

**UNCERTAINTY OF THERMAL DIFFUSIVITY MEASUREMENTS BY LASER
FLASH METHOD¹**

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

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ABSTRACT

The laser-pulse method is a well-established non steady state measurement technique for measuring 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" [1].

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 some cases (Armco Iron, Pyroceram 9606) on the temperature range from 20 °C 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 : thermal diffusivity, uncertainty, flash method, Armco Iron, Pyroceram

1. INTRODUCTION

The flash method is by far the most frequently method used to measure thermal diffusivity a for good thermal conductive materials, particularly 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 accurate knowledge of thermal properties, especially thermal diffusivity. Their objectives are to check the adequacy of materials with 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 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.

Present 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 flash method is based upon the measurement of the temperature rise on the back face of a thin disk sample caused by a short energy pulse on the front surface. The sample (10 mm in diameter and about 1 to 5 mm thick) is placed in a vacuum furnace and isothermally heated at a uniform temperature (see figure 1). Then, a short (450 μ s) laser pulse of 1.06 μ m 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 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 surrounding into account. The identification of the diffusivity is carried out from the two temporal moments of order 0 and -1 from the 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 identification 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:

$$a = \frac{e^2 F(m_1)}{m_0} \quad (1)$$

3. THEORETICAL MODEL AND IDENTIFICATION METHOD

A solid cylindrical, homogeneous, isotropic and opaque sample is submitted to a heat pulse on its front face in $z = 0$. 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 :

$$\frac{\partial^2 T}{\partial z^2} + \frac{1}{r} \cdot \frac{\partial T}{\partial r} + \frac{\partial^2 T}{\partial r^2} = \frac{1}{a} \cdot \frac{\partial T}{\partial t} \quad (2)$$

Boundary and initial conditions

$$\text{in } z = 0 \quad \lambda \cdot \frac{\partial T}{\partial z} = h_1 \cdot (T(r, z, t) - T_0) - Q\delta(0) \quad (3)$$

$$\text{in } z = e \quad -\lambda \cdot \frac{\partial T}{\partial z} = h_3 \cdot (T(r, z, t) - T_0) \quad (4)$$

$$\text{in } r = R \quad -\lambda \cdot \frac{\partial T}{\partial r} = h_2 \cdot (T(r, z, t) - T_0) \quad (5)$$

$$\text{in } t = 0 \quad T(r, z, 0) = T_0 \quad (6)$$

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

- the model is linear (thermophysical properties are considered as 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 Table I) and solved [4] in order to obtain the dimensionless temperature $\theta^*(r^*, l, t^*) = f(t^*, H, r^*, R^*)$ in a point of the rear face $z = e$ ($z^* = 1$).

Theoretical and experimental thermograms normalized by their maxima are written:

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

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

$$\left\{ \begin{array}{ll} m_0^* = \int_{t_{0.1}^*}^{t_{0.8}^*} f^*(t^*) dt^* & \text{and} \quad m_{-1}^* = \int_{t_{0.1}^*}^{t_{0.8}^*} \frac{f^*(t^*)}{t^*} dt^* \\ m_0 = \int_{t_{0.1}}^{t_{0.8}} f(t) dt & \text{and} \quad m_{-1} = \int_{t_{0.1}}^{t_{0.8}} \frac{f(t)}{t} dt \end{array} \right. \quad (8)$$

The limits of integration are equal to the time $t_{0.1}$ 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:

$$m_0 = m_0^* \cdot e^2 / a \quad \text{and} \quad m_{-1} = m_{-1}^* \quad (9)$$

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

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

The expression (1) is then given by combining relations (9) and (10). The relation F , called 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.

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

4. IDENTIFICATION OF UNCERTAINTY SOURCES

Uncertainties evaluation requires a fine analysis of the measurement process. The use of the Ishikawa method allows to identify all possible causes of uncertainty. Causes can be mainly distributed in five major categories which are *Measurement means*,

Method, Materials, Medium (surroundings) and *Manpower* (user). Within each category, uncertainty causes are identified and presented in the following paragraphs.

