INTERNATIONAL STANDARD

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Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies —

Part 3:

Test methods

Plastiques — Évaluation des performances de l'interface d'adhérence dans les assemblages plastique-métal —

Partie 3: Méthodes d'essai





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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 (see www.iso.org/directives).

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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

For an explanation on the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the WTO principles in the Technical Barriers to Trade (TBT) see the following URL: Foreword - Supplementary information

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

ISO 19095 consists of the following parts, under the general title *Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies*:

- Part 1: Guidelines for the approach
- Part 2: Test specimens
- Part 3: Test methods
- Part 4: Environmental conditions for durability

Introduction

Structures of heterogeneous materials are being manufactured in the automobiles and aerospace industry sectors where higher safety margins are required.

The existing test methods are not appropriate because the evaluation of the adhesion performance of plastic-metal assemblies is difficult as the polymer material has a relatively low mechanical strength and therefore fractures outside the joints. Therefore, it is necessary to develop a methodology for the evaluation of the plastic-metal interface performance.

A test method to evaluate accurately the adhesion interface performance or standardization of long-term evaluation under harsh environments is also necessary.

The method in ISO 19095 is intended to ensure the integrity of the interface is realized through the interface and that traceability of the value improves the data comparison.

This part of ISO 19095 defines the test conditions, quoting test methods related to mechanical properties that can be evaluated by the existing ISO standards and also adding other test methods that are supposed to be needed.

Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies —

Part 3:

Test methods

SAFETY STATEMENT — Persons using this this part of ISO 19095 should be familiar with normal laboratory practice, if applicable. This part of ISO 19095 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. It is recognized that some of the materials permitted in this part of ISO 19095 might have a negative environmental impact. As technological advances lead to more acceptable alternatives for such materials, they will be eliminated to the greatest extent possible. At the end of the test, care should be taken to dispose of all waste in an appropriate manner in accordance with local regulations.

1 Scope

This part of ISO 19095 specifies the test methods for the adhesion interface performance in plastic-metal assemblies.

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 8256, Plastics — Determination of tensile-impact strength

ISO 14125, Fibre-reinforced plastic composites — Determination of flexural properties

ISO 19095-1, Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies — Part 1: Guidelines for the approach

ISO 19095-2:2015, Plastics — Evaluation of the adhesion interface performance in plastic-metal assemblies — Part 2: Test specimens

IEC 60068-2-17, Basic environmental testing procedures — Part 2-17: Tests — Test Q: Sealing

3 Conditioning

See ISO 19095-1.

4 Conditions of the specimens

The specimens shall be free from twist and shall have mutually perpendicular pairs of parallel surfaces. The surfaces and edges shall be free from scratches, pits, sink marks, and flashes. The specimens shall be checked for conformity with these requirements by visual observation against straight-edges, squares, and flat plates and by measuring with micrometre callipers. Specimens showing measurable or observable departure from one or more of these requirements shall be rejected or machined to the required size and shape before testing.

5 Test procedure

5.1 Tensile strength

5.1.1 Apparatus

Tensile test machine should be selected so that the rupture of the specimen falls between 10 % and 80 % of the full-scale capacity. The response time of the machine shall be short enough so as to enable the force applied at the time of rupture to be measured accurately. The recorded force shall not differ from the true applied force by more than 1 %. The machine shall be capable of maintaining the constant speeds at 10 mm/min (see 5.1.3.2, 5.2.3.2, and 5.3.3). A machine which allows a constant rate of load application may be used. In this case, the machine shall be capable of maintaining a rate of loading of 8,3 MPa/min to 9,7 MPa/min. The machine shall be provided with a suitable pair of self-aligning grips to hold the specimen. The grips and attachments shall be constructed so that they move into alignment with the specimen as soon as the load is applied so that the long axis of the specimen will coincide with the direction of the applied force through the centerline of the grip assembly.

5.1.2 Specimens

See ISO 19095-2:2015, 4.1.

5.1.3 Procedures

5.1.3.1 Test atmosphere

Conduct the test in the same atmosphere used for conditioning the test specimen, unless otherwise agreed upon by the interested parties, for example, for testing at elevated or low temperatures.

5.1.3.2 Test speed and set up specimens

Crosshead speed is 10 mm/min, unless otherwise noted. Distance between the grips shall be 50 mm and the distance above and below the joint surface shall be 25 mm.

NOTE As a result of tests at several different crosshead speeds from 0,5 mm/min to 50 mm/min, tensile strength does not depend on the crosshead speed, however, 10 mm/min is the most suitable in order to inhibit the variability of data.

