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**Measurement methods of thermal
diffusivity, specific heat capacity,
and thermal conductivity for fine
ceramics by flash method**

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Foreword

This translation has been made based on the original Japanese Industrial Standard revised by the Minister of Economy, Trade and Industry through deliberations at the Japanese Industrial Standards Committee as the result of proposal for revision of Japanese Industrial Standard submitted by Japan Fine Ceramics Association (JFCA)/ Japanese Standards Association (JSA) with the draft being attached, based on the provision of Article 12 Clause 1 of the Industrial Standardization Law applicable to the case of revision by the provision of Article 14.

Consequently **JIS R 1611**:1997 is replaced with this Standard.

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Measurement methods of thermal diffusivity, specific heat capacity, and thermal conductivity for fine ceramics by flash method

Introduction

This Japanese Industrial Standard has been prepared based on the first edition of **ISO 18755** published in 2005 with some modifications of the technical contents made to reflect the recent advancement in the measurement technology.

The portions given sidelines or dotted underlines are the matters in which the contents of the corresponding International Standard have been modified. A list of modifications with the explanations is given in Annex JE. Annexes JA to JD contain matters that are not given in the corresponding International Standard.

1 Scope

This Standard specifies the test method for the determination of thermal diffusivity from room temperature to 1 700 K, specific heat capacity from room temperature to 1 000 K, and thermal conductivity from room temperature to 1 000 K by the flash method for homogeneous ceramics with porosity 10 % or less.

NOTE : The International Standard corresponding to this Standard and the symbol of degree of correspondence are as follows.

ISO 18755:2005 *Fine ceramics (advanced ceramics, advanced technical ceramics)—Determination of thermal diffusivity of monolithic ceramics by laser flash method* (MOD)

In addition, symbols which denote the degree of correspondence in the contents between the relevant International Standard and **JIS** are IDT (identical), MOD (modified), and NEQ (not equivalent) according to **ISO/IEC Guide 21-1**.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Standard. The most recent editions of the standards (including amendments) indicated below shall be applied.

JIS B 0601 *Geometrical Product Specifications (GPS)—Surface texture: Profile method—Terms, definitions and surface texture parameters*

JIS B 7502 *Micrometer callipers*

NOTE : Corresponding International Standard: ISO 3611 *Micrometer callipers for external measurement* (NEQ)

JIS C 1602 *Thermocouples*

JIS R 1600 *Glossary of terms relating to fine ceramics*

JIS R 1634 *Test methods for density and apparent porosity of fine ceramics*

JIS R 1672 *Determination of specific heat of fiber-reinforced ceramics composite by differential scanning calorimetry methods*

JIS Z 8401 *Guide to the rounding of numbers*

ISO/IEC Guide 98-3 *Uncertainty of measurement—Part 3: Guide to the expression of uncertainty in measurement*

3 Terms and definitions

For the purpose of this Standard, the terms and definitions given in **JIS R 1600** and the following apply.

3.1 pulse width, τ_p

full width of half maximum (FWHM) which is the time duration when the pulsed light energy is larger than the half of its maximum value on time basis

3.2 non-uniformity of pulsed light

the non-uniformity of intensity distribution of pulsed light on a plane vertical to the irradiation direction

3.3 transient temperature curve

transient change of the spectral radiance from the rear face of the specimen after the pulse heating (see figure 1)

3.4 steady-state temperature, T_0

specimen temperature maintained in a steady state before pulse heating

3.5 maximum temperature rise, ΔT_m

difference between the maximum temperature and the steady-state temperature on the transient temperature curve

3.6 measurement temperature, T_e

effective temperature of a specimen when the temperature which rises due to pulse heating is taken into consideration

3.7 time origin of analysis, t_0

time to which impulse heating is made to correspond, in the data analysis that calculates the thermal diffusivity from a transient temperature curve

3.8 rise time of pulse, t_p

time when emission of pulsed light starts

3.9 centroid of laser pulse, t_g

observed chronological centroid of pulsed light energy

3.10 characteristic time of thermal diffusion, τ_0

time defined by $\tau_0 = d^2/\alpha$ when heat is diffused from the front face to the rear face of a flat plate specimen of thickness d and thermal diffusivity α