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 comes 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 with which the model was determined. Although the method presented previously aims at correcting their effects, uncertainty factors come from badly controlled boundary conditions : finite pulse-time, non-uniformity of the laser, thermal losses, non-linearity.

Uncertainty due to the pulse duration depends on the thermal properties and the thickness of the sample and on the pulse characteristics (duration, energy and temporal shape). The time when the laser pulse occurs is generally taken as origin ($t=0$). When the duration of the pulse is not negligible in comparison with the rise time, the origin of time is shifted by a suitable value t_g [5] [6].

The uncertainty due to the non-uniformity 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 non-linearity effects are due to the variation of the thermophysical properties of the sample with the temperature during the test though 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 at 5 K maximum.

The uncertainty on the assumptions associated to 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 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 5K.

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 on the thickness for a temperature higher than room temperature.

4.4. Medium (surroundings)

The uncertainty factors relating 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 instance).

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 for example the thermal diffusivity of metals at very low temperatures. In this case, the diffusivity considerably varies with the temperature (up to 10 % per degree). We also can note the case of iron about which thermal diffusivity strongly varies with the temperature near the Curie point (≈ 770 °C). The determination of thermal diffusivity is then very sensitive to an error on the temperature measurement.

4.5. Manpower (user)

The uncertainty sources due to the people depend on expertise of the physician 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

Uncertainty sources being identified, the analytical expression of the uncertainty on 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 thermal diffusivity due to uncertainty on the test temperature T .

Assuming that first 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 , finally the variance of a calculated from the equation (1) is expressed as follows:

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

with

$$\left\{ \begin{array}{l} c_e = \frac{2 \cdot e \cdot \sum_{i=0}^n b_i \cdot m_{-1}^i}{m_0} \quad c_F = \frac{e^2}{m_0} \\ c_{m_0} = \frac{-e^2 \cdot \sum_{i=0}^n b_i \cdot m_{-1}^i}{m_0^2} \quad c_{m_{-1}} = \frac{e^2 \cdot \sum_{i=1}^n i \cdot b_i \cdot m_{-1}^{i-1}}{m_0} \end{array} \right.$$

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 (13)

$$m_k = \int_{t_{0,1}}^{t_{0,8}} f(t) \cdot t^k dt \quad (13)$$

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

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

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 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:

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

with

$$\left\{ \begin{array}{l} c_{U(t)} = \frac{1}{U_{\max} - U_0} = \frac{1}{\Delta U_{\max}} \qquad c_{U_{\max}} = \frac{-(U(t) - U_0)}{(U_{\max} - U_0)^2} = \frac{-f(t)}{\Delta U_{\max}} \\ c_{U_0} = \frac{[(U(t) - U_0) - (U_{\max} - U_0)]}{(U_{\max} - U_0)^2} = \frac{f(t) - 1}{\Delta U_{\max}} \end{array} \right.$$

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

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

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.

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

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

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

The two lastest terms of the formula (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.

$$e = e_0 + \Delta_e \quad (18)$$

Uncertainty on the thickness e results from the combination of the uncertainty of measurement thickness e_0 and uncertainty on the correction Δ_e .

$$u^2(e) = u^2(e_0) + u^2(\Delta_e) + 2 \cdot u(e_0, \Delta_e) \quad (19)$$

Uncertainty on the thickness e_0 results from the combination of the uncertainties due to the repeatability 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:

$$u^2(e_0) = u_c^2(e_0) + u_r^2(e_0) + u_R^2(e_0) \quad (20)$$

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

$$\Delta_e = \alpha_l \cdot e_0 \cdot (T - T_a) \quad (21)$$

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.

