INTERNATIONAL STANDARD

ISO 16525-3

First edition
2014-05-15

Adhesives — Test methods for isotropic electrically conductive adhesives —

Part 3: Determination of heat-transfer properties

Adhésifs — Méthodes d'essai pour adhésifs à conductivité électrique isotrope —

Partie 3: Détermination des propriétés de transfert de chaleur

Reference number
ISO 16525-3:2014(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

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.

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. 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. 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.

The committee responsible for this document is ISO/TC 61, *Plastics*, Subcommittee SC 11, *Products*.

ISO 16525 consists of the following parts, under the general title *Adhesives — Test methods for isotropic electrically conductive adhesives*:

— *Part 1: General test methods*

— *Part 2: Determination of electric characteristics for use in electronic assemblies*

— *Part 3: Determination of heat-transfer properties*

— *Part 4: Determination of shear strength and electrical resistance using rigid-to-rigid bonded assemblies*

— *Part 5: Determination of shear fatigue*

— *Part 6: Determination of pendulum-type shear impact*

— *Part 7: Environmental test methods*

— *Part 8: Electrochemical migration test methods*

— *Part 9: Determination of high-speed signal-transmission characteristics*

Adhesives — Test methods for isotropic electrically conductive adhesives —

Part 3: Determination of heat-transfer properties

SAFETY STATEMENT — Persons using this part of ISO 16525 should be familiar with normal laboratory practice. This part of ISO 16525 does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to ensure compliance with any regulatory conditions.

IMPORTANT — Certain procedures specified in this part of ISO 16525 might involve the use or generation of substances, or the generation of waste, that could constitute a local environmental hazard. Reference should be made to appropriate documentation on safe handling and disposal after use.

1 Scope

This part of ISO 16525 specifies test methods for heat-transfer properties such as effective thermal conductivity and thermal resistance by a steady-state comparative longitudinal heat-flow method (SCHF method) using cartridge-type specimen for isotropic electrically conductive adhesives used in wiring, die attach, and surface assembly.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 13385-1, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 1: Callipers; Design and metrological characteristics*

ISO 13385-2, *Geometrical product specifications (GPS) — Dimensional measuring equipment — Part 2: Calliper depth gauges; Design and metrological characteristics*

ISO 17212, *Structural adhesives — Guidelines for the surface preparation of metals and plastics prior to adhesive bonding*

ISO 80000-1, *Quantities and units — Part 1: General*

IEC 60584-1, *Thermocouples — Part 1: Reference tables*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

NOTE Thermal conductivity of heterogeneous materials is called “apparent thermal conductivity”, “equivalent thermal conductivity” or “effective thermal conductivity”. For those materials whose compositions are nearly homogeneous, “thermal conductivity” can be used if it does not lead to confusion.

3.1
heat flow rate
Q
amount of themal energy transferring from/to one system from/to another in a given time

Note 1 to entry: It is expressed in watts (W).

3.2
heat flow rate over surface area
heat flux
q
amount of themal energy being transferred over a given surface area

Note 1 to entry: It is expressed in watts per square metre (W/m^2).

3.3
heat transfer property
collective term for thermal resistance, thermal conductivity, effective thermal conductivity and thermal conductance

3.4
thermal resistance
R
temperature difference between the front and back sides in the steady-state divided by heat flux

Note 1 to entry: It is expressed in square metre kelvin per watt ($m^2 \cdot K/W$).

Note 2 to entry: This parameter indicates how heat is transferred, with a high value denoting reduced heat transfer.

3.5
thermal conductance
K
heat flux in the steady state divided by the temperature difference between front and back sides

Note 1 to entry: It is expressed in watt per square metre kelvin ($W/m^2 \cdot K$).

Note 2 to entry: The reciprocal of thermal resistance.

3.6
thermal conductivity
k
proportionality coefficient that represents the relationship of heat flux and temperature gradient, where heat flux on an isothermal surface is proportional to the temperature gradient in the normal direction on the isothermal surface

Note 1 to entry: It is expressed in watt per metre kelvin ($W/m \cdot K$).

3.7
effective thermal conductivity
k_{eff}
<system consisting of two substances or more> heat flux in the steady state that is divided by a temperature difference between the front and back sides, and multiplied by the distance between the front and back sides, *L*

Note 1 to entry: It is expressed in watt per metre kelvin (W/m·K).

Note 2 to entry: Effective thermal conductivity includes interfacial thermal resistance.

Note 3 to entry: It is expressed by *K* x *L*.

