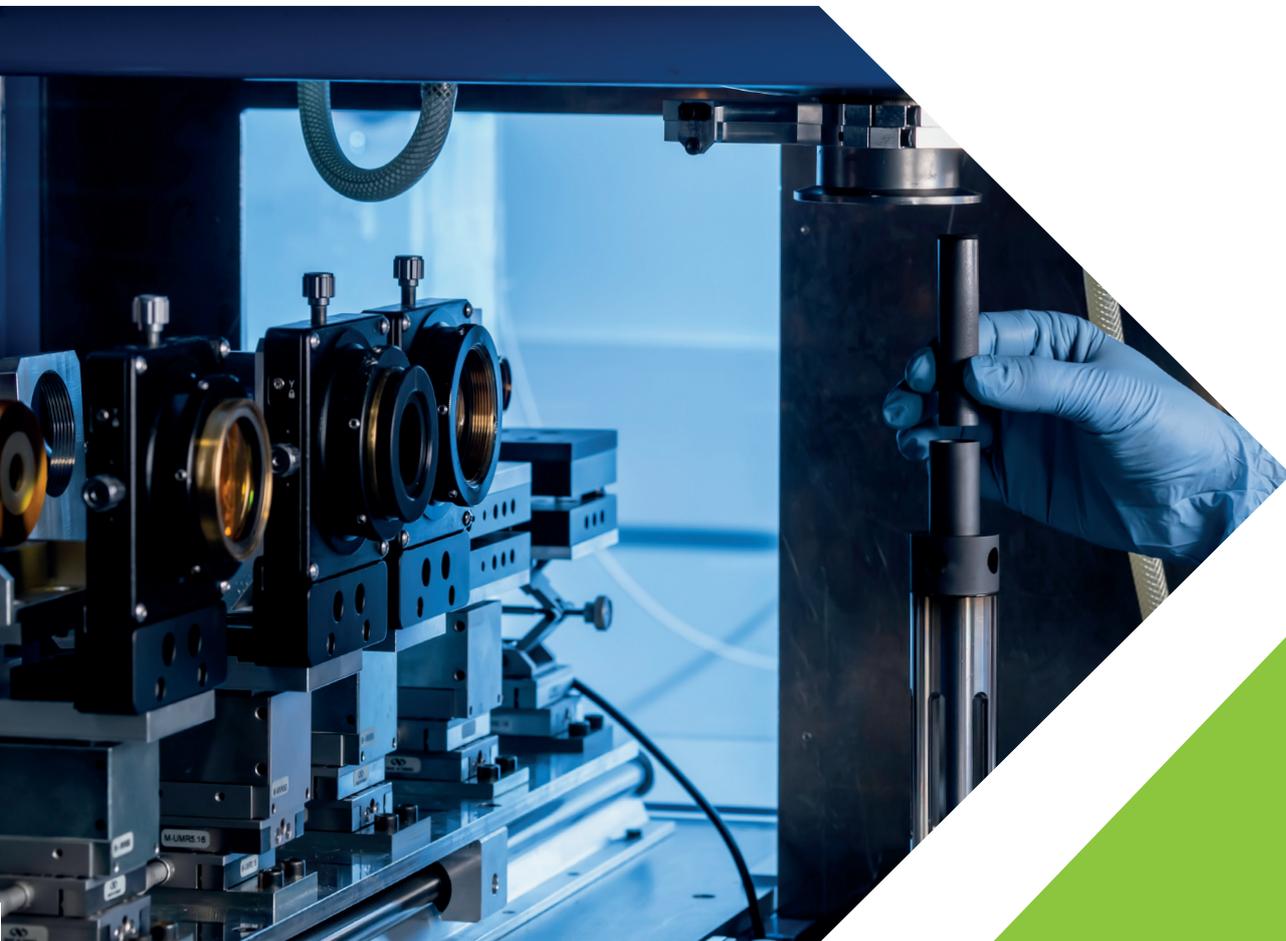


High Temperature Thermal Diffusivity Measurements by the Laser Flash Method



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Thermometry

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Technical Guide on High Temperature Thermal Diffusivity Measurements by the Laser Flash Method

Purpose

This document has been produced to enhance the equivalence and mutual recognition of high temperature thermal diffusivity measurement results obtained by laboratories.