3.11 half rise-time, $t_{1/2}$

time required for the temperature rise from the time origin of analysis to 1/2 the maximum temperature rise in the transient temperature curve

3.12 temperature increasing domain

time domain in which temperature is increasing with time in a transient temperature curve

3.13 temperature decreasing domain

time domain in which temperature is decreasing with time in a transient temperature curve

3.14 time constant of cooling, τ_c

time constant of relaxation determined when the temperature decreasing domain of the transient temperature curve is fitted with an exponential function which converges the temperature decreasing domain to the steady-state temperature

3.15 extrapolated temperature rise, ΔT_{ext}

the exponential function extrapolated to the time origin of analysis when the temperature decreasing domain is fitted with an exponential function which cools the temperature decreasing domain to the steady-state temperature

3.16 adiabatic temperature rise, ΔT_0

maximum temperature rise under ideal conditions where there is no heat loss from the specimen, which is observed on the transient temperature curve

3.17 initial noise

signal that changes in the shape of a spike on the transient temperature curve, observed with the pulse heating time at the centre

3.18 signal change by extraordinary light, ΔT_r

change of a transient temperature curve generated by the pulsed light transmitted and scattered without being absorbed in a specimen surface

3.19 trigger signal

signal that transmits the time of pulse heating to a recording device

3.20 Biot number, Y

dimensionless number indicating the magnitude of the heat loss from a specimen to an outside

3.21 heat capacity

heat quantity required to raise the temperature of an object such as a specimen by 1 K (J/K)

3.22 differential method

method of measurement, in flash method, which is performed as follows: a reference specimen and a measurement specimen are adjacently installed, and being set at a measurement temperature, subjected to pulse heating, then the change of the rear surface temperature of the specimens is measured, the results of which are then compared to determine the heat capacity and specific heat capacity of a measurement specimen at the set temperature

3.23 specimen exchange method

method of measurement, in flash method, which is performed as follows: a reference specimen and a measurement specimen are each subjected to a series of measurements with the specimen temperatures ranging from the room temperature to the elevated temperature as necessary, and the results of the two specimens at the same temperature, or of one specimen at two temperatures (the room temperature and the elevated temperature) are compared to determine the heat capacity and specific heat capacity of a measurement specimen

3.24 standard specimen of thermal diffusivity

specimen in which thermal diffusivity is valued for the purpose of performing the confirmation of the measurable range of a thermal diffusivity measuring apparatus and the correction of a thermal diffusivity value

Standard materials of thermal diffusivity, pure materials in which recommended values of thermal diffusivity are given, etc. supplied by public institutions such as National Metrology Institute of Japan are used (see Annex E).

3.25 reference specimen of (specific) heat capacity

specimen of which specific heat capacity and mass (or heat capacity) are known

It is used when specific heat capacity (or heat capacity) is obtained by the comparison measurement of the flash method. Standard materials of (specific) heat capacity, pure materials in which recommended values of specific heat capacity are given, etc. supplied by public institutions such as National Metrology Institute of Japan are used.

3.26 variation of the specimen thickness

difference between the maximum and the minimum of the thickness of a specimen at an arbitrary position

