INTERNATIONAL STANDARD

**ISO
21901**

Thermal insulation — Test method for thermal diffusivity — Periodic heat method

Isolation thermique — Méthode d'essai pour la diffusivité thermique — Méthode de chauffage périodique

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Foreword

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This document was prepared by Technical Committee ISO/TC 163, *Thermal performance and energy use in the built environment*, Subcommittee SC 1, *Test and measurement methods*.

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Thermal insulation — Test method for thermal diffusivity — Periodic heat method

1 Scope

This document specifies a periodic heat method for measurement of the thermal diffusivity of thermal insulation material in the shape of a flat plate.

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 8302:1991, *Thermal insulation — Determination of steady-state thermal resistance and related properties — Guarded hot plate apparatus*

3 Terms and definitions

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

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

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

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

3.1
phase
quantity that shows the stage of progress in one *period* (3.5) of wave motion and other periodic phenomenon

EXAMPLE $\omega t + \varphi_1$ in $y = A\sin(\omega t + \varphi_1)$ is the phase.

3.2
phase lag
difference between two *phases* (3.1) provided by periodic temperature changes measured at two different points on the surface and inside a test specimen

EXAMPLE   When the periodic temperature changes 1 and 2 are given by $y_1 = A_1\sin(\omega t + \varphi_1)$ and $y_2 = A_2\sin(\omega t + \varphi_2)$, respectively, the phase lag between them is $\varphi_2 - \varphi_1$.

3.3
amplitude
half of the difference between the maximum and minimum values of the amount of displacement in a periodic temperature change

EXAMPLE A in a sine wave given by $A\sin(\omega t + \varphi)$ or $A\exp[i(\omega t + \varphi)]$ refers to the amplitude.

3.4
amplitude ratio
ratio of two *amplitudes* (3.3) provided by periodic temperature changes measured at any point on the surface and inside a test specimen.

EXAMPLE   When the periodic temperature changes y_1 on the surface and y_2 inside the test specimen are given by $y_1 = A_1 \sin(\omega t + \varphi_1)$ and $y_2 = A_2 \sin(\omega t + \varphi_2)$, respectively, the amplitude ratio between the two amplitudes is A_2 / A_1.

3.5
period
reproduction interval of a periodic phenomenon (or a periodic function)

Note 1 to entry: When the time interval is constant, the period f is given by $f = 1/v = 2\pi/\omega$, where v is the *frequency* (3.6) and ω is the *angular frequency* (3.7).

3.6
frequency
number of repetitions of the same state within a certain time interval of any temporal periodic phenomena

Note 1 to entry: It is expressed as $v = 1/f$, where f is the *period* (3.5).

3.7
angular frequency
rotation angle per second

Note 1 to entry: Angular frequency is obtained by multiplying the *frequency* (3.6) v by 2π ($2\pi v$) or by multiplying the reciprocal of the *period* (3.5) f by 2π ($2\pi/f$).

4   Symbols

The symbols used in this document are given in Table 1.

Table 1 — Symbols

Symbol	Quantity	Unit
a	Thermal diffusivity	m^2/s
c	Specific heat capacity	$J/(kg \cdot K)$
D	Difference	%
d	Thickness of test specimen (total thickness of the two pieces when using two stacked test pieces in a measurement)	m
f	Period	s
i	Imaginary unit	-
k	Damping coefficient	1/m
L	Length of one side of test specimen	m
l	Distance from periodic heating surface ($d - x_m$)	m
m	Mass prior to measuring thermal diffusivity (prior to grooving)	kg
T	Absolute temperature	K
V	Volume prior to measuring thermal diffusivity (prior to grooving)	m^3
x	x coordinate	m
x_m	Location of temperature measurement point inside a test specimen	m
ε	Phase lag on $x = 0$ surface	rad
η	Arbitrary phase	rad