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

$$u^2(\Delta_e) = (e_0 \cdot (T - T_a))^2 \cdot u^2(\alpha_l) + (\alpha_l \cdot (T - T_a))^2 \cdot u^2(e_0) + (\alpha_l \cdot e_0)^2 \cdot (u^2(T) + u^2(T_a)) \quad (22)$$

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

$$u(e_0, \Delta_e) = (\alpha_l \cdot (T - T_a))^2 \cdot (u_R^2(e_0) + u_c^2(e_0)) \quad (23)$$

5.3. Variance of the identification function F

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

$$u^2(F) = u_{\text{mod}}^2(F) + u_{\text{hyp}}^2(F) \quad (24)$$

The variance $u_{\text{mod}}^2(F)$, calculated from data resulting from the fit of the function F , is a combination of the three following 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.

$$u_{\text{mod}}^2(F(m_{-1}^*)) = \sum_{j=0}^n \left(\frac{\partial F}{\partial b_i} \right)^2 \cdot u^2(b_i) + 2 \cdot \sum_{i=1}^{n-1} \sum_{j=i+1}^n \frac{\partial F}{\partial b_i} \cdot \frac{\partial F}{\partial b_j} \cdot u(b_i, b_j) + SE^2 \quad (25)$$

The variance $u_{\text{hyp}}^2(F)$, 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 paragraph 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. These three components being independent, the variance $u_{\text{hyp}}^2(F)$ is written as:

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

5.4. Variance on 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 on T results from the uncertainty due to the resolution and calibration of the temperature measuring chain, the uncertainty due to the stability and the homogeneity of the furnace temperature. These four uncertainty factors being independent, the variance $u^2(T)$ is thus written:

$$u^2(T) = u_r^2(T) + u_s^2(T) + u_h^2(T) + u_c^2(T) \quad (27)$$

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

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

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 II 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 III and IV.

The thermal diffusivity values and the associated uncertainties given in table III 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, Netzsch) involved in this study are presented in figure II. The deviation of all mean values determined by each laboratory from the certified value is less than ± 3.7 %. The expanded uncertainties of the certified values are estimated 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 to 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 analytical expression of measurement uncertainty was obtained by establishing the assessment of uncertainties for the several components. The uncertainty was calculated on the range [20 °C - 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 measurement.

Round robin and certification study show that the uncertainty measurement evaluated by BNM-LNE are in agreement with those calculated by the other european metrological laboratories.

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Table I Nomenclature

a	thermal diffusivity [$\text{m}^2 \text{s}^{-1}$]	t	physical time [s]
c_p	specific heat [$\text{J kg}^{-1} \text{K}^{-1}$]	t^*	dimensionless time ($= a t / e^2$)
e	thickness of the sample [m]	T	temperature [K]
F	identification fonction	T_0	reference temperature [K]
H	Biot number ($= h e / \lambda$)	U	voltage delivered by IR detectors [V]
m_0	experimental temporal moment of order 0	z	depth of the measurement point [m]
m_{-1}	experimental temporal moments of order -1	z^*	dimensionless depth ($= z / e$)
m_0^*	theoretical temporal moment of order 0	<u>Greek symbols</u>	
m_{-1}^*	theoretical temporal moments of order -1	α_l	thermal expansion coefficient [K^{-1}]
Q	heat pulse surface density [J m^{-2}]	θ^*	dimensionless temperature ($= \rho c_p e (T - T_0) / Q$)
r	abscissa of the measurement point [m]	λ	thermal conductivity [$\text{W m}^{-1} \text{K}^{-1}$]
r^*	dimensionless abscissa ($= r / e$)	ρ	density [kg m^{-3}]
R	radius of the sample [m]	<u>Superscript</u>	
R^*	dimensionless radius ($= R / e$)	* refers to dimationless quantities	

Table II Uncertainty budget of thermal diffusivity determination (Armco Iron)