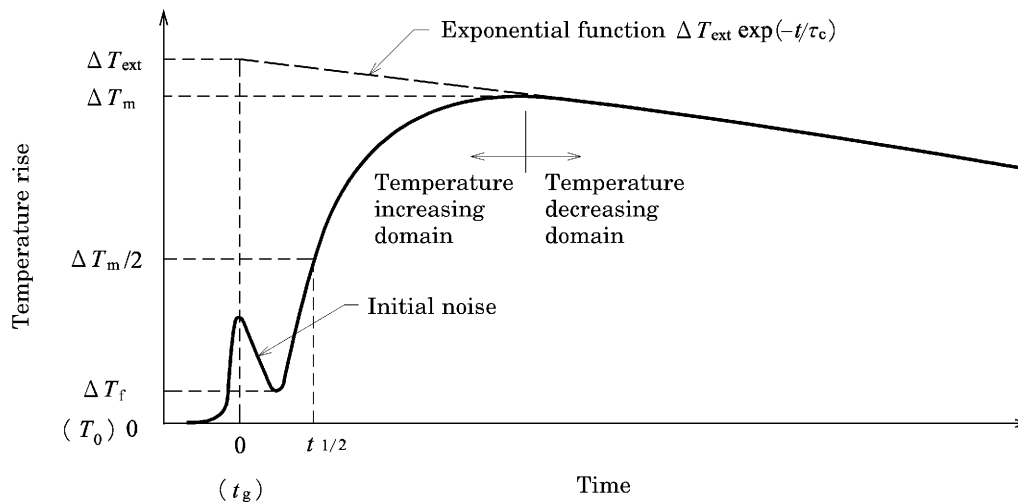


Figure 1 Transient temperature curve observed by the flash method

4 Principle

Thermal diffusivity is obtained from an observed transient temperature curve according to the flash method in which the surface of a flat plate specimen is heated momentarily by spatially uniform pulsed light and the temperature rise on the rear face of the specimen is observed.

Specific heat capacity is obtained by the comparison measurement in which the heat capacity of the measurement specimen is calculated from the ratio between the adiabatic temperature rise at the time of pulse heating of the reference specimen having the same absorptance to heating pulsed light, the same spectral emissivity in the radiation thermometry wavelength and the heat capacity known and the adiabatic temperature rise at the time of pulse heating of the measurement specimen. The comparison measurement is performed by the differential flash method (see figure 2) or the specimen exchange flash method (see figure 3).

Thermal conductivity (λ) is obtained as the product of thermal diffusivity (α), specific heat capacity (c) and bulk density (ρ), $\lambda = \alpha c \rho$.

Specific heat capacity is obtained by measuring in accordance with the Differential Scanning Calorimetry (DSC) of **JIS R 1672**, etc., or may use literature values. Furthermore, bulk density may be obtained in accordance with **JIS R 1634**, etc.

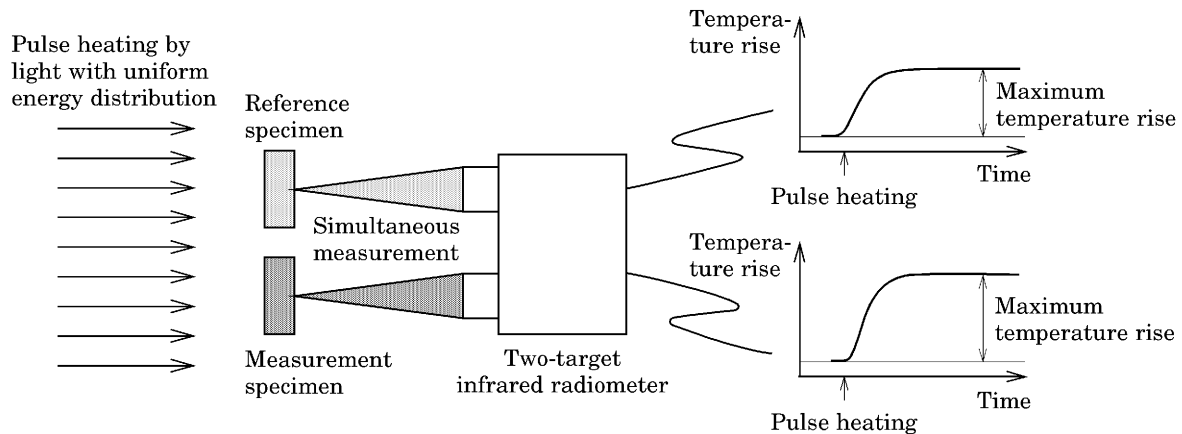


Figure 2 Example of arrangement in which two specimens installed adjacently are measured simultaneously in flash method (differential method)

