signal. Paper describes a method that achieves a minimum deviation between estimated and experimental curves and gives results with the highest possible accuracy, comprising at the same time uncertainties of known parameters. Also, an influence of new parameter, which represents the onset of temperature response, and its estimation, is studied and performed.

**Keywords:** laser flash method, parameter estimation, thermal diffusivity

## 1 INTRODUCTION

The original laser pulse method of measuring thermal diffusivity proposed by Parker et al. [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 realizing these idealized conditions. Thanks to theoretical works of many researchers, the original concept has been gradually improved to account for real experimental conditions.

In that sense, regarding the homogeneous samples, Cape and Lehman [2], Heckman [3], et Dusza [4] investigated the effects of radiation (heat exchange coefficients different from zero) and finite laser pulse duration. They concluded that these effects have been opposite regarding the half-rise time, which is used for thermal diffusivity estimation, and that an optimal sample thickness should be found. The influence of radiation was studied separately by Cowan [5] and Clark and Taylor [6]. Cowan considered the heat loss using the descending part of experimental signal, while Clark and Taylor proposed an improved correction procedure given previously by Cowan. The finite laser pulse effect was theoretically and experimentally analyzed by Taylor and Clark [7], and Azumi and Takahashi [8] proposed a correction procedure for this effect. Larson and Koyama [9] studied the same phenomenon for thin samples. One important theoretical contribution regarding the laser flash method is given by Watt [10]. He offered several analytical solutions for 1D and 2D heat transfer through the sample, considering at the same time the different effects. Also, he presented some simplified

equations one can use in practice. Laplace, Fourier, and cosine transformations were utilized for data reduction in the flash method by Gembarovič et Taylor [11] and [12], while a logarithmic technique was proposed by Thermitus and Laurent [13]. Beedham and Dalrymple [14] investigated errors originated from the non-uniformity of the laser pulse energy, while McKay and Schriempf [15] gave a correction procedure for the same purpose. The influence of sample transparency in the flash method and its measurement is given by Tischler et al. [16] and Srinivasan et al. [17]. Maglić and Maršičanin [18] and Heckman [19] computed an influence of the characteristic response of intrinsic thermocouple had been used for transient temperature measurements in the laser flash method. The effects of non-linearity of the infrared detector are analyzed by Tang et al. [20]. Data acquisition and data reduction with computers was firstly described by Perović and Maglić [21] and Koski [22].

Two-dimensional version of heat transfer in homogeneous sample was proposed, beside the theoretical analysis from Watt [10], and applied by Donaldson and Taylor [23] and consecutively developed by Chu et al. [24], Amazouz et al. [25], Lachi and Degiovanni [26], Shibata et al. [27], and recently by Sheikh et al. [28]. This version is particularly suitable for thermal diffusivity measurements of thin films and coatings.

The most of publications following the original work were directed toward correcting measured half-rise time or some characteristic points of the transient response. In mathematical sense, these procedures can be named 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.

Accordingly, data reduction procedure implying the analysis of whole temperature response or its part was made by Balageas [29]. He proposed a procedure taking a corresponding part of the signal where existing heat loss could be neglected. Considering the heat losses, however, Degiovanni and Laurent [30] presented a partial time moments technique of order 0 and  $-1$ , where simultaneous identification of characteristic time and heat loss coefficient take place. Pawlowski L. and Fauchais P. [31] applied the least square method for fitting the data of whole response, but thermal diffusivity was still determined using the characteristic points. Gembarovič et al. [32] reported an improvement of technique given by [31] using the least-square method. A contribution of thermal diffusivity estimation using the flash method is given by Raynaud et. al. [33]. They applied the sequential estimation procedure that accounts for whole experimental signal and heat loss effect leading to increased accuracy and reliability of results. At the same time, using the estimation analysis, a design of optimal experiment was suggested.

In this work, the Gauss parameter estimation procedure, being very convenient to use in the laser flash method, is proposed. It enables simultaneous determination of more than one parameter from the same temperature response, such as Biot number, laser pulse-width, or the onset of the temperature response. Estimation possibilities are influenced by the sensitivity coefficient of each single parameter, whose analysis will be given in this paper. Also, the accuracy of results obtained by proposed procedure

comprises the influence of known parameters uncertainties, which was usually neglected in literature.

## 2 GAUSS ESTIMATION PROCEDURE

Generally, all estimation techniques are based on minimizing the difference between the measured and corresponding values obtained by the mathematical model. Among several different approaches, the Gauss parameter estimation procedure, whose detailed description is given by Beck and Arnold [34], is most frequently used. In this paper only relevant information about the procedure will be pointed out.

The Gauss approach 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 must be complied if one wishes reliable results for a given parameter. In that sense, sensitivity coefficients play the most important role, giving information about estimation possibilities of desired parameter. The matrix of sensitivity coefficients are defined by

$$\mathbf{X} \equiv \begin{bmatrix} \partial T_1 / \partial b_1 & \dots & \partial T_1 / \partial b_p \\ \vdots & & \vdots \\ \partial T_n / \partial b_1 & \dots & \partial T_n / \partial b_p \end{bmatrix} \quad (1)$$

where  $T_j$  is a value calculated from the model at the time  $\tau_j$  ( $j=1, \dots, n$ ),  $n$  is the number of measured values,  $b_i$  is the  $i^{\text{th}}$  parameter for estimation ( $i=1, \dots, p$ ), and  $p$  is the number of parameters for estimation. Sensitivity coefficient for parameter  $b$  is presented by the single column of the matrix (1). They are analyzed for each model in particular, and also for different parameter values of the same model.

