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Plastics — Determination of thermal conductivity and thermal diffusivity —

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

Plastiques — Détermination de la conductivité thermique et de la diffusivité thermique —
Partie 2: Méthode de la source plane transitoire (disque chaud)

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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 22007-2 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*

In this corrected version of ISO 22007-2:2008, Figure 3 has been amended to remove a horizontal line running between ΔU and the earth connection.

Introduction

A significant increase in the development and application of new and improved materials for broad ranges of physical, chemical, biological and medical applications has necessitated better performance data from methods of measurement of thermal-transport properties. The introduction of alternative methods that are relatively simple, fast and of good precision would be of great benefit to the scientific and engineering communities [1].

A number of measurement techniques described as contact transient methods have been developed and several have been commercialized. These are being widely used and are suitable for testing many types of material. In some cases, they can be used to measure several properties separately or simultaneously [2],[3].

A further advantage of some of these methods is that it has become possible to measure the true bulk properties of a material. This feature stems from the possibility of eliminating the influence of the thermal contact resistance (see 8.1.1) that is present at the interface between the probe and the specimen surfaces [1],[3],[4],[5],[6].

Plastics — Determination of thermal conductivity and thermal diffusivity —

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

1 Scope

1.1 This part of ISO 22007 specifies a method for the determination of the thermal conductivity and thermal diffusivity, and hence the specific heat capacity per unit volume, of plastics. The experimental arrangement can be designed to match different specimen sizes. Measurements can be made in gaseous and vacuum environments at a range of temperatures and pressures.

1.2 This method is suitable for testing homogeneous and isotropic materials, as well as anisotropic materials with a uniaxial structure. In general, the method is suitable for materials having values of thermal conductivity, λ, in the approximate range $0{,}01\ \mathrm{W{\cdot}m^{-1}{\cdot}K^{-1}} < \lambda < 500\ \mathrm{W{\cdot}m^{-1}{\cdot}K^{-1}}$ and values of thermal diffusivity, α, in the range $5 \times 10^{-8}\ \mathrm{m^2{\cdot}s^{-1}} \leqslant \alpha \leqslant 10^{-4}\ \mathrm{m^2{\cdot}s^{-1}}$, and for temperatures, T, in the approximate range $50\ \mathrm{K} < T < 1\ 000\ \mathrm{K}$.

NOTE The specific heat capacity per unit volume, C, can be obtained by dividing the thermal conductivity, λ, by the thermal diffusivity, α, i.e. $C = \lambda/\alpha$, and is in the approximate range $0{,}2\ \mathrm{MJ{\cdot}m^{-3}{\cdot}K^{-1}} < C < 5\ \mathrm{MJ{\cdot}m^{-3}{\cdot}K^{-1}}$. It is also referred to as the volumetric heat capacity.

1.3 The thermal-transport properties of liquids can also be determined, provided care is taken to minimize thermal convection.

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 472, *Plastics — Vocabulary*

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

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 472 and ISO 22007-1 and the following apply.

3.1
penetration depth
Δp_{pen}
measure of how far into the specimen, in the direction of heat flow, a heat wave has travelled

NOTE 1 For this method, the penetration depth is given by

$$\Delta p_{\text{pen}} = \kappa\sqrt{\alpha \cdot t_{\text{tot}}}$$

where

t_{tot} is the total measurement time for the transient recording;

α is the thermal diffusivity of the specimen material;

κ is a constant dependent on the sensitivity of the temperature recordings.

NOTE 2 It is expressed in metres (m).

3.2
probing depth
Δp_{prob}
measure of how far into the specimen, in the direction of heat flow, a heat wave has travelled during the time window used for calculation

NOTE 1 The probing depth is given by

$$\Delta p_{\text{prob}} = \kappa\sqrt{\alpha \cdot t_{\text{max}}}$$

where t_{max} is the maximum time of the time window used for calculating the thermal-transport properties.

NOTE 2 It is expressed in metres (m).

NOTE 3 A typical value in hot-disc measurements is $\kappa = 2$, which is assumed throughout this document.

3.3
sensitivity coefficient
β_q
coefficient defined by the equation

$$\beta_q = q\frac{\partial\left[\Delta T(t)\right]}{\partial q}$$

where

q is the thermal conductivity, λ, the thermal diffusivity, α, or the volumetric specific heat capacity, C;

$\Delta T(t)$ is the mean temperature increase of the probe

NOTE 1 Different sensitivity coefficients are defined for thermal conductivity, thermal diffusivity and specific heat per unit volume [8].

NOTE 2 To define the time window that is used to determine both the thermal conductivity and diffusivity from one single experiment, the theory of sensitivity coefficients is used. Through this theory, which deals with a large number of experiments and considers the constants, q, as variables, it has been established that

$$0{,}30 \leqslant t_{max} \cdot \alpha / r^2 \leqslant 1{,}0$$

where r is the mean radius of the outermost spiral of the probe.

