FINAL
DRAFT

INTERNATIONAL
STANDARD

**ISO/FDIS
22007-7**

ISO/TC **61**/SC **5**

Secretariat: **DIN**

Voting begins on:
2023-01-30

Voting terminates on:
2023-03-27

Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 7:
Transient measurement of thermal effusivity using a plane heat source

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO/FDIS 22007-7
https://standards.iteh.ai/catalog/standards/sist/0dcbee0d-e207-4e02-9955-3288331c6c7a/iso-fdis-22007-7

RECIPIENTS OF THIS DRAFT ARE INVITED TO SUBMIT, WITH THEIR COMMENTS, NOTIFICATION OF ANY RELEVANT PATENT RIGHTS OF WHICH THEY ARE AWARE AND TO PROVIDE SUPPORTING DOCUMENTATION.

IN ADDITION TO THEIR EVALUATION AS BEING ACCEPTABLE FOR INDUSTRIAL, TECHNOLOGICAL, COMMERCIAL AND USER PURPOSES, DRAFT INTERNATIONAL STANDARDS MAY ON OCCASION HAVE TO BE CONSIDERED IN THE LIGHT OF THEIR POTENTIAL TO BECOME STANDARDS TO WHICH REFERENCE MAY BE MADE IN NATIONAL REGULATIONS.

Reference number
ISO/FDIS 22007-7:2023(E)

© ISO 2023

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO/FDIS 22007-7
https://standards.iteh.ai/catalog/standards/sist/0dcbee0d-e207-4e02-9955-3288331c6c7a/iso-fdis-22007-7

COPYRIGHT PROTECTED DOCUMENT

© ISO 2023

All rights reserved. Unless otherwise specified, or required in the context of its implementation, no part of this publication may be reproduced or utilized otherwise in any form or by any means, electronic or mechanical, including photocopying, or posting on the internet or an intranet, without prior written permission. Permission can be requested from either ISO at the address below or ISO's member body in the country of the requester.

ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Email: copyright@iso.org
Website: www.iso.org

Published in Switzerland

© ISO 2023 – All rights reserved

Contents

Page

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO/FDIS 22007-7
https://standards.iteh.ai/catalog/standards/sist/0dcbee0d-e207-4e02-9955-3288331c6c7a/iso-fdis-22007-7

© ISO 2023 – All rights reserved

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.

The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular, the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

iTeh Standards
(standards.iteh.ai)

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

ISO/FDIS 22007-7
https://standards.iteh.ai/catalog/standards/sist/...-9955-3288331c6c7a/iso-fdis-22007-7

A list of all parts in the ISO 22007 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

© ISO 2023 – All rights reserved

Introduction

The developments of so-called transient measurement methods since the 1990's[1]-[4], has provided the scientific community with tools capable of quickly and accurately testing thermophysical properties of small- and irregular-shaped specimens[5]-[9].

A regularly-shaped probe (square, rectangle, circle, ellipse, etc.), consisting of a metal heating pattern, is sandwiched between two pieces of a specimen material. The probe simultaneously functions as an ohmic heater – providing approximately equal heat production per unit area across its surface – and also as a resistance thermometer. In experimental configurations discussed in the following, the thermal effusivity in the normal direction to the probe surface can be estimated from a single experiment[2]-[4],[9].

The specimens that can be tested using this method are homogeneous isotropic specimens and homogeneous anisotropic specimens (with uniaxial structure[10]). The effusivity is obtained for the bulk of the specimen material, because of the possibility to eliminate the influence from the thermal contact resistance between the probe sensing metal pattern and the substrate surface.

Some experimental features on testing thermal effusivity with present approach are, first, the ability to significantly reduce the overall specimen geometry size. Secondly, the normal-direction heat flow allows for analysing specimen geometries of major industrial importance, for instance, a layered- or composite structure, with repeated intrinsic geometric features.

One industrial application considered is the TIM-stacked setup, consisting of a repeated structure incorporating thermal interface material (TIM) layers between solid slabs. The many drawbacks and uncertainties of testing a single-layer TIM layer applied in alternative measurement approaches, is here replaced with an experimental stack setup allowing to precisely measure the final application intended for a specific TIM layer material.

Parameters to consider when testing thermal effusivity in a rod-shaped specimen are: differences in probe cross-section and rod specimen cross-section. At least a rough estimation on the volumetric specific heat of the specimen is also advantageous to know, when estimating the probing depth (important for controlling of the transient experiment). In addition, potential effects of heat losses to surroundings should also be assessed.

