INTERNATIONAL STANDARD

ISO
6872

Fourth edition
2015-06-01

Dentistry — Ceramic materials

Médecine bucco-dentaire — Matériaux céramiques

Reference number
ISO 6872:2015(E)

ISO copyright office
Ch. de Blandonnet 8 • CP 401
CH-1214 Vernier, Geneva, Switzerland
Tel. +41 22 749 01 11
Fax +41 22 749 09 47
copyright@iso.org
www.iso.org

Contents

Page

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 fourth edition cancels and replaces the third edition (ISO 6872:2008), which has been technically revised with the following changes:

- new edition of ISO 23146:2012 for fracture toughness by SEVNB has been added as an alternative in Annex A. It has a rigorous procedure developed by ISO/TC 206, *Fine ceramics*;
- a restriction on the use of the SEVNB method for fracture toughness determination for 3Y-TZP has been added. In most cases, the notch cannot be made sharp enough with a razor blade;
- maximum chamfer size on bend bars has been reduced for the case of the thin specimens;
- recommendations to grind lengthwise were added to the bend bar preparation step in 7.3.1.2.2;
- the Y equations for SEVNB fracture toughness in 3–point have been refined and expanded to cover more configurations;
- modification to Table 1 changing “aesthetic” to “monolithic”.

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 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 3696, *Water for analytical laboratory use — Specification and test methods*

ISO 1942, *Dentistry — Vocabulary*

ISO 13078, *Dentistry — Dental furnace — Test method for temperature measurement with separate thermocouple*

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
add-on ceramic
correction ceramic
dental ceramic material which is fired at a reduced temperature and is normally applied to restore contact areas on a dental restoration or prosthesis

3.1.2
aesthetic ceramic
dental porcelain (3.1.5) or *glass ceramic* (3.1.10) having appropriate translucency and colour used to mimic the optical properties of natural teeth

3.1.3
chromatic dentin ceramic
dentine ceramic having a high strength or saturation of the hue (color)

3.1.4
dental ceramic
inorganic, non-metallic material which is specifically formulated for use when processed according to the manufacturers' instructions to form the whole or part of a dental restoration or prosthesis

3.1.5
dental porcelain
predominantly, glassy *dental ceramic* (3.1.4) material used mainly for aesthetics in a dental restoration or prosthesis

3.1.6
dentine ceramic
dental ceramic (3.1.4) material used to form the overall shape and basic colour of a dental restoration or prosthesis simulating the natural tooth dentine

3.1.7
enamel ceramic
dental ceramic (3.1.4) material used to overlay, either partially or wholly, the *dentine ceramic* (3.1.6) and also, to form the more translucent incisal third of a dental restoration or prosthesis simulating the natural tooth enamel

3.1.8
flame-sprayed dental ceramic
dental ceramic core or substructure layer formed via the technique of flame-spraying

3.1.9
fluorescent ceramic
dental ceramic (3.1.4) material that absorbs radiant energy and emits it in the form of radiant energy of a different wavelength band all or most of whose wavelengths exceed that of the absorbed energy

EXAMPLE Absorption of ultraviolet light with emission of blue light.

3.1.10
glass ceramic (dental)
dental ceramic (3.1.4) 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

3.1.11
glass-infiltrated dental ceramic
dental ceramic core or substructure layer which is porous and is subsequently densified by the infiltration of specialised glass at elevated temperature

3.1.12
glaze ceramic
dental ceramic (3.1.4) material which is overlayed and fired at a lower temperature compared to *dentine ceramic* (3.1.6) or *enamel ceramic* (3.1.7) to produce a thin coherent sealed surface, the level of gloss being determined by the firing conditions

3.1.13
liner
dental ceramic (3.1.4) material used on all ceramic substructure forming a layer that provides a background colour upon which dentine or opaceous dentine can be applied to achieve overall aesthetics

3.1.14
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

3.1.15
modifying enamel ceramic
enamel ceramic (3.1.7) used to modify the surface contour of a restoration, for example, add a contact, often fired at a lower temperature than the enamel ceramic or dentine ceramic

3.1.16
monolithic ceramic
dental ceramic (3.1.4) that is substantially made of a single uniform material

Note 1 to entry: A thin layer of *glaze* (3.3.4) (staining technique) can be applied.