Assuming $\kappa = 2$, this expression can be rewritten as

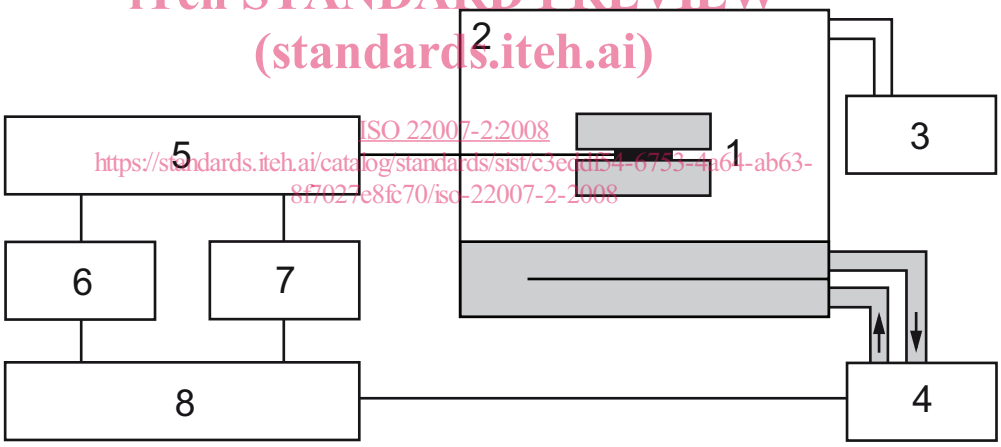
$$1{,}1r \leqslant \Delta p_{prob} \leqslant 2{,}0r$$

4 Principle

A specimen containing an embedded hot-disc probe of negligible heat capacity is allowed to equilibrate at a given temperature. A heat pulse in the form of a stepwise function is produced by an electrical current through the probe to generate a dynamic temperature field within the specimen. The increase in the temperature of the probe is measured as a function of time. The probe operates as a temperature sensor unified with a heat source (i.e. a self-heated sensor). The response is then analysed in accordance with the model developed for the specific probe and the assumed boundary conditions.

5 Apparatus

5.1 A schematic diagram of the apparatus is shown in Figure 1.



Key

1	specimen with probe	5	bridge circuit
2	chamber	6	voltmeter
3	vacuum pump	7	voltage source
4	thermostat	8	personal computer

Figure 1 — Basic layout of the apparatus

5.2 A typical hot-disc probe is shown in Figure 2. Convenient probes can be designed with diameters from 4 mm to 100 mm, depending on the specimen size and the thermal-transport properties of the material to be tested. The probe is constructed as a bifilar spiral etched out of a (10 ± 2) μm thick metal foil and covered on both sides by thin (from 7 μm to 100 μm) insulating film. It is recommended that nickel or molybdenum be used as the heater/temperature-sensing metal foil due to their relatively high temperature coefficient of electrical resistivity and stability over a wide temperature range. It is recommended that polyimide, mica, aluminum nitride or aluminum oxide be used as the insulating film, depending on the ultimate temperature of use. The arms of the bifilar spiral forming an essentially circular probe shall have a width of (0,20 ± 0,03) mm for probes with an overall diameter of 15 mm or less and a width of (0,35 ± 0,05) mm for probes of larger diameter. The distance between the edges of the arms shall be the same as the width of the arms.