5.1.3.3 Expression of results

In test report, breaking stress is expressed as the average value in MPa about valid specimens. Tensile strength (MPa) is calculated by dividing breaking load (N) by joint area (mm²). Breaking stress is calculated using Formula (1).

$$\sigma_B = \frac{F_B}{A_i} \tag{1}$$

where

 σ_B is breaking stress (MPa);

 F_B is breaking load (N);

 A_i is joint area (mm²).

5.2 Tensile lap-shear strength

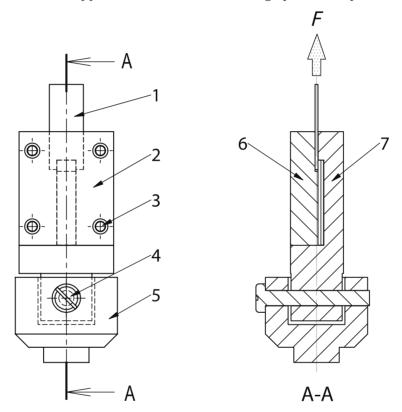
5.2.1 Apparatus

5.2.1.1 Tensile testing machine

See 5.1.1.

5.2.1.2 Specimen retainer

One example of specimen retainer is shown in <u>Figure 1</u>. This retainer shall be fixed in such a way that the fixture attached to the test apparatus and the load line at grips on the specimen lie on a straight.



Key

- 1 specimen
- 2 retainer
- 3 bolt for fixation
- 4 connecting pin
- 5 the fixture attached to the test apparatus
- 6 cover of retainer
- 7 base of retainer

Figure 1 — One example of specimen retainer

The cover (key 6) and the base (key 7) of the specimen retainer which are bolted together to grip the metal portion of the specimen to be tested shall have a gap within 0,1 mm larger than the specimen along both the width and length of the metal portion of the specimen to keep any friction from tainting the test results.

However, the part of the specimen retainer which grips the plastic part of the specimen to be tested shall have a gap less than 0,1 mm along both the width and length of the plastic.

NOTE When carrying out a tensile test holding both edge of the specimen, lap-shear strength could not be measured precisely because the resin part breaks first if the joint strength is too great. If using the retainer, it is possible to put shear stress around the joint interface directly.

5.2.2 Test specimen

See ISO 19095-2:2015, 4.2.

5.2.3 Procedure

5.2.3.1 General

As shown, place the test specimen in the test retainer, taking care to align the planes of loading in Figure 1, and record the maximum load before breaking by the test machine.

5.2.3.2 Test speed and width of grips of metal part

Crosshead speed is 10 mm/min unless otherwise noted. Width of gripping of metal part shall be less than 15 mm from the top of the metal specimen.

NOTE As a result of tests at several different crosshead speeds from 1 mm/min to 50 mm/min, tensile strength does not depend on the crosshead speed, however, 10 mm/min is the most suitable in order to make test time shorter and inhibit the variability of data.

5.2.4 Expression of results

In test report, breaking load is expressed as N or breaking stress is expressed as the average value in MPa about valid specimens. Shear stress (MPa) is calculated by dividing breaking load (N) by joint area [mm²]. Breaking stress is calculated using Formula (2).

$$\sigma_B = \frac{F_B}{A_i} \tag{2}$$

where

 σ_B is breaking stress (MPa);

 F_B is breaking load (N);

 A_i is joint area (mm²).

5.3 Peel strength

5.3.1 Apparatus

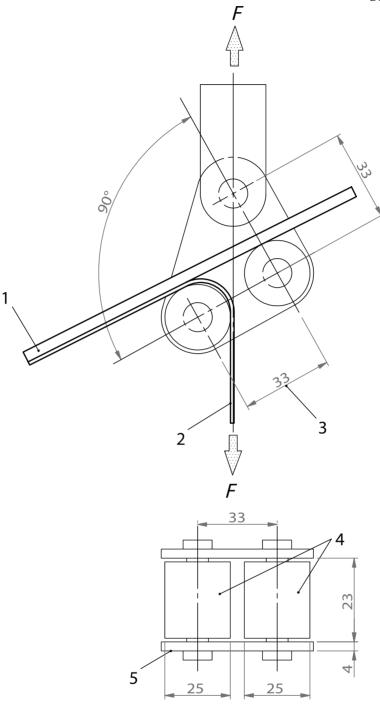
5.3.1.1 Tensile test machine

See <u>5.1.1</u>.

