INTERNATIONAL STANDARD

ISO 9693-2

First edition
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Dentistry — Compatibility testing —

Part 2: Ceramic-ceramic systems

Médecine bucco-dentaire — Essais de compatibilité —

Partie 2: Systèmes céramo-céramiques

Reference number
ISO 9693-2:2016(E)

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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 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 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 2, *Prosthodontic materials*.

This first edition, together with ISO 9693-1, cancels and replaces ISO 9693:1999.

This part of ISO 9693 replaces the bi-material portions of ISO 9693:1999 to focus only on the compatibility of veneering porcelains fired onto substrate ceramics. Tests of all ceramic materials for either metal or ceramic substructures are now contained in a global ceramics standard ISO 6872. Some elements of ISO 9693:1999 remain for all materials (e.g. measurement of thermal expansion coefficients) and one remains only for porcelain fired to zirconia (Schwickerath bond characterization test). New requirements have been added for porcelain-ceramic systems, including thermal shock testing for ceramic-ceramic compatibility (allowing many protocols that are in widespread use within industry).

ISO 9693 consists of the following parts, under the general title *Dentistry — Compatibility testing*:

— *Part 1: Metal-ceramic systems*

— *Part 2: Ceramic-ceramic systems*

Introduction

Dental porcelains and substructure ceramics are suitable for use in fabrication of all-ceramic dental restorations. Their compatibility under mechanical and thermal loading is essential if they are to function in a prosthetic construction. This part of ISO 9693 sets out requirements and test methods for allowing the risks associated with masticatory forces and the oral environment to be assessed.

Specific qualitative and quantitative requirements for freedom from biological hazards are not included in this International Standard, but, in assessing possible biological hazards, reference can be made to ISO 10993-1 and ISO 7405.

Dentistry — Compatibility testing —

Part 2:
Ceramic-ceramic systems

1 Scope

This part of ISO 9693 specifies requirements and test methods to assess the compatibility of ceramic-ceramic materials used for dental restorations by testing composite structures.

The requirements of this part of ISO 9693 apply when different ceramic components are used in combination. Compliance cannot be claimed for either ceramic alone.

For requirements of ceramic materials, see ISO 6872.

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 6872:2015, *Dentistry — Ceramic materials*

3 Terms and definitions

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

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

3.2
conditioning
process of treating the ceramic substructure to enhance the bonding of the veneer ceramic

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

4 Requirements

4.1 Biocompatibility

See the Introduction for guidance on biocompatibility.

4.2 Physical properties

4.2.1 General

The individual materials shall fulfil the requirements of ISO 6872 and the thermo-mechanical compatibility tests shall be performed where applicable. The materials shall also comply with the requirements of 4.2.2 to 4.2.4.

4.2.2 Thermal expansion

The coefficients of thermal expansion of the substructure ceramic and the veneering ceramic shall have been determined according to ISO 6872:2015, 7.4.

It is imperative that the same protocol be used for both the veneering and ceramic substructure (e.g. same lowest temperature).

Test in accordance with 6.1.

4.2.3 De-bonding/crack-initiation test (zirconia-porcelain only)

When tested according to 6.3, the de-bonding/crack-initiation strength of the zirconia material and at least one specified (named) dental veneering ceramic present shall be greater than 20 MPa. Test in accordance with 6.3.

4.2.4 Thermal shock resistance

At least one test for resistance to thermal shock shall be performed according to 6.4.2 or 6.4.3.

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 substructure and ceramic veneer are compatible.

5 Sampling

5.1 Substructure dental ceramic

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

5.2 Dental porcelain

Take a sufficient amount of veneering ceramic to carry out the necessary tests in accordance with this International Standard. Perform test with a colour/shade most commonly used. All of the material tested shall be from the same lot.

6 Test methods

6.1 Linear thermal expansion

See ISO 6872:2015, 7.4.

6.2 Glass transition temperature

See ISO 6872:2015, 7.5.

6.3 De-bonding/crack-initiation test (zirconia-porcelain only)

6.3.1 Preparation of test specimens

Prepare six zirconia specimens (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, observing the manufacturer's instructions. According to the manufacturer's instructions, add dental porcelain 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 porcelain 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 zirconia in order to keep its overall shape.

Submit each specimen to a glaze firing in accordance with the manufacturer's instructions.

Dimensions in millimetres

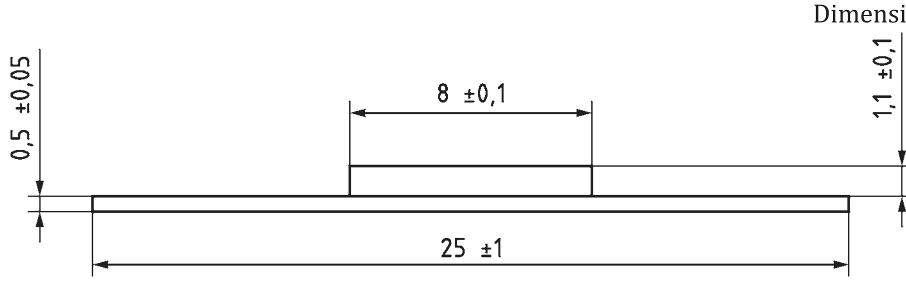

Figure 1 — Test specimen configuration

6.3.2 Determination of fracture force

6.3.2.1 Apparatus

Flexural-strength testing machine for three-point bending, having a span between supports of 20 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 mm.

