International Journal of Thermophysics, Vol. 26, No. 6, November 2005 (© 2005)
DOI: 10.1007/s10765-005-8603-6

Uncertainty of Thermal Diffusivity Measurements by Laser Flash Method[1]

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The laser-pulse method is a well-established nonsteady-state measurement technique for measuring the thermal diffusivity, a, of solid homogeneous isotropic opaque materials. BNM-LNE has developed its own bench based on the principle of this method in which the thermal diffusivity is identified according to the "partial time moments method." Uncertainties of thermal diffusivity by means of this method have been calculated according to the ISO/BIPM "Guide to the Expression of Uncertainty in Measurement." Results are presented for several cases (Armco iron, Pyroceram 9606) in the temperature range from 20 to 800°C. The relative expanded ($k = 2$) uncertainty of the thermal diffusivity determination is estimated to be from ±3 to ±5%, depending on the material and the temperature.

KEY WORDS: Armco iron; laser flash method; Pyroceram; thermal diffusivity; uncertainty.

1. INTRODUCTION

The laser flash method is by far the most frequently used method to measure the thermal diffusivity, a, for high thermal conductivity materials; and in particular, to calculate the thermal conductivity λ from the thermal diffusivity when the density ρ and the specific heat c_p are known. More and more, industrial and research laboratories require an accurate knowledge of thermal properties, especially thermal diffusivity. Their objectives

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[1] Paper presented at the Fifteenth Symposium on Thermophysical Properties, June 22–27, 2003, Boulder, Colorado, U.S.A.

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are to check the adequacy of materials within a specific application, to solve problems of thermal transfer, or to calibrate their own equipment. In this context, BNM-LNE has developed a bench based on the principle of the laser flash method in which the thermal diffusivity is identified according to the "partial time moments method" proposed by Degiovanni [1].

The flash method is an absolute method because only measurements of basic quantities such as time, temperature, length, and electrical quantities are required. So, it can be used for metrological traceability and for the certification of reference materials. BNM-LNE took part with this setup in round robins and in the certification of a ceramic reference material involving many European laboratories [2]. To compare results of measurements performed by the different partners, it was necessary to determine the uncertainty associated with each measurement. Uncertainties of thermal diffusivity by means of this method have been calculated according to the ISO/BIPM "Guide to the Expression of Uncertainty in Measurement" [3], which gives a general method for the evaluation of measurement uncertainties.

This paper deals with the estimation of measurement uncertainties performed by BNM-LNE on their laser flash bench used for thermal diffusivity measurements.

2. PRINCIPLE OF MEASUREMENT

The laser flash method is based upon the measurement of the temperature rise on the back face of a thin disk sample resulting from a short energy pulse on the front surface. The sample (10 mm in diameter and about 1–5 mm thick) is placed in a vacuum furnace and isothermally heated at a uniform temperature (see Fig. 1). Then, a short (450 μs) laser pulse of 1.06 μm wavelength irradiates one side of the sample. The temperature rise on the opposite sample face is measured by an IR detector (HgCdTe or InSb depending on the temperature range). A high-speed recorder collects data representing the temperature rise. The diffusivity is calculated from the shape of the temperature–time curve (thermogram) and the thickness, e, of the sample. The absolute values of the energy absorbed, the temperature rise, and the emissivity of the back face of the sample are not necessary.

The thermal diffusivity is calculated by identification of the experimental thermogram with a theoretical model. BNM-LNE uses an identification method, which takes the heat losses between the sample and its surroundings into account. The identification of the diffusivity is carried out from the two temporal moments of order 0 and -1 from the

