TECHNICAL SPECIFICATION

ISO/TS 11405

Second edition
2003-02-01

Dental materials — Testing of adhesion to tooth structure

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

Reference number
ISO/TS 11405:2003(E)

© ISO 2003

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS

Not for Resale

PDF disclaimer

This PDF file may contain embedded typefaces. In accordance with Adobe's licensing policy, this file may be printed or viewed but shall not be edited unless the typefaces which are embedded are licensed to and installed on the computer performing the editing. In downloading this file, parties accept therein the responsibility of not infringing Adobe's licensing policy. The ISO Central Secretariat accepts no liability in this area.

Adobe is a trademark of Adobe Systems Incorporated.

Details of the software products used to create this PDF file can be found in the General Info relative to the file; the PDF-creation parameters were optimized for printing. Every care has been taken to ensure that the file is suitable for use by ISO member bodies. In the unlikely event that a problem relating to it is found, please inform the Central Secretariat at the address given below.

© ISO 2003

All rights reserved. Unless otherwise specified, no part of this publication may be reproduced or utilized in any form or by any means, electronic or mechanical, including photocopying and microfilm, without permission in writing from either ISO at the address below or ISO's member body in the country of the requester.

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

Published in Switzerland

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS

© ISO 2003 — All rights reserved

Not for Resale

Contents

Page

© ISO 2003 – All rights reserved

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

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.

© ISO 2003 — All rights reserved
Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

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.

© ISO 2003 – All rights reserved

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

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 14155-1[1)] , *Clinical investigation of medical devices for human subjects — Part 1: General requirements*

ISO 14155-2[1)], *Clinical investigation of medical devices for human subjects — Part 2: Clinical investigation plan*s

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)

--`,,`-`-`,,`,,`,`,,`---

© ISO 2003 – All rights reserved

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

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.

5 Test methods

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).

© ISO 2003 — All rights reserved
Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

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.

--`,,`-`-`,,`,,`,`,,`---

© ISO 2003 – All rights reserved

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

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.

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.

© ISO 2003 — All rights reserved
Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

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 ± 2) °C and stored in water at (37 ± 2) °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.

© ISO 2003 – All rights reserved

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

— Test type 2: thermocycling test comprising 500 cycles in water between 5 °C and 55 °C, starting after 20 h to 24 h storage in water at 37 °C. The exposure to each bath should be at least 20 s, and the transfer time between baths should be 5 s to 10 s.

— Test type 3: long-term test after six months storage in water at 37 °C.

The specimens should be tested for bond strength immediately after removal from water.

5.2.4.6 Tensile loading

The test should be performed at (23 ± 2) °C and (50 ± 5) % relative humidity. Mount the tensile test specimen in the testing apparatus. Do not apply any bending or rotational forces to the adhesive material during mounting. Apply the tensile load in accordance with 5.2.4.7.

5.2.4.7 Strain rate for bond breakage

The recommended standard strain rate for testing a bonded specimen is (0,75 ± 0,30) mm/min crosshead speed, or a load rate of (50 ± 2) N/min.

NOTE The stiffness of the various testing machines and bond assemblies varies widely and hence load rate is more meaningful than crosshead speed.

5.2.5 Shear bond strength

5.2.5.1 General requirements

Two critical parameters should be carefully considered when designing test equipment and in the preparation of specimen for shear testing of bond strength:

a) a defined and limited bonding area;

b) an apparatus and a specimen assembly which allows a reproducible positioning of the shearing force as close as possible to the adhesive interface of each specimen.

5.2.5.2 Tooth surface preparation

See 5.2.2.5.

For the shear bond strength test, the prepared tooth surface should protrude not more than 1 mm from the top surface of the mounting material in order to avoid bending moments (see also 5.2.2.5).

5.2.5.3 Application of adhesive material

5.2.5.3.1 Adhesive and/or adherend material in bulk

If it is intended that the adhesive material be applied as a thin film with the adherend material in bulk, or that the adhesive be applied in bulk, a limitation of the bonding area is important. This can be achieved by firmly clamping a split mould of polytetrafluoroethylene (PTFE) or other suitable material (see Figure 1), to the tooth specimen using a suitable device (see Figure 2). Alternatively, an adhesive tape with a hole of the same geometry and size as the determined bonding area can be applied to the tooth surface. However, ensure that the adhesive does not affect the tape.

