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ISO 13356

Third edition
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Implants for surgery — Ceramic materials based on yttria-stabilized tetragonal zirconia (Y-TZP)

Implants chirurgicaux — Produits céramiques à base de zircone tétragonal stabilisée à l'yttrium (Y-TZP)

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Foreword

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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 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

This third edition cancels and replaces the second edition (ISO 13356:2008), which has been technically revised.

Introduction

No known surgical implant material has ever been found to cause absolutely no adverse reactions in the human body. However, long-term clinical experience regarding the use of the material referred to in this International Standard has shown that an acceptable level of biological response can be expected if the material will be used in appropriate applications.

Implants for surgery — Ceramic materials based on yttria-stabilized tetragonal zirconia (Y-TZP)

1 Scope

This International Standard specifies the requirements and corresponding test methods for a biocompatible and biostable ceramic bone-substitute material based on yttria-stabilized tetragonal zirconia (yttria tetragonal zirconia polycrystal, Y-TZP) for use as a material for surgical implants.

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 3310-1, *Test sieves — Technical requirements and testing — Part 1: Test sieves of metal wire cloth*

ISO 3611, *Geometrical product specifications (GPS) — Dimensional measuring equipment: Micrometers for external measurements — Design and metrological characteristics*

ISO 7500-1, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

ISO 13383-1, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Microstructural characterization — Part 1: Determination of grain size and size distribution*

ISO 14704, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for flexural strength of monolithic ceramics at room temperature*

ISO 14705, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for hardness of monolithic ceramics at room temperature*

ISO 17561, *Fine ceramics (advanced ceramics, advanced technical ceramics) - Test method for elastic moduli of monolithic ceramics at room temperature by sonic resonance*

ISO 18754, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of density and apparent porosity*

ISO 20501, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Weibull statistics for strength data*

ISO 22214, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for cyclic bending fatigue of monolithic ceramics at room temperature*

EN 623-2, *Advanced technical ceramics — Monolithic ceramics — General and textural properties — Part 2: Determination of density and porosity*

EN 843-2, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 2: Determination of Young's modulus, shear modulus and Poisson's ratio*

EN 843-4, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 4: Vickers, Knoop and Rockwell superficial hardness*

EN 843-5, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 5: Statistical analysis*

ASTM C1161, *Standard Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature*

ASTM C1198, *Standard Test Method for Dynamic Young's Modulus, Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Sonic Resonance*

ASTM C1239, *Standard Practice for Reporting Uniaxial Strength Data and Estimating Weibull Distribution Parameters for Advanced Ceramics*

ASTM C1259, *Standard Test method for Dynamic Young's Modulus. Shear Modulus, and Poisson's Ratio for Advanced Ceramics by Impulse Excitation of Vibration*

ASTM C1327, *Standard Test Method for Vickers Indentation Hardness of Advanced Ceramics*

ASTM C1331, *Standard Test Method for Measuring Ultrasonic Velocity in Advanced Ceramics with Broadband Pulse-Echo Cross-Correlation Method*

ASTM E112, *Standard Test Method for Determining Average Grain Size*

3 Physical and chemical properties

The physical and chemical properties, when tested as specified in Clause 4, shall comply with the values specified in Table 1.

3.1 Test category

3.1.1 General

The required tests are divided into two categories.

3.1.2 Category 1

The following test shall be performed for periodical production control:

a) bulk density;

b) chemical composition;

c) microstructure;

d) strength (including Weibull modulus);

e) accelerated aging (monoclinic fraction).

3.1.3 Category 2

The manufacturer shall define the general materials specification. In addition to all tests in 3.1.2, the following tests shall be performed to demonstrate compliance with the material specification:

a) hardness;

b) Young's modulus;

c) fatigue strength;

d) accelerated aging (strength);

e) quantity of monoclinic phase;

f) radioactivity.

Table 1 — Limits for material properties

Property	Unit	Test category	Requirement	Reference	Subclause
Bulk density	g/cm^3	1	≥ 6,00	ISO 18754 EN 623–2	4.2
Chemical composition					4.3
ZrO_2+HfO_2+Y_2O_3[a]	% mass fraction	1	≥ 99,0		
Y_2O_3			> 4,5 to ≤ 6,0		
HfO_2			≤ 5		
Al_2O_3			≤ 0,5		
Other oxides			≤ 0,5		
Microstructure					4.4
Grain size	µm	1	Intercept distance ≤ 0,4 Standard deviation < 0,2	ISO 13383-1 ASTM E112	
Amount of monoclinic phase	% molar fraction	2	≤ 20	See 4.4.3	
Strength: alternative of 1) or 2)					
1a) Biaxial flexure[a]	MPa	1	≥ 500	ASTM C1499	4.5
1b) Weibull modulus		1	≥ 8	ISO 20501 EN 843–5 ASTM C1239	4.7
2a) 4-point bending[a]		1	≥ 800	ISO 14704 EN 843–1 ASTM C1161	4.6
2b) Weibull modulus		1	≥ 8	ISO 20501 EN 843–5 ASTM C 1239	4.7
Young's modulus	GPa	2	≥ 200	ISO 17561 EN 843–2 ASTM C1198 ASTM C1259 ASTM C1331	4.8
Hardness	GPa	2	≥ 11,8	ISO 14705 EN 843–4 ASTM C1327	4.9
Cyclic fatigue limit stress at 10^6 cycles	MPa	2	≥ 320	ISO 22214	4.10
Radioactivity[b]	Bq/kg	2	≤ 200	—	4.11
Accelerated aging					4.12

NOTE The number of fractional digits given for each limit value in this table indicates the appropriate number of fractional digits which should be given for the respective measured values.