Table 1 *(continued)*

Symbol	Quantity	Unit
θ	Temperature	°C
λ	Thermal conductivity	W/(m·K)
v	Frequency	1/s
π	Circular constant	-
ρ	Density, bulk density	kg/m³
φ	Phase lag at $x = x_m$ cross section	rad
ω	Angular frequency	1/s

5 Principle

5.1 General

The periodic heat method is a method for obtaining thermal diffusivity by measuring the temperature at two different points in a test specimen. Thermal diffusivity is evaluated by the phase lag between two phases provided by periodically changing the temperature of one side surface of the test specimen. Here, those temperatures are measured at any point on the heating surface and inside (the internal plane perpendicular to the direction of thermal diffusion) the test specimen.

This measurement method uses a solution of the heat conduction equation that is obtained under the condition that the temperature on one side of a test specimen changes as a trigonometric function of time and the change propagates in a one-dimensional direction, and further, the temperature on the opposite side is kept constant.

The periodical temperature change on the heated surface shall be expressed by a trigonometric function of time.

It is contrary to the measurement principle to generate a distorted temperature change on the heated surface. In addition, in reality, the waveform easily changes in a distorted temperature change during propagation in a test specimen, and this becomes a cause of measurement errors.

5.2 Calculation of thermal diffusivity

As shown in Figure 1, the x-axis is taken upward along the thickness direction of a test specimen, and the heat dissipation surface (the surface in contact with the low-temperature side heater) of the test specimen with thickness d is set as the origin, while the heating surface is set at $x = d$. Accordingly, as shown by the thick arrow, heat flows downward from the heating surface to the heat dissipation surface. At the heat dissipation surface, the temperature is kept constant, and the temperature of the heating surface is changed periodically based on $\theta_H + \theta_0 \sin(\omega t + \eta)$. Under these conditions, the phase lag (rad) between the point at $x = d$ and an arbitrary point at $x = x_m$ is obtained by solving the one-dimensional heat conduction equation. As a result, Formula (1) is given (see Figure 2).

Key
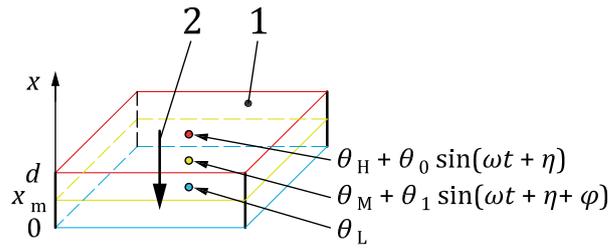
1   test specimen

2   heat flow

Figure 1 — Relation between test specimen and coordinate (one-dimensional heat flow, in the case θ_L is kept constant)

Key
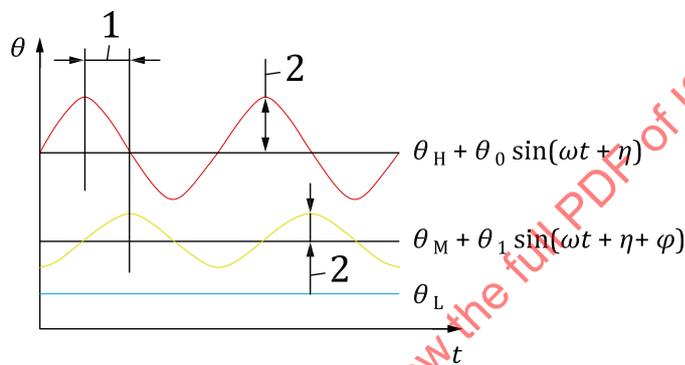
1   phase lag

2   amplitude

Figure 2 — Periodic temperature change, in the case θ_L is kept constant

$$\varphi = \arg\left\{\frac{\sinh kx_m\,(1+i)}{\sinh kd\,(1+i)}\right\} \tag{1}$$

The damping coefficient k in Formula (1) is defined by Formula (2).

$$k = \sqrt{\frac{\omega}{2a}} \tag{2}$$

Also, the angular frequency ω is defined by Formula (3).

