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**ISO 14916**

Second edition
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# Thermal spraying — Determination of tensile adhesive strength

*Projection thermique — Mesure de l'adhérence par essais de traction*

Reference number
ISO 14916:2017(E)

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# Contents

Page



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# Foreword

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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).

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For an explanation on the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see the following URL: www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 107, *Metallic and other inorganic coatings*.

This second edition cancels and replaces the first edition (ISO 14916:1999), which has been technically revised.


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# Introduction

The determination of the tensile adhesive strength of a thermal spray coating can play an important role in the quality control of production. Deviations from the normal and qualified procedure can be recognized when preparing and spraying a component.

If the fracture occurs cohesively in the coating when applying the tensile adhesive strength test, the coating's strength in the direction normal to the surface is supplied. Influences of variations in spray conditions can be identified via proper interpretation of tensile test results. Microscopic investigations of the fractured surface can supply further information for judging the quality of the coating's structure.

A revision of the existing document had been required as a result of the identification of significant influences on the test results caused by the tensile test bonding procedure and by the properties of the adhesive itself. These findings were not adequately covered in the previous version of this document.

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# Thermal spraying — Determination of tensile adhesive strength

## 1 Scope

This document specifies the procedure to determine the tensile adhesive strength of thermally sprayed coatings under tension in the direction normal to the surface of the coating by applying a tensile test. By using this procedure, comparability of the test results is ensured.

The test is intended to determine the tensile adhesive strength between the thermally sprayed coating and the substrate material or between the bond and top coat and/or of the cohesive strength of the related coat of the coating system. In some cases, thermally sprayed coatings might have more than two layers. The method specified in this document applies also to determine the tensile adhesive strength between the interfaces of different layers in a coating system which consists of more than two layers.

This test is sufficient to compare coatings manufactured using same or similar feedstock materials and thermal spray processes with each other. The tensile adhesive strength test is not intended to provide absolute values for evaluation of the durability of coatings under operational use.

The test is used to assess the influence of substrate preparation, the spraying conditions and the process parameter on the tensile adhesive strength of thermally sprayed coatings. It can also be employed in order to monitor the consistency of the manufacturing and spraying processes.

NOTE This tensile test can also be applied to very thin coatings. Moreover, the infiltration of bonding agent into the thermally sprayed coatings containing a required level of porosity can be minimized using an appropriate bonding agent (foil rather than liquid). For further instructions, please refer to 6.5.3. This tensile test is inappropriate for determining the adhesive strength of fused spray coatings deposited using self-fluxing alloys due to their inherent high adhesion strength values.

## 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 7500-1, *Metallic materials — Calibration and verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Calibration and verification of the force-measuring system*

ISO 14917, *Thermal spraying — Terminology, classification*

EN 13507, *Thermal spraying — Pre-treatment of surfaces of metallic parts and components for thermal spraying*

## 3 Terms and definitions

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

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— IEC Electropedia: available at http://www.electropedia.org/

— ISO Online browsing platform: available at http://www.iso.org/obp

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**3.1**
**adhesive strength**
$R_H$
tensile strength measured in the tension test, which is calculated from the quotient of the maximum load $F_{max}$ and the cross-section area of the fractured face

**3.2**
**adhesive failure**
fracture, which runs along the interface between coating and substrate

Note 1 to entry: The coating will be totally separated away from the substrate.

Note 2 to entry: The inter-particle bonding (cohesive strength) is higher than the adhesive strength of the coating.

**3.3**
**cohesive failure**
fracture, which takes place within the coating

Note 1 to entry: The inter-particle bonding (cohesive strength) is lower than the adhesion strength of the coating.

**3.4**
**adhesive-cohesive failure**
fracture, which is partially located in the interface of the coating to the substrate and partially within the coating

**3.5**
**internal adhesive strength**
adhesive strength between the layers of a coating system, e.g. between a bond and top coat

**3.6**
**test disc**
test specimen formed like a disc coated on one side

Note 1 to entry: This is to be positioned and glued between two loading blocks when preparing the tensile test specimen.

**3.7**
**reference specimen**
specimen for determination of the strength of bonding agent

Note 1 to entry: This consists of two uncoated loading blocks glued together using the same joining procedure as for all other tested specimens.

## 4 Principles

The test methods listed in this document are recommended for quality control or characterization of coatings and/or coating systems in order to improve thermal spray processes or to develop coatings with increased adhesive and cohesive strength as well as with improved microstructure.

Thermally sprayed coatings exhibit as a thumb of rule — inherent to the characteristics of the process — a porous microstructure. Due to the requirements of their targeted application areas, they may contain a high level of porosity. Due to the possibility of infiltration of the coating by the bonding agent, the porous character of a coating can be unfavourable. The development of incorrect types or quantities of bonding agents can lead to significant changes in the coating properties so that the measurement results can be invalid.

Furthermore, only loads normal to the coating surface, which are free of bending or torsion moments, shall be applied during tensile loading. Therefore, adequate clamping and centring devices shall be used during the entire manufacturing process of the test specimens and during the testing.


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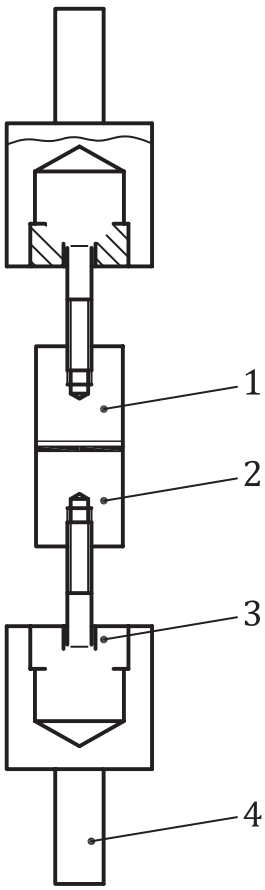
# 5 Equipment for testing and measuring and auxiliary equipment

## 5.1 Test instruments

A tensile testing machine according to ISO 7500-1, class 1, with a suitable clamping system shall be used, which ensures clamping and loading of the specimens through the centre line following no bending and torsion moments.

This can be achieved by a ball joint which is specified in this document (for details, see Figure 1) or by a universal suspension [examples are according to ASTM C633-13 (see Figure B.4), EN 13144 and ISO 13779-4). The M16 threaded drill hole shall bear and transfer the load to the specimen.].




**Key**

1 loading block
2 substrate block
3 ball joint
4 clamping part

**Figure 1 — Arrangement for the tensile adhesion test with test specimen according to form A**

## 5.2 Measurement instrument

Measurements can be carried out, when preparing the loading block and test specimens, using measurement instruments in accordance with standard commercial practices. Recommendations and examples for special gauges to measure the concentricity (see Figure B.2) and parallelism of specimens with a 25,0 mm diameter are given in Annex B.

