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ISO 6872

Third edition
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Dentistry — Ceramic materials

Art dentaire — Produits céramiques

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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 6872 was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 2, *Prosthodontic materials*.

This third edition cancels and replaces the second edition (ISO 6872:1995) and Amendment 1:1997 which have been technically revised.

Introduction

Specific qualitative and quantitative requirements for freedom from biological hazard are not included in this International Standard, but it is recommended that, in assessing possible biological or toxicological hazards, reference be made to ISO 10993-1 and ISO 7405.

Dentistry — Ceramic materials

1 Scope

This International Standard specifies the requirements and the corresponding test methods for dental ceramic materials for fixed all-ceramic and metal-ceramic restorations and prostheses.

2 Normative references

The following referenced documents are indispensable for the application 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 3696, *Water for analytical laboratory use — Specification and test methods*

3 Terms and definitions

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

3.1 Material

3.1.1
addition ceramic
dental ceramic material that is fired at a reduced temperature and is normally applied to restore contact points on a dental restoration or prosthesis

3.1.2
dental ceramic
inorganic, non-metallic material that is specifically formulated for use, when processed according to manufacturer's instructions, to form the whole or part of a dental restoration or prosthesis

3.1.3
dental porcelain
predominantly glassy dental ceramic material used mainly for aesthetics in a dental restoration or prosthesis

3.1.4
dentine ceramic
dental ceramic material used to form the overall shape and basic colour of a dental restoration or prosthesis, simulating the natural tooth dentine

3.1.5
enamel ceramic
dental ceramic material used to overlay either partially or wholly the dentine ceramic and also to form the more translucent incisal third of a dental restoration or prosthesis, simulating the natural tooth enamel

3.1.6
flame-sprayed dental ceramic
dental ceramic core or substructure layer formed via the technique of flame spraying

3.1.7
glass-ceramic (dental)
dental ceramic material formed by the action of heat treatment on a glass, in order to cause initiation and growth of a wholly or predominantly crystalline microstructure

3.1.8
glass-infiltrated dental ceramic
dental ceramic core or substructure layer, which is porous and is subsequently densified by the infiltration of specialized glass at elevated temperature

3.1.9
glaze ceramic
dental ceramic material that is overlayed and fired at a reduced temperature compared to dentine or enamel ceramic, to produce a thin coherent sealed surface, the level of gloss being determined by the firing conditions

3.1.10
liner
dental ceramic material used on all-ceramic substructure forming a layer that provides a background colour upon which dentine or opaceous dentine ceramic can be applied to achieve overall aesthetics

3.1.11
modelling fluid
liquid with which a dental ceramic powder is mixed, in order to shape or model it into its required form prior to firing

3.1.12
opaceous dentine ceramic
dental ceramic material having a higher opacity than a dentine ceramic material, but which can still be used to contribute to the overall shape and basic colour of a dental restoration or prosthesis, simulating the natural tooth dentine

3.1.13
opaque dental ceramic
dental ceramic material that, when applied to a metallic substructure in accordance with manufacturer's instructions, acts to bond to the metal surface forming a layer that provides a background colour and interface upon which other dental ceramic materials can be applied to achieve overall aesthetics

3.1.14
shoulder ceramic
dental ceramic material used to form shape and colour at the marginal area of the dental restoration or prosthesis, simulating natural tooth dentine in this area

3.1.15
stain ceramic
dental ceramic powder or paste that is normally intensely coloured and that is formulated to be used either internally or externally, during the build up of a dental restoration or prosthesis, to simulate details within or on the surface as are respectively found in natural teeth

3.1.16
substructure (core) dental ceramic
predominantly polycrystalline dental ceramic material that forms a supporting substructure upon which one or more layers of dental ceramic or dental polymer material are applied, either partially or totally, to form a dental restoration or prosthesis

3.2 Processing

3.2.1
air firing dental ceramic
firing of dental ceramics under ambient atmospheric pressure

3.2.2
CAD/CAM dental ceramic
computer aided design/computer aided manufacture (CAD/CAM) procedures to manufacture a dental restoration or prosthesis normally including the following stages:

1) a digital scanning procedure of the model or wax-up to produce a 3D data set

2) software manipulation of the 3D data set to design the prosthesis

3) a computer directed machine tool that performs the manufacturing process

3.2.3
condensation of dental ceramic
powder process whereby a slurry of dental ceramic powder is vibrated to compact the powder prior to sintering

3.2.4
injectable, castable or pressable dental ceramic
dental ceramic material, normally in the form of a pellet or ingot (often pre-sintered), designed for use in a specialized furnace, which enables the pellet or ingot to be injected/cast/pressed into a mould, prepared via the lost wax technique

3.2.5
sintering of a dental ceramic
process whereby heat and potentially other process parameters (e.g. pressure and atmosphere) are applied to a ceramic powder or powder compact, in order to densify the ceramic into its required form

NOTE "Firing" and "sintering" are used interchangeably in this document ("firing" connotating the application of heat to drive sintering).

3.2.6
vacuum firing dental ceramic
firing of dental ceramics at reduced pressure (i.e. under vacuum) to yield the required density and associated aesthetics, especially the degree of translucency

NOTE Dental ceramics for vacuum firing have a specific particle size distribution to reduce the entrapment of porosity.

3.3 Properties

3.3.1
class of dental ceramic
classification of a dental ceramic material according to its intended function

3.3.2
fracture toughness
conventional fracture mechanics parameter indicating the resistance of a material to crack extension (propagation)

3.3.3
glass transition temperature
approximate mid-point of the temperature range over which a glass transforms between elastic and viscoelastic behaviour, characterized by the onset of a rapid change in its coefficient of thermal expansion

3.3.4
glaze (medium)
surface appearance obtained when the gloss is clinically and aesthetically acceptable

4 Types, classes and their identification

For the purposes of this International Standard, dental ceramics are designated into two types:

— Type I: ceramic products that are provided as powders, pastes or aerosols;

— Type II: all other forms of ceramic products.

Ceramics are divided into six classes according to their intended clinical use according to descriptions in Table 1. If colour is added to a ceramic powder for identification purposes, the colour coding given in Table 2 is recommended.