$$\omega = \frac{2\pi}{f} \tag{3}$$

At this time, mean temperature of a test specimen θ_S is calculated by Formula (4).

$$\theta_S = (\theta_H + \theta_L)/2 \tag{4}$$

When using Formula (1), the surface temperature on the low-temperature side of a test specimen shall always be controlled so as to be constant.
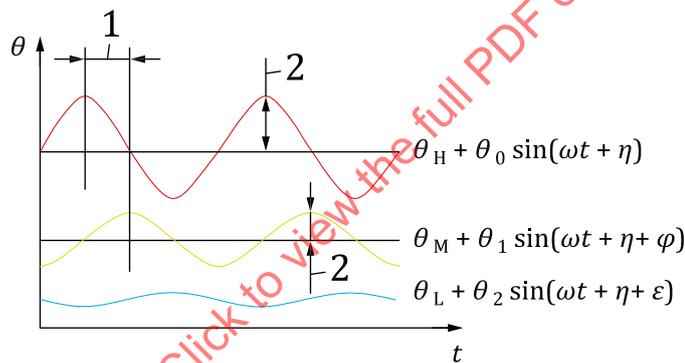
If the periodical temperature changes on the low-temperature side, as described in Figure 3, it is also possible to solve the phase lag between $x = d$ and $x = x_m$ by solving the one-dimensional heat conduction equation in the same manner as shown above. However, in this case, thermal diffusivity is calculated using the phase lag and the amplitude ratio.

Key
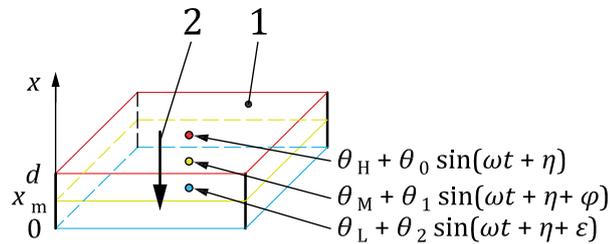
1 test specimen

2 heat flow

Figure 3 — Relation between test specimen and coordinates (one-dimensional heat flow, in the case θ_L changes periodically)

Key

1 phase lag

2 amplitude

Figure 4 — Periodic temperature change, in the case θ_L changes periodically

As a result, Formula (5) is given (see Figure 4). The coefficients used in Formula (5) are shown in Formulae (6), (7), (8) and (9). The damping coefficient k and the angular frequency ω are defined in Formulae (2) and (3).

If the temperature on the low-temperature side is constant, Formula (5) is equal to Formula (1).

$$\varphi = \arctan\left\{\frac{\theta_{1H}\sin(\varphi_H)+\theta_{1L}\sin(\varphi_L+\varepsilon)}{\theta_{1H}\cos(\varphi_H)+\theta_{1L}\cos(\varphi_L+\varepsilon)}\right\} + \begin{cases} 0 \ \text{if} \ \theta_{1H}\cos(\varphi_H)+\theta_{1L}\cos(\varphi_L+\varepsilon) \geq 0, \\ \pi \ \text{if} \ \theta_{1H}\cos(\varphi_H)+\theta_{1L}\cos(\varphi_L+\varepsilon) < 0, \end{cases} \tag{5}$$

$$\theta_{1H} = \theta_0\sqrt{\frac{\cosh(2kx_m)-\cos(2kx_m)}{\cosh(2kd)-\cos(2kd)}} \tag{6}$$

$$\theta_{1L} = \theta_2\sqrt{\frac{\cosh(2kl)-\cos(2kl)}{\cosh(2kd)-\cos(2kd)}} \tag{7}$$

$$\varphi_{H} = \arg\left(\frac{\sinh\{kx_{m}(1+i)\}}{\sinh\{kd(1+i)\}}\right) \tag{8}$$

$$\varphi_{L} = \arg\left(\frac{\sinh\{kl(1+i)\}}{\sinh\{kd(1+i)\}}\right) \tag{9}$$