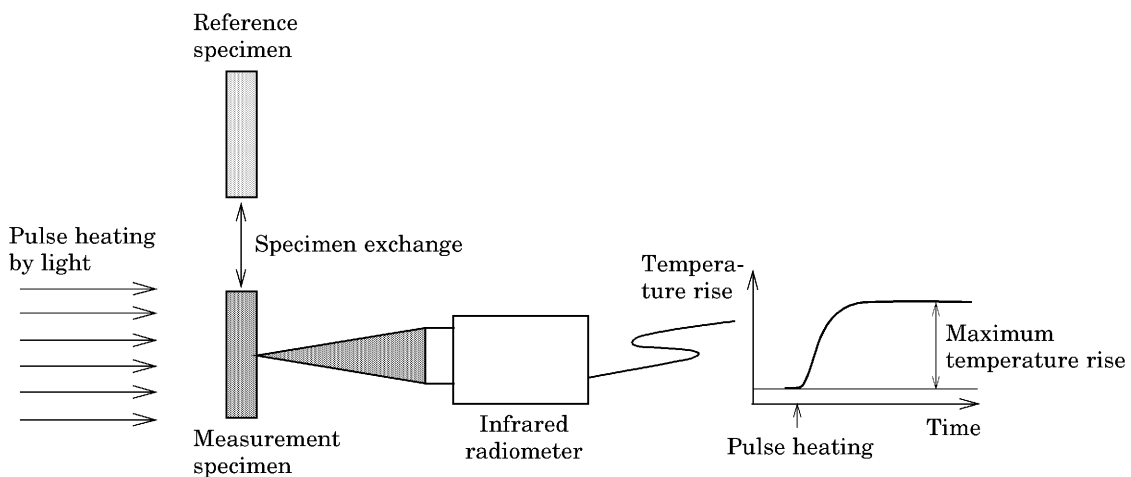


Figure 3 Example of arrangement in which specimens are measured by exchanging them in flash method (specimen exchange method)

5 Thermal diffusivity measurement

5.1 Apparatus and instrument

The apparatus and instrument shall be designed for obtaining the thermal diffusivity from the transient temperature curve of the rear face of a specimen after the pulsed light is irradiated onto the front face of the specimen, and shall consist of the following principal components (see figure 4).

- Specimen holder** The specimen holder shall hold the specimen vertical to the incident direction of the pulse light, with minimum light scattered to the side and rear surface of the specimen, and shall be designed such that the thermal conductivity loss from the specimen is minimum.
- Temperature measuring apparatus** The temperature measuring apparatus as follows shall be used:

- 1) **Temperature response measuring apparatus** The temperature response measuring apparatus, used for obtaining the transient temperature curve of the thermal diffusivity measurement, shall be the infrared radiometer. For the infrared radiometer, the response shall be not less than 10 kHz, and the output shall be capable of being converted into temperature as necessary. For the measured value, the effect of the heat loss from the side face of a specimen shall be avoided, and the domain in which a one-dimensional thermal-diffusion phenomenon becomes effective near the centre of the rear face of a specimen shall be obtained.

NOTE : It is specified in **ISO 18755** that the size of the domain which an infrared radiometer observes should be smaller than, for the disk specimen, the circle of which the diameter is taken as its radius, and for the flat plate specimen of convex polygon, the circle of which the diameter is taken as the radius of its inscribed circle.

- 2) **Contact thermometer** The temperature shall be measured by a thermocouple specified in **JIS C 1602**. The thermocouple shall be positioned in contact with the specimen or the nearest specimen holder, such that it does not interrupt the measurement of the transient temperature curve by the temperature response measuring instrument.
- c) **Pulse heating apparatus** The pulse heating apparatus shall consist of a pulse emitting device (pulse laser or flash lamp) capable of emitting the pulsed light, in vicinity of the specimen, with beam diameter 10 mm or greater and pulse width (τ_p) 0.1 ms to 1 ms, and an optical system.

NOTE : Use of optical fibre is effective for homogenization of energy distribution of the pulsed light.
- d) **Measurement container and electric heater** The measurement container and the electric heater shall be as follows.