6.3.2.2 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.

6.3.2.3 Evaluation of de-bonding/crack-initiation strength

6.3.2.3.1 General

The fracture force F_{fail} has to be multiplied with a coefficient k. Coefficient k can be read from Figure 2. The coefficient k is a function of the thickness of the zirconia substrate d_Z (0,5 ± 0,05) mm, and the value of Young's modulus E_Z of the zirconia 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 equation:

$$\tau_b = k \times F_{fail}$$

The ceramic-ceramic system passes the test if four specimens out of six, or more, comply with 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 %).

Dimensions in millimetres

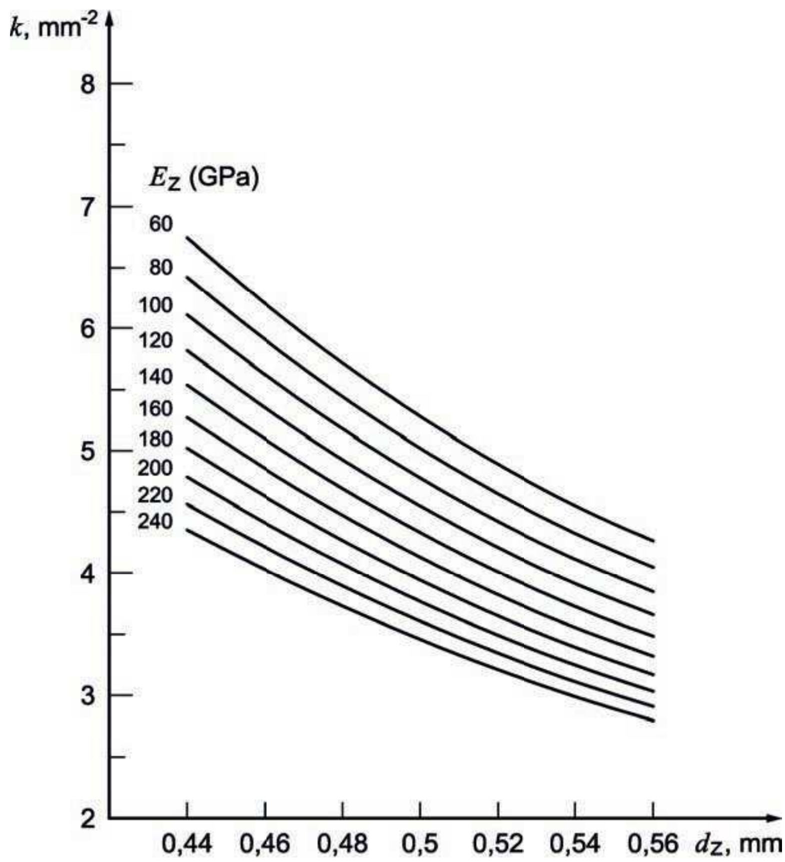

Figure 2 — Diagram to determine the coefficient *k* as a function of zirconia substrate thickness d_Z and Young's modulus E_Z of the zirconia

6.3.2.3.2 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.

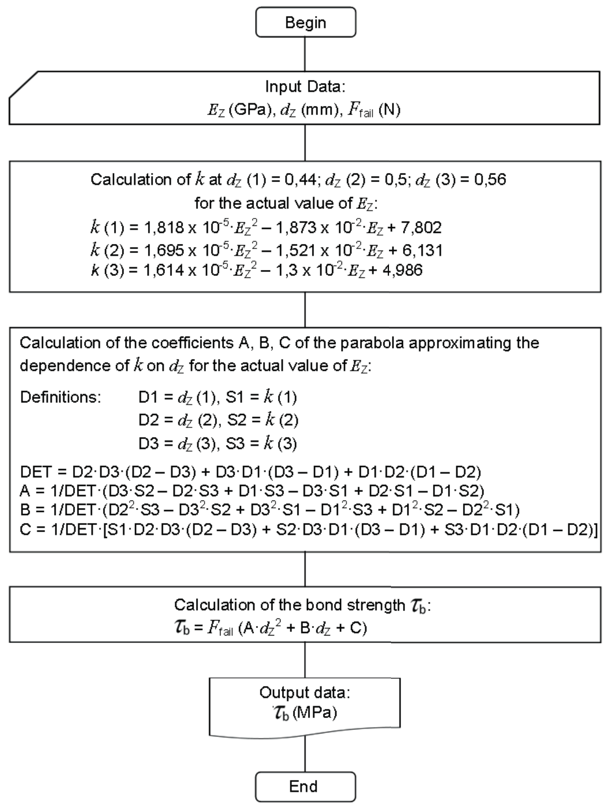

Figure 3 — Flow chart for numerical calculation of de-bonding/crack-initiation strength

6.3.3 Test report

For test report see Clause 7.