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SCOPE

Measurement practices in the laboratories for determining the thermal diffusivity of solid materials are in many cases based on national or international standards. The most important standards related to this topic are given below:

- EN 821-2 (1997) “Advanced technical ceramics - Monolithic ceramics - Thermophysical properties - Part 2: Determination of thermal diffusivity by the laser flash (or heat pulse) method”
- ISO 18755 (2022) “Fine ceramics (advanced ceramics, advanced technical ceramics) - Determination of thermal diffusivity of monolithic ceramics by laser flash method”
- ASTM E1461-13 (2022) “Standard test method for thermal diffusivity by the flash method”
- ISO 22007-4 (2024) “Plastics - Determination of thermal conductivity and thermal diffusivity - Part 4: Light flash method”
- ISO 19629 (2018) “Fine ceramics (advanced ceramics, advanced technical ceramics) - Thermophysical properties of ceramic composites - Determination of unidimensional thermal diffusivity by flash method”

These standards describe the general methodology to be applied to measure the thermal diffusivity according to the laser flash method, without giving specific recommendations for measurements performed at high temperatures.

The purpose of this guide is to outline the basic technical requirements for performing thermal diffusivity measurements of solid materials at high temperature (between 500 °C and 3000 °C) by applying the laser flash method. The aim of this document is not to replace or harmonize the existing standards, but it could serve as a reference for future standard revisions.

1 GENERAL COMMENTS

The laser-flash method is by far the most frequently used method to measure the thermal diffusivity of solid materials [1]. In this method, a thin disk shaped specimen is maintained at a uniform temperature in a furnace operating either by resistive or inductive heating. One side of the specimen is then subjected to a short thermal excitation generated by a laser pulse (with sufficient energy to create a measurable temperature change). The laser pulse should have a uniform spatial energy distribution. The induced transient temperature rise on the opposite side is measured versus time with an infrared detector. An example of the laser flash experiment is presented in Figure 1 for inductive heating.

The thermal diffusivity is obtained by comparing the measured temperature-time curve (thermogram) with a theoretical model describing the transient heat conduction through the specimen. Various models and identification methods can be used to calculate the thermal diffusivity from the measured thermograms. The original method proposed by Parker [1], which assumes ideal conditions (adiabatic experiment, energy uniformity and infinitesimal duration of the laser pulse, homogenous and opaque specimen), shall not be applied in particular at high temperatures. Alternative methods enabling correction for non-ideal initial and boundary conditions, are more appropriate, especially at high temperatures, and shall be used (cf. section 4.2).

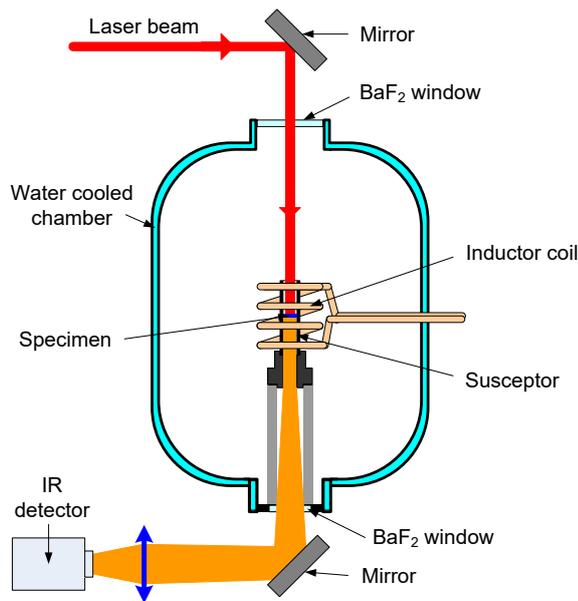


Figure 1. Schematic diagram of a laser flash experiment

The following sections give specific recommendations and guidance for thermal diffusivity measurements with the laser flash method above 500 °C.

