Dental materials — Guidance on testing of adhesion to tooth structure

Produits dentaires — Lignes directrices pour l'essai d'adhésion à la structure de la dent
## Contents

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>1 Scope</td>
<td>1</td>
</tr>
<tr>
<td>2 Normative references</td>
<td>1</td>
</tr>
<tr>
<td>3 Definitions</td>
<td>1</td>
</tr>
<tr>
<td>4 Requirements</td>
<td>1</td>
</tr>
<tr>
<td>5 Sampling</td>
<td>1</td>
</tr>
<tr>
<td>6 Test methods</td>
<td>1</td>
</tr>
<tr>
<td>6.1 Screening tests</td>
<td>2</td>
</tr>
<tr>
<td>6.2 Bond strength measurements</td>
<td>3</td>
</tr>
<tr>
<td>6.3 Gap measurement test for adhesion to dentin</td>
<td>11</td>
</tr>
<tr>
<td>6.4 Microleakage test</td>
<td>12</td>
</tr>
<tr>
<td>6.5 Clinical usage tests</td>
<td>13</td>
</tr>
</tbody>
</table>

### Annex

<table>
<thead>
<tr>
<th>Section</th>
<th>Page</th>
</tr>
</thead>
<tbody>
<tr>
<td>A Bibliography</td>
<td>14</td>
</tr>
</tbody>
</table>
Foreword

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

The main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

- type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;
- type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;
- type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 11405, which is a Technical Report of type 2, was prepared by Technical Committee ISO/TC 106, Dentistry, Subcommittee SC 1, Filling and restorative materials.

This document is being issued in the type 2 Technical Report series of publications (according to subclause G.4.2.2 of part 1 of the ISO/IEC Directives, 1992) as a "prospective standard for provisional application" in the field of adhesion to tooth structure in dentistry because there is an urgent need for guidance on how standards in this field should be used to meet an identified need.

This document is not to be regarded as "International Standard". It is proposed for provisional application so that information and experience of its use in practice may be gathered. Comments on the content of this document should be sent to the ISO Central Secretariat.
A review of this type 2 Technical Report will be carried out not later than two years after its publication with the options of: extension for another two years; conversion into an International Standard; or withdrawal.

Annex A of this Technical Report is for information only.
Introduction

The increasing importance of adhesion in restorative dentistry has made it evident that information is needed on the relative performance of materials which 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 clinical behaviour, they are valuable for screening.

Adhesive materials are used in many types of restorative and prophylactic 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, the intention of this Technical Report to standardize as far as possible different procedures whereby the effect or quality of a bond between a dental material and the tooth structure may be substantiated. By gaining experience with a specific testing system, a correlation between laboratory and clinical performance of the materials should be sought. Data from such correlations may then form the basis for revision of the document and simplification of appropriate testing.
Dental materials — Guidance on testing of adhesion to tooth structure

1 Scope

This Technical Report specifies test methods for evaluation of the adhesive bond between dental materials and tooth structure, i.e. enamel and dentine. It describes two bond strength measurement tests, tensile and shear, a test for measurement of marginal gaps around fillings, a microleakage test, and gives guidance on clinical usage tests for such materials.

2 Normative references

The following standards contain provisions which, through reference in this text, constitute provisions of this Technical Report. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this Technical Report are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.


3 Definitions

For the purposes of this Technical Report, the following definitions apply. [11, 12]

3.1 adhere: 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.

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 by adhesion.

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 substrate: Material upon the surface of which an adhesive-containing substance is spread for any purpose, such as bonding or coating.

4 Requirements

This Technical Report contains no requirements for dental materials.

5 Sampling

The amount of test material shall be sufficient for all planned tests and have the same lot or batch number.

6 Test methods

This Technical Report describes various types of tests:

a) screening tests;

b) bond strength measurements;

c) gap measurement test for adhesion to dentine;
d) microleakage test;
e) clinical usage tests.

For some types, specific tests are described in detail. For other types, guidelines are given. It is not the intention to recommend testing each material by every test; 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 has to be a clinical usage test.

6.1 Screening tests

6.1.1 Introduction

Many screening tests may be necessary in the development of new adhesive materials or for production control. Such tests may be performed on bovine teeth.

However, the chemistry and especially the structure of bovine teeth are not identical to those of human teeth, and results from bovine teeth cannot replace those from human teeth. The results of such tests should not be used for advertising or promotion of the material.

6.1.2 Tooth substrate, storage and preparation

The condition, storage and preparation of teeth, either human or bovine, should be as described in 6.2 to 6.4.

6.1.3 Test methods

6.1.3.1 Type of test

Many types of screening tests are available, for example bond strength measurements, microleakage tests, marginal gap measurements. If bond strength measurements are used, the method adopted should be either

a) tensile (bond broken by a force perpendicular to the tooth surface), or

b) shear (bond broken by a force parallel to the tooth surface).

Methods which introduce “peel” stresses are not acceptable. Many current test methods fail to exert purely tensile or shear forces on the bond. A special problem is the alignment in the tensile test and the correct and reproducible loading in the shear test. It is recommended that similar tests for screening purposes to those described for bond strength measurement on human teeth (6.2.2 and 6.2.3) should be used.

All forces exerted on a bond can be resolved into shear and tensile components and, since there is no direct relation between the results obtained from the two measurements, it is preferable to measure both.

6.1.3.2 Condition of adhesive

Bonding materials may be used in a thin film or in bulk.

As far as possible, materials should be applied in a manner which duplicates their clinical use.

6.1.3.3 Storage of test specimens

Test specimens shall be prepared at (23 ± 2) °C and stored in water at (37 ± 2) °C prior to testing at (23 ± 2) °C. Storage in water for 24 h is normally sufficient to discriminate between those materials which cannot and those which 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 bond.

The recommended procedure is as follows:

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 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 5 s to 10 s.

Test type 3: Long term test after six-month storage in water at 37 °C.

6.1.3.4 Strain rate for bond breakage

The standard rate of loading a bonded specimen is recommended to be (0.75 ± 0.30) mm/min crosshead speed, or more accurately (50 ± 2) N/min. (The stiffness in the various testing machines and bond assemblies varies widely and hence 50 N/min is more meaningful than 0.75 mm/min.)

6.1.3.5 Treatment of results

The bond strength values obtained by tensile or shear testing show large coefficients of variation, i.e. 20 % to 50 %. If the variation is above 50 %, a thorough inspection of the overall procedure is recommended. For screening purposes, six to ten specimens are re-