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Dentistry — Machinable ceramic blanks

Médecine bucco-dentaire — Ébauches en céramique usinables

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Foreword

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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).

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For an explanation of the voluntary nature of standards, the meaning of ISO specific terms and expressions related to conformity assessment, as well as information about ISO's adherence to the World Trade Organization (WTO) principles in the Technical Barriers to Trade (TBT), see www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 106, *Dentistry*, Subcommittee SC 9, *Dental CAD/CAM systems,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 55, *Dentistry,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Introduction

A variety of ceramic blank materials are being used in machining systems for fabrication of various restorations. Although all these materials can have different chemical and microstructural makeup, there are some unique and common concerns for machining and performance of these materials. Machining damage, minimum machined thickness, and machining tolerances all are common concerns for these materials.

The overwhelming use of zirconia and alumina is in the form of green or partially sintered blanks with shrinkage values of 20 % to 35 % by volume when sintered to full density. In order for the restoration to be fabricated with proper accuracy, the blank density should be carefully measured and conveyed to the computer controlled milling unit. This allows for proper oversizing and shrinkage to provide an accurate fit. Furthermore, the blank should be homogeneous throughout the body, otherwise differential shrinkage occurs resulting in significant warping and departure from linearity.

With respect to glass ceramics, a subset requires crystallization post-machining during which distortion can occur placing the machined part out of the tolerance specified for the restoration. Also, another subset is machined in the crystallized state that can cause significant machining damage affecting the properties of the material.

The machining process can cause surface and subsurface damage that can decrease the flexural strength of the material. Furthermore, damage can limit the minimum thickness of the material that can be achieved with the machining process and affect the accuracy of the final part with respect to the original designed dimensions.

This document provides guidance for evaluating the effects of machining on ceramic materials, the dimensional changes occurring after crystallization and after sintering, and assessing machining damage.

Specific qualitative and quantitative recommendations for freedom from biological hazard are not included in this document, however when assessing possible biological or toxicological hazards, reference should be made to ISO 10993-1 and ISO 7405. Basic material properties are not included in this document, however when assessing material properties, reference should be made to ISO 6872.

Dentistry — Machinable ceramic blanks

1 Scope

This document specifies test methods for machinable ceramic blanks used for the fabrication of dental fixed restorations. This document also specifies the contents of the test report.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements 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 6872, *Dentistry — Ceramic materials*

3 Terms and definitions

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

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

3.1 Materials

3.1.1
feldspathic ceramic
inorganic, non-metallic material which is predominantly a glassy material that consists of aluminum silicates with either potassium, sodium or calcium

3.1.2
polymer infiltrated ceramic
dental ceramic which is an interconnected network of a ceramic and polymer formed by infiltration of a porous ceramic network with a monomer

3.1.3
zirconia
ZrO_2
oxidized form of the metal zirconium (Zr), exhibiting three well-defined crystal structures (polymorphs or phases) that can be monoclinic, tetragonal or cubic

3.1.4
glass ceramic
material manufactured by melting a glass, cooling it to the amorphous state, forming nuclei by controlled heat treatment and then growing the nuclei into the crystalline phase(s) by a second controlled heat treatment

3.2 Properties

3.2.1
homogeneity
degree to which the density and properties are uniform throughout the entirety of the dental blank

3.2.2
shrinkage factor
volumetric or linear change in dimension during sintering of a *green blank* (3.3.1) or a *partially sintered blank* (3.3.2) as labelled with a bar code or stated in the packaging

3.2.3
warpage
degree to which sections of the *fully dense blank* (3.3.3) or *partially sintered blank* (3.3.2) has a uniform flat surface after final sintering to full density or post machining processing

3.2.4
machining damage
effect on surface and sub surface structure occurring during machining the blank to form the final part or device

3.2.5
crystallization distortion
change in dimension of the machined part due to crystallization from a glass or a partially crystallized glass ceramic to a fully crystallized glass ceramic

3.2.6
minimum machined thickness
minimum thickness that an intact part can be machined from a given blank of material

3.2.7
machinable ceramic blank
piece of material subjected to subtractive methods to remove material from the piece leaving the final desired part

3.3 Types of blanks

3.3.1
green blank
blank in which powder has been pressed or cast to form the structure

3.3.2
partially sintered blank
blank which has been subjected to heating to cause partial sintering of the blank resulting in a blank with improved mechanical properties but that is still porous and not fully dense

3.3.3
fully dense blank
blank which has been subjected to heating to cause full sintering of a ceramic powder to achieve full density such as feldspathic, leucite and glass ceramic materials

3.4 Test piece

3.4.1
merlon
free standing wall of the test piece after the milling

4 Homogeneity of partially sintered zirconia blanks

4.1 Classification

For the purposes of this document, machinable ceramic blanks shall be classified into the following types:

— type 1: green blank (3.3.1);

— type 2: partially sintered blank (3.3.2);

— type 3: fully dense blank (3.3.3).

