INTERNATIONAL STANDARD

ISO 13356

Second edition
2008-06-01

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

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

Reference number
ISO 13356:2008(E)

ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
Web www.iso.org

Published in Switzerland

Contents

Page

Foreword iv
Introduction v
1 Scope 1
2 Normative references 1
3 Physical and chemical properties 2
4 Test methods 3
4.1 Bulk density 3
4.2 Chemical composition 3
4.3 Microstructure 3
4.4 Biaxial flexural strength 6
4.5 Four-point bending strength 9
4.6 Cyclic fatigue 9
4.7 Radioactivity 10
4.8 Accelerated Aging Test 12
Bibliography 13

Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 2.

The main task of technical committees is to prepare International Standards. Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this document may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

ISO 13356 was prepared by Technical Committee ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

This second edition cancels and replaces the first edition (ISO 13356:1997) which has been technically revised.

Introduction

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

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

1 Scope

This International Standard specifies the characteristics of, 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 material for surgical implants.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 3611, *Micrometer callipers for external measurement*

ISO 7500-1:2004, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

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

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

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

EN 623-3, *Advanced technical ceramics — Monolithic ceramics — General and textural properties — Part 3: Determination of grain size and size distribution (characterized by the Linear Intercept Method)*

ASTM C1499, *Standard Test Method for Monotonic Equibiaxial Flexural Strength of Advanced Ceramics at Ambient Temperature*

ASTM E112-96, *Standard Test Methods for Determining Average Grain Size*

ASTM G136-03, *Standard Practice for Determination of Soluble Residual Contaminants in Materials by Ultrasonic Extraction*

ASTM F1873-98 [1)], *Standard Specification for High-Purity Dense Yttria Tetragonal Zirconium Oxide Polycrystal (Y-TZP) for Surgical Implant Applications*

1) Standard since withdrawn.

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.

Table 1 — Limits for material properties

Property	Unit	Requirement	Test method according to subclause
Bulk density	g/cm^3	⩾ 6,00	4.1
Chemical composition: $ZrO_2 + HfO_2 + Y_2O_3$ Y_2O_3 HfO_2 Al_2O_3 Other oxides	percent mass fraction	 ⩾ 99,0 > 4,5 to ⩽ 6,0 ⩽ 5 ⩽ 0,5 ⩽ 0,5	4.2
Microstructure: grain size	µm	Intercept distance ⩽ 0,4	4.3
Microstructure: amount of monoclinic phase		Standard deviation < 0,18 ⩽ 20 %	4.3.7
Strength [a]: biaxial flexure or 4-point bending	MPa	⩾ 500 ⩾ 800	4.4 4.5
Cyclic fatigue limit stress at 10^6 cycles	MPa	⩾ 320	4.6
Radioactivity [b]	Bq/kg	⩽ 200	4.7
Accelerated aging: maximum amount of monoclinic phase after accelerated aging residual biaxial flexure strength residual 4-point bending strength		 ⩽ 25 % ⩾ 500 MPa, and decrease not more than 20 % ⩾ 800 MPa, and decrease not more than 20 %	4.8

[a] Measured on a minimum of 10 samples.

[b] The radioactivity, defined as the sum of the mass activity of ^{238}U, ^{226}Ra, ^{232}Th 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.

4 Test methods

4.1 Bulk density

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

4.2 Chemical composition

The chemical compositions should be determined by ICP-OES (Inductively Coupled Plasma — Optical Emission Spectrometry), fluorescent X-ray, or atomic absorption spectrum analysis methods.

NOTE ISO 12677[1] can be used.

4.3 Microstructure

4.3.1 Principle

For describing the microstructure, the average grain size is determined by measuring the linear intercept size in accordance with EN 623-3 or ASTM E112.

4.3.2 Apparatus

The apparatus shall consist of the following items:

4.3.2.1 **Grinding and polishing devices**, for preparing plane and smooth surfaces.

4.3.2.2 **Furnace**, capable of maintaining a temperature of 1 400 °C.

4.3.2.3 **Scanning electron microscope**.

4.3.3 Preparation of test piece

Test pieces shall be prepared in accordance with EN 623-3 and the following instructions.

a) Prepare test pieces of the zirconia ceramic using methods representative of the method of production of parts for surgery, using the same precursor powder, pressing technique, pressure and firing conditions.

b) Grind one surface plane, polish it until the percentage of interpretable area is at least 90 % and thermally etch in air at a low temperature of less than 200 °C from sintering temperature. The etching conditions shall be specifically determined for each zirconia material.

c) Coat the polished surface by sputtering with a thin conductive layer, for example, gold or carbon.

