INTERNATIONAL STANDARD

ISO 13779-4

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Implants for surgery — Hydroxyapatite —

Part 4: Determination of coating adhesion strength

Implants chirurgicaux — Hydroxyapatite —

Partie 4: Détermination de la résistance à l'adhésion du revêtement

Reference number
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ISO copyright office
Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.ch
Web www.iso.ch

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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 3.

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 part of ISO 13779 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 13779-4 was prepared by Technical Committee ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

ISO 13779 consists of the following parts, under the general title *Implants for surgery — Hydroxyapatite*:

— *Part 1: Ceramic hydroxyapatite*

— *Part 2: Coatings of hydroxyapatite*

— *Part 3: Chemical analysis and characterization of cristallinity and phase purity*

— *Part 4: Determination of coating adhesion strength*

Future parts will deal with other relevant aspects of implant material based on hydroxyapatite.

Introduction

No known surgical implant material has ever been shown to cause absolutely no adverse reactions in the human body. However, long-term clinical experience of the use of the material referred to in this part of ISO 13779 has shown that an acceptable level of biological response can be expected, if the material is used in appropriate applications.

The biological response to hydroxyapatite ceramic has been demonstrated by a history of clinical use and by laboratory studies. See the Bibliography for further information.

Implants for surgery — Hydroxyapatite —

Part 4:
Determination of coating adhesion strength

1 Scope

This part of ISO 13779 specifies test methods for measurement of the adhesion strength of hydroxyapatite coatings intended for use on components of surgical implants.

2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 13779. For dated references, subsequent amendments to, or revisions of, any of these publications do not apply. However, parties to agreements based on this part of ISO 13779 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 7500-1:1999, *Metallic materials — Verification of static uniaxial testing machines — Part 1: Tension/compression testing machines — Verification and calibration of the force-measuring system*

3 Terms and definitions

For the purposes of this part of ISO 13779, the following terms and definitions apply.

3.1
ceramic hydroxyapatite
hydroxyapatite which has been sintered into a coherent crystalline mass by subjecting it to conditions at which the crystals in the powder fuse together

[ISO 13779-1]

3.2
coating
distinct layer of material deposited onto the surface of a metallic or non-metallic substrate by either a thermal, vapour or aqueous route

3.3
hydroxyapatite
chemical compound with a crystallographic structure characterized by the powder diffraction file PDF 9-432 of the International Committee for Diffraction Data ICDD, Newton Square, Pennsylvania, USA

NOTE The chemical formula is $Ca_5(OH)(PO_4)_3$.

[ISO 13779-1]

3.4
sintering
process for production of ceramics in which the application of heat causes a significant reduction of particle surface area and bulk volume to achieve densification and consequent increase in mechanical properties

[ISO 13779-1]

4 Determination of hydroxyapatite coating adhesion strength

4.1 Principle

Coating adhesion strength is determined by applying a uniaxial tensile load to a cylindrical test assembly composed of one hydroxyapatite-coated specimen bonded to an uncoated component.

4.2 Apparatus

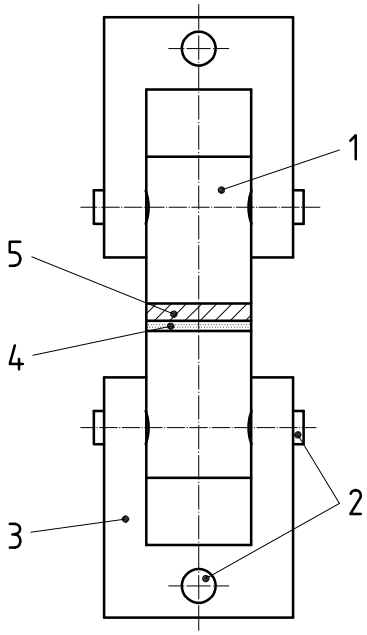
4.2.1 Mechanical testing machine, of load capacity not less than 30 kN and accuracy ± 2 % of full scale reading. The tensile load shall be applied normal to the plane of the coating.

Verification and calibration of the force-measuring system shall be in accordance with ISO 7500-1.

4.2.2 Loading assembly, used to transmit the load from the testing machine to the test assembly.

The loading assembly shall ensure that the axis of the test assembly does not deviate from that of the testing machine by more than ± 1° and ± 0,1 mm, in order that the coating test plane is perpendicular to the axial load. Eccentric loading of the test assembly shall invalidate the result.

The yoke and dowel pin assembly illustrated in Figure 1 is an example of a loading assembly which meets these requirements The coated specimen and uncoated component are each secured by two perpendicular pins which minimize off-axis loading. The dimensions of the loading assembly, as specified in Table 1, are suitable for the testing of coatings with a maximum predicted adhesion or cohesion strength of 50 MPa.