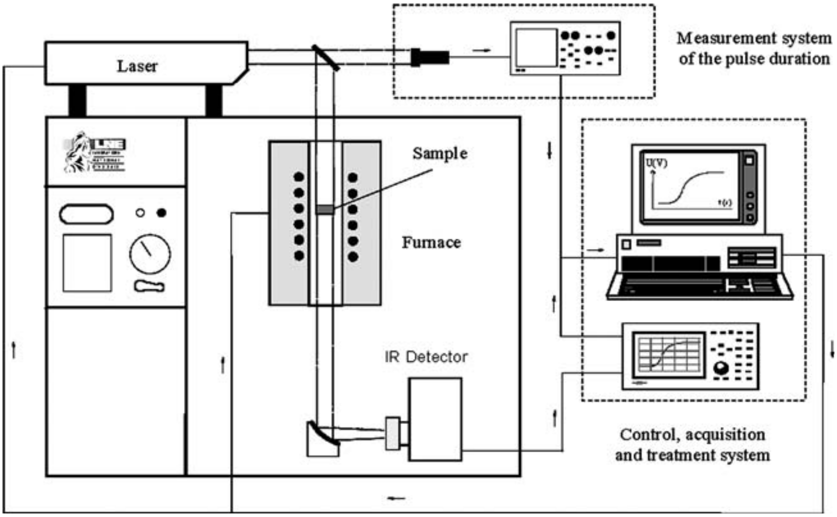


Fig. 1. Schematic diagram of BNM-LNE thermal diffusivity apparatus.

experimental thermogram (m_0 and m_{-1}) and the model (m_0^*, m_{-1}^*). This model is obtained by solving the heat conduction equation in the case of a homogeneous isotropic sample. An identication function F (expressed in m_{-1}) is determined with theoretical thermograms obtained using the model. The thermal diffusivity is then given by the following relationship:

$$a = \frac{e^2 F(m_{-1})}{m_0} \tag{1}$$

3. THEORETICAL MODEL AND IDENTIFICATION METHOD

A solid cylindrical, homogeneous, isotropic and opaque sample is subjected to a heat pulse on its front face at $z = 0$. The heating of the sample involves heat losses on its three faces, characterized by three coefficients of exchange, h_1, h_2, and h_3. The fields of temperature in the sample are described by the following system of equations:

$$\frac{\partial^2 T}{\partial z^2} + \frac{1}{r}\frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial r^2} = \frac{1}{a}\frac{\partial T}{\partial t} \tag{2}$$

Boundary and initial conditions

$$\text{at } z = 0 \qquad \lambda \frac{\partial T}{\partial z} = h_1 (T(r, z, t) - T_0) - Q\,\delta(0) \tag{3}$$

$$\text{at } z = e \qquad -\lambda \frac{\partial T}{\partial z} = h_3(T(r,z,t) - T_0) \tag{4}$$

$$\text{at } r = R \qquad -\lambda \frac{\partial T}{\partial r} = h_2(T(r,z,t) - T_0) \tag{5}$$

$$\text{at } t = 0 \qquad T(r,z,0) = T_0 \tag{6}$$

The assumptions used to establish the analytical model are as follows:

- The model is linear (thermophysical properties are considered independent of the temperature),
- The thermal losses between the sample and its surrounding are characterized by a uniform, and constant in time, heat-exchange coefficient,
- The laser pulse is considered as a Dirac pulse.

The system of equations is expressed in classical dimensionless parameters (see Nomenclature) and solved [4] in order to obtain the dimensionless temperature $\theta^*(r^*, l, t^*) = f(t^*, H, r^*, R^*)$ at a point on the rear face $z = e$ or $z^* = 1$).

Theoretical and experimental thermograms normalized by their maxima are written as follows:

$$f^*(t^*) = \frac{\theta^*(t^*)}{\theta^*_{\max}} \quad \text{and} \quad f(t) = \frac{T(t) - T_0}{T_{\max} - T_0} \tag{7}$$

where $\theta^*_{\max}$ is the maximum of the dimensionless temperature and $T_{\max}$ is the maximum experimental temperature reached during the test. In the Degiovanni method [1], the thermal diffusivity is determined using the partial temporal moments which are expressed as

$$\begin{cases} m_0^* = \int_{t^*_{0.1}}^{t^*_{0.8}} f^*(t^*)\,dt^* & \text{and} \quad m^*_{-1} = \int_{t^*_{0.1}}^{t^*_{0.8}} \frac{f^*(t^*)}{t^*}\,dt^* \\ m_0 = \int_{t_{0.1}}^{t_{0.8}} f(t)\,dt & \text{and} \quad m_{-1} = \int_{t_{0.1}}^{t_{0.8}} \frac{f(t)}{t}\,dt \end{cases} \tag{8}$$

