INTERNATIONAL STANDARD

ISO 6474-1

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Implants for surgery — Ceramic materials —

Part 1: Ceramic materials based on high purity alumina

Implants chirurgicaux — Produits céramiques —

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

Reference number
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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 6474-1 was prepared by Technical Committee ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

This first edition, together with ISO 6474-2, cancels and replaces ISO 6474:1994, which has been technically revised.

ISO 6474 consists of the following parts, under the general title *Implants for surgery — Ceramic materials*:

— *Part 1: Ceramic materials based on high purity alumina*

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

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 ISO 6474 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 part of ISO 6474 specifies the characteristics of, and corresponding test methods for, a bio-compatible and bio-stable ceramic bone substitute material based on high purity alumina for use as bone spacers, bone replacements and components of orthopaedic joint prostheses.

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 31 (all parts), *Quantities and units*

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 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 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 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 Classification

3.1 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).

3.2 Test categories

3.2.1 General

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

The tests in 5.6, 5.8 and 5.9 shall only be applied for type A materials.

3.2.2 Category 1: required tests representative for the periodical production control

The following tests shall be performed for periodical production control:

a) bulk density;

b) chemical composition;

c) microstructure;

d) strength.

3.2.3 Category 2: required tests representative for the general material specification

The manufacturer shall define the general material specification. In addition to all tests in 3.2.2, the following tests shall be performed for the qualification of the material specification:

a) Young's modulus;

b) fracture toughness;

c) hardness;

d) wear resistance;

e) fatigue limit.

3.3 Material properties

To fulfil the requirements of this part of ISO 6474, the material shall meet the limits for properties as given in Table 1. Documentation of the test results shall be according to the International System of Units (SI), ISO 31.

Table 1 — Limits for material properties

Property	Unit	Property category	Requirement		Subclause	References
			Type A	Type B		
Average bulk density	kg/m^3 × 10^3	1	⩾ 3,94	⩾ 3,90	5.1	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	5.2	ISO 12677
Limits of impurities, total amount of $SiO_2 + CaO + Na_2O$	% mass fraction	1	⩽ 0,1	⩽ 0,3		
Microstructure:						EN 623-3
Linear intercept grain size	µm	1	⩽ 2,5	⩽ 3,5	5.3	ASTM E112
Standard deviation	%	1	⩽ 25	⩽ 25		ISO 13356
Material strength; alternatives 1) or 2):					5.4	
1a) Mean biaxial flexural strength	MPa	1	⩾ 300	⩾ 150	5.4.2	ASTM C1499
1b) Weibull modulus	-	1	⩾ 8	⩾ 8	5.4.4	ISO 20501 EN 843-5 ASTM C1239
2a) Mean 4-point flexural strength	MPa	1	⩾ 500	⩾ 250	5.4.3	ISO 14704 EN 843-1 ASTM C1161
2b) Weibull modulus	-	1	⩾ 8	⩾ 8	5.4.4	ISO 20501 EN 843-5 ASTM C1239
Young's modulus	GPa	2	⩾ 380	⩾ 370	5.5	ISO 17561 EN 843-2 ASTM C1331 ASTM C1198 ASTM C1259
Fracture toughness, alternatives 1) to 3)					5.6	
1) SEVNB	MPa $\sqrt{m}$	2	⩾ 2,5	n.a.	5.6.2	ISO 23146 CEN/TS 14425-5
2) SEPB	MPa $\sqrt{m}$	2	⩾ 2,5	n.a.	5.6.3	ISO 15732
3) SCF	MPa $\sqrt{m}$	2	⩾ 2,5	n.a.	5.6.4	ISO 18756 ASTM C1421
Average hardness, Vickers HV1	GPa	2	⩾ 18	⩾ 17	5.7	ISO 14705 EN 843-4 ASTM C1327
Wear resistance		2	Info	n.a.	5.8	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.	5.9	ISO 22214

4 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 5.4);

b) fracture toughness (see 5.6);

c) cyclic fatigue (see 5.9).

5 Test methods

5.1 Bulk density

The bulk density shall be determined in accordance with ISO 18754.

NOTE An equivalent procedure can be found in EN 623-2.

5.2 Chemical composition

The chemical composition shall be determined either by X-ray fluorescence in accordance with ISO 12677 or by induced coupled plasma (ICP) emission spectroscopy.

5.3 Microstructure

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

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:

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

2) the position of the micrographs shall represent regions at the centre and at the surface of the selected specimens;

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

4) 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;

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

6) 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.

5.4 Determination of strength properties

5.4.1 General

The strength properties shall be determined using either the biaxial flexural strength test, as described in 5.4.2 or the 4-point bending strength test (see 5.4.3).

5.4.2 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 part of ISO 6474, 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.			

5.4.3 4-point flexural strength

The 4-point flexural strength shall be determined in accordance with ISO 14704. The surfaces of the specimen shall be ground. Within the scope of this part of ISO 6474, the dimensions of specimen and test rig listed in Table 3 shall be used.

NOTE Equivalent procedures can be found in EN 843-1 or ASTM C1161.

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.			

5.4.4 Weibull modulus

To evaluate the distribution of the material strength, the Weibull modulus shall be calculated either from the biaxial bending test results or the 4-point bending test results in accordance with ISO 20501.

NOTE Equivalent procedures can be found in EN 843-5 or ASTM C1239.

5.5 Young's modulus

The Young’s modulus shall be determined in accordance with ISO 17561.

NOTE Equivalent procedures can be found in EN 843-2, ASTM C1331, ASTM C1198 and ASTM C1259.

5.6 Fracture toughness

5.6.1 General

The fracture toughness of the material shall be determined using the SEVNB test according to 5.6.2, the SEPB test according to 5.6.3 or the SCF test according to 5.6.4.

5.6.2 SEVNB

The single edge V-notch bending test method (SEVNB) shall be used in accordance with ISO 23146 or CEN/TS 14425-5.

5.6.3 SEPB

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

5.6.4 SCF

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

5.7 Hardness

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

NOTE Equivalent procedures can be found in EN 843-4 and ASTM C1327.

5.8 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 part of ISO 6474. 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.

5.9 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 tests shall be performed under the following conditions:

- the same test specimen and test rig geometry as described in 5.4.3 (4-point bending strength);
- in a physiological saline solution at 18°C to 40°C;
- cyclic rate $\leqslant$ 20 Hz;
- stress ratio = 0,1 ($\sigma_{min}/\sigma_{max}$);
- sinusoidal waveform;
- duration of the test $\geqslant 10^7$ cycles;
- number of samples $\geqslant$ 5.

6 Test report

The test report shall contain at least the following information:

a) reference to this International Standard, i.e. ISO 6474-1:2009;

b) identity of the ceramic material, details of the batch/lot number and other codes sufficient to identify the test piece uniquely;

c) method(s) of preparing the test pieces;

d) details of the test(s) performed;

e) description of the test apparatus;

f) date(s) when the test(s) was (were) performed;

g) name(s) of the person(s) who performed the test(s);

h) reference value and actual value;

i) comments, if applicable.

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

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

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