Following the manufacturer's instructions, apply the adhesive to the tooth surface in the hole. If adhesive tape is used, position the mould over the hole in the adhesive tape and clamp in place. Fill the mould with the adherend material using a method involving minimal risk for entrapment of air along the contact area of adhesive. After curing, remove the specimen from the apparatus and the adhesive tape without applying any adverse force on the specimen — i.e. shear, bend or rotation.

© ISO 2003 — All rights reserved
Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

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

When using a preformed rod as the adherend material, fix a thin tape non-reactive with the adhesive in accordance with 5.2.4.4. Apply a thin layer of the adhesive material inside the hole in the tape and place the adherend rod exactly on the hole. Fix the rod in exact position and as close as possible to an angle 90° to the tooth surface during the necessary curing time, and apply a load of 10 N on top for 10 s. The total procedure from application of the material to the fixation of the rod shall be performed within the manufacturer's stated working time. Remove the tape after curing, without applying any adverse force on the bonded specimen. To control the angle between the long axis of the rod and the tooth surface, the rod may be positioned and fixed in the same apparatus used for preparation of specimens in 5.2.5.3.1 (split mould and apparatus inFigures 1 and 2). See also 5.2.4.4.

5.2.5.3.3 Adhesive material used as bulk

Use a split mould and fill completely with the adhesive material as described in 5.2.5.3.1.

5.2.5.4 Storage of specimens

See 5.2.4.5 for tests of types 1, 2 and 3: respectively, short-term, thermocycling and long-term storage.

5.2.5.5 Shear loading

Position the specimen in the loading rig. Fix the specimen in the correct position with the adhesive interface within 0,5 mm of the shearing blade in order to prevent displacement during loading. Mount the rig in a universal testing machine and apply the load in accordance with 5.2.4.6.

5.2.5.6 Strain rate

See 5.2.4.6.

5.3 Gap measurement test for adhesion to dentine

5.3.1 Overview

The gap measurement test is another approach to demonstrating the efficacy of an adhesive material intended to bond a filling material to dentine [4]. This type of test involves the laboratory preparation of a tooth cavity and its subsequent filling with the test material or combination of materials. The resulting “restoration” and tooth are sectioned or ground to reveal the cavity wall/restoration interface.

If the filling has been placed correctly, the principal reason for the formation of a gap or gaps around it is the polymerization shrinkage of the restorative material system. The dentine-bonding agent is intended to withstand the forces of this shrinkage and if totally effective no gap will be formed.

If the bond is partially effective at withstanding the forces, some of the polymerization shrinkage will be manifested by external dimensional changes before the interface breaks down. Therefore, a small gap will demonstrate a more effective agent in contrast to one associated with a large gap. The test can be used to evaluate the effectiveness of the adhesive at various times after completion of the restoration.

If a particular bonding agent is recommended for a specific restorative material, then this particular combination should be tested. The test is technique-sensitive and the tester needs good training in handling and application of all the materials used in the procedure, as well as proficiency in dental cavity preparation [4].

The test should be performed at (23 ± 2) °C in order to limit influences from thermal changes.

--`,,`,-`-`,,`,,`,`,,`---
© ISO 2003 – All rights reserved
Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

5.3.2 Tooth substrate and storage

See clause 5.2.2.

5.3.3 Cavity preparation

The teeth should be conditioned in distilled water at (23 ± 2) °C for a minimum of 12 h prior to the experimental procedure.

Plane the buccal surface of the tooth on wet silicon carbide paper (see 5.2.2.5), fixed to a hard, plane surface, to expose a dentine area of at least 4 mm diameter. Prepare a dentine cavity (3,0 ± 0,1) mm in diameter, approximately 1,5 mm deep, with a cavosurface angle of approx. 90°. Use a carbide bur with a straight flat fissure head with flat end and without cross-cuts (ISO 3823-1:1997, clause 5.3.2.4) at approx. 4 000 r/m and with liberal water-cooling. The specimen should be assessed at 5× magnification to ensure that the entire cavosurface margin is surrounded by dentine.

