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**Dentistry — Testing of adhesion to  
tooth structure**

*Art dentaire — Essais d'adhésion à la structure de la dent*

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# Contents

	Page
<b>Foreword</b> .....	<b>iv</b>
<b>Introduction</b> .....	<b>v</b>
<b>1 Scope</b> .....	<b>1</b>
<b>2 Normative references</b> .....	<b>1</b>
<b>3 Terms and definitions</b> .....	<b>1</b>
<b>4 Sampling</b> .....	<b>2</b>
<b>5 Test methods</b> .....	<b>2</b>
5.1 Bond strength tests.....	2
5.1.1 General.....	2
5.1.2 Tooth substrate and storage.....	3
5.1.3 Treatment of results.....	4
5.1.4 Tensile bond strength.....	5
5.2 Gap measurement test for adhesion to dentine.....	6
5.2.1 General.....	6
5.2.2 Tooth substrate and storage.....	7
5.2.3 Cavity preparation.....	7
5.2.4 Filling procedure.....	7
5.2.5 Storage of specimen.....	7
5.2.6 Gap measurement.....	7
5.3 Microleakage test.....	7
5.3.1 General.....	7
5.3.2 Tooth substrate and storage.....	8
5.3.3 Cavity preparation.....	8
5.3.4 Filling procedure.....	8
5.3.5 Storage of specimens.....	8
5.3.6 Measurement of microleakage.....	8
5.3.7 Treatment of results.....	9
5.4 Clinical usage tests.....	9
5.4.1 Introduction.....	9
5.4.2 Method.....	9
5.4.3 Restorations.....	9
5.4.4 Study duration.....	9
5.4.5 Sample size.....	9
5.4.6 Clinical procedures.....	9
5.4.7 Evaluation.....	9
5.4.8 Treatment of results.....	10
<b>Annex A (informative) Examples of test methods for measurement of bond strength</b> .....	<b>11</b>
<b>Bibliography</b> .....	<b>12</b>

## 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](http://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](http://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 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

This third edition cancels and replaces the second edition (ISO/TS 11405:2003), which has been technically revised.

## Introduction

Adhesion in restorative dentistry is an important topic. It is the intention of this Technical Specification to describe different laboratory and clinical procedures whereby the effect or quality of a bond between a dental material and tooth structure may be substantiated. By gaining experience with different testing methods, a correlation between laboratory and clinical performance of the materials may be sought.

Adhesive materials are used in many types of restorative and preventive work. Even if the stress on the bond in most circumstances may be defined as either tensile, shear, or a combination of these, there are no specific laboratory or clinical tests which may be valid for all the various clinical applications of adhesive materials.

The relative performance of materials that are claimed to bond to tooth structure has been examined by laboratory assessment of bond strength. While bond strengths may not predict exact clinical behaviour, they could be useful for comparing adhesive materials.

ISO 29022<sup>[1]</sup> describes the notched-edge shear bond strength test which is an important publication in the subject.

[Annex A](#) lists several published laboratory methods for tensile bond strength measurement.

Adhesion testing is also common in general materials in science and a publication listing where many systems have been provided with information.<sup>[2]</sup>

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# Dentistry — Testing of adhesion to tooth structure

## 1 Scope

This Technical Specification gives guidance on substrate selection, storage, and handling as well as essential characteristics of different test methods for quality testing of the adhesive bond between restorative dental materials and tooth structure, i.e. enamel and dentine. It includes a tensile bond strength measurement test, a test for measurement of marginal gaps around fillings, a microleakage test, and gives guidance on clinical usage tests for such materials. Some specific test methods for bond strength measurements are given for information in [Annex A](#).

This Technical Specification does not include requirements for adhesive materials and their performance.

## 2 Normative references

The following referenced 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 1942:2009, *Dentistry — Vocabulary*

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods*

ISO 3823-1:1997, *Dental rotary instruments — Burs — Part 1: Steel and carbide burs*

ISO 6344-1:1998, *Coated abrasives — Grain size analysis — Part 1: Grain size distribution test*

ISO 14155, *Clinical investigation of medical devices for human subjects — Good clinical practice*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1942 and the following definitions apply.