The limits of integration are equal to the times $t_{0.1}$ and $t_{0.8}$, corresponding to the times taken by the back face of the sample to reach 10 and 80% of its maximum temperature. The links between the theoretical and experimental moments are expressed as

$$m_0 = m_0^* e^2/a \quad \text{and} \quad m_{-1} = m^*_{-1} \tag{9}$$

A relation between the theoretical moments is determined from couples of values (m_0^*, m_{-1}^*) obtained with the expressions (Eq. (8)) from thermograms calculated for various values of H by using the ideal model,

$$m_0^* = F(m_{-1}^*) \tag{10}$$

Equation (1) is then obtained by combining Eqs. (9) and (10). The relation F, called the identification function, is determined by a polynomial fit of the calculated couples of values (m_0^*, m_{-1}^*). The coefficients b_i depend on the geometry of the sample.

$$F(m_{-1}^*) = \sum_{i=0}^{n} b_i (m_{-1}^*)^i \tag{11}$$

4. IDENTIFICATION OF UNCERTAINTY SOURCES

Uncertainty evaluation requires a careful analysis of the measurement process. The use of the Ishikawa method allows identification of all possible sources of uncertainty. Sources of uncertainty can be mainly distributed into five major categories, which are *measurement means, method, materials, medium* (surroundings), and *manpower* (user). Within each category, uncertainty sources are identified and discussed in the following subsections.

4.1. Measurement Means

The term "*measurement means*" gathers the devices used for the measurement of the physical quantities involved in the determination of thermal diffusivity (thickness and temperature of the sample, voltage delivered by the IR detector, timebase). Uncertainties in the thickness, temperature, time, and voltage measurements result mainly from the combination of uncertainties due to the noise of measurement, the resolution, and calibration of the equipment. Another source of uncertainty arises during the experiment from the possible drift of the baseline of the signal delivered by the IR detector, due to the electromagnetic disturbances induced by the laser pulse.

4.2. Method

Uncertainties related to the measurement method are mainly due to the identification process of thermal diffusivity and to the differences between the experimental conditions and the assumptions upon which the model was based. Although the method presented previously aims at correcting their effects, uncertainty factors come from badly controlled boundary conditions: finite pulse-time, nonuniformity of the laser, thermal losses, and nonlinearity. Uncertainty due to the pulse duration depends on the thermal properties, the thickness of the sample, and on the pulse characteristics (duration, energy and temporal shape). When the duration of the pulse is not negligible in comparison with the rise time, the origin of time is shifted by a value T_g, corresponding to the chronological centroid of the laser pulse [5, 6].

The uncertainty due to the nonuniformity of the laser depends on the energy spatial distribution, on the radius/thickness ratio, and on the measurement method of the back-face temperature rise. This uncertainty becomes negligible when the average temperature of all the back face is measured [7] with an IR detector. Uncertainties on the nonlinearity effects are due to the variation of the thermophysical properties of the sample with the temperature during the test, although they are assumed to be constant in the model. These effects are neglected by limiting the energy pulse in order to minimize the temperature increase of the rear face to 5 K maximum.

The uncertainty on the assumptions associated with the theoretical model used for the determination of the identification function has been estimated from simulations (different boundary conditions have been introduced in the model describing the heat transfer in the sample). In addition to the imperfect agreement between the experimental conditions and the boundary conditions of the model, uncertainties on the determination method depend also on the identification method (calculation of the experimental moments by numerical integration, determination of the identification function F). Another uncertainty term concerns the assumption of the linearity of the voltage delivered by the IR detector with the temperature although the thermal radiation from a grey body (a body with a spectral emissivity independent of wavelength) is proportional to the fourth power of its absolute temperature. This uncertainty component is calculated considering a maximum temperature rise of the rear face of 5 K.

4.3. Materials

The sources of uncertainty concerning the sample are its geometrical quality (flatness and parallelism of the faces) and its chemical, optical, and thermophysical properties (isotropy, homogeneity, opacity…).

The thickness of the sample at a temperature T is calculated from the thickness measured at room temperature corrected by a term taking the expansion of the sample between these two temperatures into account. The uncertainty of the thermal expansion gives an additional uncertainty component for the thickness for a temperature higher than room temperature.