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### 5.3 Specimen fixing device for bonding of the tensile adhesive specimen

A fixing apparatus, which keeps the angular and axial deviation of the blocks as low as possible, shall be used in order to glue the loading block to the substrate block (or to the test disc). Furthermore, fixing the apparatus shall ensure the application and maintaining of the required contact pressure over the entire gluing process.

NOTE These objectives can be achieved by using a V-block fixture for centring. Using the fixture, the coaxial alignment and a sufficient contact pressure can be maintained so that substrate and loading block (see Figure 2 for details) or two loading blocks and a test disc (see Figure 3 for details) can be joined together to form a tensile adhesion specimen with required tolerances.

When applying the required contact pressure (generally low), it shall be ensured that neither bending nor torsion moments affect the bonding joint.

Consideration should be given to defining the value of the required contact pressure. The contact pressure shall be possibly constant over the entire temperature range during application and hardening of the bonding agent. If the weight of the top loading block, with respect to the substrate block, is inadequate to create the required load, then loading by means of a spring is recommended.

NOTE In case of a spring with an appropriate thread as an intermediate element, the displacement is controlled, set by the number of screw revolutions, and is transferred into force. In this way, the contact pressure can be set precisely by controlling the total number of screw revolutions. Formula (1) is valid for the calculation of the required revolutions of the screw for setting-up of the necessary contact pressure.

$$n = \frac{p \times \pi \times d^2}{4 \times c \times s} \tag{1}$$

where

- $n$ number of revolutions of the screw;
- $p$ necessary contact pressure (Pa);
- $d$ diameter of the substrate block (m);
- $c$ spring constant (N/m);
- $s$ pitch length (m).

In order to avoid deviations of the spring's force during heating and hardening of the bonding agent, the spring shall be made out of a suitable steel. An adequate device is shown in Figure B.1.

## 6 Specimens

### 6.1 Shape of specimens

For the determination of the tensile strength when applying the tensile test, specimens of shape A or B with diameters of 25,0 mm (respectively 25,4 mm) or 40 mm shall be used. The smaller diameter (25,0 mm or 25,4 mm) shall be preferred where possible.

Specimen A (see Figure 2) consists of a substrate block, to which the coating is frontally applied, and the loading block which is glued to the surface of the thermally sprayed coating.

Specimen B (see Figure 3) consists of two loading blocks and a test disc. The test disc coated on one side is glued to the two loading blocks.


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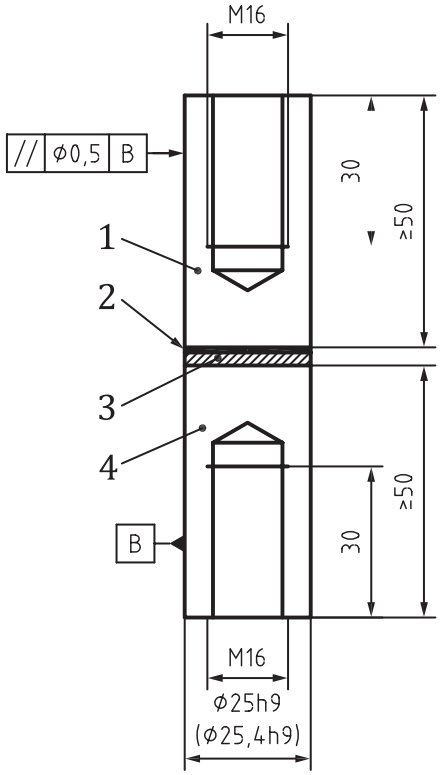
Dimensions in millimetres




**Key**

1 loading block
2 adhesive bond
3 coating
4 substrate block

**Figure 2 — Specimen A for tensile test**

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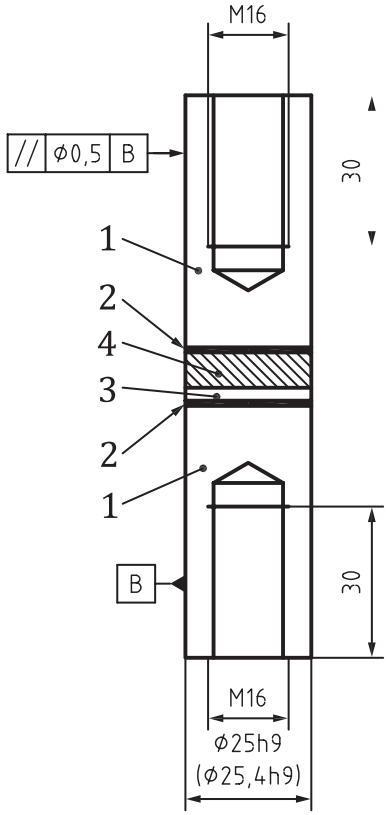
Dimensions in millimetres




**Key**

1 loading block
2 adhesive bond
3 coating
4 disc (according to Figure 6)

**Figure 3 — Specimen B for tensile test**

## 6.2 Material of the specimen

The same material (and surface preparation) shall be used for the specimens as for the actual component. If this is not feasible, a material shall be used which is comparable in strength, chemical and physical properties to the material of the component. Identical surface preparation and coating conditions shall be used.

If the substrate and loading blocks used are made of materials which are prone to work hardening, the deformation of screw threads inside the blocks might lead to invalid results. The acceptance should be arithmetically proofed.

## 6.3 Preparing the substrate and loading blocks

The parts for the tensile test specimen (loading and substrate blocks) are to be manufactured according to Figure 4. In the case of the test disc, manufacture shall be realized according to Figure 6.

The frontal faces of the blocks or that of the test disc shall be perpendicular to the longitudinal axis. This can be assured using a bevelling edge square.

The flat faces of the test disc according to form B shall be flat and parallel. For details, see Figure 6.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The M16 threaded hole shall be concentric and coaxial to the axis of the cylinder in order to achieve a pure tensile loading.

Each substrate and loading block as well as each test disc used shall be clearly designated for identification. Designation should be carried out in such a way that this does not have a negative influence on the test procedure. Identification and material designation of the specimen shall be recorded in the test report. The coating face shall not be marked.

Proof that the test blocks are within the required tolerances can be realized using commercial measuring instruments (for details, see Figure 4 and Figure 5). The gauge shown in Figure B.3 can be employed in order to measure the parallelism. Repeated use of substrate and loading blocks is acceptable, as long as the outer diameter is within the tolerances. The length shall not fall below 50 mm and deformation of the M16 thread due to work hardening shall not influence the results.