6 Test specimen

6.1 Sample

The test specimen taken from the sample shall satisfy the homogeneity prescribed in ISO 8302:1991, 1.8.2. Here, the term "homogeneous" means that raw materials such as fibres and particles constituting the specimen are distributed evenly along the direction of heat flow. Namely, the distribution of fibres and particles is independent of their location in the specimen, and there are no extreme voids inside the specimen. The thermal diffusivity of various kinds of materials like fibrous insulations such as ceramic fibre, rock wool, glass fibre and fibrous board, and furthermore, styrene foam, urethane foam, brick and concrete can be measured by applying the periodic heat method. However, the thermal diffusivity of materials which undergo phase change, release gases, expand or shrink excessively or deform as a result of phenomena such as cracks cannot be measured by this method.

6.2 Details of test specimen

The details of the test specimen are as follows.

a) The dimensions of the test specimen shall be the same as those of the heater for periodic heating with the dimensional tolerance of ±2 mm with respect to the heater dimensions.

b) The relationship between the length L of one side and the thickness d of the test specimen should be restricted so as to reduce the error due to heat inflow (outflow) from the edges of the test specimen. In addition, the minimum thickness d of the test specimen should be restricted so as to reduce the error due to the diameter of the thermocouples on the upper/middle/lower surfaces of the test specimen. For example, when using a 3 mm diameter insulator tube, the minimum thickness d is 20 mm.

c) The heating and low-temperature surfaces of the test specimen shall be smooth, and if necessary, test specimens made of deformable materials shall be held by a support frame or a spacer.

d) The test specimen shall be dried in a drier at 105 °C ± 2 °C until it reaches a constant mass. When there is a risk of alteration or deformation by heat, the test specimen shall be dried until it reaches a constant mass at a temperature that causes no alteration, deformation or other property changes.

e) The bulk density of the test specimen can be calculated by using Formula (10) based on the volume at the time of measurement of thermal diffusivity and on the mass prior to measurement of thermal diffusivity.

$$\rho = \frac{m}{V} \tag{10}$$

It is recommended that the ratio of length to width of the specimen (L/d) be 6 or more, as L/d may affect the results of the measurement. If it is necessary to use a smaller value of L/d, the accuracy of the measurement shall be verified by the method described in Annex A.
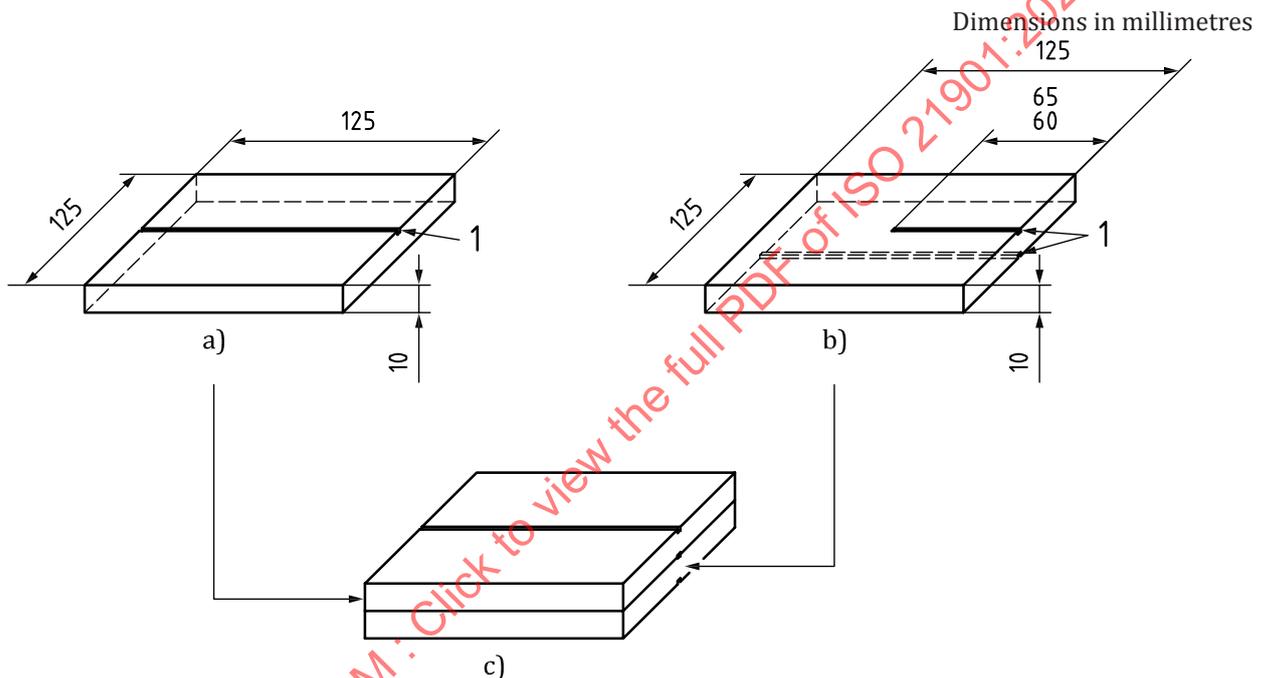
6.3 Machining of test specimen

