INTERNATIONAL STANDARD

ISO 16525-6

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Adhesives — Test methods for isotropic electrically conductive adhesives —

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

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

Partie 6: Détermination de la résistance au choc du type pendule

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing Part of ISO 16525s 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

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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 6: Determination of pendulum-type shear impact

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 the pendulum-type test methods for impact strength of isotropic electrically conductive adhesives used in mounting components products on substrates.

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

ISO 472, *Plastics — Vocabulary*

ISO 10365, *Adhesives — Designation of main failure patterns*

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*

3 Terms and definitions

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

3.1
impact strength
ability of a material to resist shock loading

3.2
pendulum-type impact test
test using apparatus consisting of a pendulum swinging at a sample of material

3.3
accelerometer
pendulum-type device that applies impact force to a specimen

3.4
displacement sensor
instrument to measure the movement distance of the hammer in an impact test

3.5
total impact energy
E
total energy required to break a specimen in an impact test

Note 1 to entry: It is expressed in joule (J).

3.6
maximum force
L
maximum force generated during an impact test

Note 1 to entry: It is expressed in Newton (N).

3.7
maximum impact energy
I
energy to reach the maximum impact force

Note 1 to entry: It is expressed in joule (J).

4 Principle

This part of ISO 16525 specifies procedures for measuring the impact properties of adhesively bonded joints manufactured with isotropic electrically conductive adhesives by pendulum-type impact test.

An apparatus, which consists of a pendulum head swinging at a sample of material, is used. The energy transferred to the material is measured by an accelerometer installed in the pendulum head or hammer.

The total impact energy is calculated by measuring the passing speed of the hammer.

5 Test apparatus and circuit

5.1 Basic performance of the impact tester

The impact tester shall meet the following requirements.

a) potential energy of the pendulum: E_P = 0,1 J ± 5 %;

b) hammer speed: v_1 = 1,0 m/s to 1,2 m/s.

5.2 Structure of the hammer

A pendulum-type hammer consists of a striker (see Figure 1), which applies impact force to the specimen, and an arm, which connects the rotating shaft and the striker. The structure of the arm shall make the elastic deformation energy loss of the arm negligible.

The striker shall be manufactured using durable materials, which are unlikely to be damaged when the specimen is broken. To calculate the energy accurately, the centre of gravity should be as close to the point of impact as possible. It is preferable to use a material with high specific gravity (for example tungsten).

Dimensions in millimetres

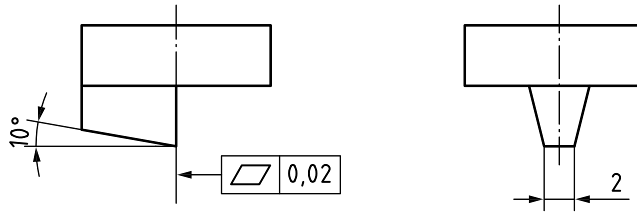


Figure 1 — Form of the striker

5.3 Measurement with the hammer

To calculate the potential energy, E, measure the vertical force, F_H, and the distance, L_H, from the point of impact.

Measure F_H with the hammer raised so that it is perpendicular to the rotation shaft after the tester is assembled.

Use Vernier callipers or a height gauge to measure L_H from the point of impact.

Use measurement devices of sufficient accuracy such that precision of the measurements is within ±1 % of each result.

Calculate the potential energy, E_P, in joules, of the pendulum using Formula (1):

$$E_P = F_H \cdot L_H (1 - \cos\alpha) \quad (1)$$

where

α is the initial arm angle to vertical.

Calculate the hammer speed, v_1, (m/s) using Formula (2):

$$v_1 = 4{,}43\sqrt{L_H(1 - \cos\alpha)} \quad (2)$$

where

α is the initial arm angle to vertical.