- 1) **Measurement container** For measurement at an elevated temperature, the measurement container used shall be of a highly airtight structure which enables measurements in vacuum state, so that chemical reactions, such as oxidation, of specimens, etc. do not occur, and convective heat loss of the specimen is restrained. The container may be of a structure which enables measurement in inert gas atmosphere. For those measurements where the effects of chemical reactions or the convective heat loss of the specimens, etc. are not required to be considered (e.g. measurements at a comparatively low temperature), the container need not be of a airtight structure.
- 2) **Electric heater and temperature control unit** The electric heater shall be with the performance and thermal resistance to be capable of heating the specimen to the measurement temperature, and provided with the temperature control unit capable of setting the temperature of the specimen. The stability in keeping the temperature of the specimen shall be confirmed by the drift and fluctuation of the output of the temperature response measuring apparatus (0.01 K/s maximum).

- e) **Measurement circuit and recording device** The measurement circuit shall have the responsivity of 10 kHz or more, and shall be capable of automatically recording the transient temperature curve. The resolution of the AD conversion should be larger than 10 bits, and more than 1 000 data points should be sampled with the sampling time faster than 1/100 the half rise-time.
- f) **Standard specimen of thermal diffusivity** It is a specimen in which thermal diffusivity is valued for the purpose of confirming the measurable range of a thermal diffusivity measuring apparatus and correcting thermal diffusivity values (see Annex E).

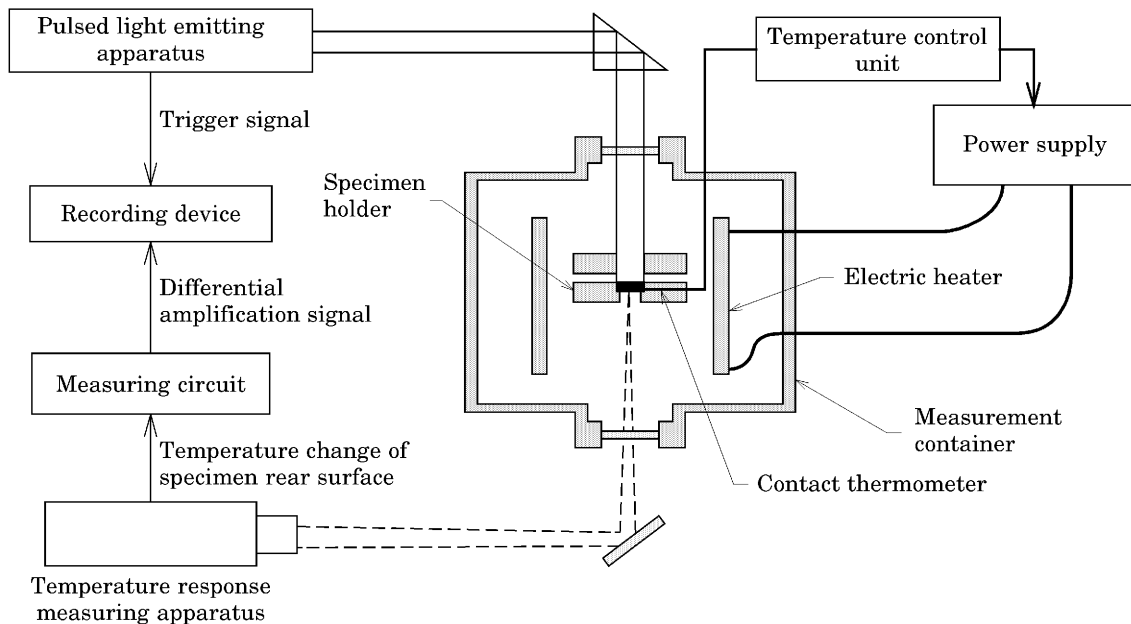


Figure 4 Example of flash method measuring apparatus

5.2 Specimen

5.2.1 Shape of specimens

The specimen shall be either a disk or a convex polygon, between 4 mm and 15 mm in diameter of the inscribed or circumscribed circle. The circumscribed circle of the specimen shall be such that it is within the irradiation range of the pulsed light. The thickness of the specimen shall be 0.5 mm or over up to and including 5 mm, and the half rise-time value shall be $5\tau_p$ or larger. The variation of the specimen thickness shall be 1 % maximum with respect to the thickness, and the front and rear surface roughness of the specimen shall be not more than $0.40 \mu mRa$.