6.3.1 When using two test specimens

Prepare two test specimens with approximately the same bulk density, and perform the machining work required to set the thermocouples for temperature measurement according to the procedure

shown below. In this case, the total thickness of the specimen composed of the two specimens shall be restricted as described in 6.2.

a) On one specimen, cut a groove about 3 mm wide and 1,5 mm deep at the centre of one surface so that the groove runs across the specimen; see Figure 5 a).

b) On the other specimen, cut a groove about 3 mm wide and 1,5 mm deep from the side to the centre of the specimen surface. In Figure 5 b), the groove length is 60 mm to 65 mm. Also cut a groove with the same dimensions on the other surface so that the groove runs across the specimen; see Figure 5 b).

c) As shown in Figure 5 c), place the test specimen in Figure 5 a) over the test specimen in Figure 5 b), with no space between them.

Key

1 groove

Figure 5 — Test specimen (machining process required when using two test specimens) (example)

6.3.2 When using a single test specimen

When using a single test specimen, the thickness of the specimen shall be restricted as described in 6.2.

As shown in Figure 6, cut grooves about 3 mm wide and 1,5 mm deep at the centre of the top and bottom surfaces so that the grooves run across the specimen. In addition, drill a hole having a diameter of about 2,5 mm toward the centre perpendicular to the side surface from the centre of the thickness direction on the side surface. In Figure 6, the length of the hole is about 60 mm to 65 mm.

Dimensions in millimetres
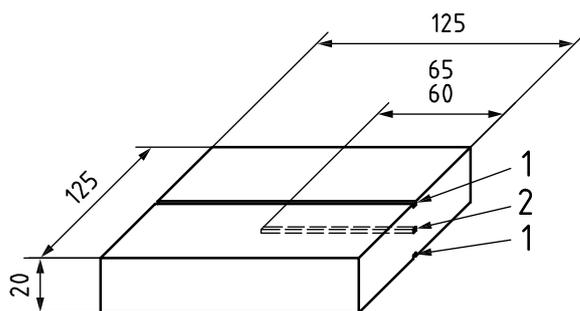
Key

1   groove

2   hole

Figure 6 — Test specimen (machining process required when using a single test specimen) (example)

7   Apparatus

7.1   General

An example of the measurement device is shown in Figure 7. This device is composed of three units, which are periodic heater (3), low-temperature heater (5) with water cooling pump, and atmospheric furnace (2).

