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Plastics — Determination of thermal conductivity and thermal diffusivity —

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

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

Partie 7: Mesure transitoire de l'effusivité thermique à l'aide d'une source de chaleur plane

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Foreword

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

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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.

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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 $40\ \mathrm{W{\cdot}s^{1/2}{\cdot}m^{-2}{\cdot}K^{-1}} < b_\mathrm{n} < 40\,000\ \mathrm{W{\cdot}s^{1/2}{\cdot}m^{-2}{\cdot}K^{-1}}$, and temperatures in the range of $50\ \mathrm{K} < T < 1\,000\ \mathrm{K}$.

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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*

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 $\mathrm{W{\cdot}s^{1/2}{\cdot}m^{-2}{\cdot}K^{-1}}$.

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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 $W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1}$.

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.

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

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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 $W·s^{1/2}·m^{-2}·K^{-1}$ 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.

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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.

7 Procedure

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;

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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):

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

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

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the edge of the effusivity probe. Following outlined procedures, this error can be controlled to within the specified errors.

7.12 The thermal effusivity shall be reported along with the conditions under which it was measured, such as temperature and pressure. The cardinal directions of the material and their orientations in relation to the plane surface of the specimen shall be reported whenever a material is anisotropic.

Table 1 — Summary of recommended experimental parameters for a range of polymer composites with different thermal effusivities

	Material A	Material B	Material C	Material D
Thermal effusivity $\left[W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1}\right]$	$1{,}8 \cdot 10^4$	$9{,}2 \cdot 10^3$	$4{,}4 \cdot 10^3$	$1{,}3 \cdot 10^3$
Thermal conductivity $\left[W \cdot m^{-1} \cdot K^{-1}\right]$	170	40	14	1,5
Specific heat per unit volume $\left[J \cdot m^{-3} \cdot K^{-1}\right]$	$2{,}0 \cdot 10^6$	$2{,}1 \cdot 10^6$	$1{,}4 \cdot 10^6$	$1{,}2 \cdot 10^6$
Temperature increase in specimen $[K]$	0,3	0,6	0,8	1,6
Measurement time $[s]$	1,2	5,3	10	80
Power output $[W]$	0,9	0,4	0,2	0,04
Probing depth $[mm]$	20	20	20	20
NOTE Rod cross-section is 10 mm × 10 mm (probe cross-section assumed to be similar), and rod length is 20 mm.				

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Table 2 — Summary of recommended experimental parameters for two examples of materials with different thermal effusivities

	Polymer	Insulating material
Thermal effusivity $\left[W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1}\right]$	$5{,}7 \cdot 10^2$	$3{,}2 \cdot 10^2$
Thermal conductivity $\left[W \cdot m^{-1} \cdot K^{-1}\right]$	0,19	0,28
Specific heat per unit volume $\left[J \cdot m^{-3} \cdot K^{-1}\right]$	$1{,}7 \cdot 10^6$	$3{,}7 \cdot 10^5$
Temperature increase in specimen $[K]$	1,4	2,5
Measurement time $[s]$	34	5
Power output $[W]$	0,6	1,6
Probing depth $[mm]$	4	4
NOTE Probe cross-section is 50 mm × 50 mm (probe is completely embedded), specimen width is 90 mm × 90 mm and specimen thickness is 20 mm.		

8 Calculation of thermal effusivity

8.1 Computations

8.1.1 An identical procedure, as outlined and described in detail in ISO 22007-2, is employed for calculating the mean temperature increase of the probe $\Delta T = T(t) - T_0$ from the experimental recording.

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8.1.2 The temperature increase can be seen as consisting of two parts. One part represents the temperature difference across the intercalated insulating layer and the other part the temperature increase of the specimen surface during the transient measurement. This can be expressed as shown in Formula (3):

$$\Delta T(t)=\Delta T_i(t)+\Delta T_s(t) \tag{3}$$

where

$\Delta T_i(t)$ is the increase in temperature over the insulating layers of the probe;

$\Delta T_s(t)$ is the increase in the temperature of the specimen surface.