4.2 Determination of the shrinkage factor, *d*

4.2.1 Blanks characterized by one shrinkage factor for all three dimensions in space

4.2.1.1 Bar-size test specimen — Large zirconia blanks

Blanks of this type are discs and blocks that can be used to fabricate a wide variety of crown- and bridgework, mostly covering multiple units up to full arches (if indicated by the manufacturer for the provided zirconia material).

Mill five bar-size specimens with the following dimensions, w_1, b_1 and l_1, out of the original blank (type 2) using the same thickness (e.g. 18 mm):

— width for specimen 1, w_1 = (7,5 ± 2,5) mm;

— thickness for specimen 1, b_1 = (7,5 ± 2,5) mm;

— length for specimen 1, l_1 = (60 ± 10) mm.

Width and thickness can vary within the given limits. However, it is advised to manufacture specimen with a square cross section to further improve the reproducibility of the measured shrinkage factor.

NOTE The specimen is positioned evenly within the blank geometry (avoid milling in extreme edge locations) and does not include the surface of the blank.

Determine the exact dimensions (at least ±0,005 mm) of the milled partially sintered zirconia specimens in all three directions in space by using a calibrated micrometre screw gauge or another appropriate device accurate to at least ±0,005 mm. Repeat each measurement three times and calculate the average value for all three directions in space respectively.

Afterwards sinter all five specimens according to the manufacturer's instruction for use (including recommendations for correct sintering support of the specimen).

Determine the dimensions of the fully sintered zirconia specimens in all three directions, width, w_2, thickness, b_2, and length, l_2, in space (at least ±0,005 mm) by using the calibrated micrometre screw gauge or another appropriate device accurate to at least ±0,005 mm to yield the following values: w_2, b_2 and l_2.

Finally, calculate the resulting shrinkage factors, *d*, for all three directions in space with an accuracy of at least at least 0,001 mm by using the following formulae:

— shrinkage factor width, $d_w = w_1/w_2$;

— shrinkage factor thickness, $d_b = b_1/b_2$;

— shrinkage factor length, $d_l = l_1/l_2$.

Calculate the average shrinkage factor, d_{av}, for each bar-size specimen by using Formula (1) for specimen 1:

$$d_{av1} = (d_{w1} + d_{b1} + d_{l1}) / 3 \quad (1)$$

Calculate the final average shrinkage factor of the large zirconia blank by averaging the individual results of all five test bars as given in Formula (2):

$$d_{av} = (d_{av1} + d_{av2} + d_{av3} + d_{av4} + d_{av5}) / 5 \quad (2)$$

Compare d_{av} to the official value stated by the manufacturer for the given blank.

An example of a resulting shrinkage factor, d_{av}, is 1,229 5. Blanks of this type are blocks and can be used to fabricate, for example, three-unit bridges (medium-size blanks) or single crowns (small-size blanks) and are usually supplied in various (block-size) rectangular geometries.

Randomly choose five partially sintered zirconia blanks of the same lot of a given geometry, determine the outer dimensions and sinter them to complete density.

Ensure that the provided energy of the sintering furnace which follows the originally supplied sintering program by the manufacturer, ensures complete sintering and guarantees elimination of all porosities within the examined blank. Details concerning the characterization of the used furnace shall be given in the final report (see 4.4).

If it is uncertain that the large zirconia block can be sintered to full density in the available furnace then fabricate smaller specimens (with the dimensions as defined in this subclause) and sinter those five specimens (one specimen per zirconia blank, five blanks overall) to complete the density by using the sintering program as provided by the manufacturer. Always apply appropriate sintering support of the specimen according to the manufacturer's recommendation.

