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Dentistry — Testing of adhesion to tooth structure

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National foreword

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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 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[1] 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.[2]

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

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 ± 2) °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 ± 1,5) 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 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.

5.1.2.6 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 instructions given by the manufacturer. The procedure should be performed at (23 ± 2) °C and (50 ± 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 ($P_{f\,10}$) and that to give 90 % failure ($P_{f\,90}$) 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[4] (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,[1] 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.

5.1.4.5 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 materials that may withstand a wet environment and those that may not. Thermocycling 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. Simple water storage has been found to mimic clinically observed restoration degradation.[19]

The recommended procedures are the following:

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

— test type 2: thermocycling test comprising 500 cycles in water between 5 °C and 55 °C starting after (20 – 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 – 10) s.

— test type 3: long term test after six months storage in water at 37 °C (medium changed every seven days to avoid contamination).

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

5.1.4.6 Tensile loading

Perform the test at (23 ± 2) °C and (50 ± 10) % RH. 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 as described in 5.1.4.7.

5.1.4.7 Strain rate for bond breakage

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

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

5.2 Gap measurement test for adhesion to dentine

5.2.1 General

The gap measurement test is another approach that may demonstrate the efficacy of an adhesive material that is intended to bond a filling material to dentine.[5],[6] This type of test involves the laboratory preparation of a tooth cavity and its subsequent filling by 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 it is 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 compared to the one associated with a large gap. The test may be used to evaluate the effectiveness of the adhesive at various times after completion of the restoration.

It is important that 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 being proficient at dental cavity preparation.[5]

Perform the test at (23 ± 2) °C and (50 ± 10) %RH to limit influences from thermal changes.

5.2.2 Tooth substrate and storage

See 5.1.2.

5.2.3 Cavity preparation

Condition the teeth 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 5.1.2.5) fixed to a hard, plane surface to expose a dentine area of at least 4 mm diameter. Prepare a dentine cavity (3,0 ± 0,5) mm diameter approximately 1,5 mm deep with a cavosurface angle of approximately 90 °. Use a carbide bur with a straight flat fissure head with flat end and without cross-cuts in accordance with ISO 3823-1:1997, 5.3.2.4 at approximately 4 000 rpm and liberal water-cooling. The specimen should be assessed at 5 × magnification to ensure that the entire cavosurface margin is surrounded by dentine.

5.2.4 Filling procedure

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

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

5.2.5 Storage of specimen

After completion of the restoration, store the specimen in water of grade 3, in accordance with ISO 3696:1987, 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.

5.2.6 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 of grade P2500 in accordance with ISO 6344-1:1998. 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 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.

5.3 Microleakage test

5.3.1 General

Microleakage testing is another way to test the efficacy of a material or a combination of materials to establish bonds to both enamel and dentine. Many methods have been described with some variation 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.

In addition, the phenomenon of "nanoleakage" has been described.[7] This is a specific type of leakage within the dentine margins of restorations which appears as a consequence of the acid etching procedure allowing the penetration of oral and pulpal liquids, such as acids, into porosities within or adjacent

to the hybrid layer. Nanoleakage is independent of microleakage. The amount of penetration depends on the type of bonding agent and the application technique. Nanoleakage is much less extensive than microleakage and has probably no short-term clinical relevance. The long-term stability of the adhesive bond between dentine and restorative material, however, could be adversely affected.

While there have been many papers reporting microleakage studies, a correlation with clinical performance has not been established.

5.3.2 Tooth substrate and storage

See 5.1.2.

5.3.3 Cavity preparation

Condition teeth 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 hand piece using a small cylindrical diamond bur. Finish cavity walls to a diameter of (3 ± 0,2) mm with a carbide bur with a straight flat fissure head with flat end and without cross-cuts in accordance with ISO 3823-1:1997, 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 5.2.3. A minimum of 10 cavities should be examined.

5.3.4 Filling procedure

Follow the manufacturer's instructions. See also 5.2.4.

5.3.5 Storage of specimens

Immediately after completion of the filling procedure, immerse the specimen in the chosen tracer solution and store at (37 ± 2) °C for 24 h.

If the effect of thermocycling is part of the test, start the thermocycling procedure according to 5.1.4.5 after 24 h storage at (37 ± 2) °C. After the end of thermocycling, immerse the tooth in a tracer solution for (2 – 4) h.

NOTE Many tracer solutions have been used including inorganic dyes, organic dyes, electrolytes, and silver nitrate. Suspensions of pigment particles are not recommended.

5.3.6 Measurement of microleakage

Cut the tooth longitudinally twice to 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.

Use the following scoring system:

— 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:

— 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.

5.3.7 Treatment of results

If the data collected are in the form of scores, non-parametric tests should be used when comparing products or procedures.

5.4 Clinical usage tests

5.4.1 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.

5.4.2 Method

Clinical usage tests should follow the general principles given in ISO 14155 or appropriate protocols such as those that may be available from FDI World Dental Federation [8] or as described below.

5.4.3 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.

5.4.4 Study duration

Study duration should be decided by an assessment of the likely incidence of adverse observations. This will depend upon the material being investigated and any particular property being specified, e.g. marginal staining or whether it is simply “restoration present”. Observations should be made and recorded at baseline and at appropriate intervals during the study.

5.4.5 Sample size

Sample size will depend upon the predicted incidence of changes to the restoration. The reasons for loss of patients and/or failure of restorations should be identified.

5.4.6 Clinical procedures

A detailed description of the clinical procedures comprising design, instruments used, isolation procedures, pre-treatment of surfaces, mixing and placement of material, polymerization method, finishing, etc. should be given.

5.4.7 Evaluation

The evaluation should include both direct and, if possible, indirect clinical methods. Where possible, the evaluator should not be the person that placed the restorations.

Direct clinical methods were traditionally based on the USPHS criteria.[9] However, this type of assessment, while simple, is somewhat non-discriminatory and more refined methods are now available [8].

5.4.8 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 clauses.[10]

Annex A
(informative)

Examples of test methods for measurement of bond strength

A.1 General

This Annex lists several examples of published bond strength tests with a short description of principles and a reference to publications for a more complete description.

A.2 Tensile tests

A.2.1 Kemper and Killian test

Consists of 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.[11]

A.2.2 Bencor test

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 described in the previous test (A.2.1). The material holder (metal) allows the use of light-curing materials.[12]

A.2.3 Dumb-bell test

A dumb-bell shaped specimen with a rectangular cross-section of bonded area cut from a larger tooth/adhesive-composite specimen which allows a good control of the bonding area and guides 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],[15]

A.2.4 Micro tensile test

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 fewer defects and therefore, a more valid measured bond strength.[16],[17]

A.3 Other bond strength tests

A.3.1 Fracture toughness test

A fracture toughness test designed for a more controlled test of the energy required to break an adhesive bond. It consists of a notch less triangular prism specimen placed in a special testing holder and includes a mounting block for preparation of specimens.[18]

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