For qualitative study, one computes usually their reduced form, defined as

$$X_{ji}^* = b_i \frac{\partial T_j}{\partial b_i} \quad (2)$$

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

In theoretical model that correspond to the laser flash method, sensitivity coefficients are very complex functions of both proper and also other parameters. In such case, their values must be numerically calculated using an approximate formula

$$X_{ji}^* = b_i \frac{\partial T_j}{\partial b_i} \approx b_i \frac{T_j(b_1, b_2 \dots b_i + \delta b_i \dots b_p) - T_j(b_1, b_2 \dots b_i - \delta b_i \dots b_p)}{2\Delta b_i} \quad (3)$$

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

The minimization of the difference between theoretical and measured values is performed using the *maximum a posteriori* (MAP) criteria (Beck and Arnold [34]). Linearizing theoretical values  $\mathbf{T}$  using the Taylor series, the Gauss iterative equation obtains the following form:

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

where  $\mathbf{T}$  is the matrix of calculated values from the model  $[n \times 1]$ ,  $\mathbf{Y}$  is the matrix of measured values  $[n \times 1]$ ,  $\mathbf{b}$  is the matrix of parameters for estimation  $[p \times 1]$ ,  $\boldsymbol{\mu}$  is the matrix with *a priori* parametric values  $[p \times 1]$ ,  $\mathbf{W}$  is the variance-covariance matrix of measured values  $[n \times n]$ , and  $\mathbf{U}$  is the variance-covariance matrix of parameters *a priori*  $[p \times p]$ . Diagonal elements of the matrix  $\mathbf{W}$  are the function of variances of each measured value,  $\sigma$ , and variances of known parameter  $m$ ,  $\sigma_m$ , since other elements represent a correlation degree among measured values. If there is no correlation between the measured values, which is the case in this method, the characteristic diagonal element of the variance-covariance matrix  $\mathbf{W}$  is [35]

$$(W)_{jj} = \left[ \sigma_j^2 + \sum_l (\sigma_{m_l} \frac{\partial T_j}{\partial m_l})^2 \right]^{-1} \quad (5)$$

while those extra-diagonal are equal to zero. One can notice that the expression in parenthesis under the summation in (5) represents the reduced sensitivity coefficient of known parameter  $m$ . Calculating these coefficients one can also analyze the influence of known parameter on the model. Through the matrix  $\mathbf{W}$  such influence is directly involved in the estimation process.

The iterative procedure (4) should be ceased when the following condition is satisfied:

$$\frac{b_i^{(k+1)} - b_i^{(k)}}{|b_i^{(k)}| + \xi} < \vartheta \quad (6)$$

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

Standard deviation of parameters estimated by (4) as the criterion of estimation accuracy can be found from the *a posteriori* variance-covariance matrix of the final iteration,

$$\mathbf{S}^{(\text{final})} = \left[ \mathbf{X}^{\text{T}(\text{final})} \mathbf{W} \mathbf{X}^{(\text{final})} + \mathbf{U} \right]^{-1} \quad (7)$$

whose diagonal elements represent the variances of the estimated parameters.

Beside the standard deviation, one could use the normalized sum of relative differences between estimated theoretical and experimental curve as another convenient criterion for the reliability of estimated parameters. Mathematically, this sum can be expressed as:

$$s(T) = \frac{1}{n} \sum_{j=1}^n \bar{\varepsilon}_j^2(T) \quad (8)$$

with

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

Therefore, estimated parameters are more reliable if the sum  $s(T)$  has a smaller value.

### 3 ESTIMATION PROCEDURE APPLIED IN THE LASER FLASH METHOD

#### 3.1 Theoretical model

In the laser flash method the theoretical model is represented by temperature response of the rear sample side. Let the laser pulse with a short duration  $\tau_p$  be absorbed uniformly in a very thin layer of the front sample side. If one measures transient temperature over the all rear sample side, analytical solution of the temperature response is (Watt [10], Yamane et al. [36]):

$$T(\tau) = \frac{8T_m}{a\tau_p} \sum_{n=1}^{+\infty} \frac{\beta_n (\beta_n \cos \beta_n + Bi_L \sin \beta_n)}{\beta_n^2 + Bi_L^2 + 2Bi_L} \sum_{i=1}^{+\infty} \frac{Bi_R J_1(Z_i)}{Z_i (Z_i^2 + Bi_R^2) (Z_i^2 / R^2 + \beta_n^2 / L^2) J_0(Z_i)} \times \\ \times \exp \left[ - \left( \frac{Z_i^2}{R^2} + \frac{\beta_n^2}{L^2} \right) a\tau \right] \left\{ \exp \left[ \left( \frac{Z_i^2}{R^2} + \frac{\beta_n^2}{L^2} \right) a\tau_p \right] - 1 \right\} \quad (9)$$

where  $\tau$  is time ( $\tau > \tau_p$ ),  $Bi_L = h_L L / \lambda$  and  $Bi_R = h_R R / \lambda$  are Biot numbers for two base and one lateral sample sides respectively,  $h_L$  and  $h_R$  are radiative heat transfer coefficients (axial and lateral heat losses),  $L$  is sample thickness,  $R$  is sample radius,  $\lambda$  is thermal conductivity,  $a$  is thermal diffusivity, and  $f(\tau, \tau_p)$  is dimensionless function that describes 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  $Q / (L\rho c)$ , and represents the maximum temperature rise when  $h_L = h_R = 0$ .  $Q$  is absorbed laser energy per square meter,  $c$  is specific heat of sample material, and  $\rho$  is density. Coefficients  $\beta_n$  and  $Z_i$  ( $n, i = 1, 2, 3, \dots$ ) are positive roots of correspondent transcendental equations (Watt [10]).