6.4 Thermal shock testing

6.4.1 General

The two procedures described below (6.4.2 and 6.4.3) 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 official test report (see Clause 7).

6.4.2 Thermocycling test with fixed temperature interval

6.4.2.1 Preparation of test specimens

Prepare, according to the manufacturers' instructions, either at least one veneered prosthesis of three units (molar bridge, geometry of tooth positions 14 to 16) or, if multiple units are not indicated, at least five anterior crowns (geometry of tooth position 11). Apply the veneer to the framework with at least two firings.

6.4.2.2 Apparatus

6.4.2.2.1 Container with cold water maintained at 0 °C to 20 °C by an external temperature control element.

6.4.2.2.2 Container with boiling water maintained by an external heating element.

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

6.4.2.3 Procedure

a) Place test objects in the wire basket so that they are not in contact or under mechanical stress.

b) Place the basket in boiling water; this process is counted as first thermal shock [only when executing b) for the first time]. Dwell time: (30 ± 5) s.

c) Transfer the basket from the boiling water to the cold water (0 °C to 20 °C) within 3 s; this process is counted as second thermal shock [only when executing c) for the first time]. Dwell time: (30 ± 5) s.

d) Return the basket to the boiling water [transfer time: < 3 s; dwell time: (30 ± 5) s].

e) Repeat until the test objects have been quenched 10 times or until (obvious) failure.

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

g) Examine specimens according to 6.4.2.4.

6.4.2.4 Examination

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

Repeat the examination after 48 h to test for retarded cracking (if cracking is not observed immediately after the test).

6.4.2.5 Test report

For test report, see Clause 7.

6.4.3 Thermocycling test with increasing temperature interval

6.4.3.1 Preparation of test specimens

Prepare specimens according to 6.4.2.1.

6.4.3.2 Apparatus

6.4.3.2.1 Container with cold water maintained at 0 °C to 20 °C by an external temperature control element

6.4.3.2.2 Furnace (hot air) with starting temperature at 80 °C and a capability to reach at least 165 °C.

6.4.3.2.3 Wire basket suitable for rapidly transferring the test specimens between the containers and preventing them coming in direct contact with the walls or bottom while ensuring that the test piece remains submerged.

6.4.3.3 Procedure

a) Place test objects in the wire basket so that they are not in contact or under mechanical stress.

b) Place the basket in hot air (furnace) at 80 °C. Dwell time > 10 min. This process is not to be counted as thermal shock. Apply 80 °C only when starting the procedure. Throughout the process the temperature of the furnace is subsequently increased when specimen shows no defects [compare steps d) and e)].

c) Transfer the test objects in the basket into the cold water with transfer time of less than 3 s. Keep the test objects in this condition for (30 ± 5) s. This process is counted as thermal shock.

d) Remove the specimens from the cold water and dry them. Test for cracks at room temperature by light microscopy at magnifications up to 10 × with transillumination. If no cracks appear, continue with step e). If any specimens are defective, directly go to 6.4.3.4.

e) Increase the temperature of the furnace by 10 °C to 15 °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 is earlier.

6.4.3.4 Test report

For test report see Clause 7.

7 Test report

Report the following parameters:

a) tested materials;

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 ceramic according to 6.1;

d) results obtained from the de-bonding/crack initiation test in MPa (ZrO_2 systems only) according to 6.3, including at least the following information:

   1) tested materials,

   2) results obtained from the crack initiation test in MPa;

e) results obtained from the thermocycling test with fixed temperature interval according to 6.4.2 (if applicable) including the following:

   1) dimensions and thickness ratios of test specimens,

   2) detailed test set-up covering number and preparation of test specimens (6.4.2.1), apparatus (6.4.2.2) and procedure (6.4.2.3),

   3) how many specimens have failed during the test (after how many thermal shocks),

   4) how many specimens have failed immediately after performing the test,

   5) how many specimens have failed 48 h after performing the test;

f) results obtained from the thermocycling test with increasing temperature interval according to 6.4.3 (if applicable) including the following:

   1) dimensions and thickness ratios for specimens used in this test,

2) detailed test set-up covering number and preparation of test specimens (6.4.3.1), apparatus (6.4.3.2) and procedure (6.4.3.3),

3) how many specimens have failed at which temperature difference (and after how many thermal shocks);

g) name of the responsible person and the testing laboratory;

h) date of test report and signature of responsible person.

Bibliography

[1] ISO 7405, *Dentistry — Evaluation of biocompatibility of medical devices used in dentistry*

[2] ISO 9693-1, *Dentistry — Compatibility testing — Part 1: Metal-ceramic systems*

[3] ISO 10993-1, *Biological evaluation of medical devices — Part 1: Evaluation and testing within a risk management process*

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