If the characterized zirconia blank does not allow the fabrication of test specimen with the dimensions, w_1, b_1 and l_1, (because the outer dimensions of the blank are too small), the manufacturer may modify the dimensions of the test specimen as follows:

— w_1 = (7,5 ± 2,5) mm;

— b_1 = (7,5 ± 2,5) mm;

— $l_1 \geq 2 \times w_1$ (or $\geq 2 \times b_1$, whichever is larger).

Width and thickness may vary within the given limits. However, it is advised to manufacture the specimen with a square cross section to further improve the reproducibility of the measured shrinkage factor.

The dimensions of these individual test specimens shall be reported (before and after sintering, see 4.4).

Finally, calculate the resulting five shrinkage factors with an accuracy of at least ±0,005 mm following the routine and formulae and compare to the values stated by the manufacturer for those five individual zirconia blanks.

4.2.1.2 Cubic test specimen

Mill five cubic specimens each with the dimension 10 mm × 10 mm × 10 mm out of the originally partially sintered blank using a common thickness (e.g. 18 mm).

Determine the exact dimensions (at least ±0,005 mm) of the milled partially sintered zirconia specimens in all three directions in space by using a calibrated micrometer screw gauge or another appropriate device accurate to at least ±0,005 mm. Repeat each measurement three times and calculate the average value for all three directions in space respectively.

Afterwards, sinter all five specimens to complete the density according to the sintering program provided by the manufacturer in the official instruction for use (including recommendations for correct sintering support of the specimen). If necessary, adjust the sintering program slightly to ensure complete elimination of any residual porosity.

Determine the volume before sintering (v_{BS}) and after sintering (v_{AS}) for each individual cube. The shrinkage factor for each specimen (d_V) is the volume before sintering determined as given in Formula (3) (here, it is given for cube 1):

$$d_{V1} = (v_{BS1} / v_{AS1})^{1/3} \quad (3)$$

where

d_{V1} is the shrinkage factor for specimen 1;

v_{BS1} is the volume before sintering for specimen 1;

v_{AS1} is the volume after sintering for specimen 1.

Calculate the resulting five individual shrinkage-factors of the cubes with an accuracy of at least ±0,005 mm.

Calculate the final average shrinkage factor of the large zirconia blank by averaging the individual results of all five test cubes as given in Formula (4):

$$d_V = (d_{V1} + d_{V2} + d_{V3} + d_{V4} + d_{V5}) / 5 \quad (4)$$

Compare d_v to the official value stated by the manufacturer for the given blank. An example of a resulting shrinkage factor d_V is, for example, 1,229 5.

4.2.1.3 Medium and small-size zirconia blanks

Prepare specimens by sectioning five randomly selected blanks into cubes 10 mm × 10 mm × 10 mm. Mark each cube for the *x*, *y* and *z* sides. Determine the exact dimensions (at least ±0,005 mm) of the milled partially sintered zirconia specimens in all three directions in space by using a calibrated micrometre screw gauge or another appropriate device accurate to at least ±0,005 mm. Repeat each measurement three times and calculate the average value for all three directions in space respectively.

Afterwards, sinter all five specimens to complete density according to the sintering program provided by the manufacturer in the official instruction for use (including recommendations for correct sintering support of the specimen). If necessary, adjust the sintering program slightly to ensure complete elimination of any residual porosity.

Determine the volume before sintering (v_{BS}) and after sintering (v_{AS}). The shrinkage factor (d_V) is determined as given in Formula (5) for each cube:

$$d_V = (v_{BS}/v_{AS})^{1/3} \quad (5)$$

Calculate the resulting five shrinkage factors with an accuracy of at least ±0,005 mm and compare to the values stated by the manufacturer for those five individual zirconia blanks.

4.2.2 Blanks characterized by two or three shrinkage factors

For discs, when shrinkage factors are indicated by the manufacturer for *x*, *y*, and *z* direction, the shrinkage factor shall be measured in each direction (bar-size specimen) or 4.2.1.2 (cubic specimen). It shall be ensured, that the x-y-z direction of the milled test specimen correctly reflects the *x*-*y*-*z* direction defined by the manufacturer.

For each of the five specimens milled per large blank, the following individual shrinkage factors per specimen, d_{x1}, d_{y1} and d_{z1}, result [here, it is given for specimen 1, length determined before sintering (BS) and after sintering (AS)]:

— d_{x1} shrinkage factor x direction specimen 1, x_{1BS}/x_{1AS};

— d_{y1} shrinkage factor y direction specimen 1, y_{1BS}/y_{1AS};

— d_{z1} shrinkage factor z direction specimen 1, z_{1BS}/z_{1AS}.