Table 1 — Classification of ceramics for fixed prostheses by intended clinical use

Class	Recommended clinical indications	Mechanical and chemical properties	
		Flexural strength minimum (mean) MPa	Chemical solubility maximum $\mu g \cdot cm^{-2}$
1	a) Aesthetic ceramic for coverage of a metal or a ceramic substructure. b) Aesthetic-ceramic: single-unit anterior prostheses, veneers, inlays, or onlays.	50	100
2	a) Aesthetic-ceramic: adhesively cemented, single-unit, anterior or posterior prostheses.	100	a) 100
	b) Adhesively cemented, substructure ceramic for single-unit anterior or posterior prostheses.	100	b) 2 000
3	Aesthetic-ceramic: non-adhesively cemented, single-unit, anterior or posterior prostheses.	300	100
4	a) Substructure ceramic for non-adhesively cemented, single-unit, anterior or posterior prostheses. b) Substructure ceramic for three-unit prostheses not involving molar restoration.	300	2 000
5	Substructure ceramic for three-unit prostheses involving molar restoration.	500	2 000
6	Substructure ceramic for prostheses involving four or more units.	800	100

Table 2 — Recommended colour coding for the identification of Type I dental ceramic powders

Material	Colour coding
Dentine	Pink
Enamel	Blue
Fluorescent	Yellow
Highly chromatic dentine	Orange
Opalescent enamel	Blue-green
Modifying enamel (e.g., translucent, clear)	Purple

5 Requirements

5.1 Uniformity

The inorganic pigment(s) used to produce the colour of a fired dental ceramic and any organic colorants (for colour coding) shall be uniformly dispersed throughout the dental ceramic material and, in powdered ceramic products, no segregation of the pigment(s) shall take place when the powder is mixed as described in 7.1.4. Check by visual inspection.

5.2 Freedom from extraneous materials

5.2.1 Dental ceramic materials shall be free from extraneous materials, when assessed by visual inspection.

5.2.2 Dental ceramic materials shall not have an activity concentration of more than 1,0 $Bq \cdot g^{-1}$ of uranium^{238}. Test in accordance with 7.2.2.

5.2.3 Any colorants used to colour code the ceramic powder as listed in Table 2 are recommended to be food quality organic materials.

5.3 Mixing and condensation properties, type I ceramics

When mixed as described in 7.1.4, with water or the modelling fluid recommended by the manufacturer, a dental ceramic powder shall not form lumps or granules when assessed by visual inspection.

The paste so formed shall be suitable for making the indicated restorations and prostheses by condensation of successive layers. When the paste is condensed as in 7.1.5, it shall not crack or crumble during drying, as later assessed by visual inspection.

5.4 Physical and chemical properties

The physical and chemical properties of ceramic test specimens tested in accordance with the relevant methods, detailed for Type I and Type II ceramics in Clause 7, shall comply with the requirements specified in Table 1. 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 8.2.2 d)]. The glass transition temperature of the ceramics shall not deviate by more than 20 °C from the value stated by the manufacturer [see 8.2.2 d)].

5.5 Biocompatibility

See the Introduction for guidance on biocompatibility.

6 Sampling

6.1 Type I ceramics

Take a sufficient amount of ceramic to carry out the necessary tests. Where there is more than one shade in a class of dental ceramic, combine equal quantities of each shade.

Sufficient quantities of essential modelling fluids shall be obtained, if their use is recommended by the manufacturers. The quantities shall be those recommended by the manufacturer concerned.

6.2 Type II ceramics

All of a material procured for testing in accordance with this International Standard shall be of the same lot.

7 Test methods

7.1 Preparation of test specimens

7.1.1 General

For detailed instructions, see the individual test methods.

For Type I specimens (unless otherwise stated or inconsistent with the text) the apparatus detailed in 7.1.3 along with the conditions for mixing, condensation and firing (7.1.4 to 7.1.6) apply to all test methods.

7.1.2 Components of test specimens, Type I ceramics

The liquid used in the preparation of test specimens shall be water that complies with the requirements for grade 3 water (see ISO 3696) or, when applicable, the modelling fluid recommended by the manufacturer of the dental ceramic powder. The required amount of powder shall be taken from the appropriate pool of powder obtained in accordance with 6.1.

7.1.3 Apparatus for mixing

All apparatus for mixing shall be clean and dry.

7.1.3.1 **Glass slab or mixing palette**.

7.1.3.2 **Spatula**, made from a material that is not readily abraded by the dental ceramic powder (glass is recommended). Instruments used for the mixing procedure shall be made of materials that do not contaminate the ceramic material.

7.1.3.3 **Open multipart mould**, from which the condensed specimen can be removed without distortion.

7.1.3.4 **Vibration system (vibration table or mechanical brush)**, capable of vibrating at a frequency of 50 Hz to 60 Hz or in accordance with the manufacturer's instructions.

7.1.4 Method of mixing

Combine the water or modelling liquid and the ceramic powder in the proportions recommended by the manufacturer. Avoid vigorous mixing which will tend to incorporate air bubbles with the paste and, both during and after mixing, examine for compliance with 5.1 and 5.2.1.

7.1.5 Procedure for specimen fabrication

Overfill the mould (7.1.3.3) with dental ceramic paste, and vibrate. When excess liquid appears at the free surface of the specimen, place a paper tissue (or similar absorbent material) on the surface of the specimen, and remove the excess liquid continually by replacing the tissue as soon as it becomes saturated with liquid. Continue vibration and absorption until no further liquid can be removed, and then level the free surface of the condensed specimen by means of a suitable instrument (a bevelled glass microscope slide is ideal for this purpose). After removing the specimen from the mould, place it on a firing tray, dry it and check for compliance with 5.3.

NOTE Other forming methods, such as dry pressing, are acceptable for specimen fabrication.

7.1.6 Firing

Position the specimens in the furnace so that they will be uniformly fired, and on a substrate to which they will not adhere and from which there will be no pick-up of material. Obtain guidance from the manufacturer for the firing of test specimens. These specimens shall be fired according to manufacturer's instructions so that their final density and thermal history is representative of that found for indicated restorations or prostheses.

7.2 Radioactivity of dental ceramic

7.2.1 Preparation of samples

7.2.1.1 Type I ceramics

A 50 g sample as-manufactured is suitable, collected as described in 6.1.

7.2.1.2 Type II ceramics

Mill powder using tungsten carbide milling media or other appropriate media (to avoid contamination by radioactive species). Sieve and obtain 50 g of powder with a particle size less than 75 µm.

7.2.2 Counting procedure

Use a sample volume of 50 g bulk powder and determine the activity concentration of uranium^{238} by neutron activation or gamma spectroscopy (with gamma spectroscopy, techniques shall be used to screen for adulteration).

7.2.3 Assessment of results

Each sample tested shall comply with the requirement in 5.2.2.

7.3 Flexural strength

7.3.1 General

Three flexural test methods are acceptable:

a) three-point bending;

b) four-point bending;

c) biaxial flexure (piston-on-three-ball).

