INTERNATIONAL STANDARD

**ISO 18755**

Second edition
2022-12

# Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of thermal diffusivity of monolithic ceramics by flash method

*Céramiques techniques — Détermination de la diffusivité thermique des céramiques monolithiques par la méthode flash*



Reference number
ISO 18755:2022(E)



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Published in Switzerland

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# Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 206, *Fine ceramics*.

This second edition cancels and replaces the first edition (ISO 18755:2005), which has been technically revised.

The main changes are as follows:

— a change of title and scope to enable the use of flash lamps to generate the energy pulse;

— the addition of three new informative annexes: one dealing with the determination of the intrinsic thermal diffusivity; the second with the determination of specific heat and thermal conductivity of the samples tested; and the third providing precision data for the method on the basis of an inter-laboratory study carried out by seven European laboratories in 2020-2021 in the framework of the project Hi-TRACE;

— an additional normative reference to provide clear instructions on the determination of the density of the materials to be analysed;

— relevant specifications added concerning the size and the density of the specimen;

— improvement of Annex F, with an updated list of potential reference material and incorporation of a validation method.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

# Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of thermal diffusivity of monolithic ceramics by flash method

## 1 Scope

This document specifies the test method for the determination of thermal diffusivity from room temperature to at least 1 700 K by the flash method for homogeneous monolithic ceramics with porosity less than 10 %.

Flash methods, like laser flash, are applicable to homogeneous isotropic materials with thermal diffusivity values ranging from 0,1 to 1 000 $mm^2 s^{-1}$ within the temperature range from approximately 100 K to 2 300 K.

The method described in Annex G describes how to estimate, on the basis of the thermal diffusivity test, the specific heat capacity and the thermal conductivity of homogeneous monolithic ceramics with porosity less than 10 %.

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 18754, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of density and apparent porosity*

## 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

**3.1**
**thermal diffusivity**
thermal conductivity divided by the product of specific heat capacity and density

**3.2**
**thermal conductivity**
density of heat flow rate divided by temperature gradient under steady state condition

**3.3**
**specific heat capacity**
heat capacity per unit mass

**3.4**
**pulse width**
$\tau_p$
full width at half maximum (FWHM), which is the time duration when the laser or light pulse intensity is larger than half of its maximum value on time basis

**3.5**
**centroid of laser pulse**
chronological centroid of laser light energy

**3.6**
**centroid of light pulse**
chronological centroid of light energy

**3.7**
**spatial energy distribution of pulse laser beam**
energy density of the laser beam or light flash incident at each point on the front face of the specimen

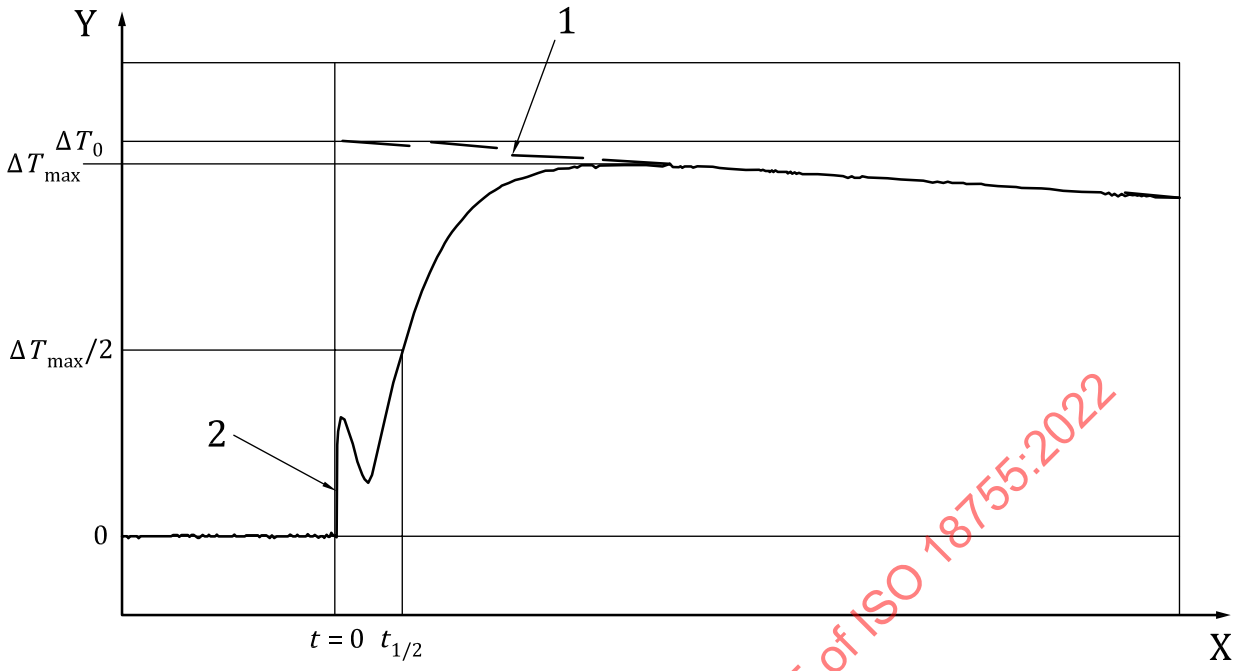
**3.8**
**transient temperature curve**
transient temperature change of the rear face of the specimen after the light pulse heating

**3.9**
**transient radiance curve**
transient change of the spectral radiance from the rear face of the specimen after the light pulse heating

Note 1 to entry: It should be noted that the observed transient curve is proportional to the change of the spectral radiance rather than the change of temperature when a radiation thermometer or a radiation detector is used to observe the transient temperature rise of the specimen after the light pulse heating.

**3.10**
**maximum temperature rise**
$\Delta T_{max}$
difference between the steady temperature before the pulse heating and the maximum temperature of the rear face of the specimen after the pulse heating

Note 1 to entry: See Figure 1.




**Key**

X time

Y temperature rise

1 exponential function $[\Delta T_0 \ exp(-t/\tau_c)]$

2 initial noise

**Figure 1 — Transient temperature curve of the rear face of the specimen after a light pulse heating onto the front face of the specimen**



**3.11**
**half rise-time**
$t_{1/2}$
time until $\Delta T_{max}/2$ is attained from the pulse heating

**3.12**
**characteristic time of heat loss**
$\tau_c$
time of heat loss determined when the cooling region is fitted with an exponential function, $[\Delta T_0 \ exp(-t/\tau_c)]$

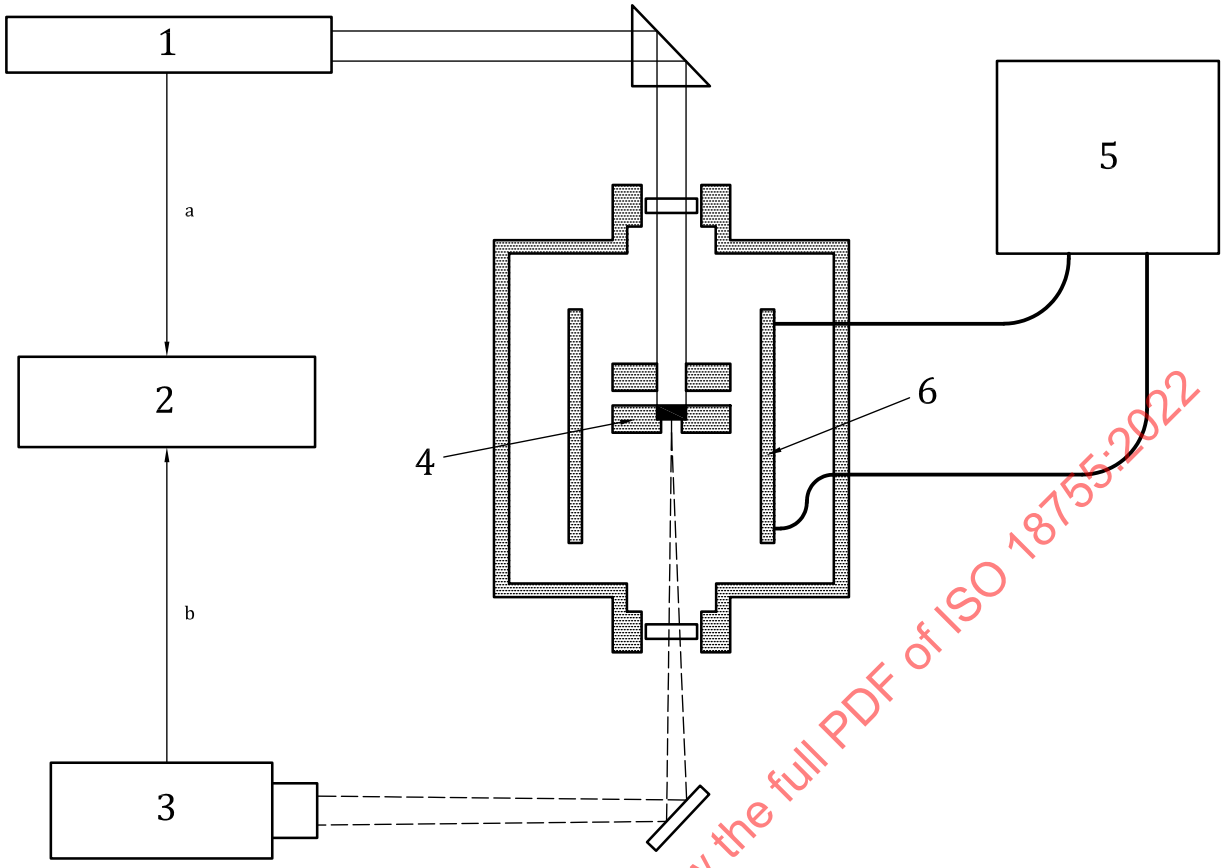
Note 1 to entry: See Figure 1.

**3.13**
**extrapolated temperature rise**
$\Delta T_0$
temperature rise determined when the cooling region is fitted with an exponential function, $[\Delta T_0 \ exp(-t/\tau_c)]$

## 4 Apparatus

### 4.1 General

The apparatus shall be designed for obtaining the thermal diffusivity from the transient temperature curve of the rear face of a specimen after the light pulse is irradiated onto the front face of the specimen. It shall consist of the principal components as shown in Figure 2.




