INTERNATIONAL STANDARD

ISO 9693

Second edition
1999-12-15

Metal-ceramic dental restorative systems

Systèmes pour restaurations dentaires métallo-céramiques

Reference number
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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 3.

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.

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

Attention is drawn to the possibility that some of the elements of this International Standard may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

This second edition cancels and replaces the first edition (ISO 9693:1991), which has been technically revised.

Significant differences between this second edition and the first edition are:

— the scope includes metallic materials (not only alloys) processed by casting or machining;

— a requirement (4.3.3) and a test (6.3.3) on the debonding/crack-initiation strength for the metal-ceramic system have been added, replacing the previous requirement and test related to metal-ceramic bond characterization.

Introduction

Dental metallic materials and ceramics are suitable for use in fabrication of metal-ceramic dental restorations.

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.

Requirements and test methods for tarnish and corrosion resistance for the components and for the metal-ceramic system will be included in the future when they are available.

Metal-ceramic dental restorative systems

1 Scope

This International Standard specifies requirements and test methods for dental metallic materials processed by casting or machining, and for ceramics suitable for use in the fabrication of metal-ceramic dental restorations, together with requirements and test methods for the composite structure.

The requirements of this International Standard apply to the metallic materials and ceramics when used in combination, and compliance may not be claimed for either metallic materials or for ceramics alone.

2 Normative references

The following normative documents contain provisions which, through reference in this text, constitute provisions of this International Standard. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the normative documents indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

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

ISO 6872:1995, *Dental ceramic*.

ISO 6892:1998, *Metallic materials — Tensile testing at ambient temperature.*

ASTM B 265-95, *Standard specification for titanium and titanium alloy strip, sheet and plate*.

ASTM B 348-93, *Standard specification for titanium and titanium alloy bars and billets.*

3 Terms and definitions

For the purposes of this International Standard, the following terms and definitions apply.

3.1
alloy/metal conditioning
process of treating the metal substructure to enhance the bonding of ceramic to metal

3.2
coating agent
bonding agent
substance which, when applied to the metal substructure and fired under appropriate time-temperature conditions, improves aesthetics and may enhance the adherence of ceramic to the coated metal surface

NOTE Examples of such substances are electroplated layers, or agents containing ceramic and/or alloy particles.

3.3
dental dentine ceramic
slightly translucent, pigmented dental ceramic used to impart the overall shape and basic colour of the ceramic part of a metal-ceramic restoration or prosthesis

3.4
dental enamel ceramic
translucent, lightly pigmented dental ceramic, used on a core or a base of dentine ceramic to simulate the natural tooth enamel

3.5
firing schedule
temperature-time cycle stating the initial temperature, the time period (if any) at the initial temperature, the heating rate, the final temperature, the time period (if any) at the final temperature and, in the case of vacuum firing, the temperature of vacuum application and the point of release

3.6
heating rate
rate of temperature increase

NOTE It is expressed in degrees Celsius per minute.

3.7
metallic material
alloy or metal suitable for use as the substructure of a metal-ceramic restoration

3.8
opaque bonding dental ceramic
ceramic product that, when mixed with distilled water or other appropriate liquid, applied to a dental metallic material and treated according to the firing schedule for the opaque ceramic, will bond to the metal surface to form a layer that visibly masks the metallic colour

4 Requirements

4.1 Chemical composition

4.1.1 Metallic material

For noble metal alloys, the percentage of each alloy constituent shall not deviate from the value stated on the package label or insert by more than 0,5 % (mass fraction) [see 8.2.2 c)].

For base metal alloys, the content of constituents present in excess of 20 % (mass fraction) shall not deviate from the value stated on the package label or insert by more than 2 % (mass fraction). The content of constituents present in excess of 2 % (mass fraction) but not in excess of 20 % (mass fraction) shall not deviate from the value stated on the package label or insert by more than 1 % (mass fraction) [see 8.2.2 c)].

The percentage content of nickel, beryllium and cadmium shall not be greater than the amounts indicated on the package label or insert [see 8.2.2 d)].

Testing shall be carried out in accordance with 6.1.1.

For unalloyed titanium, the grade according to ASTM Designation B 265-95 (strip, sheet and plate) or B 348-93 (bars and billets) shall be certified, i.e. standard specification data apply. A certificate from the supplier of the titanium shall be required.

