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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

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Foreword

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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).

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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
τ_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

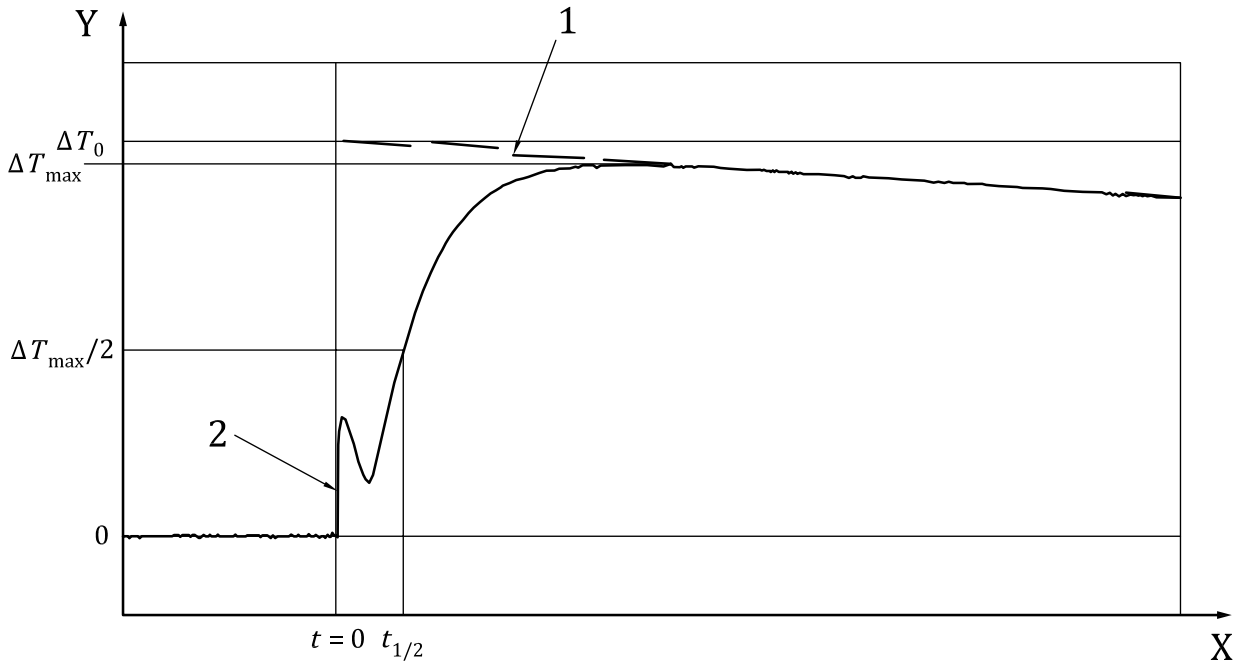
3.8
transient temperature curve
transient temperature change of the rear face of the specimen after the light pulse heating

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

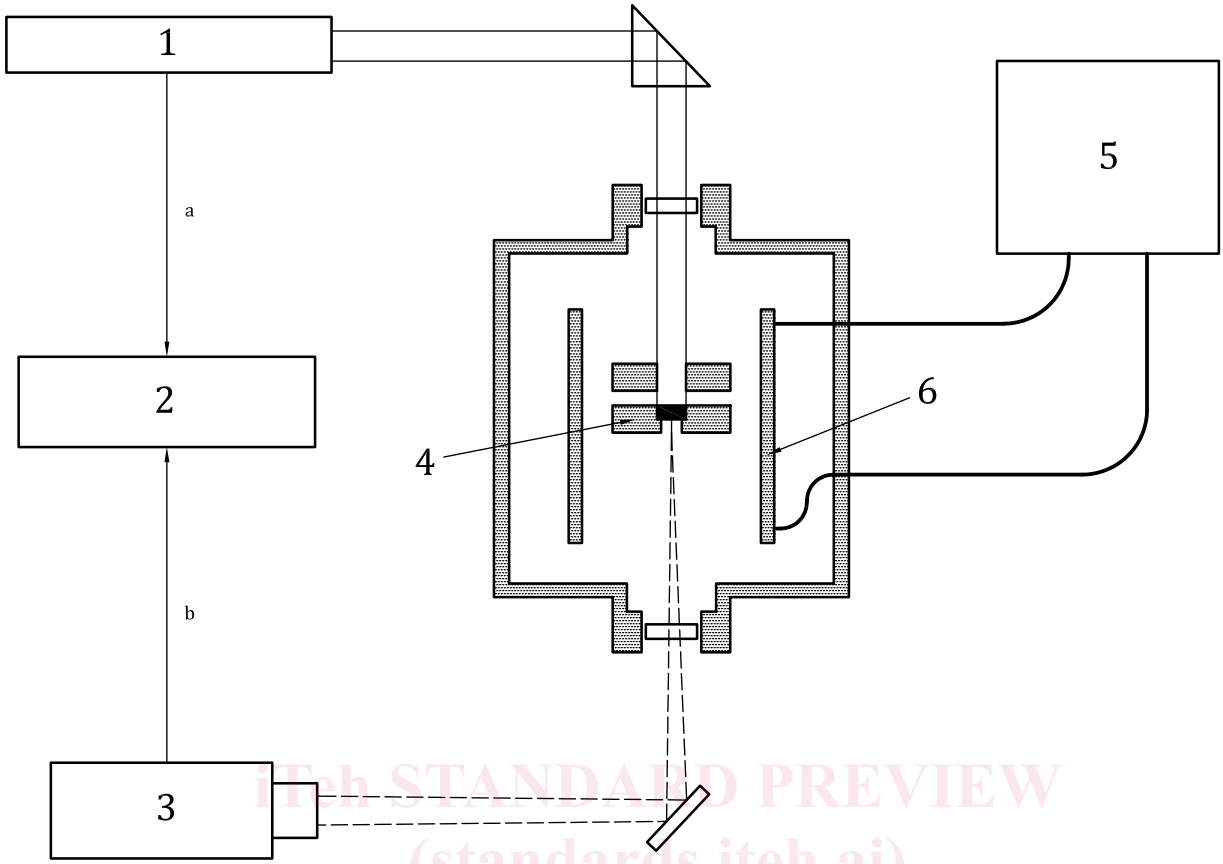
Note 1 to entry: See Figure 1.

