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Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 3: Temperature wave analysis method

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

Partie 3: Méthode par analyse de l'oscillation de la température

Reference number
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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 22007-3 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*

Introduction

Thermal-transport properties of plastics are indispensable not only in the plastics industry but also in other fields. Plastics are used in various manufacturing processes in new application areas, such as nanotechnologies, and in the biomedical industry. Accurate but simple small-scale measurements are required which can be performed quickly.

High sensitivity and excellent temperature resolution are peculiar to the modulation techniques used for the measurement of thermal-transport properties. Temperature wave analysis is a method of measuring the thermal diffusivity of thin specimens and is also suitable for use with small specimens.

Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 3:
Temperature wave analysis method

1 Scope

This part of ISO 22007 specifies a temperature wave analysis method for the determination of the thermal diffusivity of thin films and plates of plastics in the through-thickness direction. The method can be used on plastics in either the solid or molten state, and having either an isotropic or an orthotropic structure.

The method covers values of the thermal diffusivity, α, in the range $1{,}0 \times 10^{-8}\ m^2 \cdot s^{-1} < \alpha < 1{,}0 \times 10^{-4}\ m^2 \cdot s^{-1}$.

Measurements can be performed either in air or in another atmosphere, e.g. an inert gas, at atmospheric pressure or at other, reduced or elevated, pressures, or under a vacuum, at a variety of temperatures.

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*

ISO 80000-5, *Quantities and units — Part 5: Thermodynamics*

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 purposes of this document, the terms and definitions given in ISO 472, ISO 22007-1 and ISO 80000-5 and the following apply.

3.1
temperature wave
temperature oscillation produced by a power-modulated heat source

3.2
phase shift
$\Delta\theta$
phase difference of the temperature wave between the front and rear surfaces of a specimen

NOTE A delay is defined as a negative phase shift.

4 Symbols and units

Symbol	Meaning	Unit
A	slope of a plot of phase shift, $\Delta\theta$, versus the square root of the angular frequency, ω, of the temperature wave	$s^{1/2}$
C	heat capacity per unit volume	$J/(m^3 \cdot K)$
d	thickness of specimen	m
f	frequency of temperature wave	Hz
k	the quantity $(\omega/2\alpha)^{1/2}$	
α	thermal diffusivity	m^2/s
λ	thermal conductivity	$W/(m \cdot K)$
ω	angular frequency of temperature wave	rad/s
ω_c	angular frequency that satisfies the condition $kd = 1$	rad/s

5 Principle

5.1 Temperature wave analysis is a method of measuring thermal diffusivity in the through-thickness direction of a thin, flat specimen by measuring the phase shift of a temperature wave between the front and rear surfaces of the specimen.

5.2 Electrical resistors, sputtered or contacted on both surfaces of the specimen, are used, one as a heater to generate the temperature wave by a.c. Joule heating and the other as a thermometer to detect the temperature wave.

