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Dentistry — Adhesion — Notched-edge shear bond strength test

Médecine bucco-dentaire — Adhérence — Essai de résistance au cisaillement sur échantillons à bord entaillé

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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.

ISO 29022 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 1, *Filling and restorative materials*.

Introduction

The International Organization for Standardization (ISO) draws attention to the fact that it is claimed that compliance with this document may involve the use of a patent concerning testing shear bond strength.

ISO takes no position concerning the evidence, validity and scope of this patent right.

The holder of this patent right has ensured ISO that he/she is willing to negotiate licenses under reasonable and non-discriminatory terms and conditions with applicants throughout the world. In this respect, the statement of the holder of this patent right is registered with ISO. Information may be obtained from:

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Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights other than those identified above. ISO shall not be held responsible for identifying any or all such patent rights.

The purpose of this International Standard is to establish a simple and easy to use method for documenting a claim that a material adheres to tooth substance. While the method described in this International Standard has been used for comparing dental adhesive materials, users of this International Standard should evaluate usefulness of this method for their particular application(s). There is a variety of other dental adhesion test methods that may also be suitable or preferable, depending on the objective (e.g. academic research).

Dentistry — Adhesion — Notched-edge shear bond strength test

1 Scope

This International Standard specifies a shear test method used to determine the adhesive bond strength between direct dental restorative materials and tooth structure, e.g. dentine or enamel. The method as described is principally intended for dental adhesives. The method includes substrate selection, storage and handling of tooth structure, as well as the procedure for testing.

NOTE 1 Testing adhesion to tooth structure is technique sensitive and experience with the test method is required.

NOTE 2 With modification, it may be possible to use this method for adhesive restorative materials (e.g. glass-ionomer materials).

2 Normative references

The following 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, *Dentistry — Vocabulary*

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

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

3 Terms and definitions

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

3.1
bond strength
force per unit area required to break a bonded assembly with failure occurring in or near the adhesive/adherend interface

3.2
direct dental restorative material
material used to restore a tooth that is placed in the plastic or unset state and sets intraorally when used clinically

3.3
substrate
material upon the surface of which an adhesive is spread for any purpose, such as bonding or coating

4 Tooth substrate and storage

4.1 General

Use bovine incisors or human erupted permanent third molars for the measurement of bond strength. Record the type of substrate used.

For bovine teeth, use the labial surface. For human teeth, use the buccal, mesial, distal or lingual surface.

NOTE The occlusal surface of human teeth should not be used due to difficulty in finding a suitable bonding area.

Use superficial dentine, i.e. as close to enamel as possible, in order to reduce variations. It is important that the planed dentine surface follow the anatomical plane of the dentine-enamel junction (interface) as closely as possible in order to produce a uniform dentine structure.

4.2 Time after extraction

Use teeth within 6 months after extraction.

NOTE Teeth extracted more than 6 months prior to use may undergo degenerative changes in dentinal proteins.

4.3 Condition of teeth

Use caries-free teeth for bond strength measurement. Do not use human teeth which are restored or are root-filled teeth (endodontically treated).

4.4 Initial preparation and storage of teeth

For bovine teeth, cut off the roots at the cemento-enamel junction (CEJ) and remove the pulp. For human teeth, wash thoroughly in running water and remove all blood and adherent tissue as soon as possible after extraction.

Place the teeth in water (ISO 3696, grade 3) at (4 ± 4)°C. To minimize deterioration, replace the storage medium at least once every 2 months.

Preservatives which do not react with dentine, e.g. 1 % aqueous solution of chloramine-T, may be used. Do not use preservatives which can react with dentine, e.g. aldehydes, or which can inhibit radical polymerization, e.g. phenols. If storage in a preservative has taken place, thoroughly rinse the teeth in running water to remove any preservative solution prior to tooth surface preparation.

5 Tooth surface preparation

5.1 General

A standard, reproducible, flat surface is required. Keep tooth surfaces wet at all times.

NOTE Exposure of a tooth surface to the air for several minutes may cause irreversible changes in bonding character. Dentine is especially sensitive to dehydration.

5.2 Potting (mounting) of teeth

Prior to potting the teeth, block the opening to the pulp chamber with wax or dental cement. Alternatively, use a high viscosity potting medium that does not penetrate the pulp chamber. This can be verified by preparing a set of potted teeth, sectioning them and examining the pulp chambers. Discard the sectioned specimens.

Place the tooth bonding side down (labial side down for bovine teeth; buccal, mesial, distal or lingual side down for human teeth) in a cylindrical mould on a level working surface. Plastics or metal rings of approximately 25 mm internal diameter may be used. The height of the mould may vary; 10 mm to 25 mm is suggested. If rings are used, a sheet of polyester or similar film may be placed under the rings for easy removal and clean up. Pour a mixed slow-setting viscous self-curing resin or dental die stone into the mould. Remove the potted tooth from the mould as soon as possible after the potting medium has set. Store the potted tooth immediately in water (ISO 3696, grade 3) at (4 ± 4)°C.

