
**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

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Published in Switzerland

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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.

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 the following URL: 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 13779-4:2002), which has been technically revised.

A list of all parts in the ISO 13779 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

Hydroxyapatite coatings are designed to promote adhesion to bone and to be colonized by bone. As a consequence, the mechanical strains in the coating and mechanical properties of the coating after implantation will change with time during the osteointegration of the coating. The aim of initial measurements of the adhesion properties of the coating, in dry conditions, detailed in this document is to guarantee minimum mechanical properties of the coating during implantation. Those minimal properties defined in ISO 13779-2 are for the coating to keep its morphology (thickness, roughness, etc.) during the implantation and to avoid chipping and spalling of the coating during implantation.

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Implants for surgery — Hydroxyapatite —

Part 4: Determination of coating adhesion strength

1 Scope

This document specifies a test method for measurement of the adhesion strength of hydroxyapatite coatings intended for use on metallic-substrate components of surgical implants.

NOTE Requirements for the competence of testing laboratories can be found in ISO/IEC 17025.

2 Normative reference

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 5832-1, *Implants for surgery — Metallic materials — Part 1: Wrought stainless steel*

ISO 5832-3, *Implants for surgery — Metallic materials — Part 3: Wrought titanium 6-aluminium 4-vanadium alloy*

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

ISO 13779-3, *Implants for surgery — Hydroxyapatite — Part 3: Chemical analysis and characterization of crystallinity and phase purity*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 13779-3 and the following apply.

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/>

3.1 adhesion strength

tensile strength at the interface between the coating and the substrate or within the coating

Note 1 to entry: For the purpose of this document, both adhesion and cohesion strength are summarized under the term adhesion strength.

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 coupon bonded to an uncoated counterface.

4.2 Apparatus

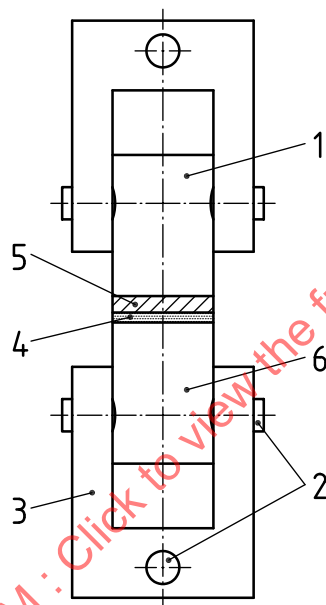
4.2.1 Mechanical testing machine, of load capacity not less than 30 kN and accuracy $\pm 2\%$ of full scale reading. The tensile load shall be applied normal to the plane of the coating.

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, in order that the coating test plane is perpendicular to the axial load.

The yoke and dowel pin assembly illustrated in [Figure 1](#) is an example of a loading assembly which meets these requirements. The coated coupon and uncoated counterface are each secured by two perpendicular pins which minimize off-axis loading.



Key

- 1 coated coupon
- 2 pins
- 3 yoke
- 4 adhesive
- 5 tested coating
- 6 counter-part

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

The loading assembly shall be inspected before use to ensure it is mounted correctly and it is not damaged.

4.2.3 Test assembly

The parts of the test assembly (coated coupon and counter-part) are bonded together by the use of an adhesive.

The counter-part shall be fabricated from stainless steel complying with ISO 5832-1, Ti 6-Al 4-V alloy complying with ISO 5832-3 or from the same material as the substrate of the coated coupon.

Coated coupons shall be rods with nominal diameter comprised between 24,8 mm and 25,6 mm. Tolerances on the diameter shall be less than 0,2 mm.

NOTE Careful clamping of the coupon can minimize stress effects in the coating due to deformation of the substrate which might impact the interface being tested.

The diameter of the counter-part shall be the same as that of the coated coupon. The bond surface of the uncoated counterface can be roughened in order to aid bonding with the adhesive.

All test specimens and test assemblies shall be handled with care and be kept clean as the test results can be affected by contamination such as from oil and debris at the coating adhesive interface.