4.1.2 Ceramic

The ceramic shall meet the requirements of ISO 6872:1995/Amd.1:1997, 5.1 and 5.2. Test in accordance with ISO 6872:1995/Amd. 1:1997.

4.2 Biocompatibility

See the Introduction for guidance on biocompatibility.

4.3 Properties

4.3.1 Metallic material

The mechanical properties of the metallic materials as processed shall comply with the requirements of Table 1.

Table 1 — Mechanical properties of metallic materials

Proof stress of non-proportional elongation, $R_{p0,2}$ MPa min.	**Percentage elongation after fracture** % min.
250	3

The solidus and liquidus temperatures of the alloys shall not deviate by more than 25 °C from the values stated on the package label or insert [see 8.2.2 g)].

The coefficient of linear thermal expansion of the alloys shall not deviate by more than $0,5 \times 10^{-6}$ K^{-1} from the value stated on the package label or insert [see 8.2.2 h)].

The density of the alloy as supplied by the manufacturer shall not deviate by more than 0,5 g/cm^3 from the value stated on the package label or insert [see 8.2.2 i)].

Testing shall be carried out in accordance with 6.1.2, 6.1.3, and 6.3.1, respectively. Standard testing procedures shall be used for determining the density.

NOTE For titanium and other pure metals, controlled literature data for melting point, linear thermal expansion and density can be used; see e.g. [8].

4.3.2 Ceramic

The flexural strength and the chemical solubility of the fired ceramics shall meet the requirements of Table 2.

Table 2 — Properties of ceramics

Ceramic type	Flexural strength MPa min.	Chemical solubility, loss in mass $\mu g \cdot cm^{-2}$ max.
Opaque	50	100
Dentine	50	100
Enamel	50	100

The coefficient of thermal expansion of the ceramics shall not deviate by more than $0,5 \times 10^{-6}$ K^{-1} from the value stated by the manufacturer [see 8.2.3 e)].

The glass transition temperature of the ceramics shall not deviate by more than 20 °C from the value stated by the manufacturer [see 8.2.3 f)].

Testing shall be carried out in accordance with 6.2.1, 6.3.1, and 6.3.2, respectively.

4.3.3 Metal-ceramic system

The debonding/crack-initiation strength of the metallic material and at least one specified (named) ceramic present shall be greater than 25 MPa. The debonding/crack-initiation strength of the ceramic and at least one specified (named) metallic material present shall be greater than 25 MPa.

Testing shall be carried out in accordance with 6.3.3.

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 alloy and ceramic are compatible.

5 Sampling

5.1 Metallic material

The sample shall be adequate to prepare the specimens for testing in accordance with this International Standard. All of the metallic materials procured shall be unused and obtained from the same batch.

5.2 Ceramic

Take a sufficient amount of 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, respectively, take equal quantities of each shade.

6 Test methods

6.1 Metallic material

6.1.1 Chemical composition

Use standard analytical procedures for determining the composition.

For unalloyed titanium, specification data in accordance with 4.1.1 shall be provided by the manufacturer or distributor of the metal.

6.1.2 Mechanical properties

6.1.2.1 Preparation of test specimens

Prepare six test specimens, as depicted in Figure 1 or 2, in accordance with the manufacturer´s procedure for processing the substructures for metal-ceramic prostheses. Discard and replace specimens with visible defects.

Preheat the furnace. Place the metallic specimens in the furnace for 15 min with the atmosphere recommended for the metallic material concerned. Use the highest temperature allowed with respect to the ceramic(s) recommended for fusing to that metallic material. Remove the specimens from the furnace, place them on a refractory plate and allow them to cool. Use the specimens for the tests in 6.1.2.2 and 6.1.2.3.

6.1.2.2 Proof stress of non-proportional elongation

Determine the proof stress of non-proportional elongation in accordance with ISO 6892 using the test specimens, processed and conditioned in accordance with 6.1.2.1. Load the test specimens in a universal mechanical testing instrument at a cross-head speed of (1,5 ± 0,5) mm/min until fracture occurs.