3.8
interfacial thermal resistance
R_i
specific type of thermal resistance arising on the contact surface of the material (contact thermal resistance), where thermal resistance caused by heat conduction of the filling between a gap and the material is removed

Note 1 to entry: It is expressed in square metre kelvin per watt (m^2·K/W).

Note 2 to entry: It is generated mainly between the surface of the material and the contact surface.

3.9
mean temperature of specimen
T_m
arithmetic mean of temperatures of the high-temperature surface and the low-temperature surface of a specimen in the steady state

Note 1 to entry: It is expressed in kelvin (K).

Note 2 to entry: It is simply called mean temperature.

4 Principle

Effective thermal conductivity of isotropic electrically conductive adhesives is measured by the steady-state temperature distribution in the cartridge-type specimen and standard rods under longitudinal heat-flow condition. The cartridge-type specimen is sandwiched by the standard rods made of square or cylindrical poles with known thermal conductivity.

NOTE This method is called the steady-state comparative longitudinal heat-flow method (SCHF), which uses standard rods to measure heat flux, *q*, to calculate effective thermal conductivity. It is useful for isotropic electrically conductive adhesives whose thermal conductivity varies due to interfacial thermal resistance between the isotropic electrically conductive adhesive and the heating surface.

5 Scope of test

The measurement range of thermal conductivity of an isotropic electrically conductive adhesive is determined by its thermal conductance. The lower and upper limits of thermal conductance are calculated using Formulae (1) and (2), respectively.

$$K_s \geq 2760 t_r^{1,85} w_r^{-1,4} \quad (1)$$

where

$25 \leq t_r \leq 60$, $10 \leq w_r \leq 60$

$$K_s \leq 100\,000 \qquad (k_{eff} > 20)$$
$$K_s \leq 5\,000 k_{eff} \qquad (k_{eff} \leq 20) \qquad (2)$$

where

K_s is the thermal conductance of the isotropic electrically conductive adhesive [W/(m²·K)] (= k_{eff}/t_s);

k_{eff} is the effective thermal conductivity of the isotropic electrically conductive adhesive [W/(m·K)];

t_r is the thickness of standard rods (mm);

t_s is the thickness of the isotropic electrically conductive adhesive (mm);

W_r is the length of the side of the specimen (mm).

EXAMPLE The lower limits of measurable thermal conductance of the isotropic electrically conductive adhesive with t_r of 25 mm are shown below:

a) 2 000 W/(m²·K), when W_r = 10 mm;

b) 800 W/(m²·K), when W_r = 20 mm;

c) 400 W/(m²·K), when W_r = 30 mm;

d) 200 W/(m²·K), when W_r = 60 mm.

NOTE The upper limit of measureable thermal conductance of the isotropic electrically conductive adhesive is shown below:

a) 100 000 W/(m²·K), when k_{eff} = 50 W/(m·K);

b) 100 000 W/(m²·K), when k_{eff} = 30 W/(m·K);

c) 100 000 W/(m²·K), when k_{eff} = 20 W/(m·K);