#### 3.2 Estimation possibilities

Theoretically all constants in (9) could be treated as parameters for estimation. However, by increasing number of parameters for estimation the estimating 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, the laser pulse length  $\tau_p$  could be also known accurately for a given laser type, or determined in advance. However, when the laser pulse length varies from pulse to pulse, this parameter should also be estimated.

In practice, heat losses from base and lateral sample sides are same so  $Bi_L=Bi_R \cdot R/L=Bi$ . It can be shown that sensitivity coefficient of  $Bi$  is greater than sensitivity coefficients of separate Biot numbers, which gives a better possibility for the estimation of  $Bi$ .

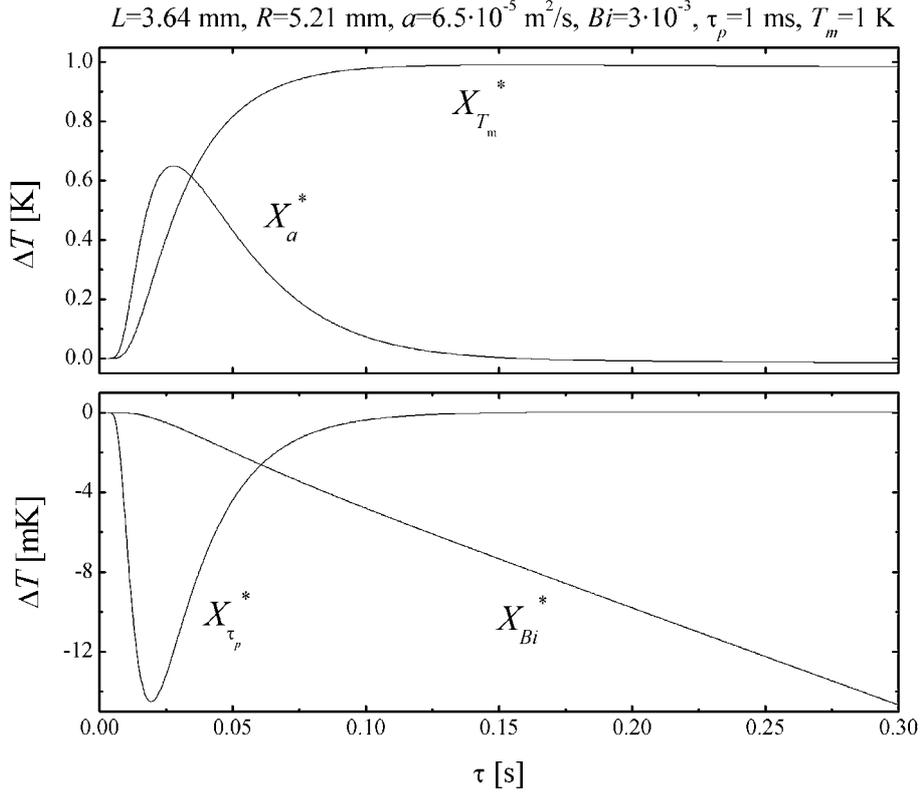


Fig. 1. Normalized sensitivity coefficients of parameters  $a$ ,  $T_m$ ,  $L$ ,  $Bi$ , and  $\tau_p$

Selected values of sensitivity coefficients  $X^*$  of four parameters from (9) are shown in Fig. 1. Sensitivity coefficients were calculated using (3), with  $a=6.5 \cdot 10^{-5} \text{ m}^2/\text{s}$ . This figure 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 complicates simultaneous estimation of these four parameters. However, sensitivity coefficients of all 4 parameters in both examples are linearly independent in a certain time range, especially in the period of temperature rise. This allows their simultaneous estimation despite considerable differences in absolute values, particularly when small uncertainty of parameter  $\tau_p$  and  $Bi$  is not required.

Fig. 1 also shows that sensitivity coefficient of  $T_m$  has the same form and values as the temperature response. This is expected because  $T_m$  affects the model only as a multiple factor.

All these four parameters have influence on temperature response according to (9). There are some parameters, however, which are not visible from (9), but who also affect the experimental signal. Namely, for purpose of estimation procedure, temperature

response must begin from zero-level, for both temperature and also time values. Since experimental signals are measured from certain non-zero level, there is a need for a re-scaling of the signal. Therefore, in practice one has to determine referential signal level,  $T_{ref}$ , in the period prior to the laser discharge (Fig. 2). As far as the high signal-to-noise ratio is concerned, the influence of uncertainty in  $T_{ref}$  on determination of other parameters can be neglected in most cases. In this paper, parameter  $T_{ref}$  was taken as exact.

