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Implants for surgery — Ceramic materials —

Part 2: Composite materials based on a high-purity alumina matrix with zirconia reinforcement

Implants chirurgicaux — Produits céramiques —
Partie 2: Matériaux composites à matrice alumine de haute pureté renforcée par des grains de zircone

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Contents

Page

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.

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).

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. Details of any patent rights identified during the development of the document will be in the Introduction and/or on the ISO list of patent declarations received (see www.iso.org/patents).

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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*, Subcommittee SC 1, *Materials*.

This second edition cancels and replaces the first edition (ISO 6474-2:2012) which has been technically revised.

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 found to be completely free of adverse reactions in the human body. However, long-term clinical experience of use of alumina and zirconia (the main components of the material referred to in this document) as biomaterials 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 2: Composite materials based on a high-purity alumina matrix with zirconia reinforcement

1 Scope

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

This document is intended for composite materials which are based on an alumina matrix, i.e. alumina as the dominating phase in the composite with a mass fraction of >60 %, similar to the material described in ISO 6474-1, but extended by means of a certain amount of zirconia and other defined ingredients.

NOTE The required properties in this document differ from those in ISO 6474-1 with respect to strength and fracture toughness. Furthermore, there are requirements specifically applicable for zirconia-containing materials (see ISO 13356).

In the material composition as defined in this document, additional additives are listed. Typical additives for alumina or zirconia ceramics are Mg, Y, Ce and others. Such additives can be useful in order to improve the mechanical properties and/or the chemical stability of the alumina-zirconia composite material.

This document does not cover biocompatibility (see ISO 10993-1). It is the responsibility of the manufacturer to evaluate the biocompatibility of the specific ceramic composite material which is 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 13356, *Implants for surgery — Ceramic materials based on yttria-stabilized tetragonal zirconia (Y-TZP)*

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

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*

ISO 14243-1, *Implants for surgery — Wear of total knee-joint prostheses — Part 1: Loading and displacement parameters for wear-testing machines with load control and corresponding environmental conditions for test*

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*

CEN/TS 14425-5, *Advanced technical ceramics — Test methods for determination of fracture toughness of monolithic ceramics — Part 5: 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 843-1, *Advanced technical ceramics — Monolithic ceramics — Mechanical properties 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*

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 Method 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*

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 X or Type S:

— Type X: extra-high strength;

— Type S: standard high strength.

Ceramic materials of Type X are intended for applications where extra-high strength of the material is required (e.g. thin-walled bearings for hip or knee joint replacements).

Ceramic materials of Type S are intended for applications where an improved strength in comparison to pure alumina is recommended (e.g. standard hip joint replacement).

In particular, the strengths of ceramic materials of type X and type S are higher than for materials according to type A as defined in ISO 6474-1.

4.2 Test categories

4.2.1 General

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

4.2.2 Category 1: required tests representative for 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 the tests listed in 4.2.2, the following tests shall be performed for qualification of the material specification:

a) radioactivity (see 6.5).

b) fracture toughness (see 6.6);

c) hardness (see 6.7);

d) Young's modulus (see 6.8);

e) cyclic fatigue (see 6.9);

f) accelerated ageing, including strength, cyclic fatigue and wear (see 6.10).

4.3 Material properties

To fulfil the requirements of this document, the material shall meet the limits for material properties as specified in Tables 1 and 2.

Table 1 — Limits for material property category 1

Property	Unit	Property category	Requirement		Subclause	References
			Type X	Type S		
Average relative bulk density	%	1	≥99	≥99	6.1	ISO 18754 EN 623-2
Chemical composition:						
Alumina, Al_2O_3	% mass fraction	1	60 to 90	60 to 90	6.2	ISO 12677
Zirconia, ZrO_2 + HfO_2	% mass fraction	1	10 to 30	10 to 30		
Amount of HfO_2 in ZrO_2	% mass fraction	1	≤5	≤5		
Intended additives	% mass fraction	1	≤10	≤10		
Total amount of impurities	% mass fraction	1	≤0,2	≤0,2		
Microstructure:						
Alumina linear intercept grain size	µm	1	≤1,5	≤1,5	6.3	ISO 13383-1 EN 623-3
Relative standard deviation alumina linear intercept grain size	%	1	≤25	≤25		
Zirconia linear intercept grain size	µm	1	≤0,6	≤0,6		
Relative standard deviation zirconia linear intercept grain size	%	1	≤40	≤40		
Material strength; alternative 1) or 2):					6.4	
1 a) Mean biaxial flexural strength	MPa	1	≥600	≥450	6.4.2	ASTM C1499
1 b) Weibull modulus		1	≥8	≥8	6.4.4	ISO 20501 EN 843-5 ASTM C1239
2 a) Mean 4-point flexural strength	MPa	1	≥1 000	≥750	6.4.3	ISO 14704 EN 843-1 ASTM C1161
2 b) Weibull modulus		1	≥8	≥8	6.4.4	ISO 20501 EN 843-5 ASTM C1239

Table 2 — Limits for material property category 2

Property	Unit	Property category	Requirement		Subclause	References
			Type X	Type S		
Radioactivity (measured on raw materials)						
Zirconia Other intended additives	Bq/kg	2 See 6.5	≤200	≤200	6.5	ISO 13356
Fracture toughness, alternatives 1) to 3)					6.6	
1) SEVNB	MPa $\sqrt{m}$	2	≥4,0	≥3,5	6.6.2	ISO 23146 CEN/TS 14425-5
2) SEPB	MPa $\sqrt{m}$	2	≥4,0	≥3,5	6.6.3	ISO 15732
3) SCF	MPa $\sqrt{m}$	2	≥4,0	≥3,5	6.6.4	ISO 18756 ASTM C1421
Hardness, Vickers HV1	GPa	2	≥16,0	≥15,5	6.7	ISO 14705 EN 843-4 ASTM C1327
Young's modulus	GPa	2	≥320	≥320	6.8	ISO 17561 EN 843-2 ASTM C1331 ASTM C1198 ASTM C1259
Cyclic fatigue: Cyclic loading in 4-point bending, 10^7 cycles		2	No failure at 400 MPa	No failure at 300 MPa	6.9	ISO 22214
Accelerated ageing: 10 h in autoclave (0,2 MPa, 134 °C) after autoclaving:					6.10	
Strength		2	Degradation ≤ 20 % in comparison to value before autoclaving *and* conformity with values given in Table 1		6.10.2	See 6.4
Cyclic loading in 4-point bending, 10^7 cycles		2	No failure at 320 MPa	No failure at 240 MPa	6.10.3	See 6.9
Wear		2	Increase ≤ 20 % in comparison to value before autoclaving		6.10.4	ISO 14242-1 ISO 14243-1 or other tests

5 Preparation of specimens

Specimens shall be produced in a similar way to the regular production of implants. The same feedstock and 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 and surface finishing of the specimens shall be accomplished according to the requirements of the test.

The manufacturer shall declare and justify that the production of the specimens is 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 to the risk of damage during specimen preparation, it is not