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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

TECHNICAL ISO

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Contents

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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.

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 Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.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 refarded 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.

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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.

ISO 3696:1987, *Water for analytical laboratory use — Specification and test methods.*

ISO 3823-1:1986, *Dental rotary instruments — Part 1: Steel and carbide burs.*

DIN 69176:1985, *Körnungen aus Elektrokorund und Siliciumkarbid für Schleifmittel auf Unterlagen — Teil 1: Bezeichnung und Korngrössenverteilung.*

3 Definitions

For the purposes of this Technical Report, the following definitions apply. [1], [2]

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 surface 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-

quired to give a meaningful average. Of greater importance at this stage may be inspection of fracture surfaces visually at × 10 magnification and to determine whether the fracture pattern is of an adhesive, cohesive or mixed nature.

6.2 Bond strength measurements

6.2.1 Introduction

The measurement of bond strength may be important when evaluating an adhesive material. However, the technique-sensitivity of such tests and the scatter of results due to variations in the substrate, adhesive and handling limit the importance and use of the results.

Adhesive materials are used for many different purposes in the mouth. The choice of test shall be considered according to the intended use of the material.

This Technical Report describes two types of tests, tensile and shear. In addition, several variations such as application in thin film and bulk, and short or long exposure time to a wet environment are described. A set of tests may be necessary properly to evaluate the bond strength of a material.

When bond strength is to be measured, the raw data are in units of force (newtons). It is desirable to convert this into stress units, i.e. force per unit area (megapascals). Hence, control of the surface for application of the adhesive material is paramount.

6.2.2 Tooth substrate and storage

6.2.2.1 Substrate

Human permanent teeth are required for the measurement of clinically relevant bond strength. When measuring bond strength to dentine, this Technical Report recommends that the superficial dentine, i.e. as close to enamel as possible, at the buccal aspect of the third permanent molar from patients in the age group 18 to 25 years be used to reduce variations. However, if this restriction is difficult to fulfil, the other molars or premolars may be used, provided the teeth used are listed in the report of the investigation.

6.2.2.2 Time after extraction

There is increasing evidence that changes in dentine occur after extraction which 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 clearly 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. Do not use root-filled teeth. Teeth which have been extracted for longer than six months may undergo degenerative changes in dentinal protein.

6.2.2.3 Condition of teeth

The 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. There is some evidence to suggest that different areas of the dentition may give different results with bonding to dentine and enamel. Bearing in mind the number of measurements required, it is not possible to control variables such as age of the donating patient, cultural and dietary history, state of health, or to standardize the composition and structure of the teeth.

6.2.2.4 Storage of teeth

Prior to storage, the teeth should be thoroughly washed in running water and all blood and adherent tissue removed, preferably by the clinician. Teeth should be placed immediately after extraction in distilled water (grade 3, ISO 3696) or in a 0,5 % chloramine bacteriostatic/bactericidal solution for a maximum of 1 week and thereafter stored in distilled water in a refrigerator at a nominal 4 °C. To minimize deterioration, the storage medium should be replaced periodically. It is essential that no other chemical agents be used. Such agents may be absorbed by, and alter, tooth substance.

6.2.2.5 Tooth surface preparation

A standard, reproducible, flat surface is required. Tooth surfaces shall be kept wet at all times. Exposure of a tooth surface to the air for more than 15 minutes may cause irreversible changes in bonding character. Dentine is especially sensitive to dehydration. The use of high-speed handpieces to prepare tooth surfaces for bonding studies is to be avoided. A standard surface is recommended such as that produced against a silicon carbide abrasive paper with a mean grit size of approximately 18 µm (Grade 1000, DIN 69176) under running water. To control the planing and the angle of the surface during surface preparation, the tooth should be mounted in a holder by means of a dental die stone or a cold-curing resin. The absorption of resin and the heat of polymerization may adversely affect the tooth. Avoid smearing of embedding material on the tooth surface during final preparation against the 1000 grade silicon carbide, e.g. by

mounting the tooth above the surface of the embedding material.

6.2.2.6 Re-use of teeth

Fresh teeth should preferably be used. If it is not possible to use a fresh tooth for every measurement, it is essential that "control" measurements using a standard material be undertaken frequently to check for irreversible changes caused by previous treatments.

6.2.3 Tensile bond strength

6.2.3.1 Apparatus

The various parts of the tensile test specimen are shown in figure 1: a) is a holder for mounting of teeth (tooth cup); b) is the counterpart either as a holder for the adhesive materials applied in bulk, i.e. the material cup or as the second substrate, i.e. a small rod, when applying the adhesive in thin film. For light-curing systems, the material cup may be made totally of a transparent material, or alternatively it may be split into two parts with a smaller cup in a transparent resin and the connection to c) in metal, where c) is the coupling to the alignment rod.

Figure 2 shows the bonding alignment apparatus with alignment rods, a), and the bonding alignment block, b).

Figure 3 shows the measurement alignment block, b), and the necessary alignment rods, a).

Figure 4 shows the polishing block.