7.3.2 Three-point and four-point bending tests

7.3.2.1 Apparatus

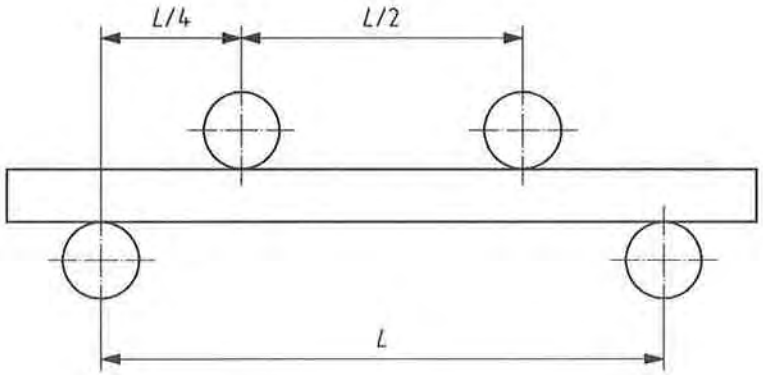
7.3.2.1.1 Universal mechanical testing machine, capable of a cross-head speed of (1 ± 0,5) mm/min and able to measure applied loads of between 10 N and 1 000 N (± 0,1 N).

7.3.2.1.2 Flexural test fixtures

a) **for three-point bending**, consisting of support rollers 1,5 mm to 5 mm (± 0,2 mm) in diameter positioned with their centres 12,0 mm to 40,0 mm (± 0,5 mm) apart, the load shall be applied at the midpoint between the supports by means of a third roller 1,5 mm to 5 mm (± 0,2 mm) in diameter. Rollers shall be made from hardened steel or other hard material having a hardness greater than 40 HRC (Rockwell C scale) and have a smooth surface with a roughness less than 0,5 µm Ra.

b) **for four-point bending**, consisting of a ¼-point test configuration, the test piece is loaded by two inner bearing rollers located ¼ of the total span, L, from the outer support bearing rollers (see Figure 1).

Support rollers 1,5 mm to 5 mm (± 0,2 mm) diameter shall be positioned with their centres 16,0 mm to 40,0 mm (± 0,5 mm) apart. Rollers shall be made from hardened steel or other hard material having a hardness greater than 40 HRC and have a smooth surface with a roughness less than 0,5 µm Ra. The two loading rollers, of identical material and size to the support rollers, shall be located at the quarter points yielding an inner span (L/2 in Figure 1) of 8,0 mm to 20,0 mm (± 0,2 mm). The loading arrangement shall ensure that equal forces are applied to the loading rollers and that torsional loading is minimized.



NOTE Moment arm = L/4.

Figure 1 — Schematic of the four-point-1/4-point fixture configuration

7.3.2.2 Preparation of test specimens

7.3.2.2.1 Test specimen dimensions and test parameters

7.3.2.2.1.1 Dimensions

Specimens for three-point and four-point bending testing have a rectangular cross section and most preferably an edge chamfer as shown in Figure 2 and given in the dimensions listed below.

Width w = 4,0 mm ± 0,2 mm (dimension of the side at right angles to the direction of the applied load)

Thickness b = 1,2 mm to 3,0 mm ± 0,2 mm (with 3,0 mm recommended; dimension of the side parallel to the direction of the applied load)

Chamfer c = 0,09 mm to 0,15 mm

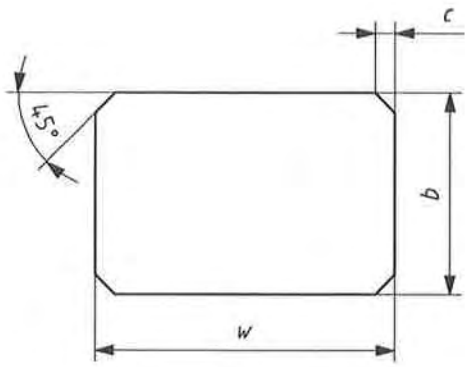


Figure 2 — Specification of indicated dimensions

7.3.2.2.1.2 Test parameters

Test span:

For four-point bending, L in millimetres (centre-to-centre distance between outer support roller, see Figure 1. In the four-point ¼ configuration specified, the moment arm = L/4).

For three-point bending, l in millimetres (centre-to-centre distance between support rollers).

Breaking load, P in Newtons

Specimen lengths shall be at least 2 mm longer than the test span (L or l) and the ratio of thickness to length (b/L or b/l) shall be $\leqslant$ 0,1.

7.3.2.2.2 Type I ceramics

Prepare at least ten and preferably thirty specimens of dimensions as specified in 7.3.2.2.1. Use a mould appropriately sized to allow for dimensional changes resulting from sintering and finishing. Fire the specimens at least once under vacuum and once at atmospheric pressure in air in accordance with the manufacturer's instructions modified as needed due to specimen dimensions. Grind each specimen so as to produce a rectangular test piece as specified in 7.3.2.2.1. Final grind on diamond-embedded media, having a nominal grit size of 30 µm to 40 µm, and final polish on media having 15 µm to 20 µm diamond grit. Ensure that opposing faces of the test pieces are flat and parallel to within 0,05 mm. Thoroughly clean the test pieces, ensuring that all traces of grinding debris are removed.

7.3.2.2.3 Type II ceramics

Prepare according to the manufacturer's instructions at least ten and preferably thirty specimens of dimensions as specified in 7.3.2.2.1. In the case of ceramic material produced for machining, prepare the specimens from ceramic blocks made by the manufacturer. Grind each specimen to produce test pieces using the protocol specified in 7.3.2.2.2.

7.3.2.3 Procedure

Measure the cross-sectional dimensions of each test piece to ± 0,01 mm, then place a test piece centrally on the bearers of the test machine so that the load is applied to a 4 mm wide face along a line perpendicular to the long axis of the test piece, and determine to ± 0,1 N, the load required to break the test piece. Use a crosshead speed of (1 ± 0,5) mm/min. Repeat the procedure with the remaining test pieces.

7.3.2.4 Calculation of strength

7.3.2.4.1 Three-point flexure

From Equation (1) calculate the flexural strength, σ, in megapascals, and report the mean and standard deviation of the strength data. Means shall be equal to or exceed the requirements listed in Table 1. In addition, if at least fifteen specimens have been tested, the Weibull characteristic strength and Weibull modulus may also be reported as indicated in Annex B.

$$\sigma = \frac{3Pl}{2wb^2} \tag{1}$$

where

P is the breaking load, in newtons;

l is the test span (centre-to-centre distance between support rollers), in millimeters;

w is the width of the specimen, i.e. the dimension of the side at right angles to the direction of the applied load, in millimeters;

b is the thickness of the specimen, i.e. the dimension of the side parallel to the direction of the applied load, in millimeters.