3.1.17
opaceous dentine ceramic
dental ceramic (3.1.4) material having a higher opacity than a *dentine ceramic* (3.1.6) 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

3.1.18

opalescent enamel ceramic

enamel ceramic (3.1.7) material that scatters shorter wavelengths of light (e.g. blue) and transmits longer wavelengths of light (e.g. red)

3.1.19

opaque dental ceramic

dental ceramic (3.1.4) material which when applied to a metallic substructure, according to the 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

3.1.20

shoulder ceramic

margin ceramic

dental ceramic (3.1.4) material used to form shape and colour at the marginal area of the dental restoration or prosthesis simulating natural tooth dentine in this area

3.1.21

stain ceramic

dental ceramic powder or paste which is normally intensely coloured and which 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 found in natural teeth

3.1.22

substructure (core) dental ceramic

predominantly, polycrystalline dental ceramic material that forms a supporting substructure upon which one or more layers of *dental ceramic* (3.1.4) 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* (3.1.4) under ambient atmospheric pressure

3.2.2

dental CAD/CAM

computer-aided design/computer-aided manufacture (CAD/CAM) procedures to manufacture a dental restoration or prosthesis normally including the following stages: a) a digital scanning procedure of the model, wax-up, or intra-orally to produce a 3D data set; b) software manipulation of the 3D data set to design the prosthesis; c) 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 (3.1.4) material, normally in the form of a pellet or ingot (often pre-sintered), designed for use in a specialised furnace which enables the ingot to be injected/cast/pressed into a mould prepared through the lost wax technique

3.2.5
sintering of a dental ceramic
process whereby heat and potentially other process parameters, for example, mechanical or gas pressure, are applied to a ceramic powder or powder compact in order to densify the ceramic into its required form

Note 1 to entry: “Firing” and “sintering” are used interchangeably in this International Standard (“firing” connoting the application of heat to drive sintering).

3.2.6
vacuum firing dental ceramic
firing of *dental ceramics* (3.1.4) at reduced pressure (i.e. under vacuum) to yield the required density and associated aesthetics especially the degree of translucency

Note 1 to entry: 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* (3.1.4) 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 midpoint 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
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 five classes according to their intended clinical use and according to the 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 with required mechanical and chemical properties

Class	Recommended clinical indications	Mechanical and chemical properties	
		Flexural strength [MPa] minimum value for mean (see 7.3.1.4)	Chemical solubility [µg/cm^2]
1	a) Monolithic ceramic for single-unit anterior prostheses, veneers, inlays, or onlays adhesively cemented.	50	<100
	b) Ceramic for coverage of a metal framework or a ceramic substructure.	50	<100
2	a) Monolithic ceramic for single-unit anterior or posterior prostheses adhesively cemented.	100	<100
	b) Partially or fully covered substructure ceramic for single-unit anterior or posterior prostheses adhesively cemented.	100	<2 000
3	a) Monolithic ceramic for single-unit anterior or posterior prostheses and for three-unit prostheses not involving molar restoration adhesively or non-adhesively cemented.	300	<100
	b) Partially or fully covered substructure for single-unit anterior or posterior prostheses and for three-unit prostheses not involving molar restoration adhesively or non-adhesively cemented.	300	<2 000
4	a) Monolithic ceramic for three-unit prostheses involving molar restoration.	500	<100
	b) Partially or fully covered substructure for three-unit prostheses involving molar restoration.	500	<2 000
5	Monolithic ceramic for prostheses involving partially or fully covered substructure for four or more units or fully covered substructure 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 ceramic	Pink
Enamel ceramic	Blue
Fluorescent ceramic	Yellow
Highly chromatic dentine ceramic	Orange
Opalescent enamel ceramic	Blue-green
Modifying enamel ceramic (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 in 7.1.3. 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·g^{-1} of ^{238}U. Test in accordance with 7.2.2.

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

5.3 Mixing and condensation properties of type I ceramics

When mixed as in 7.1.3 with water or the modelling fluid recommended by the manufacturer, a dental ceramic powder shall neither form lumps, nor granules, when assessed by visual inspection.

The paste formed shall be suitable for making the indicated restorations and prostheses by condensation of successive layers. When the paste is condensed as in 7.1.4, it shall neither crack, nor crumble, when assessed by visual inspection during drying.

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). 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).

5.5 Biocompatibility

See the introduction for guidance on biocompatibility.

5.6 Shrinkage factor

The absolute accuracy of the shrinkage factor by which the dimensions of the partially sintered material is to be divided as provided under 9.2.2 c) shall be ±0,002.

6 Sampling

6.1 Type I ceramics

Use retail packages from the same batch containing enough material to carry out the specified tests plus an allowance for repeated tests, if necessary. Where there is more than one shade in a class of dental ceramic, perform test with a colour/shade most commonly used. All of the materials tested shall be of the same lot.