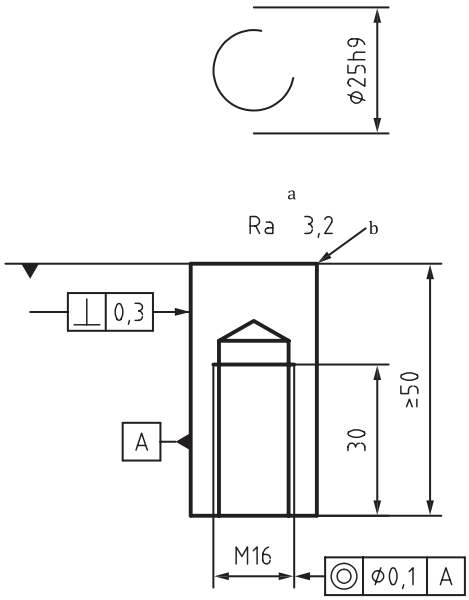
In the case of repeated use of substrate and loading blocks made of materials prone to work hardening, thread inserts shall be used.

NOTE The steps required for manufacture of the test blocks, according to form A and form B, and their tensile adhesive test procedures are listed in Annex A. This includes different categories of bonding agents:

— A.1: Instructions for single-part and multi-part epoxy bonding agents;

— A.2: Instructions for hardening epoxy-foil bonding agents.

Dimensions in millimetres




**Key**

a Flat and square.

b Sharp-edged.

**Figure 4 — Dimensions and tolerances of substrate and loading block (for 25 mm specimen diameter)**

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Dimensions in millimetres

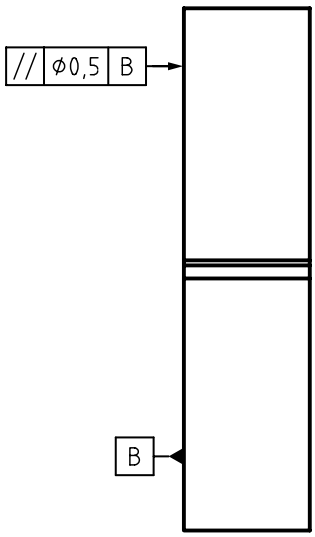


**Figure 5 — Tolerance of parallelism for the tensile adhesive specimen form A exemplary shown**

Dimensions in millimetres

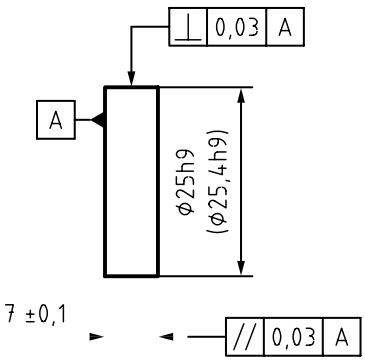


**Figure 6 — Dimensions and tolerances for test disc with rated diameters 25,0 mm and 25,4 mm**

## 6.4 Preparing and spraying of the specimen

The front face of the substrate block (made out of the specified substrate material) shall be prepared for deposition of the coating according to the specifications of the corresponding part, respectively the corresponding component, or according to EN 13507. Contamination (e.g. dirt, fat, and oil) shall be removed carefully. A rounding of the edges due to the blasting process is to be avoided. Subsequently, the coating shall be applied. If the outer cylindrical surface of the specimen cannot be kept free of powder particles, it has to be cleaned. The sequence of spraying process, spraying parameters and spraying material (for both bond and top coat) shall be used according to the specifications of the corresponding part, respectively that of the component.

To avoid negatively affecting the tensile testing procedure when substrate and loading blocks are used repeatedly, any bonding agent residues on the outer cylindrical surfaces of blocks shall be removed carefully; otherwise, the required centring of the specimen in the V-block may not be possible. Machined/mechanical removal of remaining bonding agent would inadmissibly reduce the diameter of the block and is therefore not suggested.

Residues of coating and bonding agent on the front faces are to be removed by means of suitable methods mechanically, chemically, or thermally, providing that the substrate's dimensions or physical properties are not altered.


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## 6.5 Manufacture of the specimens for tensile testing

### 6.5.1 Machining of the coated substrate block (or test disc) and preparation for bonding

When preparing the specimens, care shall be taken that coating thickness is consistent across the specimen's front face. The resulting surface shall be at right angles to the axis of the specimen. Coating and its adhesion to the surface shall not be affected by machining operations. ISO 14924 provides adequate instructions for post-treatment of thermally sprayed coatings.

The surface of the coating shall be cleaned appropriately to fulfil the gluing requirements. The thread hole shall be cleaned carefully as well, e.g. by blowing in a stream of compressed air followed by degreasing. Impurities, i.e. swarf, oil, or residues of cooling media from previous operations, shall not be permitted to remain on the blocks. The instructions of the bonding agent supplier shall be followed.

### 6.5.2 Gluing the specimen

Subsequent to the preparation of the coating surface, the loading block will be glued to the thermally sprayed coating (specimen A) or to the coated side of the test disc (specimen B) which itself shall be glued to the other loading block. If specimen B is used, consideration should be given to the blasting of the rear face and the mating surfaces of both loading blocks to ensure adequate adhesive bonding. The need for blasting also applies to the face of the one loading block used for specimen A.

For the gluing process and hardening of the bonding agent, the instructions provided by the supplier of the bonding agent shall be followed. An adequate fixture, i.e. according to Figure B.1, shall be applied to ensure correct positioning of the substrate and loading block and test disc for the specimen form B. The axes of all parts of the test specimen shall be coaxial and the outer surfaces of all parts shall be parallel.

During the application of bonding agent, the clamping device shall be set in such a way that the specimen remains in the vertical position and can be loaded vertically with a constant load during the entire gluing and hardening cycle. A constant amount of load shall be maintained during cooling down of the specimens in the oven (if applicable).

In case of oven hardening, in order to avoid additional residual stresses, all the specimens shall remain in the oven during the cooling cycle until a temperature of 40 °C is reached at the centre of the oven. The instructions provided by the supplier of the bonding agent shall be followed.

All the details of the test specimen preparation shall be an integral part of the test report.

The diameter of the bonding agent foil or the mass of the liquid bonding agent is to be selected so that a clearly visible bulge of bonding agent around the joint is created after hardening. The surfaces of the faces to be glued shall be entirely wetted by bonding agent.

After completion of hardening, the specimens shall be checked for concentricity and parallelism. The parallelism gauge shown in Figure B.3 can assist with this.

### 6.5.3 Bonding agent

The bonding agent shall make possible the coating to be glued with the loading block. The bonding agent should be selected in relation to the expected adhesive and cohesive strength of the coating. Further criteria for selection are the highest admissible temperature of coating and substrate, i.e. in case of polymer coatings on a carbon filler-reinforced polymer (CFRP) substrate, as well as their chemical resistance. The strength of the bonding agent in the joint should be at least as high as the adhesive or cohesive strength of the coating in the direction normal to the surface. The actual tensile strength of the bonding agent should be checked using a reference specimen. Proof tests performed less than 1 month ago can be accepted.