For the definition of roughness, see **JIS B 0601**.

NOTE 1 The circumscribed circle of the specimen should be in the range in which the energy distribution of pulsed light is uniform.

NOTE 2 The inscribed circle of the specimen should be wider than the observation visual field in the rear face of the specimen of an infrared radiometer.

5.2.2 Surface treatment

The surface treatment shall be performed as follows.

- a) **Nontranslucent specimen** If the specimen front surface (surface irradiated with the pulsed light) does not have a high absorption coefficient for the pulsed light, or if its rear surface (measured surface) does not have a high emissivity for the observed wavelength of a temperature response measuring apparatus, the surfaces of the specimen should be coated with a thin and uniform black layer by spraying, evaporating, or sputtering material containing carbon fines.
- b) **Translucent specimen** The same surface treatment as **a)** shall be performed on the front and rear faces of a specimen. For the specimen of which the translucency is not negligible even after the surface treatment, the treatment in **a)** shall be performed after a metal film such as gold is deposited so that $\Delta T_f/\Delta T_m$ becomes 0.05 or less.
- c) **Evaluation of effect of surface treatment** The evaluation of the effect of the surface treatment (blackening treatment) on the measurement should be performed in accordance with Annex JC.

5.3 Measurement procedure

5.3.1 Measurement of standard specimen of thermal diffusivity¹⁾

Measurement using a standard specimen shall be performed to evaluate the characteristics of the apparatus, such as heat loss and non-uniformity of pulsed light. The results shall be corrected as necessary (see Annex JB).

The standard specimen shall be a flat plate having a shape equivalent to that of the specimen, i.e. either a disk or a convex polygon. Several specimens, each different in thickness, should be used. The measurement of a standard specimen for the transient temperature curve shall be performed according to the procedure in **5.3.3**.

Note ¹⁾ If it is considered that the characteristics of an apparatus have not changed, the results from the previous measurement may be used.

5.3.2 Determination of centroid of pulsed light intensity in chronological measurement

If the chronological centroid obtained from the pulsed light waveform at the time origin of analysis is used for the analysis of the transient temperature curve, the pulsed light waveform shall be measured referring to Annex D, and the chronological centroid of pulsed light (t_g) shall be determined. If the pulse width (τ_p) is larger than 1 % of the half rise-time ($t_{1/2}$), t_g should be regarded as the time origin of analysis.

The transient temperature curve may be analyzed using the theoretical formula which considers the time variation of emission intensity without approximating pulsed light by delta function located at the chronological centroid.

5.3.3 Measurement of specimen

Specimen shall be measured under the following procedures.

- a) **Measurement of thickness of specimen** Measure the thickness of the specimen to an accuracy of 1 μm , using a micrometer caliper in accordance with **JIS B 7502**.

- b) **Surface treatment** Carry out the surface treatment in accordance with 5.2.2.
- c) **Control of atmosphere** The atmosphere shall be such that the specimen is not subjected to any chemical change under the measured temperature range.
- d) **Control of specimen temperature** The specimen temperature shall be controlled with drift not greater than 0.2 K/s per duration $100t_{1/2}$.
- e) **Measurement of steady-state temperature** Measure the steady-state temperature of the specimen (T_0) using a contact thermometer.
- f) **Measurement of the transient temperature curve** Measure the transient temperature curve according to the following.
 - 1) **Pulse heating** Irradiate pulsed light with such an intensity that ΔT_m of the specimen does exceed 3 K.

NOTE : A thermocouple with a diameter 0.15 mm or smaller may be directly contacted to the specimen to estimate (ΔT_m to see whether it exceeds 3 K.

- 2) **Recording** The transient temperature curve measured by means of a temperature response measuring apparatus should be recorded for at least until 10 times the half rise-time is reached.

