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Dentistry — Compatibility testing for metal-ceramic and ceramic-ceramic systems

Médecine bucco-dentaire — Essais de compatibilité pour systèmes métallo-céramiques et céramo-céramiques

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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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT) see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials*.

This third edition cancels and replaces ISO 9693-1:2012 and ISO 9693-2:2016, which have been technically revised.

The main changes compared to the previous editions are as follows:

— this document focuses only on the compatibility of veneering ceramics fired on to metallic or ceramic substrate materials. Tests of dental veneering ceramics themselves, whether for either metal or ceramic substructures, are now contained in ceramics standard ISO 6872;

— some clauses are relevant for all materials (e.g. measurement of thermal expansion coefficients);

— the de-bonding test (formerly denoted the Schwickerath bond characterization test) for veneering ceramic fired to a substrate is retained for metallic substrates and for ceramic substrates with an elastic modulus less than 250 GPa;

— a new requirement has been added for metal-ceramic systems to undergo thermal shock testing according to either of two protocols.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

Dental veneering ceramics and metal alloys or substructure ceramics are suitable materials for the fabrication of dental restorations. Compatibility between the veneering ceramic and the substructure material under mechanical and thermal loading is essential if they are to function in a prosthetic construction.

This document specifies requirements and test methods for assessing the risk of failure associated with masticatory forces and the oral environment.

Dentistry — Compatibility testing for metal-ceramic and ceramic-ceramic systems

1 Scope

This document specifies requirements and test methods to assess the thermomechanical compatibility between a veneering ceramic and a metallic or ceramic substructure material used for dental restorations.

This document applies only to the materials used in combination. Conformity cannot be claimed for a single material.

For requirements for ceramic materials, see ISO 6872. For requirements for metallic materials see ISO 22674.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 1942, *Dentistry — Vocabulary*

ISO 6872:2015, *Dentistry — Ceramic materials*

ISO 22674:2016, *Dentistry — Metallic materials for fixed and removable restorations and appliances*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 1942, ISO 6872, ISO 22674 and the following apply.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at http://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

3.1
veneering ceramic
full structure of fired ceramic layers applied to a substrate material

3.2
conditioning
process of treating the substructure to enhance the bonding of the *veneering ceramic* (3.1)

3.3
liner
substance, applied to the substructure and fired under appropriate time-temperature conditions, with the purpose to improve aesthetics and/or adherence of the veneering ceramic to the coated substructure-surface

4 Requirements

4.1 Biocompatibility

Specific qualitative and quantitative test methods for demonstrating freedom from unacceptable biological risks are not included in this document, but it is recommended that, for the assessment of such biological risks, reference be made to ISO 10993-1 and ISO 7405.

4.2 Physical properties

4.2.1 General

The individual component materials shall fulfil the requirements of ISO 6872 for ceramics or ISO 22674 for metallic materials. The materials shall also be in accordance with the requirements of 4.2.2 to 4.2.4 where applicable.

In particular, the elastic modulus of the substrate material shall be determined in order to interpret the measurements in the de-bonding test.

4.2.2 Thermal expansion

The coefficients of thermal expansion of the substructure ceramic and the veneering ceramic shall be determined according to ISO 6872.

The coefficients of thermal expansion of the metallic substructure material shall be determined according to ISO 22674.

The same measurement protocol shall be used for both the veneering and substructure materials (e.g. same lowest temperature).

Test in accordance with 6.1.

NOTE The measured values for coefficients of linear thermal expansion are compared with the manufacturer's values as a means of quality control, but the values cannot provide an assurance that the ceramic or metallic substructure and ceramic veneer are compatible.

4.2.3 De-bonding/crack-initiation test

4.2.3.1 Metallic substructure material

The debonding/crack-initiation strength of the metallic substructure material and at least one, nominated selected dental veneering ceramic shall be greater than 25 MPa.

Test in accordance with 6.4.

4.2.3.2 Ceramic substructure material

For ceramic-ceramic combinations this test shall be used for zirconia-veneering ceramic only.

The de-bonding/crack-initiation strength of the ceramic substrate material and at least one nominated dental veneering ceramic present shall be greater than 20 MPa.

