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Implants for surgery — Ceramic materials —

Part 1: Ceramic materials based on high purity alumina

Implants chirurgicaux — Matériaux céramiques —

Partie 1: Matériaux céramiques à base d'alumine de haute pureté

Reference number
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ISO copyright office
CP 401 • Ch. de Blandonnet 8
CH-1214 Vernier, Geneva
Phone: +41 22 749 01 11
Fax: +41 22 749 09 47
Email: copyright@iso.org
Website: www.iso.org

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

This second edition cancels and replaces the first edition (ISO 6474-1:2010), which has been technically revised in Clause 6.

A list of all parts in the ISO 6474 series can be found on the ISO website.

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

No known surgical implant material has ever been shown to be completely free of adverse reactions in the human body. However, long-term clinical experience of use of the material referred to in the ISO 6474 series 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 —

Part 1: Ceramic materials based on high purity alumina

1 Scope

This document specifies the characteristics of, and corresponding test methods for bio-stable ceramic bone substitute material based on high purity alumina for use as bone spacers, bone replacements and components of orthopaedic joint prostheses.

This document does not cover biocompatibility (see ISO 10993-1). It is the responsibility of the manufacturer to evaluate the biocompatibility of ceramic materials which are produced within the framework of this document.

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 12677, *Chemical analysis of refractory products by X-ray fluorescence (XRF) — Fused cast-bead method*

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 15732, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test method for fracture toughness of monolithic ceramics at room temperature by single edge precracked beam (SEPB) method*

ISO 16428, *Implants for surgery — Test solutions and environmental conditions for static and dynamic corrosion tests on implantable materials and medical devices*

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 18756, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Determination of fracture toughness of monolithic ceramics at room temperature by the surface crack in flexure (SCF) method*

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*

ISO 23146, *Fine ceramics (advanced ceramics, advanced technical ceramics) — Test methods for fracture toughness of monolithic ceramics — Single-edge V-notch beam (SEVNB) method*

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)*

EN 725-1, *Advanced technical ceramics — Methods of test for ceramic powders — Part 1: Determination of impurities in alumina*

EN 843-1, *Advanced technical ceramics — Mechanical properties of monolithic ceramics at room temperature — Part 1: Determination of flexural strength*

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*

CEN/TS 14425-5, *Advanced technical ceramics — Test methods for determination of fracture toughness of monolithic ceramics — Part 5: Single-edge vee-notch beam (SEVNB) method*

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 C1421, *Standard Test Methods for Determination of Fracture Toughness of Advanced Ceramics at Ambient Temperature*

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

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

3 Terms and definitions

No terms and definitions are listed in this document.

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

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

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

4 Classification

4.1 Material types

The material shall be classified as either type A or type B.

Ceramic materials of type A are intended for implants for high load applications (e.g. bearing surfaces of joint replacements).

Type B is intended for implants for low load applications (e.g. maxillofacial and middle-ear implants).

4.2 Test categories

4.2.1 General

The required tests shall be distinguished in category 1 and category 2.

The tests in 6.6, 6.8 and 6.9 shall only be applied for type A materials.

4.2.2 Category 1: Required tests representative for the periodical production control

The following tests shall be performed for periodical production control:

a) bulk density (see 6.1);

b) chemical composition (see 6.2);

c) microstructure (see 6.3);

d) strength (see 6.4).

4.2.3 Category 2: Required tests representative for the general material specification

The manufacturer shall define the general material specification. In addition to all tests in 4.2.2, the following tests shall be performed for the qualification of the material specification:

a) Young's modulus (see 6.5);

b) fracture toughness (see 6.6);

c) hardness (see 6.7);

d) wear (see 6.8);

e) cyclic fatigue (see 6.9).

4.3 Material properties

To fulfil the requirements of this document, the material shall meet the limits for properties as given in Table 1.