4.4. Medium (Surroundings)

The uncertainty factors related to the *surroundings* correspond to the experimental conditions, especially induced by the furnace temperature (its stability, its homogeneity), the nature of the atmosphere (vacuum, inert gas...), and all other parameters conditioning the heat losses (contacts between the sample and its surrounding, for example). Thermophysical properties of the matter as well as the heat transfer coefficients depend on temperature. The variations of the thermophysical properties, often weak, can become significant, when measuring the thermal diffusivity of metals at low temperatures (below 100 K). In this case, the thermal diffusivity varies strongly with the temperature (up to 10% per °C). We also note the case for iron for which the thermal diffusivity varies strongly with temperature near the Curie point ($\approx 770°\mathrm{C}$). The determination of thermal diffusivity is then extremely sensitive to an error in the temperature measurement.

4.5. Manpower (User)

The uncertainty sources due to the people depend on the expertise of the operator who performs the thermal diffusivity measurement. They concern mainly the selection of limits U_0 and $U_{\max}$ used to normalize the experimental curve, the correction of the thermogram baseline drift, and the measurement of the sample thickness.

5. UNCERTAINTY BUDGET

With the uncertainty sources identified, an analytical expression for the uncertainty of thermal diffusivity measurement is established following the ISO guide [3]. The uncertainty on the thermal diffusivity determination results from the combination of uncertainties on calculation of the moments m_0 and m_{-1}, on the thickness determination, on the determination of the identification function F, and on the uncertainty on the test temperature T. Assuming first that the temporal moments are correlated together and are independent of the thickness e and of the identification function coefficients b_i, themselves correlated together and independent of the thickness e, the variance of a calculated from Eq. (1) is expressed as follows:

$$u^2(a) = c_e^2 u^2(e) + c_{m_{-1}}^2 u^2(m_{-1}) + c_{m_0}^2 u^2(m_0) \\ +2c_{m_0} c_{m_{-1}} u(m_0, m_{-1}) + c_F^2 u^2(F) + u_T^2(a) \tag{12}$$

with

$$\text{with} \quad \begin{cases} c_e = \dfrac{2e\sum_{i=0}^{n} b_i m_{-1}^{i}}{m_0} & c_F = \dfrac{e^2}{m_0} \\ c_{m_0} = \dfrac{-e^2\sum_{i=0}^{n} b_i m_{-1}^{i}}{m_0} & c_{m_{-1}} = \dfrac{e^2\sum_{i=0}^{n} i b_i m_{-1}^{i-1}}{m_0} \end{cases}$$

5.1. Variances and Covariances of the Temporal Moments

The temporal moments m_0 and m_{-1} are calculated from the experimental thermogram $U(t)$ according to the sequence below:

- Determination of the minimum U_0 and maximum $U_{\max}$ of the thermogram
- Normalization of the experimental thermogram by its maximum
- Determination of the limits of integration $t_{0.1}$ and $t_{0.8}$
- Calculation of the experimental moments from the following general formula:

$$m_k = \int_{t_{0.1}}^{t_{0.8}} f(t) t^k dt \tag{13}$$

The function $f(t)$, which corresponds to the normalized thermogram, is written as

$$f(t) = \frac{U(t) - U_0}{U_{\max} - U_0} \tag{14}$$

The uncertainty on $f(t)$ results from the combination of the following uncertainties:

- Uncertainty on the measurement of the voltage $U(t)$
- Uncertainty on determinations of the baseline U_0 and the maximum voltage $U_{\max}$
- Uncertainty due to the assumption of linearity of the tension $U(t)$ delivered by the IR detector with the temperature T of the back face of the sample.

By assuming that the uncertainty due to the assumption of linearity is independent of the other uncertainty factors affecting the calculation of the normalized thermogram, the variance of $f(t)$ can be presented in the following form:

$$\begin{aligned} u^2(f(t)) = {} & c^2_{U(t)}u^2(U(t)) + c^2_{U_0}u^2(U_0) + c^2_{U_{\max}}u^2(U_{\max}) + u^2_{\text{lin}}(f(t)) \\ & +2c_{U(t)}c_{U_0}u(U(t), U_0) + 2c_{U(t)}c_{U_{\max}}u(U(t), U_{\max}) \\ & +2c_{U_{\max}}c_{U_0}u(U_{\max}, U_0) + u^2_{\text{lin}} \end{aligned} \tag{15}$$

