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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:

— $w_1 = (7{,}5 \pm 2{,}5)$ mm;

— $b_1 = (7{,}5 \pm 2{,}5)$ 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):

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

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):

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

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:

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

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.