5.5. Calibration and Test Procedures

- List of Calibration Procedures

1. C-18-010-2000, Calibration procedure of melting point measuring apparatus
2. C-18-011-2000, Calibration procedure of calorimeter
3. T-04-001-2000, Test procedure of thermal conductivity of thermal insulation by guarded hot plate
4. T-04-002-2000, Test procedure of linear thermal expansion of fine ceramics by thermomechanical analysis
5. T-04-003-2000, Test procedure of linear thermal expansion and glass transition temperature of plastics by thermomechanical analysis
6. T-04-004-2000, Test procedure of thermal diffusivity for ceramics by pulse laser method*
7. T-04-005-2000, Test procedure of thermal conductivity of firebricks by hot wire method (Cross-array)
8. T-04-006-2000, Test procedure of thermal conductivity of firebricks by hot wire method(Parallel)
9. T-04-007-2000, Test procedure of transition temperature of plastics
10. T-04-008-2000, Test procedure of heat of fusion of plastics

*:written in English

- List of Attached Files

1. T-04-004-2000, Test procedure of thermal diffusivity for ceramics by pulse laser method

Contents

AMENDMENT RECORD			
Amended Item (or Section)	Version	Date	Contents
	1(E)	2000/1/1	English version of C-04-004-2000

Drafted by	Reviewed by	Authorized by
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1. Scope

This standard specifies the test method for the determination of thermal diffusivity from room temperature to 1700 K by the laser flash method for homogeneous monolithic ceramics with porosity less than 10%.

2. Normative References

The following normative documents contain provisions which, through reference in this text, constitute provisions of this part of the ISO/IEC Directives. For dated references, subsequent amendments to, of revisions of, any of these publications do not apply. However, parties to agreements based on part of the ISO/IEC Directives are encouraged to investigate the possibility of applying the most recent editions of the mormative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 468: 1982, Surface roughness – Parameters, their values and general rules for specifying requirements.

ISO 3611: 1978, Micrometer calipers for external measurement.

3. Term and Definition

For the purpose of this Standard, the following principal definitions apply.

3.1

thermal diffusivity

thermal conductivity divided by heat capacity per unit volume.

3.2

thermal conductivity

density of heat flow rate divide by temperature gradient under steady state condition.

3.3

specific heat capacity

the heat capacity per unit mass.

3.4

pulse width(τ_p)

the pulse width is defined by the full width of half maximum (FWHM) which is the time duration when the laser pulse intensity is larger than the half of its maximum value on time scale.

3.5

centroid of laser pulse

chronological centroid of laser light energy.

3.6

spatial energy distribution of pulse heating

spatial energy distribution of laser beam is define by the energy density of the laser beam incident at each point on front face of the specimen.

3.7

transient temperature curve

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

3.8

transent radiance curve

the transient change of the spectral radiance from the rear face of the specimen after the light pulse heating.

It should be noted that the observed transient curve is proportional to the change of the spectral radiance instead of the temperature when a radiation thermometer of a radiation detector is used to observe the transient temperature rise of the specimen after the light pulse heating.

3.9

maximum temperature rise(ΔT_{max})

the difference between the steady temperature before the pulse heating and the maximum temperature of the rear face of the specimen the pulse heating (see figrue1).

3.10

half rise time($t_{1/2}$)

that time until ΔTmax/2 is attained, with the centroid position of pulse being as the origin of time axis.

3.11

characteristic time of heat loss

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

3.12

extrapolated temperature rise(ΔT_0)

the extrapolated temperature rise, ΔT_0 , is determined when the cooling region is fitted with an exponential function, $\Delta T_0 \exp(-t/\tau_c)$

3.13

adiabatic temperature rise(ΔT)

the ideal temperature rise with no heat losses.

NOTE The adiabatic temperature "ΔT" is generally larger than the extrapolated temperature rise "ΔT_0".

3.14

stray light superimposed on transient temperature curve

initial spike and/or hump superimposed on the initial part of curve due to transmitted and/or scattered light from the heating laser pulse.

