INTERNATIONAL STANDARD

ISO 6474

Second edition
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Implants for surgery — Ceramic materials based on high purity alumina

Implants chirurgicaux — Produits céramiques à base d'alumine de haute pureté

Reference number
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Contents

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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.

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.

International Standard ISO 6474 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 6474:1981), of which it constitutes a technical revision.

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 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 high purity alumina

1 Scope

This International Standard specifies the characteristics of, and corresponding test methods for, a biocompatible 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 standards contain provisions which, through reference in this text, constitute provisions of this International Standard. At the time of publication, the editions indicated were valid. All standards are subject to revision, and parties to agreements based on this International Standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below. Members of IEC and ISO maintain registers of currently valid International Standards.

ISO 468:1982, *Surface roughness — Parameters, their values and general rules for specifying requirements.*

ISO 3611:1978, *Micrometer callipers for external measurement.*

ISO 5017:1988, *Dense shaped refractory products — Determination of bulk density, apparent porosity and true porosity.*

ISO 5436:1985, *Calibration specimens — Stylus instruments — Types, calibration and use of specimens.*

ASTM C573:1986, *Method for chemical analysis of fireclay and high-alumina refractories.*

ASTM E112:1988, *Methods for determining average grain size.*

3 Classification

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

4 Physical and chemical properties

The properties of type A and type B materials shall be as given in table 1.

5 Test methods

5.1 Bulk density

The bulk density shall be determined in accordance with ISO 5017.

5.2 Chemical composition

The chemical composition shall be determined either in accordance with ASTM C573, or by an equivalent method. In cases of dispute, the method given in ASTM C573 shall be the referee method.

Table 1 — Properties of type A and type B materials

Property	Unit	Requirement		Test method according to subclause
		Type A	Type B	
Bulk density	g/m^3	⩾ 3,94	⩾ 3,90	5.1
Chemical composition:				5.2
basic material, Al_2O_3	%	⩾ 99,5		
sintering additive, MgO	%	⩽ 0,3		
limits of impurities, total amount of SiO_2 + CaO + alkali metal oxides	%	⩽ 0,1		
Microstructure:				5.3
mean linear intercept size	µm	⩽ 4,5	⩽ 7,0	
standard deviation	µm	⩽ 2,6	⩽ 3,5	
Average biaxial flexural strength	MPa	⩾ 250	⩾ 150	5.4
Wear resistance[1)]				5.5
wear volume	mm^3	⩾ 0,1	not applicable	

1) This test applies only if articulation of ceramic on ceramic is intended.

5.3 Microstructure

5.3.1 Principle

As a means of describing the microstructure, the average grain size is determined by measuring the mean linear intercept size.

5.3.2 Apparatus

5.3.2.1 Grinding and polishing devices to prepare plane and smooth scratch-free surfaces.

5.3.2.2 Furnace capable of maintaining a temperature of 1 500 °C.

5.3.2.3 Light microscope having a magnification of × 500 to × 1 000.

NOTE 1 If the mean grain size is anticipated to be less than 2 µm, a scanning optical microscope may be preferred.

5.3.3 Preparation of test pieces

5.3.3.1 Prepare test pieces of the alumina ceramic by methods representative of the processing techniques to be used for the production of parts for surgery, using the same precursor powder, pressing method and pressure, and firing conditions.

5.3.3.2 Grind one surface of the test piece plane and polish it until the percentage of scratch-free interpretable area is at least 90 %.

5.3.3.3 Etch the test pieces thermally in air at a temperature in the range 1 400 °C to 1 500 °C for 1 h to 4 h.

To increase the optical contrast, the polished and etched surface may be sputter-coated with a thin gold layer.

5.3.4 Procedure

Observe the microstructure using the microscope at a magnification sufficient to delineate grain boundaries clearly. Using either lines drawn on photomicrographs or stage movement, follow the general procedure in ASTM E112 to measure the individual linear intercept sizes of at least 250 grains in total over at least six 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. Alternatively a calibrated stage micrometer may be used.

5.3.5 Calculation of results

Calculate the mean linear intercept size and the standard deviation from the individual linear intercept sizes.

5.3.6 Test report

The test report shall contain the following information:

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

b) the method of preparing the test pieces surfaces, including details of the grinding and polishing procedure used;

c) the mean linear intercept size and its standard deviation, expressed in micrometres.

5.4 Biaxial flexural strength

5.4.1 Principle

A disc of test material is placed between two coaxial rings of unequal diameter, and a compressive force is applied. The force at fracture of the test disc is recorded and the fracture nominal stress is calculated.