<i>Quantity</i>	<i>Estimate</i>	<i>Variance or covariance</i>	<i>Sensitivity coefficient</i>	<i>Relative weight</i>
X_i	x_i	$u^2(x_i)$ or $u(x_i, x_j)$	$\partial a / \partial X_i$	(%)
T [°C]	20	1.952E-01	3.919E-08	0.2
e [mm]	3.006E-03	2.633E-12	1.338E-02	0.3
$m_{.1}$	0.5353	6.619E-06	7.969E-05	29.1
m_o [s]	0.03643	2.184E-08	5.517E-04	4.6
$u(m_o, m_{.1})$	/	3.276E-07	/	20.0
F	0.08119	1.075E-06	2.478E-04	45.8
a [m ² /s]	Variance	Uncertainty		
		Standard [m ² /s]	Expanded [m ² /s]	Expanded [%]
2.012E-05	1.4430E-13	3.798E-07	7.597E-07	3.78

Table III Measurement Uncertainties of thermal diffusivity on Pyroceram 9606

Temperature (°C)	Thermal Diffusivity ($10^{-6} \text{ m}^2\text{s}^{-1}$)	Expanded Uncertainty (k=2)	
		($10^{-6} \text{ m}^2\text{s}^{-1}$)	(%)
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 IV Measurement Uncertainties of thermal diffusivity on Armco Iron

Temperature (°C)	Thermal Diffusivity ($10^{-6} \text{ m}^2\text{s}^{-1}$)	Expanded Uncertainty (k=2)	
		($10^{-6} \text{ m}^2\text{s}^{-1}$)	(%)
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