8.1.3 With the assumption that equal heating per unit area occurs across the entire cross-section area, the solution of the thermal conductivity equation used to determine the thermal effusivity is given by Formula (4):

$$\Delta T_s(t)=\left(P_0\sqrt{t-t_c}\right)/\left(\pi^{1/2}A_{cr}b_n\right) \tag{4}$$

where

P_0 is the power output of the probe;

A_{cr} is the cross-section area of rod cross-section for setup described in 6.1 (Rod-shaped specimens having a thermal effusivity above 1 000 $W\cdot s^{1/2}\cdot m^{-2}\cdot K^{-1}$), or cross-section area of the probe for setup described in 6.2 (Bulk specimens of thermal effusivity below 1 000 $W\cdot s^{1/2}\cdot m^{-2}\cdot K^{-1}$ completely embedding the probe);

t_c is a time correction.

8.1.4 A time correction, t_c, shall be introduced because of unavoidable hardware and software delays. This means that the development of the full power output of the probe does not coincide exactly with the time t = 0, and a time correction shall be introduced accordingly. Typical time corrections are a fraction of a second and shall not be larger than 0,5 % of the total measurement time.

NOTE 1 $\Delta T_i(t)$ becomes constant after a short time provided the probe insulating layer is thin and the power output is constant. The time it takes to approach this constant value is determined by the relaxation time, δ^2/α_i, where δ is the thickness of the insulating layer and a_i is the thermal diffusivity of the layer. For a typical insulated probe, the relaxation time is less than 10 ms and the time required to reach a constant temperature difference is less than 100 ms.

NOTE 2 The possibility of determining the thermal contact resistance experimentally via the initial temperature difference, $\Delta T_i(t)$, enables the true bulk properties of the specimen material to be determined.

NOTE 3 If the thermal effusivity and time correction is known (see Annex A), there is a linear relationship between the temperature increase, $\Delta T_s(t)$, and $\sqrt{t-t_c}$ [see Figure A.2)] where, in the example given, $\Delta T_i = 3{,}585\,1\,(\text{K})$ and $\Delta T_s(t)=1{,}612\,4\sqrt{t-t_c}\,(\text{K})$.

8.1.5 The calculation of thermal effusivity starts with an iteration procedure with the time correction, t_c, as optimization variable. Through iteration, a model fitting of the experimental temperature points to the linear model $\Delta T(t)=\Delta T_i + B\sqrt{t-t_c}$ visualised by plotting the experimental temperature increase data points on the y-axis against the corresponding $\sqrt{t-t_c}$ data points on the x-axis, whereby the slope B is obtained by a least-squares fitting procedure. The standard deviation of this least-squares fitting procedure varies with selected time correction t_c. The optimal time correction $t_{c,op}$ is obtained

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from the final step of the iteration procedure resulting in the best model fit, *i.e.* minimum standard deviation, with a corresponding slope B_{op}. Finally, b_n is determined according to Formula (5):

$$b_{\mathrm{n}} = P_0 / \left(\pi^{1/2} A_{\mathrm{cr}} B_{\mathrm{op}}\right) \quad (5)$$

8.1.6 The initial time window selected for the analysis can result in experimental points deviating from a straight line [see Figure A.2)]. By removing deviating data points, a correct time window is obtained for the analysis. The graph of residuals, i.e. plotting the difference between experimental temperature points and model-fitted temperature points $(\Delta T_{\mathrm{exp}} - \Delta T_{\mathrm{model}})$ *vs.* $\sqrt{t - t_{\mathrm{c}}}$ displays the deviating points. (ISO 22007-2:2022, Figure 4 illustrates an example of a graph of residuals.)

8.1.7 When selecting the time window for analysis, as the specimen is of limited size (rod length for setup 6.1 or specimen thickness for setup 6.2), the outside boundaries might, after some time, affect the temperature increase. This deviation becomes apparent in the graph of residuals and, if there are deviating points at the end of the transient, these shall also be deleted. The deviations at the end of the transient becomes apparent when the estimated probing depth [Formula (1) or (2)] exceeds the size limit of the specimen. In case other conditions are required, for instance requirements in 7.4 or 7.8, the time window for analysis should be selected accordingly. If initial scouting tests and initial computations following Clause 8 indicate a too short experimental test time has been selected, the measurement shall be redone with a longer test time.