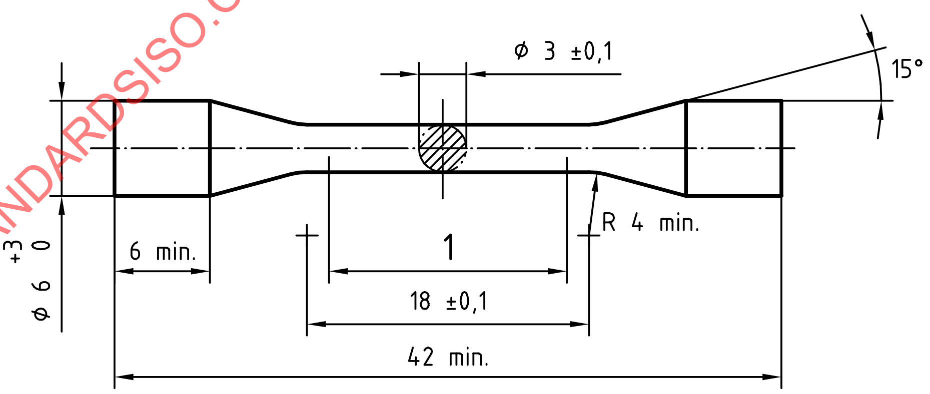
Determine the values from the resultant stress-strain curves at the 0,2 % offset level and calculate the proof stress on the basis of the original cross-sectional area.

If four or more specimens are found to comply with the requirement in Table 1, calculate the proof stress as the mean value for those specimens, and report it to the nearest 5 MPa.

If fewer than four specimens comply with the requirements specified in Table 1, repeat the test.

If again fewer than four specimens comply with the requirement specified in Table 1, the metallic material does not pass the test.

Dimensions in millimetres

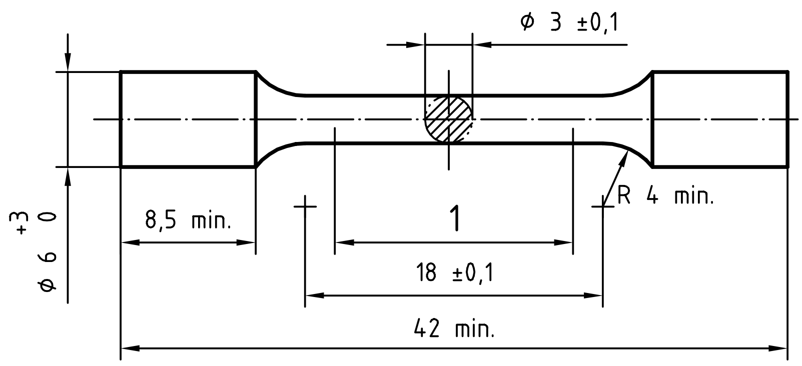


Key

1 Gauge length 15 ± 0,1

Figure 1 — Test specimen with conical shoulders

Dimensions in millimetres



Key

1 Gauge length 15 ± 0,1

Figure 2 — Test specimen with radial shoulders

6.1.2.3 Percentage elongation at fracture

Determine the percentage elongation at fracture in accordance with ISO 6892 on the specimens fractured in the test according to 6.1.2.2.

If four or more specimens are found to comply with the requirement in Table 1, calculate the elongation as the mean value for those specimens, and report it to the nearest 1 %.

If fewer than four specimens comply with the requirement specified in Table 1, repeat the test.

If again fewer than four specimens comply with the requirement specified in Table 1, the metallic material does not pass the test.

6.1.3 Solidus and liquidus temperature

Determine the solidus and liquidus temperatures by differential thermal analysis (DTA) or another established method of equivalent or better accuracy.

6.2 Ceramics

6.2.1 Flexural strength and chemical solubility

6.2.1.1 Preparation of test specimens

Prepare the specimens for testing of flexural strength and chemical solubility in accordance with ISO 6872. When water is required for mixing ceramics, it shall comply with Grade 3 in accordance with ISO 3696.

6.2.1.2 Flexural strength

Test for flexural strength in accordance with ISO 6872.

6.2.1.3 Chemical solubility

Wash the specimens with water of Grade 3 in accordance with ISO 3696, dry them at (150 ± 5) °C for 4 h, and weigh them to the nearest 0,1 mg. Calculate the total surface area to the nearest 0,5 cm^2. Use a 250 ml Pyrex glass bottle with 100 ml acetic acid (analytical grade), 4 % (volume fraction) solution in water of Grade 3 in accordance with ISO 3696. Preheat to (80 ± 3) °C and place the specimens in the bottle. Close the bottle and place it in an oven at (80 ± 3) °C for 16 h. Place the bottle on a bench and remove the specimens when the bottle has cooled to room temperature.