4.2.4 Adhesive

The adhesive shall have a minimum adhesive bond strength of 5 MPa greater than the adhesion strength of the coating.

NOTE As hydroxyapatite coatings contain pores, it is important to ensure that, by careful application to the coating and selection of a sufficiently viscous adhesive, the adhesive will not penetrate through the coating to the substrate. The viscosity and the wetting angle of the adhesive with the coating material, along with the quantity, size and distribution of pores in the coating material will influence the opportunity for penetration of the adhesive.

4.3 Test method

4.3.1 Number of test samples

A minimum of 10 valid results on coated coupons shall be obtained.

4.3.2 Preparation of coated coupon

All coupons shall be prepared using the same production methods of regular implant components, including initial powder, substrate material, production installations, substrate surface preparation process, coating process parameters, cleaning and sterilization, unless there are special reasons which shall be justified by the manufacturer.

ISO 13779-2 provides information on the thickness of the coupons' coating. Thickness of the coating shall be evaluated on each coated coupon by a non-destructive method (for example eddy current, micrometer, or other suitable method if the method is correlated to the results obtained with ASTM F1854).

A coating of greater thickness than that applied to standard product may be used for testing coating strength providing the methodology of deposition can be justified to be the same, and the thicker coating rationalised as a worst case strength coating. The thicker coating could alleviate adverse test results found with adhesive penetration of a thinner coating.

4.3.3 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 $(2,5 \pm 0,5)$ mm/min until complete separation of the components has been achieved. Record the maximum load applied, to the nearest 100 N.

NOTE Although the adhesive is selected so that the adhesive strength when tested between two metal test pieces is greater than the coating strength, this might not be the case when the adhesive is between a metal counterface and an HA coating. This might be due to poor wetting of the HA by the adhesive.

The failures shall be documented by micrographs and the failures shall be classified as:

- a) failure at the interface between the substrate and the coating,

- b) failure in the coating,
- c) failure at the interface between the coating and the adhesive,
- d) failure in the adhesive,
- e) or mixed failures of above types.

Once the failure mode has been identified, the micrographs shall be annotated identifying examples of the classified failure mode.

Case a) and b)

A valid test shall produce a failure either at the interface between the substrate and the coating or in the coating. Presence of adhesive on either fracture surface shall invalidate the result.

Case c) and d)

If the failure occurs purely within the adhesive or at the interface between the coating and the adhesive the result relating to the coupon shall be rejected.

Case e)

The only valid tests with mixed failure mode are those in which failure modes a) and b) are mixed. In such cases the proportion of each failure mode should be recorded.

4.4 Calculation of coating adhesion strength

Coating adhesion strength, σ , in megapascals, shall be calculated for each coupon using the following equation:

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

where

F is the maximum load, expressed in newtons;

A is the cross-sectional area of the coated coupon, in mm².

Mean and standard deviation of the adhesion strength obtained for the coated coupon during valid tests shall be calculated.

5 Test report for tensile testing of hydroxyapatite coatings

The following information shall be included in the test report:

- a) reference to this document (including its year of publication);
- b) name, address and certifications of the testing laboratory;
- c) identification of the materials used for the adhesive and uncoated counterface. Report the details of any surface treatment applied to the bonded surface of the uncoated counterfaces;
- d) the adhesive used;
- e) details of the method used in the manufacture of the coated specimens or any form of identification associated with the coated coupons, such as date and batch number;
- f) the method used for measuring coating thickness (see [4.3.2](#));
- g) the number of coupons tested;

- h) for each tested coupon specify the following:
 - 1) coating thickness;
 - 2) adhesion strength;
 - 3) mode of failure and micrographs of each fracture surface with appropriate scale referenced (see [4.3.3](#));
 - 4) whether the test was considered valid or non-valid;
 - 5) if the result was considered non-valid, the reason for rejection (see [4.3.3](#));
- i) for valid results specify the following values of adhesion strength of the coated coupons: mean and standard deviation (see [4.4](#));

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