NOTE An important reason for checking the tensile strength of the bonding agent using a reference specimen is the limited storage life of the bonding agent, as the strength of the bonding agent may decrease over time. Additionally, inadequate preparation of the bonding agent may be detected especially in the case of multi-part bonding agents.

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Since thermally sprayed coatings contain internal porosity, infiltration of the bonding agent into the pores and voids can significantly affect the result when applying this test method. Bonding agents with a low viscosity, those requiring a high hardening temperature or those consisting of a matrix with filler material are unsuitable, because during hardening, the viscosity of the bonding agent and that of matrix might experience significant decreases, leading to an increase in infiltration rate.

Suitable foil bonding agent avoids the problem of infiltration, but possesses under some circumstances the disadvantage that their own tensile strength can remain well below the adhesion strength of the thermally sprayed coating to the substrate or to the bond coat, respectively below the cohesion strength of the coating. In such a case, an appropriate liquid bonding agent with the highest possible viscosity can be applied. Basically, proof of minimal infiltration shall be delivered by a preliminary test, preferably by examination of cross-sections before using the selected bonding agent in the tensile adhesion strength test. The preliminary test shall show that the bonding agent did not penetrate the coating during the entire gluing process or that the infiltration took only place at the near proximity of the coating surface. This investigation shall be carried out for each coating system and for the corresponding spray parameters. Such proof can be difficult to be furnished due to the possibility of the bonding agent being transparent to visible light. If appropriate measures are taken, it can be carried out nevertheless.

Only appropriate foil bonding agent corresponding thickness shall be used for tensile adhesive testing of coatings with high porosity, for example, that of thermal barrier and abradable coatings or coatings intended for oil reservation.

Generally, the use of foil bonding agents is recommended; however, its suitability shall be checked for the particular application. A minimum thickness of the foil or foil layers shall be assured, providing that all topographical peaks and valleys on the coating surface are covered completely. A high thickness of the foil bonding agents is undesirable.

When applying appropriate foil bonding agents, coatings with a minimum thickness of 80 µm can be tested according to this document. If the coating thickness is less than 80 µm, a metallographic examination is required for each coating system and each bonding agent to make sure that the bonding agent from the foil is not forced into the coating.

If the bonding agent to be used is agreed upon between the contracting parties, the gluing procedure shall be agreed too.

### 6.6 Reference specimen

The reference specimen together with the other specimens used in the tensile adhesion strength test need to be glued, hardened and tested, undergoing the same manufacturing steps and using the same bonding agent and parameters. The preparation of the surfaces to be glued shall correspond to that of the substrate blocks of the tensile test specimens. The reference specimen serves for the control of the actual quality of the bonding agent (due to its limited storage life), the bonding method, and the specimen's preparation and is to be evaluated separately. For instructions regarding the use of reference specimen, please refer to 6.5.3.

### 6.7 Number of specimens to be tested

At least five specimens shall be tested, which were coated either in one set on a single specimen holder or together with the actual components.

## 7 Applying the testing

The specimen, with attached clamping arrangement (see Figure 1 for details), shall be inserted free of torque into the tensile testing machine and strained under tension at a constant rate and shock-free until fracture occurs. The increase in tensile loading shall be carried out motion controlled at a constant rate. The cross-head moves at a speed of (1 ± 0,24) mm/min, respectively (0,017 ± 0,004) mm/s, until fracture occurs. The maximum load $F_{max}$ shall be recorded.


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Testing is to be performed at ambient temperature. For a series of tests, equal test conditions shall be maintained.

The tensile adhesive test should be carried out as soon as possible after joining the specimen. If this is not possible, the specimens should be kept at ambient temperature in normal atmosphere. They should be secured to prevent them from falling down. If they are to be transported to an external laboratory, they should be packed in a vertical position in order to avoid damage to the coating and the bonding agent.

## 8 Measurement readings and evaluation

The diameter of the substrate block shall be measured to an accuracy of 0,1 mm. By means of these data, the specimen's cross-section shall be calculated at the gluing surface of the spray coating.

The results obtained with different specimens shall only be used for the calculation of the average tensile adhesive strength of the coatings if and only if the specimens exhibit a failure at the coating-substrate interface, at the top coat to bond coat interface (at the interface of a multilayer systems) or within the coating itself. In case of a mixture of the above-mentioned failure types, the results are included to the calculation as well. If an adhesive-cohesive failure (a mixture of the above-mentioned failure mechanisms/modes) takes place, the distinction between cohesive and adhesive strength cannot be made.

The tests with specimens for which the failure occurs within the bonding agent zone are not relevant for the calculation of an average/mean value. This result does provide a useful guideline for the maximum adhesive/cohesive strength that can be tested using this adhesive. If all fractures occur in this zone within a series, it can be concluded that the tensile strength of the coating is higher than the bonding agent strength.

If a reference specimen is tested together with the test specimens, the result of the test conducted with the reference specimen shall be excluded from the calculations.

An illustration of the individual areas of the tensile adhesive specimen (see Figure C.1) and the different possible failure/fracture locations as well as possible/proposed abbreviations are given in Annex C.

The maximum force $F_{max}$ of each individual specimen shall be taken for the evaluation. The tensile adhesion strength $R_H$ shall be calculated according to Formula (2):

$$R_H = \frac{4 \times F_{max}}{\pi \times d^2} \tag{2}$$

where

$R_H$ tensile adhesive strength in MPa (N/mm$^2$);

$F_{max}$ maximum force in N;

$d$ diameter of the test specimen in mm (tolerance ±0,1 mm).

Furthermore, the failure mode shall be determined and recorded together with the location positioning of the fracture for investigated coating systems, i.e. at interface substrate/bond coat, at interface bond coat/top coat or within the coating. Proposed/possible abbreviations for the positioning of the fracture are specified in Table C.1.

For each specimen series (same coating, same adhesive, same heating procedure), the measured tensile adhesive strength values shall be quoted and the statistical mean value shall be calculated. Additionally, the absolute standard deviation and the relative standard deviation (standard deviation/statistical mean value of the measured tensile adhesive strength in %) are to be calculated and documented unless the testing of the tensile strength shall only show that the rated value had been reached.