**Key**

| | | | |
|---|---|---|---|
| 1 | pulsed laser | 4 | specimen holder |
| 2 | data analysis | 5 | power supply |
| 3 | detector | 6 | heater |

a Trigger signal.

b Transient temperature response.

**Figure 2 — Block diagram of laser flash apparatus for measuring thermal diffusivity**



## 4.2 Specimen holder

The specimen holder shall hold the specimen stable, with minimum thermal contact, and shall be designed to suppress stray lights from the laser beam/light flash being transmitted to the transient detector.

A diaphragm with aperture diameter slightly larger than the specimen diameter should be placed close to the front face of the specimen, and another diaphragm with aperture diameter smaller than the specimen diameter and larger than the target size of radiative detection should be placed close to the rear face of the specimen.

## 4.3 Flash source

The flash source shall be a pulse laser, a flash lamp or another device capable of generating a short duration pulse of substantial energy with pulse duration preferably shorter than 1,0 ms in full width at half maximum (FWHM). The specimen should be irradiated uniformly by the light pulse.

When a pulse laser is used for the light pulse, the direct beam profile is often irregular because of multi-mode oscillation. In this case, the beam should be converted to a uniform beam by using beam-homogenizing optics.

### 4.4 Thermometer for measuring steady-state temperature of the specimen

The steady-state temperature of the specimen before pulse heating should be measured by a thermocouple, or an equally or more reliable thermometer.

The thermocouple shall be positioned such that it does not interrupt the light pulse heating onto the front face of the specimen, or the radiation from the rear face of the specimen. If the specimen does not react with the thermocouple, a thin thermocouple should be contacted with the specimen to measure the specimen temperature with minimal uncertainty. If the thermocouple junction cannot be allowed to contact the specimen because of chemical reaction with the specimen, or because it interrupts the setting of the specimen, or because of the system design, the tip should be placed as close as practical to the specimen in the same plane.

### 4.5 Detector for measuring transient temperature rise of rear face of the specimen

The transient temperature rise curve on the rear face of the specimen shall be observed with a non-contact radiation thermometer or a radiation detector. The frequency response of the detector and its associated electronics should be faster than 10 kHz. The target diameter of the radiation detector should be smaller than 50 % of the diameter for disk specimens, or 50 % of the shortest side-length for square and rectangular specimens.

### 4.6 Environment for measurements

Measurements can be performed under open air, under an inert gas atmosphere or under vacuum at room temperature. For higher temperature measurements, an appropriate inert atmosphere or vacuum shall be used, when necessary, to protect furnace parts and specimen holders from oxidation and to protect the specimen and its coating from structure or phase changes and compatibility problems.

### 4.7 Temperature control unit

For higher temperature measurements, the specimen should be kept at a stable temperature by electric heaters before pulse heating. Drift and fluctuation of the temperature should be less than 0,01 K/s.

### 4.8 Data acquisition unit

The transient detector signal should be amplified and converted to the digital signal using a digital oscilloscope or an AD converter, which is input to a personal computer for computation of the thermal diffusivity. The frequency response of the amplifier and the AD conversion should be faster than 10 kHz. The resolution of the AD conversion should be larger than 10 bits, more than 1 000 data points should be sampled with the sampling time faster than 1 % of the half rise-time “t1/2”.

## 5 Specimen

### 5.1 Shape and dimension of specimens

The specimen shall be a flat plate of circular, square or rectangular shape. The specimen diameter or side shall be larger than 5 mm and up to typically 20 mm.

The specimen thickness shall be chosen to be as follows:

a) sufficiently thick that the $t_{1/2}$ value is larger than five times the pulse width.

b) the diameter-or-side-to-thickness ratio shall be equal to or higher than 5:2.

NOTE In most cases, experience shows that the diameter-or side-to-thickness ratio is in the order of magnitude of 4:1. However, some reference materials supplied from NMIJ (see Annex F) include specimens of 10 mm diameter and 4,0 mm thickness. They would be out of the ratio of 4:1. Therefore, in order to also include the reference materials with the above-mentioned sizes, the selected ratio is identified as 5:2.

c) The uniformity of the specimen thickness shall be smaller than 1,0 %.

### 5.2 Density of the specimen

The porosity of the specimen as determined by ISO 18754 shall be lower than 10 %.

The mass of the specimen shall be measured before and after measurement in order to detect possible mass changes, in particular for high-temperature measurements, due to reactions which can occur during the measurements, even if they ought to be avoided.

NOTE If the porosity is higher than 10 % other approaches can be applied, see References [43] to [47].

### 5.3 Coating on the specimen

If the specimen does not have a high absorption coefficient for the heating laser beam/light flash or a high emissivity for radiative temperature detection, the surfaces of the specimen shall be coated with a thin, opaque, preferably black layer. The coating shall be dense enough to prevent penetration of the laser beam/light flash or thermal radiation at the observed wavelength, and should be resistive against laser/light pulse heating at high temperatures. Coating thickness should be a minimum commensurate with excluding directly transmitted laser/light pulse.

Suitable coatings for many ceramic materials include evaporated, sputtered carbon or sprayed colloidal graphite. If the test specimen reacts with carbon at high temperatures, a metal coating, such as platinum, gold or nickel, can alternatively be used. The surface of the test specimen can, with advantage, be roughened to improve adhesion of the coating. The coating thickness dependence should be evaluated for the observed thermal diffusivity, if the contribution of coatings is not negligible.

### 5.4 Reference specimen

Reference specimens can be used to evaluate uncertainty of thermal diffusivity measurements by a flash apparatus. The uncertainty is obtained as the difference between the measured value and the reference value of thermal diffusivity of the reference specimen.

NOTE Several materials are used as reference (see Annex F).

Care should be taken in the use of these references to ensure that the half rise-time and the thermal diffusivity value are similar to those of the test materials.

## 6 Measurement procedure

### 6.1 Measurement of specimen thickness

Measure the thickness of the specimen to an accuracy of 0,5 % or better, using a micrometer in accordance with ISO 3611.

### 6.2 Surface treatment

Carry out the surface treatment in accordance with 5.3.

### 6.3 Determination of the flash time of the laser or light pulse and the chronological profile of the laser or light pulse

The chronological trace of the laser or light pulse versus the same trigger signal to initiate flash thermal diffusivity measurements shall be observed. If the FWHM of the laser/light pulse duration is larger than 1 % of the half rise-time, correction for the finite pulse time shall be made following one of the procedures stated in Annex B.

### 6.4 Temperature and atmosphere control

Insert the test specimen in the apparatus and position the thermocouples. The atmosphere should be such that the specimen is not subjected to any chemical change under the measured temperature range.

### 6.5 Stability of specimen temperature

The specimen temperature shall be controlled with drift smaller than 0,01 K/s.

### 6.6 Energy of pulse heating

Irradiate the specimen with the laser or light pulse at an intensity of as low energy as possible, commensurate with an acceptable noise level.

NOTE See Annex D regarding non-linearity of spectral radiance on temperature.

### 6.7 Measurement temperature

Record the measurement temperature as $T_0 + \Delta T_{max}$, where $T_0$ is the initial steady-state temperature and $\Delta T_{max}$ is the maximum temperature rise of the specimen recorded by the thermocouple in contact with the specimen or the calibrated radiation thermometer.

NOTE A thermocouple below 0,15 mm in diameter, which is directly contacted to the rear or side surface of a specimen mechanically or with a paste, is preferable to estimate $\Delta T_{max}$.

### 6.8 Record

The transient temperature curve should be recorded for a duration at least until 10 times the half-rise-time, in order to make reliable evaluation of measurements, including heat-loss correction and evaluation of non-uniform heating effect.

## 7 Data analysis

### 7.1 Calculation based on the half-rise-time method

The standard algorithm to calculate thermal diffusivity from the flash method is the half-rise-time method, in which the analytical formula is fitted to the transient temperature curve at *t,* the height of a half of maximum temperature rise of the transient temperature or radiance response curve above the baseline $\Delta T_{max} / 2$ over the half-rise-time.

If the measurement is valid when made under the above-mentioned ideal initial and boundary conditions, the thermal diffusivity, $\alpha$, is represented by Formula (1), based on the half-rise-time method:

$$\alpha = \frac{0{,}138\ 8 d^2}{t_{\frac{1}{2}}} \tag{1}$$

where

$d$ is the specimen thickness, in metres;

$t_{\frac{1}{2}}$ is the time delay when the temperature of the rear face reaches one-half of the maximum temperature rise, $\Delta T_{max}$, after the front face was heated by the laser pulse.

## 7.2 Criteria for applicability of the half-rise-time method

In order that the rise-time can be validly applied, the following initial and boundary conditions shall be satisfied:

— The duration of the laser/light pulse is short, compared with the characteristic time of heat diffusion (FWHM < 1 % of $t_{1/2}$).

— The front face of the specimen is uniformly heated by the laser/light pulse.

— The specimen is adiabatic during the period of measurement after the laser/light pulse heating.

— The specimen is uniform (in geometry) and is homogeneous.

— The specimen is opaque (non-transparent and non-translucent) to the laser/light pulse and to thermal radiation.

If these conditions are satisfied, the heat flow becomes one-dimensional and the temperature of the rear face of the specimen changes according to an analytical formula (see Annex A).