NOTE A gold or a gold-platinum alloy can be used.

d) Five extra test pieces shall be made from the chosen flexure test sample. These test pieces shall be made in the same way as described for each according to 4.4.3 for the biaxial test and 4.4.5 for the 4-point bend test. The samples for microstructural analysis shall be chosen randomly from the flexure test samples.

e) Each microstructure test sample shall be cut in half. If it is a disc it shall be cut diametrically, if a bar it shall be cut lengthwise through the centre as shown in Figure 1. The piece may be sectioned further to permit it to fit into the microscope (4.3.2.3), the positions for the micrographs being those determined for the full section.

f) The surface shall then be thermally etched to produce appropriate grain relief, using typically temperatures in the range 1 300 °C to 1 400 °C for 30 min to 60 min.

g) Use

— either a low vacuum electron microscope with optics capable of discerning the microstructure on a sufficiently fine scale or

— sputter a thin conducting coating of Au, Au-Pd or C sufficient to allow for conductivity of the beam from the site

while retaining adequate feature resolution on the sample to allow analysis.

4.3.4 Procedure

Carry out the test in accordance with EN 623-3 or ASTM E112 and the following instructions.

a) Observe the microstructure using the scanning electron microscope (4.3.2.3) at a magnification sufficient to clearly delineate grain boundaries. Using either lines drawn on photomicrographs or stage movement, follow the general procedure described in EN 623-3 or ASTM E112 to measure the linear intercept sizes of at least 250 grains in total over at least three fields of view on lines sufficiently long to encompass at least 20 grains, taking random orientations of measurement. Calibrate the magnification employed using a certified graticule or grid. The micrographs should be of sufficient magnification, approximately 10 000 ×.

NOTE Alternatively a calibrated stage micrometer can be used.

b) Align the cross section or cut fraction thereof so that the side of the bottom of the part when firing is at the bottom of the micrograph.

c) Take micrographs for each of the five positions shown on the appropriate cross-section below. The micrographs should be of sufficient magnification, approximately 10 000 ×.

d) The location of the micrographs on the disc cross section on the left of Figure 1 (using the lower left hand corner as the origin of an x-y-coordinate system) are as given in Table 2 (tolerance ± 0,05 mm).

Table 2

Point	1	2	3	4	5
x	6	12	18	24	30
y	0,33	0,66	1,00	1,33	1,66

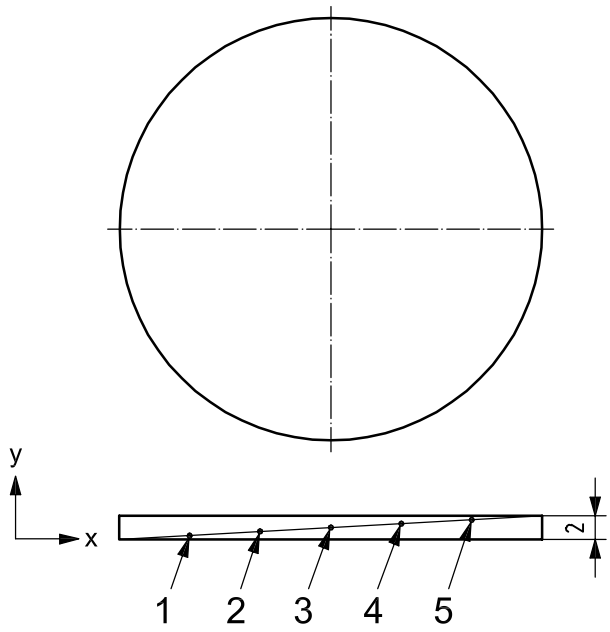
The location of the micrographs on the 4-point flexure cross section on the right of Figure 1 (using the lower left hand corner as the origin as the origin of a x-y-coordinate system) are as given in Table 3 (tolerance ± 0,05 mm).

Table 3

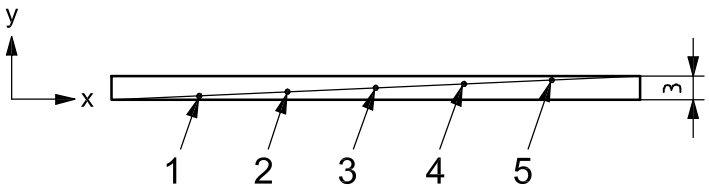
Point	1	2	3	4	5
x	7,5	15	22,5	30	37,5
y	0,5	1,0	1,5	2,0	2,5

e) For each micrograph strike no fewer than 12 lines, 4 horizontal to the diameter (bottom line), 4 at +(60 ± 5) degrees from the horizontal and 4 at –(60 ± 5) degrees from the horizontal (see Figure 1). These lines should be spread evenly across the micrograph so that as much as possible of the micrograph is represented without allowing any of the diagonal lines to be cut by the micrograph borders that are perpendicular to the diametral dimension.

f) Determine the linear intercept of each of these lines in accordance with ASTM E112 and record the intercept numbers.



a) diametral cut



b) lengthwise cut through the 45 × 4 mm surface

Figure 1 — Cuts for the cross sections of the biaxial disc (left) and 4-point flexure bar (right): bottom shows positions of micrographs to be taken

4.3.5 Calculation of results

The evaluation of the data shall be performed as follows. Determine the mean linear intercept size for each micrograph ("*micrograph mean*") in accordance with EN 623-3 or ASTM E112.

Calculate the average mean linear intercept size for test piece ("*test piece mean*") from the average of the micrograph means.

Calculate *mean value* and *standard deviation* from the 5 test piece means. These results shall be used for the test report.