3.15

homogeneity of specimen

degree of homogeneity of local thermal diffusivity over the specimen.

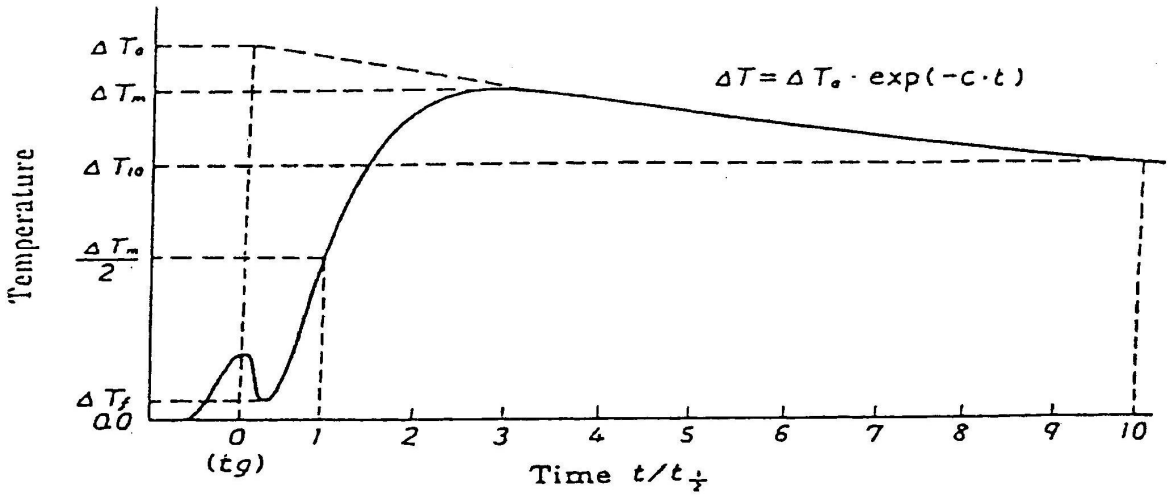

Figure 1 - Transient temperature curve of the rear face of the specimen after a light pulse heating onto the front face of the specimen

4. Apparatus

The apparatus shall be those for obtaining the thermal diffusivity from the temperature rise curve of the rear face of a specimen after the laser pulse is irradiated onto the front face of the specimen, and shall consist of the following components as shown in figure 2.