8.1.8 When selecting the time window for analysis for rods (specimens and setup according to 6.1), a similar procedure of removing initial time points where model-fitting is bad, needs to be undertaken. It can be shown from simulations and experiments that any gap distance β between the effusivity probe and the edge of the rod (in the cross-section plane) may require cutting of initial data points corresponding to $2{,}4\beta = 2\sqrt{\alpha_n t_1} = 2b_n\sqrt{t_1} / \rho c_p$, where (t_1, t_{max}) is the time window for data fitting.

Within the geometrical zone (0, 2,4β), where 0 is the probe position, the heat flux from the effusivity probe is not fully 1-dimensional but represents a 3D heat flow zone. These initial data points should be omitted from the model fitting. In the geometrical zone (2,4β, d_p), the heat flux along the length of the rod can be assumed to occur in a 1-dimensional manner. These data points generally gives good fitting of the model to experimental data.

It is advisable to use an effusivity probe that is fully embedded within a rod cross section, but with a heating zone which is as close as possible to the cross-section of the rod. Greater deviations in heating zone cross-section and the rod cross-section, will require the omittance of more data points in the beginning of the transient, which is not desirable.

8.1.9 In case data points does not give good fitting of the model to experimental data, this indicates either the specimen is not structurally homogeneous, or the initial temperature of the specimen, at the beginning of the experiment, is not isothermal.

8.1.10 After the thermal effusivity of the experiment has been estimated – and in case the volumetric specific heat ρc_p of the specimen is known from a separate measurement or from an external source – the thermal conductivity in the specimen normal direction can be computed as according to Formula (6):

$$\lambda_n = b_n^{\,2} / \rho c_p \quad (6)$$

Also, the thermal diffusivity in the specimen normal direction can be computed according to Formula (7):

$$\alpha_n = \left(b_n / \rho c_{\mathrm{p}}\right)^2 \quad (7)$$

An example of testing a homogeneous, anisotropic rod specimen is presented in informative Annex A.

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An example of testing a stacked, anisotropic rod specimen is presented in informative Annex B – demonstrating how the thermal conductivity of a thin thermal interface material (TIM) can be estimated.

8.2 Single-sided setup

For a situation representing a single-sided probe, where the backing material (side 2) has a thermal diffusivity α_2 lower than that of the unknown specimen (side 1)[8], and a thermal effusivity $b_{n,2}$ significantly smaller than that of the unknown specimen, $b_{n,1}$, assuming ideal thermal contact conditions (no thermal contact resistance at or near the effusivity probe), the model Formula (4) can be replaced by Formula (8):

$$\Delta T = A + \left(P_0 \sqrt{t - t_c}\right) / \left(\frac{1}{2}\pi^{1/2} A_{cr} \left(b_{n,1} + b_{n,2}\right)\right) \tag{8}$$

where $b_{n,1}$ is the only unknown part of the transient term on the right-hand-side of Formula (8).

It is shown in experiments that this setup has errors of less than 10 % when the backing material is a high-insulation material, and a firm mounting pressure is applied, pressing the probe against the unknown specimen material[5].

The number of situations where measurement error can be controlled with a single-sided setup is limited to a smaller range of thermal effusivities, as compared to the range of thermal effusivities possible to test by a double-sided setup.

As the requirements on the backing material and mounting pressure allows a variable selection of setup, the single-sided setup shall not be used for laboratory measurements whenever a double-sided setup can be prepared. However, for industrial situations of non-destructive quality control testing of a specimen material with identical surface conditions, shape and little internal structural variations, from experiment to experiment, it is important to perform scouting tests of the single-sided probe and compare the estimated results with corresponding results obtained with a double-sided setup. This, in order to assess the performance of the single-sided setup, and if the estimated results from the single-sided setup agrees with estimated results from the double-sided setup.