NOTE 1 The heat of polymerization of self-curing resin may adversely affect the tooth. Samples may be cooled in an ice bath during polymerization of the resin.

NOTE 2 It is preferable to pot teeth soon after cutting off the roots and removing the pulp.

NOTE 3 A small flat area which will be parallel to the final bonding surface may be ground in the enamel for easier placement and stabilization of the tooth during the potting process.

5.3 Surface preparation

Fix silicon carbide abrasive paper, complying with ISO 6344-1, to a hard, flat surface. No more than 4 h before the bonding procedure is planned, prepare a standard surface by a two-step sequential planing process under running water. First use P120 paper until a bonding area sufficient for placing a resin composite ('composite') button with a diameter of 2,38 mm has been exposed, followed by P400 paper until the surface is even and smooth when visually inspected. For dentine, stop the grinding when superficial dentine is exposed. The median grit size for P120 abrasive paper is (125 ± 1) µm and for P400 abrasive paper is (35 ± 1) µm. If abrasive papers of grit size P120 or P400 are not available, use abrasive papers with grit size as close as possible to 125 µm and 35 µm.

Grinding may be performed in an automatic grinding machine with rotating abrasive discs and running water. Use a mechanical fixture that orientates the abrasive paper perpendicular to the specimen. A suitable machine consists of a grinding mandrel with T-slot grinding plate. Alternatively, place the potted tooth into a fixture in a drill press (tooth orientated downwards), lower and press the rotating tooth against the abrasive paper on a motorized rotating lapidary wheel under running water.

In order to ensure correct alignment in the bonding and test fixtures, ensure that the ground surface is perpendicular to the sides of the potted tooth cylinder and that the top and bottom surfaces of the specimen are parallel. Discard potted ground teeth that have perforations into the pulp chamber. Rinse the potted ground teeth well with water after grinding to remove any foreign matter (e.g. residual sandpaper grit).

Do not grind too deeply into the dentine or bond strength may be reduced. The amount of area exposed by grinding can be checked by briefly drying the tooth with air for easier visualization of the tooth surface and dentine area.

5.4 Storage of prepared specimens

After grinding to expose the bonding surface, store the specimens in water at room temperature and use for the bonding procedure within 4 h.

6 Specimen production, handling and storage

6.1 General

Prepare and test the specimens under ambient conditions of (23 ± 2)°C and (50 ± 10) % relative humidity, ensuring that they are kept wet. Prepare 15 specimens for testing.

6.2 Preconditioning

Precondition the prepared tooth surface according to the adhesive manufacturer's instructions. If no instructions are given, rinse the tooth with running water for 10 s and remove visible water on the surface with a filter paper or by a light/short stream of oil- and water-free compressed air immediately before application of the adhesive material.

6.3 Application of adhesive

Apply the adhesive material according to the manufacturer's instructions over the entire prepared tooth surface ensuring an even coating of adhesive, and cure according to the manufacturer's instructions.

6.4 Insertion into bonding clamp

Once the adhesive film has been cured, insert the tooth into the bonding clamp (Figure A.2) containing a white plastics button mould with hole diameter of (2,38 ± 0,03) mm (Figure A.1). Inspect the plastics button mould visually prior to use and replace if it appears to be worn. Centre the mould opening over a suitable bonding area, ensuring that the bonding area consists only of the specified substrate, e.g. dentine, and lower the mould onto the tooth surface. If the height of the potted tooth is less than approximately 20 mm, use a spacer under the potted tooth so that the mould will contact the tooth surface when lowered onto the bonding surface. Tighten screws until one-half of the wave spring (key item 1 in Figure A.2) is compressed and there is no arching of the plastics button mould positioned on the tooth.

NOTE 1 Arching of the plastics mould can lift the mould off of the tooth and allow composite to spread beyond the mould.

NOTE 2 A stack of 12 microscope slides taped together, approximately 13 mm in height, can be used as a spacer for potted teeth less than 20 mm in height.

6.5 Composite placement

Use a high modulus composite and use the same composite when comparing adhesives. Apply the composite to the bonding surface and cure according to the manufacturer's instructions for use. Use a small flat end packing instrument of about 1 mm diameter to avoid "tug back" on the composite. Pack a thin layer of composite, approximately 0,2 mm thick, into the button mould ensuring good contact with the bonding surface without any air voids at the bonding interface. Pack additional composite until the cylindrical part of the button mould is approximately one-half to three-quarters full. Do not place composite in the upper 45-degree angled part of the mould. Cure the composite according to the manufacturer's instructions for use. Do not exceed manufacturer's recommendations for depth of cure during composite placement.

NOTE 1 A high modulus composite (flexural modulus > 9 GPa) is used to minimize the effect of deformation of the composite button during shear.