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

7 Test methods

7.1 Preparation of test specimens

For detailed instructions, see the individual test methods.

For type I specimens (unless otherwise stated or inconsistent with the text), the apparatus detailed in 7.1.2 along with the conditions for mixing, condensation, and firing (7.1.3, 7.1.4, and 7.1.5) apply to all the test methods.

7.1.1 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 (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.

7.1.2 Apparatus for mixing

All apparatus for mixing shall be clean and dry.

7.1.2.1 Glass slab or mixing palette.

7.1.2.2 Spatula, made from 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.

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

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

7.1.3 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.

7.1.4 Procedure for specimen fabrication

Overfill the mould 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.

7.1.5 Firing

Position the specimens in the furnace so that they will be uniformly fired 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 should 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 specific for the given test specimen which could be different from the instructions for use.

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 to be 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 ^{238}U by neutron activation or gamma spectroscopy.

NOTE Those with gamma spectroscopy techniques should 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

Three flexural test methods are acceptable thath are

a) three-point bending,

b) four-point bending, and

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

7.3.1 Three-point and four-point bending tests

7.3.1.1 Apparatus

7.3.1.1.1 Universal mechanical testing machine, capable of a crosshead speed of (1 ± 0,5) mm/min and an ability to measure applied loads of between 10 N and [1 000 N or 2 500 N] e.g see ISO 7500-1.

7.3.1.1.2 Flexural test fixtures.

7.3.1.1.2.1 Fixture 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 R_a. It is recommended that the actual spacing between the support roller centres (L) be measured to within 0,1 mm.

7.3.1.1.2.2 Fixture for four-point bending, consisting of 1/4-point test configuration such that the test piece is loaded by two inner bearing rollers located at 1/4 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 apart such that L = 16,0 mm to 40,0 mm. 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 R_a. 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. The loading arrangement shall ensure that equal forces are applied to the loading rollers and that torsional loading is minimized. It is recommended that the actual spacing between the roller centres (L for support and $L/2$ for inner) be measured to within 0,1 mm. It is also recommended that the inner span be centred over the support span to within 0,1 mm.

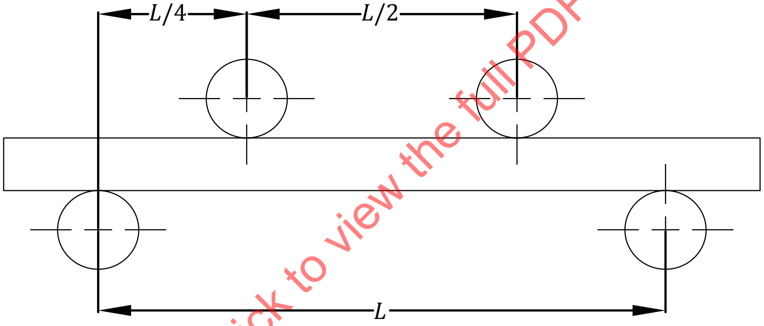


Figure 1 — Schematic of the four-point-1/4-point fixture configuration (Note: Moment arm = $L/4$)

7.3.1.2 Preparation of test specimens

7.3.1.2.1 Test specimen dimensions and test parameters

Dimensions:

Specimens for three-point and four-point bending testing have a rectangular cross section and most preferably an edge chamfer as per the diagram in Figure 2 and dimensions listed below. The edge chamfer should be ground or rounded lengthwise along the long axis of the specimen so that grinding damage and chipping is minimized. The edge chamfer can be ground prior to final sintering if kept within below values after final sintering.

Specimen dimensions:

Width	w = (4 ± 0,2) mm (dimension of the side at right angles to the direction of the applied load)
Thickness	b = (2,1 ± 1,1) mm (with 3,0 mm recommended; dimension of the side parallel to the direction of the applied load)
Chamfer	c = (0,12 ± 0,03) mm [with a maximum of 0,10 mm recommended for small thickness specimens (b < 2,0 mm)]

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 1/4 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 ≤0,1.

