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Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 4: Laser flash method

Plastiques — Détermination de la conductivité thermique et de la diffusivité thermique —

Partie 4: Méthode flash laser

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Contents

Page

Foreword

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

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 22007-4 was prepared by Technical Committee ISO/TC 61, *Plastics*, Subcommittee SC 5, *Physical-chemical properties*.

ISO 22007 consists of the following parts, under the general title *Plastics — Determination of thermal conductivity and thermal diffusivity*:

— *Part 1: General principles*

— *Part 2: Transient plane heat source (hot disc) method*

— *Part 3: Temperature wave analysis method*

— *Part 4: Laser flash method*

Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 4: Laser flash method

1 Scope

1.1 This part of ISO 22007 specifies a method for the determination of the thermal diffusivity of a thin solid disc of plastics in the thickness direction by the laser flash method. This method is based upon the measurement of the temperature rise at the rear face of the thin-disc specimen produced by a short energy pulse on the front face.

1.2 The method can be used for homogeneous solid plastics as well as composites having an isotropic or orthotropic structure. In general, it covers materials having a thermal diffusivity, α, in the range $1 \times 10^{-7}\ m^2 \cdot s^{-1} < \alpha < 1 \times 10^{-4}\ m^2 \cdot s^{-1}$. Measurements can be carried out in gaseous and vacuum environments over a temperature range from − 100 °C to + 400 °C.

NOTE For inhomogeneous specimens, the measured values may be specimen thickness dependent.

2 Normative references

The following referenced documents are indispensable for the application 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 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 22007-1, *Plastics — Determination of thermal conductivity and thermal diffusivity — Part 1: General principles*

ISO/IEC Guide 98-3, *Uncertainty of measurement — Part 3: Guide to the expression of uncertainty in measurement (GUM:1995)*

3 Terms and definitions

For the purpose of this document, the terms and definitions given in ISO 22007-1 and the following apply.

3.1
pulse width
t_p
time duration for which the laser pulse intensity is larger than half of its maximum value

NOTE It is expressed in seconds (s).

3.2
time origin
t_0
start of the laser pulse

NOTE It is expressed in seconds (s).

3.3
maximum temperature rise
ΔT_{max}
difference between the maximum temperature reached by the rear face of the specimen after the laser pulse has passed and its steady temperature before the pulse

NOTE It is expressed in kelvins (K).

3.4
half-rise time
$t_{1/2}$
time from the time origin until the rear-face temperature increases by one-half of ΔT_{max}

NOTE It is expressed in seconds (s).

3.5
thermogram
temperature versus time curve for the rear face of the specimen

3.6
thickness
d
dimension of the test specimen in the direction of heat transfer measurement

NOTE It is expressed in metres (m).

4 Principle

One side of a flat-sheet test specimen is subjected to an energy pulse which has a very short duration compared with the half-rise time (see 6.1) and a uniform spatial energy distribution. The transient temperature rise on the opposite face (rear face) is recorded as a function of time (see Figure 1). The thermal diffusivity is obtained by comparing the experimental thermogram with a theoretical model (see Clause 9 and Annex B).

5 Apparatus

5.1 General

The apparatus shall be designed to obtain the thermal diffusivity as described in Clause 4 and shall consist of the following main components as shown in Figure 2. These are the furnace or climatic chamber with a specimen holder and temperature measurement device (e.g. thermocouple), the flash source (e.g. laser), the pulse detector, the transient detector (IR detector) and the control, data acquisition and analysis unit.

5.2 Furnace or climatic chamber

The furnace or climatic chamber shall meet the following requirements:

a) The temperature range shall be appropriate to the range of materials to be studied. Depending on the range of temperature, the specimen is maintained at a constant temperature by a cryostat or by a furnace.

b) It shall be capable of maintaining the test temperature constant to within ± 0,5 K or less for at least 30 min.

c) The temperature measurement device shall be capable of measuring the furnace temperature with a resolution of ± 0,1 K and an accuracy of ± 0,5 K or better.

d) The furnace shall be fitted with two windows, one transparent to the pulse radiation and the other transparent to the working wavelength range of the IR detector.