Key

1 Test assembly
2 Pins
3 Yoke
4 Adhesive
5 Tested coating

Figure 1 — Illustration of loading assembly which minimizes off-axis loading

Table 1 — Jig and specimen dimensions for testing of coatings with adhesion/cohesion strengths of less than 50 MPa

Figure 2 symbol	Test component/jig		Dimensions mm	
			Ti6Al4V alloy	316 stainless steel
ts	Test surface diameter	Stepped test component	$25 \pm 0{,}05$	$25 \pm 0{,}05$
		Straight test component	$25^{-0{,}02}_{-0{,}041}$	$25^{-0{,}02}_{-0{,}041}$
a	Sample holder step diameter		$20^{-0{,}02}_{-0{,}041}$	$18^{-0{,}02}_{-0{,}041}$
b	Pin hole diameter		$9^{+0{,}015}_{0}$	$8^{+0{,}015}_{0}$
c	Sample hole depth		$14 \pm 0{,}1$	$12 \pm 0{,}1$
d	Pin diameter		$9^{-0{,}005}_{-0{,}041}$	$8^{-0{,}005}_{-0{,}041}$
e	Pin length		$55 \pm 0{,}1$	$50 \pm 0{,}1$
f	Yoke internal width	Stepped test component	$20^{+0{,}033}_{0}$	$18^{+0{,}033}_{0}$
		Straight test component	$25^{+0{,}033}_{0}$	$25^{+0{,}033}_{0}$
g	Yoke external diameter	Stepped test component	$40 \pm 0{,}1$	$35 \pm 0{,}1$
		Straight test component	$45 \pm 0{,}1$	$45 \pm 0{,}1$
h	Yoke hole depth		$14 \pm 0{,}1$	$12 \pm 0{,}1$
k	Yoke internal depth		$40 \pm 0{,}1$	$40 \pm 0{,}1$

4.2.3 Test assembly.

The parts of the test assembly (coated specimen and uncoated component) are bonded together by the use of a polymeric adhesive.

The uncoated component shall be fabricated from the same material as the substrate of the coated specimen. Recommended materials are listed in Table 1.

The dimensions of the uncoated component shall be the same as those of the coated specimen, with dimensions and tolerances as in Table 1.

Loading and test components are illustrated in Figure 2. The dimensions of the components specified are suitable for the testing of coatings with a maximum predicted adhesion or cohesion strength of 50 MPa.

NOTE 1 The accuracy of this test method is highly sensitive to preparation of the coated specimen, application of the polymeric adhesive and proper alignment of the coated specimen and uncoated component. Improper alignment will result in eccentric loading and the application of bending stresses on the assembly, leading to an invalid result.

NOTE 2 The bond surface of the uncoated component may be roughened in order to aid bonding with the polymeric adhesive.

4.2.4 Polymeric adhesive, having a minimum adhesive bond strength at least 5 MPa greater than either the adhesion or cohesion strength of the coating, or 30 MPa, whichever is greater. The polymeric adhesive used shall be identified in any test report.

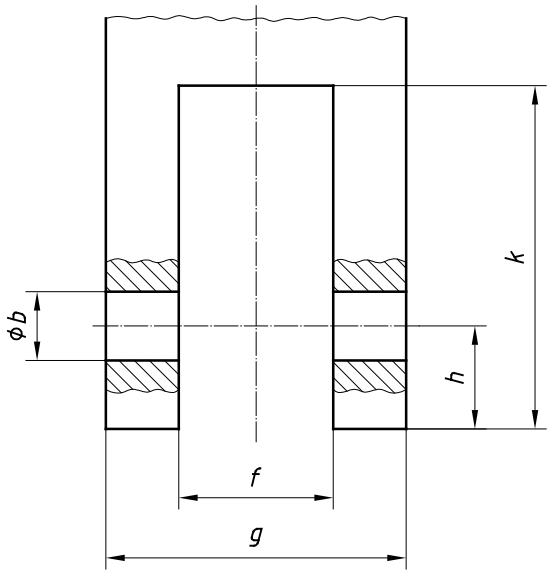
Where porosity extends to the coating/substrate interface, it shall be ensured that, by careful application to the coating and selection of a sufficiently viscous polymeric adhesive, the polymeric adhesive will not penetrate through the coating to the substrate. Penetration of the adhesive through the coating shall invalidate the result.

The penetration of the adhesive shall be assessed by examining cross-sections of a trial test piece.