2 RECOMMENDED REQUIREMENTS FOR THE LASER FLASH APPARATUS

2.1 FURNACE

The test chamber should be either a resistive or an inductive furnace capable of operation within the required temperature range. It should be equipped with two windows, one transparent to the pulse radiation and the other transparent to the working wavelength range of the IR detector measuring the thermogram. BaF₂ (Barium fluoride) windows are recommended for thermal diffusivity measurements performed at very high temperatures, as their transmission is higher than 90 % from 0.25 μm to 10 μm wavelength (CaF₂ (Calcium fluoride) windows with transmission higher than 90 % from 0.4 μm to 8 μm can also be used).

Tests may be performed either under vacuum or under inert gas (for example argon or helium). Even if measurements under vacuum reduce convection losses, it is recommended to work with a high purity inert gas at very high temperatures (argon or helium up to 2000 °C, and preferably helium above 2000 °C) in order to avoid sublimation phenomena of the tested specimens. If the tests are performed with inert gas, the specimen should be in a horizontal position in order to reduce the effect of gas convection on it.

Additional precautions should be taken if an inductive system is used to heat the specimen. The specimen should be placed in a graphite holder (also named susceptor) which is heated by induction from a surrounding coil connected to a high frequency generator. A filter should be placed at the output of the high frequency generator for radio frequency interference protection of power lines. The inductive coil, which is usually made of copper, should be coated with an electrically insulating ceramic deposit (few hundred micrometer thick) in order to avoid the occurrence of sparks between the susceptor and the coil that can damage the susceptor and disturb the measurements.

2.2 SPECIMEN HOLDER

The specimen holder should hold the specimen stable with minimum thermal contact in order to limit conductive heat losses and should be designed to suppress stray light from the laser beam being transmitted to the IR detector.

Depending on the tested material, the specimen holder should be made of either compatible refractory metallic materials (if the tested material is a pure metal or a metallic alloy) or graphite to limit chemical interactions between the holder and the specimen at high temperatures. To avoid contact between a metallic specimen and a graphite specimen holder, an alternative solution is to put a thin washer made of molybdenum or tungsten between the specimen and the holder. If available, it is recommended to consult relevant material phase diagrams when choosing the appropriate type of material for the specimen holder.

2.3 DETECTOR

The transient temperature rise of the specimen rear side (thermogram) should be measured by optical means with an infrared detector, which shall be carefully chosen depending on the temperature range. HgCdTe (Mercury cadmium telluride) or InSb (Indium antimonide) IR detectors are recommended from 23 °C to 1000 °C, and InGaAs and Si detectors for test temperatures ranging from 1000 °C to 3000 °C.

2.4 THERMOCOUPLE AND RADIATION THERMOMETER CALIBRATION

Below 1200 °C the steady-state temperature of the specimen should be measured before the laser pulse with a calibrated thermocouple (for example type K, N, S or R thermocouples) located close to the specimen. For higher temperatures, it should be measured with infrared radiation thermometers (or pyrometers) having an operating temperature range covering that of the furnace. Preferably bi-chromatic radiation thermometers should be used, as in that case the knowledge of the emissivity of the specimen is not needed. However, even the application of a bi-chromatic radiation thermometer needs careful evaluation, because rapid changes in emissivity might critically affect the measured temperature [2].

The error on the temperature measurements by radiation thermometry in the laser flash apparatus can be greater than 50 °C at 3000 °C if the pyrometers are not calibrated. Even if the sensitivity of the thermal diffusivity to temperature is low at high temperatures, this error has an influence on the uncertainty of thermal diffusivity measurements. Therefore, radiation thermometers, which can be subjected to drift with time, need to be periodically calibrated to ensure trueness of the temperature measurements.

The temperature calibration of the applied radiation thermometer can be done in-situ by using metal-carbon eutectic (for example Co-C, Pd-C, Pt-C, Ir-C, etc.) high temperature fixed points (HTFP). The cross-section of a small HTFP crucible (main part of the cell and cap) is shown in Figure 2 (left). In the temperature calibration configuration, the laser is disabled, and the radiation thermometer is aimed at the aperture of the HTFP blackbody cavity positioned in the furnace at the location of the specimens (cf. Figure 2 - right). The HTFP cell is thermally stabilized at a temperature higher or lower than the phase change temperature of the metal carbon eutectic used, and its temperature is continuously measured by the radiation thermometer while heating or cooling the HTFP until the material is fully melted or solidified. The in-situ calibration of the radiation thermometer is performed by comparing the value of the phase change temperature (freezing or melting temperature) measured and the reference value. More details about this procedure can be found in [3].