Test in accordance with 6.4.

NOTE According to 6.4, the de-bonding test only applies to materials with an elastic tensile modulus less than or equal to 250 GPa. This excludes some stiff ceramic materials such as alumina.

4.2.4 Thermal shock resistance

At least one thermocycling test for resistance to thermal shock shall be performed according to 6.5.3 or 6.5.4 and the results be reported according to Clause 7.

5 Sampling

5.1 Metallic substructure material

The sample shall be adequate for preparing the test specimens in accordance with this document. All of the metallic material shall be from the same lot and unused.

5.2 Ceramic substructure material

The sample shall be adequate to prepare the specimens for testing in accordance with this document. All of the ceramic material shall be from the same lot.

5.3 Veneering ceramic

The sample shall consist of a sufficient amount of veneering ceramic to carry out all tests in accordance with this document, including the tests performed according to ISO 6872. Perform the tests on the colour/shade variant most commonly used. All of the material tested shall be from the same lot.

6 Test methods

6.1 Linear thermal expansion

6.1.1 Metallic materials

Test methods for metallic materials shall be in accordance with ISO 22674:2016, 8.13.

6.1.2 Ceramic materials

Test methods for ceramic materials shall be in accordance with ISO 6872:2015, 7.4.

6.2 Glass transition temperature

The glass transition temperature shall be determined in accordance with ISO 6872:2015, 7.5.

6.3 Young's modulus

Test methods for elastic modulus of metallic materials shall be in accordance with ISO 22674:2016, 5.5.

6.4 De-bonding/crack-initiation test

6.4.1 Apparatus

6.4.1.1 Flexural-strength testing machine for three-point bending, having a span between supports of (20 ± 0,1) mm and capable of a cross-head-speed of (1,5 ± 0,5) mm/min. Supports and bending piston shall be rounded to a radius of (1,0 ± 0,1) mm and shall be made from hardened steel or other hard material having a hardness greater than 40 HR (Rockwell C-scale) and have a smooth surface with a roughness less than 0,5 µm Ra.

6.4.1.2 Furnace appropriate to fire the veneering ceramic to the substructure acc. to the manufacturer's recommendation.

6.4.2 Preparation of test specimens

Prepare six specimens (either six metallic or six zirconia) with a size of (25 ± 1) mm × (3 ± 0,1) mm × (0,5 ± 0,05) mm in accordance with the manufacturer's procedure for processing the substructures for prostheses. Condition the specimens and follow the procedure described in the instructions for use (e.g. cleaning, sandblasting, oxidation).

Before applying the ceramic to the test specimens, calibrate the furnace according to the manufacturer's recommendation and test-fire the ceramic material to obtain the appropriate firing grade and surface gloss of the veneering ceramic materials. If necessary, adjust the firing temperatures or holding times.

According to the instructions for use, add dental veneering ceramic to each specimen to form a total ceramic thickness of (1,1 ± 0,1) mm after firing (see Figure 1). The ceramic layer shall have a rectangular shape and extend the full 3 mm width of the substrate.

If necessary add additional dental veneering ceramic to obtain the required thickness and shape, and fire it. Carefully trim the rectangular shape with a disc. If necessary remove ceramic from the side of the substructure material in order to keep its overall shape.

Submit each specimen to a glaze firing in accordance with the instructions for use.