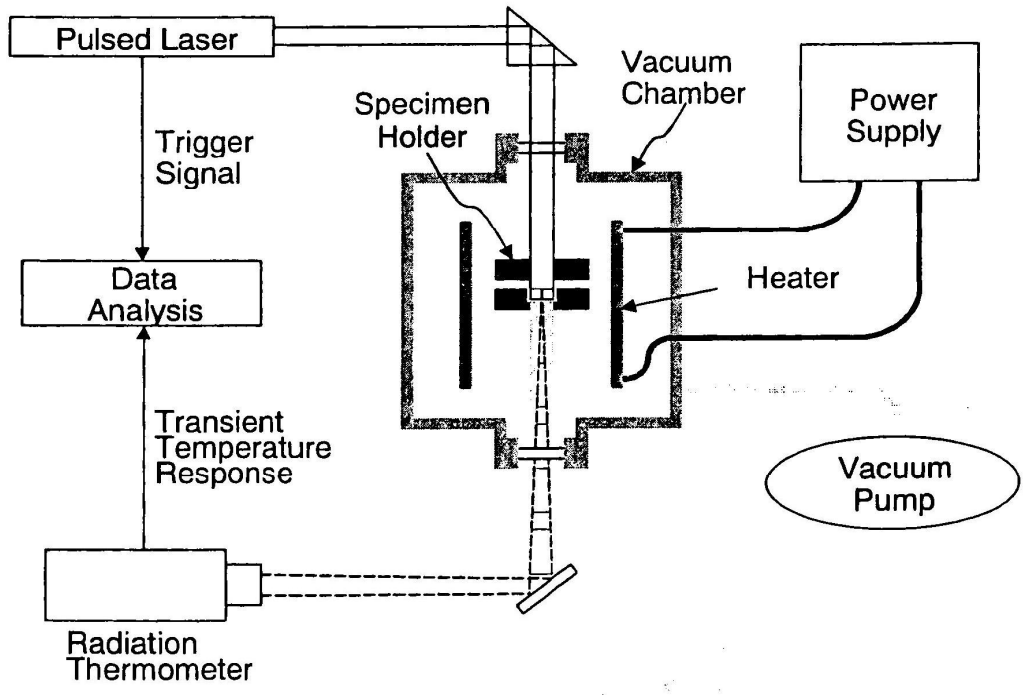

Figure2 - Block diagram of laser flash apparatus for measuring thermal diffusivity

4.1 Specimen holder

The specimen holder shall hold the specimen stable with minimum thermal contact and shall designed suppress stray lights from the laser beam.

NOTE A diaphragm with aperture diameter of 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.

4.2 Pulse laser

The pulse laser shall be capable of emitting the light pulse with pulse duration shorter than 0.5 ms in full width at half maximum (FWHM).

NOTE A pulse laser should be operated under normal oscillation. A Q-switch laser should not be used except for measurements of specimens thinner than 0,1 mm

The specimen should be irradiated uniformly by the light pulse. Evaluation methods to measure energy distribution of a light pulse over the area corresponding to the specimen surface are described in C.1.1 in annex C.

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

4.3 Thermometer for measuring steady state temperature of the specimen

The steady state temperature of the specimen before pulse heating shall be measured by a thermocouple contact with the specimen. The thermocouple shall be set without interrupting the light pulse heating onto the front face of the specimen of the radiation from the rear face of the specimen. If the thermocouple junction cannot contact with the specimen because of chemical reaction with the specimen or inconvenience for setting the specimen, or the system design, the calibration table between the specimen temperature and the thermocouple junction temperature should be presented with information about reproducibility of the calibration.

4.4 Thermometer for measuring transient temperature rise of the specimen rear face

The transient temperature rise curve on the rear face of the specimen shall be observed noncontactly by a radiation thermometer of a radiation detector. The frequency response of the detection should be faster than 10 kHz. The target diameter of the radiation detection should be smaller than 50% of the specimen diameter.

NOTE Evaluation methods of frequency response and target size of a radiation thermometer of a radiation detector are described in C.4.1.

4.5 Environment for measurements

Measurements can be performed under open air, under inert gas atmosphere, or under vacuum from room temperature to 400K. Measurements above 400K should be made under vacuum better than 5×10^{-3} Pa or under inert gas without oxygen impurity.

4.6 Temperature control unit

For higher temperature measurements, the specimen should be kept at stable temperature by electric heaters before pulse heating. Drift of the temperature should be smaller than 1% of the maximum temperature rise "ΔT_{max}" over the half rise time "$t_{1/2}$".

4.7 Data acquisition

The signal from the radiation thermometer or detector should be subtracted by the DC level before the pulse heating and differentially amplified. The amplified signal should be converted to the digital signal using a digital oscilloscope of an AD converter. The AD converted signal is transferred to a personal computer. 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 1000 data points should be sampled with the sampling time faster than 1% of the half rise time "$t_{1/2}$".

5. Specimen

5.1 Shape and dimension of specimens

The specimen shall be a flat plate of circular, square or rectangular shape. specimen diameter or side length should be from 5 mm to 20 mm. Specimen thickness should be from 0.5 mm to 10 mm. The thickness to diameter ratio should be smaller than on half. The specimen thickness shall be such that the half rise time of specimen is between $10\tau_p$ and 100 ms. The parallelism of the front and rear faces shall be smaller than 0.5 % of the thickness, and the roughness of front and rear faces shall be smaller than 0.40 micro m Ra. (see ISO 468).