9 Verification procedures

9.1 Calibration of apparatus

This method is an absolute method, allowing measurements to be performed which are directly traceable to primary SI units (length, time, temperature and voltage). Hence, calibration against reference materials is not required.

A couple of experimental aspects of the setup can be calibrated separately, as listed in ISO 22007-2:2022, 9.1.

9.2 Verification of apparatus

Verification of the apparatus shall be performed periodically, preferably by measuring the thermal effusivity of one or more reference materials (if available) giving values within the range covered by the materials to be tested. If the measured values differ from the reference values by more than the limits specified in Clause 10, recalibration in accordance with 9.1 shall be performed. If no appropriate certified reference materials are available for thermal effusivity, verification can be performed by measurements on materials which have isotropic, well-known and reproducible thermal-transport properties, such as stainless steels, Pyroceram™ 9606 1), Perspex™ 1), and polystyrene.

1) Pyroceram™ 9606 and Perspex™ are examples of suitable products available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of these products.

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10 Precision and bias

10.1 In routine measurements at or around room temperature, the accuracy for thermal effusivity is estimated at 4 % to 10 %. The accuracy at higher temperatures is estimated at 6 % to 12 % for thermal effusivity. The ranges in uncertainty indicated here relate to probes with polyimide insulations (thickness of insulation between 7 µm and 40 µm), different minimum cross-section distances across the probe (from 2 mm to 60 mm) and transient recordings of different durations (from 0,5 s to 1 000 s).

NOTE A covering factor of 2 has been used when estimating the uncertainties from a large number of tests.

10.2 If tests are repeated at the same temperature using the same probe and test equipment, the deviation from the first measurement is very small, since the same TCR, the same probe radius, the same power output, and preferably also the same time window for the transient measurement is used to evaluate the data. In such experiments, the repeatability of the thermal effusivity is between 2 % and 4 %.

11 Test report

In addition to the information required in ISO 22007-1 and ISO 22007-2, the following information shall be supplied:

a) a reference to this document, including the year of publication;

b) thermal effusivity b_n of the specimen tested;

c) relevant details of the specimen, such as specimen geometry, mounting conditions;

d) in case a rod setup (see 7.3) is used, information on the surrounding insulation;

e) in case heat losses through lateral boundaries of a rod setup (see 7.3) can be estimated, an estimation of these heat losses should be presented;

f) a description of the test apparatus, including details of the power supply and digital voltmeter in the bridge circuit;

g) relevant details of the probe, the measurement time, the power output, the scanning rate and the time windows used in the analysis of the test data;

h) deviations from the procedure;

i) unusual features observed;

j) the date of the test.

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Annex A
(informative)

Example of testing a homogeneous, anisotropic rod

A.1 Overview

The following example demonstrates the testing of a rod cut out from a carbon nanotubes epoxy composite specimen.

For this specimen, it is of interest to determine the thermal effusivity (and thermal conductivity) across a sheet of this specimen material.

In order to apply the rod configuration (see 6.1), the specimen is cut into two rectangular pieces with dimensions 21 mm × 18 mm (thickness 3,75 mm). These dimensions were chosen to leave a margin of 1 mm between the rectangular-shaped probe and the specimen edges (dimensions of the rectangular-shaped probe is 18 mm × 16 mm). The probe is sandwiched between two identical pieces of the specimen (see Figure A.1).