5.3.4 Filling procedure

Follow the manufacturer's instructions closely, including the choice of other necessary materials and all other steps necessary for completion of the total filling procedure.

NOTE Syringing high viscosity materials into the cavity reduces the risk of voids along the cavity walls.

5.3.5 Storage of specimen

After completion of the restoration, store the specimen in water (ISO 3696, grade 3) at (23 ± 2) °C. To test the initial effect of an adhesive in preventing gaps due to contraction of the restorative material, specimens should be inspected at (10 ± 2) min of storage. Other storage times will be appropriate for long-term assessment of an adhesive.

5.3.6 Gap measurement

Remove approximately 0,1 mm of the surface of the filling and dentine by gentle, wet grinding on silicon carbide paper with a median particle size of 8 µm (grade P2500, ISO 6344-1). The surface of the specimen should be kept wet continuously and at a temperature of (23 ± 2) °C.

Rinse the specimen surface thoroughly with a water spray to remove debris in the gaps. Measure the maximum width of the widest gap observed along the circumference of the cavity wall using a device such as a measuring microscope. The measurement should be performed without dehydration of the tooth/filling surface, (e.g. in a water-saturated chamber). A minimum of 10 cavities should be examined.

5.4 Microleakage test

5.4.1 Overview

A microleakage test is another way of testing the efficacy of a material or a combination of materials to establish bonds to both enamel and dentine. A variety of methods have been described with some variations in results. Standardization of such methods is therefore necessary in order to obtain comparable results from different laboratories. In this respect it seems important to standardize quality of teeth, type of cavity and the quantification of leakage. The type of tracer substance does not seem to be of major importance, apart from radioactive tracers that will show diffusion of water through closed interfaces in addition to leakage along patent interfaces.

5.4.2 Tooth substrate and storage

See clause 5.2.2.

--`,,`,-`-`,,`,,`,`,,`---
© ISO 2003 — All rights reserved
Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

5.4.3 Cavity preparation

Teeth should be conditioned in distilled water at (23 ± 2) °C for a minimum of 12 h prior to use.

Several cavity types are of interest when studying leakage. When testing the quality of a particular material or combination of materials to prevent leakage, a standard 3 mm diameter cavity with a depth of at least 1 mm into the dentine in the mid-part of the buccal surface of a third molar should be used.

Start cavity preparation in enamel with a high-speed handpiece using a small cylindrical diamond bur. Finish cavity walls to a diameter of (3 ± 0,2) mm with a carbide bur having a straight flat fissure head with flat end and without cross-cuts (ISO 3823-1:1997, clause 5.3.2.4) at approximately 4 000 rpm and with liberal water cooling.

If a cavity solely surrounded by dentine is of interest, follow the procedure described in 5.3.3. A minimum of 10 cavities should be examined.

5.4.4 Filling procedure

See 5.3.4.

5.4.5 Storage of specimens

Immediately after completion of the filling procedure, immerse the specimen in the tracer solution and store at (23 ± 2) °C for 24 h.

If the effect of thermocycling is part of the test, start the thermocycling procedure according to 5.2.4.5 after 24 h storage at (23 ± 2) °C. After the end of thermocycling, immerse the tooth in a tracer solution for 10 min.

5.4.6 Measurement of microleakage

Cut the tooth longitudinally twice at either side of midline of the cavity with a slow-speed diamond saw under water-cooling. Score all four surfaces, if possible, for microleakage. Inspect under a microscope at 10× magnification for penetration of tracer along the cavity walls.

Normally, use the following quantification.

— No penetration = 0.

— Penetration into the enamel part of the cavity wall = 1.

— Penetration into the dentine part of the cavity wall but not including the pulpal floor of the cavity = 2.

— Penetration including the pulpal floor of the cavity = 3.

If using a dentine cavity only, use the following quantification.

— No penetration = 0.

— Penetration into the dentine/material interface, but not including the pulpal floor of the cavity = 1.

— Penetration including the pulpal floor of the cavity = 2.

5.4.7 Treatment of results

Count the number of observations and use non-parametric statistics when comparing products or procedures.