### 3.1

#### **adhere**

to be in a state of *adherence* (3.2)

### 3.2

#### **adherence**

state in which two surfaces are held together by interfacial forces

### 3.3

#### **adherend**

body that is held or is intended to be held to another body by an *adhesive* (3.5)

### 3.4

#### **adhesion**

state in which two surfaces are held together by chemical or physical forces, or both, with the aid of an *adhesive* (3.5)

### 3.5

#### **adhesive**

substance capable of holding materials together

**3.6  
bond strength**

force per unit area required to break a bonded assembly with failure occurring in or near the *adhesive* (3.5)/*adherend* (3.3) interface

**3.7  
microleakage**

passage of substances such as saliva, ions, compounds, or bacterial by-products between a cavity wall and the restorative material

**3.8  
substrate**

material upon the surface of which an *adhesive* (3.5) is spread for any purpose such as bonding or coating

## 4 Sampling

The amount of test material should be sufficient for all planned tests and be from the same batch.

## 5 Test methods

This Technical Specification describes essential characteristics of various types of tests such as:

- a) tensile bond strength measurement;
- b) gap measurement tests for adhesion to dentine;
- c) microleakage tests;
- d) clinical usage tests.

NOTE See Reference [1] for shear bond strength.

For substrate selection, storage, and handling, specific characteristics are described in detail. For the apparatus used for bond strength measurements, general guidelines are given. It is not the intention to recommend the testing of each material by every test as some tests will not be appropriate. However, the quality and sophistication of a laboratory test may not compensate for the fact that the final evidence of adhesive properties should be a clinical usage test.

### 5.1 Bond strength tests

#### 5.1.1 General

Adhesive materials are used for many different purposes in the mouth. The choice of test should be considered according to the intended use of the material. ISO 29022[1] describes the ISO standard shear bond strength test for evaluating direct dental restorative materials. This Technical Specification describes a tensile bond strength test. In addition, several variations are described such as application in thin film and bulk, short, or long exposure time to a wet environment. A set of tests may be necessary to evaluate properly the bond strength of a material. When bond strength is to be measured, the raw data will be in units of force (N). It is necessary to convert this into stress units, i.e. force per unit area (MPa). Hence, control of the area and smoothness of the surface for application of the adhesive material is important.

Several pieces of apparatus are available for measuring the tensile or shear bond strength of an adhesive system. The critical requirements for selection of a suitable instrument for the small and sometimes, fragile specimens are the following:

- the ability to mount the tooth/material specimen in the apparatus and the universal testing machine without application of load (tensile, bending, shear, or torsion) on the specimen;



- a rigid construction in order to avoid elastic deformation (or displacement) of the apparatus and the connection to the testing machine;
- for tensile testing, the ability to apply a slowly increasing and unidirectional tensile load and the ability to align the specimen to avoid an uneven stress distribution during loading.

Large differences in bond strength results between different laboratories are common. Absolute values should therefore be treated with caution and it may be more appropriate to compare the ranking of materials.

In some circumstances, bond strength tests are only useful for screening. They may allow only rough guidance with respect to the clinical performance of an adhesive system. Low values are more likely correlated with poor clinical performance namely retention in adhesive cavities. However, bond strength values above a certain threshold value might not indicate better clinical performance.

## 5.1.2 Tooth substrate and storage

### 5.1.2.1 Substrate

Use either human permanent premolars/molars or bovine mandibular incisors of animals for the measurement of bond strength. The donor bovine animals should not be more than five years old.

When measuring bond strength to human dentine, this Technical Specification recommends to use the buccal superficial dentine that is as close to enamel as possible in order to reduce variations. It is preferable to use third permanent molars from 16-year-old to 40-year-old individuals, if possible.

