BS ISO 13179-1:2014

BSI Standards Publication

Implants for surgery — Plasma-sprayed unalloyed titanium coatings on metallic surgical implants

Part 1: General requirements

National foreword

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A list of organizations represented on this committee can be obtained on request to its secretary.

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Implants for surgery — Plasma-sprayed unalloyed titanium coatings on metallic surgical implants —

Part 1: General requirements

Implants chirurgicaux — Revêtements en titane non-allié des implants chirurgicaux métalliques, obtenus par projection plasma —

Partie 1: Exigences générales

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Case postale 56 • CH-1211 Geneva 20
Tel. + 41 22 749 01 11
Fax + 41 22 749 09 47
E-mail copyright@iso.org
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Foreword

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The committee responsible for this document is ISO/TC 150, *Implants for surgery*, Subcommittee SC 1, *Materials*.

ISO 13179 consists of the following parts, under the general title *Implants for surgery — Plasma-sprayed unalloyed titanium coatings on surgical implants*:

— *Part 1: General requirements*

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 the use of the material referred to in this Standard has shown that an acceptable level of biological response can be expected, if the material is used in appropriate applications.

INTERNATIONAL STANDARD **ISO 13179-1:2014(E)**

Implants for surgery — Plasma-sprayed unalloyed titanium coatings on metallic surgical implants —

Part 1: General requirements

1 Scope

This part of ISO 13179 specifies general requirements for plasma-sprayed unalloyed titanium coatings on metallic surgical implants.

This part of ISO 13179 applies to plasma spraying in air and in vacuum.

This part of ISO 13179 does not apply to coatings made of other materials than unalloyed titanium or coatings realized by another technology than plasma spraying.

NOTE 1 A quality management system can be useful, e.g. as described in ISO 13485. Requirements for the competence of testing laboratories can be found in ISO/IEC 17025.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

ISO 4287, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Terms, definitions and surface texture parameters*

ISO 5832-2, *Implants for surgery — Metallic materials — Part 2: Unalloyed titanium*

ISO 10993-1, *Biological evaluation of medical devices — Part 1: Evaluation and testing within a risk management process*

ISO 14971, *Medical devices — Application of risk management to medical devices*

ASTM F1044, *Standard Test Method for Shear Testing of Calcium Phosphate Coatings and Metallic Coatings*

ASTM F1147, *Standard Test Method for Tension Testing of Calcium Phosphate and Metallic Coatings*

ASTM F1160, *Standard Test Method for Shear and Bending Fatigue Testing of Calcium Phosphate and Metallic Medical and Composite Calcium Phosphate/Metallic Coatings*

ASTM F1580, *Standard specification for Titanium and Titanium-6 Aluminium-4 alloy powders for coatings of surgical implants*

ASTM F1854, *Standard Test Method for Stereological Evaluation of Porous Coatings on Medical Implants*

ASTM F1978, *Standard Test Method for measuring abrasion resistance of metallic thermal spray coatings by using the Taber Abraser*

ASTM E2371, *Test Method for analysis of Titanium and Titanium Alloy by Atomic Emission Plasma Spectrometry*

3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

3.1
plasma spraying
method of thermal spraying that produces coatings using a plasma jet

3.2
plasma-sprayed unalloyed titanium coatings
titanium which has been deposited onto the surface of a substrate, by means of a plasma spraying process

4 Requirements

4.1 Powder for plasma spraying

The powder used for plasma spraying shall comply with ASTM F1580.

4.2 Chemical analysis

4.2.1 Chemical composition

NOTE Although this standard applies to both atmospheric and vacuum plasma spraying coatings the vacuum plasma sprayed coating chemical composition values are likely to be lower than for atmospheric plasma sprayed coatings.

The chemical composition of the unalloyed titanium plasma sprayed coatings shall be in accordance with Table 1.

Table 1 — Chemical composition

Element	Composition limits (mass fraction)
Carbon (C)	≤ 0,10 %
Hydrogen (H)	≤ 0,20 %
Iron (Fe)	≤ 0,60 %
Nitrogen (N)	≤ 5,00 %
Oxygen (O)	≤ 10,00 %
Titanium (Ti)	Balance
The sum of the nitrogen, oxygen, hydrogen, carbon and iron, contents shall not be greater than 10 % mass fraction. NOTE If the mechanical properties specified in 4.4 are met and if there are no detrimental effects on the biocompatibility it can be acceptable to have higher limits for Hydrogen and Nitrogen content than those specified above.	

A risk analysis of the plasma spraying process shall be conducted to determine the elements not listed in Table 1 and likely to have a mass fraction greater than 0,5 %. The exact mass fraction shall be determined and the limit shall be specified by the manufacturer of the coating. The influence of these impurities on biocompatibility shall be assessed in accordance with ISO 10993-1.