The thermal diffusivity value shall be determined by fitting this formula to the observed transient temperature curve. Theoretically, if the measurement is made under the above-mentioned ideal conditions, the calculated thermal diffusivity value should be independent of the position along the transient curves. Therefore, any point on the transient temperature curve can be analysed to yield the thermal diffusivity, $\alpha$. This is given by Formula (2).

$$\alpha = \frac{K_x d^2}{t_x} \tag{2}$$

where

$d$ is the specimen thickness, in metres;

$t_x$ is the time for the specimen rear face to reach a fraction of the maximum temperature rise, in seconds (see Table 1);

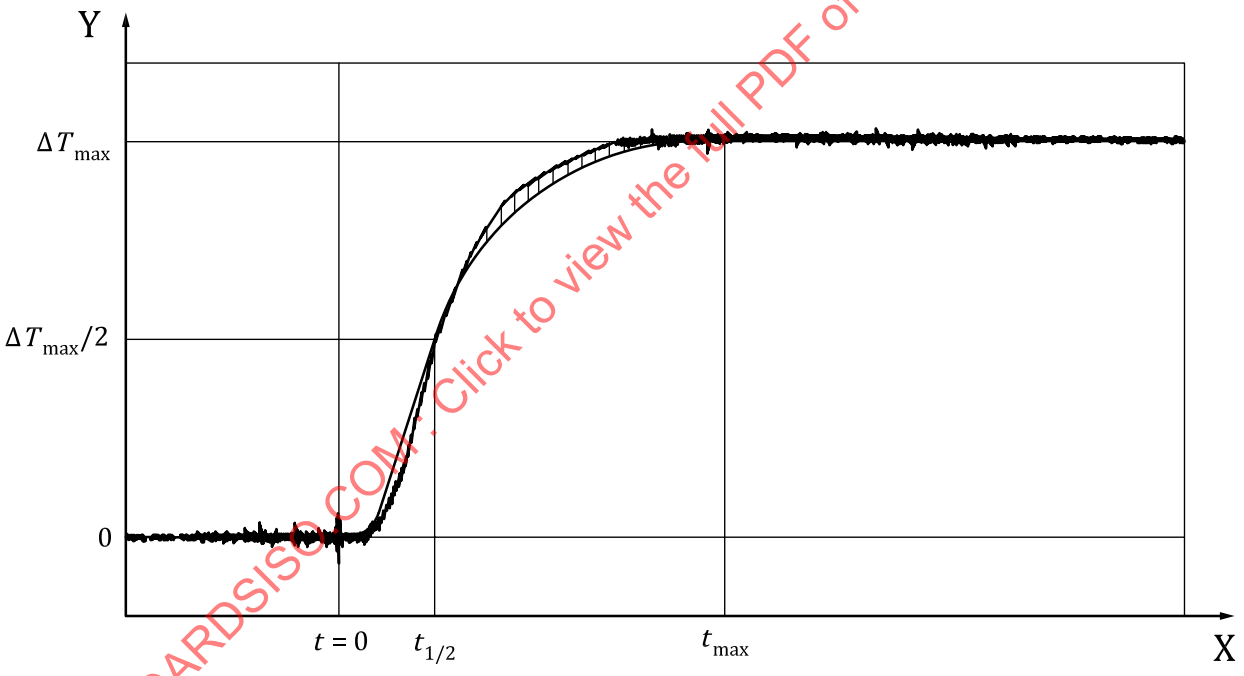
$x$ is the percentage of the maximum rise in temperature;

$K_x$ is a constant relating $\alpha$ to $d$ and $t_x$, in the case of ideal measurements.

Calculate the thermal diffusivity at fractional temperature rises other than $t_{1/2}$. If the values at $t_{0,3}$, $t_{0,5}$ and $t_{0,7}$ calculated using the relevant values of $K_x$ in Table 1 are all within ±2 %, then it can be assumed that the half-rise-time method is applicable without any correction. If the spread of thermal diffusivity values so calculated is greater than ±2 %, the possibility of non-ideal initial and/or boundary conditions, imperfect design and/or operation of the flash apparatus, or problems associated with the specimen, shall be considered.

**Table 1 — Values of constant $K_X$ for a range of transient times**

| $x$ % | $K_x$ | $t_x$ |
|---|---|---|
| 10 | 0,066 2 | $t_{0,1}$ |
| 20 | 0,084 3 | $t_{0,2}$ |
| 30 | 0,101 2 | $t_{0,3}$ |
| 40 | 0,119 0 | $t_{0,4}$ |
| 50 | 0,138 8 | $t_{1/2}$ |
| 60 | 0,162 2 | $t_{0,6}$ |
| 70 | 0,191 9 | $t_{0,7}$ |
| 80 | 0,233 2 | $t_{0,8}$ |
| 90 | 0,303 6 | $t_{0,9}$ |




**Key**

| | |
|---|---|
| X | time |
| Y | temperature rise |
| solid curve | transient temperature curve |
| broken curve | observed half rise-time |

**Figure 3 — Averaged deviation of the transient temperature curve from the Parker's formula having the observed half rise-time**

The applicability of the half-rise-time method can alternatively be checked through the averaged deviation of the transient temperature curve from the Parker's formula corresponding to the experimentally determined half rise-time as shown in Figure 3. The averaged deviation is calculated over the region from the half rise-point to the maximum point normalized by the maximum temperature rise $\Delta T_{max}$. If the averaged deviation is within ±1 % then it can be assumed that no corrections apply.

If the averaged deviation is greater than ±1 %, the possibility of non-ideal initial and/or boundary conditions, imperfect design and/or operation of the flash apparatus, or problems associated with the specimen, shall be considered as follows:

a) Imperfect design and/or operation of the flash apparatus:

   1) superposition of stray light or electrical noise on the transient temperature response curve;

   2) excessive drift of steady-state temperature;

   3) insufficient response time of radiation detector and/or amplifier;

   4) non-negligible heat exchange with the specimen holder;

   5) effect of non-linearity of spectral radiance.

b) Problems associated with the specimen:

   1) thermal resistance of coating;

   2) poor flatness of the specimen;

   3) large void or inhomogeneous distribution of pores.

c) Non-ideal initial and boundary conditions:

   1) non-uniform heating effects;

   2) finite pulse time effect;

   3) radiation heat loss.

First, check items a), b) and c) 1) and improve them to make the measurements closer to the ideal conditions.

NOTE During practical measurement by the flash method it is difficult always to satisfy the ideal initial and boundary conditions. Annex C and the Bibliography give examples of analyses for non-ideal conditions which can be applied as appropriate. Annex D gives information on other sources of error. If the position is still not acceptable, then corrections for items c) 1) and c) 2) are necessary via appropriate algorithms.

Examples of such analyses are given in Annex C. Details of all the procedures employed shall be given in the measurement report.

Annex F gives information on a method which allows evaluating the intrinsic thermal diffusivity regardless of the measurement conditions.

Finally, Annex H provides details concerning the precision data, reproducibility and repeatability of the thermal diffusivity measurements.



## 8 Measurement report

The following information should be recorded in the measurement report:

a) General information

   1) the name and address of the testing establishment;

   2) the date of measurements;

   3) a unique identification of the report;

   4) a reference to this document;

5) the name of the flash apparatus used.

b) Light pulse

1) the type of the pulse light source;

2) duration of the light pulse in full width at half maximum (optional);

3) energy of one light pulse (optional);

4) statement of spatial profile of the laser beam.

c) Specimen

1) a description of the material (material type, manufacturing code, batch number, date of receipt);

2) method of cutting, grinding and/or polishing specimens from supplied material;

3) shape of the specimen (disk, square plate or rectangular plate);

4) density or porosity of the specimen;

5) diameter or side length of the specimen;

6) thickness of the specimen.

d) Coating

1) use of coating (yes or no);

2) coated material;

3) coating procedure;

4) thickness of coating (optional).

e) Thermometry

1) thermometer used for steady-state temperature measurements;

2) thermometer or radiation detector used for measuring transient temperature or radiance rise of the specimen rear face after light pulse heating.

f) Data acquisition (optional)

1) response time of the transient temperature measurements;

2) response time of the transient temperature measurements.

g) Data analysis

1) the type of the analytical solution on which the data analysis is founded;

2) the data analysis algorithm (half-rise-time method, least-square-fit method, non-linear least-square method, equiareal method or logarithmic method).

h) Corrections

1) calculated values of heat-loss corrections, if any, giving full details if the methods are not given in Annexes B or C;

2) calculated values of non-uniform heating corrections, if any, giving full details if the methods are not given in Annexes B or C;

3) calculated values of finite pulse time correction, if any, giving full details if the methods are not given in Annex B;

4) calculated values of non-linearity of spectral radiance correction, if any, giving full details if the methods are not given in Annexes B or C;

5) calculated values of coating thermal resistance corrections, if any, giving full details.

i) Measured results

1) the measurement temperature(s), in kelvins or degrees Celsius;

2) the half rise-time, in seconds;

3) the calculated thermal diffusivity value(s), in $m^2/s$;

j) Other important information

1) discussion of errors and correction procedures;

2) comments about the measurement and measurement results.

# Annex A
(informative)

# Principle of flash thermal diffusivity measurements

## A.1 Ideal condition

The analytical solution for flash thermal diffusivity measurements is given by Parker et al.[1] under the following conditions.

a) The duration of the pulse is negligibly short compared to the characteristic time of heat diffusion.

b) The front face of the specimen is uniformly heated by a light pulse.

c) The specimen is adiabatic during the measurement after the light pulse heating.

d) The specimen is uniform (in geometry) and homogeneous.

e) The specimen is opaque (non-transparent and non-translucent) to the light pulse and to thermal radiation.

If these conditions are satisfied, the heat flow becomes one-dimensional and the temperature of the rear face of the specimen changes according to Formula (A.1):

$$T(t) = \Delta T\left[1 + 2\sum_{n=1}^{\infty}(-1)^n \; exp\left(-(\pi n)^2 \frac{t}{\tau_0}\right)\right] \tag{A.1}$$

where

$\Delta T = \frac{Q}{C}$

$Q$ is the total energy absorbed by the specimen;

$C$ is the heat capacity of the specimen;

$\tau_0 = \frac{d^2}{\alpha}$ is the characteristic time of heat diffusion across the specimen;

$\alpha$ is the thermal diffusivity of the specimen.

# Annex B
(normative)

# Correction for non-ideal initial and boundary conditions

## B.1 General

The calculation of thermal diffusivity using Formula (1) is modified if the pulse transit time is less than 100 times the duration of the heat pulse, or if heat is lost from the specimen. Examples of modifications applying to the calculation method are given in B.2 and B.3.

The mathematical derivation given in Annex A assumes that no heat is lost from the specimen during the time taken for the heat pulse to pass through it.[2-5] For good conductors at temperatures close to ambient, this is a reasonable approximation, but for poor conductors and for most samples at high temperatures, corrections for heat losses will almost certainly be applicable.

Provided that use of a suitable holder design has minimized heat lost from the specimen by conduction, and that the duration of the transient is short enough for heat lost by convection to be neglected (there are no convective losses if the measurement is performed in a vacuum), the main source of heat loss is by radiation from the specimen surfaces. The best way to analyze heat loss is to compare the entire experimental curve with one or more of the many theoretical models available. Examples of analytical methods are given in Annex C.