Dimensions in millimetres

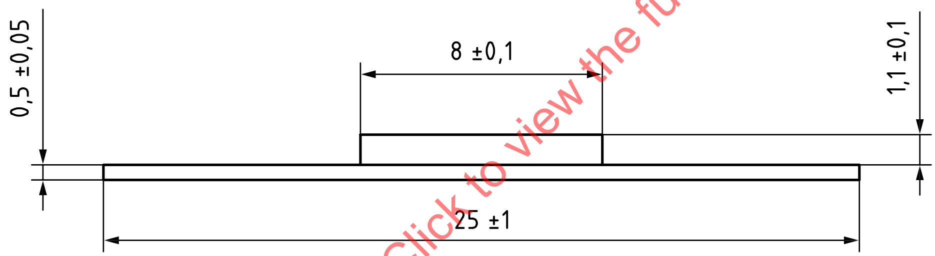


Figure 1 — Test specimen configuration

6.4.3 Determination of fracture force

6.4.3.1 Procedure

The fired specimens are placed in the bending apparatus with the veneering ceramic positioned symmetrically on the side opposite to the applied load. The force is applied at a constant cross-head speed of (1,5 ± 0,5) mm/min and recorded up to failure. The fracture force F_{fail} (in newtons) for each of six specimens is measured for specimens failing by a de-bonding crack occurring at one end of the ceramic layer. Specimens failing by cracks in the middle of the ceramic layer shall be replaced until six appropriate specimens are obtained.

The failure (abortion) criteria is the abrupt decline of the force by 5 % or more.

6.4.3.2 Evaluation of the de-bonding/crack-initiation strength

6.4.3.2.1 General procedure

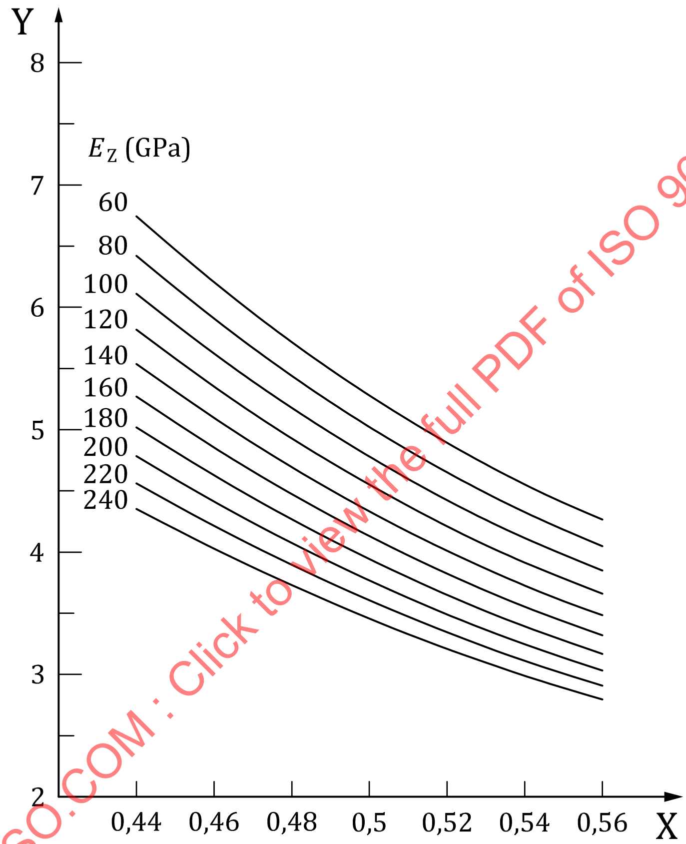
Multiply the fracture force F_{fail} with the coefficient k. Determine coefficient k by reading from Figure 2. The coefficient k is a function of the thickness of the metal or ceramic substrate d_Z (0,5 ± 0,05) mm, and the value of Young's modulus E_Z of the metal or ceramic substrate.

To read the value k for a certain thickness d_Z, first pick the curve for the proper value E_Z, then read the value k from the picked curve for the thickness d_Z.

The de-bonding/crack-initiation strength τ_b is calculated using the following formula:

$$\tau_b = k \times F_{fail} \quad (1)$$

The system passes the test if four specimens out of six, or more, are in conformity the requirement specified in 4.2 (≥66 %). If only two or fewer comply, the system fails. If three pass, repeat the test with another six specimens. If five or six pass, the system complies (= 8 out of 12 or 66 %).



Key

X d_Z [mm]

Y k [mm^{-2}]

Figure 2 — Diagram to determine the coefficient k as a function of substrate thickness d_Z and Young's modulus E_Z of the substrate

6.4.3.2.2 Alternative procedure

The de-bonding/crack-initiation strength τ_b can also be calculated numerically on the basis of the flow chart shown in Figure 3.