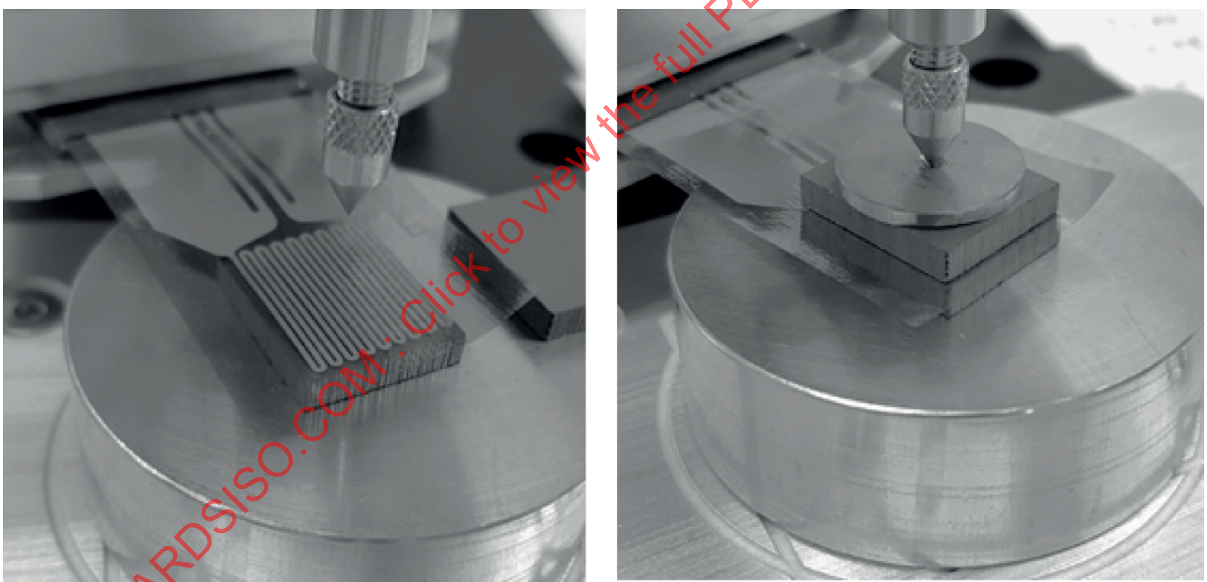

Figure A.1 — Measurement setup for one-dimensional hot square experiment

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A.2 Measurement parameters

— measurement time: 200 data points collected during 0,5 s

— heating power: 4 W

— probe model: Rectangular probe 18 mm × 16 mm cross section

— temperature: 22,4 °C.

— assumed volumetric specific heat capacity: 1,5 $\mathrm{MJ \cdot m^{-3} \cdot K^{-1}}$

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A.3 Results

The properties are measured in normal direction to the probe plane (across the thickness of the specimen). The presented results in Table A.1 are an average of 5 measurements with 20 min waiting time between measurements. Figure A.2 depicts the model fitting for the specific point selection for one of the measurements.

Table A.1 — Results of one-dimensional measurements (normal direction) across a sheet of a carbon nanotubes epoxy composite specimen

Points	Thermal conductivity, $W \cdot m^{-1} \cdot K^{-1}$	Thermal effusivity, $W \cdot s^{1/2} \cdot m^{-2} \cdot K^{-1}$	Probing depth, mm
101-195	9,063	3 687	3,43
St. dev., %	1,3	0,64	

In order to assess the heat losses from lateral surfaces, note that the maximum temperature increase in the specimen is 0,341 K, while the temperature drop across the insulating layer is 3,585 K. The lateral heat losses β (at room temperature conditions) – via combined radiation and thermal convection in air – can be estimated to correspond to around β = 20 W/m^{2}K. Hence, the heat losses can be assumed to be around $Q_{loss} = \beta \text{ x } A \text{ x } (T_{max} - T_0)/2$ = 20 × 0,000 292 5 × 0,341/2 = 0,001 W, while the experimental heating power is 4 watts, hence relative errors due to lateral heat losses is approximately 0,001/4 = 0,03 % for this setup.