Calculate the resulting average shrinkage factor in x, y and z directions with an accuracy of at least ±0,005 mm by averaging all five specimens and applying Formula (6) (here, it is given for x direction):

$$d_x = (d_{x1} + d_{x2} + d_{x3} + d_{x4} + d_{x15}) / 5 \quad (6)$$

Compare d_x, d_y and d_z to the official value stated by the manufacturer for the given blank.

For blocks, when shrinkage factors are indicated by the manufacturer for x, y, and z directions, the shrinkage factor shall be measured in each direction by applying a rectangular geometry (as described in 4.2.1.1) or a cubic test geometry (see 4.2.1.2).

For each of the five individual specimens (that are independent of each other), the following shrinkage factors, d_{x1}, d_{y1} and d_{z1}, result [here, it is given for specimen 1, length determined before sintering (BS) and after sintering (AS)]:

— d_{x1} shrinkage factor x direction specimen 1, x_{1BS}/x_{1AS};

— d_{y1} shrinkage factor y direction specimen 1, y_{1BS}/y_{1AS};

— d_{z1} shrinkage factor z direction specimen 1, z_{1BS}/z_{1AS}.

Calculate the resulting shrinkage factors in x, y and z directions for each specimen with an accuracy of at least ±0,005 mm after the comma and compare to the values given by the manufacturer for each of the five individual blanks.

4.3 Recommendations

If the assortment of a manufacturer comprises different sizes of partially sintered zirconia blanks as defined above [large (L), medium (M), small (S)], a characterization of at least one blank geometry per type of blank (L/M/S) shall be performed.

4.4 Test report

The documentation of the test shall include at least the following information:

a) brand name, name of manufacturer, colour (if applicable), type of zirconia (if applicable), other characteristics;

b) size of characterized zirconia blank (L, M or S, if applicable);

c) lot number of each characterized blank;

d) outer dimensions of an original (partially sintered) blank as provided by the manufacturer;

e) dimensions of partially sintered test specimen used to determine shrinkage factor, either original blank or milled specimen;

f) dimensions of test specimen after sintering;

g) resulting shrinkage factors for large zirconia blanks (if applicable): d_{av}, d_{av1}, d_{av2}, d_{av3}, d_{av4}, d_{av5} (specimen one to five milled out of the same large zirconia blank — see 4.2.1.1);

h) resulting shrinkage factors for medium- and small-sized zirconia blanks (if applicable): d_{av1}, d_{av2}, d_{av3}, d_{av4}, d_{av5} (specimen one to five resulting from five different medium- or small-sized zirconia blanks — see 4.2.1.3);

i) for large zirconia blanks, report comparison of d_{av} to the official shrinkage factor provided by the manufacturer for the characterized blank(s);

j) for medium- and small-sized zirconia blanks, report the comparison of d_{av}, d_{av1}, d_{av2}, d_{av3}, d_{av4}, d_{av5} to the official shrinkage factors provided by the manufacturer for each of the five characterized blanks;

k) for blanks determined using the cube method, report d_V, d_{V1}, d_{V2}, d_{V3}, d_{V4}, d_{V5} and compare them to the official value reported by the manufacturer;

l) for blanks with shrinkage values reported by the manufacturer in *x*, *y*, and *z* directions, report the specimen shrinkage values for each direction $d_{x1\text{-}5}$, $d_{y1\text{-}5}$, $d_{z1\text{-}5}$, report the comparison of the average values, d_{avx}, d_{avy}, d_{av}, to the official shrinkage factor provided by the manufacturer for the characterized blank(s);

m) details concerning the characterization of the furnace used for firing of the specimens (e.g. manufacturer, brand name, applied sintering program, used sintering support);

n) characterization (e.g. manufacturer, brand name, accuracy) of the micrometer gauge or another appropriate device used to perform all necessary length measurements;

o) International Standard used (including its year of publication);

p) method of fabrication and measurement of test specimens;

q) any deviation from recommended test procedure and unusual features observed;

r) date of test.