© ISO 2023 – All rights reserved

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO/FDIS 22007-7
https://standards.iteh.ai/catalog/standards/sist/0dcbee0d-e207-4e02-9955-3288331c6c7a/iso-fdis-22007-7

Plastics — Determination of thermal conductivity and thermal diffusivity —

Part 7:
Transient measurement of thermal effusivity using a plane heat source

1 Scope

This document specifies a method for the determination of the thermal effusivity.

This document is applicable to materials with thermal effusivity in the approximate range 40 $W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1} < b_n < 40\,000$ $W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1}$, and temperatures in the range of 50 K < T < 1 000 K.

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

ISO 22007-2, *Plastics — Determination of thermal conductivity and thermal diffusivity — Part 2: Transient plane heat source (hot disc) method*

iTeh STANDARD PREVIEW
(standards.iteh.ai)
ISO/FDIS 22007-7
https://standards.iteh.ai/catalog/standards/sist/0dcbee0d-e207-4e02-9955-3288331c6c7a/iso-fdis-22007-7

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 22007-1, ISO 22007-2 and the following apply.

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

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

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

3.1
thermal effusivity
b
quantity, possible to express in terms of the square root of the product of the material's bulk thermal conductivity and volumetric specific heat of a specimen, $b = \sqrt{\lambda \cdot \rho c_p}$

Note 1 to entry: In its most general form, this is a second-rank tensor property.

Note 2 to entry: The thermal effusivity in the normal direction to the plane of the probe is represented by the scalar b_n.

Note 3 to entry: It is expressed in $W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1}$.

© ISO 2023 – All rights reserved

4 Principle

4.1 A specimen with an internally-positioned thermal effusivity probe – assumed to have a negligible heat capacity – is set to thermally equilibrate at a certain temperature. A measurement is conducted by applying a single-step heat pulse (generated by Ohmic heating). A temperature field around the probe develops with time (from the onset of the single-step heat pulse). The temperature increase in the probe is recorded at different time points.

4.2 The probe represents a combined heater and temperature sensor – which is sometimes referred to as a self-heated sensor. The temperature vs. time response is then analysed for the model developed and the assumed boundary conditions. Two principally different configurations are possible for testing normal-direction thermal effusivity.

4.3 Configuration A: Specimens and an experimental setup designed to allow the heat flow to occur essentially in a 1-dimensional manner, in the normal direction from the probe, for a comparably long period of experimental time. It is suitable for small and narrow specimens with a thermal effusivity above approximately 1 000 $W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1}$.

4.4 Configuration B: Specimens and an experimental setup designed to allow the heat flow to occur essentially in a 1-dimensional manner, in the normal direction from the probe, for a comparably short period of experimental time. It is suitable for large and wide specimens having a thermal effusivity less than approximately 1 000 $W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1}$.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

5 Apparatus

The measuring apparatus shall be in accordance with ISO 22007-2.

However, the shape of the probe can differ appreciably as long as an even heat distribution across the probe cross-section area can be established, see 6.1.3 and 6.2.3.

ISO/FDIS 22007-7
https://standards.iteh.ai/catalog/standards/sist/0dcbeeed-e207-4e02-9955-3288331e6c7a/iso-fdis-22007-7

6 Test specimens

6.1 Configuration A: Rod-shaped specimens having a thermal effusivity above 1 000 $W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1}$

6.1.1 Typical specimen geometry is a cross-section area of minimum from approximately 7 mm^2 (corresponding to approximately 3 mm diameter) to a maximum of approximately 1 000 mm^2. There is no requirement regarding the exact shape of the cross-section of the rod, as long as this cross-section geometry is identical along the length of the rod. Advantageous geometries are circular, square or rectangular-shaped cross-sections. The rod length, which represents the orientation in which the heat flow occurs during an experiment and in which orientation the thermal effusivity is to be estimated, is normally selected depending on the thermophysical properties of the material from which the specimen is made, and a direct connection is made with the probing depth (see Clause 7). While all examples in Table 1 have a probing depth of 20 mm, the method described in this subclause is capable of analysing specimens for rod lengths in the approximate range from minimum length around 3 mm to a maximum length around 100 mm. In case several repeat-structure components make up the material (see for example Annex B), the rod length should be selected to at least 10 times the repeat-structure length scale in order to reduce measurement errors and improve stability in the estimated results.

6.1.2 The specimen geometry is adapted to the geometry of the probe heating area. The cross-section of the rod shall closely resemble the cross-section of the probe heating area. The cross-section of the rod shall however be large enough to closely, but completely, embed the probe in a way that no part of the heating elements of the probe is allowed to stick out from the lateral boundary. A margin (or tolerance) of 0,5 mm to 1 mm is often acceptable between the edge of the probe and the lateral boundary and is

© ISO 2023 – All rights reserved

compensated for in computations (see Clause 8). In addition, the rod lengths covered by the present method are limited to within 20 times the minimum rod cross-section distance.