Wash the specimens with water of Grade 3 in accordance with ISO 3696, dry them at (150 ± 5) °C to constant mass, and reweigh the specimens.

6.2.2 Assessment of results

6.2.2.1 Flexural strength

Calculate the value of flexural strength in accordance with ISO 6872. To pass the test, at least eight of the 10 specimens shall meet the requirements for flexural strength stated in Table 2.

If five or less pass, the material fails. If six or seven pass, test an additional 10 specimens. Of these, 10 or nine respectively shall pass, i.e. at least a total of 16 specimens shall pass.

6.2.2.2 Chemical solubility

Calculate the mass loss, in micrograms per square centimetre, of the specimens. Check for compliance with the requirement stated in Table 2.

6.3 Metal-ceramic system

6.3.1 Linear thermal expansion

6.3.1.1 Apparatus

6.3.1.1.1 Equipment for manufacturing dental metal framework, in accordance with the manufacturer's processing instructions.

6.3.1.1.2 Dental ceramic furnace.

6.3.1.1.3 Calibrated dilatometer.

6.3.1.2 Preparation of test specimens

Follow the processing instructions concerned, and prepare two metal specimens and four opaque, four dentine and four enamel ceramic specimens. Prepare the specimens as rods or bars having cross-sectional areas not greater than 30 mm^2. Grind the ends of the specimens flat, and parallel and square to the specimen axis.

6.3.1.3 Procedure for metallic material

Heat-treat specimens according to 6.1.2.1. Make dilatometric measurements on each of the two specimens respectively at a heating rate of (5 ± 1) °C/min, up to 550 °C. Determine the coefficient $\alpha_{(25\ °C\ to\ 500\ °C)}$ of linear thermal expansion between 25 °C and 500 °C for each specimen from plotted curves or recorded values of expansion versus temperature.

For titanium and other pure metals, see note in 4.3.1.

6.3.1.4 Procedure for ceramic

Prepare the four opaque specimens and the four dentine and four enamel specimens by firing two specimens of each type of ceramic once in vacuum and once at atmospheric pressure in air, and firing the other two specimens of each type three times in vacuum and once at atmospheric pressure in air in accordance with the manufacturer´s instructions.

Make a dilatometer measurement on each annealed specimen at a heating rate of 5 °C/min up to the dilatometric softening point. Determine for each specimen the coefficient of thermal expansion between 25 °C and 500 °C (or between 25 °C and the glass transition temperature if that temperature is lower than 500 °C) from plotted curves or recorded values of expansion versus temperature.

6.3.1.5 Assessment of results

Calculate and report a mean value for $\alpha_{(25\ °C\ \text{to}\ 500\ °C)}$ for the coefficient of thermal expansion from 25 °C to 500 °C for the metallic material [8.2.2 h)] and from 25 °C to 500 °C (or from 25 °C up to the glass transition temperature, if that temperature is lower than 500 °C) for the ceramic specimens fired twice and four times. Report the mean coefficient of thermal expansion in 10^{-6} K^{-1}, rounded to the nearest 0,1 × 10^{-6} K^{-1} [8.2.3 e)].

6.3.2 Glass transition temperature

6.3.2.1 Procedure

Determine graphically the glass transition temperature for each ceramic specimen from plotted curves of expansion versus temperature, prepared in accordance with 6.3.1.4.

6.3.2.2 Assessment of results

Calculate and report the mean glass transition temperatures, in degrees Celsius, for opaque, dentine and enamel ceramics fired twice and four times [8.2.3 f)].

6.3.3 Metal-ceramic bond characterization (Schwickerath crack initiation test)

6.3.3.1 Apparatus

6.3.3.1.1 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.

6.3.3.2 Preparation of test specimens

Prepare six alloy/metal 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 metal-ceramic prostheses. Condition the specimens, observing the manufacturer´s instructions (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 both opaque and body ceramic. If necessary, adjust the firing temperatures or holding times.

According to the manufacturer´s instructions apply opaque ceramic over a length of (8 ± 0,1) mm symmetrically on one 3 mm wide side of each specimen.

Add body ceramic to each specimen to form a total ceramic thickness of (1,1 ± 0,1) mm after firing (see Figure 3). The ceramic layer shall have a rectangular shape.

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 metal strip.