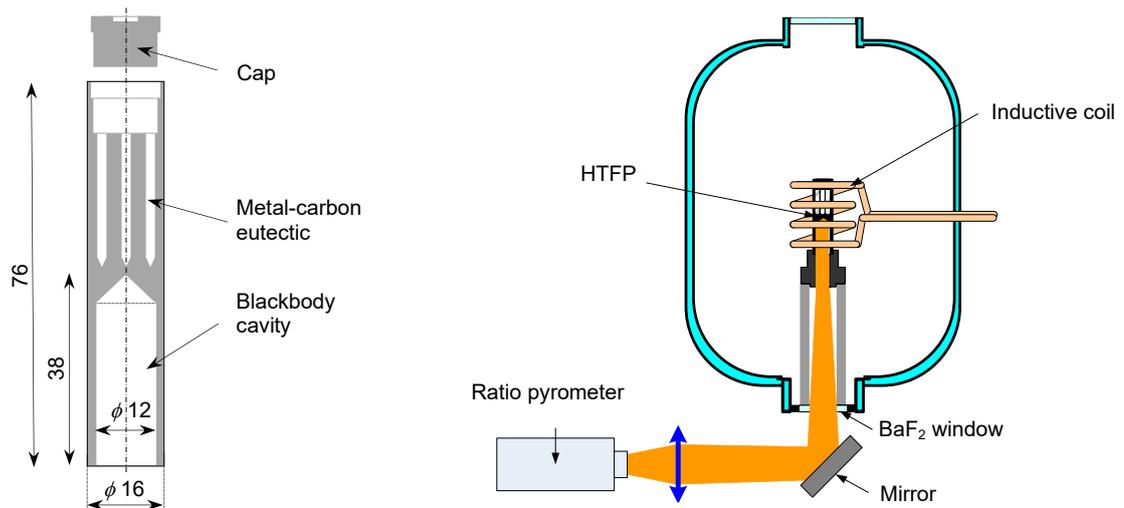


Figure 2. In-situ calibration of radiation thermometer using small HTFP

Note: It is usually advised to use the melting point of metal carbon eutectics as temperature reference instead of the freezing point which is less reproducible. However, due to technical constraints linked to the use of inductive furnace, it is sometimes needed to use the freezing point knowing that the error induced is negligible with respect to the other uncertainty sources.

3 PREPARATION OF THE TEST

3.1 TEST SPECIMEN PREPARATION

The specimen is typically a disk whose faces perpendicular to the axis of the optical measurement system shall be flat and parallel. The parallelism error of the two faces shall be less than 2 % of the specimen thickness. The specimen thickness shall be larger than 1 mm (thickness of 2 mm or 3 mm are typically used) in order to avoid a specimen deformation which can be observed at high temperature if the specimen is too thin.

The specimen should be representative of the material being examined and shall be prepared and handled with care (preferably with gloves) to avoid any contamination.

In the case of a specimen with low emissivity, it may be necessary to sandblast, grind or graphite coat the specimen surface in order to increase its emissivity and therefore to improve the signal-to-noise ratio of the infrared detector output. However, the use of graphite coating can have an influence on the measurements due to potential interaction with the specimen material at high temperatures.

3.2 CLEANING OF THE FURNACE

It is recommended to clean the furnace and the specimen holder after a series of measurements above 2000 °C. At these high temperatures particles can indeed be released from parts of the furnace and the specimen holder when they are made of graphite and can then settle on the windows or contaminate the tested specimens.

In the case of inductive heating, the presence of graphite particles in the furnace drastically increases the risk of sparks between the susceptor and the coil at very high temperatures.

4 MEASUREMENT PROCEDURE

As for the previous sections, the general recommendations given in the CEN, ASTM and ISO standards listed in Section 1 regarding the measurement procedures of thermal diffusivity should be applied. The present section gives additional advice for thermal diffusivity measurements performed from 500 °C to 3000 °C.