5.4.2 Apparatus

5.4.2.1 Mechanical testing machine suitable for applying a compressive load of at least 5 kN at a nominal loading rate of (500 ± 100) N·s^{-1} and equipped to record the peak force applied to an accuracy of better than 1 %.

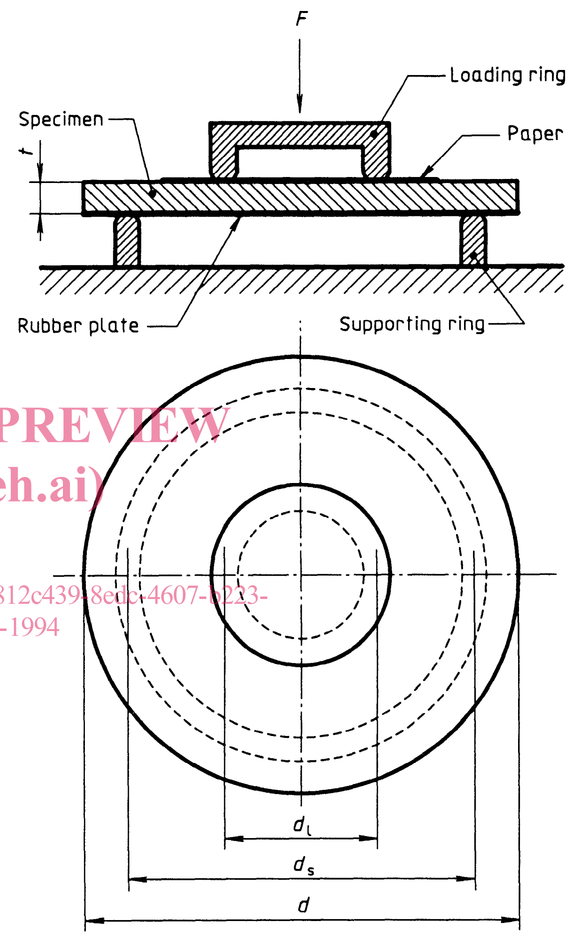
Calibration of the test machine shall be carried out according to agreed procedures, e.g. ASTM E4-83.

5.4.2.2 Test jig comprising unequal diameter loading rings and having a geometry typically as shown in figure 1. The jig shall have an outer, support ring diameter of (30 ± 0,1) mm at the diameter of contact with the test piece, and a loading ring diameter of (12 ± 0,1) mm at the diameter of contact with the test piece. The radius of curvature of the specimen contact surface shall be (2 ± 0,2) mm. The jig shall have a means of centring the loading and support rings and the test piece on a common axis to within ± 0,2 mm.

The rings should preferably be made from hardened steel (greater than 500 HV or 40 HRC) in order to minimize damage or roughness caused by the fracturing test pieces.

In order to accommodate slight departures from flatness in the surfaces of the test pieces, a rubber plate, (0,6 ± 0,1) mm thick, of 60 ± 5 Shore hardness shall be placed between the support ring and the test piece, and a piece of paper shall be placed between the test piece and the loading ring.

5.4.2.3 Micrometer in accordance with ISO 3611 capable of measuring to an accuracy of ± 0,01 mm.



See NOTE 2.

Figure 1 — Schematic diagram of biaxial flexural strength test device with concentric loading and support rings

5.4.3 Preparation of test pieces

5.4.3.1 Prepare billets or discs of the test material using methods representative of the production methods of parts for surgery, using the same precursor powder, pressing method and pressure, and firing conditions.

5.4.3.2 The test pieces (see figure 1) shall be circular plates of diameter (36 ± 1,0) mm and thickness (2 ± 0,1) mm. The surface to be tested shall be in a fired state.

5.4.3.3 At least 10 test pieces shall be prepared for determination of mean strength or at least 30 test pieces if a Weibull statistical analysis is required.

5.4.4 Procedure

NOTE 2 This test procedure can also be used for test pieces with other surface treatments not complying with 5.4.3, e.g. ground or polished. In every case the preparation method should be recorded as stated in 5.4.6 b).

5.4.4.1 Measure the diameter of the test piece to the nearest 0,1 mm and the thickness to the nearest 0,05 mm, each in at least three random positions. Calculate the mean diameter and mean thickness.

5.4.4.2 Place the rubber sheet on the support ring of the test jig. Place the test piece on the rubber sheet with the surface to be tested in contact with the rubber and centre it. Place a paper disc on the top of the test piece and place the loading ring on the paper and centre relative to the test piece and support ring.