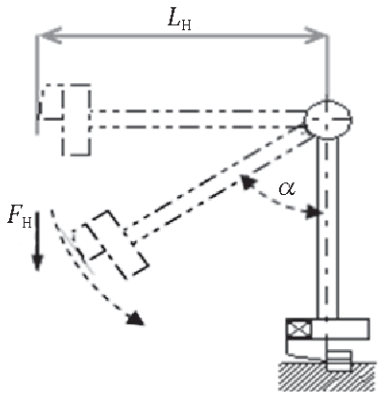


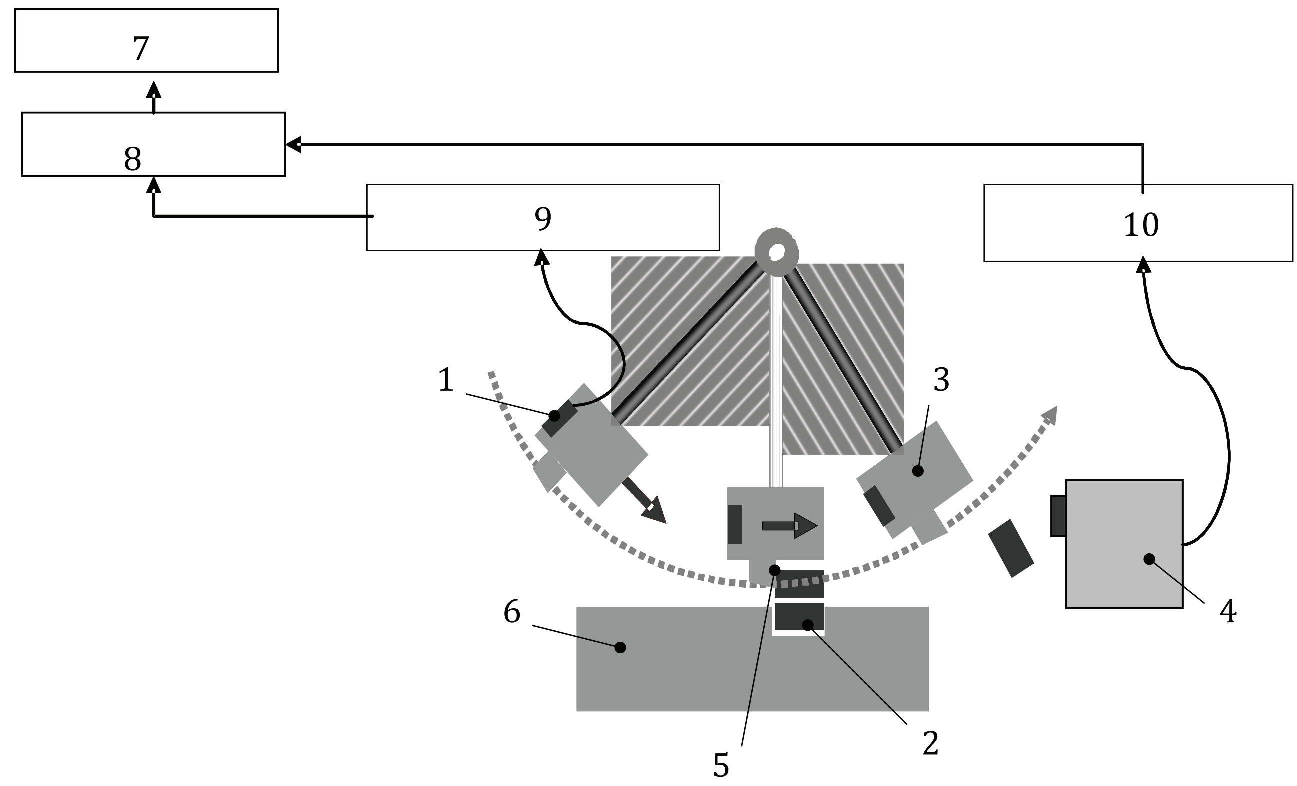
Figure 2 — Measurements necessary for calculation of potential energy

5.4 Scope of test

The angle, α, of the hammer arm can be set arbitrarily as shown in Figure 2. The total impact energy, E, of the specimen is less than 80 % of the initial potential energy, E_P. Note that the hammer speed is 1 m/s or greater.

5.5 Structure of the tester

The structure of the tester is shown in Figure 3.



Key

1 accelerometer
2 specimen
3 hammer
4 displacement sensor
5 impact position
6 specimen-fixing stage
7 PC
8 AD converter
9 accelerometer charge amp.
10 displacement sensor amp.

Figure 3 — Structure of the tester

5.6 Specimen-fixing stage

The specimen-fixing stage should be stable so that it does not vibrate or release the specimen upon impact. The specimen-fixing stage should be equipped with a micromanipulator to adjust the specimen in the X-, Y-, and Z-directions. It is also preferable that it has a mechanism to adjust the centre of the striker face to the centre of the specimen face to be hit.