with
$$\begin{cases} c_{U(t)} = \dfrac{1}{U_{\max} - U_0} = \dfrac{1}{\Delta U_{\max}} \qquad c_{U_{\max}} = \dfrac{-(U(t) - U_0)}{(U_{\max} - U_0)^2} = \dfrac{-f(t)}{\Delta U_{\max}} \\ c_{U_0} = \dfrac{[(U(t) - U_0) - (U_{\max} - U_0)]}{(U_{\max} - U_0)^2} = \dfrac{f(t) - 1}{\Delta U_{\max}} \end{cases}$$

The experimental temporal moments are estimated by numerical integration of Eq. (13) as follows:

$$m_k = \frac{\Delta t}{2}\left[f(t_{0.1})t^k_{0.1} + f(t_{0.8})t^k_{0.8} + 2\sum_{i=1}^{n-1} f(t_i)t^k_i \right] \tag{16}$$

The uncertainty on the calculation of the moments results from the following uncertainties:

- Uncertainty on the measurement of $f(t_i)$ and the determination of $f(t_{0.1})$ and $f(t_{0.8})$,
- Uncertainty on the time step Δt and the determination of the times t_i, $t_{0.1}$ and $t_{0.8}$,
- Uncertainty due to the numerical integration method (trapezoidal method).

The variance of the experimental temporal moments m_k is calculated using the following formula:

$$\begin{aligned} u^2(m_k) = {} & c^2_{\Delta t}u^2(\Delta t) + \sum_{i=1}^{n-1}(c^2_{t_i}u^2(t_i)) + c^2_{t_{0.1}}u^2(t_{0.1}) + c^2_{f(t_{0.1})}u^2(f(t_{0.1})) \\ & + \sum_{i=1}^{n-1}(c^2_{f(t_i)}u^2(f(t_i))) + c^2_{t_{0.8}}u^2(t_{0.8}) + c^2_{f(t_{0.8})}u^2(f(t_{0.8})) \\ & + u^2_{int}(m_k) + 2\sum_{p=1}^{r-1}\sum_{q=p+1}^{r} \frac{\partial m_k}{\partial x_p}\frac{\partial m_k}{\partial x_q}u(x_p, x_q) \end{aligned} \tag{17}$$

$$\text{with}\quad\begin{cases} c_{\Delta t}=\frac{1}{2}\left[f(t_{0.1})t_{0.1}^{k}+f(t_{0.8})t_{0.8}^{k}+2\sum\limits_{i=1}^{n-1}f(t_i)t_i^{k}\right] & \\ c_{t_i}=\Delta t\left[f(t_i)kt_i^{k-1}\right] \quad c_{f(t_i)}=\Delta t t_i^{k} & c_{f(t_{0.8})}=\frac{\Delta t}{2}t_{0.8}^{k} \\ c_{t_{0.1}}=\frac{\Delta t}{2}\left[f(t_{0.1})kt_{0.1}^{k-1}\right] \quad c_{t_{0.8}}=\frac{\Delta t}{2}\left[f(t_{0.8})kt_{0.8}^{k-1}\right] & c_{f(t_{0.1})}=\frac{\Delta t}{2}t_{0.1}^{k} \end{cases}$$

The last two terms of Eq. (17) correspond, respectively, to the variance due to the integration method and to the covariance terms.

5.2. Variance on the Thickness e_0

The thickness e_0 of the sample at room temperature T_r is measured using a calibrated micrometer. The thickness e of the sample at the test temperature T is equal to the thickness e_0 measured at room temperature T_r corrected with a term Δ_e taking the expansion of the sample between these two temperatures into account.

$$e=e_0+\Delta_e \tag{18}$$

The uncertainty in the thickness e results from the combination of the uncertainty of the measurement thickness e_0 and the uncertainty in the correction Δ_e.

$$u^2(e)=u^2(e_0)+u^2(\Delta_e)+2u(e_0,\Delta_e) \tag{19}$$

The uncertainty on the thickness e_0 results from the combination of the uncertainties due to the repetability of measurements $u_R(e_0)$, the calibration $u_c(e_0)$, and the resolution $u_r(e_0)$ of the micrometer. These three components being considered as independent, the variance $u^2(e_0)$ is written as

$$u^2(e_0)=u_c^2(e_0)+u_r^2(e_0)+u_R^2(e_0) \tag{20}$$

The correction of the thickness Δ_e represents the thickness variation of the sample between room temperature T_a and the test temperature T.