© ISO 2003 – All rights reserved

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

5.5 Clinical usage tests

5.5.1 Introduction

A clinical usage test is so far the only real basis for judgment of clinical efficacy and lifetime of an adhesive material. Such tests should be designed and performed according to accepted clinical procedures and the intended use of the material.

5.5.2 Method

Clinical usage tests should be in accordance with ISO 14155-1 and ISO 14155-2 or appropriate protocols such as those that may be available from [5] or as follows.

5.5.3 Restorations

The type of restoration used should be decided according to the intended use of the material. If cavities are prepared, a cavity type with limited variations in form and size is preferred.

5.5.4 Observation time

Total observation time should be three years, with restorations observed at baseline and at least once a year during this time.

5.5.5 Sample size

Sample size will depend upon recall rate. In general, the number of restorations at the end of three years should be not less than 80 % of the number at baseline and at least 25. The reasons for loss of patients or failure of restorations or both should be identified.

5.5.6 Clinical procedures

A detailed description of the clinical procedures comprising design, instruments used, pretreatment of surfaces, mixing and placement of material, finishing etc., should be given.

5.5.7 Evaluation

The evaluation should include both direct and, if possible, indirect clinical methods.

Direct clinical methods should be based on [5], [6] or the “modified Ryge-criteria”.[7]

Indirect methods should be used to assess marginal staining, loss of material etc.

5.5.8 Treatment of results

Consideration should be given to the use of a life-table analysis in order to take into account loss of patients or loss of restorations due to unrelated causes [8], [9].

© ISO 2003 — All rights reserved
Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

Dimensions in millimetres

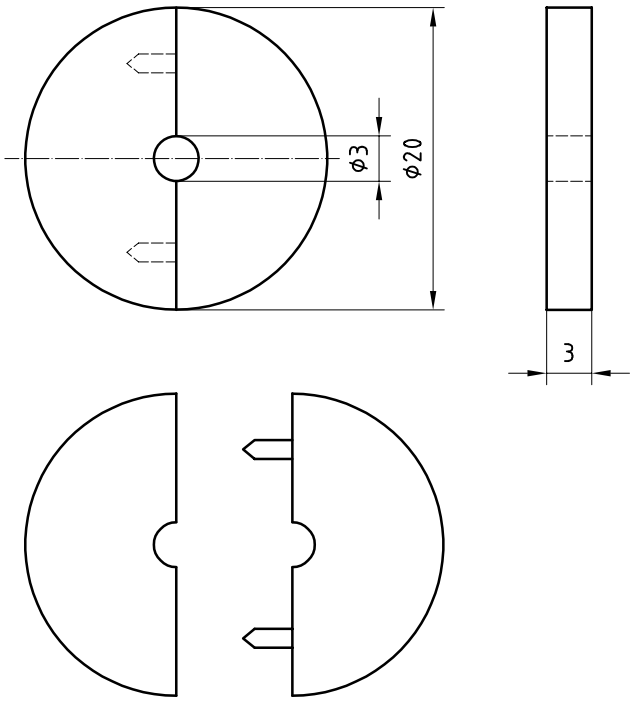
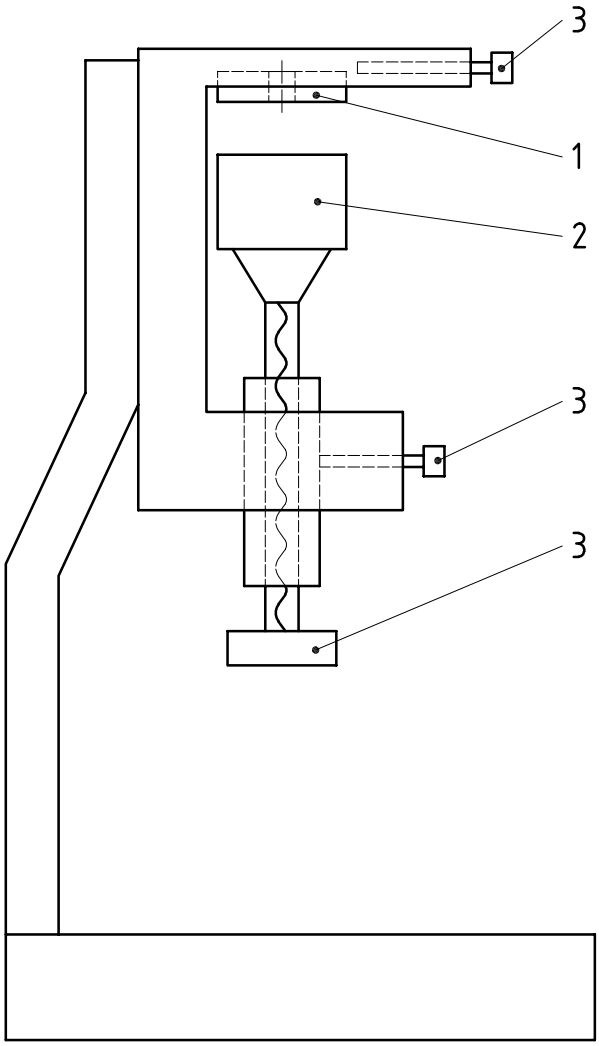