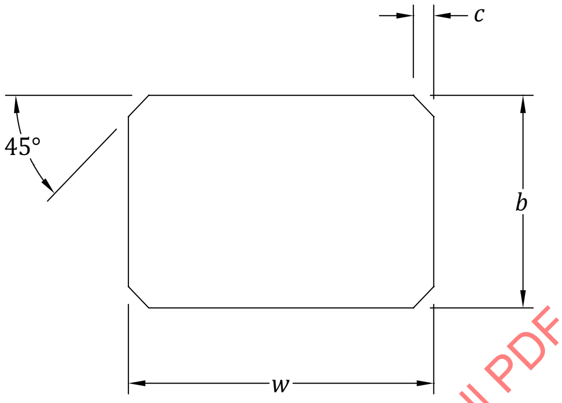


Figure 2 — Specification of indicated dimensions

7.3.1.2.2 Type I ceramics

Prepare at least 10 and preferably 30 specimens of dimensions as specified in 7.3.1.2.1. Use a mould appropriately sized to allow for dimensional changes resulting from sintering and finishing. Fire the specimens 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 7.3.1.2.1. 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. Grinding of all four long faces should be done lengthwise parallel to the specimen long axis wherever possible to minimize grinding damage that could alter the strength. Polishing may not necessarily remove prior grinding damage unless the polishing removes 20 µm to 30 µm of the material. Ensure that opposing faces of the test pieces are flat and parallel within 0,05 mm. Where appropriate, testing of specimen "as fired" (without further grinding at the edge or on the surface) may be done so long as the shape and dimensions are within the limits specified. Thoroughly clean the test pieces ensuring that all traces of grinding debris are removed.

7.3.1.2.3 Type II ceramics

Prepare according to the manufacturer's instructions at least 10 and preferably 30 specimens of dimensions as specified in 7.3.1.2.1. 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 7.3.1.2.2. Where appropriate, testing of specimen "as fired" (without further grinding at the edge or on the surface) could be done as long as the shape and dimensions are within the limits specified.

7.3.1.3 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.

7.3.1.4 Calculation of strength

7.3.1.4.1 Three-point flexure

From Formula (1), calculate the flexural strength, σ, in megapascals and report the mean and standard deviation of the strength data. Means should equal or exceed the requirements listed in Table 1. In addition, if at least 15 specimens have been tested, the Weibull characteristic strength and Weibull modulus may be reported as well, per 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 millimetres;

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 millimetres;

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

7.3.1.4.2 Four-point flexure

From Formula (2), calculate the flexural strength, σ, in megapascals and report the mean and standard deviation of the strength data. Means should equal or exceed the requirements listed in Table 1. In addition, if at least 15 specimens have been tested, the Weibull characteristic strength and Weibull modulus may be reported as well, per 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 millimetres;

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 millimetres;

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

NOTE Formula (3) can be used if the spans are such that the loading points are not exactly at $L/4$.

$$\sigma = \frac{3P\left(L_o - L_i\right)}{2wb^2} \tag{3}$$

where

L_0 is the outer loading span;

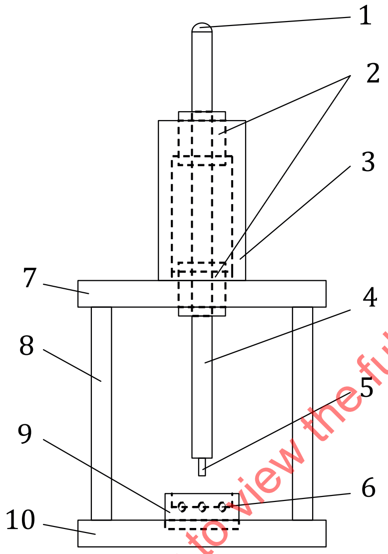
L_i is the inner loading span.

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

7.3.2.1 Apparatus

7.3.2.1.1 Universal mechanical testing machine, capable of a crosshead speed of (1 ± 0,5) mm/min and an ability to measure applied loads of between 10 N and 2 500 N (±1 %).

7.3.2.1.2 Biaxial flexural strength test fixture. For the support of the test specimen, three hardened steel balls with a diameter of (4,5 ± 2) mm positioned 120° apart on a support circle with a diameter of (11 ± 1) 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	ram, hardened, precision ground rod
5	hardened, precision ground dowel pin (1,4 ± 0,2) mm diameter
6	three (4,5 ± 2) mm steel balls 120° apart on (11 ± 1) mm diameter circle
7	top plate
8	three separator posts
9	sample holder
10	bottom plate

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

7.3.2.2 Preparation of test specimens

7.3.2.2.1 Type I ceramics

Prepare at least 10 and preferably 30 discs having a thickness of (1,2 ± 0,2) mm and a diameter of (14 ± 2) mm. Use a mould appropriately sized to allow for dimensional changes resulting from sintering and finishing. Final grind each specimen through 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 within 0,05 mm. Thoroughly clean the test pieces ensuring that all traces of grinding debris are removed. Where appropriate, testing of specimen “as fired” (without further grinding at the edge or on the surface) may be done so long as the shape and dimensions are within the limits specified.

