XIX IMEKO World Congress
Fundamental and Applied Metrology
September 6–11, 2009, Lisbon, Portugal

CALCULATED UNCERTAINTY OF THE THERMAL DIFFUSIVITY MEASUREMENT BASED ON FLASH LASER METHOD

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Abstract – Flash methods have become one of the most commonly used techniques for measuring the thermal diffusivity and thermal conductivity of solids and liquids. This method has received standard status for the measurement of thermal diffusivity of materials such as metals, carbon materials, ceramics and polymers. An uncertainty analysis of the thermal diffusivity measurement using the laser flash method will be presented in this paper. This metrological investigation follows the general rules for evaluating and expressing the uncertainty in the *LMPT – Laboratório de Medição de Propriedades Termofísicas* (Thermophysical Properties Measurement Laboratory), summarizing the main sources of uncertainties. Experiments were carried out on Pyroceram 9606 at room temperature to determine the uncertainty assign to the thermal diffusivity measurements.

Keywords: Flash method; Thermal diffusivity; Uncertainty analysis

1. INTRODUCTION

The flash method for measuring thermal diffusivity has been increasingly used since its introduction in 1961, by Parker et al [1]. Numerous corrections have been taken into account for radiation heat losses during process, the finite width of the laser pulse, the non-uniform heating of the sample, and other non-measurement errors [2-9]. Nowadays, it is a widespread technique for measuring the thermal diffusivity and thermal conductivity of solid and liquid materials (metals, carbon materials, ceramics, polymers, including radioactive materials). Easy sample preparation, small sample dimensions, fast measurements times, and high accuracy are only some of the advantages of this non-destructive measurement technique.

In every measurement, the difference between the real value and the measured value for a quantity, in this case thermophysical properties, is affected by the measurement errors. All measurements are inexact and therefore requires a statement of uncertainty to quantity that inexactness. The uncertainty of a measurement is the doubt assign to the measurement result. By quantifying the level of inexactness of a measurement, we can estimate the significance level of the expressed results. An uncertainty budget is required in order to verify if the result is adequate for its intended purpose and consistent with other similar results. It does not matter how accurate a measuring instrument is considered to be, the measurements will always be subject to a certain amount of uncertainty. In order to express the uncertainty of a measurement, we need to evaluate as accurately as possible the errors assign to each source of uncertainty of that particular measurement.

This paper deals with the uncertainties estimation of the thermal diffusivity measurements performed by the Nuclear Technology Development Center using a laser flash apparatus of the Thermophysical Properties Measurement Laboratory. Uncertainty was estimated according to the ISO/BIPM Guide to the Expression of Uncertainty in Measurement [10], which gives a general method for the evaluation of measurement uncertainties.

2. EXPERIMENTAL

The laser flash apparatus (Fig. 1) is regularly used for measurements of the thermal diffusivity of solids at the Nuclear Technology Development Center - CDTN. It consists of CO_2 Laser working at 10,6 µm wave length. A pulse of energy is applied into the sample and usually set to keep the sample temperature rise below 3 °C. An infrared thermometer measures the transient temperature. The acquisition of temperature versus time curve is performed with a NI-6210 multifunction Data Acquisition. A LabView program is used to acquire data. The sample (8 mm in diameter and about 1 mm to 2,5 mm thick) is placed in a vacuum furnace and isothermally heated. The sample holder consists of three molybdenum screws that fix the sample in vertical position in the central zone of the furnace. The system allows the irradiation of the sample in its frontal face obtaining a register of the transient of temperature on the sample opposite face.

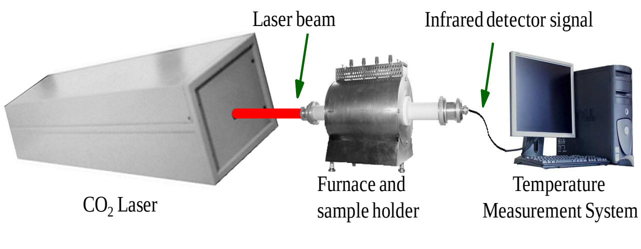

Fig. 1. Thermophysical Properties Measurement Laboratory Experimental Apparatus.