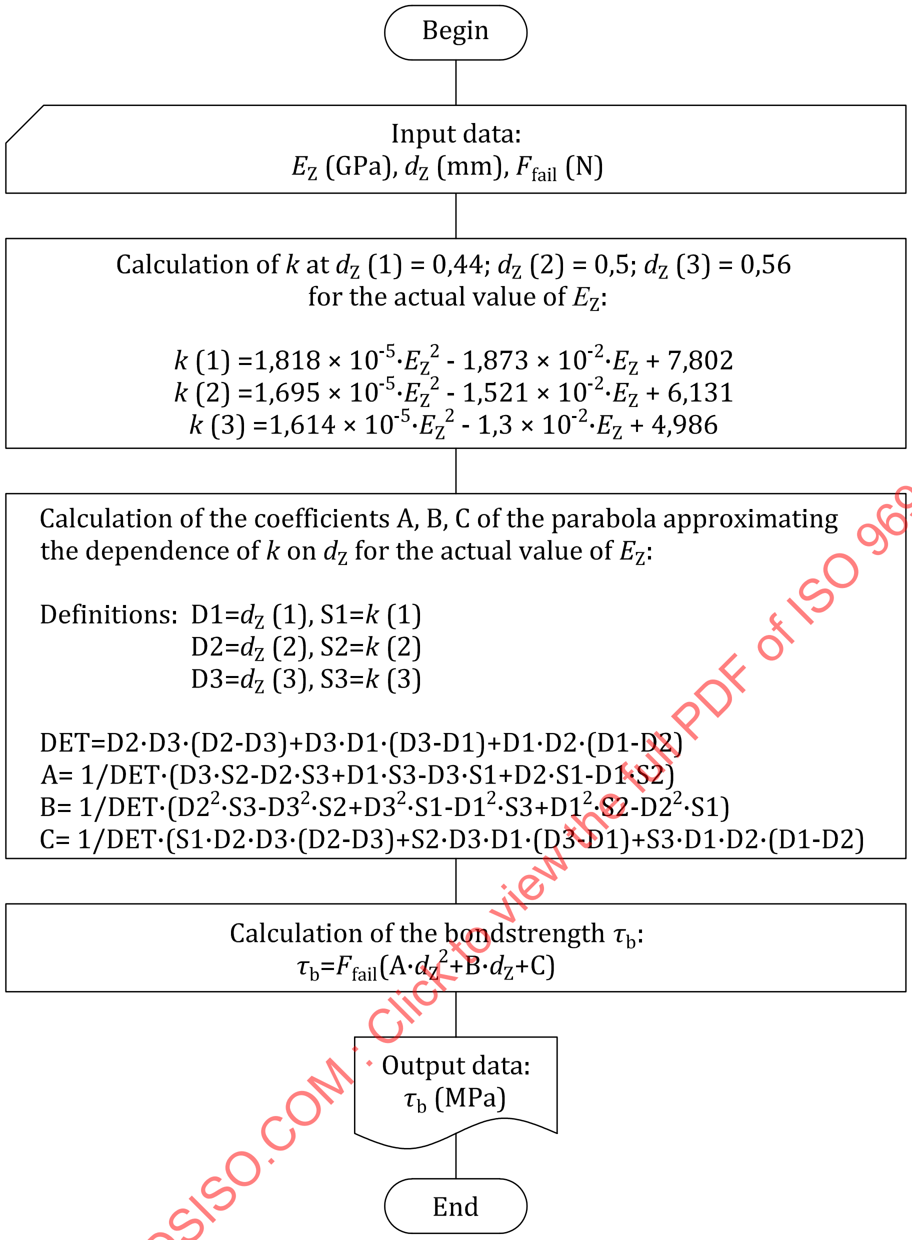


Figure 3 — Flow chart for numerical calculation of de-bonding/crack-initiation strength

6.4.4 Test report

Prepare a test report in accordance with Clause 7.

6.5 Thermal shock testing

6.5.1 General

The following two procedures (6.5.3 and 6.5.4) shall be taken as examples on how to conduct the test properly. The test set-up may however be modified according to individual material combinations tested and overall experience of the test house involved. The whole detailed test set-up and result shall be part of the test report (see Clause 7).