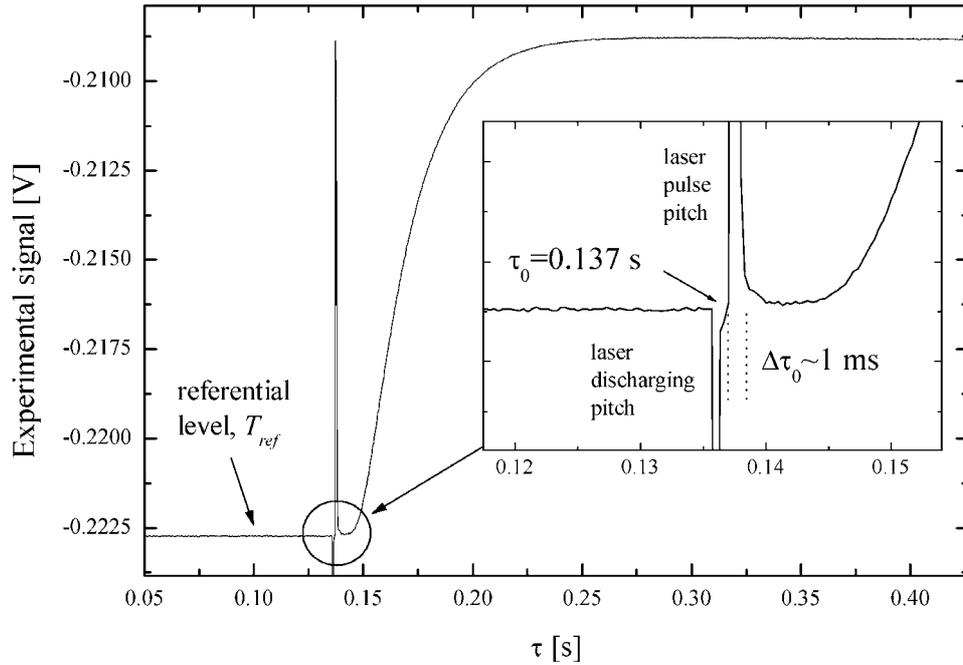


Fig. 2. Experimental signal and the onset pulse time  $\tau_0$  with its incertitude

For the same reason of signal re-scaling, one must also evaluate the onset pulse time,  $\tau_0$ . This evaluation is necessary if the laser discharge is not exactly defined (Fig. 2). In some cases, a small uncertainty in  $\tau_0$  may cause significant uncertainty in thermal diffusivity,  $a$ . This occurs when either sample is thin, or it has high thermal diffusivity. Perturbation of the signal, frequently met in the region of the laser discharge, may also introduce significant uncertainty in determining  $\tau_0$ . Generally, when a ratio of the approximate temperature half-rise time and the uncertainty in time  $\tau_0$  is over 0.1%,  $\tau_0$  should be estimated.

An estimation process for the parameter  $\tau_0$  is somewhat different from the procedure for others. Instead of varying values of the model by varying parametric values, the signal time base is varied in respect to the onset time,  $\tau_0$ . For the same parametric values as in Fig. 1, Fig. 3 shows the sensitivity coefficient of  $\tau_0$  together with the sensitivity coefficient of  $a$ . It is visible that the variation of parameter  $\tau_0$  produces remarkable variation of temperature response. This allows estimation of  $\tau_0$  with a high reliability. Also, one can notice that the sensitivity coefficients of  $\tau_0$  and  $a$  have a similar form, but they are linearly independent in the range of their maximal values (see a dotted line), allowing thus the simultaneous estimation of these two parameters.