4.1 MEASUREMENTS

The steady-state temperature T assigned to the measured thermal diffusivity value is that of the specimen measured before the laser pulse. Nevertheless, the energy deposited by the laser pulse generates an increase ΔT of the specimen temperature by few degrees, which depends on the emissivity and specific heat of the material. In fact, the thermal diffusivity assigned to the test temperature T is thus that corresponding approximately to the temperature $T + \Delta T$. The uncertainty associated with this bias is usually negligible compared to the other uncertainty components for temperatures higher than 500 °C, where the sensitivity of the thermal diffusivity to temperature is usually weak (cf. example given in Figure 3 for isotropic graphite).

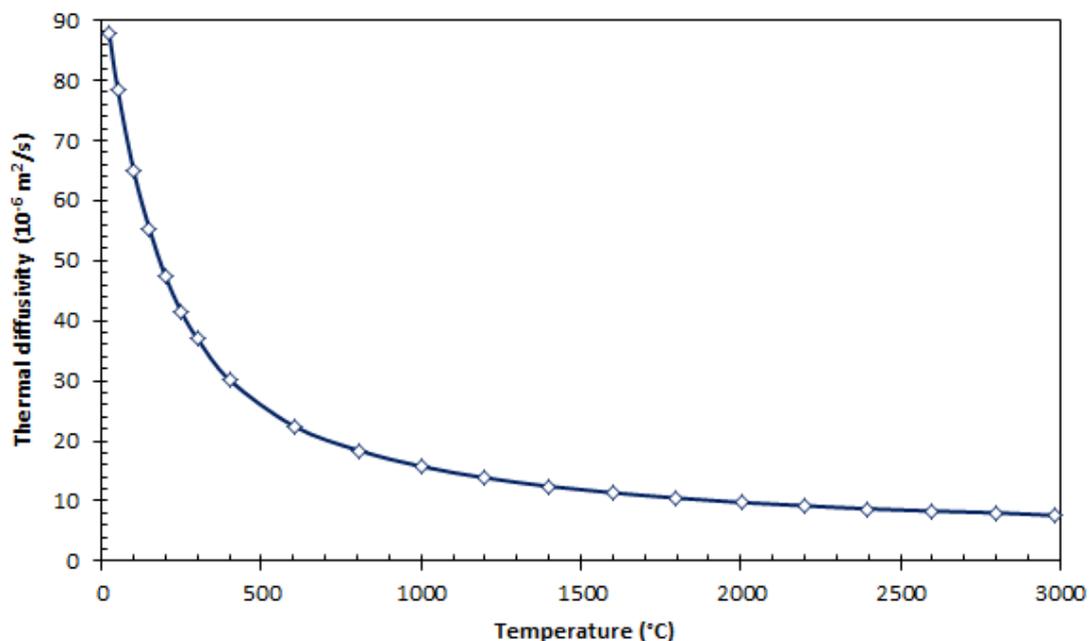


Figure 3. Thermal diffusivity of the isotropic graphite IG210 [4]

However, this bias needs sometimes to be corrected when transitions occur in the tested material at high temperature and generate strong variation of thermal diffusivity in the vicinity of the transition temperature. A typical example is shown in Figure 4 for pure iron for which the thermal diffusivity strongly varies with temperature near the Curie point (≈ 770 °C) [5].

When needed, the influence of the laser pulse energy can be directly corrected by performing thermal diffusivity measurements at the same temperature T for different energy levels of the laser pulse and by extrapolating the obtained results to those for a zero energy pulse [6]. This extrapolated value corresponds then to the thermal diffusivity at the temperature T .