5.3.1.2 Test fixture

Peel test fixture is shown in <u>Figure 2</u>. The fixture shall be attached to one of the cross-arms of the testing machine (5.1). The 25 mm rollers on the test fixture shall roll freely. The angle determined by the rollers and the use of dual bearings are critical and the rollers shall therefore be carefully maintained.

Dimensions in millimetres



Key

- 1 plastic specimen
- 2 flexible metal specimen
- 3 peeling zone
- 4 dual roller bearing type
- 5 mild steel

Figure 2 — Peel test fixture

NOTE See the same fixtures for ISO 4578.

5.3.2 Test specimens

See ISO 19095-2:2015, 4.3.

5.3.3 Procedure

Insert the test specimen into the peel fixture, with the unbounded end of the flexible adherend gripped in the free jaw of the testing machine. Peel the specimen at a constant crosshead separation rate of $100 \text{ mm/min} \pm 5 \text{ mm/min}$, unless otherwise specified.

During the peel test, make an autographic recording of force versus crosshead movement (i.e. force versus distance peeled) over a length of at least 115 mm of the bond line, disregarding the first 25 mm of peel.

5.3.4 Expression of results

Determine from the autographic curve, for at least 115 mm of peeling (disregarding the first 25 mm and the last 20 mm), the average peeling force, in kilonewtons per metre of test specimen width, required to separate the adherends. This average force can be determined from the curve by one of the methods indicated in ISO 4578.

5.4 Bending strength

5.4.1 Apparatus

5.4.1.1 General

See ISO 14125.

5.4.1.2 Loading and holding apparatus

Place supporting point (fulcrum point) and centre pressing point in the way shown in <u>Figure 3</u>. The axis of pressing point and supporting point shall be parallel.

F/2 F/2

R2 27 R2 1

R2 81 R2

100

Dimensions in millimetres

Key

1 specimen

Figure 3 — Four-point loading arrangement

5.4.2 Test specimens

See ISO 19095-2:2015, 4.1.

5.4.3 Procedure

5.4.3.1 General

See ISO 14125.

5.4.3.2 Test speed and width of grips of metal part

Crosshead speed is 1 mm/min, unless otherwise noted.

NOTE As a result of tests at several different crosshead speeds from 0,5 mm/min to 50 mm/min, tensile strength does not depend on the crosshead speed, however, 1 mm/min is the most suitable in order to inhibit the variability of data.

5.4.4 Expression of results

In test report, breaking stress is expressed as the average value in MPa about valid specimens. Flexural stress (MPa) is given by Formula (3).

$$\sigma_f = \frac{F_f L}{hh^2} \tag{3}$$

where

 σ_f is breaking stress (MPa);

 F_f is breaking load (N);

L is the span (distance between the supports) (mm);

b is the width of the specimen (mm);

h is the thickness of the specimen (mm).

5.5 Impact strength

5.5.1 Apparatus

5.5.1.1 General

See ISO 8256.

5.5.1.2 Clamping tools

Use ISO 8256.

NOTE Precise strength is not measured when measuring in the way written in ISO 179-1 because the specimen breaks at the resin part. Variability of impact strength becomes wide, depending on the way of notching.

5.5.2 Test specimens

Use ISO 19095-2:2015, 4.1.

5.5.3 Procedure

See ISO 8256.

5.5.4 Calculation and expression of results

Use ISO 8256.

5.6 Sealing properties

5.6.1 Apparatus

Use ISO 19095-1.

5.6.2 Test specimens

See ISO 19095-2:2015, 4.4.

5.6.3 Procedure

The test methods shall be in accordance with <u>Annex A</u> and <u>Annex B</u>.

5.6.4 Expression of results

See IEC 60068-2-17.

6 Test report

The test report shall include the following information in addition to test report of ISO 19095-2 as needed:

6.1 Tensile strength

- a) accuracy of test machine and elongation meter;
- b) expression of elongation, strain and length of gauge, *L*0;
- c) shape of clamping tool and distance between clamps, *L*;
- d) test speed;
- e) average stress at break.

6.2 Tensile lap-shear strength

- a) test speed;
- b) average stress at break;
- c) failure patterns.

6.3 Peel strength

- a) test speed;
- b) average, maximum, and minimum peel resistance of each test specimen;
- c) load displacement curve.

6.4 Bending strength

- a) test speed;
- b) average flexural stress at break;
- c) failure patterns.