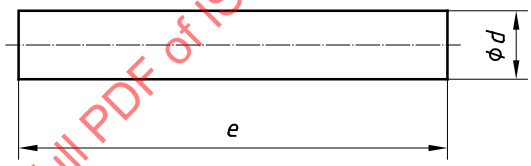
4.3 Preparation of coated specimen

Exercise care in the preparation of the coated specimen. Inspect all specimens prior to both assembly and testing. Reject any found to be non-representative of the particular coating under test. Test specimens shall not be machined after the hydroxyapatite coating has been applied. The area of coating to be tested shall be nominally circular with a diameter of 25 mm and tolerances as in Table 1, or shall possess an equivalent cross-sectional area. Measure and record the cross-sectional area and the coated specimen dimensions.

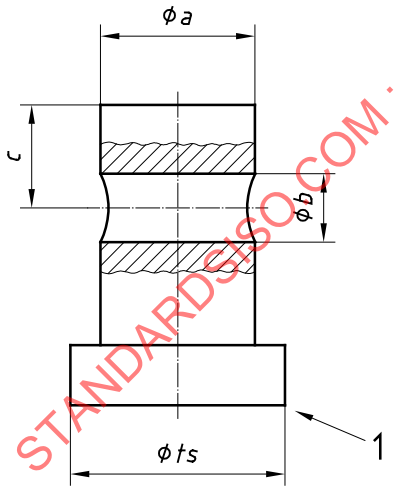
If specimens of other dimensions or cross-sectional area are used, the data shall be demonstrated to be equivalent to that obtained for a nominally circular specimen with a diameter of 25 mm.



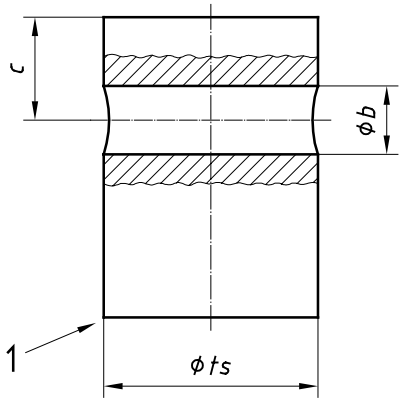
a) Yoke



b) Pin



c) Stepped test component



d) Straight test component

Key

1 Hydroxyapatite coating surface (when applicable)

See Table 1 for meaning of symbols and dimensions.

Figure 2 — Dimensions of loading and test components

4.4 Application of polymeric adhesive and alignment of test assembly

Apply the polymeric adhesive, in a single action, to the axial centre of the coating. Regulate the amount of adhesive applied and the force used to bring the parts together so as to ensure that excess adhesive does not form a collar around the coated specimen/uncoated component joint. Remove any excess adhesive after curing has occurred, in a manner which does not compromise the integrity of the test assembly. Bring the uncoated component into contact with the adhesive/coated specimen so as to ensure a plane parallel, uniform thickness of adhesive over the cross-sectional area to be tested.

In order to ensure uniaxial alignment of the test assembly, use a device to rigidly hold the parts of the assembly in position during the curing of the polymeric adhesive. This device shall enable a constant axial tension (typically, 0,2 MPa) to be applied to the assembly during curing, in order to compensate for any shrinkage of the adhesive.

4.5 Test procedure

Place the test assembly in the grips of the tensile testing machine, so that the long axis of the test assembly coincides with the direction of applied tensile loading. Apply the tensile load at a constant cross-head velocity of (1,0 ± 0,01) mm/min until complete separation of the components has been achieved. Record the maximum load applied, to the nearest 100 N.

Failure shall be confirmed to have occurred through the coating, rather than at the coating-adhesive interface, by using examination at 10 × optical magnification.

4.6 Calculation of coating adhesion strength

Coating adhesion or cohesion strength, σ, in megapascals, shall be calculated using the following equation:

$$\sigma = \frac{F}{A}$$

where

F is the maximum load, expressed in newtons;

A is the cross-sectional area (nominally 490,87mm^2), calculated to within ± 0,5 mm^2.

5 Test report for tensile testing of hydroxyapatite coatings

The following information shall be included in the test report:

a) reference to this part of ISO 13779;

b) identification of the materials used in the test assembly, including the type of polymeric adhesive and uncoated component. Report the details of any surface treatment applied to the bonded surface of the uncoated components and the resulting surface roughness;

c) number of specimens tested;

d) details of the method used in the manufacture of the coated specimens and any form of identification associated with the coated specimens, such as date and batch number;

e) all dimensional data associated with each coated specimen and the uncoated component, in addition to the bond cross-sectional area and the thickness of the hydroxyapatite coating;

f) all values of the maximum load and mode of failure (for example adhesive or cohesive);