The temperature change prior to the heating pulse shall be recorded for the duration equal to 10 % of the entire measurement, so that it can be used for information such as the stability of the steady-state temperature prior to the heating pulse.

- 3) **Determination of the measurement temperature²⁾** Determine the measurement temperature (T_c) as $T_0 + \Delta T_m$, where T_0 is the steady-state temperature of the specimen immediately before pulse heating.

In the measurement of a thick specimen, etc., when the effect of noise is not negligible because signals are small, the SN ratio may be improved by integrating, at every given time, the transient temperature curves measured multiple times (overstriking method).

Note ²⁾ In the case of determining the thermal diffusivity independent of the measuring conditions, from the measurement in which the pulse heating strength is varied (see Annex JA), the measurement temperature may be the steady-state temperature (T_0). Even when the temperature dependence of the thermal diffusivity is known, and the difference between the thermal diffusivity at the steady-state temperature and that at the measurement temperature is judged to be 1 % or smaller, the measurement temperature may be the steady-state temperature (T_0).

5.4 Calculation

5.4.1 Calculation method of thermal diffusivity

Thermal diffusivity shall be calculated by any of the following methods. In any formula, d shall be the thickness of the specimen without a blackened film.

- a) **Half rise-time method** Thermal diffusivity shall be calculated according to formula (1).

$$\alpha = 0.1388 \times k_{bc} k_m \frac{d^2}{t_{1/2}} \dots\dots\dots (1)$$

where, α : thermal diffusivity (m²/s)
 k_{bc} : correction factor for blackening treatment (see Annex JC)
 k_m : correction factor associated with measurement conditions (see Annex B)
 d : thickness of specimen (m)
 $t_{1/2}$: half rise-time (s)

Under the measurement condition where the room temperature measurement and heat loss can be disregarded, $k_m = k_{nuh}$ (see Annex B) shall apply, and in the apparatus in which pulsed light is uniformed, $k_m = k_{rhl}$ (see Annex B) can apply.

- b) **Least-square method** The thermal diffusivity shall be calculated according to formula (2).

$$\alpha = k_{bc} \times \alpha_{lsf} \dots\dots\dots (2)$$

where, α : thermal diffusivity (m²/s)
 k_{bc} : correction factor for blackening treatment (see Annex JC)
 α_{lsf} : when a square deviation is constituted using a transient temperature curve and a theoretical formula, the thermal diffusivity that makes this square deviation minimal (m²s⁻¹)

Since in the measurement at an elevated temperature, square deviation serves as a function of Biot number (Y) that indicates thermal diffusivity and radiative loss, the method that determines thermal diffusivity making the square deviation minimal and Biot number is effective.

In this calculation method, it is possible to perform the correction for non-uniform heating and heat loss simultaneously by consideration based on theoretical formula. Furthermore, thermal diffusivity with the effect of finite pulse width corrected can be calculated by applying the theoretical formula which considers the waveform of pulsed light (time dependence of intensity).

NOTE : In **ISO 18755**, this calculation method is described in Annex C (informative).

- c) **Equiareal method** The thermal diffusivity shall be calculated according to formula (3).

$$\alpha = k_{bc} \times \alpha_{areal} \dots\dots\dots (3)$$

where, α : thermal diffusivity (m²/s)
 k_{bc} : correction factor for blackening treatment (see Annex JC)

α_{areal} : when the theoretical curve under the initial condition and boundary condition that are realized in a transient temperature curve and its measurement is integrated by the set time domain ($t_1 < t < t_2$), thermal diffusivity determined so that both values become the same (m^2/s) (see Annex C)

Under the ideal measurement condition, analysis shall be performed on the theoretical formula by Parker et al. and others (see Annex A). In the measurement at higher temperature than room temperature, analysis shall be performed on the theoretical formula that takes radiative heat loss into consideration, and thermal diffusivity and Biot number (Y) shall be determined (see Annex C).

NOTE : This calculation method is described in Annex C (informative) of **ISO 18755**.

