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Dentistry — Compatibility testing —

Part 2:
Ceramic-ceramic systems

Médecine bucco-dentaire — Essais de compatibilité —

Partie 2: Systèmes céramiques-céramiques

[Revision of second edition (ISO 9693:1999) and of ISO 9693:1999/Amd 1:2005]

ICS: 11.060.10

ISO/CEN PARALLEL PROCESSING

This draft has been developed within the International Organization for Standardization (ISO), and processed under the **ISO lead** mode of collaboration as defined in the Vienna Agreement.

This draft is hereby submitted to the ISO member bodies and to the CEN member bodies for a parallel five month enquiry.

Should this draft be accepted, a final draft, established on the basis of comments received, will be submitted to a parallel two-month approval vote in ISO and formal vote in CEN.

To expedite distribution, this document is circulated as received from the committee secretariat. ISO Central Secretariat work of editing and text composition will be undertaken at publication stage.

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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.

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.

ISO 9693 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials*.

This first edition cancels and replaces the bi-material portions of the last edition (ISO 9693:1999) to focus only on the compatibility of veneering porcelains fired onto substrate ceramics. Tests of all ceramic materials for either metal and 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 as are in widespread use within industry).

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 standard 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 it is recommended that, in assessing possible biological hazards, reference 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, and compliance may not 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, *Dentistry — Ceramic materials*

ISO 9693-1 (Dentistry-Compatibility testing – Part 1: Metal-ceramic systems)

3 Terms and definitions

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

3.1
conditioning
process of treating the ceramic substructure to enhance the bonding of the veneer ceramic

3.2
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 Properties

4.2.1 Physical properties

The individual materials shall fulfill the requirements of ISO 6872, the thermo-mechanical compatibility tests shall be performed where applicable, including compliance with relevant requirements:

4.2.1.1 Thermal expansion

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

The coefficient of thermal expansion for the substructure ceramic shall exceed the coefficient of linear thermal expansion of the veneering ceramic over the temperature range from 50° C to the onset of the glass transition temperature (T_g) of the veneering ceramic or 500° C, whichever is the lesser temperature.

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

Test in accordance with 6.1.

4.2.1.1.1 Bond characterization (zirconia-porcelain only)

When tested according to 6.3, the debonding/crack-initiation strength of the zirconia material and at least one specified (named) dental veneering ceramic present shall be greater than 25 MPa. Test in accordance with 6.4.

4.2.1.2 Thermal shock resistance

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.

It is therefore required that at least one test for resistance to thermal shock be performed according to 6.4.1 or 6.4.2. Manufacturers shall supply dimensions and thickness ratios for specimens used in this test.

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 batch.

5.2 Dental porcelain

Take a sufficient amount of veneering ceramic to carry out the necessary tests in accordance with this International Standard. If there is more than one shade of opaque, dentine and enamel ceramics take equal quantities of each shade and mix thoroughly, respectively.

6 Test methods

6.1 Linear thermal expansion

See ISO 6872, 7.4.

6.2 Glass transition temperature

See ISO 6872, 7.5.

6.3 Bond characterization (Schwickerath crack initiation test) for zirconia systems

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.

Place tab or disc underneath assembled split mould. Use the circular opening of the latter to create a cylinder of packed veneering ceramic powder on the surface of the tab or disc. Gently remove both parts of the mould from the specimen. Repeat the procedure for each tab or disc.

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 body ceramic. If necessary, adjust the firing temperatures or holding times.

Add body 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 body 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 zirconia in order to keep its overall shape.

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

Dimensions in mm

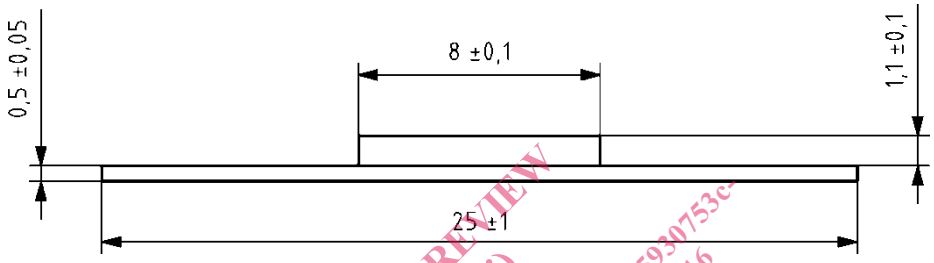

Figure 1 — Test specimen configuration

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 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 debonding 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.

6.3.2.2.1 Evaluation of debonding/crack-initiation strength

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 debonding/crack-initiation strength τ_b is calculated using the equation:

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

The zirconia-porcelain system passes the test if four or more specimens comply with the requirement specified in 4.3.3. If fewer than four specimens comply with the requirement specified in 4.3.3, repeat the test.