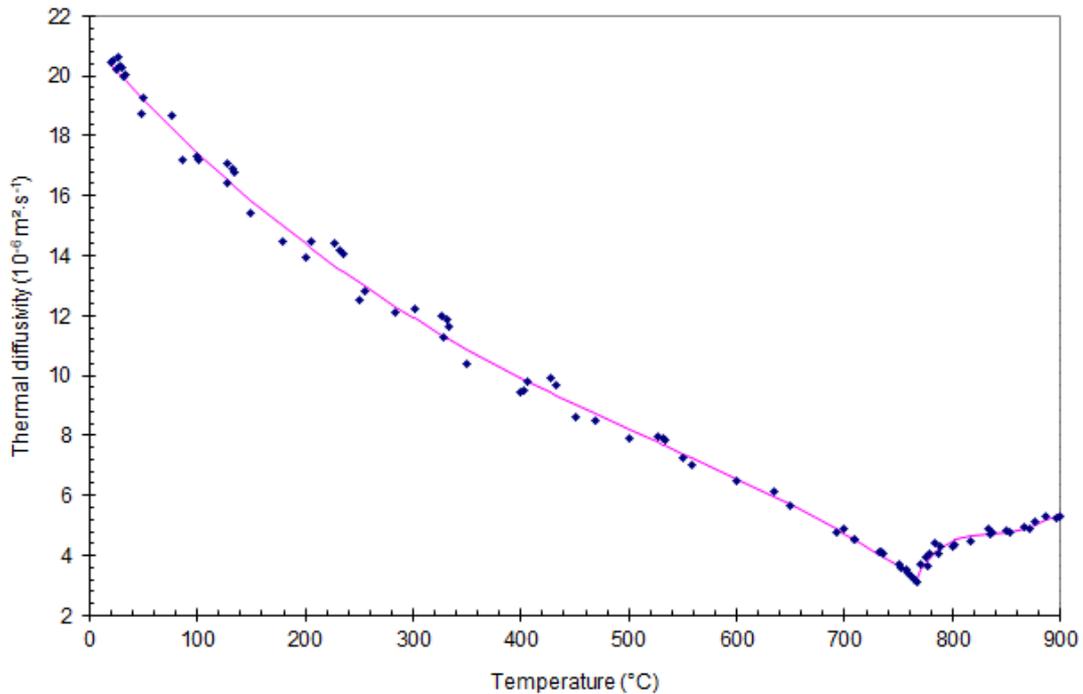


Figure 4. Thermal diffusivity of pure iron [5]

Two different measurement procedures can be applied depending on the sensitivity of the thermal diffusivity of the tested material to the temperature.

Procedure 1 (without correction of the influence of the laser pulse energy)

- 1) Measure the thickness and the mass of the specimen at room temperature before starting the test.
- 2) The thermal diffusivity measurements should be performed initially at 23 °C and subsequently at different temperatures covering the investigated temperature range when heating up and/or cooling down the specimen. This series of measurements should be repeated at least during a second heating/cooling cycle and a final measurement should be made at 23 °C.

The aim of this protocol is to check the stability of the material when heated to a high temperature and to detect potential modifications of the specimen microstructure, which can be different depending on the heating/cooling rates and the holding times at high temperatures.

It is recommended to perform at least three consecutive measurements at each level of temperature under repeatable conditions, the average of the three obtained results giving the thermal diffusivity value.

- 3) Measure the thickness and the mass of the specimen at room temperature after each thermal cycle.

Procedure 2 (with correction of the influence of the laser pulse energy)

- 1) Measure the thickness and the mass of the specimen at room temperature before starting the test.
- 2) Measure the thermal diffusivity for each temperature level covering the investigated temperature range when heating up and/or cooling down the specimen by applying the following protocol:
 - Perform at least three repeated measurements for five different energy levels of the laser pulse using the same amplifier parameters (for some laser flash apparatus, the automatic amplifier gain function needs to be switched off to avoid an auto optimisation of the output signals);
 - Determine the thermal diffusivity and the maximum amplitude of the output signal for each obtained thermogram (after smoothing of the experimental data or fitting to a theoretical model to mitigate noise peaks, if needed) - cf. Figure 5 left;
 - Plot the thermal diffusivity values as a function of the output signal amplitude (cf. Figure 5 right);
 - Determine the linear function passing through these points using the least squares method;
 - Determine the intercept of the linear function that corresponds to the thermal diffusivity value at the temperature of test, *i.e.* without influence of the energy level of the laser pulse (cf. Figure 5 right).