NOTE 2 Over-filling the mould can make it difficult to remove the mould and may disturb the bond.

NOTE 3 The composite application step is one of the most important in the bonding process. Deviating from the above composite application protocol may result in high variability.

6.6 Specimen handling and storage

Loosen the screws on the bonding clamp and remove the bottom spacer (if used). If a spacer has been used, remove the sample from the button mould using a small hand instrument (e.g. packing instrument/condenser) to apply pressure directly downward onto the cured composite button until the sample is released. Avoid applying any adverse force on the specimen, e.g. shear, bending or rotational. If a taller specimen has been used, remove the specimen from the mould by holding down the composite button with the hand instrument and lifting the top of the clamp off.

NOTE Use special care when removing the mould from a 'low-shrink' composite material because it is likely to be a tighter fit and more difficult to remove.

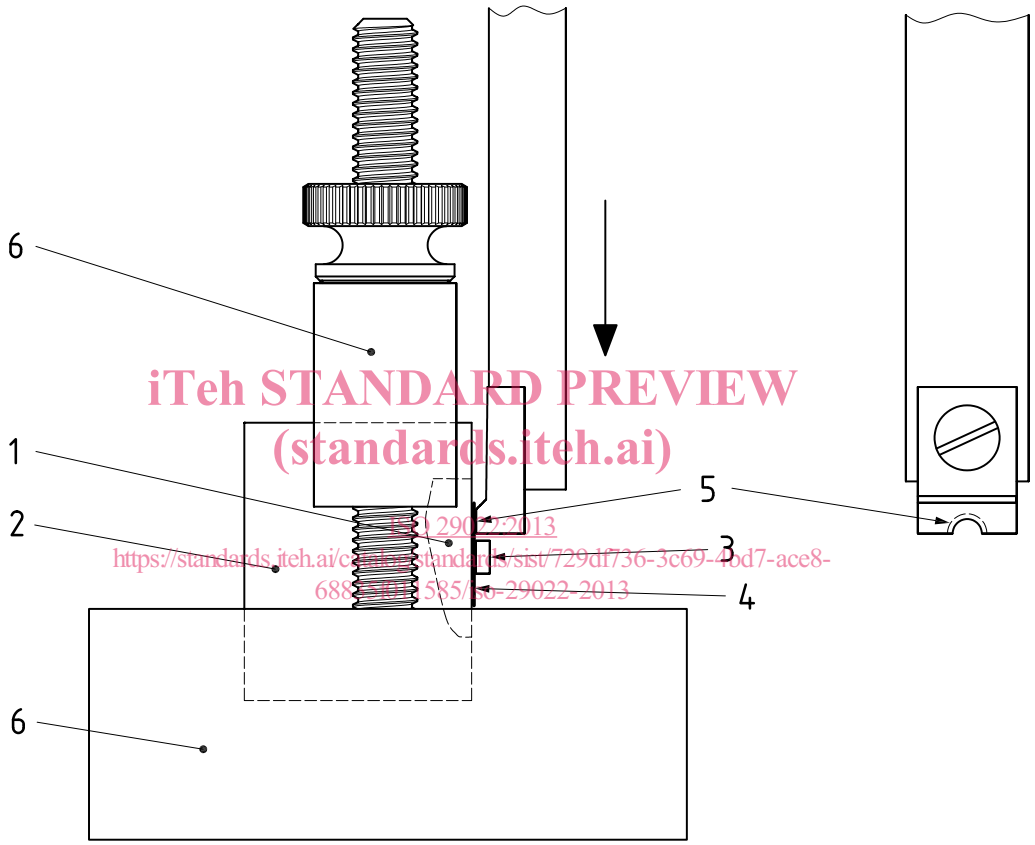
Measure the diameter of at least one composite button (adherend) per set of 15 specimens, as near to the bonding surface as possible, in order to confirm the diameter of the bonding area. Store the bonded samples in water at (37 ± 2) °C for (24 ± 2) h prior to debonding. Test the specimens for bond strength immediately after removal from water.

7 Notched-edge shear test

7.1 Apparatus

The notched-edge shear testing apparatus (see Annex A) for measurement of bond strength consists of a notched-edge shear fixture (a notched-edge crosshead assembly is shown in Figure A.3) mounted to a universal testing machine. Immediately after removal from water, place the bonded sample into the metal sample holder (a test base clamp is shown in Figure A.4). Align the bonded sample in the holder under the testing crosshead with the notched edge centred over the composite button and flush against the tooth.

NOTE An important feature of the crosshead is the thin curved lip edge that applies the load to the specimen [Figures 1 and A.3 b)]. Using a crosshead that does not have this lip edge will increase variability of the results.



Key

1 tooth
2 cured potting material
3 cured composite button
4 cured adhesive
5 notched-edge shear blade
6 test base clamp

Figure 1 — Bonded sample in test base clamp with notched-edge crosshead aligned over composite button