7.3.2.4.2 Four-point flexure

From Equation (2) calculate the flexural strength, σ, in megapascals, and report the mean and standard deviation of the strength data. Means shall be equal to or exceed the requirements listed in Table 1. In addition, if at least fifteen specimens have been tested, the Weibull characteristic strength and Weibull modulus may also be reported as indicated in Annex B.

$$\sigma = \frac{3PL}{4wb^2} \tag{2}$$

where

P is the load at failure, in newtons;

L is the centre-to-centre distance between outer support rollers, in millimeters;

w is the width of the specimen, i.e., the dimension of the side at right angles to the direction of the applied load, in millimeters;

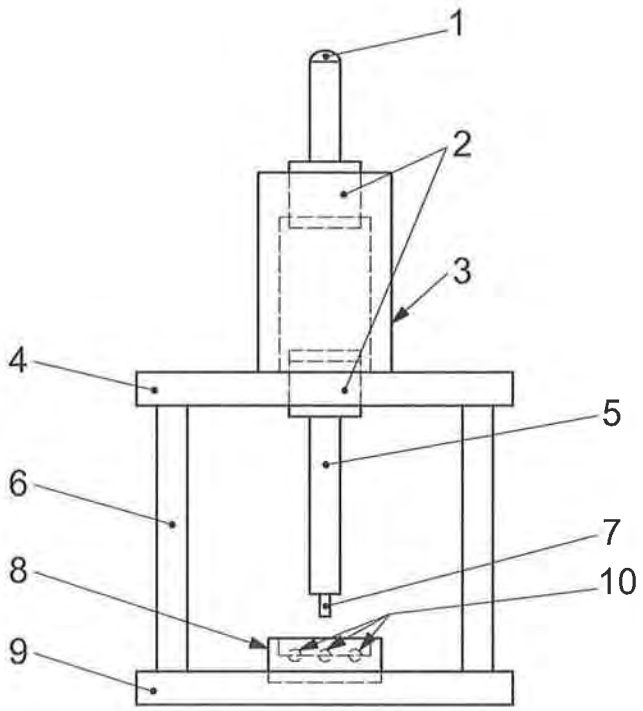
b is the thickness of the specimen, i.e., the dimension of the side parallel to the direction of the applied load, in millimeters.

7.3.3 Biaxial flexure test (piston-on-three-ball test)

7.3.3.1 Apparatus

7.3.3.1.1 Universal mechanical testing machine, capable of a cross-head speed of (1 ± 0,5) mm/min and able to measure applied loads of between 10 N and 1 000 N (± 0,1 N).

7.3.3.1.2 Biaxial flexural strength test fixture, for the support of the test specimen; three hardened steel balls with a diameter between 2,5 mm and 6,5 mm, positioned 120° apart on a support circle with a diameter of 10 mm to 12 mm, shall be provided. The sample shall be placed concentrically on these supports and the load shall be applied with a flat punch with a diameter of (1,4 ± 0,2) mm at the centre of the specimen (see Figure 3).



Key

1 12,5 mm steel ball
2 precision ball bushings
3 upper bushing holder
4 top plate
5 ram, hardened, precision ground rod
6 three separator posts
7 hardened, precision ground dowell pin, (1,4 ± 0,2) mm diameter
8 sample holder
9 bottom plate
10 three 2,5 mm to 6,5 mm steel balls 120° apart on a 10 mm to 12 mm diameter circle

Figure 3 — Schematic of a piston-on-three-ball test fixture

7.3.3.2 Preparation of test specimens

7.3.3.2.1 Type I ceramics

Prepare at least ten and preferably thirty discs having a thickness 1,2 mm ± 0,2 mm and a diameter of 12 mm to 16 mm. Use a mould appropriately sized to allow for dimensional changes resulting from sintering and finishing. Fire the specimens once under vacuum and once at atmospheric pressure in air in accordance with the manufacturer's instructions modified as needed due to specimen dimensions. Final grind each specimen using a diamond-embedded media having a nominal grit size of 30 µm to 40 µm, and final polish with media having 15 µm to 20 µm diamond grit. Ensure that opposing faces of the test pieces are flat and parallel to within 0,05 mm. Thoroughly clean the test pieces, ensuring that all traces of grinding debris are removed.

7.3.3.2.2 Type II ceramics

Prepare according to the manufacturer's instructions at least ten and preferably thirty discs having a diameter between 12 mm and 16 mm and an approximate thickness of 1 mm to 2 mm. In the case of ceramic material produced for machining, prepare the specimens from ceramic blocks made by the manufacturer. Grind each specimen so as to produce a test piece of thickness 1,2 mm ± 0,2 mm and a diameter of 12 mm to 16 mm. Grind and finish specimens using the protocol specified under 7.3.3.2.1.

7.3.3.3 Procedure

For the biaxial flexural strength test, a fixture as shown in Figure 3 may be used. Measure the dimensions of each test piece, and of all other relevant variables, to ± 0,01 mm then place a test piece concentrically on the supporting balls of the testing machine so that the load is applied at the centre of the test piece. Place a film of non-rigid material between the supporting balls and the specimen and another film between the loading piston and specimen to evenly distribute contact pressures (for example polyethylene sheet, thickness 0,05 mm). Determine to within ± 0,1 N, the load required to break the test piece. Use a crosshead speed of (1 ± 0,5) mm/min. Repeat the procedure with the remaining test pieces.

7.3.3.4 Calculation of strength

From Equation (3) calculate the flexural strength, σ, in megapascals, and report the mean and standard deviation of the strength data; means shall equal or exceed the requirements listed in Table 1. In addition, if at least fifteen specimens have been tested, the Weibull characteristic strength and Weibull modulus may also be reported as indicated in Annex B.

$$\sigma = -0,238\ 7P(X - Y)/b^2 \tag{3}$$

where

σ is the maximum center tensile stress, in megapascals;

P is the total load causing fracture, in Newtons;

$$X = (1+\nu)\ln(r_2/r_3)^2 + [(1-\nu)/2](r_2/r_3)^2$$

$$Y = (1+\nu)[1+\ln(r_1/r_3)^2] + (1-\nu)(r_1/r_3)^2$$

b is the specimen thickness at fracture origin in millimeters.

in which

ν is Poisson's ratio (If the value for the ceramic concerned is not known, use $\nu = 0,25$);

r_1 is the radius of support circle in millimeters;

r_2 is the radius of loaded area, in millimeters;

r_3 is the radius of specimen, in millimeters;

7.4 Linear thermal expansion coefficient

7.4.1 Apparatus

7.4.1.1 Equipment for making bar specimens, from Type I and Type II ceramics.

7.4.1.2 Dental ceramic oven, for firing Type I ceramics and controlling thermal history for Type I and Type II ceramics.

7.4.1.3 Calibrated dilatometer.

7.4.2 Preparing test specimens (Type I and Type II ceramics)

Prepare four test specimens in the form of rods or bars, having a length between 5 mm and 50 mm with a cross-sectional area not exceeding 30 mm^2. Fire two specimens once in a vacuum and once at atmospheric pressure in air and the other two specimens three times in a vacuum and once at atmospheric pressure in air. Grind the ends of the test specimens so that they are flat, parallel and perpendicular to the axis of the test specimens.