### 5.1.2.2 Time after extraction (standards.iteh.ai)

There is increasing evidence that changes in dentine occurring after extraction that may influence bond strength measurements. The effect may vary with different types of bonding materials. Ideally, bond strengths should be measured immediately post-extraction, but this is not generally feasible. It appears that most changes occur in the initial days or weeks after extraction and therefore, teeth one month, but not more than six months, after extraction should be used. Teeth that have been extracted for longer than six months may undergo degenerative changes in dentinal protein.

### 5.1.2.3 Condition of teeth

Human teeth used for bond strength measurement should be caries-free and preferably unrestored. However, small and superficial restorations not in the adhesion test area may be acceptable. Root filled teeth should not be used.

There is some evidence to suggest that different teeth in the dentition may give different results with bonding to dentine and enamel. It is not possible to have complete control of variables such as the age of the donating patient, cultural and dietary history, state of health, or to standardize the composition and structure of the teeth.

### 5.1.2.4 Storage of teeth

Immediately after extraction, human teeth should be thoroughly washed in running water and all blood and adherent tissue removed, preferably by the clinician using sharp hand instruments. Bovine teeth should be cleaned as soon as possible after extraction and the soft tissue in the pulp chamber should be removed in a similar fashion.

Teeth should then be placed in distilled water of grade 3 in accordance with ISO 3696:1987 or in a 1,0 % chloramine-T trihydrate bacteriostatic/bacteriocidal solution for a maximum of one week and thereafter, stored in distilled water (ISO 3696:1987, grade 3) in a refrigerator, i.e. nominal 4 °C. To minimize deterioration, the storage medium should be replaced at least once every two months. It is essential that no other chemical agents be used as they may be absorbed by tooth substance and alter its behaviour.

### 5.1.2.5 Tooth surface preparation

A standard, reproducible, flat surface is required. Tooth surfaces should be kept wet at all times during preparation because exposure of a tooth surface to the air for several minutes may cause irreversible changes in bonding character. Dentine is especially sensitive to dehydration.

To control the planing and the angle of the surface during preparation, the tooth should be mounted in a holder by means of dental die stone or cold-curing resin.

NOTE The absorption of resin and the heat of polymerization may adversely affect the tooth. Use a slow setting, viscous resin. The pulp chamber of bovine teeth should be blocked, for example, by wax, to prevent penetration of resin into dentine. Alternatively, use a high viscosity potting medium that does not penetrate the pulp chamber. This may be verified by preparing a set of potted teeth and examining the pulp chambers for the presence of polymerized resin.

Ensure that the tooth has form (undercuts, holes, or retentive pins) that will secure retention in the mounting medium. Place the mounted tooth in water at  $(23 \pm 2) ^\circ\text{C}$  as soon as possible.

Resins will set under water. Die stone should be allowed to set in 100 % RH.

A standard surface should be prepared by planing against silicon carbide abrasive paper with a grit size of P400 as defined in ISO 6344-1:1998 [median grain size  $(35,0 \pm 1,5) \mu\text{m}$ ] under running water.

Plane the exposed surface of the tooth on the wet carborundum paper fixed to a hard, plane surface. Grind until the surface is even and smooth when inspected visually. Discard teeth that have perforations into the pulp chamber. Ensure that the surface is confined to superficial coronal dentine and that the surfaces of all teeth have been prepared to a similar depth.

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### 5.1.2.6 Application of adhesive (standards.iteh.ai)

The tooth surface prepared for application of adhesive material should be preconditioned according to the manufacturer's instructions. If no instructions are given, rinse with running water for 10 s and remove visible water on the surface with a filter paper or by a light/brief stream of oil-free compressed air immediately before application of the adhesive material. Mix if necessary and apply the adhesive material according to the instructions given by the manufacturer. The procedure should be performed at  $(23 \pm 2) ^\circ\text{C}$  and  $(50 \pm 10) \%$  RH.

### 5.1.3 Treatment of results

The bond strength values obtained by tensile or shear testing generally show large coefficients of variation, i.e. (20 - 50) %, and should be tested statistically by an appropriate method. If the coefficient of variation is above 50 %, a thorough inspection of the overall procedure is recommended.