7.3.2.2.2 Type II ceramics

Prepare according to the manufacturer’s instructions at least 10 and preferably 30 discs having a diameter between (14 ± 2) mm and an approximate thickness of (1,5 ± 0,5) 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 ± 0,2) mm and a diameter of (14 ± 2) mm. Grind and finish specimens using the protocol specified under 7.3.2.2.1. Where appropriate, testing of specimen “as fired” (without further grinding at the edge or on the surface) may be done so long as the shape and dimensions are within the limits specified.

7.3.2.3 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.

EXAMPLE Polyethylene sheet, thickness 0,05 mm.

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

From Formula (4), 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 15 specimens have been tested, the Weibull characteristic strength and Weibull modulus may be reported as well, per Annex B.

$$\sigma = -0{,}2387 P (X - Y) / b^2 \tag{4}$$

where

σ is the maximum centre tensile stress, in megapascals;

P is the total load causing fracture, in newtons;

$$\begin{aligned} X &= (1+\upsilon)\ln(r_2/r_3)^2 + [(1-\upsilon)/2](r_2/r_3)^2 \\ Y &= (1+\upsilon)[1+\ln(r_1/r_3)^2] + (1-\upsilon)(r_1/r_3)^2 \end{aligned} \tag{5}$$

where

v is Poisson’s ratio (if the value for the ceramic concerned is not known, use Poisson’s ratio = 0,25);

r_1 is the radius of support circle, in millimetres;

r_2 is the radius of loaded area, in millimetres;

r_3 is the radius of specimen, in millimetres;

b is the specimen thickness at fracture origin, in millimetres.

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 of 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 vacuum and once at atmospheric pressure in air and the other two specimens three times in vacuum and once at atmospheric pressure in air. Multiple firing is not obligatory for materials where the linear thermal expansion coefficient is not influenced by multiple firing. 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 is at 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 either 25 °C or 50 °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 (as per Figure 4). For each test specimen, determine the linear expansion coefficient between either 25 °C or 50 °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 either 25 °C or 50 °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).