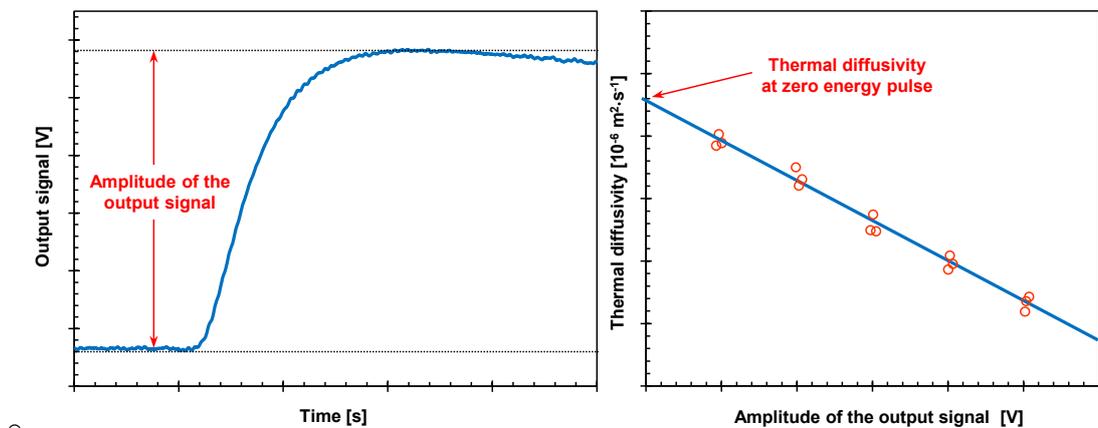


Figure 5. Principle of the extrapolation procedure [6]

This series of measurements should be repeated at least during a second heating/cooling cycle and a final measurement should be made at 23 °C.

- 3) Measure the thickness and the mass of the specimen at room temperature after each thermal cycle.

4.2 DATA ANALYSIS

The calculation of the thermal diffusivity value from the obtained experimental thermogram shall be performed according to methods enabling to take into account the unavoidable heat losses associated to high temperature experiments. These methods can be based on:

- An improvement of Parker's method by introducing correction factors in the calculation of the thermal diffusivity (typically the method proposed by Cape and Lehman [7]);
- An analysis of a part of the thermogram (e.g. the partial time moments method [8]) or of the whole temperature response (e.g. parameter estimation using a least-squares method [9]).

More details can be found in the CEN, ASTM and ISO standards listed in section 1.

This thermal diffusivity value $a_0(T)$ is calculated using the thickness of the specimen d_0 measured before the test at room temperature with a calibrated micrometer.

The correct thermal diffusivity value $a(T)$ based on the specimen thickness d_T at the measurement temperature T is determined from thermal expansion measurements with the following equation.

$$a(T) = \left(\frac{d_T}{d_0}\right)^2 \cdot a_0(T)$$

The higher the temperature, the more important the correction. It is shown in [4] that the error on thermal diffusivity values is about 3 % for measurements performed on molybdenum and tungsten respectively at 2200 °C and 2400 °C and about 4 % for measurements carried out at 3000 °C on graphite specimens, if the corresponding thermal expansion is neglected.

5 UNCERTAINTY OF MEASUREMENT

The relative expanded uncertainty associated with thermal diffusivity measurement should be calculated in accordance with the ISO/BIPM Guide to the expression of uncertainty in measurement [10] by considering the following sources of uncertainties:

- Measurement of the physical quantities involved in the determination of thermal diffusivity (thickness and temperature of the specimen, output voltage of the infrared detector, time base).

Uncertainties associated with the measurements of these physical quantities result mainly from the combination of the uncertainties due to the noise of measurement, the resolution and calibration of the equipment. Another source of uncertainty could be a possible drift of the baseline of the signal delivered by the IR detector due to electromagnetic disturbances induced by the laser pulse.

- Geometrical quality of the specimen (flatness and parallelism of the faces), characteristics (homogeneity, isotropy and opacity) and physical properties (thermal expansion vs temperature and sensitivity of the thermal diffusivity to temperature) of the material.

- Operator interventions to select the minimum and maximum of the experimental curve to be normalized, to correct potential thermogram baseline drift and to measure the specimen thickness at room temperature.
- Experimental conditions such as furnace temperature (stability and homogeneity) and nature of the test atmosphere (vacuum, inert gas, etc.).
- Differences between the experimental conditions and hypotheses used to establish the theoretical model (finite pulse-time, non-uniformity of the laser beam and thermal losses). Another uncertainty source related to the measurement method is the assumption of the linearity of the infrared detector output with respect to temperature.

Among these different factors, the main uncertainty components associated with thermal diffusivity measurement at high temperatures are those related to:

- the “quality” of the experimental curve (noise, drift of baseline during the test, positive or negative shift of the baseline after the laser pulse);
- the differences between the experimental conditions and the assumptions upon which the model has been based.