5.2 Coating on the specimen

If the specimen does not have high absorption coefficient against the heating laser beam of high emissivity for radiative temperature detection, the surfaces of the specimen must be coated with black thin layer. The coating must be dense enough to prevent penetration of the laser beam of thermal radiation at the observed wavelength, resistive against laser pulse heating at high temperatures. The thickness of the coatings should be thin enough lest the heat diffusion time from the front face to the rear face of the specimen should not be increased by thermal resistance of the coatings.

5.3 Reference specimen

Reference specimens can be used to evaluate uncertainty of thermal diffusivity measurements by a laser flash apparatus. The uncertainty is obtained as the difference between the measured value and the reference value of thermal diffusivity of the reference specimen. The materials which can be used as reference specimens are described in annex B.

6. Measurement procedure

6.1 Determination of chronolgical centroid of laser pulse

The chronological centroid of the laser pulse (t_g) shall be determined by one of the following procedures:

6.1.1 Real time method

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

6.1.2 Integration method

Prepare a metallic sheet thin enough across which the heat diffusion time is shorter than 3 μs. Then, 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 pulse. The waveform of the laser pulseis derived as the derivative of the chronological trace of the temperature rise.

6.2 Measurement of specimen

6.2.1 measurement of the specimen thickness

Measure the thickness of specimen by a micrometer to the unit of 1 ㎛ (see ISO 3611).

6.2.2 Surface treatment

Carry out the surface treatment in accordance with 5.2

6.2.3 Temperature and atmosphere control

Prepare the atmosphere in which the specimen is not subjected to chemical change under the measurement temperature range.

6.2.4 Stability of specimen temperature

6.2.5 Energy of pulse heating

Irradiate the laser pulse at such intensity that the maximum temperature rise ΔT_{max} of the specimen does not exceed 5K.

6.2.6 Record

Transient temperature curve should be recorded at least until 10 times of the half rise time in order to make reliable evaluation of measurements including heat loss correction and evaluation of nonuniform heating effect.

6.3 Measurements of apparent thermal diffusivity valules 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 nonlinearity of Planck's equation and temperature dependence of thermal diffusivity of the specimen. Generally, the apparent thermal diffusivity changes as a function of temperature most sensitivity at the lowest measurement temperature. Thus, it is recommened that laser flash measurements should be made under different levels of the laser beam energy.

6.4 Measurement of apparent thermal diffusivity values dependent of specimen thickness

In order to evaluate the characteristics of equipment such as the non-uniformity of pulses and centroid, measure the temperature rise curves of several standard specimens different in thickness at the room temperature, in accordance with 6.3.

6.5 Measurement of reference specimen

When the characteristics of laser pulse are judged as unchangeable, the same temperature rise curve as that in the preceding measurement may be allowed to substitute.

The one that has been processed to nearly the same shape (thickness and radius) as the specimen shall be included.

7 Data analysis

7.1 Half rise time method

The standard algorithm to calculate thermal diffusivity from the laser flash method is the half rise time method and thermal diffusivity, α, is represented by the following equation:

$$\alpha = \frac{0.13888 d^2}{t_{1/2}} \qquad (1)$$

where $t_{1/2}$ is the time delay when the temperature of the rear face reaches one half of the maximum temperature increase, ΔT_{max}, after the front face was heated by the laser

pulse. If measurements are made under the ideal condition with no heat losses under uniform heating, ΔT_{max} is equal to ΔT .

When the ideal conditions are not satisfied in real measurements, the thermal diffusivity shall be calculated from following equation (2) with introducing correction factors considering deviation

from the ideal initial and boundary conditions.

$$\alpha = 0.1388 k_1 k_2 (d+e)^2 / t_{1/2} \qquad (2)$$

where

α is the thermal diffusivity(m^2/s)

d: thickness of specimen at room temperature (m)

k1: correction factor relating to unevenness of pulse

k2: correction factor relating to heat loss form the specimen

e: thermal expansion in thickness direction of specimen caused by temperature change from room temperature to test temperature (m)

7.2 Logarithmic method

The thermal diffusivity shall be calculated from following equation (3) based on the logarithmic method.

$$\alpha = -\frac{(d+e)^2}{4h} \qquad (3)$$

where h is the inclination of strength line obtained when $\ln(\sqrt{t} \cdot \Delta T)$ 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).