5.7 Measurement instruments

5.7.1 Accelerometer

Measure the acceleration of the hammer at the time of impact using the accelerometer. Fix the accelerometer near the point of impact. Choose an accelerometer with a mass that does not influence the mass of the hammer.

The mass of the accelerometer should be less than 1 % of the mass of the hammer.

When selecting an accelerometer, assume that the maximum acceleration generated during an impact test is 100 G. Sampling frequency should preferably be 100 kHz at least.

5.7.2 Displacement sensor

Measure the speed of the hammer using the displacement sensor. Use a non-contact type, such as a laser-positioning sensor. Install the displacement sensor within 5 mm of the point of impact of the hammer.

Resolution of measurement should not exceed 1/100 mm and sampling frequency should be 25 kHz or higher.

5.7.3 Observation instruments

Before carrying out a test, ensure that the specimen and the tip of the striker are at the centre.

Use an instrument with 5x magnification or higher to facilitate accurate location.

5.7.4 Hammer releasing mechanism

The hammer releasing mechanism should be automatic (for example electromagnet-based) so that it does not influence the hammer speed. The rotation shafts of the swing arm and the hammer arm should preferably be independent so as to reduce mechanical friction. Measure the speed of wide swing 10 times beforehand using the built-in displacement sensor. The range of 10 measurements should be within 2 %.

6 Specimen

6.1 Form of specimens

Specimens should consist of the following layers: a square adherend, an isotropic electrically conductive adhesive, and a square adherend. An example of specimens is shown in Annex A.

6.2 Dimensions and tolerances of specimens

Dimensions and tolerances of specimens are specified as follows.

6.2.1 Length of the side and its measurement

a) The length of the side is in the range of 2 mm to 3 mm; its tolerance shall be within ±0,5 mm.

b) To measure the length of the side, use Vernier callipers with a precision of 0,05 mm, as specified in ISO 13385-1 and ISO 13385-2, or equivalent in terms of precision.

6.2.2 Thickness and its measurement

The range of thickness and its measurement are specified as follows.

a) The thickness of a specimen column base is 0,8 mm and its tolerance is within ±0,1 mm. The thickness of an isotropic electrically conductive adhesive is 0,1 mm and its tolerance is within ±0,02 mm.

b) To measure the thickness of the side, use Vernier callipers with a precision of 0,02 mm, as specified in ISO 13385-1 and ISO 13385-2, or equivalent in terms of precision.

6.3 Standard atmospheric conditions

The test atmospheric conditions should, in principle, be standard temperature class 2 (23 °C ± 5 °C) specified in ISO 291. Alternative atmospheric conditions may be used upon mutual agreement between the delivering and receiving parties. In such cases, record the temperature used in a test report.

7 Operation

7.1 Loading of specimen and position of impact

When placing a specimen on the specimen-fixing stage, ensure that there is no gap between the stage and the bottom of the specimen. Adjust the specimen and the centre of impact within 1/10 of the dimensions of the specimen. Before starting a test, confirm that the physical relationship between the fixed side of the specimen and the specimen-fixing stage does not allow them to interfere with each other.

7.2 Conformance of specimen

Check the fracture mode after testing to judge whether or not the specimen contained exceptional defects, such as large voids or inclusions.

Note For preparation of a specimen, see Annex A.