$$\Delta_e=\alpha_l e_0\,(T-T_a) \tag{21}$$

The uncertainty on Δ_e results from the combination of the following uncertainties:

- Uncertainty on the determination of the average coefficient of linear expansion

- Uncertainties on room temperature T_a and test temperature T measurements
- Uncertainty on the measurement of the sample thickness at room temperature

With these four uncertainty factors being considered as independent, the variance $u^2(\Delta_e)$ is written as

$$u^2(\Delta_e) = (e_0\,(T-T_a))^2\,u^2(\alpha_l) + (\alpha_l\,(T-T_a))^2\,u^2(e_0) + (\alpha_l e_0)^2\,(u^2(T)+u^2(T_a)) \tag{22}$$

The correction Δ_e and the thickness e_0 are correlated by the means of uncertainties of calibration and repetability, uncertainties due to the resolution being assumed independent. Finally, the covariance $u(e_0,\Delta_e)$ is written as

$$u(e_0,\Delta_e) = (\alpha_l\,(T-T_a))^2\,(u_R^2(e_0)+u_c^2(e_0)) \tag{23}$$

5.3. Variance of the Identification Function *F*

The variance of the identification function F results from the variance $u^2_{\mathrm{mod}}(F)$ due to the determination of the function F and the variance $u^2_{\mathrm{hyp}}(F)$ due to the use of this function under experimental conditions different from the assumptions for which it was determined. With these two terms being independent, the variance of the function F can be expressed as

$$u^2(F) = u^2_{\mathrm{mod}}(F) + u^2_{\mathrm{hyp}}(F) \tag{24}$$

The variance $u^2_{\mathrm{mod}}(F)$, calculated from data resulting from the fit of the function F, is a combination of the following three terms:

- Sum of the variances of the coefficients b_i
- Sum of covariances between coefficients b_i and b_j
- Residual variance SE^2 of the regression due to the error of the model

$$u^2_{\mathrm{mod}}(F(m^*_{-1})) = \sum_{j=0}^{n}\left(\frac{\partial F}{\partial b_i}\right)^2 u^2(b_i) + 2\sum_{i=1}^{n-1}\sum_{j=i+1}^{n}\frac{\partial F}{\partial b_i}\frac{\partial F}{\partial b_j}u(b_i,b_j) + \mathrm{SE}^2 \tag{25}$$

The variance $u^2(F)$, due to the use of the function F under experimental conditions different from the assumptions with which it was determined, is estimated from simulations according to the assumptions described in Section 3. It results from the combination of the variance $u^2(t_p)$ due to the variation of the laser pulse duration, the variance $u^2(H)$ due to the nonhomogeneity of the Biot numbers H, and the variance $u^2(\phi)$ due to the variation of the laser beam diameter. With these three components being independent, the variance $u^2_{\text{hyp}}(F)$ is written as

$$u^2_{\text{hyp}}(F) = u^2(t_p) + u^2(\phi) + u^2(H) \tag{26}$$

5.4. Variance of the Sample Temperature T

The sample temperature T is measured by a thermocouple fixed on the sample holder and located near the sample. The uncertainty of T results from the uncertainty due to the resolution and calibration of the temperature measuring chain, and the uncertainty due to the stability and the homogeneity of the furnace temperature. With these four uncertainty factors being independent, the variance $u^2(T)$ is thus written as

$$u^2(T) = u_r^2(T) + u_s^2(T) + u_h^2(T) + u_c^2(T) \tag{27}$$

Finally, the thermal diffusivity varies with the temperature T according to a general law $a = G(T)$. The variance on the thermal diffusivity due to the variance on the temperature T can be presented in the following form:

$$u_T^2(a) = \left(\frac{\partial G(T)}{\partial T}\right)^2 u^2(T) \tag{28}$$

The analytical expression of the uncertainty of thermal diffusivity being established, each of its components has been calculated. The uncertainty budget for the thermal diffusivity measurement has been evaluated for measurements carried out at various temperatures on Pyroceram 9606 and on Armco iron [8]. Table I gives a summary of the results of this analysis in the case of Armco iron at 20°C. Expanded uncertainties ($k = 2$) are presented in Tables II and III.

