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Adhesives — Test methods for isotropic electrically conductive adhesives —

Part 1:
General test methods

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

Partie 1: Méthodes d'essai générales

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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

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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.

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 1: General test methods

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 general test methods for isotropic electrically conductive adhesives used in wiring, die attach of semiconductors, and surface assembly of printed circuit boards.

This part of ISO 16525 does not specify the materials and the performance of the isotropic electrically conductive adhesives. This part of ISO 16525 does not specify the performance of the test pieces bonded by using the adhesive concerned.

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 62, *Plastics — Determination of water absorption*

ISO 291, *Plastics — Standard atmospheres for conditioning and testing*

ISO 472, *Plastics — Vocabulary*

ISO 1183-1, *Plastics — Methods for determining the density of non-cellular plastics — Part 1: Immersion method, liquid pyknometer method and titration method*

ISO 2528, *Sheet materials — Determination of water vapour transmission rate — Gravimetric (dish) method*

ISO 2555, *Plastics — Resins in the liquid state or as emulsions or dispersions — Determination of apparent viscosity by the Brookfield Test method*

ISO 3219, *Plastics — Polymers/resins in the liquid state or as emulsions or dispersions — Determination of viscosity using a rotational viscometer with defined shear rate*

ISO 4664-1, *Rubber, vulcanized or thermoplastic — Determination of dynamic properties — Part 1: General guidance*

ISO 10364, *Structural adhesives — Determination of the pot life (working life) of multi-component adhesives*

ISO 11357-2, *Plastics — Differential scanning calorimetry (DSC) — Part 2: Determination of glass transition temperature and glass transition step height*

ISO 11359-2, *Plastics — Thermomechanical analysis (TMA) — Part 2: Determination of coefficient of linear thermal expansion and glass transition temperature*

ISO 15184, *Paints and varnishes — Determination of film hardness by pencil test*

ISO 15605, *Adhesives — Sampling*

EN 923, *Adhesives — Terms and definitions*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 923 and ISO 472 and the following apply.

3.1
isotropic electrically conductive adhesive
adhesive consisting of conductive fillers that provide electrical conduction and resin that serves for adhesion

3.2
cure schedule
T_C
period of time necessary for an adhesive in an assembly or for a polymer composition to cure under specified conditions of temperature or pressure, or both

Note 1 to entry: It is expressed in minutes (min).

Note 2 to entry: Pot life is also expressed as "working life"'; however this part of ISO 16525 uses "pot life" exclusively in accordance with ISO 10364.

4 Lists of test requirements

4.1 Tests for properties before curing

a) Appearance

b) Density

c) Viscosity

4.2 Tests for conditions of use

a) Pot life

b) Curing conditions

4.3 Tests for properties after curing

a) Glass transition temperature

b) Moisture absorption ratio

c) Modulus of elasticity

d) Surface hardness

e) Thermal expansion

f) Moisture permeability

5 Test conditions and sampling

5.1 Atmospheric conditions

Carry out all any tests under the standard conditions [temperature (23 ± 2) °C; relative humidity (50 ± 10) %] specified in ISO 291 unless otherwise specified.

5.2 Conditioning of the specimens

Specimens manufactured at least 12 h before testing shall be used. Condition them under the atmospheric conditions specified in 5.1 for 1 h or longer before testing.

5.3 Definition of lot

"One lot" refers to a set of products that are regarded as having identical condition because they were manufactured in one batch or manufactured under identical conditions.

5.4 Sampling

For sampling, comply with ISO 15605.

6 Tests for properties before curing/drying

6.1 Appearance

Carry out visual inspection, and record the colours and viscosity ("high" or "low") of the specimens collected.

6.2 Density

For measurement of the density of specimens, use the equipment and the procedure specified in ISO 1183-1.

6.3 Viscosity

For measurement of the viscosity of specimens, use the equipment and the procedure specified in ISO 2555 and ISO 3219.

7 Tests for conditions of use — Pot life

For measurement of the pot life of specimens, use the equipment specified in ISO 10364.

8 Test for properties after curing

8.1 Glass transition temperature, T_g

For measurement of the glass transition temperature, T_g, of specimens, use the equipment and the procedure specified in ISO 11357-2.

8.2 Moisture absorption ratio

For measurement of the moisture absorption ratios, ISO 62 shall be used. When calculating a moisture absorption ratio from vapour, use the following equipment and procedure.

8.2.1 Apparatus

8.2.1.1 Scales, of accuracy to within ±0,1 mg.

8.2.1.2 Oven, forced ventilation type, which allows temperature of (50 ± 2) °C or the temperature agreed between the delivering and receiving parties.

8.2.1.3 Humidity chamber, forced ventilation type, which allows temperature and humidity of (85 ± 2) °C and (85 ± 5) %, respectively, or as agreed between the delivering and receiving parties.

8.2.1.4 Means for measuring dimensions, to an accuracy of ±0,1 mm.

Prepare three specimens or more for each test. To achieve the desired dimensions, die forming may be used. Record the method used to prepare specimens and their dimensions in a test report.

8.2.2 Procedure

Dry all specimens for (24 ± 1) h using an oven with temperature set at 50 °C. Take the specimens out of the oven and, within 1 min, weigh them with a precision to 0,1 mg. These measurements are referred to as initial mass, m_1. Condition the specimens using a humidistat bath with temperature and humidity set at 85 °C and 85 %, respectively. Take the specimens out of the humidistat bath at selected of time periods. The representative test periods are 24 h, 48 h, 96 h and 192 h. Measure their mass, m_2, within 1 min. Put the specimens back into the humidistat bath. Repeat this procedure until the mass measurements are constant.

8.2.3 Displaying results

Calculate the moisture absorption ratio of each specimen to its initial mass:

$$C = \frac{m_2 - m_1}{m_1} \times 100 \quad (1)$$

where

C is the moisture absorption ratio (%);

m_1 is the mass of specimen after initial drying (mg);

m_2 is the mass of specimen after moisture absorption (mg).

8.3 Modulus of elasticity

For measurement of the modulus of elasticity, use the method specified in ISO 4664-1. For displaying the results, either static modulus of elasticity or dynamic modulus of elasticity may be used.

8.4 Surface hardness

For measurement of the surface hardness, use a pencil hardness tester or penetrometer specified in ISO 15184.

8.5 Thermal expansion

For measurement of the thermal expansion, use the equipment specified in ISO 11359-2. Calculate results using the compression mode of thermomechanical analysis (TMA).

8.6 Moisture permeability

For measurement of the moisture permeability, use the equipment and the procedure specified in ISO 2528.

9 Test report

The test report shall contain the following items. Some items may be selected from items b) to f) upon agreement between the delivering and receiving parties:

a) a reference to this part of ISO 16525, i.e. ISO 16525-1;

b) the name of the isotropic electrically conductive adhesive and its data, including the kinds of resin, material of fillers, manufacturer code, lot number and others;

c) types and preparation of specimens;

d) the number of specimens;

e) the date, institution and atmospheric conditions of the test;

f) conditions of each test.

Bibliography

[1] ISO 554, *Standard atmospheres for conditioning and/or testing — Specifications*

[2] ISO 2811, *Paints and varnishes — Determination of density*

[3] ISO 3251, *Paints, varnishes and plastics — Determination of non-volatile-matter content*

[4] EN 1245, *Adhesives — Determination of pH — Test method*

[5] EN 12092, *Adhesives — Determination of viscosity*

[6] ASTM E96/E96M,*Standard Test Methods for Water Vapour Transmission of Materials*

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