8 Calculation of impact strength

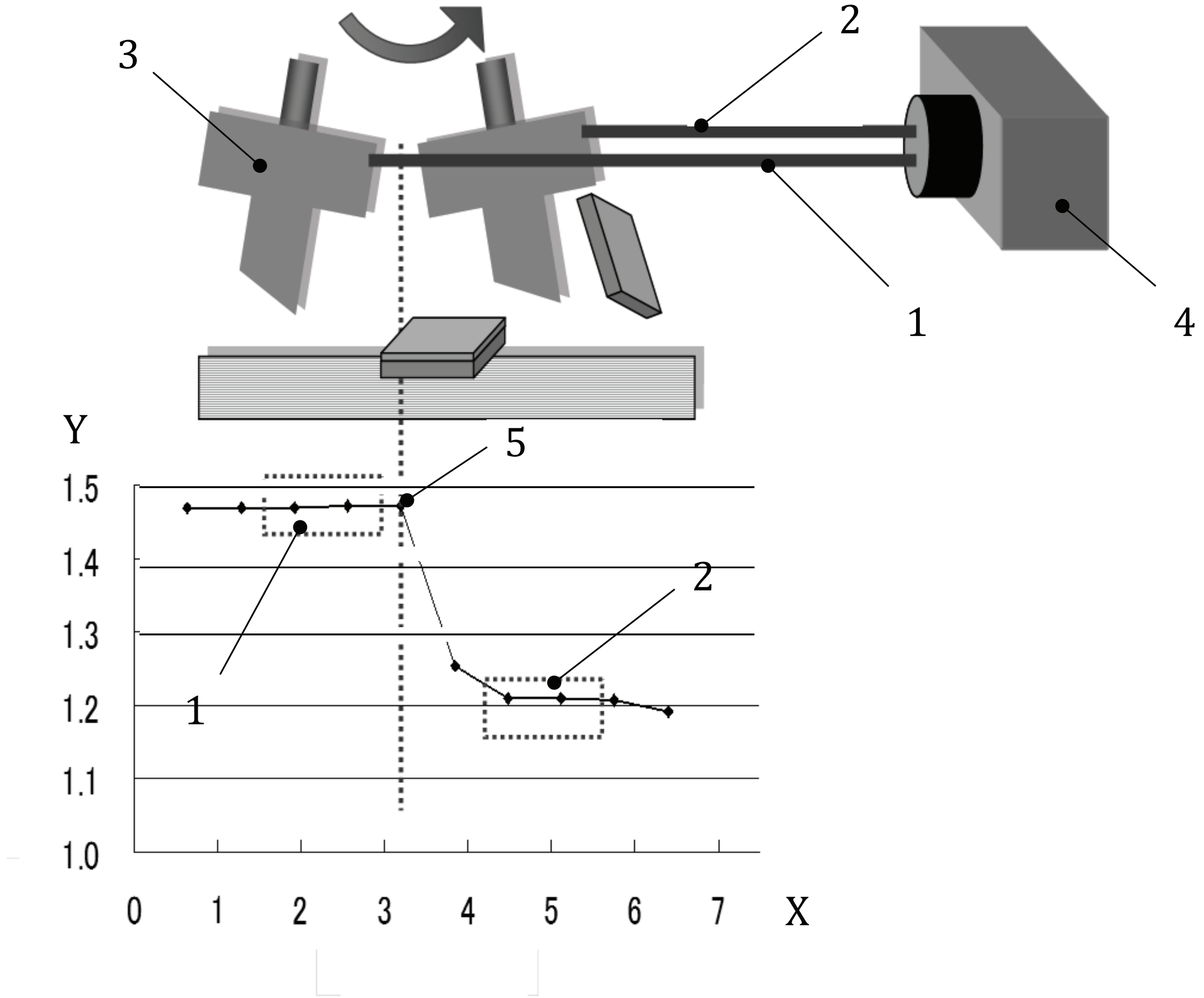
8.1 Calculation — General

The calculation of impact strength is specified as follows.

8.2 Calculation of total impact energy

The calculation of total impact energy is specified as follows.

Measure the travelling time and distance of the hammer before and after the impact using a laser-displacement sensor and determine the speed by differentiating the travelling distance over time. Based on the speed before and after the impact and mass of the hammer, and Formula (1), calculate total impact energy, E, using Formula (3). Figure 4 shows an example of measurement. For correction of measurements, see Annex B.



Key

X time (m/s)

Y hammer speed (m/s)

1 speed after impact

2 speed before impact

3 hammer

4 displacement sensor

5 impact position

Figure 4 — Example of measurement using a laser-displacement sensor

$$E = \frac{1}{2} \cdot m \cdot v_1^2 - \frac{1}{2} \cdot m \cdot v_2^2 \quad (3)$$

where

E is the total impact energy (J);

v_1 is the speed immediately before impact (m/s);

v_2 is the speed immediately after impact (m/s);

m is the mass of the hammer (kg).

8.3 Calculation of maximum force and maximum impact energy

The calculations of the maximum force and maximum impact energy are specified as follows.

a) Measure the travelling time and acceleration of the hammer using an accelerometer (see Figure 5) and calculate loads using Formula (4). Among those calculated, let the maximum load be the maximum force.