4.2.2 Sampling

In order to perform the chemical analysis of the coating, a minimum of 5 g of coating, which has been deposited onto a titanium substrate coupon produced in accordance with ISO 5832-2, shall be removed. If the removal of 5 g of coating is not possible or not sensible, the sample weight may be decreased provided that the chemical analysis is not affected. The technique used to remove the coating shall not generate contamination of the coating. If a cleaning step is performed on the implants after coating, the same cleaning step shall be applied on the sample before analysis.

4.2.3 Chemical analysis procedure

Iron content shall be determined by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES) in accordance with ASTM E2371.

Nitrogen, oxygen, carbon and hydrogen content, shall be determined by combustion using a recognized validated method.

The analysis accuracy with a level of confidence of 95 % shall be taken into account when claiming the conformity of the chemical analysis to the limits specified in Table 1.

EXAMPLE If the uncertainty for measuring the oxygen content is 1 % with a level of confidence of 95 %, the conformity shall be stated as ≤ 9 % for the measured values.

4.3 Morphology

For validation purposes the morphology tests shall be performed on the final device, if possible. If the morphology tests are not performed on the final device because the requirements of the applied standard cannot be met due to geometry of the device, coupons may be used and their representativeness to the final device shall be justified.

The average thickness and the tolerances, in µm, shall be determined in accordance with ASTM F1854.

Roughness (*Ra* or *Rt*), in µm, shall be determined in accordance with ISO 4287. The evaluation length shall be at least 8 mm.

For coatings having an average thickness ≥ 300 µm, the average volume % void content and mean void intercept length, and/or average void content and intercept length at distinct levels through the coating thickness ("tissue interface gradient method") shall be tested in accordance with ASTM F1854.

4.4 Coating mechanical properties

4.4.1 General

At least 5 test samples, manufactured from the same substrate as the final implant, shall be used for the tests specified in 4.4.2 to 4.4.4. The same pre-treatments (e.g. cleaning, sand-blasting) and post-treatments (e.g. cleaning, sterilization) shall be applied to the test samples and the final implant.

4.4.2 Static shear strength

When tested in accordance with ASTM F1044, the mean static shear strength of the coating shall be greater than 20 MPa.

4.4.3 Shear fatigue strength

When tested in accordance with ASTM F1160 with a shear fatigue maximum strain of at least 10 MPa, the coating shall withstand at least 10^7 cycles without any failure.

4.4.4 Static tensile strength

When tested in accordance with ASTM F1147, the mean static tensile strength of the surface coating shall be greater than 22 MPa.

4.4.5 Abrasion resistance

When tested in accordance with ASTM F1978, the abrasion loss of the surface coating shall be less than 65 mg after 100 cycles.

4.5 Audit control

The chemical composition, morphology and tensile strength of the coating shall be controlled periodically with a periodicity determined during the risk analysis in accordance with ISO 14971.

Routine control procedures of the coating shall specify the type and time interval of each control test.

4.6 Significance of numerical limits

For determination of compliance to all limits specified in any condition, the calculated or observed values shall be rounded to the nearest two significant digits after the decimal sign expressing the specified limit.

NOTE For rounding of numbers see ISO 80000-1:2009, Annex B.

5 Test report

The test report shall include at least the following information:

a) identification of the test sample including source, date of receipt, form;

b) number of test samples used for each test;

c) the reference to the test method used;

d) deviations from the test method used, if relevant;

e) any unusual features observed during the tests;

f) justification for any results removed from the data;

g) chemical analysis results (see 4.2);

h) morphology analysis results (see 4.3);

i) mean static shear strength (see 4.4.2);

j) shear fatigue strength (see 4.4.3);

k) mean static tensile strength (see 4.4.4);

l) abrasion loss (see 4.4.5);

m) dates of tests;

n) identification of the laboratory carrying out the test;

o) signature of the laboratory manager and operator.

Bibliography

[1] ISO 5961, *Water quality — Determination of cadmium by atomic absorption spectrometry*

[2] ISO 13485, *Medical devices — Quality management systems — Requirements for regulatory purposes*

[3] ISO 16429, *Implants for surgery — Measurements of open-circuit potential to assess corrosion behaviour of metallic implantable materials and medical devices over extended time periods*

[4] ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories*

[5] ISO 80000-1, *Quantities and units — Part 1: General*

[6] ASTM F67, *Standard Specification for Unalloyed Titanium for Surgical Implant Applications*

[7] ASTM E1409, *Standard Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Technique*

[8] ASTM E1447, *Standard Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by Inert Gas Fusion Thermal Conductivity/Infrared Detection Method*

[9] ASTM E1941, *Standard Test Method for Determination of Carbon in Refractory and Reactive Metals and Their Alloys by Combustion Analysis*

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BSI Group Headquarters

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