7.3 Regression analysis methods

An observed temperature response curve after pulse heating is fitted with the analytical solution of the thermal diffusion equation under the initial and boundary conditions corresponding to the measurement condition.

8. Report

The following information should be recorded in the measurement report:

a) General information

1) Date and time of measurements
2) Organization where the measurements were made
3) The name of the laser flash apparatus used
4) The operator who made made measurement
5) Room temperature when the measurement was made
6) Measurement number in successive measurements

b) Light pulse

1) Type of the pulse light source
2) Duration of the light pulse in full width at half maximum
3) Wavelength of the light pulse
4) Energy of one light pulse
5) Charging voltage for the flash lamp of the pulse laser

6) Spatial profile of the light pulse

c) Measured specimen

1) Material

2) Manufacturer of the material:

3) Grade of the material

4) Name of the specimen

5) Thickness of the specimen

6) Shape of the specimen (disk, square plate or rectangular plate)

7) Diameter or side length of the specimen

8) Weight of the specimen before and after the series of measurements

9) Color of the specimen before and after the series of measurements

10) Appearance of the specimen before and after the series of measurements

d) Coating

1) Use of coating (Yes or No)

2) Coated material

3) Coating procedure:

4) Thickness with coating before and after the series of measurements

5) Weight with coating before and after the series of measurements

e) Thermometry

1) Thermometer used for steady state temperature measurement

2) Thermometer used for measuring transient temperature rise of the specimen rear face after light pulse heating

3) Temperature scale of the radiation thermometer (Yes or No)

4) If Yes, calibration procedure

f) Data acquisition

1) Response time of the transient temperature measurements:

2) Shot add. Of the laser pulse

3) Sampling clock period

4) Resolution of the DA converter

5) Data Count

j) Data analysis

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

2) Data analysis algorithm (half rise time method, logarithmic method or regression analysis method)

3) Type of corrected items and corrected amount

g) Measured results

1) Steady temperature of the specimen

2) The maximum temperature rise of the specimen

3) The effective temperature of the specimen

4) The half rise time

5) The characteristic time of heat loss

6) Thermal diffusivity determined by the half rise time method under them ideal condition

7) The final thermal diffusivity with corrections for real measurement

8) Difference of the final thermal diffusivity with corrections for real measurement conditions from that alculated by the half rise time method under the ideal condition

9) Biot number

i) Other important information

5.6. Typical Certificate of Calibration

시 험 성 적 서

TEST REPORT

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page of pages

① 의뢰기관 Applicant	ABC		② 시험번호 Test No.	01-00000-02
③ 시험대상 Test Item	시험품명 Description	Glassy Carbon		
	제작회사 및 형식 Manufacturer & Model	DEF	기 기 번 호 Serial No.	
④ 접수일자 Date of Receipt	2001. 6. 29	⑤ 시험일자 Test Date: 2001. 7.12	⑥ 시험장소 Test Site	Standard Lab.
⑦ 시험환경 Environment	온 도 Temperature	(22 ± 2) °C	상대습도 Relative Humidity	below 55 % R.H.
⑧ 시험방법 Test Method	KS L 1604, Pulse laser method	⑨ 담당자 Tested by	042-868-5191, Jong-Chul Kim	

⑩ 시험결과 (Test Results)

The overall measurement uncertainty is estimated to be within 6 %, based on a uncertainty multiplied by a coverage factor k=2, providing a level of confidence of approximately 95 %.

Temperature (°C)	Thermal diffusivity (x 10^{-6} m^2/s)
24.8	6.031
203.0	5.157
420.1	4.640
661.1	4.275
781.3	4.156
967.8	3.993
1210.0	3.984

The end

o 위 내용은 의뢰자가 제공한 시험품의 결과이며, 시료명은 의뢰자가 제공한 것임.
o 시험결과는 상기 시험품에 한하여 유효하며, 한국표준과학연구원장 승인 없이 수정 또는 재 발행 할 수 없음.