5 Warpage

5.1 Test method

5.1.1 Large zirconia blanks

Blanks of this type (diameter of 98 mm ± 10 mm, thickness of at least 10 mm) can be used to fabricate a wide variety of crown and bridgework, mostly covering multiple units up to full arches (if indicated by the manufacturer for the provided zirconia material).

Mill five bar-size specimens out of the original blank according to the following dimensions:

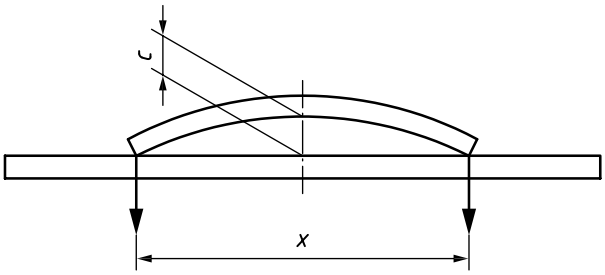
— width: (10 ± 1,0) mm;

— thickness: (2 ± 0,5) mm;

— length: (70 ± 2,0) mm.

Afterwards, sinter all five specimens to complete the density.

An engineer's parallel block metallic ruler is then placed in alignment with all four sides of the fully sintered ceramic bar in such a way that (in case a warpage occurs) the two ends of the bar touch the ruler, whereas the "middle section" shows no contact to the block (see Figure 1).



Key

x difference of the length between two ends of the ceramic bar on the ruler

c clearance between ruler and maximum point of warpage

Figure 1 — Scheme of warpage, *e*

Determine the values of "*c*" and "*x*" as defined in Figure 1 by using a calibrated micrometer screw gauge or another appropriate device with an accuracy of at least ±0,005 mm. Alternatively, use an optical microscope or laser scanning methods.

Calculate the warpage, *e*, in percentage by applying the following formula (here, it is given for specimen 1):

$$e_1 = (c_1 / x_1) \times 100$$

Calculate the final warpage by averaging the individual results of all five test bars as given in Formula (7):

$$e_{av} = (e_1 + e_2 + e_3 + e_4 + e_5) / 5 \quad (7)$$

5.1.2 Medium- and small-size zirconia blanks

Blanks of this type can be used to fabricate, for example, three-unit bridges (medium-size blanks) or single crowns (small-size blanks) and are usually supplied in various (block-size) rectangular geometries.

Mill five bar-size specimens out of the original blank according to the following dimensions:

— *w* is width, (6,0 ± 0,5) mm;

— *b* is thickness, (2,0 ± 0,2) mm;

— *l* is length, 12 mm to 35 mm.

Afterwards, sinter all five specimens to complete density.

Determine warpage for each of the five individual test specimens (e_1, e_2, e_3, e_4, e_5) as described in 5.1.1.

5.2 Recommendations

If the assortment of a manufacturer comprises different sizes of partially sintered zirconia blanks as defined above [Large (L), Medium (M), Small (S)], a characterization of at least one blank geometry per type of blank (L/M/S) shall be performed.

5.3 Test report

The documentation of the test shall include at least the following information:

a) brand name, name of manufacturer, colour (if applicable), type of zirconia (if applicable), other characteristics;

b) size of characterized zirconia blank (L, M or S, if applicable);

c) lot number of each characterized blank;

d) outer dimensions of original partially sintered (type 2) blank as provided by the manufacturer;

e) description (dimensions) of partially sintered test specimen used to determine warpage (either original blank or milled specimen as described in 5.1.1 and 5.1.2);

f) resulting warpage (of fully sintered test specimen);

g) characterization of the furnace used to perform the sintering (e.g. manufacturer, brand name, applied sintering program, used sintering support);

h) characterization (e.g. manufacturer, brand name, accuracy) of the micrometer gauge or another appropriate device used to perform all necessary length- (or distance-) measurements;

i) International Standard used (including its year of publication);

j) method of fabrication and measurement of test specimens;

k) any deviation from recommended test procedure and unusual features observed;

l) date of test.

6 Dimensional stability post machining crystallization of glass ceramics

6.1 General

Many dental glass ceramic materials are subject to one or more heat treatment steps after the milling process. Although no sintering shrinkage is to be expected, a small shrinkage can occur because of crystallization distortion or relaxation processes. In addition, during heat treatment a softening of the glassy matrix can lead to geometrical deformation. The test is to ensure that the shrinkage and deformation is within tolerable limits.