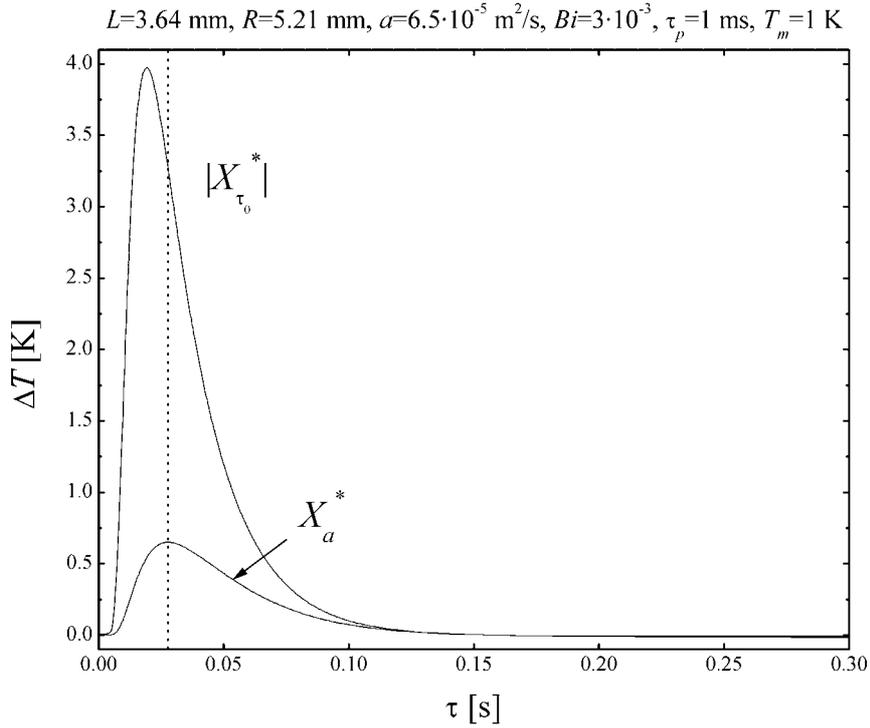


Fig. 3. Normalized sensitivity coefficients of  $\tau_0$  and  $a$

That is not a case, however, for parameters  $\tau_0$  and  $\tau_p$ , whose sensitivity coefficients are virtually linearly dependent (Fig. 1 and Fig. 3). This means that there is no possibility to estimate simultaneously onset pulse time and pulse duration with acceptable accuracy. One of these two parameters, therefore, must be treated as known, during the whole estimation procedure. Depending on particular experimental settings and conditions, sometime one can know more accurately  $\tau_0$  than  $\tau_p$ , and vice versa.

### 3.3 Procedure for determining thermal diffusivity

According to the above analysis, a procedure for determining thermal diffusivity from re-scaled experimental signal is proposed in following three steps (Fig. 4), where the second has two alternatives:

1. The first step involves the estimation of  $Bi$  from the time just before its maximum to a certain value along its descent. This should be performed simultaneously with parameters  $a$ , and  $T_m$  having their *a priori* starting values. Estimation of last two parameters is only temporary, so their estimated values are not final in this step. This “temporary estimation” improves fitting procedure between theoretical and experimental curves. The estimation of  $Bi$  alone would not give a good fit due to its small influence on the temperature response. The uncertainty of estimated parameter  $Bi$  might be relatively high, but its accuracy could be sufficiently good as the new *a priori* value in next steps. In this step parameters  $\tau_0$  and  $\tau_p$  are fixed at their supposed values.
2. Second step refers to estimating whether the onset pulse time,  $\tau_0$ , or the pulse duration,  $\tau_p$ , in the range of the signal rise. This is effected simultaneously with parameters  $a$  and  $T_m$ , whose estimation is also temporary. Parameters  $\tau_p$  in first or  $\tau_0$  in second case and  $Bi$  in both cases are fixed on their supposed, i.e. estimated values.

Like in step 1, the uncertainty of estimated  $\tau_p$  could be relatively high when it has a small influence on the temperature response.

3. Finally, the third step presents the estimation of thermal diffusivity  $\alpha$ , simultaneously with  $T_m$ . Parameters  $Bi$  and  $\tau_0$ , or  $\tau_p$  and are fixed at values determined in the previous steps. Estimation should be performed in the time scale that covers a range from about 10% of the response's maximum and a certain time after the maximum is reached (Fig. 4).

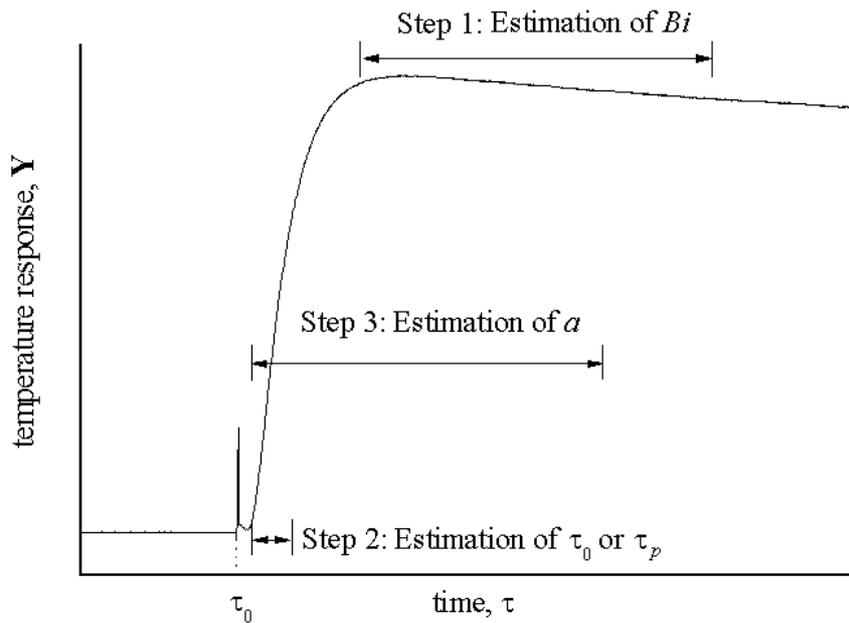


Fig. 4. Three-step parameter estimation procedure applied to temperature response

### 3.4 Uncertainty of estimated thermal diffusivity

As explained above, the proposed parameter estimation procedure intends to obtain the highest accuracy and reliability of thermal diffusivity from both rise and also descending portion of the signal.

The maximal measured uncertainty of some parameter  $\delta$  can be found from the calculated standard deviation  $\sigma$  (7), using a simple approximate relation:  $\delta \approx 3\sigma$ . As the influence of known parameters is involved in the calculated standard deviation through the matrix  $\mathbf{W}$  (5) and (7), the uncertainty of thermal diffusivity  $\delta_\alpha$  already comprises uncertainties of known parameters, such as thickness,  $\delta_L$ , and radius  $\delta_R$ , although the latter is usually neglected due to a very small influence of this parameter on the model.