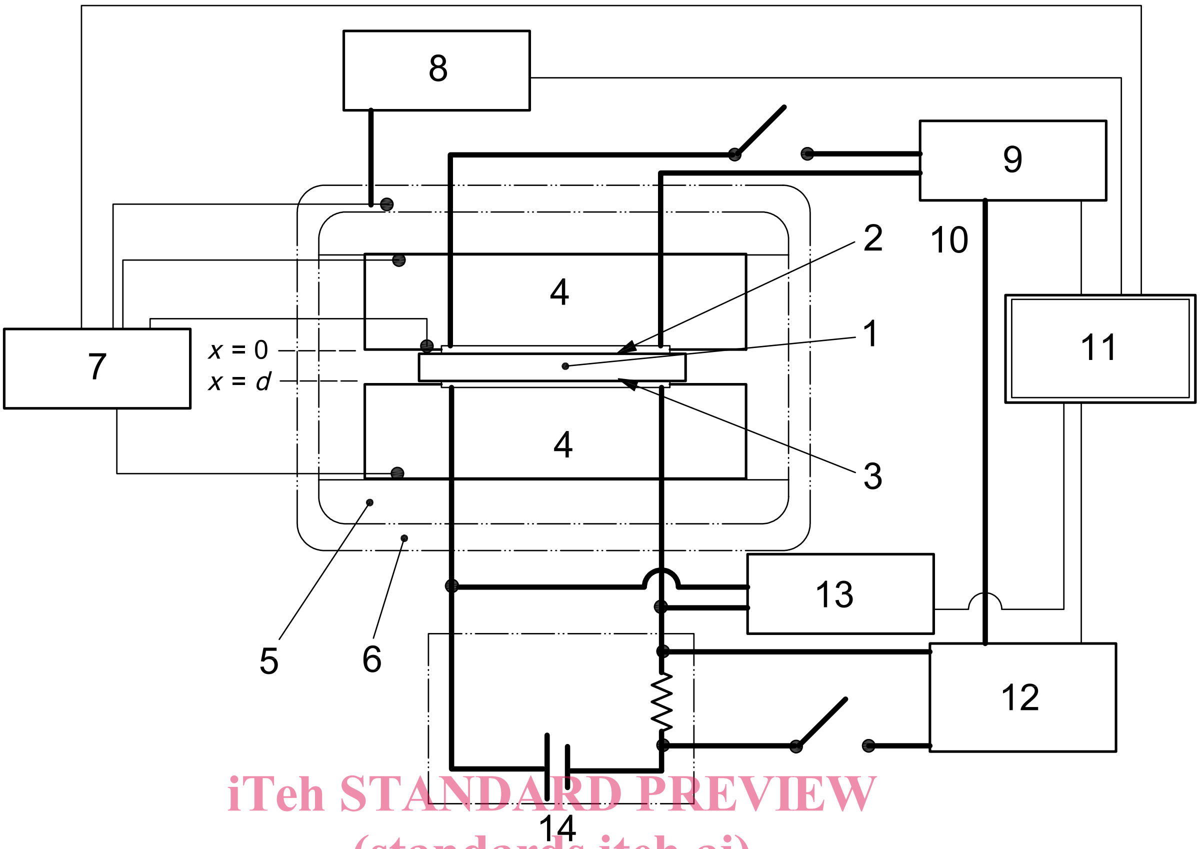
5.3 This method involves analysis of the phase shift of the temperature wave, which is propagated through the specimen, as a function of the square root of the angular frequency of the temperature wave.

NOTE Further details of the theoretical background are given in Annex A and the references in the Bibliography.

6 Apparatus

6.1 General

The apparatus shall be designed to determine the thermal diffusivity as described in Clause 5 and shall consist of the following main components. An example of a suitable apparatus is shown in Figure 1.



Key

1 specimen
2 heater
3 sensor
4 backing plate
5 specimen holder
6 constant-temperature enclosure
7 thermometer
8 temperature controller
9 function synthesizer
10 reference signal
11 personal computer
12 lock-in amplifier
13 digital multimeter
14 bias current circuit

Figure 1 — Schematic diagram showing an example of a suitable apparatus

6.2 Constant-temperature enclosure

The temperature range of the constant-temperature enclosure shall be appropriate to the materials to be tested.

It shall be possible to control the enclosure temperature such that the specimen temperature does not change by more than ± 1 K throughout the duration of the measurement.

6.3 Heater and sensor elements

The heater element used to generate the temperature wave by passing alternating current through an electrical resistor attached to the front surface of the specimen is assumed to be located at $x = 0$ (see Figure 1).

The sensor element used to detect the temperature wave by measuring the oscillation of the resistance of an electrical resistor attached to the rear surface of the specimen is assumed to be located at $x = d$.

The heater and sensor should preferably be sputtered directly onto opposite surfaces of the specimen in order to achieve high sensitivity and quick response. The heat capacities of the heater and sensor should be negligible.