Table 1 — Limits for material properties

Property	Unit	Property category	Requirement		Subclause	References
			Type A	Type B		
Average bulk density	$kg/m^3 \times 10^3$	1	≥ 3,94	≥ 3,90	6.1	ISO 18754 EN 623–2
Chemical composition:						
Basic material, Al_2O_3	% mass fraction	1	≥ 99,7	≥ 99,5		
Sintering additive, MgO	% mass fraction	1	≤ 0,2	≤ 0,2	6.2	ISO 12677 EN 725-1
Limits of impurities, total amount of $SiO_2 + CaO + Na_2O$	% mass fraction	1	≤ 0,1	≤ 0,3		
Microstructure:						
Linear intercept grain size	µm	1	≤ 2,5	≤ 3,5	6.3	ISO 13383-1 ASTM E112 EN 623-3
Relative standard deviation linear intercept grain size	%	1	≤ 25	≤ 25		
Material strength; alternatives 1) or 2):					6.4	
1a) Mean biaxial flexural strength	MPa	1	≥ 300	≥ 150	6.4.2	ASTM C1499
1b) Weibull modulus	—	1	≥ 8	≥ 8	6.4.4	ISO 20501 EN 843–5 ASTM C1239
2a) Mean 4-point flexural strength	MPa	1	≥ 500	≥ 250	6.4.3	ISO 14704 EN 843–1 ASTM C1161
2b) Weibull modulus	—	1	≥ 8	≥ 8	6.4.4	ISO 20501 EN 843–5 ASTM C1239
Young's modulus	GPa	2	≥ 380	≥ 370	6.5	ISO 17561 EN 843–2 ASTM C1331 ASTM C1198 ASTM C1259
Fracture toughness, alternatives 1) to 3)					6.6	
1) SEVNB	$MPa\sqrt{m}$	2	≥ 2,5	n.a.	6.6.2	ISO 23146 CEN/TS 14425-5
2) SEPB	$MPa\sqrt{m}$	2	≥ 2,5	n.a.	6.6.3	ISO 15732
3) SCF	$MPa\sqrt{m}$	2	≥ 2,5	n.a.	6.6.4	ISO 18756 ASTM C1421

Table 1 *(continued)*

Property	Unit	Property category	Requirement		Subclause	References
			Type A	Type B		
Hardness, Vickers HV1	GPa	2	≥ 18	≥ 17	6.7	ISO 14705 EN 843–4 ASTM C1327
Wear		2	Info	n.a.	6.8	e.g. ISO 14242-1
Cyclic fatigue: 10 million cycles endurance limit strength in 4-point bending	MPa	2	No failure at 200 MPa	n.a.	6.9	ISO 22214

5 Preparation of specimens

Specimens shall be produced equivalent to the regular production of the implants. The same feedstock, comparable shaping technology (e.g. axial pressing, isostatic pressing), high temperature process (e.g. sintering, hot isostatic pressing) and hard machining (e.g. grinding, polishing) shall be applied. The shaping of specimens shall be accomplished according to the requirements of the test.

The manufacturer shall declare and justify whether the production of the specimens can be assessed as equivalent to the regular production.

Finished products or portions of them can be used for the evaluation of material properties. However, due to geometric restrictions and the risk of damage during specimen preparation, it is not recommended to produce specimens as portions of finished products for evaluation of the following material properties:

a) strength (see 6.4);

b) fracture toughness (see 6.6);

c) cyclic fatigue (see 6.9).

6 Test methods

6.1 Bulk density

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

6.2 Chemical composition

The chemical composition shall be determined in accordance with EN 725-1 or either by X-ray fluorescence in accordance with ISO 12677 or by Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) or Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS).

The upper limits of impurities (total amount of SiO_2 + CaO + Na_2O) shall have a % mass fraction in accordance with the requirements specified in Table 1.

6.3 Microstructure

For determination of the alumina grain size, ISO 13383-1 or EN 623-3 or ASTM E112 shall be applied (linear intercept method).

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

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

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

a) the wall thickness of the selected specimens shall represent maximum and minimum of the manufacturer's products;

b) the position of the micrographs shall represent regions at the centre and at the skin of the selected specimens;

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

d) using regular products as specimens for microstructure evaluation is recommended; if other specimens are used, they shall be produced equivalent to the normal manufacturing of the products;

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

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

The determination of linear intercept grain size shall be organized such that homogeneity of the regular production can be assessed to a sufficient statistical relevance. The manufacturer shall justify the organization of grain size determination for his 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 these data for the organization of the regular production control. If this detailed analysis is accomplished successfully, the regular production control of the microstructure can be performed with a reduced number of specimens and micrographs.

6.4 Strength properties

6.4.1 General

The strength properties shall be determined using either the biaxial flexural strength test, as described in 6.4.2 or the 4-point bending strength test (see 6.4.3). A total of at least 30 specimens for each test shall be used. The data shall be analysed in accordance with Weibull statistics (see 6.4.4).

It is recommended that the surface finish which was used for the test for ease of data interpretation in terms of the product’s intended use be specified.

For an as-fired surface, specify whether the surface was made by pressing of green machining.