As another criteria for estimation accuracy, normalized sum  $s(T)$  (8) has a smaller value if one estimates  $\tau_0$ . This can be easily seen on Fig. 5 showing a typical relative difference between experimental and theoretical responses  $\bar{\epsilon}(T)$ . It is visible that additional estimation of the onset time improves agreement between the signal and the model.

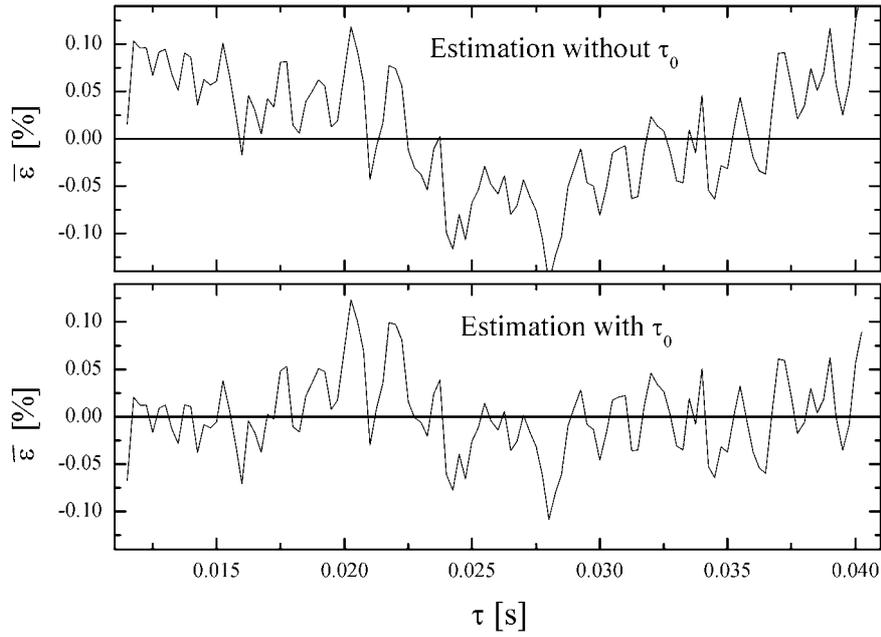


Fig. 5. Difference between the experimental curve and the estimated theoretical model in two cases of estimation procedure

The uncertainty of others parameters can vary from case to case. In some, for example, the uncertainty of parameter  $T_m$  in the last step could be relatively high. However, this doesn't affect the accuracy of  $\alpha$ ; on the contrary, estimation of  $T_m$  always improves the fitting process which leads to less deviation between theoretical and experimental curve. It can be proved by using the value  $T_m$  as a fixed parameter. Then, the value  $s(T)$  is much higher than in the previous case. It might be said, therefore, that in the third step estimation of the parameter  $T_m$  is also a bypassing process.

## 4 RESULTS AND DISCUSSION

In order to test foregoing, the estimation procedure was applied on signals measured on samples of tungsten and alumina ( $\text{Al}_2\text{O}_3$ ). 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 also contribute to the inaccuracy of obtained values. Tungsten sample was NBS SRM-1468 thermal conductivity reference material, and alumina was studied within NPL organized inter-laboratory comparisons of thermal diffusivity measurement techniques (Maglić and Milošević [37]). Both samples had 10 mm in diameter. Thickness of tungsten and alumina samples was 3.64 mm and 1 mm, respectively.

Temperature responses were analyzed using described inverse technique, with and without the estimation of parameter  $\tau_0$ . Parameters  $L$  and  $R$  were considered to be invariant in both cases. *A priori* value for  $\tau_p$  was 1 ms. Other *a priori* values were taken corresponding to each single temperature response and the sample referential signal level. Correction for thermal expansion was not applied for either of materials.