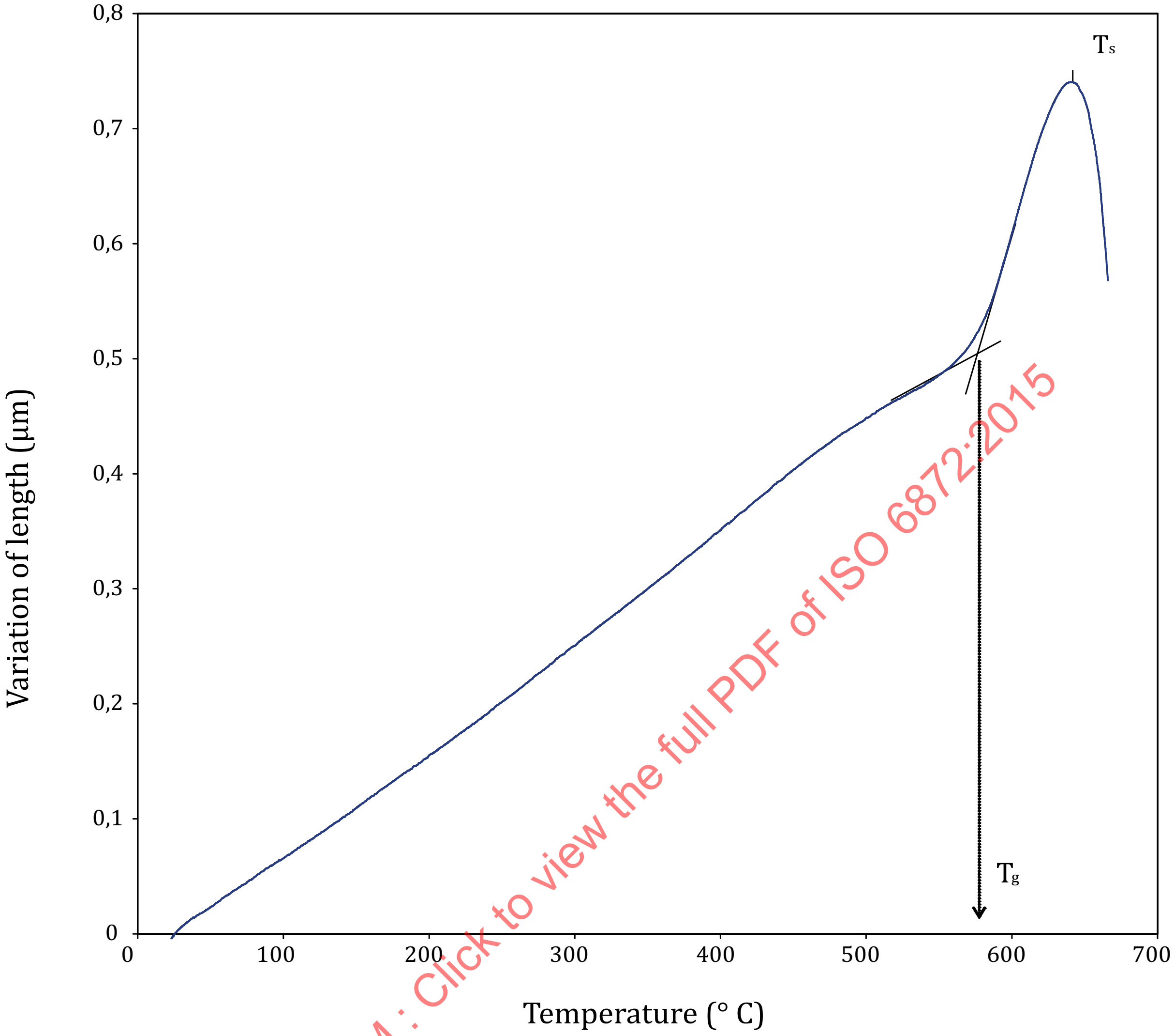


Figure 4 — Typical dilatometry curve demonstrating graphical method of determining glass transition temperature (T_g) (T_S is the dynamic softening temperature of the ceramic under the dilatometer load and heating rates used)

7.5.2 Assessment of results

Report the mean and standard deviation of the glass transition temperature measured in 7.5.1 in degree Celsius.

7.6 Chemical solubility

7.6.1 Reagent

Acetic acid (analytical grade), 4 % (V/V) 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 the powder collected as per 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 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 as per ISO 3696, dry them at (150 ± 5) °C for 4 h, and weigh them to the nearest 0,1 mg. Determine the total surface area to the nearest 0,5 cm^2 . Use a 250 ml or several 100 ml Pyrex glass or plastic bottles with 100 ml acetic acid (analytical grade) and 4 % (V/V) solution in water of grade 3 as specified in ISO 3696. Preheat to (80 ± 3) °C and place specimens into the bottle. Close the bottle and place it in an oven at (80 ± 3) °C for 16 h. Wash the specimens with water of grade 3 as per ISO 3696, dry them at (150 ± 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.

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 should be held, and the rate of heating and cooling) 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. In the case of ceramic material produced for machining, information on recommended type of grinding and polishing equipment.

For partially sintered ceramics and glass ceramics that require further heat treatment, this shall be specified by heating rates and holding times with temperatures and times assigned permissible ranges appropriate to furnaces calibrated according to ISO 13078 or equivalent.

8.2 Instructions for use

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

8.2.2 The following information shall accompany the instruction materials:

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

b) name and address of the manufacturer and/or his authorized representative;

c) the type and class of dental ceramic;

d) the coefficient of thermal expansion between either 25 °C or 50 °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), for example, 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 nor 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 and/or his authorized representative (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 millilitres or the number of dose units such as preformed ceramic tablets, or ceramic blocks;

f) a general warning regarding the potential health hazards (if any), for example, 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 refer to the manufacturer's records for the particular lot or batch of ceramic;

b) product trade name;

c) for partially sintered ceramic materials, the shrinkage factor by which the dimensions of the partially sintered material is to be divided shall be quoted as a factor to three decimal places.

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 Fracture Toughness

A.1.1 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 in accordance with ISO 23146 or alternatively, by a simplified version as described in A.1.2. The SEVNB-method has undergone international evaluation for standardization and was found to be user-friendly, easy, reliable, and accurate (see References [1] and [11]). According to ISO 23146, the SEVNB method states that it should not be used to measure the fracture toughness of fine grained materials with grain sizes of less than 1 µm since the interlaboratory test exercise[11] showed that most participants were not able to obtain sharp notch-tip radius with the razor sharpening method. The majority of fracture toughness data overestimated the true fracture toughness due to notch bluntness[11] Also, ISO 23146 specifically states in Clause 1 that the method should not be used for yttria-tetragonal polycrystal zirconia because of the difficulties in forming sharp notch-tip radius.