7.4.3 Dilatometric measurement

Place each specimen in the dilatometer's oven at room temperature and wait 15 min so that the sample has attained the same temperature as the oven.

Set the sample's "preloading" to the value specified by the instrument manufacturer for measuring ceramics.

Perform an expansion measurement of the test specimen at 5 °C/min to 10 °C/min between 25 °C and approximately 500 °C (or approximately 30 °C above the T_g, lower or higher than 500 °C as required), sufficient to determine T_g graphically (see Figure 4). For each test specimen determine the linear expansion coefficient between 25 °C and 500 °C (or T_g) by referring to plotted curves or recorded values indicating the expansion in relation to temperature.

7.4.4 Assessment of results

Report the average value and standard deviation of the linear thermal expansion coefficient for the four specimens (fired twice and four times) between 25 °C and 500 °C (or T_g). Report the mean coefficient of thermal expansion rounded-off to the nearest $0{,}1 \times 10^{-6}\ K^{-1}$.

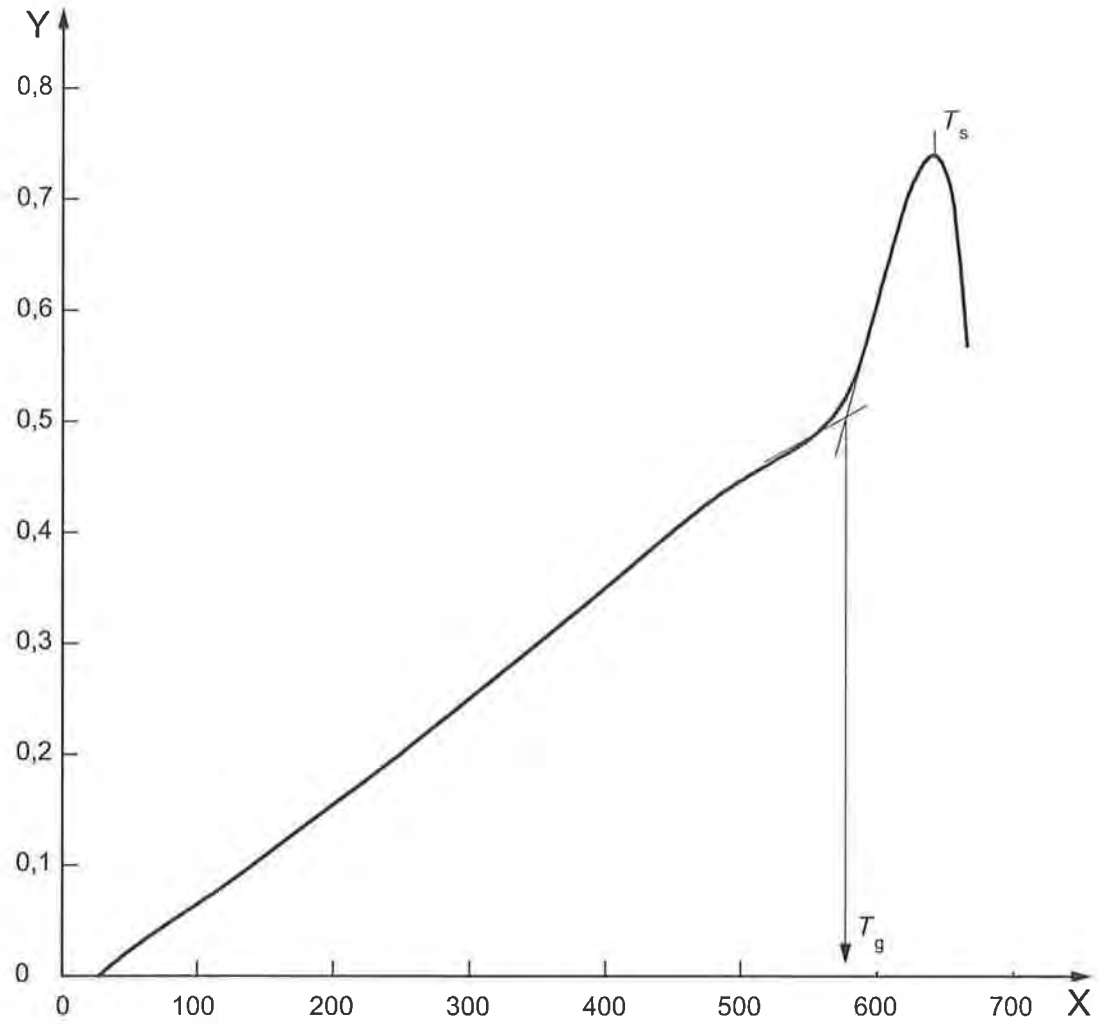
7.5 Glass transition temperature

7.5.1 Operating procedure

Graphically determine the glass transition temperature, T_g, for each specimen (where applicable) by referring to the expansion curves obtained in accordance with 7.4.3 (as illustrated in Figure 4).

7.5.2 Assessment of results

Report the average and standard deviation of the glass transition temperature, measured in 7.5.1, in degrees centigrade.



Key

X temperature (°C)

Y variation in length (µm)

NOTE T_s is the dynamic softening temperature of the ceramic under the dilatometer load and heating rates used.

Figure 4 — Typical dilatometry curve demonstrating graphical method of determining glass transition temperature

7.6 Chemical solubility

7.6.1 Reagents

7.6.1.1 Acetic acid (analytical grade), 4 % by volume solution in water of Grade 3 as specified in ISO 3696.

7.6.2 Apparatus

7.6.2.1 Balance, accurate to 0,1 mg.

7.6.2.2 Drying oven, capable of being controlled at (150 ± 5) °C.

7.6.3 Preparation of test specimens

7.6.3.1 Type I ceramics

Prepare specimens using powder collected as described in 6.1. Fire these specimens in accordance with the manufacturer's instructions, modified as needed due to specimen dimensions. Prepared specimens shall provide at least 30 cm^2 ($\pm$ 0,5 cm^2) of exposed surface area freely accessible to the test solution.

7.6.3.2 Type II ceramics

Prepare specimens according to the manufacturer's instructions providing at least 30 cm^2 of exposed surface area freely accessible to the test solution. If applicable, fire the specimens to achieve a medium glaze or give an appropriate surface finish using polishing procedures as specified by the manufacturer to simulate the surface finish used for clinical indications.