$$N = m \cdot a \quad (4)$$

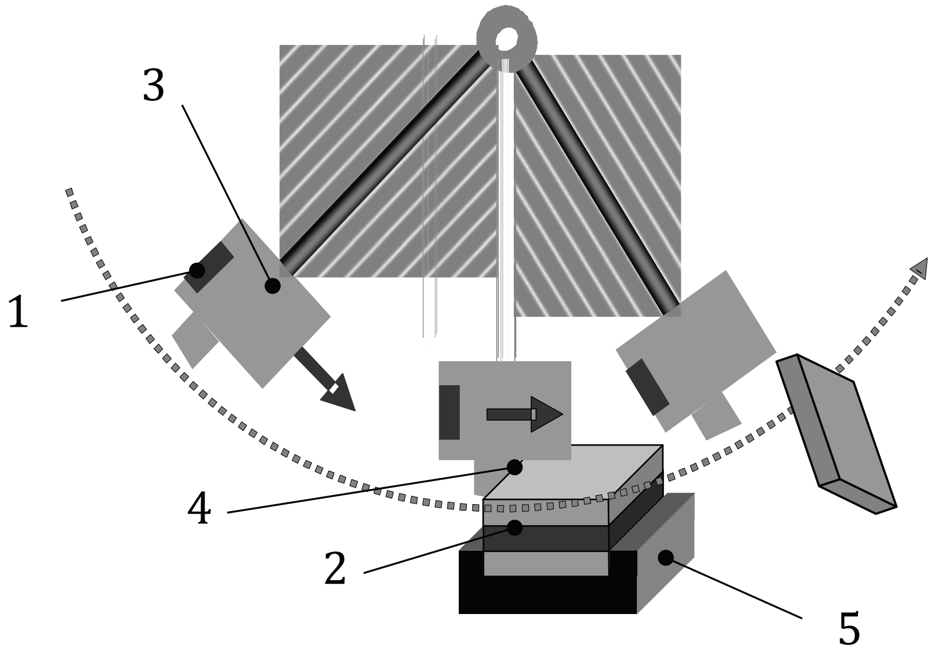
where

N is the load (kg·m/s^2);

m is the mass of hammer (kg);

a is the acceleration (m/s^2).

b) Determinion of the maximum impact energy: determine the displacement by integrating twice the mass, m, of the hammer and measured acceleration, a, over time, and integrate loads up to the maximum force over displacement (see Figure 6).



Key

1 accelerometer

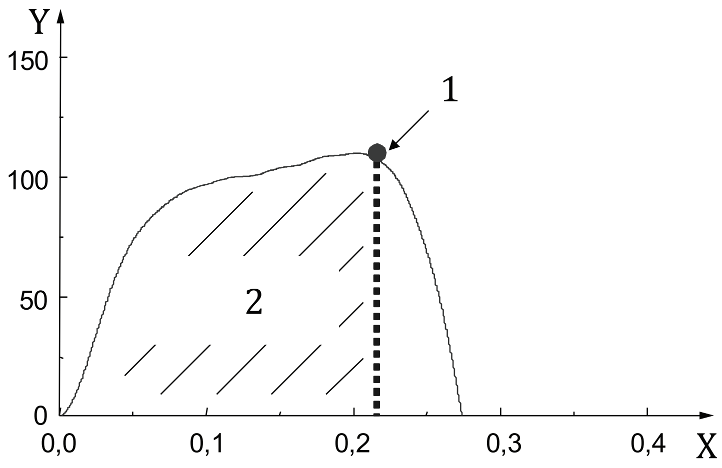
2 specimen

3 hammer

4 impact position

5 specimen-fixing stage

Figure 5 — Example of measurement using an acceleration sensor



Key

X displacement (mm)

Y force (N)

1 maximum force

2 maximum impact energy

Figure 6 — Example of load-displacement curve

8.4 Checking the fracture mode

Check the fracture after the test and determine the fracture mode in accordance with ISO 10365.

9 Test report

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

a) a reference to this part of ISO 16525, i.e. ISO 16525-6;

b) name of the isotropic conductive adhesive and its data, including kinds of resin, filler material, manufacturer’s code, and lot number;

c) assembly procedure of the specimens (curing temperature, time, and pressure of the adhesive procedure);

d) dimensions (length of side) and the number of specimens;

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

f) total impact energy, maximum force and maximum impact energy;

g) conditions used for calculation of total impact energy, maximum force and maximum impact energy (for example mass of hammer and acceleration).