More details about the assessment of uncertainty associated with high temperature thermal diffusivity measurements can be found in [4].

6 REFERENCE MATERIALS

Reference materials with known thermal diffusivities may be used to check laser flash apparatus. As there are no reference materials with certified values of thermal diffusivity above 1000 °C, three refractory materials (molybdenum, tungsten and IG210 isotropic graphite) have been studied as candidate reference materials in the framework of the European project 17IND11 Hi-TRACE [11] and can be recommended for apparatus checks.

The thermal diffusivity of these three materials has been measured as functions of temperature by seven laboratories on a batch of 2 mm or 3 mm thick specimens machined from the same blocks of materials. The mean values as well as the relative expanded uncertainty ($k=2$) calculated from the results obtained by the participant laboratories [12] are presented in Table 1. These uncertainty values take into account the inhomogeneity of the batches of specimens, the standard deviations of reproducibility as well as the uncertainty on thermal diffusivity measurements reported by the participants [4]. The thermal diffusivity values given for temperature higher than 23 °C have been corrected for the thermal expansion of each studied material.

The isotropic graphite may be used up to 3000 °C, while the use of molybdenum and tungsten should be limited to temperatures lower than 2200 °C and 2715 °C, respectively, due to possible eutectic formation at the surface of the specimens in the case of laser flash apparatus containing graphite parts.

Table 1. Thermal diffusivity of isotropic graphite IG210, molybdenum and tungsten versus temperature [12]

| Temperature (°C) | Isotropic graphite IG210 | | Molybdenum | | Tungsten | |
|---------------------|--|--------------------------------------|--|--------------------------------------|--|--------------------------------------|
| | Thermal diffusivity (10 ⁻⁶ m ² ·s ⁻¹) | Expanded uncertainty (k=2) (%) | Thermal diffusivity (10 ⁻⁶ m ² ·s ⁻¹) | Expanded uncertainty (k=2) (%) | Thermal diffusivity (10 ⁻⁶ m ² ·s ⁻¹) | Expanded uncertainty (k=2) (%) |
| 23 | 84.94 | 7,0 | 54.96 | 7,0 | 67.65 | 4,8 |
| 50 | 76.69 | 6,9 | 53.90 | 7,0 | 66.03 | 4,8 |
| 100 | 63.79 | 6,9 | 51.51 | 6,8 | 62.68 | 5,9 |
| 150 | 54.31 | 6,9 | 49.98 | 6,4 | 60.11 | 5,4 |
| 200 | 46.58 | 5,4 | 48.07 | 7,0 | 57.18 | 5,7 |
| 250 | 40.77 | 5,3 | 46.81 | 6,5 | 55.11 | 5,4 |
| 300 | 36.14 | 5,8 | 45.47 | 6,8 | 52.83 | 6,6 |
| 400 | 29.74 | 5,8 | 42.89 | 4,3 | 49.21 | 5,2 |
| 600 | 22.30 | 5,4 | 39.74 | 4,0 | 44.35 | 5,3 |
| 800 | 18.25 | 5,4 | 37.21 | 4,1 | 41.14 | 5,8 |
| 1000 | 15.63 | 5,6 | 34.78 | 4,9 | 39.12 | 6,1 |
| 1200 | 13.80 | 5,5 | 32.27 | 4,2 | 37.05 | 6,3 |
| 1400 | 12.50 | 5,3 | 29.74 | 4,3 | 35.42 | 6,6 |
| 1600 | 11.53 | 5,4 | 27.18 | 4,2 | 33.77 | 6,6 |
| 1800 | 10.66 | 6,0 | 24.79 | 4,7 | 31.91 | 6,6 |
| 2000 | 9.96 | 5,6 | 22.75 | 5,2 | 29.89 | 6,8 |
| 2200 | 9.39 | 6,0 | 21.09 | 6,0 | 27.79 | 7,0 |
| 2400 | 8.93 | 6,6 | | | 26.90 | 7,0 |
| 2600 | 8.44 | 6,8 | | | | |
| 2800 | 8.11 | 7,0 | | | | |
| 3000 | 7.64 | 7,0 | | | | |

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