6.1.3 As probes can be designed to be in different shapes, such as cylindrical, square or rectangular, the shape of the specimen cross-section shall have similar shape.

6.1.4 In the basic setup, two specimen halves with symmetric rod geometry facing the probe are assumed. A flat surface (see 6.3.2) on each of the two specimen halves facing the probe is required.

6.2 Configuration B: Specimens of thermal effusivity below 1 000 $W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1}$ completely embedding the probe

6.2.1 Typical bulk specimen geometry is a cross-section area of minimum from approximately 2 000 mm^2 (corresponding to approximately 50 mm diameter) to a maximum of approximately 50 000 mm^2. The thickness of the bulk specimen required, which due to the low thermal effusivity requirements, is limited depending on the thermophysical properties of the material from which the specimen is made, and a direct connection is made with the probing depth (see Clause 7). As the thickness direction represents the orientation of heat flow assumed in the experiment, the thermal effusivity in the thickness direction is estimated. While the examples in Table 2 have a probing depth of 4 mm, the specimen thickness for these examples would require a minimum thickness of 4 mm for a corresponding experiment to be performed. The described method is capable of analysing specimens of thicknesses in the approximate range from minimum thickness around 3 mm to a maximum thickness around 30 mm. In case several repeat-structure components make up the material, preferably the specimen thickness should be selected to at least 10 times the repeat-structure length scale in order to reduce measurement errors and improve stability in the estimated results.

6.2.2 The specimen geometry is adapted to the geometry of the probe heating area. The cross-section of the specimen shall completely embed the probe cross-section, and with a margin on each edge that exceeds the specimen thickness on the sides of the probe, i.e. the cross-section of the specimen in one direction shall be at least equal to the cross-section of the probe in the same direction plus 2 x specimen thickness. For example, a probe of 30 mm × 30 mm cross section, and a specimen thickness of 5 mm, requires a specimen cross-section of a minimum 40 mm × 40 mm cross section. Note that in case a specific probing depth is required to achieve, for instance if according to 6.2.1 a specific thickness is required to reach at least 10 times the repeat-structure length scale in the thickness direction, the cross-section of the specimen geometry as well as the geometry of the probe might need to be selected differently: According to 6.2.3, the minimum cross-section distance across the probe area should be selected at least 10 times the specimen thickness (assuming near-isotropic specimen conditions), or at least 30 times the specimen thickness (in case anisotropy may be at hand) – which with the additional requirement of a margin at each edge results in a different minimum specimen cross-section area.

6.2.3 Probes can be designed to be in different shapes, such as cylindrical, square or rectangular. However, it should be noted that specimen thickness probed in the experiment will not exceed 1/10 of the minimum distance across the cross-section of an effusivity probe, and hence the specimen thickness can be adapted accordingly following the cross-section design of the probe.

6.2.4 In the basic setup, two specimen halves with symmetric setup facing the probe are assumed. A flat surface (see 6.3.2) on each of the two specimen halves facing the probe is required.

6.2.5 In case in-plane thermal conductivity is estimated to be more than ten times the through-plane thermal conductivity, the setup in 6.2 should not be used.

6.3 Specimen preparation

NOTE These specimen preparations apply to the setups described in 6.1 or 6.2.

© ISO 2023 – All rights reserved

6.3.1 The specimen should be conditioned in accordance with the standard specification which applies to the type of material and its particular use.

6.3.2 The specimen surfaces which are in contact with the probe should be plane and smooth. The specimen halves shall be clamped on to both sides of the effusivity probe.

6.3.3 It is important to consider specimen materials prone to significant dimensional changes – whether caused by measurements over large temperature ranges, thermal expansion, change of state, phase transition, or other causes.

6.3.4 Care should be taken to ensure that the applied load does not affect the properties of the specimen. For instance, for soft specimens tested according to setup 6.2, the clamping pressure should not compress the specimen and thus change its thermal transport properties.

6.3.5 Heat sink contact paste shall not be used since:

a) it is difficult to obtain a sufficiently thin layer of paste which will actually improve the thermal contact;

b) the paste obviously increases the heat capacity of the insulating layer and delays the development of the constant temperature difference between the sensing material and the specimen surface;

c) it is difficult to obtain exactly the same thickness of paste on both sides of the probe and achieve a strictly symmetrical flow of heat from the heating/sensing material through the insulation into the two specimen halves.

iTeh STANDARD PREVIEW
(standards.iteh.ai)

7 Procedure

ISO/FDIS 22007-7
https://standards.iteh.ai/catalog/standards/sist/0dcbee0d-e207-4e02-9955-3288331e6c7a/iso-fdis-22007-7

7.1 The procedure for performing measurements shall be in accordance with ISO 22007-2, with the following additional considerations.