The thermal diffusivity values and the associated uncertainties given in Table II were used in an European study concerning the certification of Pyroceram 9606 as a reference material [2]. The mean values obtained by the six partners (NPL, ARCS, BNM-LNE, KE, INSA, Netszch) involved in this study are presented in Fig. 2. The deviation of all mean values determined by each laboratory from the certified value is less than ±3.7%.

Table I. Uncertainty Budget of Thermal Diffusivity Determination (Armco Iron)

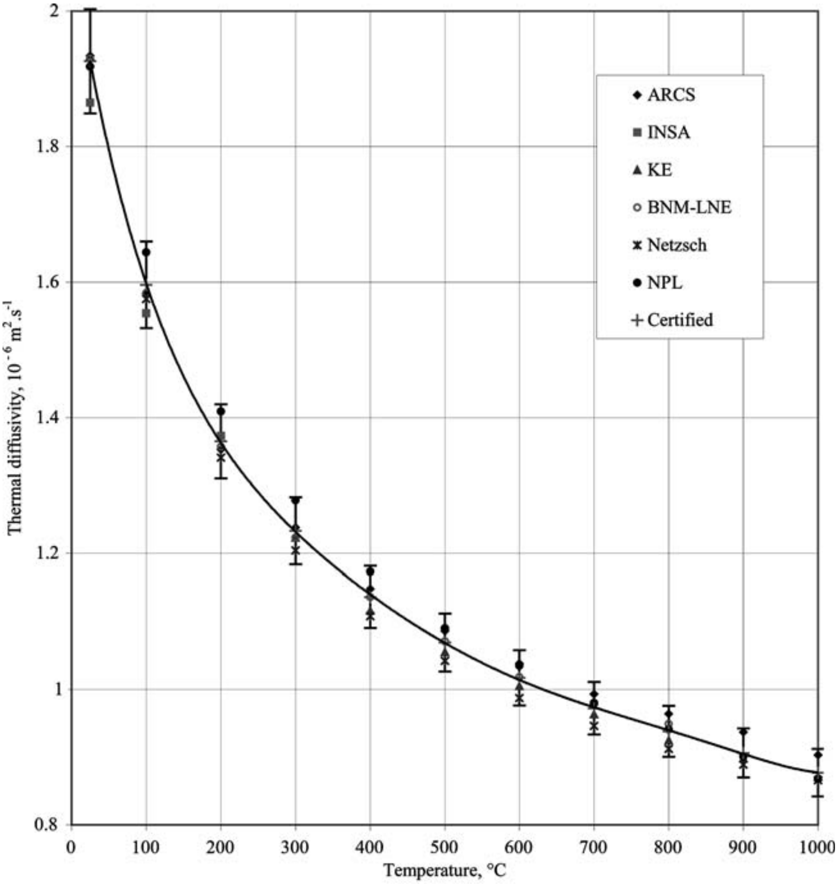
Quantity X_i	Estimate x_i	Variance or covariance $u^2(x_i)$ or $u(x_i, x_j)$	Sensitivity coefficient $\partial a/\partial X_i$	Relative weight (%)
T(°C)	20.0	1.95×10^{-1}	3.92×10^{-8}	0.2
e (mm)	3.006×10^{-3}	2.63×10^{-12}	1.34×10^{-2}	0.3
m_{-1}	0.535	6.62×10^{-6}	7.97×10^{-5}	29.1
m_0 (s)	0.0364	2.18×10^{-8}	5.52×10^{-4}	4.6
$u(m_0, m_{-1})$	–	3.28×10^{-7}	–	20.0
F	0.081	1.08×10^{-6}	2.48×10^{-4}	45.8
		Uncertainty		
a ($m^2 \cdot s^{-1}$)	Variance	Standard ($m^2 \cdot s^{-1}$)	Expanded ($m^2 \cdot s^{-1}$)	Expanded (%)
2.012×10^{-5}	1.44×10^{-13}	3.80×10^{-7}	7.60×10^{-7}	3.78