Alternatively, fracture toughness can be evaluated by the following methods in accordance with ISO TC 206: single-edge precracked beam (ISO 15732), surface crack in flexure (ISO 18756), or chevron-notched beam (ISO 24370). The SEPB, CNB, and SCF methods can be used for yttria-tetragonal polycrystal zirconia. Each of these methods has pros and cons and some experience may be needed to readily obtain precise and accurate results.

Fracture toughness should 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.1.2 Single-edge v-notched beam method-apparatus

A.1.2.1 Equipment, for producing parallel-sided beams by firing or machining followed by grinding and polishing.

A.1.2.2 Diamond saw, for cutting a starter notch in one surface of the beams.

A.1.2.3 Single-edged razor blade, and diamond polishing paste to refine the starter notch.

A.1.2.4 Three-point or four-point test fixture, as described in 7.3.1.1.2.

A.1.3 Preparation of test specimens

A.1.3.1 Five beam specimens, are used to measure fracture toughness in accordance with this method. Specimens have a rectangular cross section as per the diagram and dimensions listed below and in Figure A.2. The edge chamfer specified for strength test specimens should not be used.

Specimen dimensions:

Thickness $w = (4{,}0 \pm 0{,}2)$ mm

Width $b = (3{,}0 \pm 0{,}2)$ mm

Specimen lengths should be at least 2 mm longer than the support span used for testing. Note that this specimen is tested 90° to strength test specimens.

A.1.3.2 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 onto a flat holder that allows uniform cutting in your 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.

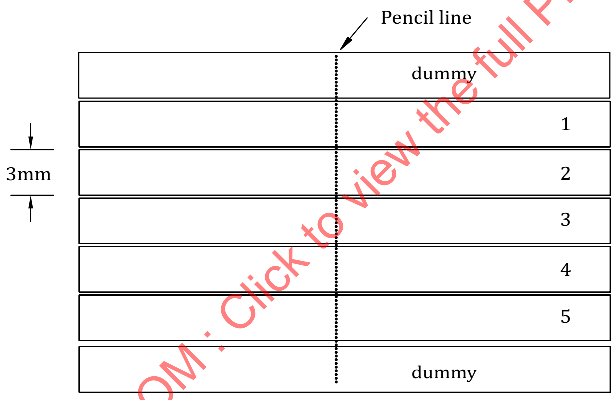


Figure A.1 — Starter notch formation

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.

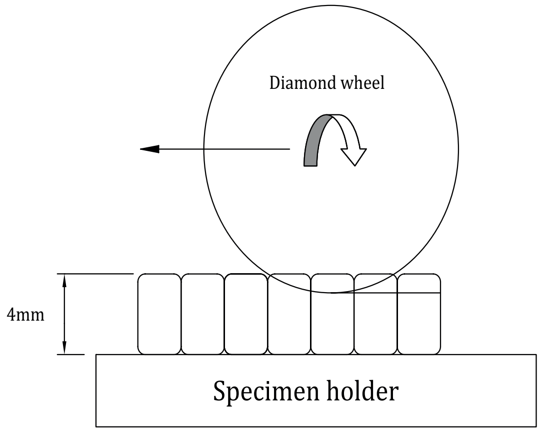
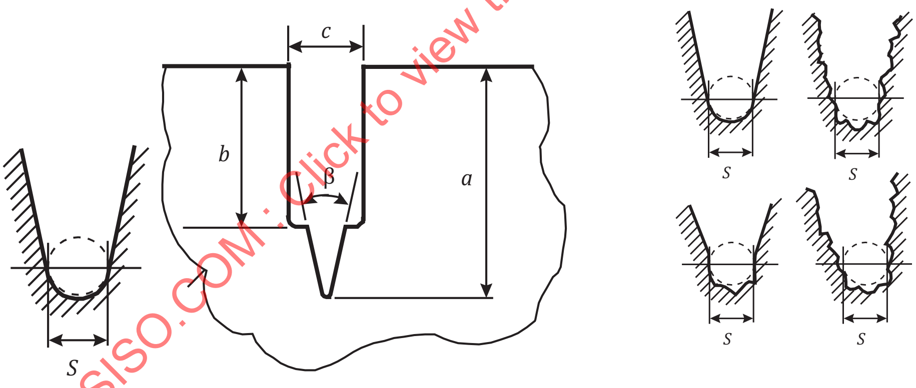