6.5 Impact strength

- a) energy of pendulum;
- b) weight of crosshead;
- c) tensile-impact strength expressed in kilojoules per square metre, reported as the mean of the results;
- d) failure patterns.

6.6 Sealing properties

- a) test method;
- b) pressure and time of test;
- c) detecting method;
- d) maximum leak rate.

Annex A

(normative)

Spray method of sealing properties

A.1 General

The test shall be carried out under the following test procedure.

A.2 Procedure

A.2.1 Apparatus

A.2.1.1 General

See ISO 19095-1.

A.2.1.2 Suction cup

Small size vacuum box, named as the suction cup, is sealed with the test specimen using rubber packing. The inside of the suction cup shall be kept in vacuum to allow the helium leak detector to be operated.

A.2.2 Preparation

The preparation of leak testing is in accordance with the following.

- a) Arrange suction cup, calibration leak, vacuum exhaust system, helium leak detector, hood for enclosing helium gas, and helium gas. Select the necessary vacuum exhaust system according to the volume of the suction cup to be evacuated and the desired pressure.
- b) Perform the pre-treatment of the test specimen. In this case, remove the materials attaching to the surface that is likely to influence the leak detection such as grease, oil, fat, and paint.
- c) Attach the calibration leak directly to the suction cup, or to the position as close as possible to the suction cup (the position most distant from the helium leak detector).

A.2.3 Evacuation

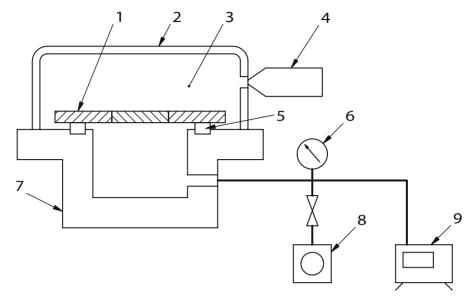
Attach the suction cup to the location to be tested and evacuate. Initial vacuum shall be lower than 5.0×10^{-4} Pa and initial helium partial pressure shall be lower than 5.0×10^{-9} Pa.

A.2.4 Measurement of leakage

The measurement of leakage shall be carried out in accordance with the following.

- a) Example of composition of the test specimen, the suction cup, the calibration leak, the hood, the auxiliary exhaust system, and the helium leak detector are shown in Figure A.1.
- b) Attach the suction cup to the object and evacuate the inside of cup.
- c) Spray helium gas to introduce helium to the hood covering the test specimen as shown in <u>Figure A.1</u> after exhausting the inside of the suction cup.

d) Observe or record the indicated value of the helium leak detector for more than the response time at least from fill the inside of the hood with helium.



Key

- 1 specimen
- 2 hood
- 3 helium gas
- 4 spray probe of helium gas
- 5 seal
- 6 vacuum gauge
- 7 suction cup
- 8 vacuum pump
- 9 helium leak detector

Figure A.1 — Spray method of sealing properties

A.2.5 Precautions

The following precautions shall be taken.

- a) If organic high polymeric material is used for the sealed portion of the test specimen, the helium gas might permeate through the sealed portion and might result in the erroneous recognition to be a leak. In order to prevent this, after spraying the helium gas, the compresses air and the like shall be immediately sprayed to remove the helium gas at the sealed position.
- b) The work shall be done in the circumstance of good ventilation to avoid the residence of helium gas.
- c) The indicated value of the helium leak detector is affected by the concentration of helium near the leak position. For quantification, be careful of the concentration of helium.

A.2.6 Opening and post-treatment

Carry out the opening and treatment in accordance with the following.

- a) Release the helium gas within the hood and complete the evacuation of the suction cup. Introduce air or dry nitrogen to the suction cup to return its inside to the atmospheric pressure.
- b) Remove the sealing agent if it was used to seal the rubber packing of the suction cup for helium leak test.

Annex B

(normative)

Bell jar method of sealing properties

B.1 General

The test shall be carried out under the following test procedure.

B.2 Procedure

B.2.1 Apparatus

B.2.1.1 General

See ISO 19095-1.

B.2.1.2 Vacuum chamber (bell jar)

This shall be of the size that can contain the test specimen and equipped with the connection port for pressure gauge, calibration leak, and vacuum exhaust system. The bell jar shall be confirmed in advance to be free from leak by leak inspection.

B.2.2 Preparation

The preparation of leak testing is in accordance with the following.

- a) Arrange helium leak detector, helium gas, pressure gauge, vacuum chamber, vacuum exhaust system, etc.
- b) Carry out the pre-treatment of the test specimen. In this case, remove the materials attaching to the surface that is likely to influence the leak detection such as grease, oil, fat, etc. and dry the surface.