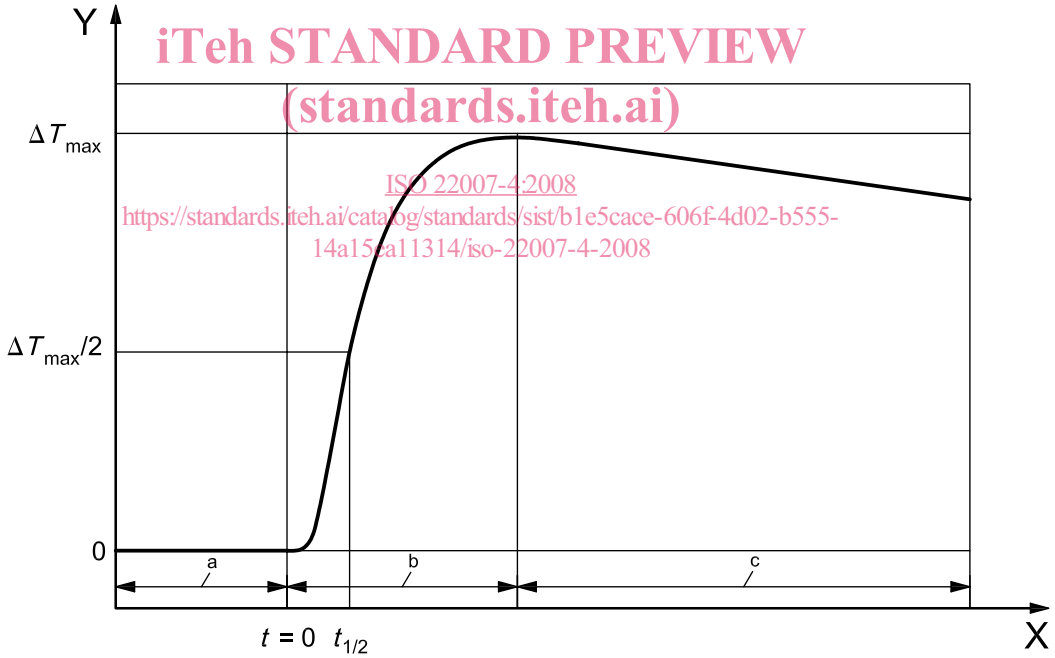
e) If required, the environment in the furnace shall be a vacuum or an inert-gas atmosphere to avoid oxidative degradation during heating and testing of the specimen. For cryoscopic measurements, care shall be taken to avoid water condensation on the windows.

NOTE Measurement under vacuum will eliminate convection effects.

The specimen holder shall be designed to minimize thermal contact with the specimen and to suppress stray light transmitted from the laser beam to the IR detector.

The test temperature shall be measured using a calibrated temperature measurement device that is preferably in contact with the specimen or the specimen holder but at least within 1 mm of the specimen holder.

The temperature measurement device shall be designed so as not to significantly disturb the temperature field generated in the specimen by the laser pulse.

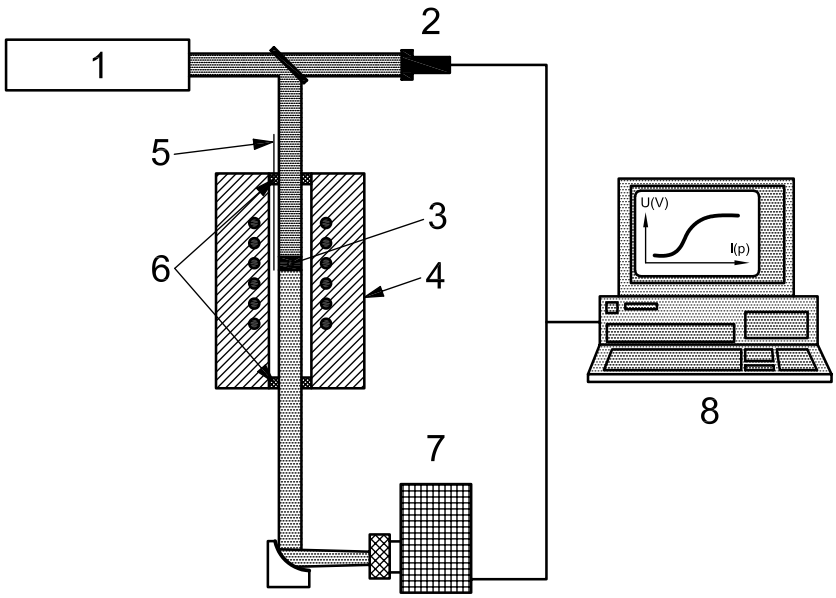


Key

X time
Y temperature rise

a Baseline.
b Transient-rise period.
c Cooling period.

