
**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 zirconie*



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Contents

Page

Foreword	iv
Introduction	v
1 Scope	1
2 Normative references	1
3 Terms and Definitions	3
4 Classification	3
4.1 Material types	3
4.2 Test categories	3
4.2.1 General	3
4.2.2 Category 1: required tests representative for periodical production control	3
4.2.3 Category 2: required tests representative for the general material specification	3
4.3 Material properties	4
5 Preparation of specimens	5
6 Test methods	6
6.1 Bulk density	6
6.1.1 General	6
6.1.2 Calculation of ultimate density	6
6.1.3 Empirical determination of the ultimate density	6
6.2 Chemical composition	7
6.3 Microstructure	7
6.4 Strength properties	8
6.4.1 General	8
6.4.2 Biaxial flexural strength	8
6.4.3 4-point flexural strength	8
6.4.4 Weibull modulus	9
6.5 Radioactivity	9
6.6 Fracture toughness	9
6.6.1 General	9
6.6.2 SEVNB	9
6.6.3 SEPB	9
6.6.4 SCF	9
6.7 Hardness	10
6.8 Young's modulus	10
6.9 Cyclic fatigue	10
6.10 Accelerated ageing	10
6.10.1 General	10
6.10.2 Strength	11
6.10.3 Cyclic fatigue	11
6.10.4 Wear	11
Bibliography	12

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).

Any trade name used in this document is information given for the convenience of users and does not constitute an endorsement.

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.

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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 ₂ O 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

recommended to produce specimens as portions of finished products for evaluation of the following material properties:

- strength (6.4);
- fracture toughness (6.6);
- cyclic fatigue (6.9).

6 Test methods

6.1 Bulk density

6.1.1 General

The bulk density shall be determined in accordance with ISO 18754 or EN 623-2. The benchmark of the bulk density depends on the selected composition of the composite. It is given as a relative density $\rho_r \geq 99\%$.

The relative density ρ_r shall be calculated in accordance with [Formula \(1\)](#):

$$\rho_r = \frac{\rho_m}{\rho_u} \quad (1)$$

where

ρ_m is the measured density, in g/cm³;

ρ_u is the ultimate density, in g/cm³.

ρ_u shall be determined either by calculation or empirically.

6.1.2 Calculation of ultimate density

For the calculation of the ultimate density ρ_u , the mass fraction and the density of each phase have to be known exactly. The ultimate density is then calculated in accordance with [Formula \(2\)](#):

$$\rho_u = \frac{100\%}{\frac{m_i}{\rho_i} + \dots + \frac{m_n}{\rho_n}} \quad (2)$$

where

ρ_i, ρ_n are the densities of the individual components (alumina, zirconia, others);

m_i, m_n are the relative mass fractions of the components, in %.

The value of $m_i + \dots + m_n$ is necessarily 100 %.

The theoretical density of each component for the application of [Formula \(2\)](#) shall be determined. Impurities can be neglected for the calculation of the theoretical density if their amount has a mass fraction of $\leq 0,2\%$.

6.1.3 Empirical determination of the ultimate density

If the ultimate density cannot be calculated to a sufficient reliability, it is recommended that the ultimate density be empirically determined in accordance with the following procedure.

- a) Choose a powder batch with a representative inorganic composition.

- b) Produce at least 10 test pieces by sintering and hot isostatic pressing. Choose the sintering and hot isostatic pressing conditions according to the experience of the manufacturer in order to achieve the highest possible density.
- c) Analyse the microstructure after this process for any evidence of residual pores.
- d) If no pores can be detected, measure the density according to ISO 18754. The outer surface of the test pieces shall be ground or polished.
- e) Round off the density of each test piece to two decimal places ($x.xx \text{ g/cm}^3$).
- f) Declare the highest value of all individual density values as the ultimate density.

6.2 Chemical composition

The chemical composition shall be determined 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).

If applicable, the manufacturer shall document all inorganic additives which are intentionally added to the composition. Upper and lower limits for these additives shall be defined by the manufacturer. The total amount of these additives is limited to a mass fraction of 10 %.

The manufacturer shall identify elements that have an adverse impact on the properties of the composite as "impurities". The upper limit of the total amount of these impurities has a mass fraction of 0,2 %.

It is recommended that the manufacturer define the upper and lower limits for the amount of alumina and zirconia which shall be measured in accordance with the definition of category 1 (see [4.2.2](#)).

6.3 Microstructure

ISO 13383-1 or EN 623-3 shall be applied for the determination of microstructure. The linear intercept grain size of the alumina grains and zirconia grains shall be determined. The grain size determination of other phases is not required.

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 the 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 in an equivalent manner to the normal manufacturing of the products;
- e) the requirement for linear intercept grain size given in [Table 1](#) shall be met 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 meet 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 amount of specimens and micrographs.