The standard size of the periodic heater shall be a 125 mm × 125 mm square. However, this shall not apply if it is confirmed that the device can ensure the accuracy stipulated in this document.
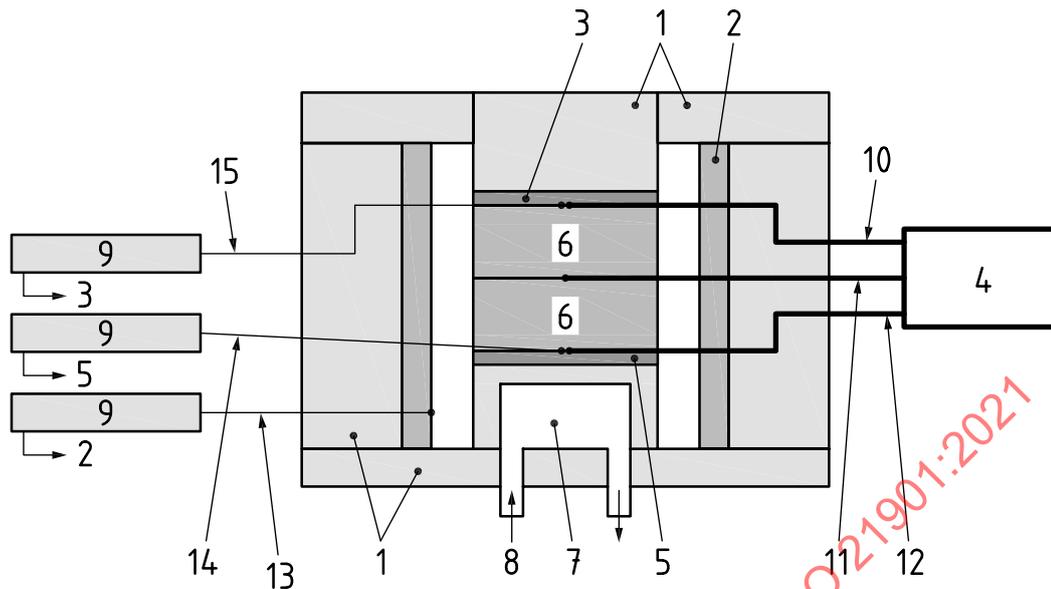
Key

1	thermal insulation	9	temperature controller
2	atmospheric furnace	10	thermocouple for measuring temperature at high-temperature surface
3	periodic heater	11	thermocouple for measuring temperature inside specimen
4	recorder	12	thermocouple for measuring temperature at low-temperature surface
5	low-temperature heater	13	thermocouple for controlling temperature of atmospheric furnace
6	specimen	14	thermocouple for controlling temperature of low-temperature heater
7	water cooling tank	15	thermocouple for controlling temperature of periodic heater
8	circulating water		

Figure 7 — Details of measurement device (example)

7.2 Temperature measurement instrument

The temperature measurement instrument to be used for measurement shall meet the following requirements.

The calibrated thermocouples with a diameter of 0,5 mm or less shall be used for temperature measurement.

8 Procedure

8.1 Rounding numbers

Measured and calculated values shall be rounded off to the specified digit.

8.2 Period of temperature change

In principle, the period of temperature change shall be set to 3 600 s.

Depending on L/d or thermal diffusivity, it is possible to use a shorter period of temperature change than 3 600 s. When a different period is to be used, refer to Annex A.

8.3   Amplitude of temperature change

The amplitude of periodic temperature change shall be approximately 3 °C to 5 °C on the heating surface. Refer to B.1 about amplitude of temperature change.