5.4.2 Correction items

The correction items shall be as follows.

- a) **Light-emitting time of pulsed light** The chronological centroid of a waveform based on the intensity waveform signal of pulsed light shall be obtained, and the time origin of analysis shall be determined by adding correction to the time origin of the apparatus. In the case of the specimen of which the half rise-time ($t_{1/2}$) is shorter than $100 \tau_p$, it is necessary to obtain the chronological centroid correctly. When half rise-time is long and pulse width is about the same degree as at the data sampling time, it is not necessary to correct the time origin.
- b) **Non-uniform heating** When the specimen surface is heated non-uniformly due to the partially non-uniform pulsed light, or the absorption distribution of the surface to be heated of the specimen, etc., one-dimensional thermal diffusion is not realized inside the specimen and uncertainty is generated. In this case, the correction factor (k_{nuh}) for non-uniform heating is obtained by the ratio of the time constant of non-uniform heating to half rise-time and the relative amplitude in the transient temperature curve of the specimen. See Annex B for the calculation method.

NOTE 1 When $0.98 < k_{\text{nuh}} < 1.02$, correction is not necessary.

NOTE 2 In the least-square method and the equiareal method, when the model function that takes non-uniform heating into consideration is adopted, correction is not necessary.

- c) **Heat loss of specimen** Since in the measurement at high temperature heat loss from a specimen to the outside is generated, when the thermal diffusivity is calculated, correction shall be performed. The correction factor (k_{rhl}) for heat loss is obtained as the function of the ratio of the time constant of cooling to half-rise time. See Annex B for the calculation method.

NOTE 1 When $0.98 < k_{\text{rhl}}$, correction is not necessary.

NOTE 2 In the least-square method and the equiareal method, when the model function that takes heat loss into consideration is adopted, correction is not necessary.

- d) **Non-linearity of heat radiation** When a transient temperature curve is observed using an infrared radiometer without temperature scales, spectral radiance change is observed. In this case, the thermal diffusivity that is not affected by non-linearity can be obtained by changing the output of the light source for pulse heating, measuring the apparent thermal diffusivity and extrapolating the output at zero (see Annex JA).
- e) **Surface treatment** When the surface treatment of a blackened film, etc. is applied to a specimen prior to the measurement, the surface treatment will have an effect on the measurement. When this effect cannot be disregarded, correction shall be performed in accordance with Annex JC. If $k_{bc} > 1$ and $k_{bc} < 1.05$ always hold true, correction is not necessary.
- f) **Thermal expansion** The thermal diffusivity calculated in 5.4.1 is the apparent thermal diffusivity $[\alpha_0(T)]$ on the basis of the thickness of a specimen at room temperature. When the thermal diffusivity on the basis of the thickness of a specimen at measurement temperature $[\alpha(T)]$ is needed, correction shall be made on the thermal expansion of the specimen according to formula (4).

$$\alpha(T) = \left(\frac{d_T}{d_0} \right)^2 \times \alpha_0(T) \dots\dots\dots (4)$$

where, d_0 : thickness of specimen at room temperature (m)
 d_T : thickness of specimen at temperature T (m)

6 Measurement of specific heat capacity

6.1 General

Heat capacity and specific heat capacity are obtained by the method of either the flash method or the Differential Scan Calorimetry (DSC), or literature values are used. Specific heat capacity is calculated by dividing the heat capacity of a specimen by the mass of a specimen.

6.2 Flash method

6.2.1 Differential method

In the differential flash method, as shown in figure 2, the measurement specimen and the reference specimen of which the heat capacity is known shall be installed closely, the measurement specimen and the reference specimen shall be simultaneously irradiated with spatially uniform pulsed light, and the heat capacity of the measurement specimen shall be obtained from the temperature rise ratio between two specimens and the heat capacity of the reference specimen.

The differential method shall be as follows.

- a) **Apparatus and instrument** The apparatuses and instruments shall be designed for measuring the specific heat capacity from the transient temperature curve of the rear face of a specimen after the pulsed light is irradiated onto the front face of a specimen (see figure 1), and shall have following elements.