Figure 1 — Split mould

© ISO 2003 – All rights reserved

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale



Key

1 split mould
2 tooth holder
3 fixation screw

Figure 2 — Apparatus for fixation of split mould to tooth specimen

© ISO 2003 — All rights reserved
Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

Annex A
(informative)

Test methods for measurement of bond strength

A.1 General

Annex A gives several examples of published bond strength tests, together with a short description of principles.

A.2 Tensile tests

A.2.1 Kemper and Killian test

This test uses a multi-part test apparatus to ensure alignment during specimen preparation and testing, including holders for material and the tooth (tooth cup, material cup), a bonding alignment block, a measurement alignment block and a set of rods for the connection to the universal testing machine. Special translucent holders are made for light-curing materials. The method has been used in several published tests [10], [11].

A.2.2 Bencor test

This is based on a commercially available apparatus (Bencor multi-T testing device) for making specimens and performing tensile tests under controlled conditions (alignment). It uses partly the same principles as the test described in A.2.1. The material holder (metal) allows the use of light-curing materials [12].

A.2.3 Dumb-bell test

This test uses a dumb-bell shaped specimen with a rectangular cross-section of bonded area, cut from a larger tooth/adhesive-composite specimen, allowing a good control of the bonding area and guiding the fracture to the adhesive interface. Specimens limited to a 3×2 mm bonding area, termed “mini-dumb-bell”, seem to give more information on the bonding surfaces and the bonding mechanism [13], [14].

A.2.4 Micro tensile test

This is a tensile test for bond strength in which hour-glass shaped specimens of approximately 1 mm^2 bonded area have been introduced. It is suggested that a smaller bonding area will have a fewer defects and therefore a more valid measured bond strength [15], [16].

A.3 Shear tests

A.3.1 Noguchi test

This test uses a simple rig for shear testing of a bonded assembly consisting of a body block with a hole for fixation of the specimen and guidance of the shearing blade. The shearing blade has three circular holes of varying diameter for different specimens, and a 0,5 mm blunt shearing edge. The total block can be placed on the table of the testing machine and the load directly applied to the shearing blade [17].

© ISO 2003 – All rights reserved

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

A.3.2 Watanabe test

The single-plane shear test consists of two plane plates, one for fixation of the substrate and the other for fixation and loading of the adhesive/adherend material, both rigidly fixed into place. The shear plates are fixed to a universal testing machine by two alignment assemblies consisting of thin plates which direct shear forces through the adhesive bond zone and loaded in tension [18], [19].

A.3.3 Shear test in accordance with ISO 10477

This shear test for the strength of bonding of polymer-based crown and bridge materials to dental alloys in accordance with ISO 10477, based on the same principles as those of the test described in A.3.1, has been used to test bond strength of dental materials to tooth structure [20].

A.4 Other bond strength tests

The fracture toughness test has been designed for a more controlled test of the energy required to break an adhesive bond. It consists of a notchless triangular prism specimen placed in a special testing holder, and includes a mounting block for preparation of specimens [21].