8.4   Measurement method

Measurement shall be performed according to the following procedure. Refer to B.2 about propagation of periodic temperature change.

a)   To measure the temperature on the surface and inside the test specimen, thermocouples as prescribed in 7.1 shall be set on the surfaces of the test specimen and inside the specimen.

b)   Figure 8 shows the locations of the thermocouples: substantially at the centre of the specimen surface ($x = d$), substantially at the centre of the interface between the upper and lower specimens ($x = x_m$) and substantially at the centre of the low-temperature surface ($x = 0$). In this case, the relative positions of the thermocouples (temperature sensors) on the surface and inside the specimen shall be aligned within the radius of 1,5 mm to the normal line AB at the centre of the test specimen.

c)   Inside the specimen refers to the tip of the hole drilled from the side surface to the centre of the specimen (see Figure 6) when using a single test specimen, and the centre of the interface of the two specimens when using two specimens (see Figure 5).

d)   The relative distances (x_m and $d – x_m$ in Figure 8) between the three temperature sensors set on the surfaces (the heating surface and the low-temperature surface) and inside the specimen shall be measured to within 0,1 mm.

e)   The ambient temperature is controlled by the atmospheric furnace and is shown in Figure 7 (key 13). The atmospheric furnace shall stabilize the ambient temperature within the temperature range of about ±1 K.

f)   After the temperatures at all positions become sufficiently stable, that is, when the temperature change is within about 0,5 K per hour, periodic heat with the period f is applied to the surface of the test specimen.

g)   The temperature changes at the three positions $x = d$, $x = x_m$, and $x = 0$ are measured at a constant interval. The temperature digits shall be measured up to 1 °C. The time interval shall be selected so as to include more than 100 measurement points in one period.

h)   The phase lags shall be obtained from the phase difference between the measured temperatures at positions $x = x_m$, and $x = d$. Therefore, the temperature changes at each x position shall be measured so as to be reproduced a sine curve. In order to obtain the sine curve, the temperature at each x position shall be measured with an optimum resolution (e.g. 0,1 °C or 0,01 °C).

i)   Thermal diffusivities are calculated from three or more phase lags. After averaging these values, the intended mean thermal diffusivity is obtained.

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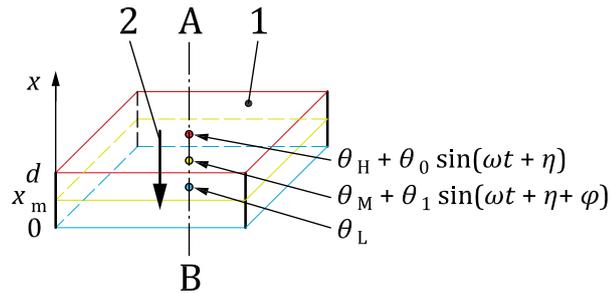
1   test specimen

2   heat flow

The three temperature sensors (thermocouples) shall be set within a radius of 1,5 mm along the normal line AB positioned at the centre of each surface.

Figure 8 — Relative positional relation between thermocouples (temperature sensors) set on and in test specimen

8.5   Calculation of thermal diffusivity

Obtain the damping coefficient k by using Formula (1) with the phase lag obtained by measurement, and calculate thermal diffusivity a by Formula (11).

$$a = \frac{\omega}{2k^2} \tag{11}$$

8.6   Calculation of thermal conductivity

When thermal conductivity λ is desired, calculate with Formula (12) using the thermal diffusivity a and specific heat capacity c.

$$\lambda = \rho c a \tag{12}$$

9   Test report

The test report shall include the following items:

a)   International Standard used (including its year of publication);

b)   frequency (1/s) or period (s);

c)   mean temperature of a test specimen (°C);

d)   details of test specimen:

— name of material;

— dimensions and thickness (mm);

— dry mass (kg);

— bulk density (kg/m^3);

e)   thermal diffusivity (m^2/s).

Annex A
(normative)

Size of test specimen and period of temperature change

A.1 General

The ratio of side length L to thickness d of the specimen (L/d) is restricted so as to reduce the error due to heat inflow (outflow) from the edges of the specimen. In addition, in some cases, it is advisable to change the period of temperature change, depending on the L/d or thermal diffusivity of the specimen.