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## 9 Test report — Documentation

The test report shall contain the following data for every specimen/series tested according to this document, as far as they are known to the inspector and no further agreements were made (see Annex D).

a) General:

- type of test and objectives;
- institution, inspector, date;
- a reference to this document, i.e. ISO 14916.

b) Specimens:

- preparation of specimens;
- material of the substrate block and/or the test disc;
- designation of specimen;
- number of specimens.

c) Preparation and spraying of the specimens:

- surface preparation of the substrate material (blasting parameters, roughness), roughening procedure and measured roughness;
- feedstock materials of bond coat and top coat, respectively;
- thermal spray process according to ISO 14917;
- spray position in front of the component, respectively the specimen's clamping device;
- thickness of the coating in µm;
- finishing process (if applicable), roughness of the machined coating.

d) Gluing of the specimens:

- manufacturer's designation for the bonding agent used, characteristics of the bonding agent and instructions for use of bonding agent;
- preparation for gluing, roughening procedure, roughness, other procedures, e.g. chemical processes;
- diameter in mm, tolerance ±0,05 mm.

e) Results:

- maximum load $F_{max}$ when fracture occurs and the tensile strength $R_H$ in MPa (N/mm$^2$) for each individual specimen and determination of the arithmetical mean tensile strength value and the absolute standard deviation and relative standard deviation too, if required;
- where measurements of the tensile strength are only used to judge the production consistency and quality assurance when manufacturing the thermal spray coating, a comparison of the readings to the rated value fulfils the task provided the set or agreed tolerance limits are kept;
- minimum value of the tensile strength;
- location of the fracture at the coating-substrate interface (adhesive fracture), in the spray coating (cohesive fracture) or as internal adhesive fracture between bond and top coat. The respective areas should be quoted in order of magnitude (e.g. 100/0 % adhesive/cohesive;


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75/25, etc.). In the case of a single fracture in the bonding agent only, this value shall not be included in the determination of the mean value. If the fractures occur in the bonding agent material predominantly and the values are above the minimum value and are not generated by unacceptable infiltration of the coating, then the coating tensile adhesive strength may be expressed as being greater than the strength of the bonding agent itself;

— tensile adhesive strength of the reference specimen is specified in MPa (if this measurement had been required, for details, see 6.5.3) and the minimum tensile adhesive strength of the bonding agent as well;

— specific observations such as observed deviations from the procedure and preferred kinds of exceptional phenomena which may influence the results and validity of tests shall also be stated;

— visualization if test requirements are fulfilled or not fulfilled.

## 10 Possible sources of fault when preparing the specimens and on testing

— contaminated cylinder wall;

— angular and/or positional displacement of substrate block and loading block;

— testing machine, loading rate, and fixed end moment of the specimen in the test machine;

— spray deposit not uniform in thickness;

— deposit damaged through inadequate post-spray machining or treatment;

— non-observance of bonding agent instructions given by the manufacturer (wetting, storage, setting, and loading for setting);

— the coating thickness is too low and/or the infiltration depth of the adhesive is too high.

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# Annex A
(informative)

# Work instructions for gluing

## A.1 Work instructions when using typical single-part and multi-part epoxy bonding agents

Specific instructions provided by the supplier of the adhesive should be followed. If these contradict the steps given in Tables A.1 and A.2, then the supplier's instructions should be given preference.

**Table A.1 — Work instructions for the manufacture of specimens using the clamping device as shown in Figure B.1**

| | Specimen shape A | Specimen shape B |
|---|---|---|
| 1 | Heating the bonding agent — if applicable — in the closed container up to ambient temperature. | |
| 2 | Mixing of multi-part bonding agents according to supplier's instructions. | |
| 3 | Checking that surfaces of all blocks of the specimen are free of contamination. | |
| 4 | Applying of the bonding agent to the coating on the substrate block. | Applying of the bonding agent to a loading block. |
| 5.1 | The bonding agent is to be evenly applied onto the spray coating using a non-returnable spatula. Care shall be taken that the entire surface is wetted to avoid air bubbles. | |
| 5.2 | Consideration should be given to grit blast non-coated surface prior to application of the bonding agent. If the uncoated surface is not prepared in this way, adhesion of the bonding agent can be compromised. Therefore, test results can become invalid (i.e. premature glue failure). | |
| 6 | | Applying of the bonding agent to the coating which is deposited on the test disc. Use of non-returnable spatula. |
| 7 | | Positioning of the test disc to the loading block in the clamping device (coating above). |
| 8 | Positioning of the loading block in the clamping device, respectively onto the coated substrate block. The shell shall be in contact with the prism. See Figure B.1 and Figure 2. | Positioning of a second loading block in the clamping device, respectively on the coating. The shell shall be in contact with the prism. See Figure B.1 and Figure 3. |
| 8.1 | OPTION: When using a clamping device with a spring bolt, according to Figure B.1, fixing of the substrate and loading block by means of a spring bolt. | |
| 9 | Positioning of the spring cup on top of the above specimen block (see Figure B.1). | |
| 10 | Setting of the spring's line by turning the screw according to Formula (1). Number of revolutions shall be set to one-eighth revolutions precisely. The calculated value shall be rounded up. | |
| 11 | Checking that a bonding agent bulge is visible. | |
| 12 | Visual checking that no misalignment is given. | |
| 13 | Positioning of the charged clamping device in the soaking temperature preheated oven[a]. The clamping device is to be put vertically, the time to be noticed. In the case of more than one specimen per heat, all specimens are to be positioned in the oven at the same time. | |

[a] Bonding temperature and curing time are to be taken from the supplier's instructions.


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**Table A.1** *(continued)*

| | Specimen shape A | Specimen shape B |
|---|---|---|
| 14 | The time shall be noticed again when all specimens reached the hardening temperature. Start of the duration time. | |
| 15 | At the end of the duration time[a], the oven is to be switched off. The specimen shall remain in the oven until cooling down to 40 °C. | |
| 16 | Unloading of each specimen out of its clamping device, when all have reached ambient temperature. Checking of the alignment of the specimen blocks using an adequate gauge, e.g. parallelism gauge according to Figure B.3. | |
| [a] Bonding temperature and curing time are to be taken from the supplier's instructions. | | |