7.2 As described in Clause 6, the preparation of specimen is connected directly with the selection of effusivity probe to be used.

7.3 For rod shaped specimens, it is important to ensure that heat losses to the surroundings can be controlled to a minimum. For specimens of thermal effusivity more than 1 000 $W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1}$ it is normally enough with air, vacuum, or styrofoam insulation applied on the lateral surfaces.

7.4 For rod shaped specimens, it is important to ensure a correct measurement time. This is obtained by ensuring that the probing depth is at least 1/3 of the rod length, but not exceeding the rod length. A couple of scouting measurement may sometimes be made, in order to find a suitable measurement time. In case there are repeat components making up the length of the rod, the probing depth into the specimen shall be at least 10 times the characteristic length of the components making up the material or of any inhomogeneity in the material, in the direction of the rod axis.

The expression for the normal-direction probing depth is given by Formula (1):

$$\Delta p_{\text{prob}} = 2\sqrt{\alpha_n t_{\max}} \tag{1}$$

where

$t_{\max}$ is the maximum time of the time window used for calculating the thermal-transport properties;

© ISO 2023 – All rights reserved

α_n is the thermal diffusivity of the specimen material in the normal direction to the probe surface.

In case the thermal diffusivity α_n is not known, the volumetric specific heat capacity of the specimen should be estimated, either from tabulated data or by direct – separate – measurement by an alternative method, in order to compute the probing depth according to Formula (2):

$$\Delta p_{\text{prob}} = 2b_n \left(t_{\max}\right)^{1/2} \left(\rho c_p\right)^{-1} \tag{2}$$

7.5 For rod-shaped specimens, the heat pulse power and test time should use Table 1 or a scouting experiment, as a guideline.

7.6 For rod-shaped specimens, in case the specimen can be considered being anisotropic, and the timescale for heat to spread across the rod cross-section is considered to be much smaller than the total experimental test time, the effects of anisotropy can be assumed to not influence the estimation of the normal-direction thermal effusivity. For an anisotropic homogeneous specimen, where equal heating can be assumed across the probe position cross-section, the conditions of the 1-dimensional setup can also be assumed.

7.7 For rod-shaped specimens, the heat losses from lateral surfaces influence the accuracy of the measurement. Estimated net heat losses at lateral surfaces (if these can be estimated) divided by total heating power input in the probe, indicate the contribution of the lateral heat losses to the absolute error of the measurement.

NOTE When making a measurement on a material with a high thermal effusivity, the temperature undergoes a rapid increase at the very beginning of the transient followed by a much more gradual increase[11]. The insulating layer, between which the sensing spiral is sandwiched, causes this rapid increase. It has been shown both experimentally and in computer simulations that the temperature difference across the insulating layer becomes constant within a very short time and remains constant throughout the measurement. The reason is that the total power output, the area of the sensing material and the thickness of the insulating layer are constant during the test.

7.8 For specimens with a probe totally embedded in the specimen having a thermal effusivity below 1 000 $W{\cdot}s^{1/2}{\cdot}m^{-2}{\cdot}K^{-1}$, it is important to ensure a correct measurement time. This is obtained by ensuring that the probing depth is at least 1/3 of the specimen thickness, but not exceeding the specimen thickness. A couple of scouting measurement may sometimes be made, in order to find a suitable measurement time. In case there are repeat components making up the thickness, the probing depth into the specimen shall be at least 10 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. Formulas (1) and (2) can be used to compute the normal-direction probing depth.

7.9 For specimens with a probe totally embedded in the specimen having a thermal effusivity below 1 000 $W{\cdot}s^{1/2}{\cdot}m^{-2}{\cdot}K^{-1}$, the heat pulse power and test time should use Table 2 or a scouting experiment, as a guideline.

7.10 For specimens with a probe totally embedded in the specimen having a thermal effusivity below 1 000 $W{\cdot}s^{1/2}{\cdot}m^{-2}{\cdot}K^{-1}$, in case the in-plane thermal conductivity is higher than the through-plane thermal conductivity, up to 10 times higher than the through-plane thermal conductivity, the normal-direction probing depth should be controlled to within less than 1/30 of the smallest cross-section width of the effusivity probe.

7.11 For specimens with a probe totally embedded in the specimen having a thermal effusivity below 1 000 $W{\cdot}s^{1/2}{\cdot}m^{-2}{\cdot}K^{-1}$, the deviation between the experimental temperature response and that predicted by the 1-dimensional model is increasing with experimental test time – due to side-ways heatflow at

© ISO 2023 – All rights reserved