B.2.3 Evacuation

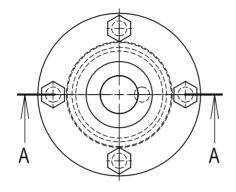
Place the test specimen in the bell jar and attach pressure gauge and calibration leak to the bell jar and then evacuate it. The bell jar shall be evacuated at least to the pressure that does not hinder the operation of helium leak detector and enables to obtain the specified sensitivity confirmed by the calibration leak.

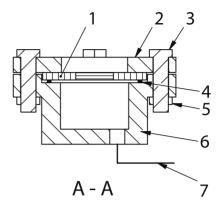
B.2.4 Measurement

The measurement of response shall be in accordance with the following.

- a) Place the test specimen in the vacuum chamber (bell jar). Connect helium piping to the test specimen if necessary. Specimen shall be fixed to the pressurized vessel without the small leakage. Otherwise, the leak from the specimen/vessel could be detected mainly. One example of test specimen with the pressurized vessel is shown in Figure B.1. One example of test condition is also shown in Figure B.2.
- b) Close the vacuum chamber (bell jar) and start evacuation. Initial vacuum shall be lower than 5.0×10^{-4} Pa and initial helium partial pressure shall be lower than 5.0×10^{-9} Pa.

- c) Fill the vessel with helium. In this case, it is recommended to evacuate the vessel using exhaust system to replace the air inside the vessel with helium. The vessel is pressurized to determined pressure.
- d) Observe or record the indicated value of the helium leak detector for more than the response time.

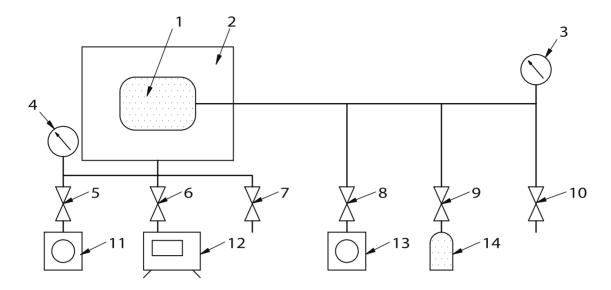




Key

- 1 specimen
- 2 cover of fixture
- 3 bolt for fixation
- 4 sealant
- 5 nut for fixture
- 6 pressurized vessel
- 7 piping to connect helium

Figure B.1 — One example of test specimen with the pressurized vessel



Key

- 1 test specimen with pressurized vessel
- 2 vacuum chamber
- 3 pressure gauge
- 4 vacuum gauge
- 5 vacuum chamber exhaust valve
- 6 test valve
- 7 vacuum chamber vent valve

- 8 pressurized vessel exhaust valve
- 9 helium valve
- 10 pressurized vessel vent valve
- 11 vacuum pump
- 12 helium leak detector
- 13 vacuum pump (for inside gas replacement)
- 14 helium gas

Figure B.2 — Bell jar method of sealing properties

B.2.5 Precautions

The following precautions shall be taken.

- a) If organic high polymeric material is used for the sealed portion of the test specimen, the helium gas might permeate through the sealed portion and might result in the erroneous recognition to be a leak. In order to prevent this, after spraying the helium gas, the compressed air and the like shall be immediately sprayed to remove the helium gas at the sealed position.
- b) The work shall be done in the circumstance of good ventilation to avoid the residence of helium gas.
- c) The indicated value of the helium leak detector is affected by the concentration of helium near the leak position. For quantification, be careful of the concentration of helium.

B.2.6 Opening and post-treatment

Carry out the opening and treatment in accordance with the following.

- a) Finish evacuating the vacuum chamber, introduce the air or dry nitrogen into the vacuum chamber for the vacuum chamber to return to the atmospheric pressure.
- b) Release the pressure inside the test specimen to return to atmospheric pressure. When carrying out the leak testing repeatedly, it is recommended to release the air inside the test object to outdoor using duct to avoid the influence on measurement by the increased helium concentration at the testing site due to releasing of the gas inside the test specimen.
- c) Carry out the necessary post-treatment.

Bibliography

- [1] ISO 179-1, Plastics Determination of Charpy impact properties Part 1: Non-instrumented impact test
- [2] ISO 4578, Adhesives Determination of peel resistance of high-strength adhesive bonds Floating-roller method
- [3] JIS Z 2331, Method for helium leak testing