© ISO 2003 — All rights reserved
Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

Bibliography

[1] *Compilation of ASTM Standard Definitions. 5th Edition*, American Society for Testing and Materials, Philadelphia, PA, USA, 1982

[2] ISO 6354, *Adhesives — Vocabulary*

[3] McCABE, J.F. and WALLS, A.W.G. *The treatment of results for tensile bond strength testing*. J Dent 1986, 14: 165-8

[4] HANSEN, E.K. and ASMUSSEN, E. *Comparative study of dentin adhesives*. Scand. J. Dent. Res. 1985, 93: 280-7

[5] *Revised American Dental Association Acceptance Program Guidelines for Dentin and Enamel Adhesive Materials*, American Dental Association, Chicago, IL, USA, 1994

[6] CVAR, J.F. and RYGE, G. *Clinical evaluation of dental materials*. Monograph GPO 790-244, U.S. Department of Health, Education and Welfare, Dental Health Center, San Francisco, CA, USA, 1973

[7] RYGE, G. *Clinical criteria*. Int Dent J 1980, 30, pp. 347-58

[8] MITCHELL, L. and WALLS, A.W.G. *Survival analysis in practice*. Dent. Update 1991, 18: 125-8

[9] THYLSTRUP, A. and ROLLING, I. *The life table method in clinical dental research*. Community Dent Oral Epidemiol 1975, 3: 5-10

[10] KEMPER K. and KILLIAN, R. *New test system of tensile bond strength testing*. J. Dent. Res. 1976, 55: Special Issue B, 148, Abstract 308

[11] ØILO G. and AUSTERHEIM, E. *In vitro quality testing of dentin adhesives*. Acta Odontol Scand. 1993, 51: 263-9

[12] DRIESSEN C.H. and COETZEE, W.J.C. *Advanced testing device to evaluate characteristics of dental materials*: The Bencor Multi-T. Transactions for the Second International Congress on Dental Materials, 1993; p. 274: Abstract P-136

[13] NAKABAYASHI, N., WATANABE, A. and ARAO, T. *A tensile test to facilitate identification of defects in dentine bonded specimens*. J. Dent. 1998, 26: 379-85

[14] ARAO, T. and NAKABAYASHI, N. *Effect of miniaturized dumbbell-shaped specimen to identify bonding of resin to bovine dentin*. J. Jpn. Dent. Mater. 1997, 16: 175-81

[15] SANO H., *et al. Relationship between surface area for adhesion and tensile bond strength – Evaluation of a microtensile bond test*. Dent. Mater. 1994, 10: 236-40

[16] PHRUKKANON, S., BURROW, M.F. and TYAS, M.J. *The influence of cross-sectional shape and surface area on the micro-tensile bond test*. Dent. Mater. 1998, 14: 212-21

[17] NOGUCHI, H., *et al. Evaluation of Japanese dental adhesives*. J. Dent. Eng. 1982, 60: 16-27

[18] WATANABE, L.G., MARSHALL, G.W. and MARSHALL S., *Dentin shear strength: Effects of tubule orientation and intratooth location*. Dent. Mater. 1996, 12: 109-15

[19] WATANABE, L.G, MARSHALL, G.W. and MARSHALL S.J., *Variables influence on shear bond strength testing to dentin*. Advanced Adhesive Dentistry, 3rd International Kuraray Symposium. Ed. Tagami J, Toledano M, Prati C. Granada International Symposium: Kuraray Co.Ltd. 1999, pp. 75-90

© ISO 2003 – All rights reserved

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

[20] ISO 10477, *Dentistry — Polymer-based crown and bridge materials*

[21] RUSE, N.D., TROCZYNSKI, T., MACENTEE, M.I. and FEDUIK, D., *Novel fracture toughness test using a notchless triangular prism (NTP) specimen*. J. Biomed. Mater. Res. 1996, 31: 456-463

[22] *Quality Evaluation for Dental Care, Guidelines for the assessments of clinical quality and professional performance*. Californian Dental Association, L.A., USA, 1979

© ISO 2003 — All rights reserved
Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS
Not for Resale

--`,,`,,-`-`,,`,,`,`,,`---

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS

Not for Resale

ICS 11.060.10

Price based on 16 pages

© ISO 2003 – All rights reserved

--`,,`,,`-`-`,,`,,`,`,,`---

Copyright International Organization for Standardization
Provided by IHS under license with ISO
No reproduction or networking permitted without license from IHS

Not for Resale