Annex A
(normative)

Example of preparation of specimens

A.1 Procedure for preparation of three-layer specimens

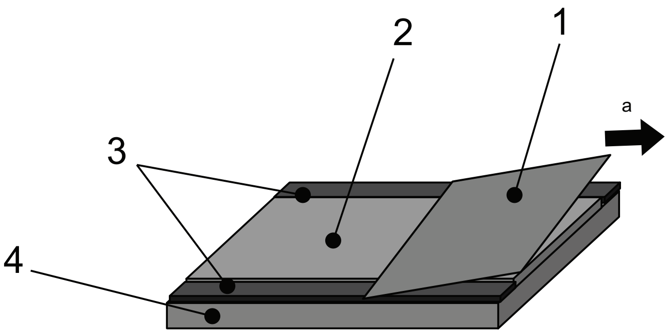
A.1.1 Procedure — General

The preparation of specimens is specified as follows.

A.1.2 Application of an isotropic electrically conductive adhesive

Application of an isotropic electrically conductive adhesive is specified as follows.

a) Use two plates whose length, width and thickness are 30 mm, 20 mm and 0,8 mm, respectively, as bases. Grind them using No. 1 500 and No. 4 000 sandpapers. Place a 0,1 mm spacer on them and apply an isotropic electrically conductive adhesive. An example is shown in Figure A.1.



Key

1 squeegee

2 isotropic electrically conductive adhesive

3 spacer

4 (Cu) copper

a Direction of application.

Figure A.1 — Application of isotropic electrically conductive adhesive

b) Remove the spacer from one of the bases and laminate the bases.

A.1.3 Curing of the assembly

The curing of the assembly is specified as follows:

a) place a 200 g weight on the three-layer structure specified in Figure A.2 and put the assembly into an oven;

b) cure in accordance with the manufacturer's recommendations for the isotropic electrically conductive adhesive;

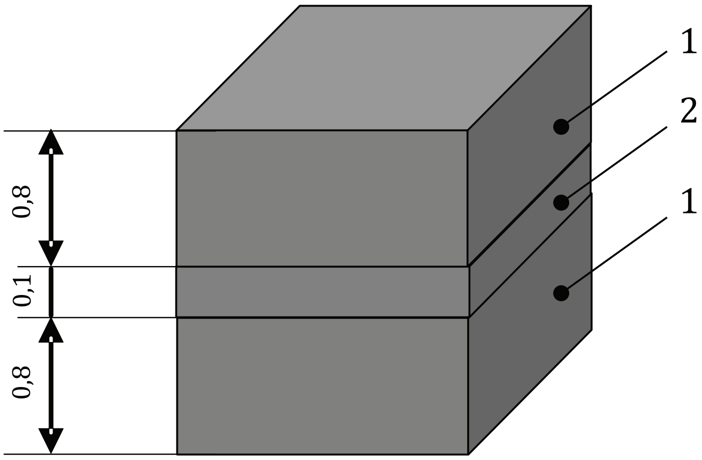
c) after curing, leave the assembly at room temperature until its temperature decreases to room temperature.

A.1.4 Cutting the assembly into specimens

The cutting of the assembly into specimens is specified as follows.

a) Cut the assembly into pieces whose dimensions are specified in 6.2.1 a). Figure A.2 shows an example.

Dimensions in millimetres



Key

1 Cu

2 isotropic electrically conductive adhesive

Figure A.2 — Example of impact test specimen

b) Decide on the size of the specimens based on the following factors.

1) Minimum size: the sizes of the specimens should be large enough to avoid the influence of cutting damage. Cut the assembly into pieces in three or more different sizes and carry out 10 shear tests, which are described in JEITA ET 7409/102:2005, on each specimen. The range in which the strength per bonded area is constant is a range in which there is no influence of cutting load. If one of the specimens breaks during a series of tests, or if one of the specimens is found to have strength per bonded area that is smaller compared to specimens in the other sizes, the specimen is said to have been influenced by the cutting load. Therefore, specimens should be larger than the broken or influenced specimen.

2) Maximum size: the sizes of the specimens should allow them to be broken by the hammer in an impact test. If the observed energy equals or exceeds 80 % of the initial potential energy of the hammer, ignore the measurements. Therefore, determine the maximum size by carrying out an impact test 10 times on each of the specimens in different sizes and measuring energy of each specimen.