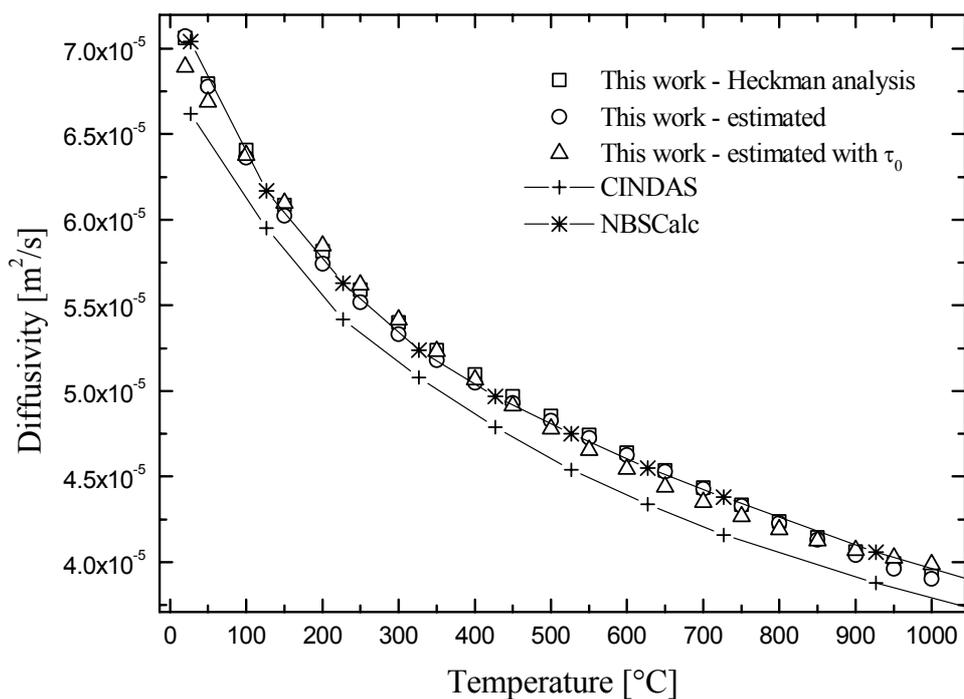


Fig. 6. Thermal diffusivity of tungsten

For comparison sake, the data reduction procedure according to Heckman [3], named a direct procedure, was also applied. This method was chosen because it uses corrections for the finite laser pulse and the heat loss effects, which were both present in the above measurements.

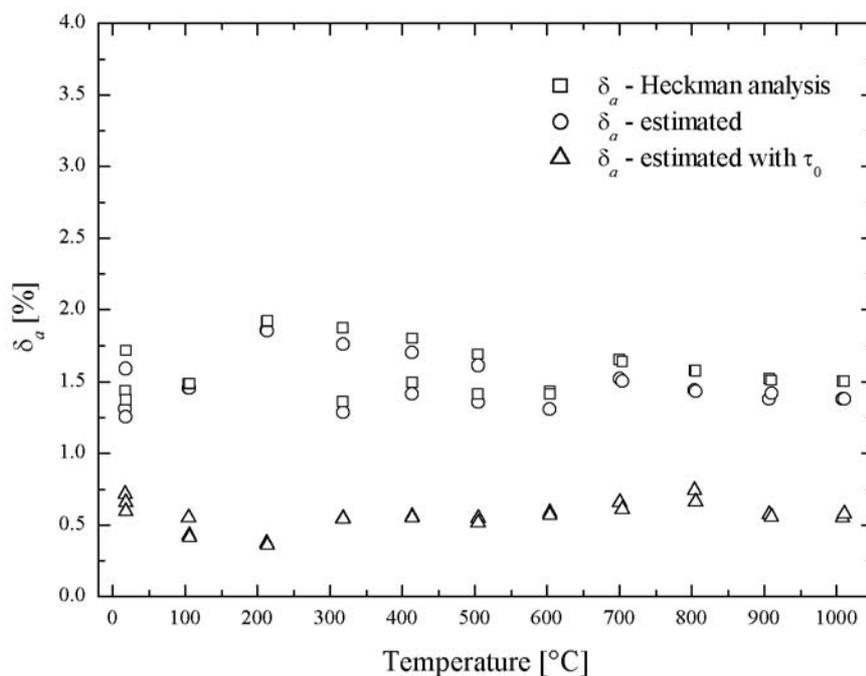


Fig. 7. Maximal uncertainty of estimated thermal diffusivity of tungsten for each measurement

Results for tungsten are shown in Fig. 6, together with thermal diffusivity values calculated from the NBS thermal conductivity reference data (Hust and Giarratano [38]) and the literature data for specific heat and density (Touloukian et al. [39]), and the CINDAS recommended thermal diffusivity values (Touloukian et al. [40]). For each data reduction procedure experimental data were fitted with respective polynomials of the 4<sup>th</sup> degree. Data points in Fig. 6 refer to these interpolated functions. Deviation of individual values from their corresponding polynomials never exceeded 1.5 %.

Thermal diffusivity values obtained with three 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 without parameter  $\tau_0$ , are relatively close. This is expected because both procedures used the same onset time. Thermal diffusivity values estimated with parameter  $\tau_0$  are somewhat different from the other two, due to significant influence of  $\tau_0$  on the temperature response.

On Fig. 7 one can see that the maximal uncertainty of estimated thermal diffusivity,  $\delta_a$ , for each individual measurement and for three types of data reduction procedure never exceeds 2% for this case, and that it falls below 1 % when one applies the estimation with the onset pulse time.