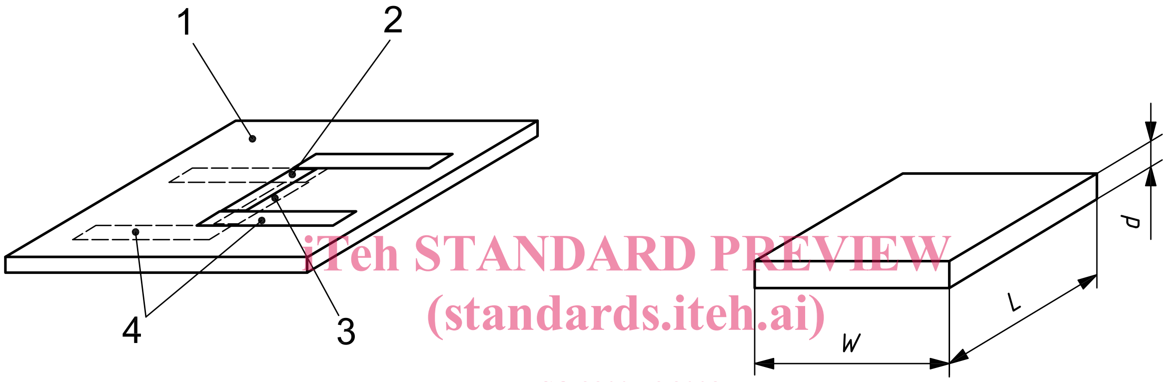
NOTE 1 An example of heater and sensor elements sputtered directly onto the front and rear surfaces of a film specimen is shown in Figure 2 a).

NOTE 2 An example of a heater and sensor set-up that can be used for liquid samples is shown in Figure 2 b). As direct sputtering is not possible in this case, a set of pre-sputtered specimen-backing plates is used.

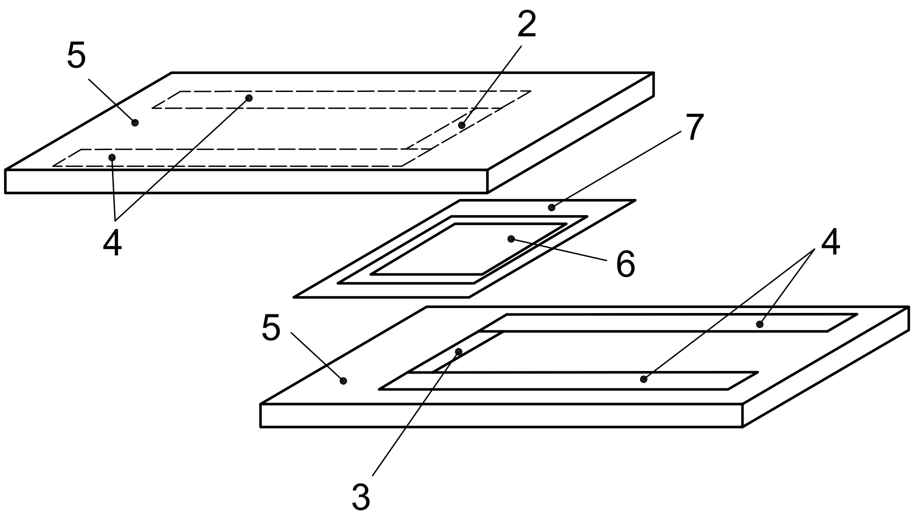
NOTE 3 Typical dimensions of a heater and sensor set-up deposited by sputtering on the specimen are 1 mm wide by 5 mm long.

Metal-layer leads are sputtered or connected using conductive paste or solder to each end of the heater and sensor elements. Alternatively, the leads can be sputtered onto the backing plates [see Figure 2 c)]. Electrical contacts from the lead layers to electric cables for connection to the power supply and measurement devices can be provided using conductive paste or solder.

Direct sputtering of the heater and sensor onto the front and rear surfaces of the specimen is recommended for good thermal contact. As an alternative, the heater and sensor can be attached to the specimen surface under constant load.