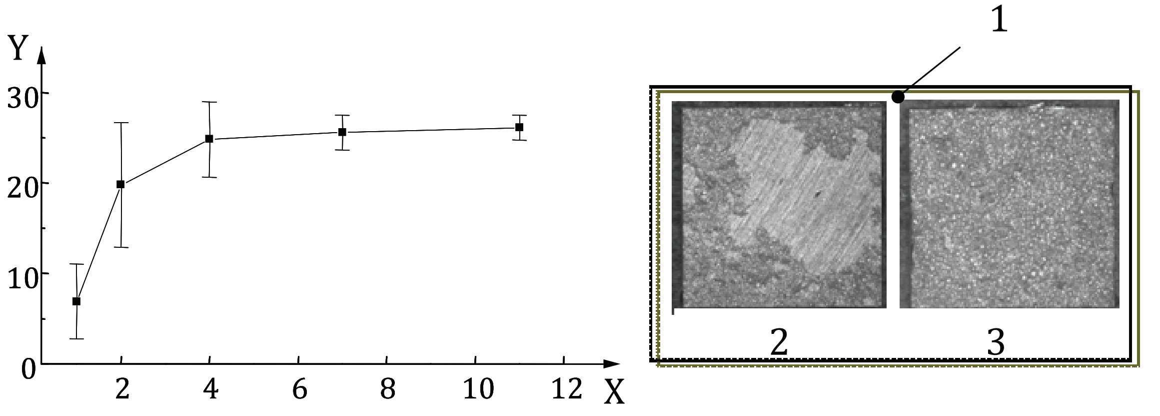
A.1.5 Drying the specimens

Drying of specimens is specified as follows:

a) Dry specimens at 100 °C for 2 h or air dry in the atmosphere for seven days to remove moisture absorbed in the isotropic electrically conductive adhesive when specimens are cut.

b) Check the relationship between the number of days of air drying and shear strength.

If the specimens are not dried perfectly, there will be great error in their strength, and the fracture mode will be complicated, as shown in Figure A.3.



Key

X time (day)

Y shear strength (MPa)

1 fracture surface (optical microscope)

2 one day

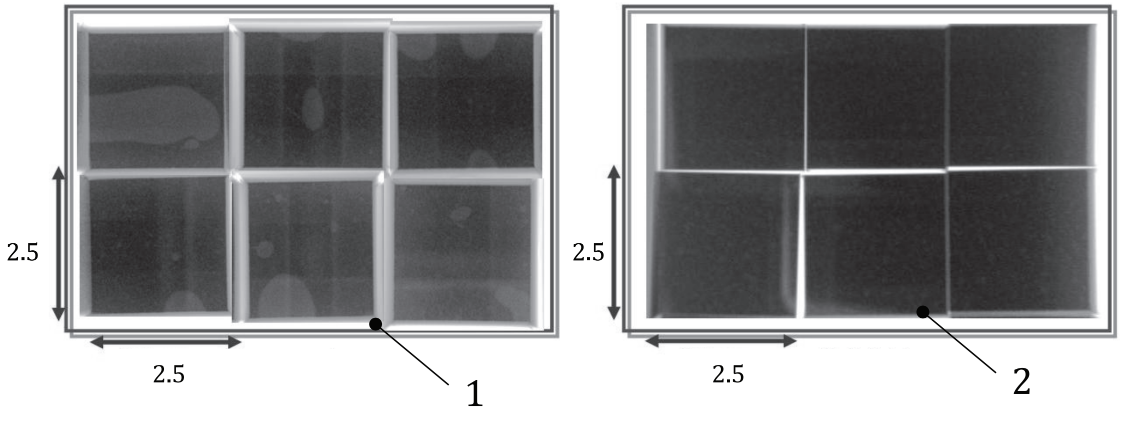
3 seven days

Figure A.3 — Drying of specimens

c) Checking the bonding: to check the bonding of the specimens, use X-ray transmission imaging.

Use only specimens with few voids (less than 10 voids or 20 % in area) as shown in Figure A.4.