## A.2 Work instructions when using warm curing epoxy-foil adhesive

**Table A.2 — Work instructions for the manufacture of specimens using the clamping device as shown in Figure B.1 when using epoxy-foil adhesives**

| | Specimen shape A | Specimen shape B |
|---|---|---|
| 1 | Determination of the required number of layers, respectively of the required foil thickness related to the coating's roughness by pre-tests. | |
| 2 | Preparing the foil by cutting, respectively punching using a hollow punch. The diameter of the bonding agent coupon shall correspond to the diameter of the substrate block/test disc. Care shall be taken that the used tool (hollow punch, stirrup, tweezers) is free of contamination (e.g. oil and grease) and the foil bonding agent is not touched by naked hands (use of non-returnable gloves). | |
| 3 | Checking that surfaces of all blocks of the specimen are free of contamination. | |
| 4 | Positioning of the coated substrate block (form A) in the clamping device. | Positioning of a loading block (form B) in the clamping device. |
| 4.1 | Therefore, the clamping device shall be put at an angle that will make easier the centring of the parts to each other. The angle sheet of the clamping device (see Figure B.1) serves for that. During positioning, care shall be taken that the shell's surface touches the prism. In order to avoid bonding of substrate block/loading block/test disc with the prism, an adequate heat resistant and if possible, not to be moistened thin intermediate layer, for example a baking parchment, is to be placed between the mentioned elements along the entire height of the prism. | |
| 5 | Positioning of the prepared foil bonding agent on the coating using pincers. | Positioning of the prepared foil bonding agent on the coating on the front face of the loading block using pincers. |
| 6 | | Positioning of the coated test disc on the coating above. The shell shall be in contact with the prism. |
| 7 | | Deposit the prepared foil bonding agent on the coating. |
| 8 | Positioning of the loading block onto the foil bonding agent. When positioning, the shell shall be in contact with the prism. | Positioning of the loading block onto the foil bonding agent. When positioning, the shell shall be in contact with the prism. |
| 8.1 | OPTION: When using a clamping device with a spring bolt, according to Figure B.1, fixing of the substrate and loading block by means of a spring bolt. | |
| 9 | Positioning of the spring cup on top of the above specimen block (see Figure B.1). | |
| 10 | Set of the spring's line by turning the screw according to Formula (1). Number of revolutions shall be set to one-eighth revolutions precisely. The calculated value shall be rounded up. | |
| 11 | Visual checking that substrate and loading blocks are not misaligned. | Visual checking that loading block and test disc are not misaligned. |
| 12 | Positioning of the charged clamping device in the soaking temperature preheated oven[a]. The clamping device is to be put vertically, the time to be noticed. In the case of more than one specimen per heat, all specimens are to be positioned in the oven at the same time. | |
| [a] Bonding temperature and hardening time are to be taken from the supplier's instructions. | | |

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**Table A.2** *(continued)*

| | Specimen shape A | Specimen shape B |
|---|---|---|
| 13 | The time shall be noticed again when all specimens reached the hardening temperature. Start of the duration time. | |
| 14 | At the end of the duration time[a], the oven is to be switched off. The specimen shall remain in the oven until cooling down to 40 °C. | |
| 15 | Unloading of each specimen out of its clamping device, when all have reached ambient temperature. Checking of the alignment of the specimen blocks using an adequate gauge, e.g. parallelism gauge according to Figure B.3. Checking that bonding agent is visible. | |

[a] Bonding temperature and hardening time are to be taken from the supplier's instructions.


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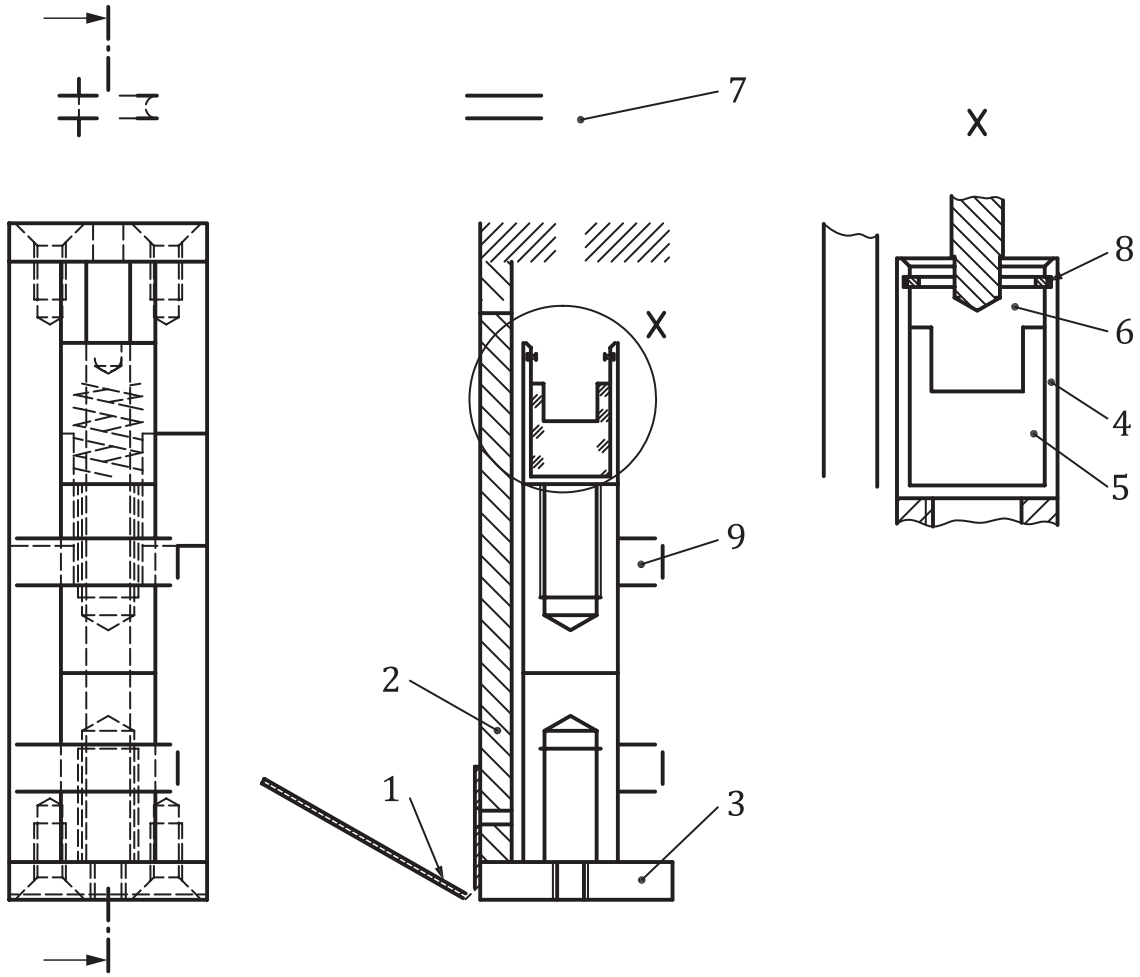
# Annex B
(informative)

# Recommendations for further auxiliary equipment

## B.1 Clamping device

The contact pressure is applied by a cylindrical pressure spring and distance setting. The centring is reached by a prism. For example, a clamping device containing a specimen according to form A is shown in Figure B.1.