2001년 월 일

시험책임자(Approved by) : Temperature-Humidity Group Leader Hyun-Soo Nham

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5.7. Typical Best Measurement Capability

Best measurement capability of measurement of thermal diffusivity by pulse laser system

In the this report, we considered only uncertainty budgets from a pulse laser system used in the glassy carbon.

1. Measurement model

$$\alpha = 0.1388\frac{d^2}{t_{1/2}} \quad (1)$$

where, α : thermal diffusivity $(m^2 \cdot s^{-1})$

d : thickness of specimen(m)

$t_{1/2}$: temperature to reach 50 % of maximum value

2. Components of uncertainty

Type A

a. Scatter of thermal diffusivity(U_{sd})

Type B

a. Thickness of specimen(U_{st})

b. Micrometer(U_{om})

c. Measurement of temperature(U_{tm})

d. Nonlinear effect of detector(U_{dn})

e. Temperature drift of furnace(U_{ft})

f. Heat loss effect(U_{he})

g. Nonunifoam heat of pulse laser(U_{pn})

h. Time scale(U_{ts})

i. Certified reference material(U_{crm})

1) Uncertainty of Type A

a) Scatter of thermal diffusivity(U_{sd})

$$U_s = s/\sqrt{n} \quad (2)$$

$$U_{sd} = 0.46\%$$

Where s : standard deviation, n : weighing number

Table 1 Test results of thermal diffusivity

Measurement point (℃)	Temperature of specimen (℃)			Thermal diffusivity $(\times 10^{-6} m^2 s^{-1})$		
	Measurement value	Mean	Standard deviation	Measurement value	Mean	Standard deviation
20	24.9 24.8 24.8	24.8	0.057	6.038 6.031 6.025	6.031	0.007
200	202.9 203.0 203.1	203.0	0.100	5.149 5.171 5.171	5.157	0.012
400	420.1 420.0 420.1	420.1	0.058	4.643 4.628 4.649	4.640	0.011
600	661.1 661.2 661.1	661.1	0.058	4.297 4.267 4.260	4.275	0.020
800	781.1 781.5 781.2	781.3	0.208	4.141 4.165 4.163	4.156	0.013
1000	967.9 967.7 967.7	967.8	0.115	4.010 3.986 3.983	3.993	0.015
1200	1210.3 1209.9 1209.8	1210.0	0.265	3.992 3.988 3.971	3.984	0.011

2) Uncertainty of Type B

Table 2 Uncertainty of Type B

Components	Uncertainty (%)
(U_{om})	0.1
(U_{st})	0.5
(U_{tm})	0.5
(U_{dn})	1.0
(U_{ft})	0.5
(U_{he})	1.0
(U_{pn})	1.0
(U_{ts})	0.5
(U_{crm})	2.0

3. Combined uncertainty

1) Combined uncertainty of Type A

$$U_{c,A} = 0.46\ \%$$

2) Combined uncertainty of Type B

$$U_{c,B} = \sqrt{(U_{om}{}^2 + U_{st}{}^2 + U_{tm}{}^2 + U_{dn}{}^2 + U_{ft}{}^2 + U_{he}{}^2 + U_{pn}{}^2 + U_{ts}{}^2 + U_{crm}{}^2)} \quad (3)$$

$$= \sqrt{(0.1^2 + 0.5^2 + 0.5^2 + 1^2 + 0.5^2 + 1^2 + 1^2 + 0.5^2 + 2^2)}$$

$$= 2.83\ \%$$

3) Combined standard uncertainty

$$U_c = \sqrt{(U_{c,A}{}^2 + U_{c,B}{}^2)} \quad (4)$$

$$= \sqrt{(0.46^2 + 2.83^2)}$$

$$= 2.87\ \%$$

4. Expanded uncertainty

$$U_{95} = 2 \times 2.87$$

$$= 5.74\ \%$$