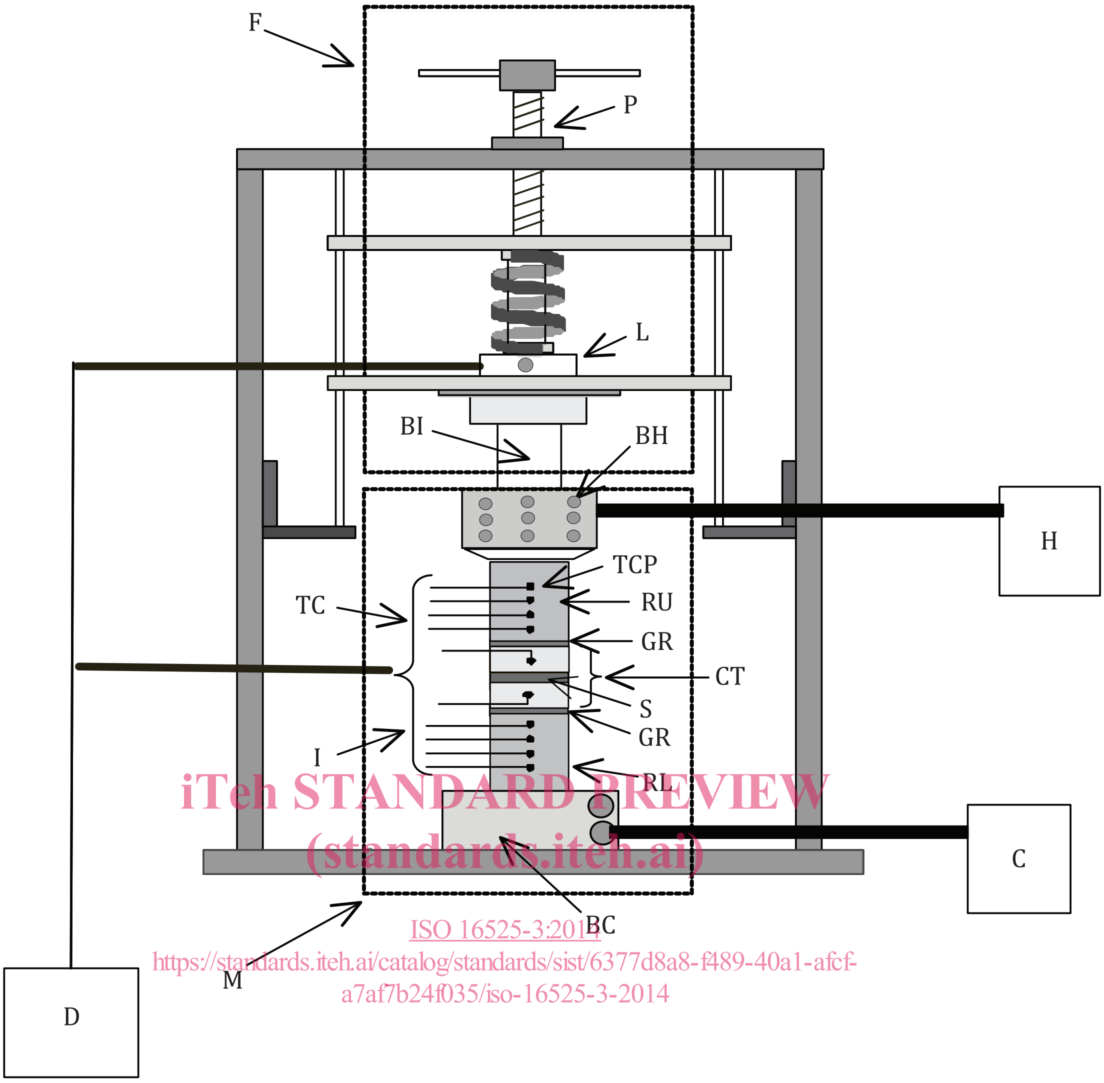
d) 50 000 W/(m²·K), when k_{eff} = 10 W/(m·K);

e) 5 000 W/(m²·K), when k_{eff} = 1 W/(m·K);

f) 2 500 W/(m²·K), when k_{eff} = 0,5 W/(m·K).

6 Apparatus

6.1 Apparatus (general), consisting of measuring, heating, cooling and pressure-regulating devices. The cartridge-type specimen is sandwiched between the standard upper and lower rods with known thermal conductivity. Heat flows through the specimen longitudinally. An example of the test apparatus is shown in Figure 1.



Key

BC cooling block
BH heating block
BI insulating block
CT cartridge-type specimen
C cooling equipment
D data acquisition device
F pressure adjustment unit
GR thermal grease
H heating equipment
L load cell
M measurement system
P screw for pressure regulation
RL lower rod
RU upper rod
S isotropic electrically conductive adhesives
TC thermocouple
TCP holes for temperature measurement
I thermal insulator

Figure 1 — Example configuration of thermal conductivity test apparatus by SCHF method

6.2 Measurement system (M), consisting of a specimen (S), upper and lower rods (RU and RL), a heating block (BH) with a built-in heater, a cooling block (BC) and a thermal insulator (I), which covers these components. The upper/lower rods (RU/RL) and the specimen have thermocouple holes (TCPs) in which thermocouples (TCs) are inserted to measure the temperature gradient. The depth of each thermocouple hole is no smaller than 5 mm.

The thermocouples are to measure the temperature distribution of the upper/lower rods (RU/RL) and specimen (S). Requirements for thermocouples are described below. When using thermoelectric devices, i.e. thermometers instead of thermocouples, they shall be equivalent to or surpass thermocouples in terms of performance.

a) They shall be stable up to the temperature specified in IEC 60584;

b) They shall have sufficient strength to be able to detect small fluctuations in temperature in a range where thermoelectromotive force does not change, and they shall have variance in the temperature displayed among thermocouples at ±0,5 K.