## B.2 Effect of finite pulse time

### B.2.1 General

Several analytical calculations have been reported in order to correct the effect of the finite duration time of the heating laser/light pulse energy.[6-8] If the pulse width is much shorter than the half rise-time of the transient temperature curve, the laser/light pulse heating can be approximated by Dirac's delta function located at the chronological centroid of laser/light pulse energy [9].

When $t_{1/2}$ is less than 100 times the heat pulse duration, the finite pulse time effect should be corrected. Two widely used correction methods are the centroid method and the triangular pulse method described as follows.

### B.2.2 Centroid method[9]

If $t_{1/2}$ is larger than three times the pulse width ($\tau_p$), the finite pulse time effect can be corrected by shifting the time origin of the data analysis to the chronological centroid of the laser/light pulse ($t_g$). Thus, $t_{1/2}$ should be replaced by $t_{1/2} - t_g$.

### B.2.3 Determination of chronological centroid of laser/light pulse

#### B.2.3.1 General

The chronological centroid of the laser beam/light flash ($t_g$) should be determined by one of the following procedures.

#### B.2.3.2 Real time method

Measure the waveform of the laser beam /light pulse by a detector of frequency response faster than 10 μs and calculate the centroid directly from the observed waveform.

#### B.2.3.3 Integration method

Prepare a metallic sheet thin enough that the heat diffusion time across it is shorter than 3 µs. Then, the chronological trace of the temperature rise of the metallic film is proportional to the integrated energy of the laser beam from the starting point of the laser beam /light pulse. The waveform of the laser beam /light pulse is derived as the derivative of the chronological trace of the temperature rise.

### B.2.4 Triangular pulse approximation[6]

The shape of the heat pulse from a neodymium-glass laser and a flash-lamp can be approximated by a triangular pulse of duration $\tau$ with a maximum occurring at $\beta$, where $\beta$ is a fraction between zero and one.

This is most easily achieved by using a fast-response photodiode (as used in several laser calorimeters) or by measuring the change in resistance of a thin (approximately 25 µm) tantalum foil strip when subjected to the heat pulse. The parameters $\tau$ and $\beta$ usually change with the heat-pulse power, and so should be determined at the power to be used.

Once $\tau$ and $\beta$ are known, the thermal diffusivity $\alpha$ is given by Formula (B.1):

$$\alpha = \frac{C_1 d^2}{C_2 t_{1/2} - \tau} \tag{B.1}$$

where

constants $C_1$ and $C_2$ are given in Table B.1 for values of $\beta$;

$d$ is the specimen thickness.

The finite pulse time correction given in Formula (B.1) should not be used for $t_{1/2}$ less than $10\tau$.

**Table B.1 — Finite pulse time correction constants**

| $\beta$ | $C_1$ | $C_2$ |
|---|---|---|
| 0,15 | 0,348 44 | 2,510 6 |
| 0,28 | 0,315 50 | 2,273 0 |
| 0,29 | 0,311 10 | 2,245 4 |
| 0,30 | 0,306 48 | 2,237 5 |

## B.3 Effect of radiative heat losses

### B.3.1 General

The thermal diffusivity shall be calculated from Formula (B.2), introducing correction factors if radiative heat losses cannot be neglected.

$$\alpha = 0{,}138\ 8\ k_{\mathrm{rhl}}\ d^2 / t_{1/2} \tag{B.2}$$

where

$\alpha$ is the thermal diffusivity ($m^2/s$);

$d$ is the thickness of the specimen at room temperature (m);

$k_{\mathrm{rhl}}$ is the correction factor relating to heat loss from the specimen.

The following boundary condition is introduced instead of the adiabatic boundary condition, in order to formulate an analytical solution under the radiative heat losses. The other assumptions are the same as the ideal conditions.

Heat losses from the specimen can be expressed by the Newtonian cooling law and the Biot number can be defined.

There are a few algorithms to calculate thermal diffusivity from the data obtained by the laser/light pulse methods which take the radiative heat loss into consideration.[10-17] Cape and Lehman[14] give a general solution for the laser flash thermal diffusivity measurements which takes the radiative heat losses into consideration. The contribution of the radiative heat loss is expressed by a non-dimensional parameter called the Biot number. Cape and Lehman present an approximate formula for their general solution. Josell et al. find that coefficients of higher order terms in Cape and Lehman's formula are not correct and present the corrected formula in Formula (B.3)[16]:

$$T(t) = \Delta T \sum_{n=0}^{\infty} A_n \exp\left(-X_n^2 \frac{t}{\tau_0}\right) \tag{B.3}$$

$$A_n = 2(-1)^n \cdot X_n^2 (X_n^2 + 2Y + Y^2)^{-1},$$

$$X_0 = (2Y)^{1/2}\left(1 - Y/12 + 11Y^2/1\ 440\right),$$

$$X_n = n\pi + \frac{2Y}{n\pi} - \frac{4Y^2}{(n\pi)^3} + \left\{\frac{16}{(n\pi)^5} - \frac{2}{3(n\pi)^3}\right\}Y^3 + \left\{-\frac{80}{(n\pi)^7} + \frac{16}{3(n\pi)^5}\right\}Y^4 \quad (n \geq 1),$$

where

$\Delta T = Q / C$;

$Q$ is the total energy absorbed by the specimen;

$C$ is the heat capacity of the specimen;

$\tau_0 = \frac{d^2}{\alpha}$ is the characteristic time of heat diffusion across the specimen;

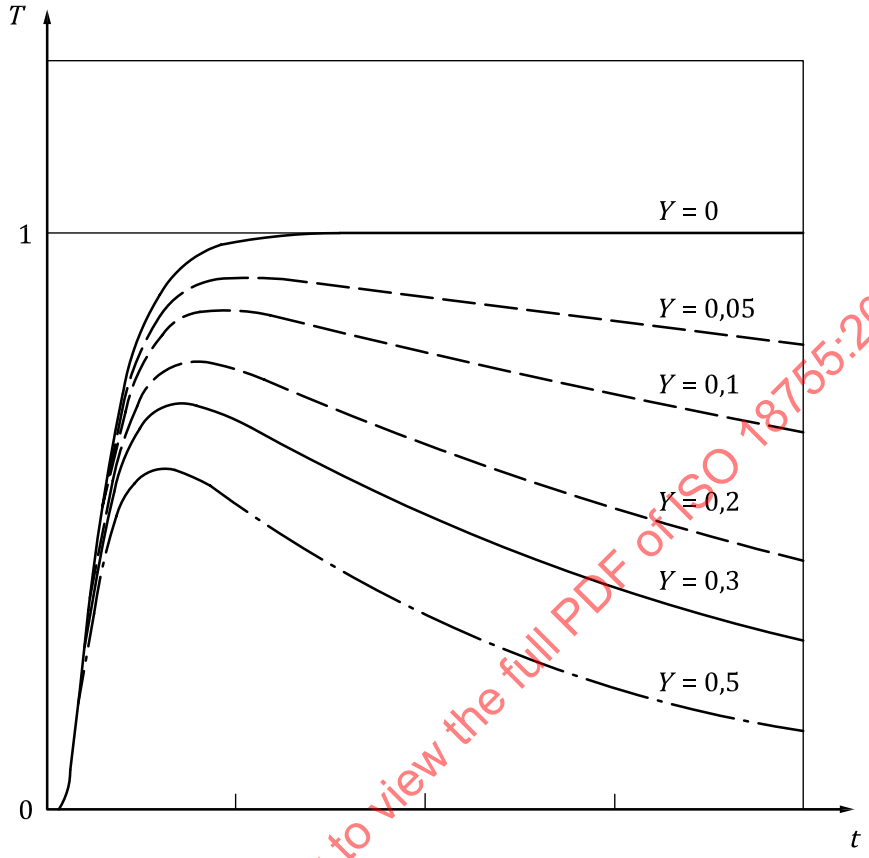
$\alpha$ is the thermal diffusivity of the specimen;

$Y = kd/\lambda$ is the Biot number.

Figure B.1 shows plots of Formula (B.3) for different values of parameter $Y$.

There are a few algorithms to calculate thermal diffusivity from the data obtained by the laser/light pulse methods which take the radiative heat loss into consideration. Among them, Cowan's method[13] and Clark and Taylor's method[15] are most commonly used. Their methods are in contrast with each other because the former uses the cooling part of the curve, whereas the latter uses the heating part of the curve to correct for the radiative heat loss. Takahashi et al.[17] proposed a correction method similar to Cowan's method. Their method calculates the correction factor for radiative heat loss from the ratio of $t_{1/2}$ to $\tau_c$, which is the characteristic time of heat loss.

In order to apply Cowan's method or Takahashi's method successfully, drift of the steady-state temperature and conduction heat loss shall be small. On the other hand, Clark and Taylor's method requires a high signal-to-noise ratio.




**Key**

$t$ time

$T$ normalized temperature rise

**Figure B.1 — Plots of Formula (B.3) for different values of the parameter $Y$**



### B.3.2 Cowen's method

According to Cowen's algorithm, the correction factor $k_{\mathrm{rhl}}$ is given by Formula (B.4):

$$k_{\mathrm{rhl}} = A + Bn + Cn^2 + Dn^3 + En^4 + Fn^5 + Gn^6 + Hn^7 \tag{B.4}$$

$$n = 2\Delta T_{10} / \Delta T_{max}$$

where

$\Delta T_{10}$ is the temperature rise at $10t_{1/2}$;

$A$ = 0,394 99;

$B$ = 1,203 01;

$C$ = −2,060 77;

$D$ = 2,042 96;

$E = -0{,}965\ 65$;

$F = 0{,}173\ 47$;

$G = 0$;

$H = 0$.

when $\Delta T_5$: temperature rise at $5t_{1/2}$

$A = -0{,}747\ 29$;

$B = 8{,}927\ 44$;

$C = -28{,}656\ 31$;

$D = 49{,}634\ 25$;

$E = -49{,}030\ 07$;

$F = 27{,}787\ 76$;

$G = -8{,}414\ 14$;

$H = 1{,}055\ 79$.

### B.3.3 Azumi and Takahashi's method [17]

According to this algorithm, the correction factor, $k_{\mathrm{rhl}}$, is given by Formula (B.5):

$$k_{rhl} = 1 - B_1\left(\sqrt{1 + A_1 \cdot c \cdot t_{1/2}} - 1\right) \tag{B.5}$$

where the following values should be substituted for $A_1$ and $B_1$:

When $L \leq 0{,}4$:

$A_1 = 96/(1 + L)$;

$B_1 = 0{,}084$.