**Key**

| | | | |
|---|---|---|---|
| 1 | angle sheet | 6 | punch |
| 2 | back plate | 7 | tommy screw M8 |
| 3 | base plate | 8 | washer (diameter 20 mm) |
| 4 | spring cup | 9 | spring sheet |
| 5 | spring | | |

**Figure B.1 — Clamping device for specimen**

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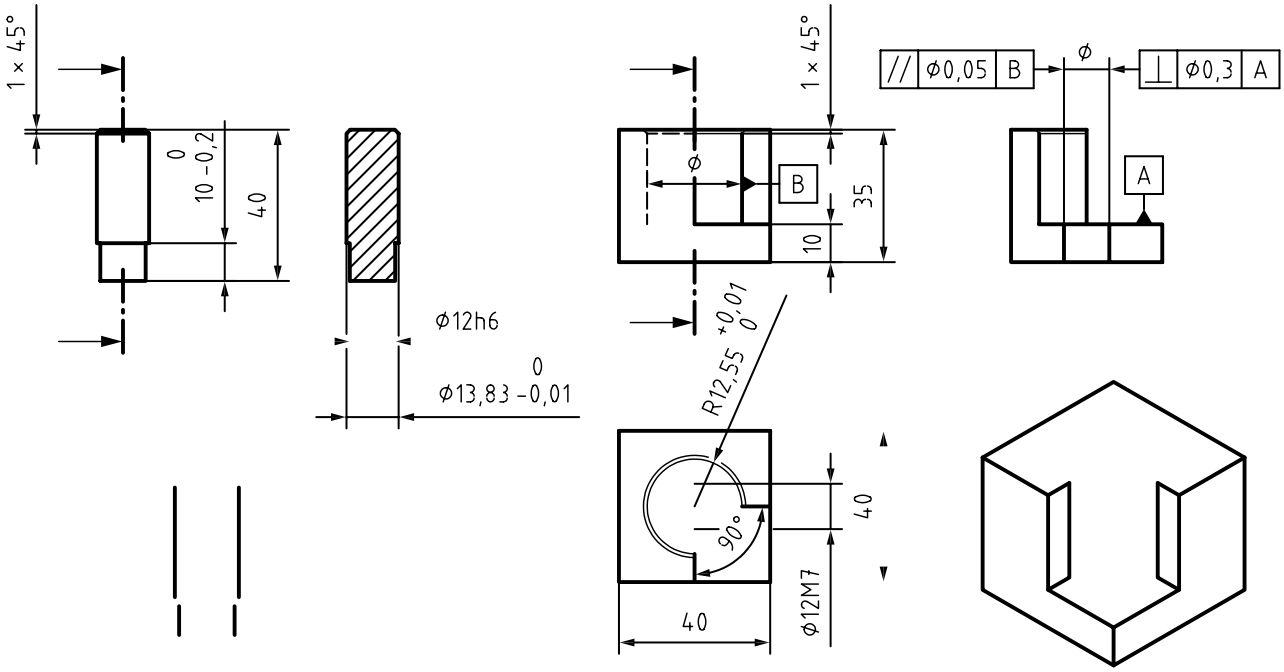
## B.2 Example for a gauge for checking the centricity

Dimensions in millimetres




NOTE The mandrel shown on the left-hand site is to be put into the drill hole 12M7.

**Figure B.2 — Gauge for checking the centricity of the specimen block for rated diameter 25 mm**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## B.3 Example for a parallelism gauge

Dimensions in millimetres

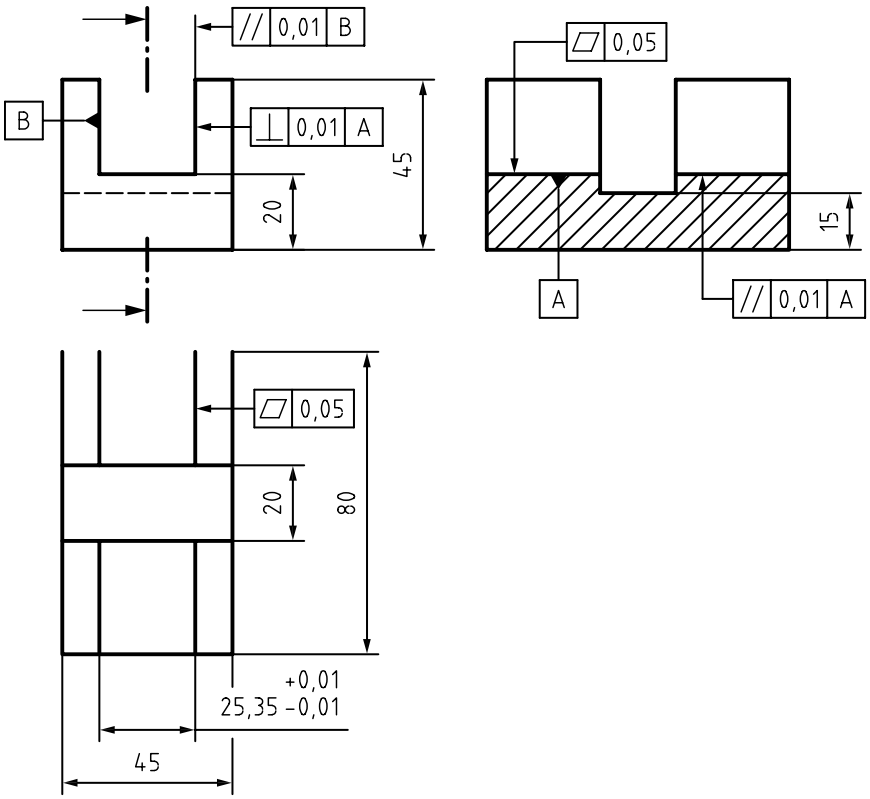


**Figure B.3 — Parallelism gauge (adequate for rated diameter 25 mm and 25,4 mm)**

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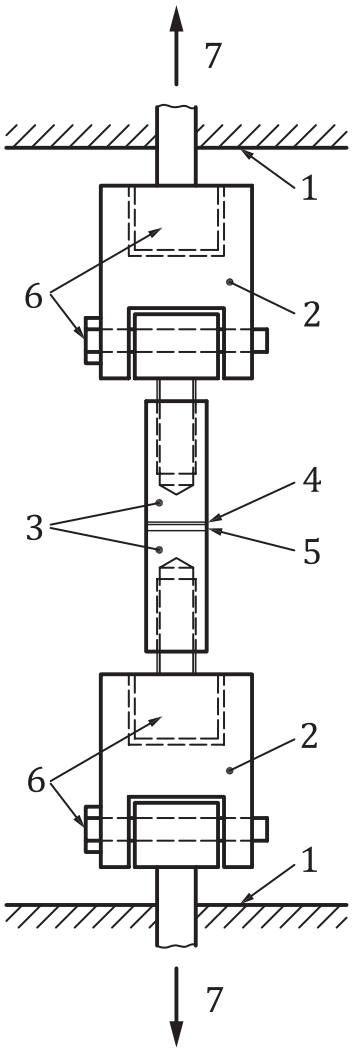
## B.4 Tensile testing using a universal suspension according to ASTM C633-13