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

Dimensions in millimetres

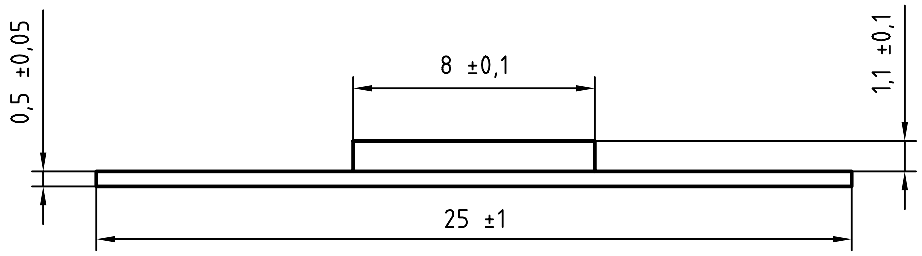


Figure 3 — Test specimen configuration

6.3.3.3 Procedure

The fired specimens are placed in the bending apparatus (distance between supports: 20 mm, radius of the bending piston: 1 mm) with the ceramic positioned symmetrically on the side opposite to the applied load. The force is applied at a constant rate 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.

6.3.3.4 Assessment of results

The fracture force F_{fail} has to be multiplied with a coefficient k. Coefficient k can be read from Figure 4. The coefficient k is a function of the thickness of the metal substrate d_M (0,5 ± 0,05) mm, and the value of Young's modulus E_M of the used metallic material.

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

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

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

The metal-ceramic 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.

If again fewer than four specimens comply with the requirement specified in 4.3.3, the metal-ceramic system does not pass the test.

6.3.3.5 Alternative procedure

The debonding/crack-initiation strength τ_b can also be calculated numerically on the basis of the flow chart shown in Figure 5.

6.3.3.6 Assessment of results

The metal-ceramic 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.

If again fewer than four specimens comply with the requirement specified in 4.3.3, the metal-ceramic system does not pass the test.

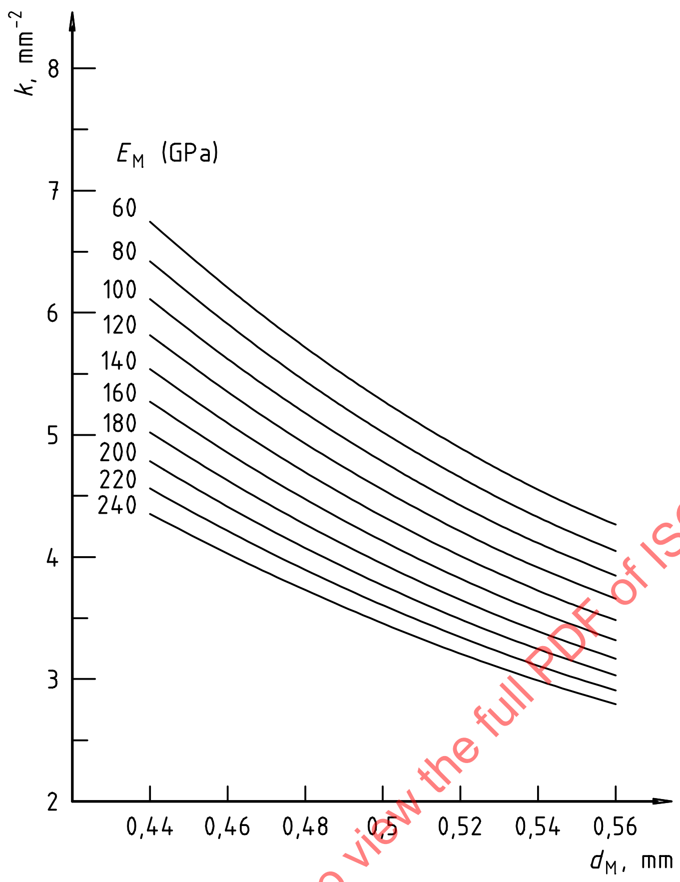


Figure 4 — Diagram to determine the coefficient k as a function of metal substrate thickness d_M and Young's modulus E_M of the metallic material

7 Information and instructions

Detailed dated instructions for processing the metallic material, including instructions for brazing or welding, and for preparing the surface to obtain satisfactory bonding to at least one specified (named) ceramic material shall be supplied by the distributor of the metallic material, or as regards titanium by the distributor(s) of the processing system(s) concerned. Detailed dated instructions for the application and the firing schedule of the ceramic material, together with the manufacturer´s recommendation of at least one (named) metallic material which can provide satisfactory bonding to the ceramic material considered, shall be supplied by the distributor of the ceramic.