6.4.2 Biaxial flexural strength

The biaxial flexural strength test shall be performed in accordance with ASTM C1499. The surfaces of the specimen can be as-fired, ground or polished. Within the scope of this document, the dimensions of specimen and test rig listed in Table 2 shall be used.

Table 2 — Dimensions of biaxial flexural strength specimens and test rig

Dimension	Value mm	Tolerances mm	Abbreviation
Circular specimen diameter	36	±1,0	D
Specimen thickness	2	±0,1	h
Support ring diameter	30	±0,1	D_S
Load ring diameter	12	±0,1	D_L
Radius of contact ring	2	±0,2	r
NOTE The abbreviations are in accordance with ASTM C1499.			

6.4.3 4-point flexural strength

The 4-point flexural strength shall be determined in accordance with ISO 14704, EN 843-1 or ASTM C1161. The surfaces of the specimen shall be ground. Within the scope of this document, the dimensions of specimen and test rig listed in Table 3 shall be used.

Table 3 — Dimensions of 4-point flexural specimens and test rig

Dimension	Value mm	Tolerances mm	Abbreviation
Specimen width	4	±0,2	b
Specimen thickness	3	±0,2	d
Specimen length	≥ 45		L_T
Support span	40	±0,1	L
Loading span	20	±0,1	L_i
NOTE The abbreviations are in accordance with ISO 14704.			

6.4.4 Weibull modulus

The strength data from the biaxial flexural tests or the 4-point bending test shall be analysed in accordance with ISO 20501, EN 843-5 or ASTM C1239 using Weibull statistics. For the test report, the mean strength and the Weibull modulus shall be used. These parameters shall meet the limits given in Table 1.

6.5 Young's modulus

The Young's modulus shall be determined in accordance with ISO 17561, EN 843-2, ASTM C1331, ASTM C1198, or ASTM C1259. At least 3 test specimens shall be prepared for determination of mean value.

6.6 Fracture toughness

6.6.1 General

The fracture toughness of the material shall be determined using the SEVNB test according to 6.6.2, the SEPB test according to 6.6.3 or the SCF test according to 6.6.4. A minimum of 5 specimens for each test shall be used. The required value refers to the mean values of the test specimens.

6.6.2 SEVNB

The single edge V-notch bending test method (SEVNB) shall be used in accordance with ISO 23146 or CEN/TS 14425-5. The notch tip radius shall be minimized, preferably to less than 10 µm.

6.6.3 SEPB

The single edge precracked beam test method (SEPB) shall be used in accordance with ISO 15732.

6.6.4 SCF

The surface crack in flexure test method (SCF) shall be used in accordance with ISO 18756 or ASTM C1421.

6.7 Hardness

For the characterization of the hardness of the material, the Vickers hardness method shall be used in accordance with ISO 14705 or EN 843-4 or ASTM C1327. A test load of 9,81 N (HV1) shall be applied.

6.8 Wear

The wear behaviour of implants is a system property and not only a material property. The wear test (e.g. the hip simulator test, ISO 14242-1) should be conducted taking into consideration the intended use of the ceramic component. The test should refer to realistic application conditions of the articulating components.

In contrast to the other material properties, there is no wear limit defined within the scope of this document. The producer shall select and perform the wear test as described above and annotate the test results in comparison to the state of the art.

6.9 Cyclic fatigue

For the characterization of the cyclic fatigue behaviour of the material, the cyclic bending fatigue method shall be used in accordance with ISO 22214. The same test specimen and test jig geometry as described in 6.4.3 (4-point bending strength) shall be used.

The test conditions shall be defined as described in Table 4:

Table 4 — Test conditions for cyclic fatigue test

Test condition	Value
Environment	Physiological saline solution[a], 18 °C to 40 °C
Cyclic rate	≤ 20 Hz
σ_{max}	200 MPa
Stress ratio	0,1 ($\sigma_{min}/\sigma_{max}$)
Waveform	Sinusoidal
Test cycles	≥ 10^7
Number of specimens	≥ 5
[a] In accordance with ISO 16428.	

Bibliography

[1] ISO 10993-9, *Biological evaluation of medical devices — Part 9: Framework for identification and quantification of potential degradation products*

[2] ISO 10993-14, *Biological evaluation of medical devices — Part 14: Identification and quantification of degradation products from ceramics*

[3] ISO 14242-1, *Implants for surgery — Wear of total hip-joint prostheses — Part 1: Loading and displacement parameters for wear-testing machines and corresponding environmental conditions for test*

[4] ASTM E4-83, *Standard Practices for Force Verification of Testing Machines*

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