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

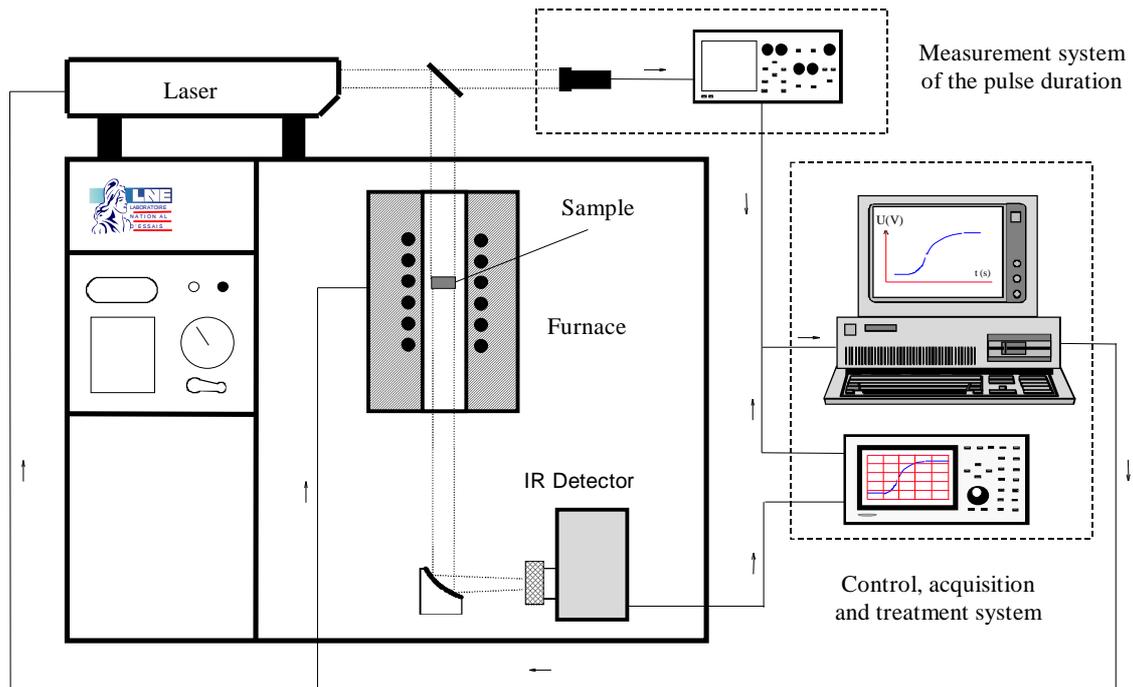
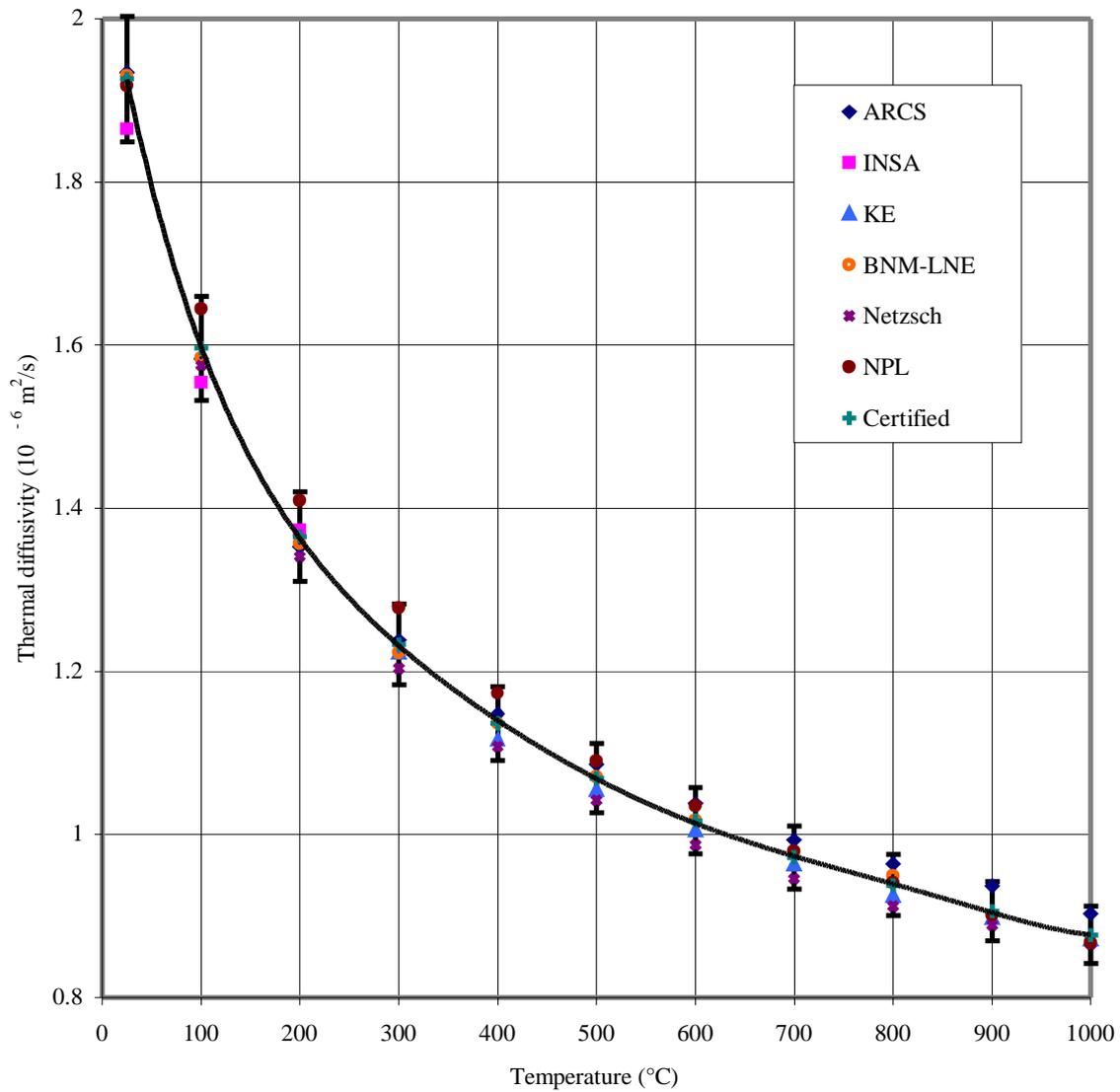


Fig.2. Certified thermal diffusivity and men values of all partners [2]



- 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 Netzsch Gerätebau GmbH (Selb/Bayern - Germany)
- NPL National Physical Laboratory (Teddington - England)