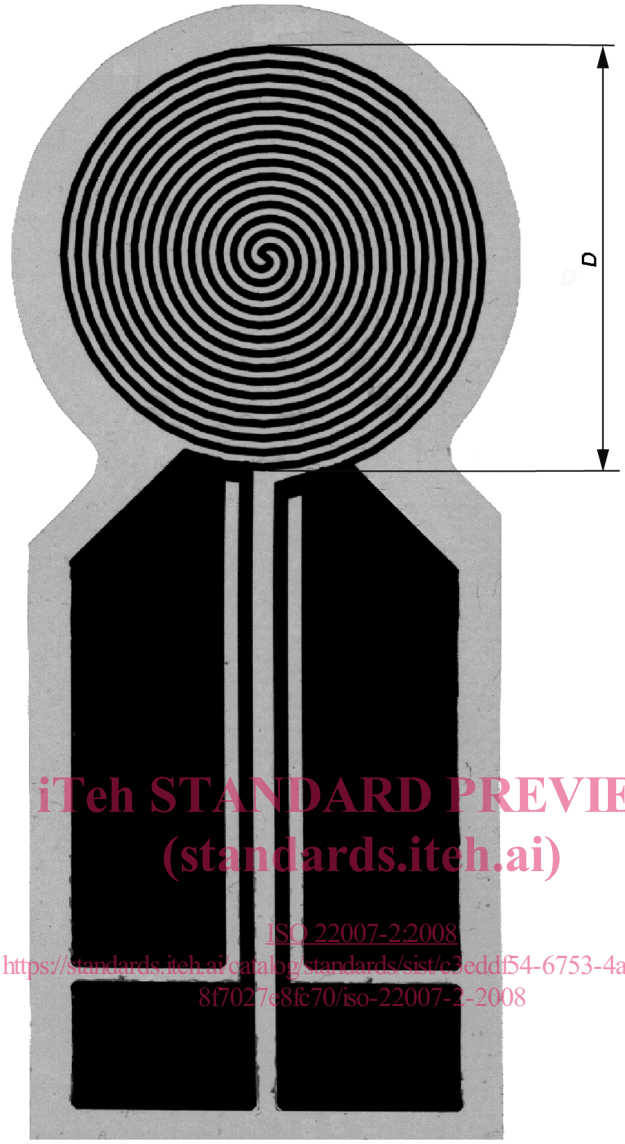
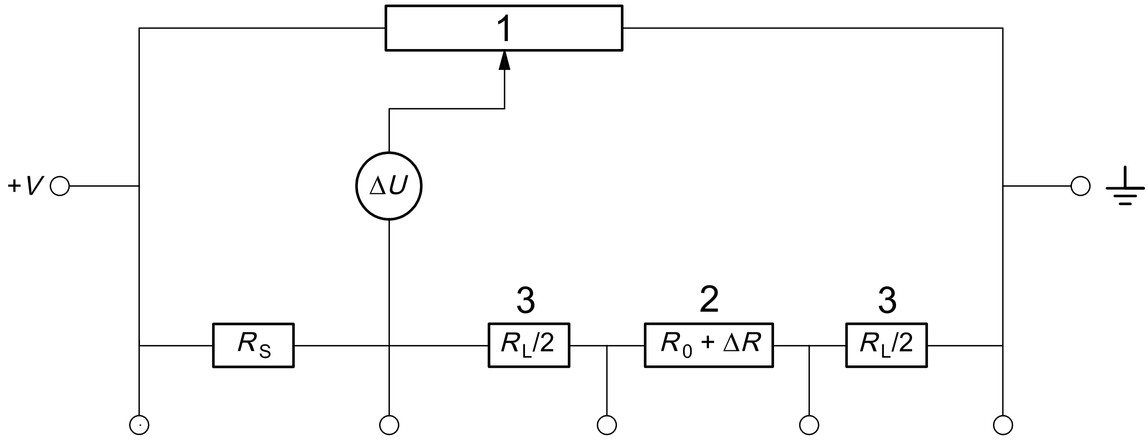


Figure 2 — Probe with bifilar spiral as heating/sensing element
(Sensor diameters, D, from 4 mm to 100 mm can be used, depending on available specimen size)

5.3 An electrical bridge shall be used to record the transient increase in resistance of the probe. Through the bridge, which is initially balanced, the successive increases in resistance of the probe shall be followed by recording the imbalance of the bridge with a sensitive voltmeter (see Figure 3). With this arrangement, the probe is placed in series with a resistor which shall be designed in such a way that its resistance is kept strictly constant throughout the transient. These two components are combined with a precision potentiometer, the resistance of which shall be about 100 times larger than the sum of the resistances of the probe and the series resistor. The bridge shall be connected to a power supply which can supply 20 V and a current of up to 1 A. The digital voltmeter by which the difference voltages are recorded shall have a resolution corresponding to 6,5 digits at an integration time of 1 power line cycle. The resistance of the series resistor, R_S, shall be close to the initial resistance of the probe with its leads, $R_0 + R_L$, in order to keep the power output of the probe as constant as possible during the measurement.



Key

1 potentiometer
2 probe
3 probe leads

Figure 3 — Diagram of bridge for recording the resistance increase of the probe

R_S is the series resistance, R_L is the total resistance of the probe leads, R_0 is the resistance of the probe before initiating the transient heating, ΔR is the increase in resistance of the probe during the transient heating and ΔU is the voltage imbalance created by the increase in the resistance of the probe.

NOTE This experimental arrangement allows the determination of temperature deviations from the iterated straight line (see treatment of experimental data in 8.1) down to or better than 50 µK.

5.4 A constant-temperature environment controlled to ± 0,1 K or better for the duration of a measurement shall be established. The chamber need only be evacuated when working with slab specimens (see 6.3).

6 Test specimens

6.1 Bulk specimens

6.1.1 For bulk specimens, the requirement for specimen thickness depends on the thermal properties of the material from which the specimen is made. The expression for the probing depth contains the diffusivity, which is not known prior to the measurement. This means that the probing depth has to be calculated after an initial experiment has been completed. If, with this new information, the probing depth is outside the limits given in 8.1.3, the test shall be repeated, with an adjusted total measurement time, until the required conditions are fulfilled.

The shape of the specimen can be cylindrical, square or rectangular. Machining to a certain shape is not necessary, as long as a flat surface (see 6.1.4) of each of the two specimen halves faces the sensor and the requirements on the sensor size given in 8.1.3 are fulfilled.

6.1.2 The thickness of the specimen shall be at least 20 times the characteristic length of the components making up the material or of any inhomogeneity in the material, e.g. the average diameter of the particles if the specimen is a powder.

6.1.3 The specimen dimensions shall be chosen to minimize the effect that its outer surfaces will have on the measurement. The specimen size shall be such that the distance from any part of the bifilar spiral of the hot-disc probe to any part of the outside boundary of the specimen is larger than the overall mean radius of the bifilar spiral (see 5.2). An increase in this distance beyond the size of the diameter of the spiral does not improve the accuracy of the results.