3.13
extrapolated temperature rise
ΔT_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 ΔT_{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 ΔT_{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, α, 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, ΔT_{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, α. 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);

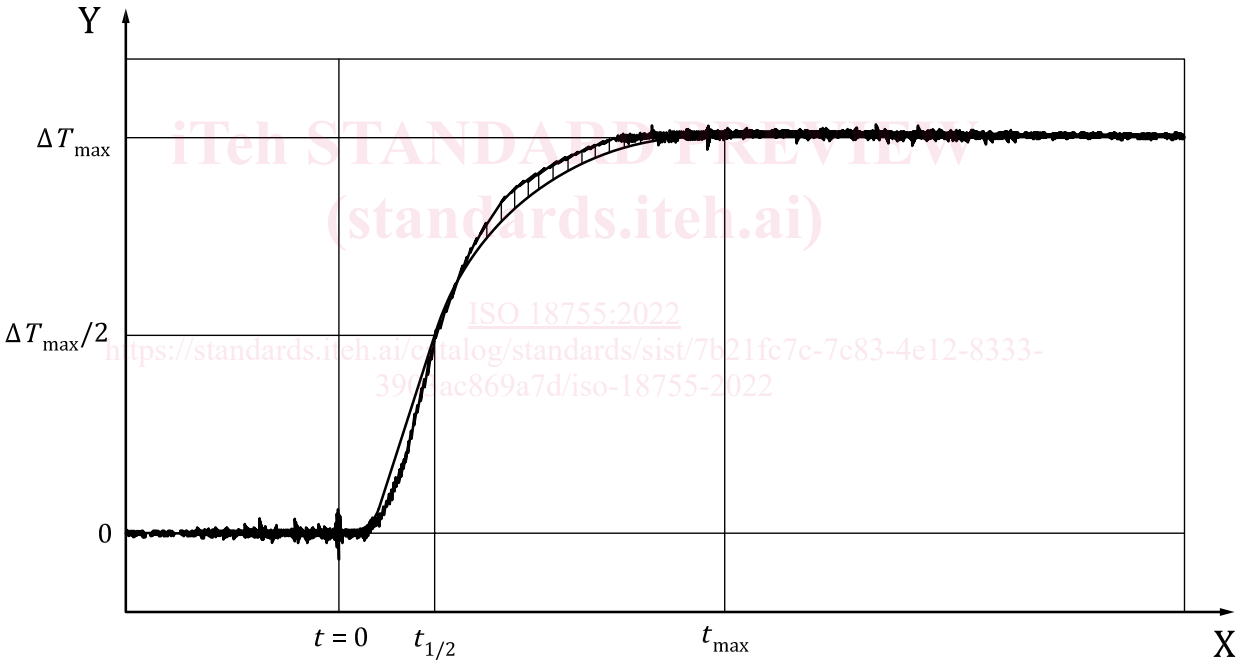
x is the percentage of the maximum rise in temperature;

K_x is a constant relating α 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 ΔT_{max}. If the averaged deviation is within ±1 % then it can be assumed that no corrections apply.