As reference, this annex presents the results of calculations of the difference between nominal thermal diffusivity and true thermal diffusivity (input value) when L/d is changed, and the difference between the true thermal diffusivity (input value) and the nominal thermal diffusivity on the period of temperature change.

Thermal diffusivity was calculated by Formula (1) using the known temperatures of a heated surface and a heat dissipation surface (set temperatures) and the temperature in the specimen centre calculated by CAE. The conditions of the calculation were as follows.

— An unsteady heat transfer analysis was performed using the finite volume method of thermal fluid analysis CAE software SCRYU/Tetra (Software Cradle Co., Ltd.).

— The analysis region was a rectangular solid specimen, and the heater and other parts were excluded from the analysis region. The boundary surfaces were three types, namely, the heated surface (specimen top side), the heat dissipation surface (specimen bottom side) and other surfaces.

— The in-plane temperature of the heated surface was changed over time at a rate of $(410 + 5 \sin(\omega t))$ °C.

— The in-plane temperature of the heat dissipation surface was fixed at 390 °C.

— At the side surfaces, the heat transfer condition of a representative temperature 400 °C and heat transfer coefficient of 100 W/(m²K) was assumed.

A.2 Size of test specimen

Table A.1 shows the difference (in percentage) between the true thermal diffusivity (input value) and the nominal thermal diffusivity (calculation results) when L/d of the test specimen was changed. Formula (A.1) was used to calculate this difference.

$$D = \left(a_n - a_t\right)/a_t \times 100 \tag{A.1}$$

where, a_t is true thermal diffusivity (input value) and a_n is nominal thermal diffusivity (calculation result).

Table A.1 — Difference of calculation results of thermal diffusivity on *L/d* of test specimen (simulation results)

a	f	ρ	c	L/d	D [%]		
m²/s	s	kg/m³	J/(kg·K)		d=20 mm	d=40 mm	d=50 mm
$5{,}0\times10^{-7}$	3 600	200	1 000	2	25	24	22
				4	2	1	1
				6	0	0	0
				8	0	0	0

A.3 Period of temperature change

Tables A.2 to A.4 show the difference between the true thermal diffusivity (input value) and the nominal thermal diffusivity (calculation value) on the period of temperature change.

Table A.2 — Difference between calculation results and input values of thermal diffusivity on the period of temperature change (simulation results [*d* = 20 mm])

a	ρ	c	L/d	d=20 mm					
				D [%]					
m²/s	kg/m³	J/(kg·K)		f=900s	f=1 800s	f=2 700 s	f=3 600 s	f=4 500 s	f=5 400 s
$1{,}0\times10^{-7}$	200	1 000	2	4	16	22	24	25	26
			4	0	0	1	1	1	2
			6	0	0	0	0	0	0
			8	0	0	0	0	0	0
$5{,}0\times10^{-7}$	200	1 000	2	23	24	25	25	25	25
			4	1	2	2	2	2	2
			6	0	0	0	0	0	0
			8	0	0	0	0	0	0
$1{,}0\times10^{-6}$	200	1 000	2	22	22	22	22	22	22
			4	2	2	2	2	2	2
			6	0	0	0	0	0	0
			8	0	0	0	0	0	0
$5{,}0\times10^{-6}$	200	1 000	2	12	12	12	12	12	12
			4	1	1	1	1	1	1
			6	0	0	0	0	0	0
			8	0	0	0	0	0	0

Table A.3 — Difference between calculation results and input values of thermal diffusivity on the period of temperature change (simulation results [*d* = 40 mm])

a	ρ	c	L/d	d=40 mm					
				D [%]					
m²/s	kg/m³	J/(kg·K)		f=900 s	f=1 800 s	f=2 700 s	f=3 600 s	f=4 500 s	f=5 400 s
$1{,}0\times10^{-7}$	200	1 000	2	0	0	1	4	8	11
			4	1	0	0	0	0	0
			6	1	0	0	0	0	0
			8	1	0	0	0	0	0