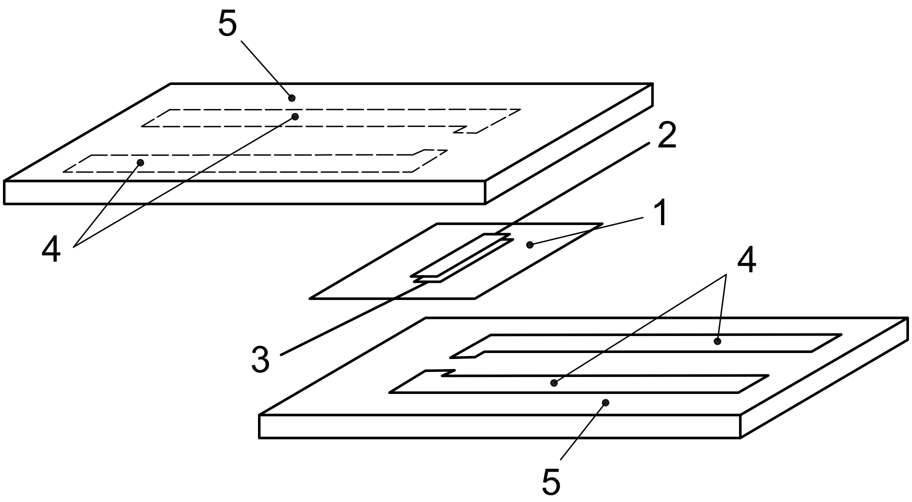


a) Heater and sensor elements and leads sputtered directly on the front and rear surfaces of a film specimen (Examples of dimensions — Specimen: $W = 10$ mm, $L = 10$ mm, $d = 100$ μm; heater: $W = 1$ mm, $L = 5$ mm; sensor: $W = 1$ mm, $L = 5$ mm)



b) Liquid specimen inserted between the backing plates on which the heater and sensor elements and leads are sputtered (Examples of dimensions — Spacer: $W = 10$ mm, $L = 10$ mm, $d = 100$ μm; heater: $W = 1$ mm, $L = 5$ mm; sensor: $W = 1$ mm, $L = 5$ mm; backing plate: $W = 30$ mm, $L = 25$ mm, $d = 2$ mm)

Figure 2 (*continued*)



c) **Heater and sensor elements sputtered directly on the specimen, with the leads on the backing plates**
(Examples of dimensions — Specimen: $W = 10$ mm, $L = 10$ mm, $d = 100$ µm; heater: $W = 1$ mm, $L = 5$ mm; sensor: $W = 1$ mm, $L = 5$ mm; backing plate: $W = 30$ mm, $L = 25$ mm, $d = 2$ mm)

Key

1 specimen
2 sputtered heater (on front)
3 sputtered sensor (on rear)
4 leads
5 backing plate
6 specimen (liquid)
7 spacer

Figure 2 — Examples of sets of heater and sensor elements

6.4 Heating circuit

The power applied to the heater shall be adjusted so as to avoid a specimen temperature rise of more than 1 K.

6.5 Measurement circuit

A bias electric current is supplied to the sensor via the layer leads for the measurement of the oscillation of the electric resistance of the sensor. A d.c. source that can supply an electric current to the sensor in the range 1 µA to 10 mA is used.

It is recommended that the accuracy of measurement of the oscillation frequency be better than 50 ppm.

6.6 Phase-shift measurement device

The oscillation of the electrical resistance of the sensor, observed as an oscillation of the voltage between the ends of the leads to the sensor, shall be measured to determine the phase shift of the temperature wave across the thickness of the specimen. The phase shift between the heater and the sensor shall be measured by a set-up such as that shown in Figure 1. The recommended accuracy for measurement of the phase shift is ± 0,01°.

6.7 Devices for measuring the specimen temperature

Thermocouples may be fixed onto the backing plates and also attached to the specimen if possible. When the temperature coefficient of the electrical resistance of the sensor is known, the temperature of the sensor can be determined by measuring its electrical resistance.