Figure 1 — Example of thermogram



Key

1 flash source
2 pulse detector
3 specimen
4 furnace or climatic chamber
5 temperature measurement device
6 windows
7 transient detector
8 control, acquisition and analysis unit

Figure 2 — Schematic diagram of laser flash set-up for measuring thermal diffusivity

5.3 Flash source

The energy level of the flash source shall produce a temperature rise not exceeding 3 K at the rear face of the specimen.

The spatial energy distribution of the pulse heating shall be as uniform as possible over the front face of the specimen.

The pulse duration shall be shorter than 1 ms.

The heat pulse source may be a laser (preferably) or a flash tube.

A photodiode can be used to determine the duration and form of the pulse and the time origin.

5.4 Transient detectors

The transient temperature rise at the rear face of the specimen shall be measured with an IR detector. The transient detector shall be able to detect a variation of 5 mK in the specimen rear face temperature. Its response shall be linear with temperature over a temperature range of at least 3 K.

The frequency response of the detector and its associated electronics (amplifiers, analogue/digital converters, etc.) shall be faster than 10 kHz. If electronic filters are used, they shall meet the requirements defined above and shall not decrease the accuracy of temperature measurement, otherwise they could distort the shape of the temperature-time curve.

NOTE The choice of IR detector depends also on the temperature range. For the range − 100 °C to + 400 °C, photovoltaic or photoconductor detectors can be used.

The temperature of the rear face, or a quantity directly proportional to it (e.g. voltage), shall be measured and recorded continuously over the duration of the test. The data acquisition system, which may be analogue or digital, shall be able to sample more than 1 000 data points on the thermogram with a sampling frequency higher than $100/t_{1/2}$. The accuracy of the time base shall be better than $\pm 1 \times 10^{-5}$ s.

5.5 Thickness measurement device

The specimen thickness shall be measured with an accuracy of ± 5 µm by a calibrated thickness measurement device having a resolution of ± 1 µm. For soft materials, a micrometer with reproducibly low compression is required.

6 Test specimen

6.1 Shape and dimension of the specimen

The specimen shall be a thin disc. The specimen diameter is usually from 5 mm to 20 mm. The specimen thickness shall be chosen according to the pulse width and the thermal diffusivity of the material. It shall be selected such that the pulse width is less than 0,01 of the half-rise time. Typically, the thickness will be between 0,5 mm and 3 mm. The aspect ratio of the specimen shall be chosen such that 2D effects are negligible during the test. The ratio of the diameter to the thickness shall be larger than 3:1.

The faces shall be flat and parallel. Any variation in the thickness of the specimen should preferably be less than 1 % of the mean thickness. The effect of greater non-uniformity can be estimated in the measurement uncertainty.

6.2 Preparation and conditioning of test specimen

The test specimen shall be representative of the material being examined and shall be prepared and handled with care. If the specimen is taken from sample pieces by cutting, care shall be taken to prevent heating, changes in molecular orientation or any other effect that may alter the sample properties.

The test specimen shall be conditioned prior to the measurement as specified in the relevant material standard or by a method agreed between the parties involved. Unless other conditions are specified, it is recommended that the specimen be conditioned in accordance with ISO 291.

NOTE Depending on the material and its thermal history, the method of test specimen preparation may be crucial to the consistency of the results and their significance.

6.3 Coating the specimen

Specimens which are not opaque to the laser radiation at the wavelength used shall be coated with an appropriate coating (a metal coating, for example) to prevent penetration of the laser beam into the specimen. The influence of the coating on the heat transfer shall be negligible (i.e. it shall have a high diffusivity and low thickness in comparison with the specimen). The total thickness of the coating shall be chosen such that the half-rise time for the coating alone is less than 2 % of the total half-rise time for the specimen.

NOTE 1 The half-rise time, $t_{1/2}$, for the coating can be simply calculated from its thickness, d, and thermal diffusivity, α, using Equation (1), a rearranged form of Equation (B.1):

$$t_{1/2} = 0{,}13879\frac{d^2}{\alpha} \tag{1}$$

NOTE 2 Both sides of the specimen can be coated with a thin opaque black layer (i.e. a layer of graphite) to optimize the absorption of the energy pulse and the emission of thermal radiation.