The resulting temperature rise of the opposite face is registered in a thermogram. The thermal diffusivity α is calculated from the sample thickness L and the time $t_{1/2}$ required to the temperature rise reach one-half of its maximum value (1):

$$\alpha = \frac{1{,}37 \cdot L^2}{\pi \cdot t_{1/2}}. \qquad (1)$$

3. UNCERTAINTY COMPONENTS

The measurements were performed on carbon-coated sample under vacuum atmosphere at room temperature. A laser beam diameter of about 8 mm and a sampling frequency of 1 kHz were used. The thermal diffusivity was obtained from the thickness of the sample and from the half-time describing the heat diffusion from the front to the opposite sample face. The sources of uncertainties in the measurement were associated with the sample itself, temperature measurement, time measurement, non-uniform heating of the sample and heat losses [11-12].

3.1. Uncertainties associated with the sample

The sources of uncertainty concerning to the sample are due to its geometrical quality and its chemical and optical properties. The thickness of the sample at a temperature (L) is calculated from the thickness measured at room temperature (L_0) corrected by a term to taking into account the expansion of the sample (Δ_L). Mathematical expression of the thickness of the sample is written as

$$L = L_0 + \Delta_L. \qquad (2)$$

The uncertainty in the thickness results from the combination of the uncertainty of measurement thickness (L_0) and the uncertainty in the correction due to expansion of the sample (Δ_L):

$$u_c^2(L) = u^2(L_0) + u^2(\Delta_L) + 2u(L_0, \Delta L). \qquad (3)$$

The sample thickness is measured with certified micrometer. The uncertainty of thickness results from the uncertainty due to repeatability of measurements, resolution, calibration, flatness, parallelism, drift of micrometer and the correction of thermal expansion. The uncertainty was calculated based on the uncertainty in thermal expansion for a typical expansion coefficient of 10^{-6} K^{-1} [12] and a 10 K temperature gradient. The uncertainty in thermal diffusivity resulting from thickness measurement was estimated in 0,11 %.

3.2. Uncertainties associated with the temperature

The temperature of the sample is not considered by the Eq. 1 but the uncertainty of the half-time and consequently the uncertainty of thermal diffusivity depends on the sample temperature. The uncertainty factors related to the sample temperature correspond to the experimental conditions especially induced by the furnace temperature, its inner atmosphere and all other sources associated with the radiation thermometer.

The uncertainty of radiation thermometer results from the uncertainty due to resolution, calibration, drift, time constant of thermometer, calibration of signal unit and other factors as effective emissivity of the sample, stability and the homogeneity of the furnace temperature. Drift in radiation thermometers between calibrations arises from changes in the optical components, in the radiation detector and in the signal processing system. Non-linearity is caused by the non-ideal performance of the detector and electronics. In our measurement s the opposite face temperature increase is always kept below 3 ºC. The non-linearity effect of the IR thermometer is assumed to be negligibly for small temperature changes (smaller than 10 ºC). The sample is coated on both sides with carbon film to improve and keep controlled the sample emissivity and absortivity. The uncertainty due to sample emissivity was estimated in 2 % [13].

Based on these characteristics, it was assumed that the uncertainty in the temperature $u(T)$ results from the combination of the uncertainties due to repeatability of measurements $u_r(T)$, the calibration u(C), the resolution $u(C_R)$, the drift of thermometer $u(C_d)$ and the emissivity $u(C_\varepsilon)$:

$$u^2(T) = u_r^2 + u^2(C) + u^2(C_R) + u^2(C_d) + u^2(C_\varepsilon). \qquad (4)$$

The uncertainty in temperature sample was estimated in 1,1 ºC.

3.3. Uncertainties associated with the time scale/finite pulse time effect

Since the measurement of thermal diffusivity is, in essence, a time measurement, it is important to know as good as possible where the time origin lies. The uncertainty on the time measurement results from the combination of the uncertainties due to measurement instruments and data acquisition board.