Dimensions in millimetres

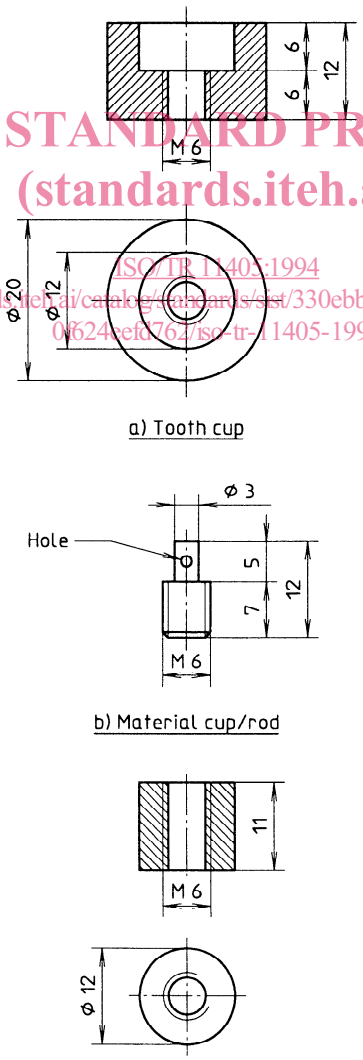
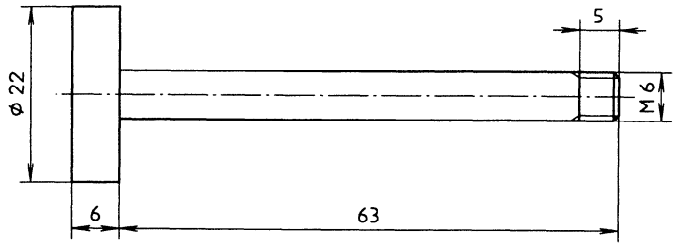
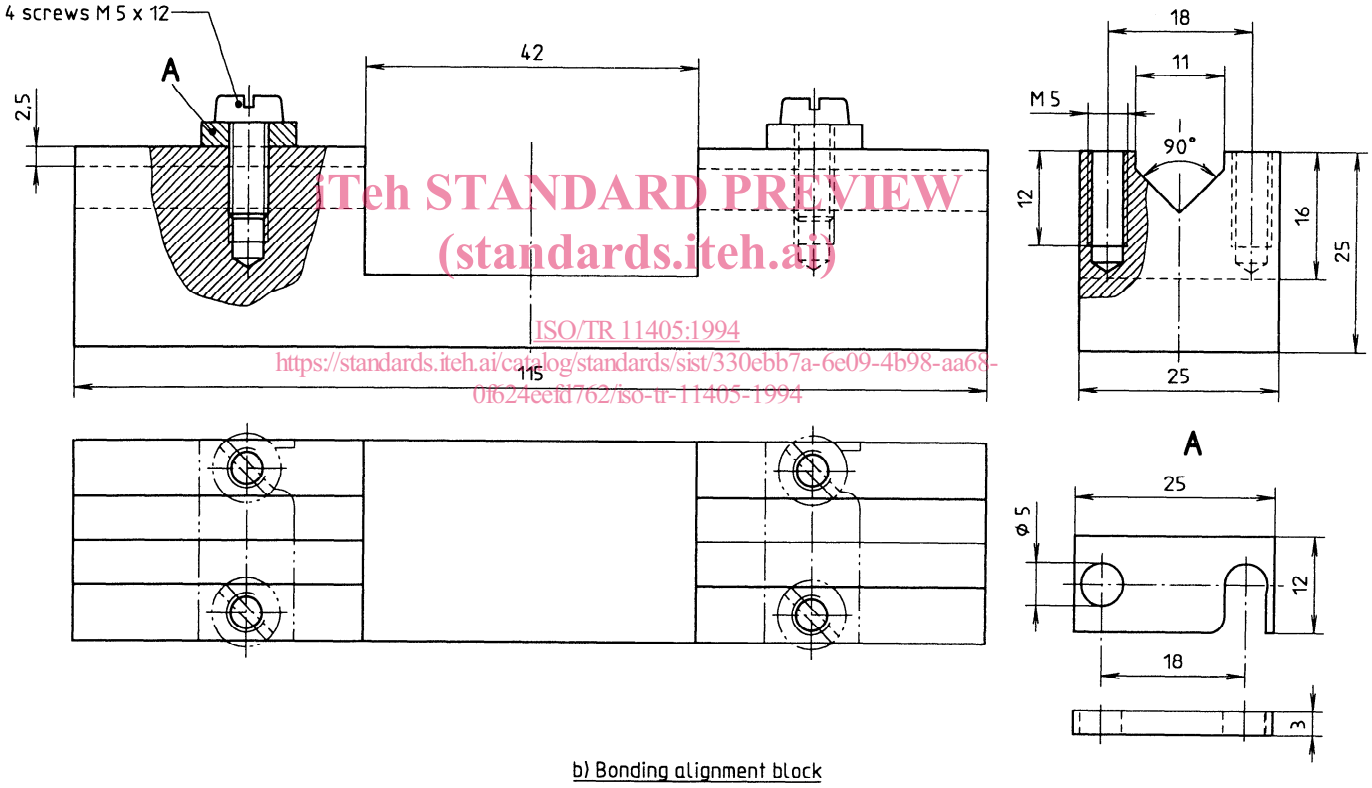


Figure 1 — Tensile specimen

Dimensions in millimetres



a) Alignment rod for bonding block (2 needed)



b) Bonding alignment block

Figure 2 — Bonding alignment apparatus