Dimensions in millimetres



Key

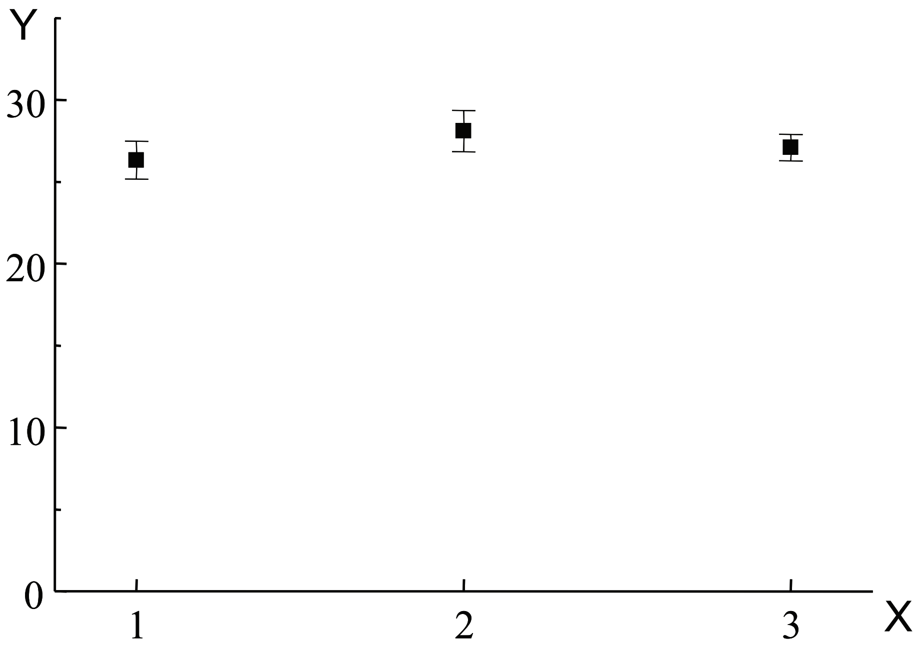
1 many voids

2 fewer voids

Figure A.4 — X-ray image of an isotropic electrically conductive adhesive

A.1.6 Reproducibility

To confirm the reproducibility of the preparation of specimens, cut an assembly into 10 specimens and carry out a shear test three times on them. Judge whether the preparation of specimens is reproducible or not according to results of the tests. See Figure A.5.



Key

X number of times: preparation of specimens

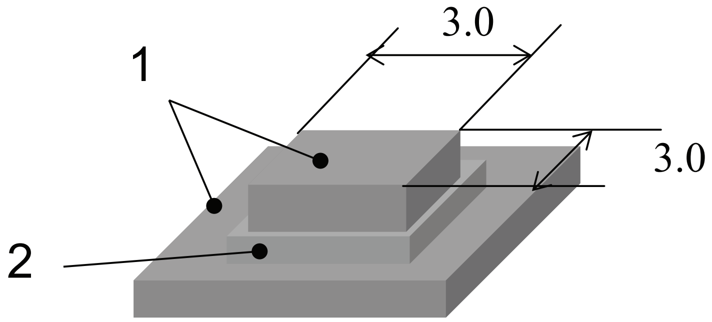
Y shear strength (MPa)

Figure A.5 — Reproducibility of the preparation of specimens

A.2 Another example of preparation of specimens

If the preparation method specified in A.1 causes fracture of the bonded portion due to the cutting load in the preparation of specimens, use copper plates as shown in Figure A.6. Apply a 0,2-mm-thick isotropic electrically conductive adhesive on it and cut it into pieces sized 3 mm x 3 mm (9 mm^2).

Dimensions in millimetres



Key

1 Cu

2 isotropic electrically conductive adhesive

Figure A.6 — Specimen type 2

Annex B
(informative)

Correction of data

B.1 Correction of data

Correct errors, which occur due to friction of pins to fix the hammer and misalignment of the position of the hammer, which is measured by the laser-displacement sensor.

On the assumption that the potential energy is perfectly converted into kinetic energy when the hammer is released, Formula (B.1) holds:

$$\frac{1}{2}\cdot m\cdot v^2 = m\cdot g\cdot h \tag{B.1}$$

where

m is the mass of the hammer;

g is the acceleration of gravity;

h is the dropping height of the hammer.

Using Formula (B.1), the relationship between the height, h, and speed, v, is derived as follows:

$$v = \sqrt{2gh} \tag{B.2}$$

where the speed is defined as the speed before the impact.

Correct the speed after impact, v_{CORR}, using Formula (B.3):

$$v_{\mathrm{CORR}} = v_2 \times \frac{v_{\mathrm{TH}}}{v_1} \tag{B.3}$$

where

v_2 is the speed immediately after impact (m/s);

v_{TH} is the theoretical speed (m/s);

v_1 is the speed before impact (m/s).

Bibliography

[1] JEITA ET 7409/102:2005, *Surface mount technology — Environmental and endurance test methods for solder joint of lead terminal type device — Part 201: Pull strength test*

[2] ISO 9653, *Adhesives — Test method for shear impact strength of adhesive bonds*

[3] ISO 13802, *Plastics — Verification of pendulum impact-testing machines — Charpy, Izod and tensile impact-testing*

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