NOTE 1 ASTM E112 cannot be applied as it is not intended for composite materials.

NOTE 2 The linear intercept grain size is inevitably smaller than the genuine grain size. Further details and references to literature are given in EN 623-3.

For improved contrast and grain boundary detection of zirconia and alumina, it is recommended that a secondary electron detector in a scanning electron microscope (SEM) at a high acceleration voltage be used.

6.4 Strength properties

6.4.1 General

The strength properties shall be determined using either the biaxial flexural strength test 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 or 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 either as fired, ground or polished. Within the scope of this document, the dimensions of the specimen and test rig specified in Table 3 shall be used.

Table 3 — Dimensions of biaxial flexural strength specimens and test rig

Dimensions in millimetres

Dimension	Value	Tolerances	Abbreviation
Circular specimen diameter	36	$\pm 1,0$	<i>D</i>
Specimen thickness	2	$\pm 0,1$	<i>h</i>
Support ring diameter	30	$\pm 0,1$	<i>D_s</i>
Load ring diameter	12	$\pm 0,1$	<i>D_L</i>
Radius of contact ring	2	$\pm 0,2$	<i>r</i>
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 either ground or polished. Within the scope of this document, the dimensions of the specimen and test rig specified in Table 4 shall be used.

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

Dimensions in millimetres

Dimension	Value	Tolerances	Abbreviation
Specimen width	4	$\pm 0,2$	<i>b</i>
Specimen thickness	3	$\pm 0,2$	<i>d</i>
Specimen length	≥ 45	—	<i>L_T</i>
Support span	40	$\pm 0,1$	<i>L</i>
Loading span	20	$\pm 0,1$	<i>L_i</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 flexural tests 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 Radioactivity

The radioactivity shall be determined in accordance with ISO 13356. Other methods are also acceptable if sufficient accuracy and reliability is provided. As test specimens, the raw material powders shall be used, i.e. the materials before mixture of other ingredients. All components shall meet the limits given in [Table 2](#).

Radioactivity is particularly expected for zirconia. Other raw materials used by the manufacturers could also be expected to show radioactivity. It is thus required that the radioactivity of these raw materials be determined in accordance with the requirements of test category 2.

Alumina and most additives for technical ceramics do not show any radioactivity. For these raw materials, no analysis of radioactivity is necessary.

Reassessment of radioactivity shall be performed in case of a substantial change in the manufacturing process of raw material, such as changes in the refining process or in the selection of the primary raw material powder.

6.6 Fracture toughness

6.6.1 General

The fracture toughness of the material shall be determined using one of the methods referred to in [6.6.2](#) to [6.6.4](#). A minimum of 5 specimens for each test shall be used. The required value refers to the mean value of the test series.

6.6.2 SEVNB

The single edge V-notch bending test method (SEVNB) in accordance with ISO 23146 or CEN/TS 14425-5 shall be used. 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) in accordance with ISO 15732 shall be used.

6.6.4 SCF

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

6.7 Hardness

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

The hardness depends on the amount of zirconia and other additives in the alumina matrix. Thus, a limit is defined in [Table 2](#) which is representative for a ceramic composite with a high zirconia content. It is recommended that the manufacturer identifies a typical value and defines an appropriate lower limit for their specific composition.

6.8 Young's modulus

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.

Young's modulus depends on the amount of zirconia and other additives in the alumina matrix. Thus, a limit is defined in [Table 2](#) which is representative for a ceramic composite with a high zirconia content. It is recommended that the manufacturer identify a typical value and define an appropriate lower limit for his specific composition.

6.9 Cyclic fatigue

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

The test conditions shall be defined as described in [Table 5](#).

Table 5 — Conditions of cyclic fatigue test

Test condition	Value
Environment	Physiological saline solution ^a , 18 °C to 40 °C
Cyclic rate	≤20 Hz
$\sigma_{\max}$	See Table 2
Stress ratio	0,1 ($\sigma_{\min}/\sigma_{\max}$)
Waveform	Sinusoidal
Test cycles	≥10 ⁷
Number of specimens	≥5
^a Physiological saline solution shall be in accordance with ISO 16428.	

6.10 Accelerated ageing

6.10.1 General

This test describes the stability of the material in a hydrous environment. In particular, the test conditions simulate the interaction of zirconia with water at an elevated temperature. The test is useful for determining any material degradation due to hydrothermal ageing.

The test shall be carried out using a suitable autoclave in water vapour at (134 ± 2) °C for a period of 10 h. The autoclave used for this test shall achieve a nominal pressure of 0,2 MPa at this temperature. Specimens shall be used as described in [6.4](#), [6.9](#) and [6.10.4](#), respectively.

In order to assess the effect of material degradation due to hydrothermal ageing, the tests specified in [6.10.2](#) to [6.10.4](#) shall be performed after autoclaving.