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Implants for surgery — Coatings on metallic surgical implants —

Part 1: Plasma-sprayed coatings derived from titanium or titanium-6 aluminum-4 vanadium alloy powders

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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 www.iso.org/iso/foreword.html.

This document was prepared by Technical Committee ISO/TC 150, *Implants for surgery*.

This second edition cancels and replaces the first edition (ISO 13179-1:2014), which has been technically revised.

The main changes compared to the previous edition are as follows:

— the title and scope of the document were changed to include coatings derived from titanium-6 aluminum-4 vanadium alloy powder;

— two new terms 3.2 and 3.3 were introduced to add a distinction between vacuum plasma spraying and atmospheric plasma spraying;

— in 4.2.1, three separate tables were introduced to distinguish the chemical composition limits of different coating addressed by this document;

— in 4.4.3, the term shear fatigue maximum strain was corrected to shear fatigue maximum stress.

A list of all parts in the ISO 13179-1 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

While no known surgical implant material has ever been shown to be completely free of adverse reactions in the human body, long term clinical experience with the material referred to in this document has shown that an acceptable level of biological response can be expected, if the material is used in appropriate applications. However, this document covers the raw material, coating structures and properties and not finished medical devices, where the design and fabrication of the device can also impact biological response.

Implants for surgery — Coatings on metallic surgical implants —

Part 1: Plasma-sprayed coatings derived from titanium or titanium-6 aluminum-4 vanadium alloy powders

1 Scope

This document specifies general requirements for plasma-sprayed titanium coatings on metallic surgical implants.

This document applies to atmospheric plasma spraying and vacuum plasma spraying.

This document does not apply to coatings made of other materials than titanium or titanium-6 aluminum-4 vanadium alloy or to coatings realized by another technology than plasma spraying.

NOTE 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 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 4288, *Geometrical Product Specifications (GPS) — Surface texture: Profile method — Rules and procedures for the assessment of surface texture*

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 Vanadium 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.

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 https://www.electropedia.org/

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

3.2
vacuum plasma spraying
plasma spraying (3.1) process carried out at pressures below atmospheric pressure in a controlled inert environment

3.3
atmospheric plasma spraying
plasma spraying (3.1) process carried out at atmospheric pressure

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

A risk assessment shall be conducted to identify and assess impurities introduced during the plasma spraying process.

For vacuum plasma-sprayed coatings and atmospheric plasma-sprayed coatings, the compositional limits for the elements listed in Tables 1 to 3 shall not exceed the mass fraction specified.

The chemical composition shall be determined using the sampling procedure in 4.2.2 and the chemical analysis procedure in 4.2.3. The results of the chemical analysis shall be included in the test report [see 5 i)].

Table 1 — Chemical composition for vacuum plasma sprayed titanium coatings

Element	Composition limits (mass fraction)
Carbon (C)	≤0,10 %
Hydrogen (H)	≤0,30 %
Iron (Fe)	≤0,60 %
Nitrogen (N)	≤0,50 %
Oxygen (O)	≤1,00 %
Titanium (Ti)	Balance

Table 2 — Chemical composition for vacuum plasma sprayed Ti-6Al-4V coatings

Element	Composition limits (mass fraction)
Carbon (C)	≤0,10 %
Hydrogen (H)	≤0,30 %
Iron (Fe)	≤0,60 %
Nitrogen (N)	≤0,50 %
Oxygen (O)	≤1,00 %
Titanium (Ti) + Aluminium (Al) + Vanadium (V)	Balance

Table 3 — Chemical composition for atmospheric plasma sprayed titanium coatings

Element	Composition limits (mass fraction)
Carbon (C)	≤0,10 %
Hydrogen (H)	≤0,30 %
Iron (Fe)	≤0,60 %
Nitrogen (N)	≤5,00 %
Oxygen (O)	≤10,00 %
Titanium (Ti)	Balance

NOTE 1 At the time of publication, the committee was not aware of any atmospheric plasma sprayed Ti-6Al-4V coatings; therefore, no table for their chemical composition is included.

NOTE 2 The above does not eliminate the necessity for biological evaluation of the final device 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 substrate coupon shall be removed. The coupon shall be representative of and made from the same material as the implant the coating is intended to be deposited on. If the removal of 5 g of coating is not possible or not sensible, the sample mass 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 Tables 1 to Table 3.

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

4.3 Morphology and microstructure

For validation purposes, tests for the morphology and microstructure of the coating shall be performed on the final device, if possible. If the morphology and microstructure 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 4288. The evaluation length shall be at least 8 mm.

For coatings having an average thickness ≥300 µm, the average volume percent void content and mean void intercept length, and/or average volume percent void content and mean void 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 10 test specimens, manufactured from the same substrate as the final implant, shall be used for the tests specified in 4.4.2 and 4.4.4.

At least 5 test specimens, manufactured from the same substrate as the final implant, shall be used for the tests specified in 4.4.3.

The same pre-treatments (e.g. cleaning, sand-blasting) and post-treatments (e.g. cleaning, sterilization) shall be applied to the test specimens and the final implant.

NOTE 1 The mechanical properties results can be influenced by the thickness of the coating. High thickness coatings can result in lower adhesion strength results. Therefore, it can be appropriate within a validation process to determine adhesion or shear to the substrate on coupons with a coating thickness within or above the highest quartile of the specification for the thickness of the coating.

NOTE 2 Process validation is typically performed on multiple lots or batches.

4.4.2 Static shear strength

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

4.4.3 Shear fatigue strength

The coating shall be tested in accordance with ASTM F1160 with a shear fatigue maximum stress of at least 10 MPa and the coating shall withstand at least 10^7 cycles without any failure. The maximum test frequency shall not exceed 10 Hz in order to reduce potential errors related to the dynamic response of the test machine and its sensors.

NOTE Higher test frequencies can be used if evidence is provided that the test results are identical to those obtained at 10 Hz or less.

4.4.4 Static tensile strength

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