[a] Measured on a minimum of 10 test specimens.

[b] The radioactivity, defined as the sum of the mass activity of U^{238}, Ra^{226}, Th^{232}, and determined by gamma spectroscopy on the ready-to-use powder should be equal or less than 200 Bq/kg. This value will be reviewed at the next revision of this International Standard and will be based upon the radioactivity data from implant ceramic manufacturers. All naturally occurring gamma emitters are to be analysed.

Table 1 *(continued)*

Property	Unit	Test category	Requirement	Reference	Subclause
Maximum amount of monoclinic phase after accelerated aging	% molar fraction	1	≤ 25	See 4.4.3	
Residual biaxial flexure strength after accelerated aging	MPa	2	≥ 500, and decrease not more than 20 %	See 4.5	
Residual 4-point bending strength after accelerated aging		2	≥ 800, and decrease not more than 20 %	See 4.6	

NOTE The number of fractional digits given for each limit value in this table indicates the appropriate number of fractional digits which should be given for the respective measured values.

a Measured on a minimum of 10 test specimens.

b The radioactivity, defined as the sum of the mass activity of U^{238}, Ra^{226}, Th^{232}, and determined by gamma spectroscopy on the ready-to-use powder should be equal or less than 200 Bq/kg. This value will be reviewed at the next revision of this International Standard and will be based upon the radioactivity data from implant ceramic manufacturers. All naturally occurring gamma emitters are to be analysed.

4 Test methods

4.1 General

All test specimens shall be prepared using the same production methods as regular implant components, including, but not limited to: the precursor powder; pressing technique; pressure; and firing conditions, unless justified by the manufacturer.

Where the requirements for the test report are not explicitly specified in this standard, the test report for a given property shall be written according to the referenced standard described in the subclause of this property and shall include a reference to this International Standard, i.e. ISO 13356:2015.

4.2 Bulk density

The bulk density shall be determined and reported in accordance with ISO 18754 or EN 623-2.

4.3 Chemical composition

The chemical compositions shall be determined by ICP-OES (Inductively Coupled Plasma – Optical Emission Spectrometry), X-ray fluorescence,[1] or atomic absorption spectrum analysis methods.

4.4 Microstructure

4.4.1 Principle

To describe the microstructure, the average grain size is determined by measuring the linear intercept size in accordance with ISO 13383-1 or ASTM E112.

Five test specimens shall be used for the determination of microstructure.

NOTE The linear intercept method results in a nominal average grain size for the selected position of micrograph, not the distribution of the size of individual grains.

For selection, preparation, and evaluation of the specimens, the following guidelines shall be followed:

a) the use of final device components as specimens for microstructure evaluation is recommended;

b) the wall thickness of the specimens shall represent the maximum and minimum of the manufacturer's device components;

c) Four micrographs on each surface shall be taken; the positions of which shall include regions in the bulk as well as at the edges of the specimen;

d) the specimen selection shall reflect the possibility of temperature deviation in the furnace;

e) the requirement for mean linear intercept grain size given in Table 1 shall be met at each selected position of the micrographs;

f) the standard deviation of the mean linear intercept grain size shall be determined from the data of all selected micrographs; the standard deviation shall meet the requirement given in Table 1.

The determination of mean linear intercept grain size shall be organized such that consistency of regular production can be assessed to a sufficient statistical relevance. The manufacturer shall justify the procedure implemented for grain size determination for its specific manufacturing process. It is recommended that the manufacturer analyse the reliability, repeatability, and maintenance of the manufacturing process with respect to microstructure (e.g. validation) and utilize this information to implement the regular routine production control. If this detailed analysis is successfully completed, regular control of the microstructure can be controlled with a reduced number of specimens and micrographs.

For improved contrast and grain boundary detection, it is recommended to use a scanning electron microscope (SEM) at a high acceleration voltage in conjunction with secondary electron detection capabilities.

4.4.2 Test report

The test report shall be prepared in accordance with ISO 13383-1 or ASTM E112 whichever is applicable.

The test report shall contain at least the following information:

a) identity of the ceramic material, details of batch number or other codes sufficient to identify the test specimens uniquely;

b) method of preparation of the test specimens, including details of the grinding and polishing procedure employed to prepare the test surfaces, as well as the etching procedure;

c) mean linear intercept size and its standard deviation shall be expressed in micrometres;

d) at least one micrograph from the specimen taken to show the microstructure after the thermal etch; the sample region from which the micrograph was taken does not have to be identified;

e) a reference to this International Standard, i.e. ISO 13356.

4.4.3 Amount of monoclinic phase

The X-ray measurement shall be conducted on the specimen prepared as described in 4.5.3 and the surface to be tested shall be in a polished state.

The amount of monoclinic phase, α, shall be determined using X-ray diffraction methods according to Formula (1):

$$\alpha = \frac{M(\bar{1}11)+M(111)}{M(\bar{1}11)+T(111)+M(111)} \quad (1)$$

where

$M(\bar{1}11)$ is the peak height of monoclinic phase at around 2θ=28,2°;