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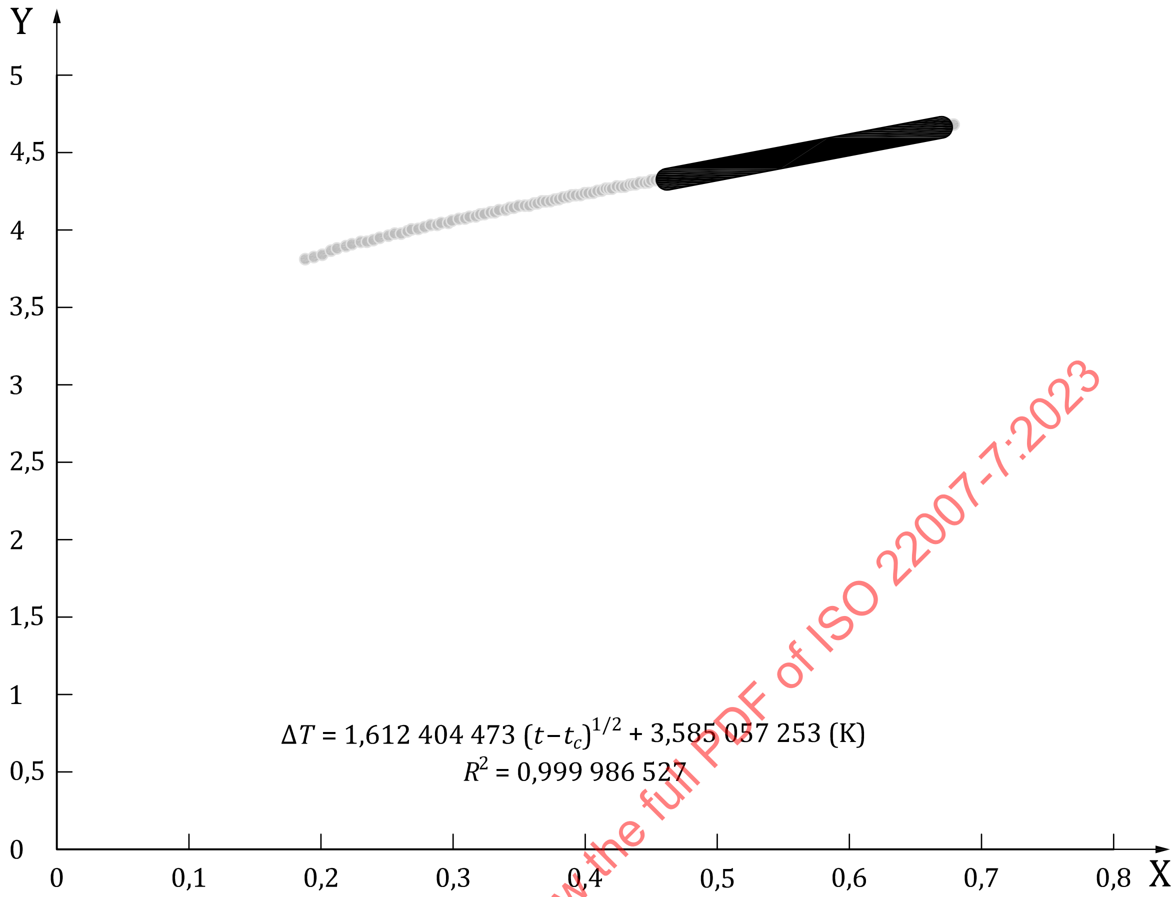

Key

X $(t-t_c)^{\frac{1}{2}}$

Y ΔT

R^2 measure of goodness of fit of linear regression

experimental temperature increase

model-fitted temperature increase

NOTE In this plot, $t_c = t_{c,\text{optimal}}$.

Figure A.2 — Experimental temperature increase versus $\sqrt{t-t_c}$, and model-fitted temperature increase versus $\sqrt{t-t_c}$

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NOTE 1 The model fitting has been performed on the solid black points. The mean deviation from the fitted straight line is 400 μK.

NOTE 2 The result obtained with ISO 22007-2 method required a larger sheet of the specimen (approximately 60 mm × 60 mm). (Surface evenness conditions were not ideal, and the specimen was possibly not fully homogeneous.) With a 20 mm diameter hot disc sensor, slab measurement results arrive at 64,35 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ in-plane (0,25 % St.dev.) thermal conductivity, while anisotropy results indicated 9,080 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ (3,6 % St.dev.) thermal conductivity in the axial direction (comparable to results obtained in Table A.1), while in-plane thermal conductivity was estimated at 62,57 $\text{W}\cdot\text{m}^{-1}\cdot\text{K}^{-1}$ (7,4 % St.dev.).

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