7.6.4 Procedure

Wash the specimens with water of grade 3 in accordance with ISO 3696, dry them at (150 $\pm$ 5) °C for 4 h, and weigh them to the nearest 0,1 mg. Determine the total surface area to the nearest 0,1 cm^2. Use a 250 ml Pyrex glass bottle with 100 ml acetic acid (analytical grade), 4 % (by volume) solution (7.6.1.1) in water of grade 3 as specified in ISO 3696. Preheat to (80 $\pm$ 3) °C and place specimens in the bottle. Close the bottle and place it in an oven at (80 $\pm$ 3) °C for 16 h. Wash the specimens with water of grade 3 in accordance with ISO 3696, dry them at (150 $\pm$ 5) °C to constant mass, and reweigh the specimens.

7.6.5 Calculation and assessment of results

Calculate the mass loss, in micrograms per square centimeter, of the specimens. Check for compliance with the requirements stated in Table 1.

NOTE The leaching solution can be analysed in accordance with ISO 6486-1, to assess the release of lead and cadmium under these accelerated test conditions.

8 Information and instructions

8.1 Information

8.1.1 General

At least the following information, supplied by the manufacturer, shall be included on the containers or in accompanying literature.

8.1.2 Type I ceramics

a) a time-temperature cycle for the firing schedule (including the final temperature, the time it shall be held, and the rate of heating) and, in the case of a vacuum-fired ceramic, the vacuum level and timing of application;

b) the glazing temperature.

8.1.3 Type II ceramics

Detailed information regarding the handling and treatment of the material considered. In the case of ceramic material produced for machining, information on recommended type of grinding and polishing equipment.

8.2 Instructions for use

8.2.1 Exact instructions for the processing shall be made available to the purchaser by the distributor.

8.2.2 The following information shall accompany instruction materials:

a) the trade-name or the brand-name of the ceramic;

b) the type and class of dental ceramic;

c) the manufacturer's name and address and/or agent in country of sale;

d) the coefficient of thermal expansion between 25 °C and 500 °C (or T_g) and the T_g (where applicable);

e) the shade as identified in the manufacturer's shade guide (if applicable);

f) any special storage conditions;

g) a general warning regarding the potential health hazards (if any), e.g., those associated with inhalation of ceramic dust.

9 Packaging, marking and labelling

9.1 Packaging

Dental ceramic powdered and non-powdered products shall be supplied in sealed containers that will not contaminate or permit contamination of the contents.

9.2 Marking and labelling

9.2.1 The following information shall be clearly marked on each container or on a label securely attached to the container:

a) a lot number or combination of letters and numbers which refers to the manufacturer's records for the particular lot or batch of ceramic;

b) the manufacturer's name (or the distributor's name if privately labelled);

c) the shade as identified in the manufacturer's shade guide (if applicable);

d) product trade name;

e) the minimum net mass, in grams, of the contained ceramic powder, the net volume in milliliters or the number of dose units such as preformed ceramic tablets or ceramic blocks;

f) a general warning regarding potential health hazards (if any), e.g., those associated with inhalation of ceramic dust.

9.2.2 The following information shall be clearly marked on ceramic blocks for CAD/CAM:

a) a lot number or combination of letters and numbers which refers to the manufacturer's records for the particular lot or batch of ceramic;

b) product trade name.

9.2.3 The following information shall be clearly marked on pressable ingots:

a) the shade as identified in the manufacturer's shade guide (if applicable).

Annex A
(informative)

Fracture toughness

A.1 General

Fracture toughness is an important property of dental ceramics, since it is often "inherent" to the material and can be used to predict other properties, such as strength (which is sensitive to flaw size and flaw population). Therefore, fracture toughness values allow meaningful comparisons to be made among ceramics used for structural purposes. Numerous methods are in use that provide good estimates of fracture toughness and vary in their degree of difficulty to execute.

This International Standard recommends the single edge V-notch beam (SEVNB) method as is described in Clause A.2. This method has undergone international evaluation for standardization and was found to be user-friendly, easy, reliable and accurate (see Reference [1]).

Alternatively, fracture toughness may be evaluated by the following methods in accordance with ISO 15732[5], single edge precracked beam; ISO 18756[6], surface crack in flexure, or ISO 24370[7], chevron notched beam. Fracture toughness may not be estimated by methods relying on indentation crack lengths (e.g., where toughness is estimated based on the surface crack lengths associated with the corners of Vickers indentations).

A.2 Single edge V-notched beam method

A.2.1 Apparatus

A.2.1.1 **Equipment for producing parallel-sided beams**, by firing or machining followed by grinding and polishing.

A.2.1.2 **Diamond saw**, for cutting a starter notch in one surface of the beams.

A.2.1.3 **Single-edged razor blade and diamond polishing paste**, to refine the starter notch.

A.2.1.4 **Three-point or four-point test fixture**, as described in 7.3.2.1.2.

A.2.2 Preparation of test specimens

A.2.2.1 Dimensions and form

Five beam specimens are used to measure fracture toughness in accordance with this method. Specimens have a rectangular cross section and dimensions here listed and shown in Figure 2. The edge chamfer specified for strength test specimens is not essential and may be ignored.

Specimen dimensions:

Width $w = 4{,}0$ mm $\pm$ 0,2 mm

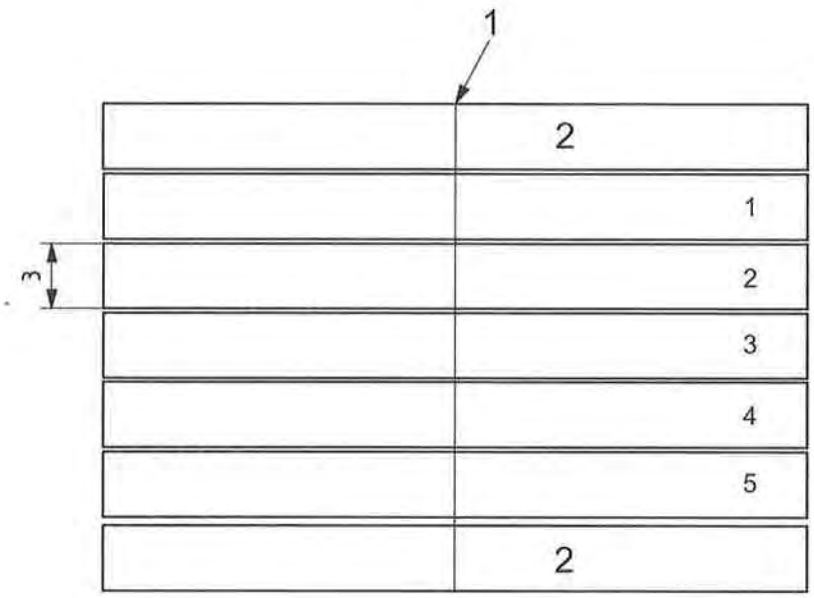
Thickness $b = 3{,}0$ mm $\pm$ 0,2 mm

Specimen lengths shall be at least 2 mm longer than the support span used for testing. Note that this specimen is tested 90° to strength test specimens, that is with the width, w, parallel to the loading direction.