When $0{,}4 < L \leq 1{,}0$:

$A_1 = 89\ [1 + 1{,}24\ (1 - L) - 2{,}70\ (1 - L)^2]$;

$B_1 = 0{,}08\ [1 - 1{,}13\ (1 - L) + 2{,}01\ (1 - L)^2]$.

When $L > 1{,}0$:

$A_1 = 89$;

$B_1 = 0{,}080$.

Where

$L = d/r$;

$r$ is the radius of the specimen, or radius of the circle having the same area as the specimen if the specimen is not a true circle.

### B.3.4 Clark and Taylor's method [15]

Heat-loss corrections based on the Clark and Taylor rise curve data also use ratio techniques. For the $t_{0,75}/t_{0,25}$ ratio, which is the time to reach 75 % of $\Delta T_{max}$ divided by the time to reach 25 % of $\Delta T_{max}$, the ideal value is 2 272. Determine this ratio from the experimental data. Then calculate the correction factor $k_{rhl}$ from Formula (B.6):

$$k_{rhl} = -0{,}346\ 146\ 7 + 0{,}361\ 578(t_{0,75}/t_{0,25}) - 0{,}065\ 205\ 43(t_{0,75}/t_{0,25})^2 \tag{B.6}$$

Corrections based on many other ratios can also be used.

### B.3.5 Least-square-fit methods

An observed transient temperature curve after pulse heating should be least-square fitted with the analytical solution of the thermal diffusion formula with variable parameters, including the thermal diffusivity value, under the initial and boundary conditions corresponding to the measurement condition.[18] The thermal diffusivity value is determined as one of the fixed parameters in the least-square-fitted analytical solution.

### B.3.6 Non-linear least-square methods[19]

Watt[11] gave the theoretical background with the wide applicability on the pulse heating methods, where the temperature response *T(x,r,t)* at time *t* in cylindrical coordinates (*x*,*r*) [see Formula (B.7)] is accompanied with two subsidiary Formulae (B.8) and (B.9).

$$T(x,r,t) = \Delta T_{o,max}\left\{\sum_{n=1}^{\infty} Y_n(x)\int_0^b f(x')Y_n(x')dx'\right\}$$

$$\left\{\sum_{j=1}^{\infty}\frac{2}{a^2}\frac{\mu_j^2 j_0\left(\mu_j\frac{r}{a}\right)}{\left(\mu_j^2+L_r{}^2\right)j_0^2(\mu_j)}\int_0^a r'g(r')J_0\left(\mu_j\frac{r'}{a}\right)dr'\right\}\cdot\int_{t_1}^{t_2}\exp\left\{-\alpha\left(\frac{\mu_j{}^2}{a^2}+\frac{\beta_n^2}{b^2}\right)(t-t')\right\}\Psi(t')dt' \tag{B.7}$$

$$Y_n(x) = \frac{\left\{2\left(\beta_n^2+L_2^2\right)^{1/2}\right\}\left\{\beta_n\cos(\frac{x\beta_n}{b})+L_1\sin(\frac{x\beta_n}{b})\right\}}{b^{1/2}\left\{\left(\beta_n^2+L_1^2\right)\left(\beta_n^2+L_2^2+L_2\right)+L_1\left(\beta_n^2+L_2^2\right)\right\}^{1/2}}$$

$$\tan\beta_n = \frac{\beta_n(L_1+L_2)}{\beta_n^2-L_1L_2} \tag{B.8}$$

$$\mu_j J_1(\mu_j) - L_r J_0(\mu_j) = 0 \tag{B.9}$$

where

the distribution of a heat pulse is expressed by $f(x)g(r)\Psi(t)$ $(0\le x\le b, 0\le r\le a, t_1\le t\le t_2)$;

$L_1$, $L_2$, and $L_r$ are Biot numbers of specimen (front, rear and side) surfaces;

*a* and *b* are the diameter and thickness of a specimen, respectively;

$\mu_j$ and $\beta_n$ are roots of Formulae (B.8) and (B.9), respectively;

$\Delta T_{0,max}$ is the maximum temperature rise of a specimen at $L = 0$ (adiabatic condition).

These relations show that thermal diffusivity, non-approximated Biot numbers, pulse width and non-uniformity of pulse can be, in principle, calculated under the assumption of axial symmetry from one transient temperature curve observed in the specified area on a specimen.

The actual data analysis procedures can be carried out on the basis of the Marquart (non-linear regression) method, as shown with the non-linear operational form of Formula (B.7), the algorithm of data analysis and some measurement results in Reference [19].

In order to avoid ambiguous estimations, the centroid of pulse $t_g$ is at first determined using a sufficiently thin metal foil, then non-uniformity of a pulse is estimated using suitable functions with one of specimens at room temperature, where heat losses are negligibly small. As the centroid position $t_g$ and non-uniformity of laser/light pulses are usually assumed to be constant during experiments, thermal diffusivity, Biot numbers and $\Delta T_{0,\max}$ (or the corresponding value in a transient radiance curve) can be precisely determined simultaneously under the predetermined parameters, including the observed temperature area (diameters). The procedures are also applicable to measure thermal diffusivity of thin films, such as those not covered by this regulation, because the laser heating and observed temperature area can be flexibly controlled and specified on a specimen so as to satisfy Formula (B.7), if the conditions are axial symmetry.

## B.4 Effect of direct radiative heat transport in the specimen

Combined conduction and radiative heat transfer occur through the specimen when it is transparent or translucent to the energy pulse. Radiation can be greatly reduced by applying an opaque coating; however, the integrity of such coating deteriorates at higher temperatures because of dissimilar thermal expansion of the specimen and the coating material. If the specimen is diathermic and optically thin, thermal diffusivity can be calculated with the model presented by Mehling et al[20] in Formula (B.10).

$$T(x,t)=2\Delta T_0\sum_{n=1}^{\infty} y_n exp\left(-\frac{\alpha y_n^2}{x^2}t\right)\frac{y_n\cos(y_n)+N_{\mathrm{Bi}}\sin(y_n)}{y_n^2+N_{\mathrm{Bi}}^2+2N_{\mathrm{Bi}}} \tag{B.10}$$

where

$\alpha$ is thermal diffusivity;

$N_{\mathrm{Bi}}$ is the Biot number;

$y_n$ is the positive root of $\tan(y_n)=\frac{2y_nN_{\mathrm{Bi}}}{y_n^2-N_{\mathrm{Bi}}^2}$; $y_1=0$.

Furthermore, multiple reflections of the heating pulse and transparency to the infrared wavelengths inside the specimen can be accounted for with the model introduced by Salazar et al.[21].

## B.5 Correction for thermal expansion

A thermal diffusivity calculated by the procedure in the main text is the apparent thermal diffusivity $\alpha_0(T)$ based on the specimen thickness at room temperature.

The correct thermal diffusivity based on the specimen thickness at the measurement temperature $\alpha(T)$ is derived from Formula (B.11).

$$\alpha(T)=\left(\frac{d_T}{d_0}\right)^2\cdot\alpha_0(T) \tag{B.11}$$

where

$d_0$ is the specimen thickness at room temperature;

$d_T$ is the specimen thickness at the measurement temperature $T$.

# Annex C
(informative)

# Data analysis algorithms to calculate thermal diffusivity from observed transient temperature curve under non-ideal initial and boundary conditions

## C.1 Logarithmic method

The thermal diffusivity is calculated from Formula (C.1) based on the logarithmic method[22-25].

$$\alpha = -d^2 / 4h \tag{C.1}$$

where

$h$ is the inclination of strength line obtained when $\ln\left(\sqrt{t}\,\Delta T\right)$ is plotted in respect to $1/t$ in the rising region ($0{,}3 < \Delta T/\Delta T_{max} < 0{,}6$) of temperature rise curve(s).

## C.2 Equiareal method

The thermal diffusivity value should be determined when the integrated areas from $t_1$ to $t_2$ are equal under the observed transient temperature curve and under the analytical solution of the thermal diffusion formula under the initial and boundary conditions corresponding to the measurement condition[5,28].

The amplitude of the analytical solution is normalized to the observed transient temperature curve at the maximum temperature if measurements are made under the ideal condition with no heat losses under uniform heating.

Under the general boundary condition with heat losses, the amplitude of the analytical solution is normalized by the asymptotic exponential function of the corresponding analytical solution.

As described in B.3, the equiareal method takes the effect of radiative heat losses from the beginning, since the observed temperature response curve is fitted by Josell's formula [Formula (B.2)], which is the solution under the heat-loss boundary condition[14,16].

The equiareal method calculates thermal diffusivity from the transient temperature curve obtained by the laser flash measurement. In contrast to the half-rise-time method, the entire set of the experimental data is fitted by the theoretical curve based on Cape and Lehman's analysis[14] corrected by Josell et al.[[16]], which considers the radiative-heat-loss effect exactly. Both thermal diffusivity and Biot number are simultaneously determined by this equiareal method as shown in Figure C.1.