The manufacturer states the following characteristics of the used data acquisition board:

- signal resolution of 16 bit (1 in 65 536 or 0,002 %);
- timing resolution 50 ns and
- timing accuracy 0,1μs.

Based on these characteristics, the uncertainty due to digital data acquisition board was evaluated in 0,01 %. The sampling frequency was set to 1 kHz. The time origin measurement error was evaluated in 1 ms. The error assign to the sample frequency is lower than 0,2 %. Therefore, the uncertainty in thermal diffusivity resulting from time scale was evaluated in 1,66%.

When the duration of the pulse is not negligible in comparison with the half-time, a finite pulse time effect correction must be performed. A computer simulation program was used to estimate the influence of finite time pulse effect on the thermal diffusivity [14]. The uncertainty in thermal diffusivity resulting from finite pulse time effect was estimated in 1,45 % for Pyroceram 9606.

3.4. Uncertainties associated with the non-uniform heating

The sample heating uniformity is directly related to the uniformity of the laser beam. The uniformity of a laser may change from shot to shot and it is also dependent on the energy level of the laser beam. A 3 % uncertainty due to the effect of non-uniform heating was assumed in this uncertainty budget [15].

3.5. Uncertainties associated with the heat losses

During a flash laser experiment, heat losses from the sample are unavoidable. The losses are very small at low temperatures, but can increase considerably at higher temperatures. The contribution of the heat losses is expressed by an overall heat transfer coefficient (U). The contribution of the heat losses is expresses by Biot number, B_{i}, defined as in (5)

$$B_i = \frac{U \cdot L_c}{k} < 0{,}1 \qquad (5)$$

where L_c is characteristic length and k is thermal conductivity.

Computational simulations were used to estimate the influence of the heat losses on the thermal diffusivity. The simulation was run for three reference samples (Pyroceram 9606, Inconel 600 and Pure Fe). In all cases, the influence of heat losses on the thermal diffusivity accuracy was estimated in 2 % [14].

3.6. Other uncertainties

There are other sources of uncertainty such as electronic noise, etc. These usually can be easily corrected or have a very small effect on the determination of thermal diffusivity, and so, were not evaluated individually.

The long-term stability is monitored by LMPT measuring periodically the thermal diffusivity of Pyroceram 9606. The measurements were performed on carbon film-coated sample (2,5 mm thick) under vacuum atmosphere at temperature room. Moreover, the reliability of the measurement is checked comparing the measured temperature rise vs. time evolution with the analytical curve as well as analyzing if the experimental conditions from those assumed in the analytical model can be easily identified.

4. RESULTS

Thermal diffusivity presented was estimated from the average of fifteen measurements carried out on repeatability conditions. The mean of the standard deviation was estimated be 0,95 % of the measured value. Thermal diffusivity measured is 1,92 % higher than the mentioned in the certificate. The Table 1 presents all quantities considered as components of uncertainty in this analysis, its assign standard uncertainty and combined standard uncertainty. This analysis followed the general rules for evaluating and expressing uncertainty in measurements, based on the GUM uncertainty framework (Guide to the Expression of Uncertainty in Measurement) [10].

Table 1. Uncertainty budget for Pyroceram 9606 at 25 ºC.

Uncertainty components	Standard Uncertainty
Repeatability	0,95 %
Sample thickness	0,11 %
Time scale	1,66 %
Finite pulse time effect	1,45 %
Non uniform heat effect	3,0 %
Heat losses	2,0 %
Combined standard uncertainty	4,33 %
Expanded uncertainty (k = 2)	8,66 %

5. CONCLUSIONS

A measurement is never guaranteed to be perfect. The uncertainty expression is important to anybody who wishes good quality measurements. This paper presents results of an uncertainty analysis of Pyroceram 9606 thermal diffusivities using the laser flash method apparatus installed in the CDTN. The relative expanded uncertainty (k = 2) of the thermal diffusivity determination is estimated to be about 8,66 % .

ACKNOWLEDGMENTS

The authors wish to thanks CNPq Conselho Nacional de Desenvolvimento Científico e Tecnológico) and FAPEMIG (Fundação de Amparo à Pesquisa do Estado de Minas Gerais), for financial support for the research.

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