Designation: F 1580 – 01

Standard Specification for Titanium and Titanium-6 Aluminum-4 Vanadium Alloy Powders for Coatings of Surgical Implants[1]

This standard is issued under the fixed designation F 1580; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers the requirements for unalloyed titanium and Ti-6Al-4V alloy powders for use in fabricating coatings on titanium alloy implants.

1.2 Powders covered under this specification may be used to form coatings by sintering or thermal spraying techniques.

1.3 This specification covers powder requirements only. It does not address properties of the coatings formed from them.

2. Referenced Documents

2.1 *ASTM Standards:*

B 214 Test Method Sieve Analysis of Granular Metal Powders[2]

B 215 Practices for Sampling Finished Lots of Metal Powders[2]

B 299 Specification for Titanium Sponge[3]

E 11 Specification for Wire-Cloth Sieves for Testing Purposes[4]

E 120 Test Methods for Chemical Analysis of Titanium and Titanium Alloys[5]

F 67 Specification for Unalloyed Titanium for Surgical Implant Applications[6]

F 981 Practice for Assessment of Compatibility of Biomaterials for Surgical Implants With Respect to Effect of Materials on Muscle and Bone[6]

F 1472 Specification for Wrought Ti-6Al-4V Alloy for Surgical Implant Applications[6]

2.2 *American Society for Quality (ASQ) Standards:*[7]

ASQ C1 General Requirements for a Quality Program

2.3 *Aerospace Material Specifications:*[8]

AMS 2249 Chemical Check Analysis Limits, Titanium and Titanium Alloys

AMS 4998A Powder, 6Al-4V, Premium Quality (noncurrent)

3. Methods of Manufacture

3.1 Powders may be manufactured by the plasma rotating electrode process, inert gas atomization, hydride-dehydride, or other method capable of producing powder meeting the requirements of this specification.

4. Chemical Requirements

4.1 The chemical analysis of the powder shall conform to the requirements set forth in Table 1. Analysis shall be performed before the addition of any processing aids.

4.1.1 Requirements for the major and minor elemental constituents for unalloyed titanium and Ti-6Al-4V alloy powders are listed in Table 1. Also listed are all important residual elements. Analysis for elements not listed in Table 1 is not required to verify compliance with this specification.

4.2 The product analysis tolerance shall conform to the requirements set forth in Table 2.

4.3 For referee purposes, Test Methods E 120 shall be used.

4.4 Intentional elemental additions other than those specified in Table 1 are not permitted.

4.5 For powder that includes particle size fractions finer than 200 mesh (74 μm), the oxygen content limits shall be agreed upon between buyer and seller.

5. Particle Size and Shape

5.1 Powder shall be sieved to the customer's requirements with stainless steel screens conforming to Specification E 11. Analysis of sieved powder for conformance to the customer's particle size range requirements shall be in accordance with Test Method B 214.

5.2 Powder made from the plasma rotating electrode process and inert gas atomization tends to be spherical in shape, powder made from the hydride-dehydride process tends to be

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[2] *Annual Book of ASTM Standards*, Vol 02.05.

[3] *Annual Book of ASTM Standards*, Vol 02.04.

[4] *Annual Book of ASTM Standards*, Vol 14.02.

[5] *Annual Book of ASTM Standards*, Vol 03.05.

[6] *Annual Book of ASTM Standards*, Vol 13.01.

[7] Available from the American Society for Quality, 600 N. Plankinton Ave., Milwaukee, WI 53203.

[8] Available from Society of Automotive Engineers, 400 Commonwealth Dr., Warrendale, PA 15096–0001.

TABLE 1 Chemical Requirements

Element	Unalloyed Ti Powder Weight Percent		Ti-6Al-4V Powder Weight Percent	
	Min	Max	Min	Max
Al			5.50	6.75
V			3.50	4.50
O		0.40		0.20
Fe		0.50		0.30
C		0.10		0.08
H		0.05		0.015
N		0.05		0.05
Cu				0.10
Sn				0.10
Si		0.04		
Cl		0.20[A]		
Na		0.19[A]		
Ti	balance[B]		balance[B]	

[A] Lower maximum chlorine and sodium contents may be agreed upon between buyer and seller.
[B] The percentage of titanium is determined by difference and need not be measured.

TABLE 2 Product Analysis Tolerances[A]

Element	Variation Under Min or Over Max
Aluminum	0.04
Vanadium	0.015
Oxygen	0.03[B]
Oxygen	0.02[C]
Iron	0.10
Hydrogen	0.002
Carbon	0.02
Nitrogen	0.02
Copper	0.05
Tin	0.15
Silicon	0.02

[A] Refer to AMS 2249.
[B] For unalloyed Ti powder.
[C] For Ti-6Al-4V alloy powder.

angular in shape and sponge powder tends to be irregular in shape.

