
**Dental materials — Testing of adhesion
to tooth structure**

Produits dentaires — Essai d'adhésion à la structure de la dent

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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

In other circumstances, particularly when there is an urgent market requirement for such documents, a technical committee may decide to publish other types of normative document:

— an ISO Publicly Available Specification (ISO/PAS) represents an agreement between technical experts in an ISO working group and is accepted for publication if it is approved by more than 50 % of the members of the parent committee casting a vote;

— an ISO Technical Specification (ISO/TS) represents an agreement between the members of a technical committee and is accepted for publication if it is approved by 2/3 of the members of the committee casting a vote.

An ISO/PAS or ISO/TS is reviewed after three years in order to decide whether it will be confirmed for a further three years, revised to become an International Standard, or withdrawn. If the ISO/PAS or ISO/TS is confirmed, it is reviewed again after a further three years, at which time it must either be transformed into an International Standard or be withdrawn.

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.

ISO/TS 11405 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

This second edition cancels and replaces the first edition (11405:1994), which has been technically revised.

Introduction

The increasing importance of adhesion in restorative dentistry has made it evident that information is needed on the relative performance of materials that are claimed to bond to tooth structure. In the absence of comparative clinical trials, much emphasis has been placed on laboratory assessment of bond strength. While bond strengths cannot predict exact clinical behaviour, they may be useful for batch quality control.

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

It is, therefore, intended with this Technical Specification to standardize as far as possible different laboratory procedures whereby the effect or quality of a bond between a dental material and tooth structure can be substantiated. By gaining experience with a specific testing system, a correlation between laboratory and clinical performance of the materials can be sought.

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Dental materials — 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 specifies two bond strength measurements tests (tensile and shear), a test for measurement of marginal gaps around fillings and a microleakage test, as well as giving recommendations on clinical usage tests for such materials. It also presents some specific test methods for bond strength measurements.

2 Normative references

The following referenced documents are indispensable for the application 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 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 14155-1¹⁾, *Clinical investigation of medical devices for human subjects — Part 1: General requirements*

ISO 14155-2¹⁾, *Clinical investigation of medical devices for human subjects — Part 2: Clinical investigation plans*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply. See also [1], [2].

3.1

adhere

to be in a state of adherence

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

1) To be published. (Replaces ISO 14155:1996)

**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
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/adherend 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 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.

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5 Test methods

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5.1 General

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

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

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

5.2 Bond strength tests

5.2.1 Overview

Adhesive materials are used for many different purposes in the mouth. The choice of test must be considered according to the intended use of the material. This Technical Specification specifies two types of tests: tensile and shear. In addition, several variations are described, such as application in thin film and bulk, short or long exposure time in a wet environment. A set of tests could be necessary for the proper evaluation of the bond strength of a material. When bond strength is to be measured, the raw data will be in units of force (newtons).

It is necessary to convert this into stress units — i.e. force per unit area in megapascals. Hence, control of the area and smoothness of the surface for application of the adhesive material are paramount.

Several 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 slow and even tensile load and to align the specimen to avoid uneven stress distribution during loading;
- for shear testing, the ability to apply a load at a clearly defined area and position on the specimen, to secure an exact position for the specimen during loading until fracture, and to have an absolute minimum of friction during movement of the load applicator (shearing blade).

5.2.2 Tooth substrate and storage

5.2.2.1 Substrate

Human permanent premolars/molars or bovine mandibular incisors of animals not more than five years old should be used for the measurement of bond strength.

When measuring bond strength to human dentine, the superficial dentine (i.e. as close to enamel as possible) on the buccal surface should be used in order to reduce variations. It is preferable to use third permanent molars from 16- to 40- year-old individuals if possible.

5.2.2.2 Time after extraction

There is increasing evidence that changes in dentine occurring after extraction could 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. Therefore, teeth one month, but not more than six months, after extraction should be used.

NOTE Teeth that have been extracted for longer than six months could undergo degenerative changes in dentinal protein.