6.2 Test method

6.2.1 Sample preparation

For each test, mill five rectangular specimens out of five original blocks from the same lot (one specimen per block). Specimens can also be prepared by saw cutting. If different colours are available, select the colour most commonly used. If there are different block sizes available, the biggest block size shall be used for the test.

The milled bar-size specimens shall have the following dimensions:

— w = (6,0 ± 0,5) mm;

— b = (2,0 ± 0,2) mm;

— l = 12 mm to 35 mm.

The length shall be as long as is practical for the given block size. A typical value for a crown block is 14 mm. A typical length for a bridge block is 30 mm.

Width and thickness can vary within the given limits. However, the dimensions shall be constant over the length so that the sample can serve as a reference before heat treatment. That means the width and the thickness shall not vary by more than 0,050 mm over the length.

Determine the dimensions and the flatness of each specimen before and after heat treatment. To prevent deformation of a glass ceramic, the firing cycle and the temperature homogeneity of the furnace is of great importance. Therefore, the firing cycle, the firing tray and the supporting structure shall be in accordance with the manufacturer's recommendation. The heat treatment of the specimens shall be performed at the highest recommended temperature. If multiple heat treatments are recommended, the highest number of heat treatments shall be performed.

6.2.2 Characterization before heat treatment

6.2.2.1 Dimensions

Determine the exact dimensions (at least ±0,005 mm) of the milled glass ceramic specimens in three directions in space by using a calibrated micrometer screw gauge or another appropriate device.

Repeat each measurement three times. Measure thickness, length and width on both ends and in the middle of the bar. Calculate the mean value and the difference between the maximum and the minimum value for all three directions respectively to get the following values:

— w_m is mean width;

— b_m is mean thickness;

— l_m is mean length;

— Δw_m is the difference between the maximum value and the minimum value widths;

— Δb_m is the difference between maximum value and the minimum value thickness;

— Δl_m is the difference between maximum value and the minimum value length.

The difference between maximum and minimum Δw_m, Δb_m and Δl_m ideally shall be less than 0,050 mm. Discard and replace all specimens that do not meet dimensional requirements.

6.2.2.2 Flatness

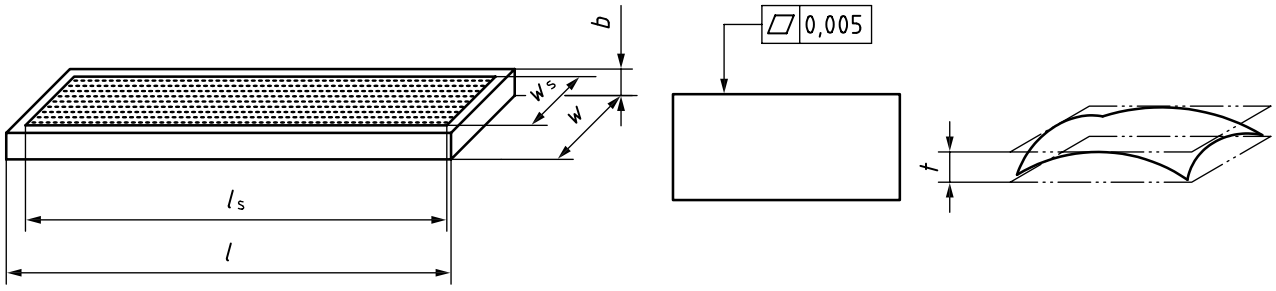
Flatness is defined as the smallest distance between two parallel planes which envelop the surface.

Measure the flatness t_0 of the original specimen on one marked side of the bar [Figure 2a)] by the optical scan method. Scan the surface and measure using a scan of 50 points × 50 points (50 lines with 50 measuring dots each). Scan the whole surface, but exclude a small rim of less than 0,5 mm. Fit the 3D raw data by least square method and run a Gaussian low pass filter to remove the roughness effects.

The surface shall be flat within 0,005 mm [Figure 2b)], so that the sample can serve as a reference. Discard all other specimens. Optical measuring system specifications are an *xy* direction measuring range of 150 mm × 100 mm, a *z* direction measuring range of 600 µm, a lateral resolution of 2 µm, and vertical resolution of 6 nm.

NOTE An example of an automatic optical measuring equipment is MicroProf® 100/200(FRT GmbH)[1)].