6.3 Heating and cooling equipment (C, H).

a) **Heating equipment** (H), supplying the heater of the heating block (BH) in the measurement system (M) with power; its temperature fluctuation shall be within ±0,5 K.

b) **Cooling equipment** (C), refrigerating the cooling block (BC) in the measuring system (M). The cooling equipment shall guarantee that the temperature of the specimen is within the range of room temperature to 125 °C and shall be able to control fluctuation of temperature of the specimen within ± 0,5 K.

6.4 Pressure adjustment unit (F), pressurizing the measurement system (M) to measure its applied force. It is composed of a pressure adjustment screw (P), load cell (L) and insulating block (BI).

6.5 Data acquisition device (D), recording steady-state temperature of the upper/lower standard rods (RU/RL) and cartridge-type specimen (CT), and applied force of the load cell (L).

7 Cartridge-type specimen

7.1 The cartridge-type specimen

The shape of the cartridge-type specimen is a square column. An example of the cartridge-type specimen is shown in Figure 2. It consists of an isotropic electrically conductive adhesive sandwiched by metal blocks with three holes each for temperature measurement.

7.2 Dimensions and tolerances of the cartridge-type specimen

Dimensions and tolerance of the specimen are specified as follows:

a) Length of the side of the specimen and its measurement: the range of the length of the side and its measurement are as follows:

1) The length of the side of the specimen shall be within the range of 10 mm to 60 mm, and its tolerance shall be ±0,05 mm.

NOTE Measurable heat conductance of a cartridge-type specimen can have a lower limit, depending on the length of its side (see Clause 5).

2) To measure the length of the side of the specimen, use vernier callipers with a precision of 0,05 mm as specified in ISO 13385-1 or those which are equivalent or higher in terms of precision.

b) Cross-section: the cross-sectional area shall be obtained by multiplying the length of one side and the length of the other side.

c) Thickness and its measurement: the range of thickness and its measurement are specified as follows.

1) The thickness of isotropic electrically conductive adhesives shall be within the range of 0,2 mm to 5 mm, and its lower limit is determined by Formula (3).

$$\begin{aligned} t_s &\geq 0{,}01 k_{eff} \quad (k > 20) \\ t_s &\geq 0{,}2 \quad (k_{eff} \leq 20) \end{aligned} \tag{3}$$

EXAMPLE The lower limits of the isotropic electrically conductive adhesive are shown below:

— 0,5 mm, when k_{eff} = 50 W/(m·K);

— 0,3 mm, when k_{eff} = 30 W/(m·K);

— 0,2 mm, when k_{eff} = 20 W/(m·K);

— 0,2 mm, when k_{eff} = 10 W/(m·K);

— 0,2 mm, when k_{eff} = 1 W/(m·K);

— 0,2 mm, when k_{eff} = 0.5 W/(m·K).

where the tolerance is ±0,010 mm.

The accuracy of the measurement value of the thermal conductance depends on the measurement tolerances of the thickness with respect to the isotropic electrically conductive adhesive. When the measurement parameter is thermal resistance only, or the tolerance is lower than 0,010 mm, the limit of thickness is less than shown in the Note above.

When the measurement parameter is not thermal resistance, which includes interfacial resistance between specimen block and an isotropic electrically conductive adhesive, the hot disc method, the temperature wave analysis method, and the laser flash method (see ISO 22007-2, ISO 22007-3 and ISO 22007-4) are available for the thinner isotropic electrically conductive adhesive layers. The accuracy depends on the thickness tolerance. For example, if the thickness of the isotropic electrically conductive adhesive is 0,03 mm, the tolerance should be ±1,0 µm.

2) The thickness of the specimen block is within the range of 2 mm to 5 mm, and its tolerance is ±0,010 mm.

3) The thickness of the isotropic electrically conductive adhesive is obtained by subtracting the thickness of the specimen blocks from that of the cartridge-type specimen.

4) Measure thickness at five points (the centre and four corners) using micrometer callipers with a precision of 0,001 mm as specified in ISO 3611 or those which are equivalent or higher in terms of precision.

d) Surface roughness and flatness of the adherend of the specimen blocks and parallelism of the upper/lower faces: surface roughness and flatness of the adherend between the specimen blocks and the isotropic electrically conductive adhesive and parallelism of the upper/lower faces are specified as follows:

1) surface roughness of the adherend, *Ra*, as specified in ISO 4287, shall be no greater than 5 µm;

2) flatness of the adherend and parallelism of the upper/lower faces shall be both no greater than 5 µm.

e) Types of specimen blocks: the specimen blocks shall be manufactured from a metal of known thermal conductivity.