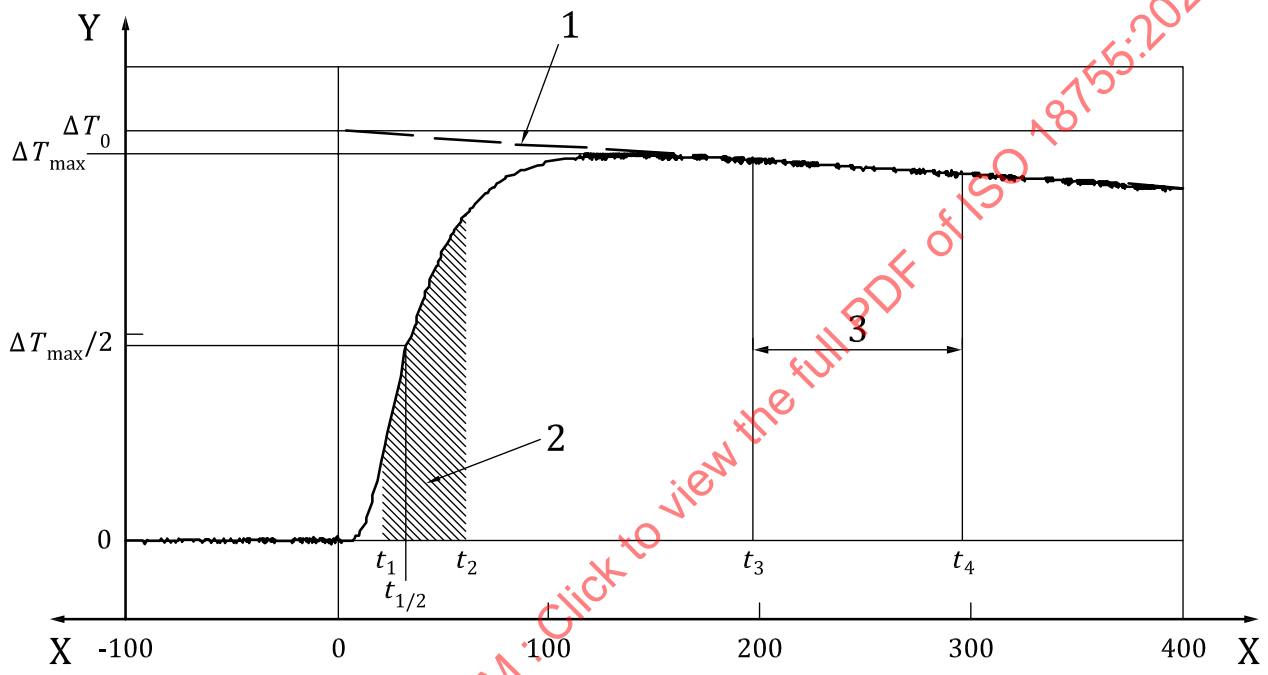
If $t > \tau_0$, the terms of order higher than second can be neglected compared with the first term in Formula (B.2). Thus, the specimen temperature converges to the steady-state temperature before the light pulse heating expressed by the single exponential Formula (C.2):

$$T(t) = \Delta T \cdot A_0 \; exp\left(-{X_0}^2 \frac{t}{\tau_0}\right) \tag{C.2}$$

Consequently, the temperature rise decreases exponentially with the characteristic cooling time of $\tau_c = \tau_0 / {X_0}^2$. The data analysis procedure to determine thermal diffusivity values by the equiareal

method is shown in Figure 2. First, $\tau_c$ is determined by a least-squares fit over the cooling region $t > \tau_0$ of the observed temperature response curve. Then, if either the thermal diffusivity or Biot number is determined, the other is simultaneously fixed. Thus, the thermal diffusivity is determined when the normalized area under Formula (B.2), as a function of $t$ with the parameter $\tau_0$ for the fixed region ($t_1 < t < t_2$), is equal to the area under the observed temperature response curve for the same time region[5].

One advantage of the equiareal method is that the quality of experimental data can be checked by observing the discrepancy between the experimental data and the theoretical curve. Data of poor quality, such as those affected by non-uniform heating, by drift of steady-state temperature or by a temperature detection system of slow response time, can be screened out immediately. Thus, only the experimental data of good quality are chosen and the thermal diffusivity values with smaller uncertainty are obtained.




**Key**

X time (ms)
Y normalised temperature rise
1 exponential function
2 equal area method
3 least-square fit

**Figure C.1 — Principle of the equiareal method to calculate thermal diffusivity from a transient temperature curve observed by the laser flash method**

# Annex D
(informative)

# Other error factors

## D.1 Effect of non-uniform heating

### D.1.1 General

There have been several investigations to analyse heat diffusion and temperature response after non-uniform pulse heating.[29-31] McKay and Schriempf[29] give a general analytical solution of temperature distribution within the specimen after the front face is heated by a laser beam of arbitrary energy distribution.

### D.1.2 Observation of spatial energy distribution of pulse laser beam

It is desirable that the spatial energy distribution of pulse laser is quantitatively measured with a beam profile measurement instrument, such as a CCD camera.[32] If those instruments are not available, the beam profile can be checked with a sheet of laser-beam foot print paper.

When the temperature profile is axially symmetric, axial energy distribution of the laser beam can be measured using a laser power meter while changing the aperture size of the field stop before the power meter. The diameter of the aperture size should be typically from 2 mm to the specimen diameter (10 mm standard) by steps of 2 mm.

The energy transmitting through the aperture of diameter $\phi$ is the integration of the energy distribution $q(z)$ of the laser beam as shown in Formulae (D.1) and (D.2):

$$Q(\phi)=\int_0^{\phi} q(z)\cdot 2\pi z dz = q_0\int_0^{\phi}\left[1+c_1 J_0\left(\mu_1\frac{z}{r}\right)\right]\cdot 2\pi z dz = q_0\left(\pi\phi^2 + c_1\int_0^{\phi} J_0\left(\mu_1\frac{z}{r}\right)\cdot 2\pi z dz\right) \tag{D.1}$$

$$c_1 = \frac{Q(\phi)\Big/ q_0 - \pi\phi^2}{\int_0^{\phi} J_0\left(\mu_1\frac{z}{r}\right)\cdot 2\pi z dz} \tag{D.2}$$

where

$\mu_1$ is the first positive root of $J_1(x)=0$

$J_1(x)$ is the first-order Bessel function

When the aperture diameter is equal to the specimen diameter the total energy of the pule incident on the specimen is provided by Formula D.3,

$$Q(r)=\pi r^2 q_0 = Q \tag{D.3}$$

where

$Q$ is the total energy of the pulse incident on the specimen.

The temperature distribution along the rear face of the specimen is provided by the function *T(z,t)*, where $0 < z < r$ and $t > 0$. Then *T(z, t)* is expressed by Formula D.4

$$T(z,t)=\Delta T\cdot\left[1+\sum_{j=1}^{\infty}c_i J_0(\mu_j\frac{z}{r})\cdot\exp\left(-\left(\frac{\pi\mu_j}{\rho}\right)^2\cdot\frac{t}{\tau_0}\right)\right]\cdot\left[1+2\sum_{n=1}^{\infty}(-1)^n\exp\left(-(\pi n)^2\frac{t}{\tau_0}\right)\right] \quad \text{(D.4)}$$

where

$\Delta T = Q / C$

$Q$ is the total energy absorbed by the specimen;

$C$ is the heat capacity of the specimen;

$\rho=\frac{\pi}{(2p)}$;

$p=\frac{d}{(2r)}$ is the thickness to diameter ratio of the specimen;

$\tau_0=\frac{d^2}{\alpha}$ is the characteristic time of heat diffusion across the specimen;

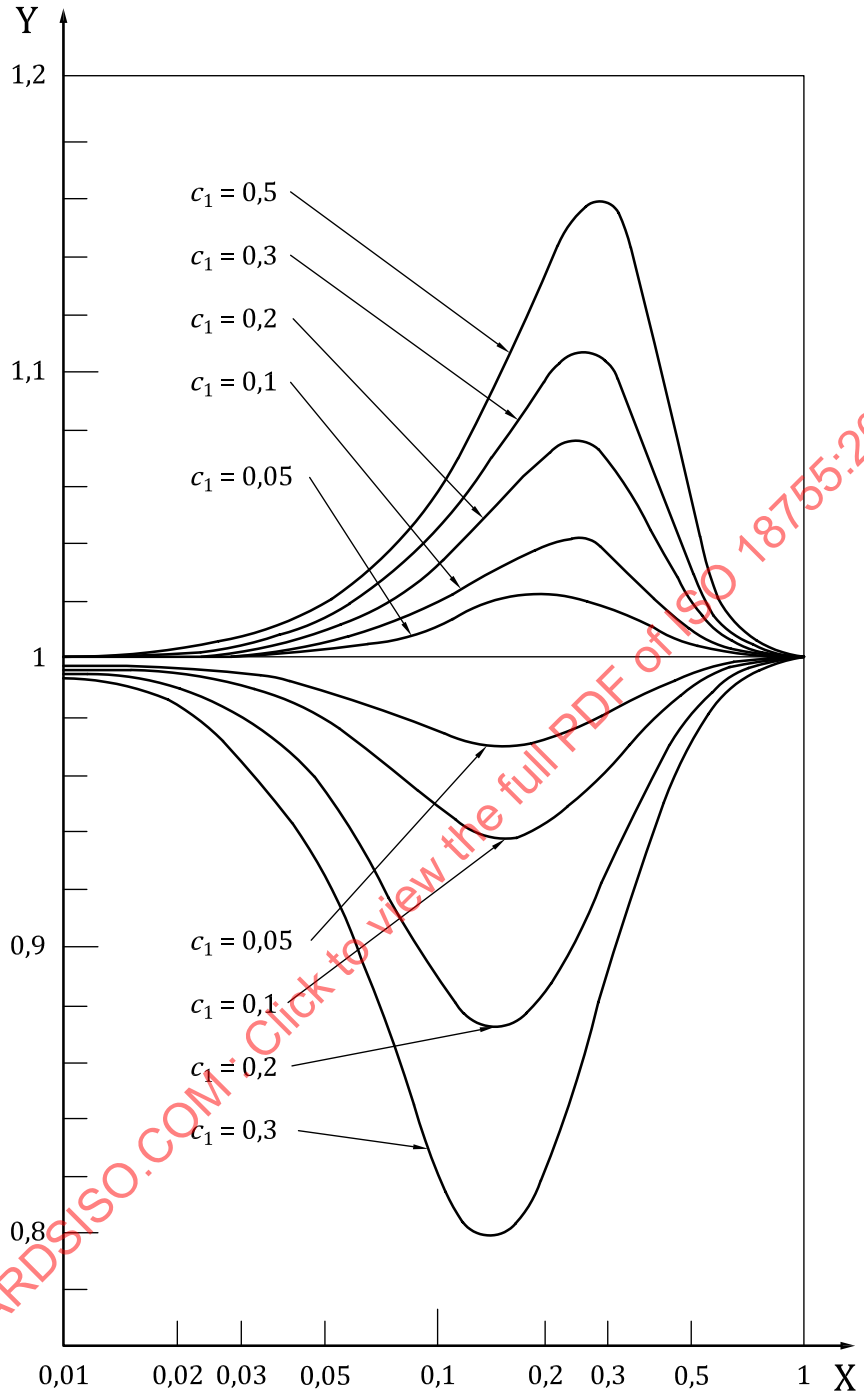
α is the thermal diffusivity of the specimen.