1) MicroProf® 100/200(FRT GmbH) is an example of a suitable product available commercially. This information is given for the convenience of users of this document and does not constitute an endorsement by ISO of this product.



a) Specimen for flatness determination **b) Illustration of the definition of flatness**

Key

w	width	l_s	scanned length after heat treatment
w_s	scanned width after heat treatment	b	thickness
l	length	t	flatness

Figure 2 — Specimen dimensions for flatness determination and flatness definition

6.2.3 Heat treatment

Apply heat treatment to all five specimens according to the sintering program provided by the manufacturer in the official instruction for use (including recommendations for correct support of the specimen). It is advised to stand the specimen on its long narrow side. This will reduce the influence of gravity. After crystallization, steam clean the specimens or clean in an ultrasonic bath. Let the specimens cool down to room temperature before measuring the final dimensions.

6.2.4 Characterization after heat treatment

6.2.4.1 Shrinkage factor

After heat treatment determine the exact dimensions (at least ±0,005 mm) of the milled glass ceramic specimens in three directions in space by using a calibrated micrometer screw gauge or another appropriate device.

Repeat each measurement three times. Measure thickness, length and width on both ends and in the middle of the bar. Calculate the average for all three directions respectively to get the following values:

— w_h is mean width for heat treatment;

— b_h is mean thickness for heat treatment;

— l_h is mean length for heat treatment.

Finally calculate the resulting shrinkage factors, d, for all three directions, d_w, d_b, and d_l, in space with an accuracy of at least ±0,005 mm by using the following formulae:

— d_w is shrinkage factor width, w_m/w_h;

— d_b is shrinkage factor thickness, b_m/b_h;

— d_l is shrinkage factor length, l_m/l_h.

Calculate the average shrinkage factor for each bar-size specimen by using the following formula (here, it is given for specimen 1) according to Formula (8):

$$d_{av1} = (d_{w1} + d_{b1} + d_{l1}) / 3 \quad (8)$$

Calculate the final average shrinkage factor of the glass ceramic by averaging the individual results of all five test bars according to Formula (9):

$$d_{av} = (d_{av1} + d_{av2} + d_{av3} + d_{av4} + d_{av5}) / 5 \quad (9)$$

EXAMPLE The resulting shrinkage factor d_{av} is less than 1,002. A shrinkage factor which is higher than 1,002 is relevant for the fit and is considered in the milling software as enlargement factor for the digital model.

It is also possible to measure the shrinkage factor by measuring the density after milling r_m and after heat treatment r_h. The density can be measured using the buoyancy method or with the pycnometer method. The linear shrinkage factor d can be calculated from the density values according to Formula (10):

$$d = r_h / r_m^{(1/3)} \quad (10)$$

6.2.4.2 Dimensional stability

Measure the flatness t_h of the marked side of the bar in the same way as described in 6.2.2.2 for the original (milled) specimen.

Calculate the dimensional stability d_s in relation to the length of the diagonal of the scanned surface.

$$d_{st} = t_h / (w_s^2 + l_s^2)^{0,5}$$

where

d_s is the dimensional stability;

t_h is the flatness of milled specimen after heat treatment;

w_s is the scanned width after heat treatment (see Figure 2);

l_s is the scanned length after heat treatment (see Figure 2).

Measure and calculate d_{st} for all five specimens ($d_{st1} - d_{st5}$).

Calculate the average dimensional stability of the five specimens according to Formula (11):

$$d_{s,av} = (d_{s,av1} + d_{s,av2} + d_{s,av3} + d_{s,av4} + d_{s,av5}) / 5 \quad (11)$$

Round down the average to the nearest 0,1 %.

The original (milled) surface is not ideally flat. To prevent an overestimation of post-machining distortion always round down to the nearest 0,1 %. For example, the resulting average dimensional stability is 0,18 %. This value can be rounded down to 0,1 %.