Pre-test failures, unless clearly due to specimen mishandling, should be ascribed bond strength value of 0 MPa.

Bond strength results should be based on appropriate statistical methods and a sufficient number of specimens. If the data are normally distributed, a mean, standard deviation, and coefficient of variation may be calculated. Means may be compared by analysis of variance (ANOVA). However, results from adhesion testing are often not normally distributed. Therefore, the use of probability of failure calculated from the Weibull distribution function provides a suitable means of comparing many materials.[3] The stress to give 10 % failure ( $P_{f10}$ ) and that to give 90 % failure ( $P_{f90}$ ) are convenient ways of characterizing the strength of a bond. A minimum of 15 specimens is required in each group for the application of Weibull statistics. If the number of specimens is smaller, non-parametric tests should be used. In general, increasing the number of specimens gives more certainty in estimating the true mean and standard deviation.

## 5.1.4 Tensile bond strength

### 5.1.4.1 General requirements

Two critical parameters should be considered when designing test equipment and preparing specimens for tensile testing of bond strength

- alignment of the tensile forces acting on the specimen;
- limitation of the bonding area.

### 5.1.4.2 Alignment

The test apparatus should secure alignment between substrate and adhesive material, i.e. the tensile force should be applied at a 90° angle to the planed substrate surface.

The connection between the apparatus and the crosshead of the universal testing machine should be by a universal joint, chain, or wire.

### 5.1.4.3 Adhesive and/or adherend material in bulk

If it is intended that the adhesive should be applied as a thin film with the adherend material in bulk or that the adhesive material should be applied in bulk, a limitation of the bonding area is an important consideration<sup>[4]</sup> (see NOTE). A clearly defined and limited area for bonding has been used by many workers. This allows demarcation of the extent of the adhesive, restriction of the substrate treatment, and permits accurate measurement of the bonded surface. This may be achieved by a material holder with a sharp edge contacting the tooth surface and able to stabilize the material(s) on the tooth surface for curing.

NOTE During the drafting of the shear test described in ISO 29022,<sup>[1]</sup> data were considered that demonstrated negligible differences when using a bonding area limitation or without one (i.e. either protocol could be used to document a claim that a dental adhesive adheres to tooth substance). In the standard shear method, therefore, no limitation is specified. This simplifies the test procedure and removes any interference that a tape limiter may create [e.g. potential contamination from adhesive on a tape limiter, artificial effects on thickness, and shape (e.g. meniscus shape) of adhesive layer, difficulty air-thinning primers and bonding agents, difficulty placing multi-step bonding agents (e.g. that require rubbing action), and difficulty centring a mould over the masked-off area].

For light-curing adhesives or adherend materials, the material holder should give sufficient access to the curing light (e.g. by being made partly or totally of a transparent material). The amount of light energy reaching the material should be in accordance with the manufacturer's instructions.

Coat the inner part of the material holder with a mould-releasing agent when using material holders several times. Avoid coating the edge of the holder. Apply a thin layer of the adhesive material onto the tooth surface. Fill the material holder to slight excess with the adhesive or the adherend material and place it firmly in the correct position on the tooth. Ensure that the material holder maintains contact with the tooth surface in the correct alignment during fixation. The fixation of the material holder should be finished within the manufacturer's stated working time of the adhesive material.

If the manufacturer recommends a particular polymer composite restorative material for use with the adhesive under investigation, then this composite should be used for all tests of that adhesive.

### 5.1.4.4 Adhesive material as thin film and adherend material as preformed rod

If it is decided to restrict the bonding area and use an adherend rod, fix a thin tape of material that is non-reactive with the adhesive with a hole of the same dimensions as the contact area of the rod to the planed tooth surface. Apply a thin layer of the adhesive material on the tooth surface inside the hole in the tape and lower the adherend rod to contact the adhesive material inside the hole. Fix the rod in exact position and alignment and place a load of 10 N on top for 10 s. The total procedure from application of the material to the fixation of the upper rod should be performed within the manufacturer's stated working time. Remove the tape after curing without applying any adverse force on the bonded specimen.