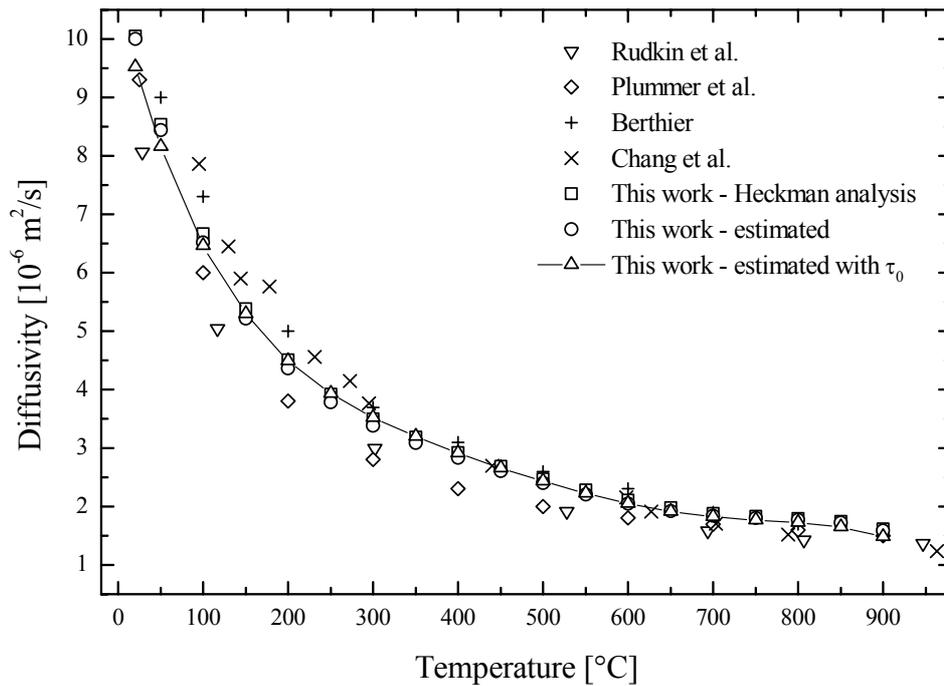


Fig. 8. Thermal diffusivity of alumina

Results for alumina are shown in Fig. 8, together with the data taken from Touloukian et al. [41] (Rudkin et al., Plummer et al., Berthier, and Chang et al.). For each reduction procedure experimental data were fitted with respective polynomials of the 5<sup>th</sup> degree. Data points in Fig. 8 refer to these interpolated functions. Deviation of individual values from their corresponding polynomials never exceeded 1%. Similarly as with tungsten sample, thermal diffusivity values were obtained with three data reduction procedures. Differences between results obtained using different procedures are similar to those in the case of tungsten sample.

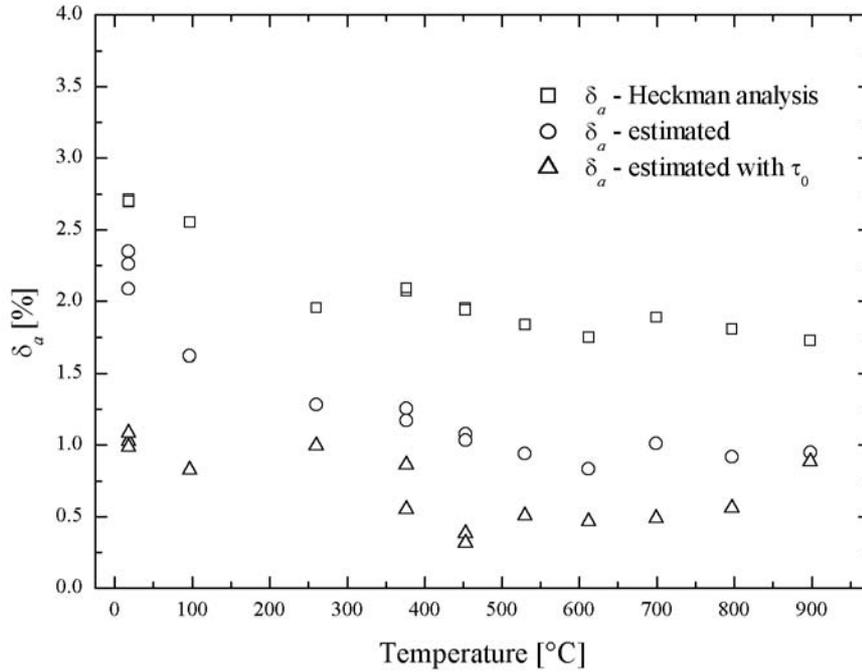


Fig. 9. Maximal uncertainty of estimated thermal diffusivity of alumina for each measurement

The maximal uncertainty of estimated thermal diffusivity of alumina is presented on Fig. 9. It goes between 1.75 and 2.75 % for the Heckman analysis, 1 and 2.5 % for the estimation without  $\tau_0$ , and from below 0.5 up to 1 % for the estimation procedure with the onset pulse time.

Therefore, the above examples show that although direct approach and estimation procedure without parameter  $\tau_0$  give similar results, the latter method is more reliable due to lesser uncertainties. Explanation of such experience could be in a fact that using the whole experimental signal instead of its few characteristic points leads to a reduction of estimation error and augmentation of the overall reliability of results. Furthermore, when one includes the estimation of the onset pulse time, uncertainty of results declines additionally making apparently this estimation technique very reliable.

In comparison to previous techniques published in literature, mathematically it is the most similar to that from Raynaud et al. [33] because the both use the whole experimental signal to make the estimation of thermal diffusivity. However, in difference to method from [33], procedure proposed in this paper accounts both for two additional parameters, laser pulse duration and onset pulse time, and also for uncertainties of known parameters such as sample thickness and diameter.

## 5 CONCLUSION

The Gauss estimation procedure used in this work belongs to inverse techniques for data processing. This procedure takes a benefit of the whole transient response, providing the information about thermal diffusivity and some other parameters such as Biot number, laser pulse duration and particularly the onset pulse time, i.e. the moment when the laser pulse begins. In practice, it could be straightforwardly applied using some common programming tool. Proposed three-step procedure, consisting of estimations of

different parameters in corresponding parts of the temperature response and involving the uncertainties of known parameters, might be a contribution to greater reliability and accuracy of thermal diffusivity measurement of single materials using the laser flash method.

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