Table II. Measurement Uncertainties of Thermal Diffusivity of Pyroceram 9606

Temperature (°C)	Thermal Diffusivity (10^{-6} $m^2 \cdot s^{-1}$)	Expanded uncertainty ($k = 2$) (10^{-6} $m^2 \cdot s^{-1}$)	Expanded uncertainty ($k = 2$) (%)
20	1.93	0.095	4.9
200	1.36	0.050	3.7
400	1.14	0.042	3.7
600	1.02	0.043	4.2
800	0.95	0.047	4.7

Table III. Measurement Uncertainties of Thermal Diffusivity of Armco Iron

Temperature (°C)	Thermal Diffusivity (10^{-6} $m^2 \cdot s^{-1}$)	Expanded uncertainty ($k = 2$) (10^{-6} $m^2 \cdot s^{-1}$)	Expanded uncertainty ($k = 2$) (%)
20	20.12	0.76	3.8
200	14.50	0.44	3.0
400	10.08	0.30	3.0
600	6.56	0.21	3.2
800	4.46	0.15	3.4



ARCS	Austrian Research Center of Seibersdorf (Seibersdorf - Austria)
INSA	Institut National des Sciences Appliquées (Villeurbanne - France)
KE	Forschungsinstitut für Kerntechnik und Energiewandlung (Stuttgart - Germany)
BNM-LNE	Bureau National de Métrologie – Laboratoire National d'Essais (Paris - France)
Netzsch	Netzch Geratebau GmbH (Selb/Bayern - Germany)
NPL	National Physical Laboratory (Teddington - England)

Fig. 2. Certified thermal diffusivity of Pyroceram 9606 and mean values of all partners [2] involved in European round-robin study.

The expanded uncertainties of the certified values are estimated to be better than ±5.3%. Pyroceram 9606 will be soon available by IRMM (Institute for Reference Materials and Measurements), European body in charge of the spreading of CRMs (Certified Reference Materials).

6. CONCLUSIONS

The evaluation of the uncertainties associated with the thermal diffusivity measurement performed with the BNM-LNE laser flash bench is presented in this paper, and the influence parameters are identified and quantified. The analytic expression of measurement uncertainty was obtained by establishing the assessment of uncertainties for the several components. The uncertainty was calculated in the range [20–800°C] for two materials (Armco iron and Pyroceram 9606).

The relative expanded uncertainty ($k = 2$) of the thermal diffusivity determination is estimated to be from ±3 to ±5% depending on the material and the temperature. The repeatability of five successive measurements lies between 0.2 and 1%. It was shown that the uncertainty components having the most weight are those related to the evaluation of temporal moments and to the determination of the identification function. Their variances represent about 90% of the total variance on thermal diffusivity measurements.

A round robin and a certification study show that the uncertainty measurement evaluated by BNM-LNE is in agreement with those calculated by the other European metrological laboratories.

NOMENCLATURE

Notation

a	thermal diffusivity, $m^2 \cdot s^{-1}$
c_p	specific heat, $J{\cdot}kg^{-1}K^{-1}$
e	thickness of the sample, m
F	identification function
H	Biot number ($= he/\lambda$)
m_0	experimental temporal moment of order 0
m_{-1}	experimental temporal moments of order -1
m_0^*	theoretical temporal moment of order 0
m_{-1}^*	theoretical temporal moments of order -1
Q	heat pulse surface density, $J \cdot m^{-2}$
r	abscissa of the measurement point, m

r^*	dimensionless abscissa $(=r/e)$
R	radius of the sample, m
R^*	dimensionless radius $(=R/e)$
t	physical time, s
t^*	dimensionless time $(=at/e^2)$
T	temperature, K
T_0	reference temperature, K
U	voltage delivered by IR detectors, V
z	depth of the measurement point, m
z^*	dimensionless depth $(=z/e)$

Greek symbols

α_l	thermal expansion coefficient, K^{-1}
θ^*	dimensionless temperature
$(=\rho c_p e(T-T_0)/Q)$	
λ	thermal conductivity, $\mathrm{W \cdot m^{-1} \cdot K^{-1}}$
ρ	density, $\mathrm{kg \cdot m^{-3}}$

Superscript

*	refers to dimensionless quantities

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