6.5.2 Preparation of test specimen

Prepare, according to the instructions for use, either at least one veneered prosthesis of three units (bridgework, with the geometry of tooth positions 11 to 13 or 14 to 16) or, if multiple units are not indicated, at least five anterior crowns (geometry of tooth position 11 or 21). Apply the veneer to the substructure material with at least two firings.

NOTE The numerical tooth designations are in accordance with ISO 3950.

6.5.3 Thermocycling test with fixed temperature interval

6.5.3.1 Apparatus

6.5.3.1.1 Container with cold water maintained at (10 ± 10) °C.

6.5.3.1.2 Container with water maintained at its boiling point.

6.5.3.1.3 Wire basket suitable for transferring the test specimen(s) rapidly between the containers, and preventing them from coming in direct contact with the walls or bottom while ensuring that the test piece or pieces remain submerged.

6.5.3.2 Procedure

a) Place test objects in the wire basket so that they are not contacting each other and are not under mechanical stress.

b) Place the basket in the boiling water for (30 ± 5) s. When executed for the first time this step is counted as the first thermal shock.

c) Transfer the basket from the boiling water to the cold water within 3 s and keep it submerged for (30 ± 5) s. When executed for the first time this step is counted as the second thermal shock.

d) Repeat steps b) and c) at least eight times or until failure of the materials is apparent. Transfer the test specimens between containers within 3 s.

e) After the last quenching, remove objects and dry them.

f) Examine specimens according to 6.5.3.3.

6.5.3.3 Examination

Examine each specimen for cracks by light microscopy at magnifications up to 10 × with trans-illumination.

If cracking is not observed immediately after the test, repeat the examination after 48 h to test for delayed cracking.

6.5.3.4 Test report

Prepare a test report in accordance with Clause 7.

6.5.4 Thermocycling test with increasing temperature interval

6.5.4.1 Apparatus

6.5.4.1.1 Container with cold water maintained at (10 ± 10) °C by an external temperature control element

6.5.4.1.2 Furnace (hot air) capable of maintaining temperatures between (80 ± 5) °C and at least (165 ± 5) °C.

6.5.4.1.3 Wire basket suitable for rapid transfer of the test specimens between the water container and the furnace and preventing them from coming in direct contact with the walls or bottom while ensuring that the specimens remain submerged in the water.

6.5.4.2 Procedure

a) Place test objects in the wire basket so that they are not contacting each other and are not under mechanical stress.

b) Place the basket in the furnace to heat the specimens to 80 °C and maintain for at least 10 min. This process is not counted as thermal shock.

c) Transfer the test objects in the basket into the cold water with transfer time of less than 3 s. Maintain the test objects in the container for (30 ± 5) s. This step is counted as a single thermal shock.

d) Remove the specimens from the cold water and dry them. Examine for cracks at room temperature by light microscopy at magnifications up to 10 × with trans-illumination. If no cracks are apparent, continue with step e). If any specimen exhibits cracking, report the result according to 6.5.4.3.

e) Increase the temperature of the furnace by (12,5 ± 2,5) °C and repeat steps b) to d).

Perform test until final failure of all specimens or until at least five thermal shocks (quenching) have been performed, whichever occurs first.

An initial furnace temperature higher than 80 °C may be used.

NOTE The holding time in the furnace of 10 min is intended to ensure that the specimen has a uniform temperature.

6.5.4.3 Test report

Prepare a test report in accordance with Clause 7.

7 Test report

Prepare a test report and report the following:

a) identification of the materials tested, including product names and lot numbers;

b) linear coefficient of thermal expansion and glass transition temperature of the veneering ceramic according to 6.1 and 6.2;

c) linear coefficient of thermal expansion of the substructure material according to 6.1;

d) the characteristic de-bonding strength obtained according to 6.4:

   1) dimensions and thickness ratios of test specimens;

   2) the value of elastic modulus E_z used in the analysis of measurements;

   3) the results of each measurement (in MPa);

e) details of the preparation of the test specimens for thermocycling, including diagrams or photographs with a scale indicating the relative thicknesses of substructure and veneer;