Figure A.2 — Formation with diamond wheel

Following cleaning, fill the notch with diamond polishing paste having a maximum grain size between 3 µm and 6 µm. Put razor blade into 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 occasionally for evenness of depth. The final V-notch depth shall be uniform and lie between 0,8 mm and 1,2 mm. The final V-notch depth should be deeper than the starting notch depth such that $a - c > b$ as shown below. 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 an hour.



a)

b)

Key

- *a* 0,8 mm up to 1,2 mm
- *b* approximately 0,5 mm
- *c* width of razor blade, $a - b < c$
- β approximately 30 degrees (°) or as small as possible
- *S* V-notch width

Figure A.3 — Schematic geometry of V-notches: (a) notch width and angle measurement and (b) interpretation of notch root shape from ISO 23146

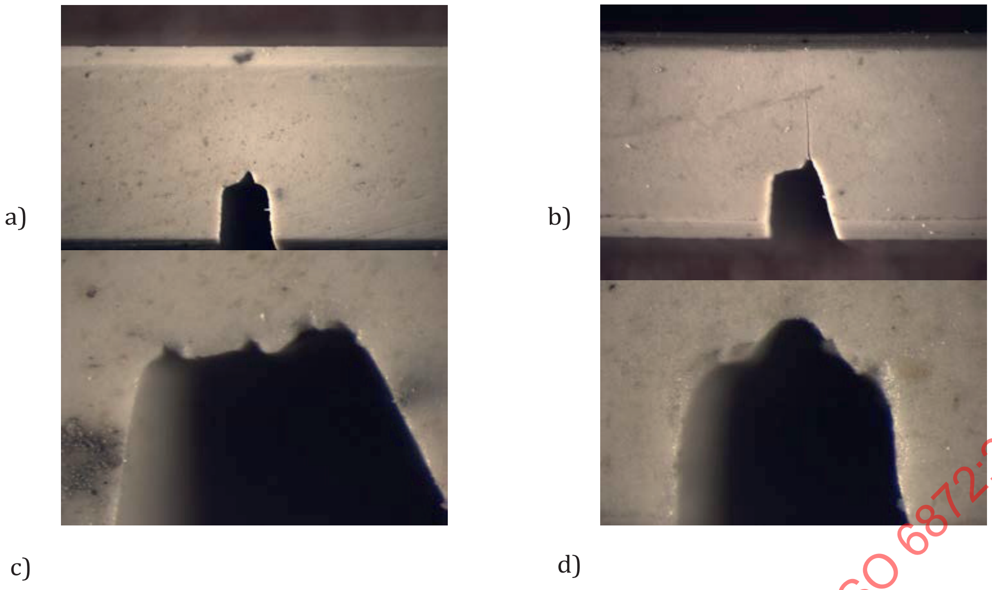


Figure A.4 — Examples of acceptable and unacceptable notch geometries: (a) an acceptable notch, (b) notch improperly centred, (c) poor notch development, and (d) notch insufficiently sharp

SEVNB method from ISO 23146 (Paragraph 6.5):

Select two test pieces from the set of five for testing. Photograph the V-notch on one side of each of the selected test pieces using a magnification of about 50x or greater if the notch tip is not resolved at this magnification. Control the V-notch geometry with the help of the photograph. Report any deviation from the geometry shown in Figure A.3.

Photograph the V-notch tip on the same two test pieces with a magnification approximately 300x. Measure the V-notch angle and width in accordance with Figure A.3. Report the V-notch angle β and width S.

The notch width, S, should be of the same order as, or smaller than, twice the average grain size of the test material. Notches of width greater than 20 µm are not acceptable in any test material irrespective of grain size.

NOTE 1 The average grain size can be determined by a linear intercept method such as that described in References [7] and [10].

NOTE 2 It is helpful to determine the acceptability of the notch width on the outermost test pieces before removing them from the mounting plate. This allows the notch honing process to be continued if the notch width is too large.

A.1.3.3 Mechanical testing

Specimens will be tested for fracture toughness in four-point bending (preferably) or in three-point bending using a fixture as described in 7.3.1.1.2.

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 width (b) and thickness (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 ≥50x. Read the depth, a_1, a_2, and a_3, to three significant figures. Check to ensure that fracture started at the bottom of the V-notch over its entire length (if this is not the case, the test is invalid). On the fracture surfaces, examine the notch root for any evidence of stable crack extension prior to fracture. If stable crack extension occurred and can be measured, include the stable extension in the calculations below. Inspect the load-displacement or load-time record from the fracture