5.4.4.3 Place the test jig in the test machine and apply a steadily increasing force to the loading ring at a rate of (500 ± 100) $N \cdot s^{-1}$ until the test piece fractures. Record the load at fracture.

5.4.4.4 Inspect the fragments for evidence of the failure origin. If this is more than 0,5 mm outside the inner loading ring, note this fact in the report (5.4.6). For the purposes of calculation of the fracture stress, assume failure within the inner loading ring. Do not discard the result in calculating the mean strength of the test batch.

5.4.4.5 Repeat the test procedure for each test piece in the batch.

5.4.5 Calculation of results

For each test piece, calculate the nominal biaxial fracture stress, σ, in megapascals, as:

$$\sigma = \frac{3F}{2\pi t^2}\left[(1+\nu)\ \ln\left(\frac{d_s}{d_l}\right) + (1-\nu)\left(\frac{d_s^2 - d_l^2}{2d^2}\right)\right]$$

where

F is the force applied at fracture, in newtons;

t is the mean test piece thickness, in millimetres;

d_s is the mean support ring contact diameter, in millimetres;

d_l is the mean loading ring contact diameter, in millimetres;

d is the mean test piece diameter, in millimetres;

ν is Poisson's ratio, which for the purposes of this test shall be taken as equal to 0,25.

Calculate the mean nominal fracture stress and the standard deviation for the batch of test pieces.

5.4.6 Test report

The test report shall contain the following information:

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

b) the method of preparing the test pieces, including details of the machining procedure used to prepare the test surfaces (see 5.4.3.1);

c) the mean value, the standard deviation and the Weibull statistical data. The position of the apparent site of fracture shall be noted if this falls more than 0,5 mm outside the loading ring diameter.

5.5 Wear resistance

5.5.1 Principle

A ring of alumina ceramic is loaded onto a flat alumina plate, and is rotated through an arc of ± 25° at a frequency of 1 Hz for a given period of time, using water as the surrounding medium. The volume of the wear track on the disc is determined and used as a measure of wear resistance for the purposes of this specification.

5.5.2 Apparatus

5.5.2.1 Ring-on-disc oscillating test device with the capability of positioning the ring test piece concentrically with respect to the disc test piece (figure 2). The ring specimen shall be able to undergo oscillatory rotation about a fixed axis by ± 25° rotation angle at a rate of 1 Hz using a sinusoidal or near-sinusoidal rate of change of angle. The disc-holding device shall include a universal joint to ensure that the plane of the disc surface coincides with the plane of the ring surface at all times.

5.5.2.2 Profilometric test device, e.g. a skidless diamond stylus instrument, to determine the wear volume removed from the disc test-piece. This device shall allow the wear track cross-sectional area to be calculated from the profile measured.

5.5.3 Preparation of test pieces

5.5.3.1 Prepare billets or discs of the test material using methods representative of the production methods of parts for surgery, using the same precursor powder, pressing method and pressure, and firing conditions. Prepare at least five pairs of test pieces.

5.5.3.2 The test pieces (see figure 3) should have the following dimensions:

Contact surface region of ring test piece:

— inner diameter ($14^{0}_{-0,1}$) mm

— outer diameter ($20^{0}_{-0,1}$) mm

Contact surface of disc test piece:

— diameter ⩾ 25 mm

Other dimensions of test pieces may however be chosen to suit the design of the test device.

Figure 2 — Schematic diagram of ring on disc wear test

Dimensions in millimetres

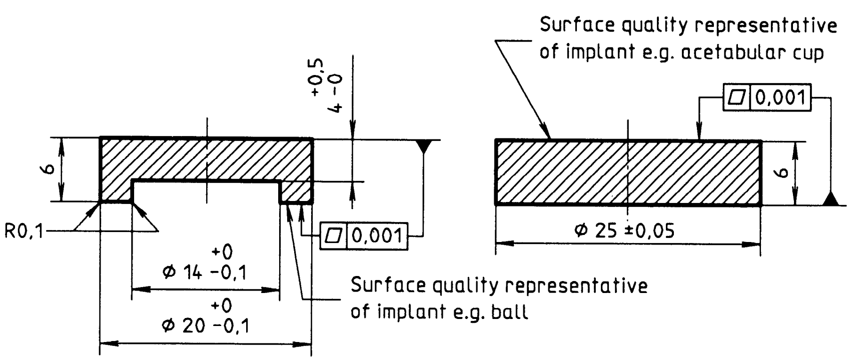


Figure 3 — Geometry of ring and disc test pieces with necessary dimensions defined