6.3 Test report

The documentation of the test shall include at least the following information:

a) name of manufacturer, product brand name, shade – if appropriate, block size, lot or batch identification;

b) dimensions of milled test specimens used to determine shrinkage factor and flatness;

c) type of the furnace used for heat treatment (manufacturer's name, furnace type, date of calibration certificate);

d) information for the heat treatment of the test specimen (details and number of firing cycles performed, orientation of the specimens, type of sintering support);

e) characterization (manufacturer, brand name, accuracy, etc.) of the micrometer gauge or another appropriate device used to perform all necessary length measurements;

f) detailed description of the method used for measuring flatness (test equipment, software if applicable, number of measuring points, precision, ...);

g) resulting shrinkage factors for the individual test specimen: d_{av1}, d_{av2}, d_{av3}, d_{av4}, d_{av5} and mean value: d_{av};

h) resulting dimensional stability for the individual test specimen: $d_{s,av1}$, $d_{s,av2}$, $d_{s,av3}$, $d_{s,av4}$, $d_{s,av5}$ and mean value: $d_{s,av}$;

i) International Standard used (including its year of publication);

j) any deviation from the recommended test procedure and unusual features observed;

k) date of test.

7 Machining damage

7.1 General

Machining can have an influence on the flexural strength of a certain material. This method was designed to test the performance of an entire system consisting of material, machine, tools, CAM-strategy and cutting parameters. It is not suitable to compare the quality of different machines, tools or materials alone. Due to the high influence of the used CAM strategy, the CAM strategy needs to be adapted to the geometries of the specimens.

7.2 Test methods

Blanks can vary greatly in size depending upon the intended use. Blanks for crowns and three-unit bridges typically range from 14 mm × 14 mm × 12 mm up to 20 mm × 20 mm × 40 mm. These are fully dense but can require post-processing, such as crystallization for glass ceramic materials or sintering for porous materials.

This test is only for fully dense blanks (type 3) such as feldspathic ceramics, polymer infiltrated ceramics, and glass ceramics. Prepare 15 control bar test specimens for three-point flexural strength testing as per ISO 6872 using a diamond sectioning device. Grind or mill a minimum of 15 flexural test specimens from one or more fully dense blank(s) using the designated machining apparatus approved by the manufacturer for fabricating final restorations. Perform post-machining processing such as full crystallization, as needed. The designated machining apparatus approved by the manufacturer shall be described in the instruction for use of the machinable ceramic blanks.

Place the ground or milled surface in maximum tension during flexural strength testing. Test according to ISO 6872. Calculate the mean and standard deviation of the flexural strength values for control and machined specimens.

7.3 Test report

The documentation of the test shall include at least the following information:

a) name of manufacturer, brand name, shade – if applicable;

b) what, if any post processing was used and the conditions of post-processing such as crystallization cycle;

c) size of the blank(s);

d) lot number of the blank(s);

e) fabrication conditions of the control specimens including sectioning methodology and finishing of the surface;

f) length, width, and height of the bar test specimens;

g) number of specimens which cannot be tested due to the machining failure;

h) characterization (e.g. manufacturer, brand name, accuracy) of the micrometer gauge or another appropriate device used to perform all necessary dimension measurements;

i) characterization (e.g. manufacturer, brand name, accuracy) of the milling machine used to fabricate the specimens as a well as machining conditions (tool type, bur grit, machining strategy, cutting parameters such as feed rate, if known) and software (manufacturer and version) used for machining;

j) instruments (e.g. manufacturer, brand name, accuracy) used for mechanical testing and conditions of the test (such as crosshead speed, load cell);

k) flexural strength values of each specimen as well as mean and standard deviation of each group — control and machined;

l) percentage change in flexural strength of the machined group as compared to the control group;

m) appropriate statistical analysis to determine significant difference;

n) International Standard used (including its year of publication);

o) any deviation from the recommended test procedure and unusual features observed;

p) date of test.

8 Machinability using the merlon fracture test

8.1 General

Various process parameters need to be compatible to a blank material that is being used to produce a dental restoration. Machine type, CAM setup, machining parameters, and tool quality, amongst others, can have a high influence on the outcome of a grinding or milling process. The herein described merlon fracture test provides further information on machining damage (see 7.1) and minimum machined thickness of one specific material-process combination by using standard merlon test geometries with a variation in wall thickness. Fractured merlons indicate that the applied machining process damages the blank material in a critical manner when a specific wall thickness was chosen.

This method was designed to test the performance of an entire system consisting of material, machine, tools, CAM-strategy and cutting parameters. It is not suitable to compare the quality of different machines, tools or materials alone. Due to the high influence of the used CAM strategy, the CAM strategy needs to be adapted to the geometries of the specimens.

The merlon fracture test can be applied to both fully dense (type 3) and partly sintered blanks (type 2).