A.2.2.2 Starter notch formation and refinement

Mount five specimens and two dummy specimens (used to protect test specimens during saw cutting and polishing of the starter notch) as close together as possible on a flat holder that allows uniform cutting in the diamond saw. Face one, 3 mm wide, side up to receive the starter notch (this side will be in tension during the fracture test). Draw a pencil line along the measured centre of the beam lengths for orientation of the saw cut.

Dimensions in millimetres



Key

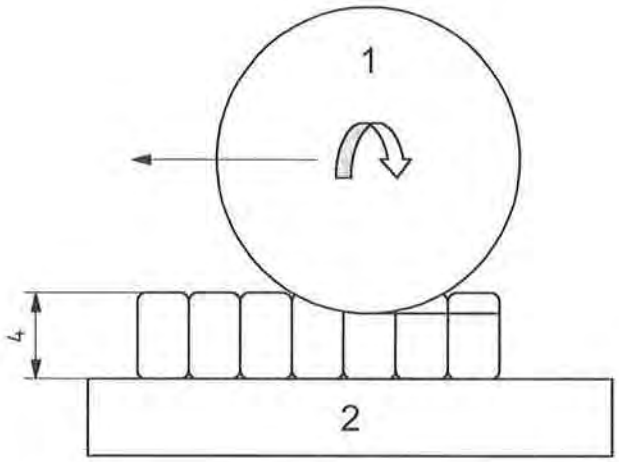
1 pencil line

2 dummy

Figure A.1 — Starter notch formation

Mount the holder in your diamond saw. Use a blade having thickness as close to or only slightly larger than the thickness of the razor blade used in order that the blade is well guided during polishing of the notch (described below). Saw a starter notch along the length of the pencil line to a uniform depth over all specimens of approximately 0,5 mm. Clean the specimens, especially the notch, following the saw cut to remove debris prior to polishing the notch.

Dimensions in millimetres



Key

1 diamond wheel

2 specimen holder

Figure A.2 — Formation with diamond wheel

Following cleaning, fill the notch with diamond polishing paste having a maximum grain size between 3 µm and 6 µm. Put the razor blade into the starter notch and apply light force (5 N to 10 N) and polish using a gentle back-and-forth motion as straight as possible. Using a light microscope examine both ends of the V-notch for evenness of depth. The final V-notch depth shall be uniform and lie between 0,8 mm and 1,2 mm. Remove the specimens from the holder and clean them with acetone in an ultrasonic bath. Dry the specimens well, for example by heating to 110 °C for 1 h.

A.2.2.3 Mechanical testing

Specimens shall be tested for fracture toughness in four-point bending (preferably), or in three-point bending, using a fixture as described in 7.3.2.1.2.

Place the 3 mm width face with the V-notch, down. Load the specimens with a crosshead speed of 0,5 mm/min at room temperature in air. Record the fracture load to three significant figures. Record the thickness, b, and width, w, of each specimen from measurements made using a micrometer capable of measuring to three decimal places. The depths of the V-notches are measured using a calibrated microscope with a magnification $\geqslant 50\times$. Read the depths a_1, a_2 and a_3 to three significant figures. Check to assure that fracture started at the bottom of the V-notch and continued over its entire length (if this is not the case the test is invalid).

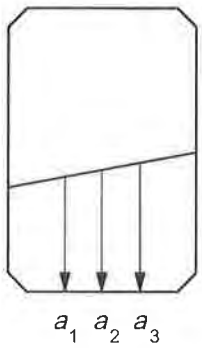


Figure A.3 — Depth of V-notch measurement

A.2.2.4 Calculations

A.2.2.4.1 Average, a, and relative, α, V-notch depths are calculated for each specimen from Equations (A.1) and (A.2). The relative V-notch depth shall be between 0,2 and 0,3 and the relationship involving the variation in notch depths shall be satisfied.

$$a = \frac{(a_1 + a_2 + a_3)}{3} \tag{A.1}$$

$$\frac{(a_{max} - a_{min})}{a} \leqslant 0,1 \text{ (this relationship is assumed to be satisfied)}$$

$$\alpha = a / w \tag{A.2}$$

where

a is the average notch depth in millimetres;

a_{max} is the maximum among a_1, a_2 and a_3 in millimetres;

a_{min} is the minimum among a_1, a_2 and a_3 in millimetres;

α is the relative V-notch depth.

A.2.2.4.2 Fracture toughness, K_{Ic}, is calculated for each specimen as follows:

$$K_{Ic} = \sigma\sqrt{a}Y = \frac{P}{b\sqrt{w}} \times \frac{S_1 - S_2}{w} \times \frac{3\sqrt{\alpha}}{2(1-\alpha)^{1,5}} Y$$

NOTE 1 There is no S_2 in three-point testing.

For four-point flexure use:

$$Y = 1,988\ 7 - 1,326\alpha - \frac{(3,49 - 0,68\alpha + 1,35\alpha^2)\alpha(1-\alpha)}{(1+\alpha)^2}$$

For three-point flexure use:

$$Y = 1,947\ 2 - 5,024\ 7\alpha + 11,895\ 4\alpha^2 - 18,063\ 5\alpha^3 + 14,598\ 6\alpha^4 - 4,689\ 6\alpha^5$$

where

K_{Ic} is the fracture toughness in megapascals by square root metre;

σ is the fracture strength in megapascals;

P is the fracture load in meganewtons;

b is the specimen thickness in millimetres;

w is the specimen width in millimetres;

S_1, S_2 are the support spans ($S_1 > S_2$) in millimetres;

Y is the stress intensity shape factor.

NOTE 2 The three-point equation is considered valid only for the case where $0,35 < a/w < 0,7$ and $S/w = 10$. Alternative equations, such as those given in ASTM C1421-01b[8], should be used for different S/w conditions.

Perform all calculations to three significant figures. Calculate the mean K_{Ic} value and standard deviation and report the result rounded to two decimal places.