Detailed instructions regarding precautions shall be given in the package or accompanying literature.

CAUTION — Appropriate protection should be used for all grinding and polishing procedures.

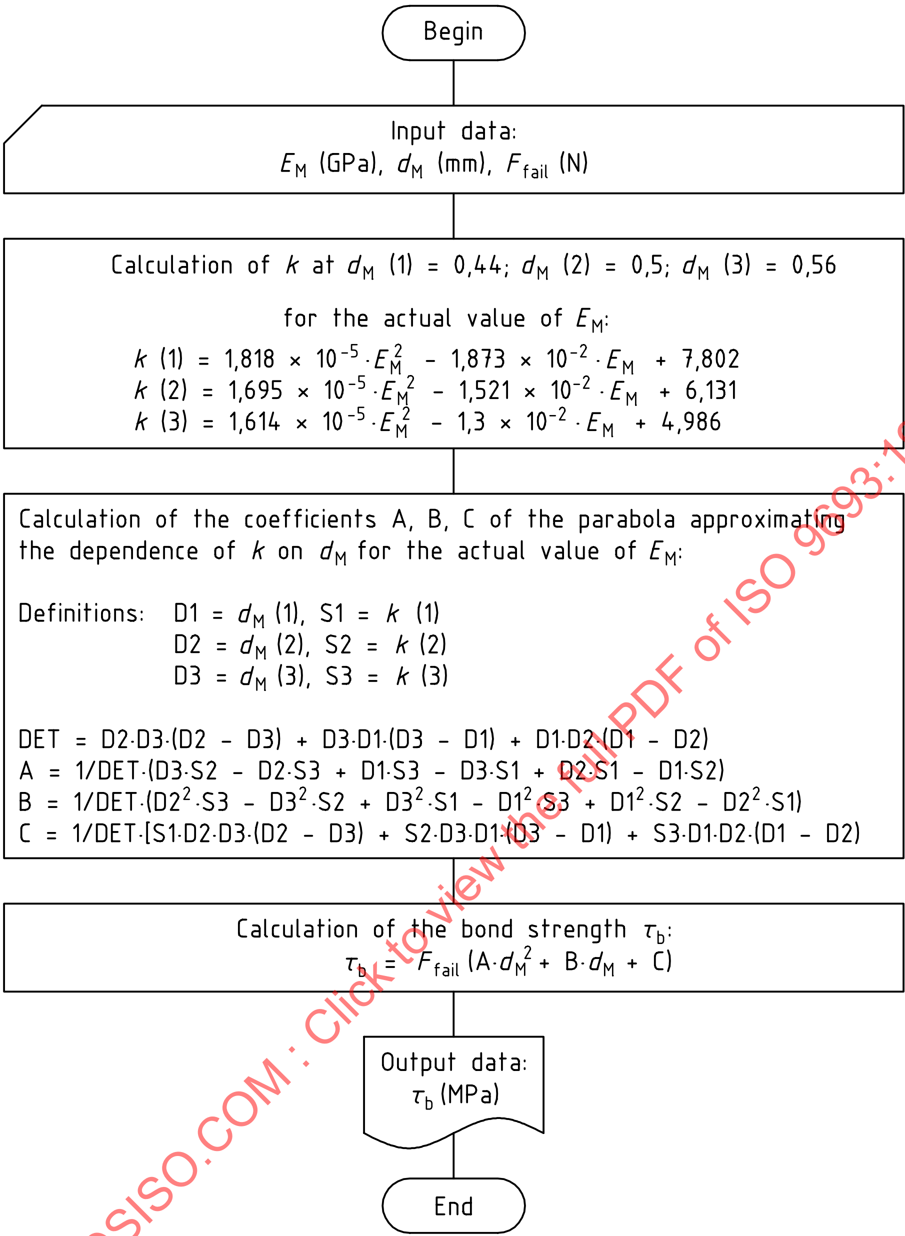


Figure 5 — Flow chart for numerical calculation of debonding/crack-initiation strength

8 Packaging, marking and labelling

8.1 Packaging

The dental ceramic powder shall be supplied in sealed containers that do not contaminate or permit contamination of the contents.