**Key**

| | | | |
|---|---|---|---|
| 1 | tensile testing machine chuck | 5 | coating |
| 2 | tensile jig (universal joint) | 6 | bolts |
| 3 | adhesion strength test piece | 7 | tensile direction |
| 4 | adhesive | | |

**Figure B.4 — Tensile testing using a universal suspension according to ASTM C633-13**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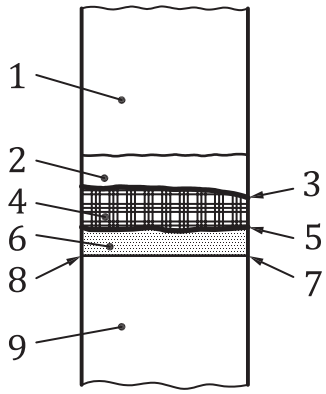
# Annex C
(informative)

# Function areas on the tensile adhesive specimen and possibilities of fracture locations according to ASTM C633-13




**Key**

1 pull-off bar
2 epoxy (cohesive)
3 epoxy-ceramic region (cohesive)
4 ceramic (cohesive)
5 ceramic-bond coat (internal adhesive)
6 bond coat (adhesive)
7 bond coat-substrate (adhesive)
8 roughened surface
9 substrate (adhesive)

**Figure C.1 — Designations of the function areas of the tensile adhesive specimen and definition for zones where fracture can occur (according to ASTM C633-13)**

**Table C.1 — Fracture locations and their abbreviation to be mentioned in the test report**

| Location of fracture | Code |
|---|---|
| Bond coat — substrate material (substrate) interface | BC-PM/I |
| Bond coat internal | BC |
| Bond coat — top coat | BC-TC/I |
| Top coat — near bond coat | TC-BC |
| Top coat — near middle | TC-M |
| Top coat — near top | TC-T |
| Epoxy | E |

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# Annex D
(informative)

# Record for the applied tensile adhesive test according to this document

## D.1 General

Manufacturer: .....

Inspection body: .....

Component designation[a]: .....

Substrate material[b]: .....

Function of the coating[a]: .....

Chemical analysis[c]: .....

Procedure qualification[d]: .....

Accompanying specimen: .....

| Sketch of the component/part (if applicable) |
| --- |
| |

[a] if known

[b] type of material

[c] main elements

[d] if applicable

## D.2 Tensile adhesive test specimen

Type A / B: .....

Substrate material: .....

Diameter: .....

Design, no chamfering, tolerances of angles checked: passed / failed

Tolerance of positioning and angle of centre hole checked: passed / failed

Designation of specimens: .....



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## D.3 Surface preparation for spraying

Programme no.: .....

Specimen degreased: passed / failed

Blasting procedure (manually/mechanized): .....

Type of grit: ..... Grain size: .....

Blasting pressure: .....bar Blasting distance/angle: .....

Visual inspection: Cleanliness, uniformity, according to ISO 8501-1: passed / failed

Roughness, e.g. according to ISO 8503-1: .....

Date/time of blasting and spraying: .....

## D.4 Spraying procedure for test specimens

Spray procedure according to ISO 14917: ..... Date/time of spraying: .....

Spray procedure specification (SPS) for part: ..... / SPS for test specimen: .....

Spraying manually: yes / no

Spraying mechanized: Spraying programme no.: ..... Rev. index: .....

Spraying material:

Designation according to standard: bond coat: ..... top coat: .....

Grain size/wire diameter: bond coat: ..... top coat: .....

Chem. compo. (main elements): bond coat: ..... top coat: .....

Preheating: yes / no

Preheating temperature: .....°C Cooling: yes / no Medium:.....

Spraying distance: .....mm

Surface speed: .....m/min (relative speed between spray torch and part)

Auxiliary spray device, if applicable, no.: .....

Location of specimen at the component/part: .....

Coating thickness: as sprayed: ..... Roughness as sprayed: $R_z$ .....

Number of specimens sprayed: .....

Thermal sprayer/operator: .....

## D.5 Preparation of specimen for tensile adhesive testing

### D.5.1 General

Number of specimens tested: .....

Reference specimen: yes / no pre-tested according to 6.5.3: yes / no

Check of centricity and parallelism: passed / failed

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### D.5.2 Bonding

Surface of shell cleaned: yes / no

Bonding surface cleaned: yes / no

Surface preparation by blasting: Type of grit: ..... Roughness: .....$R_z$

Adhesive: Type: liquid / film Designation: .....

Total film thickness: .....

Hardening: temperature: .....°C Soaking time: .....min Furnace no.: .....

Bonding device: yes/no Designation no.: .....

Contact pressure: .....N or Gravity only: yes / no

Centricity, parallelism after hardening: passed / failed

Surface of sprayed coating to be machined necessary: yes / no

Surface of sprayed coating to be cleaned: yes / no

Bulge round about the bonding zone existing: passed / failed

Infiltration metallographically inspected (if not pre-tested according to 6.5.3): passed / failed

Surface of sprayed coating to be machined necessary: yes / no

## D.6 Testing

Inspection body: .....

Transport of test specimen necessary: yes / no

Testing machine: Designation no.: ..... Type: ISO 7500-1 class 1: yes / no

Testing jig: Type: ball joint / universal joint no.: .....

Increase of load: .....N/s / .....mm/s

Condition: Ambient temperature: .....°C Humidity: .....%

Date/time of testing: .....


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## D.7 Test results

| Tensile adhesive test | Dim. | Rated value | Measured values | | | | | Reference specimen |
|---|---|---|---|---|---|---|---|---|
| Specimen no. | | — | 1 | 2 | 3 | 4 | 5 | |
| Specimen designation | | | | | | | | |
| Outer diameter $D_a$ | mm | — | | | | | | |
| Cross-sectional area | $mm^2$ | — | | | | | | |
| Coating thickness, total | µm | | | | | | | — |
| Coating thickness, top coat | µm | | | | | | | — |
| Max. force $F_{max}$ | kN | — | | | | | | |
| Location of fracture, designation | | — | | | | | | Adhesive BC-PM/I |
| Estimated proportion of fracture adhesive/cohesive | % | | | | | | | 100/0 |
| Calculated max. load | MPa | | | | | | | |

| | Dimension | Calculated results | Test result: P/F[a] |
|---|---|---|---|
| Rated value required | MPa | | |
| Arithmetic mean value, if required | MPa | | |
| Standard deviation, if required | MPa | | |
| Relative standard deviation, if required | % | | |

[a] P = passed; F = failed.

**Date of issue:** .....

**Verifying:**

For inspection body: Name: ..... Signature: .....

For manufacturer: Name: ..... Signature:.....

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