5.2.2.3 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 present. 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 neither possible to have complete control of variables such as age of the donating patient, cultural and dietary history or state of health, nor to standardize the composition and structure of the teeth.

5.2.2.4 Storage of teeth

Immediately after extraction, the teeth should be thoroughly washed in running water and, in the case of human teeth, all blood and adherent tissue removed, preferably by the clinician. The soft tissue in the pulp chamber of bovine teeth should be mechanically removed.

The teeth should then be placed in distilled water (grade 3, ISO 3696) or in a 0,5 % chloramine-T trihydrate bacteriostatic/bacteriocidal solution for a maximum of one week, and thereafter stored in distilled water either in a refrigerator (i.e. nominal 4 °C), or frozen at below –5 °C. To minimize deterioration, the storage medium should be replaced periodically. It is essential that no other chemical agents be used, as they may be absorbed by, and alter, tooth substance.

5.2.2.5 Tooth surface preparation

A standard, reproducible, flat surface is required. Tooth surfaces should be kept wet at all times. 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.

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 (e.g. by wax) to prevent penetration of resin into dentin.

Ensure that the tooth has form, undercuts, holes or retentive pins that will secure retention in the mounting medium. The part of the tooth of interest for planing, polishing and bonding should be positioned above the upper surface of the mounting material so that polishing can be performed without contaminating the tooth surface with traces of the mounting material. Place the mounted tooth in water at (23 ± 2) °C as soon as possible.

Resins will set under water. Gypsum materials should be allowed to set in 100 % relative humidity.

A standard surface should be prepared by planing against silicon carbide abrasive paper with a grit size of P600 in accordance with ISO 6344-1:1998 [median grain size $(25,8 \pm 1)$ µm] under running water.

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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 by 2× magnification. Discard teeth that have perforations into the pulp chamber.

5.2.2.6 Application of adhesive

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 manufacturer's instructions. The procedure should be performed at (23 ± 2) °C and (50 ± 5) % relative humidity.

5.2.3 Treatment of results

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

Bond strength results should be based on sound statistical methods and a sufficient number of specimens. If the data are normally distributed, a mean, standard deviation and coefficient of variation can be calculated. Means can be compared by analysis of variance (ANOVA). However, very often results from adhesion testing are 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.

5.2.4 Tensile bond strength

5.2.4.1 General requirements

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

- a) alignment of the tensile forces acting on the specimen;
- b) a clear limitation of the bonding area.

5.2.4.2 Alignment

The test apparatus should secure alignment between substrate and adhesive material, i.e. the tensile force should be applied at a 90° angle in respect of 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 string.

5.2.4.3 Adhesive and/or adherend material in bulk

If it is intended that the adhesive be applied as a thin film with the adherend material in bulk, or that the adhesive material be applied in bulk, a limitation of the bonding area is important. This can be achieved by a material holder having a sharp edge contacting the tooth surface and able to stabilize the material or materials on the tooth surface for curing.

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).

When using material holders for multiple uses, coat the inner part of the material holder with a mould-releasing agent. 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.

5.2.4.4 Adhesive material as thin film and adherend material as preformed rod

When using a preformed rod as the adherend material, fix to the planed tooth surface a thin tape of material non-reactive with the adhesive and having a hole of the same dimensions as the contact area of the rod. 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. See also 5.2.5.3.2.

5.2.4.5 Storage of test specimens

Test specimens should be prepared at $(23 \pm 2) ^\circ\text{C}$ and stored in water at $(37 \pm 2) ^\circ\text{C}$ prior to testing. Storage in water for 24 h is normally sufficient to discriminate between those materials that cannot and those that can withstand a wet environment. Thermal cycling between 5 °C and 55 °C may be used as an accelerated ageing test. Longer periods of water storage may be necessary to show durability of the bond.

The recommended procedures are the following.

- Test type 1: short-term test after 24 h in water at 37 °C.