The non-uniform heating effect is expressed by the first parenthesis and the second parenthesis corresponds to the normal solution under the uniform heating.

### D.1.3 Evaluation of non-uniform heating effect

The temperature response of any combination of $p$ and $c_1$ can be calculated from Formula (D.4), and the apparent thermal diffusivity values, $\alpha$, can be calculated by the half-rise-time method. Figure D.1 shows the calculated apparent thermal diffusivity values, when temperature is measured at the centre of the specimen as a function of thickness to diameter ratio $p$ with a parameter $c_1$.

When the coefficient $c_1$ is positive, the beam has a hot centre and when the coefficient $c_1$ is negative, the beam has a cold centre. All curves have a maximum ($c_1 > 0$) or minimum ($c_1 < 0$) between $p$ = 0,15 and $p$ = 0,3, where the non-uniform heating effect is largest under irradiation of the laser beam of the same profile.






**Key**

X ratio of thickness to diameter (in log scale), $p$

Y apparent thermal diffusivity (normalised)

**Figure D.1 — Calculated apparent thermal diffusivity when temperature is measured at the centre of the specimen as a function of "$p$" with a parameter "$c_1$"**

## D.2 Measurements of apparent thermal diffusivity values dependent on light pulse energy

The apparent thermal diffusivity derived from the observed transient radiance curve changes dependent on the light pulse energy, because of non-linearity of Planck's formula and temperature

dependence of the thermal diffusivity of the specimen.[33-37] Generally, the apparent thermal diffusivity changes as a function of temperature most sensitively at the lowest measurement temperature. Thus, it is recommended that laser/light flash measurements are made under different levels of the laser beam/light flash energy.

## D.3 Measurements of apparent thermal diffusivity values dependent on specimen thickness

In order to characterize the equipment, such as the non-uniformity of pulses, measure the transient temperature rise curves of several standard specimens different in thickness at room temperature, in accordance with 5.4[5,38].

## D.4 Uncertainty associated with radiative heat losses

If the radiative-heat-loss correction is not made, the apparent thermal diffusivity value calculated from the observed temperature response curve is larger than the correct value by the ratio $1/k_{rhl}$ according to Cowan's method, Takahashi's method, and Clark and Taylor's method.

If the thermal diffusivity is calculated based on least-square-fit methods, non-linear least-square methods or the equiareal method, the correction factor $k_{rhl}$ for the apparent thermal diffusivity value calculated from the observed temperature response curve is given by the data analysis program, simultaneously with the corrected thermal diffusivity value.

## D.5 Steady temperature of the specimen

### D.5.1 Uncertainty of thermometers

Thermocouples are commonly used in flash systems operating over the temperature range of this document (from room temperature to 1 700 K). Platinum or platinum-rhodium thermocouples are most commonly used and the uncertainty associated with these types of thermocouples is ±1,5 K or ±0,25 % according to the requirements by ISO/IEC 17025, unless they are degraded or contaminated. After a series of measurements up to high temperatures, the thermocouple should be recalibrated in order to check if its thermoelectrical response is unchanged after exposure to a high-temperature environment.

### D.5.2 Temperature difference between the specimen and the sensing part of the thermometer

If the thermal contact between the specimen and the junction of the thermocouple is not enough, there is a temperature difference between them. In this case, it is necessary to investigate the deviation of the specimen temperature from the thermocouple junction temperature systematically.

One of the recommended procedures to investigate the temperature deviation of the specimen is to prepare another thermocouple and to paste its junction to the specimen with good thermal contact. Record the indication of both the regularly used thermocouple and the temporarily pasted thermocouple, from room temperature to the highest temperature of the measurements. Then, the reproducibility of the deviation can be checked and the conversion table from the regularly used thermocouple temperature to the specimen temperature can be obtained.

### D.5.3 Effective temperature of the specimen after light pulse heating

Since thermal diffusivities of materials change dependent on temperature, there is a fundamental problem concerning how to assign the effective temperature to the transient measurement with variation of temperature distribution over the specimen after the laser/light pulse heating. According to numerical simulations, the measured thermal diffusivity value should be assigned to the effective temperature calculated as $T_0 + \Delta T_{max}$, where $T_0$ is the steady temperature of the specimen immediately before the pulse heating[39].

In order to know the value of $\Delta T_{max}$, the temperature response at the rear face of the specimen shall be measured with a radiation thermometer calibrated with the temperature scale. If a radiation detector is used for relative measurements of the temperature response, $\Delta T_{max}$ shall be estimated from the indication of other thermometers, such as a thermocouple contact with the specimen.

### D.5.4 Uncertainty of effective temperature of the specimen

Since it is difficult to evaluate the absolute value of $\Delta T_{max}$, the major factors contributing to uncertainty of effective temperature of the specimen are uncertainty of the steady-state temperature of the specimen, $T_0$, and uncertainty of the maximum temperature rise, $\Delta T_{max}$.

### D.5.5 Extrapolation of apparent thermal diffusivity value dependent on the laser/light pulse energy

As described in the previous subclause, the apparent thermal diffusivity derived from the observed transient radiance curve changes dependent on the light pulse energy, because of the non-linearity of Planck's formula and temperature dependence of thermal diffusivity of the specimen. In principle, when the light pulse energy is infinitesimal, the apparent thermal diffusivity should be equal to the intrinsic thermal diffusivity at the steady-state temperature before the pulse heating. The thermal diffusivity corresponding to the infinitesimal laser/light pulse energy can be extrapolated from the series of apparent thermal diffusivity values measured when changing the laser/light pulse energy[5,38].

## D.6 Transient temperature of the specimen

### D.6.1 Non-linearity of Planck's formula

The spectra radiance, at wavelength $\Lambda$ from a blackbody of temperature $T$, is expressed in Formula D.5 by Planck's formula as follows:

$$L(\Lambda,T)=\frac{2c_1}{\Lambda^5}\frac{1}{exp\left(-\frac{c_2}{\Lambda T}\right)-1} \tag{D.5}$$

where

$c_1$ = 5,954 8 × $10^{-17}$ $Wm^2$ is the first radiation constant;

$c_2$ = 0,014 3 88 mK is the second radiation constant.

It should be noted that this formula is highly non-linear, and linear approximation is not satisfactory even over the narrow range as small as 10 K. For example, the spectral radiance change from 25 °C to 30 °C is 18 %, larger than that from 20 °C to 25 °C[36].

### D.6.2 Calibration of radiation thermometer

In order to observe the transient "temperature" change of the specimen rear face after the impulse heating instead of transient "spectral radiance", the radiation thermometer should be calibrated using black body cavities in the temperature range from 0 °C to 200 °C, where non-linear dependence of the output temperature shall be corrected for accurate thermal diffusivity measurements[36,37].

### D.6.3 Uncertainty associated with individual measurements

Even if a radiation thermometer is calibrated to the temperature standard with small uncertainty, a number of conditions shall be satisfied as follows, dependent on a particular environment in order to measure the absolute value of temperature response after the pulse heating.

The radiation thermometer shall observe only radiation from the central part of the specimen rear face free from stray lights.

Emissivity of the observed face of the specimen shall be high and the value of emissivity shall be known.

## D.7 Time response of transient temperature measurements

### D.7.1 Observation

#### D.7.1.1 Impulse response

Overall time responsivity of transient temperature measurements, considering the response of the radiation thermometer and the electronics can be evaluated by the temperature response curve at the rear face of a metal foil thinner than 100 µm after its front face is heated by a light pulse shorter than 10 µs. The observed temperature response curve can be considered as the impulse response function $R(t)$ of the total unit for transient temperature measurements.

#### D.7.1.2 Sinusoidal response

Overall time responsivity of transient temperature as a function of frequency can be measured using an infrared light emitting diode (IR-LED) operated in current modulation mode in the frequency region from 1 Hz to 100 kHz.[40] The amplitude and the phase of the signal from a radiation thermometer should be constant at least over the frequency from 1 Hz to 10 kHz[40].

## D.8 Uncertainty associated with coating

It is not easy to evaluate uncertainty associated with coating, because it is very difficult to measure a reliable value of effective thickness and thermophysical properties of the coating layer.

The uncertainty can be reduced if a coating layer is thin enough for the heat diffusion time across it to be negligible compared with that across the specimen although the coating layer is dense, and durable enough to prevent transmission of light pulses.

# Annex E
(informative)

# Procedure to determine intrinsic thermal diffusivity

Thermal diffusivity is one of the thermophysical properties and it depends on temperature. Thermal diffusivity is determined from a heat diffusion time and specimen thickness and it is an intrinsic value of the material.

Thermal diffusivity of fine ceramics has strong temperature dependence compared with metals. In the flash method, the specimen is heated by pulsed light and the temperature of the specimen increases. When the intrinsic thermal diffusivity independent of temperature-rise during measurement is assessed, the following procedure[41] is effective:

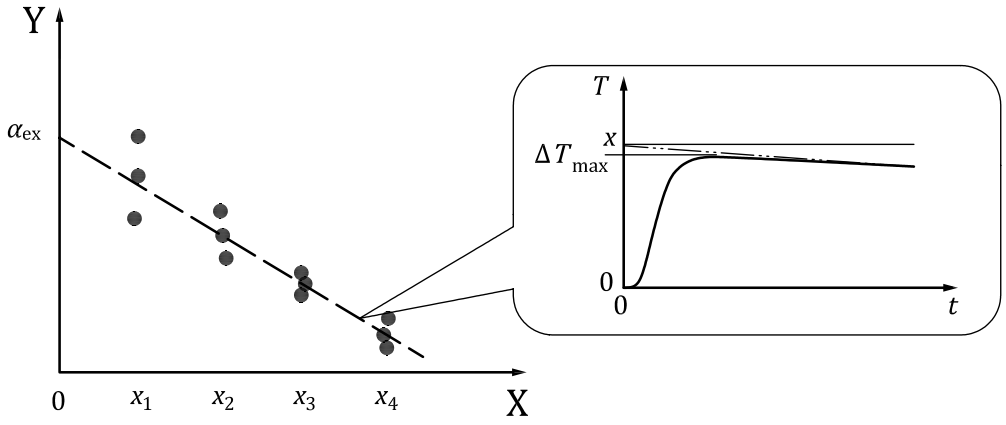
a) Specimens with different thickness should be prepared from the same batch of the material.

b) For a specimen, thermal diffusivities are measured by changing pulsed heating power at the uniform temperature. Here, pulsed heating power should be changed more than four levels. Measurements should be repeated more than twice for each pulsed heating level.

c) Thermal diffusivity value and temperature rise are obtained from each transient temperature rise curve. They are defined as apparent thermal diffusivities because it includes thermal diffusivities related to temperature dependence.

d) Apparent thermal diffusivities are plotted as a function of temperature rise. The extrapolating value of this plot is determined by the linear regression.