A.2.2.4.3 Control calculation values for four-point flexure, $K_{Ic} = 7,42$ MPa $\sqrt{m}$ when

$P = 100 \times 10^{-6}$ MN; $b = 3 \times 10^{-3}$ m; $w = 4 \times 10^{-3}$ m; $a = 2 \times 10^{-3}$ m; $S_1 = 40 \times 10^{-3}$ m and $S_2 = 20 \times 10^{-3}$ m

A.2.2.4.4 Control calculation values for three-point flexure, $K_{Ic} = 7,25$ MPa $\sqrt{m}$ when

$P = 100 \times 10^{-6}$ MN; $b = 3 \times 10^{-3}$ m; $w = 4 \times 10^{-3}$ m; $a = 2 \times 10^{-3}$ m; $S_1 = 20 \times 10^{-3}$ m

NOTE All calculations above use meganewtons and metres; equivalent values in newtons and millimetres can be used instead.

Table A.1 lists recommended fracture toughness values for six classes of ceramic.

Table A.1 — Classification of ceramics for fixed prostheses by intended clinical use, with recommended fracture toughness values

Class	Recommended clinical indications	Fracture toughness MPa $\sqrt{m}$ (minimum)
1	a) Aesthetic ceramic for coverage of a metal or a ceramic substructure. b) Aesthetic-ceramic: single-unit anterior prostheses, veneers, inlays or onlays.	0,7
2	a) Aesthetic ceramic: adhesively cemented, single-unit, anterior or posterior prostheses. b) Adhesively cemented, substructure ceramic for single-unit anterior or posterior prostheses.	1,0
3	Aesthetic ceramic: non-adhesively cemented, single-unit, anterior or posterior prostheses.	2,0
4	a) Substructure ceramic for non-adhesively cemented, single-unit, anterior or posterior prostheses. b) Substructure ceramic for three-unit prostheses not involving molar restoration.	3,0
5	Substructure ceramic for three-unit prostheses involving molar restoration.	3,5
6	Substructure ceramic for prostheses involving four or more units.	5,0

Annex B
(informative)

Weibull statistics

B.1 Weibull distribution

Ceramic strength data are generally not normally distributed about the mean, but often skewed in the high strength portion. More general distributions, such as the Weibull 2-parameter distribution, can fit both asymmetric and normally distributed data.

The Weibull 2-parameter distribution function relates the cumulative probability of failure, P_f, of an area (or volume) under tensile stress to two parameter estimates: (i) the Weibull modulus, m, (see B.2), and (ii) the Weibull characteristic strength, σ_0, (see B.3), according to the following relationship:

$$P_f = 1 - \exp\left[-\left(\frac{\sigma}{\sigma_o}\right)^m\right]$$

B.2 Weibull modulus

The Weibull modulus is the parameter describing the shape (including width) of the distribution of strength as a function of failure probability. It is similar, but inversely related to the standard deviation in a normal distribution, i.e., considering the same σ_0, the smaller the Weibull modulus the larger the scatter of the data.

B.3 Weibull characteristic strength

B.3.1 General

The Weibull characteristic strength is the strength occurring at a probability of failure of 63,2 % for a particular test specimen and loading configuration.

B.3.2 Calculation of Weibull strength parameters

The Weibull modulus and characteristic strength are estimated from flexure strength data by rank order statistics.

Begin by ranking the strengths from a batch of specimens (minimum fifteen and preferably thirty) in ascending order and assign each specimen a probability of failure based on its ranking.

$$P_f = \frac{i - 0,5}{N}$$

where

i is the 1, 2, 3, 4 ... ith;

N is the number of specimens in the batch.

Transform the variables P_f and σ to lnln [(1/1–P_f)] and lnσ, respectively. This is the double natural logarithm of 1/(1–P_f) and the natural logarithm of σ. Construct a plot with lnln[(1/1–P_f)] on the ordinate and a corresponding lnσ on the abscissa as in Figure B.1 where the slope of the curve is equal to m.

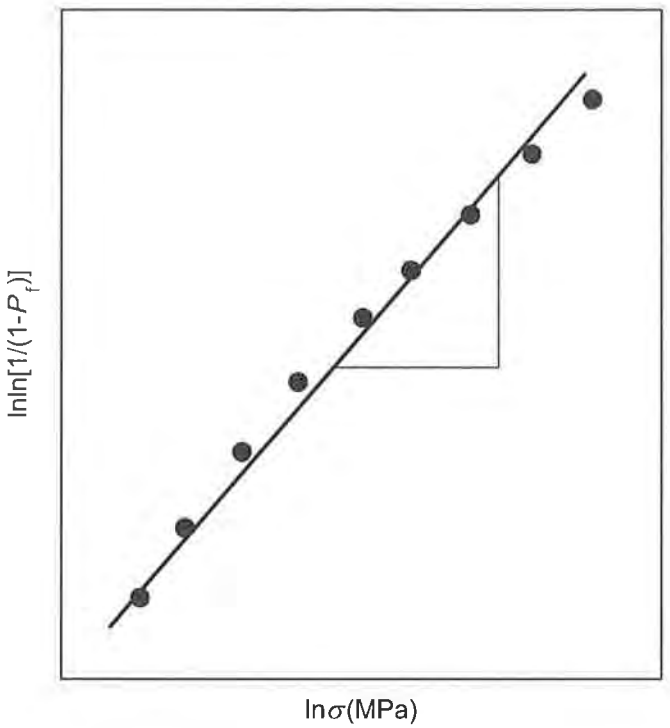


Figure B.1 — Determination of Weibull modulus

Calculate the linear regression fit to the straight line i.e., $y = Mx + b$.

The Weibull modulus, m, is equal to the slope of the linear regression fit (i.e., M).

The Weibull characteristic strength, σ_0, is calculated by setting $y = 0$. Set $y = 0$ and determine x. Since at $y = 0$, x is the natural log of the characteristic strength (at $\sigma = \sigma_0$, $P_f = 63{,}2$ %).

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[1] QUINN, G., *Fracture Toughness of Advanced Ceramics at Room Temperature: A VAMAS Round Robin*, Ceram. Eng. Sci. Proc., **14**, pp. 92-100, 1993

[2] ISO 6486-1, *Ceramic ware, glass-ceramic ware and glass dinnerware in contact with food — Release of lead and cadmium — Part 1: Test method*

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

[4] ISO 10993-1, *Biological evaluation of medical devices — Part 1: Guidance on selection of tests*

[5] ISO 15732, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for fracture toughness of monolithic ceramics at room temperature by single edge precracked beam (SEPB) method*

[6] ISO 18756, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of fracture toughness of monolithic ceramics at room temperature by the surface crack in flexure (SCF) method*

[7] ISO 24370, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for fracture toughness of monolithic ceramics at room temperature by chevron-notched beam (CNB) method*

[8] ASTM C1421-01b, *Standard Test Methods for Determination of Fracture Toughness of Advanced Ceramics at Ambient Temperature*

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