The extrapolating value ($\alpha_{ex}$) of apparent thermal diffusivity as a function of temperature rise is defined as intrinsic thermal diffusivity. It means thermal diffusivity without temperature rise. If the relation between apparent thermal diffusivity and temperature rise is known as in Figure E.1, the other function can be used to estimate the extrapolating value instead of a linear function.




**Key**

| | |
|---|---|
| X | temperature rise, $x_i$ |
| Y | apparent thermal diffusivity, $\alpha$ |
| $t$ | time |
| $T$ | temperature rise |
| $x$ | extrapolated temperature rise (see 3.12) |
| $\Delta T_{max}$ | maximum temperature rise (see 3.9) |

NOTE Insert illustrates a transient temperature curve.

**Figure E.1 — Example of apparent thermal diffusivity plot as a function of temperature rise**

The extrapolating value is calculated as follows:

The linear regression is carried out for thermal diffusivity obtained from measurements at $k$ levels of different pulsed heating powers with $r$ times repeats. When apparent thermal diffusivity $\alpha(x)$ and temperature rise $x$ are obtained for each transient temperature rise curve, the linear function is shown in Formula E.1 below.

$$\alpha(x)=\bar{\alpha}+b(x-\bar{x}) \tag{E.1}$$

where

$\bar{x}$ is the average of temperature rise $x$;

$\bar{\alpha}=\frac{1}{kr}\sum\alpha(x)$ is the average of apparent thermal diffusivity $\alpha(x)$;

$b=\frac{\sum_i\sum_j(x_i-\bar{\alpha}\bar{x})(\alpha_{ij}-\bar{\alpha})}{r\sum_i(x_i-\bar{x})}$ is slope of the linear function with $i$ =1, 2, ..., $k$ and $j$ = 1, 2, ..., $r$.

Intrinsic thermal diffusivity as an extrapolating value $\alpha_{ex}$ is provided by Formula E.2

$$\alpha_{ex} = \alpha(0) = \bar{\alpha} - b\bar{x} \tag{E.2}$$

The sum of squares of residual errors between the value of linear function and measured value is provided by Formula E.3

$$S_\alpha = \sum_{ij} \left[\alpha_{ij} - \alpha(x)\right]^2 \tag{E.3}$$

The experimental standard deviation $V\alpha$ as a square root of $S\alpha$ is provided by Formula E.4

$$V_\alpha = \sqrt{\frac{S_\alpha}{kr-2}} \tag{E.4}$$

The relevant uncertainty is provided by Formula E.5:

$$u(\alpha_{ex})^2 = \left[\frac{1}{kr} + \frac{\bar{x}^2}{r\sum_i (x_i - \bar{x})^2}\right] {V_\alpha}^2 \tag{E.5}$$

# Annex F
(informative)

# Reference data and reference materials of thermal diffusivity

## F.1 Reference data

Recommended values for thermophysical properties of some key solids have been recently updated by CODATA Task Group on Thermophysical Properties. The complete set of thermophysical property values to calculate thermal diffusivity are thermal conductivity, specific heat capacity and thermal expansion, and are only given for copper. The thermal diffusivity values calculated from Reference [42] and the density at 300 K are listed in Table F.1 for different residual resistivity ratios (RRR) as a function of temperature.

**Table F.1 — Reference values of thermal diffusivity of copper**

| Temperature K | Thermal diffusivity $10^{-4}$ m$^2$ s$^{-1}$ | | | | |
|---|---|---|---|---|---|
| | RRR=30 | RRRR=100 | RRR=300 | RRR=1 000 | RRR=3 000 |
| 300 | 1,164 | 1,197 | 1,206 | 1,209 | 1,212 |
| 400 | 1,135 | 1,159 | 1,165 | 1,168 | 1,168 |
| 500 | 1,111 | 1,129 | 1,135 | 1,138 | 1,138 |
| 600 | 1,091 | 1,106 | 1,112 | 1,112 | 1,112 |
| 700 | 1,071 | 1,083 | 1,089 | 1,089 | 1,089 |
| 800 | 1,054 | 1,063 | 1,069 | 1,069 | 1,069 |
| 900 | 1,036 | 1,045 | 1,048 | 1,048 | 1,048 |
| 1 000 | 1,017 | 1,023 | 1,026 | 1,026 | 1,029 |
| 1 100 | 0,994 | 1,002 | 1,002 | 1,005 | 1,005 |
| 1 200 | 0,968 | 0,974 | 0,977 | 0,977 | 0,977 |

## F.2 Reference materials

There are recognized and non-recognized certified reference materials supplied by national standard bodies. The reference materials recognized in 2021 are the following:

a) isotropic graphite (NMIJ CRM 5804);

b) $Al_2O_3$-TiC ceramics (NMIJ CRM 5807);

c) quartz glass (NMIJ CRM 5809);

d) glass ceramics (BCR-724);

e) alumina (JFCC TD-AL).

a) to c) are supplied by the National Metrology Institute of Japan (NMIJ). Their thermal diffusivities were determined by laser flash method traceable to the SI unit. The temperature ranges are 300 K to 1 500 K, 300 K to 1 000 K and 300 K to 800 K, respectively. They consist of three or four specimens with different thickness to check the apparatus that can measure thermal diffusivity regardless of thickness. d) is supplied by the European Commission's Joint Research Centre (JRC). The certified values were determined by flash method and guarded hot plate method. The material can be used from 298 K

to1 025 K. e) is supplied by the Japan Fine Ceramics Center (JFCC). It was experimentally confirmed that a grade of polycrystalline alumina is homogeneous and stable enough to be used as a reference material. [38] Two specimens with different thickness (2 mm and 3 mm) were measured in the temperature range from room temperature to 1 000 K and the reference formula of thermal diffusivity was determined.

Thermal conductivity reference material Electrolytic Iron (NIST SRM 8420) is supplied by the National Institute of Standard and Technology (NIST). Thermal diffusivity can be calculated on the basis of thermal conductivity, specific heat capacity and density.

Isotropic graphite (NMIJ RM 1401) has thermal conductivity as certified value, thermal diffusivity, specific heat capacity as reference values and density as information.

Other materials exist on the market and they are proposed by apparatus manufacturers.

## F.3 Validation methods of the apparatus by using reference materials

It is effective to use reference materials in order to verify the applicable region of apparatus and specimen thickness. Thermal diffusivity is a thermophysical property which is independent of specimen shape and thickness. The time response of apparatus can be checked by changing specimen thickness. For this purpose:

1) prepare different thickness specimens of the same batch of the material which has thermal diffusivity $\alpha_0$ with uncertainty $U$ with the coverage factor $k$=2. The reference materials are used as the material for the validation. If there is no reference material in the target thermal diffusivity range of validation, pure metals or ceramics which have reference thermal diffusivity values or well-known thermal diffusivity values reported in literatures and databooks are used. In order to check the lower limit of specimen thickness, different thickness between 0,1 mm to 2,0 mm specimens of high thermal diffusivity materials are useful.

2) Thermal diffusivities $\alpha_m$ of specimens of 1) are measured. The variation consisting in the repeatability of measurements is calculated as the standard deviation $s_{re}$ when a specimen is measured repeatedly. If $\alpha_m$ is obtained according to Annex E, $s_{re}$ is defined as $u(\alpha_{ex})$ in Formula (E.5).

3) Verify that measured thermal diffusivity $\alpha_m$ is within the range of $\alpha_0 \pm [(U/2)^2 + s_{re}{}^2]^{1/2}$. Here, the range where $\alpha_m \leqq \alpha_0 \pm [(U/2)^2 + s_{re}{}^2]^{1/2}$ is satisfied is defined as the applicable region of apparatus. If a systematic difference larger than $\pm[(U/2)^2 + s_{re}{}^2]^{1/2}$ from $\alpha_0$ occurs, some corrections are needed. However, when $[(U/2)^2 + s_{re}{}^2]^{1/2}$ is larger than $\alpha_0 \cdot 0{,}3$, corrections are not possible and the ranges have problems of reliability.

4) Verify the range of heat diffusion time or half time and the range of thickness where $\alpha_m \leqq \alpha_0 \pm [(U/2)^2 + s_{re}{}^2]^{1/2}$ is satisfied. The upper limit of time response of the apparatus and the lower limit of specimen thickness are known from the result.

# Annex G
(informative)

# Evaluation of specific heat capacity and thermal conductivity

## G.1 Evaluation of thermal conductivity

After directly determining thermal diffusivity ($\alpha$), it is possible to evaluate thermal conductivity ($\lambda$, lambda) on the basis of the specific heat and density of the tested samples, according to Formula (G.1).

$$\lambda(T)=\alpha(T)\cdot\rho(T)\cdot c_p(T) \tag{G.1}$$

where

$\lambda$ is the thermal conductivity;

$\alpha$ is the thermal diffusivity;

$\rho$ is the density;

$c_p$ is the specific heat capacity.

This formula applies only to materials that are homogeneous and isotropic.

a) The thermal diffusivity of the specimen shall be determined directly according to Clauses 4, 5, 6 and 7.

b) The room temperature density shall be measured according to ISO 18754. The density should be evaluated at each temperature, starting from the room temperature density, by using thermal expansion data, when available.

c) Specific heat can be determined on the basis of three different approaches:

   1) Differential scanning calorimetry (DSC): this is the primary method for determining the specific heat of the tested materials after calibration of the apparatus. The resulting data will then be employed according to Formula (G.1).

   2) Literature data: the data can be employed provided that samples have similar physical and geometrical characteristics, such as density and microstructure, and are tested under similar experimental conditions, i.e. temperature range, heating rate, test environment and coating of the specimens. The source of data shall be known.

   3) Flash method: specific heat capacity can be obtained by a comparative method according to G.2.

## G.2 Determination of specific heat capacity by flash method