6. Cleanliness

6.1 Powder shall be handled at all times so as to ensure freedom from contamination with nonmetallic materials or other metal alloy powders or both.

6.2 Powder cleanliness shall be determined by examining a representative sample, per Practices B 215 or as agreed upon between buyer and seller, comprising at least 1 in.^{2}(6.45 cm^2) of a closely packed mono-layer of powder per lot at 20× magnification. No foreign material shall be visible under these conditions. Powder cleanliness shall be determined before the addition of any processing aids.

7. Special Requirements

7.1 Various materials known as processing aids may be added to the powder to provide enhanced processibility. The powder supplier shall identify the chemical composition and weight percentage of any added processing aids on the material certification.

7.2 Processing aids shall have no detrimental effect on the corrosion resistance or biocomptability of the final coating.

NOTE 1—Finely divided titanium powder may be considered pyrophoric and should be handled in accordance with the appropriate guidelines in the Material Safety Data Sheet.

8. Certification

8.1 Powder shipped under this specification shall be accompanied by certification that includes:

8.1.1 ASTM designation and date of issue.
8.1.2 Quantity (weight).
8.1.3 Method of manufacture.
8.1.4 Chemical analysis per 4.1.
8.1.5 Sieve analysis per 5.1.
8.1.6 Powder cleanliness per 6.2.
8.1.7 Special requirement per 7.2.
8.1.8 Other requirements.

9. Quality Program Requirements

9.1 The producer shall maintain a quality program, such as that defined in the ASQ C1, for example.

9.2 The manufacturer of surgical implants shall be ensured of the producer's quality program for conformance to the intent of ASQ C1 or other recognized programs.

10. Keywords

10.1 coatings; metallic; metals (for surgical implants titanium alloys); orthopaedic medical devices (titanium/titanium alloys); powder; porous coatings; titanium/titanium alloys (for surgical implants)

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 Coatings formed from metallic powders have become widely used as a means of improving tissue attachment to uncemented orthopedic joint prosthesis. Such coatings have also been demonstrated to improve bonding of acrylic cement to prostheses. The method used to create the coatings can determine which powder size and shape is suitable for the specific application. Not all powder sizes or shapes are applicable for all coating processes.

X1.2 Chemical composition limits for O, Fe, C, and N in the unalloyed grade are taken from Specification F 67, Grade 4. Limits for Si, Cl, H, and Na are taken from Specification B 299, Grade SL.

X1.3 Chemical composition limits for Al, V, O, Fe, C, H, and N in the Ti-6Al-4V grade are taken from Specification F 1472. Limits for Cu and Sn are taken from AMS 4998.

X1.4 Product analysis tolerances are taken directly from AMS 2249. No recognized product analysis tolerances currently exist specifically for Cl or Na in titanium alloys.

X1.5 Processing aids are frequently used to facilitate powder processing and application of porous coatings to implant surfaces. It is beyond the scope of this specification to identify suitable processing aids or define their use. It is the responsibility of the implant manufacturer to ensure that any processing aid or residue of a processing aid has no detrimental effect on biocompatibility or coating properties.

X1.6 It should be recognized that the heat treatments used to form porous coatings can create microstructures that are substantially different from wrought titanium alloys. Porous coated implants also exhibit much greater surface area than monolithic implants. For these reasons, the biocompatibility and corrosion behavior must be characterized on finished coatings.

X1.7 Likewise, these heat treatments can create microstructures that give substantially different corrosion fatigue behavior from that of typical wrought titanium alloys. Corrosion fatigue behavior must be evaluated on finished coated substrates.

X1.8 Pore size and morphology are important factors influencing tissue ingrowth and acrylic penetration of porous coatings. Particle size, size distribution, and shape are critical to controlling the pore size and morphology in the final coating. Particle size and size distribution are conventionally controlled by screening. The referenced ASTM International standards allow comparison of powder to a manufacturer's specifications for a given coating process. A number of methods to characterize particle shape exists. The coating manufacturer should select a means of particle shape characterization suitable for his process.

X1.9 This specification requires sampling for particle size and powder cleanliness on each powder lot. In some cases, sampling on each shipping container of powder may be appropriate.

X1.10 Other process parameters are also critical to determining final pore size and morphology in the final coating. Because these parameters are not directly related to the chemical and physical characteristics of the starting powder, they are not addressed in this specification.

X1.11 The requirements for powder cleanliness ensure freedom from contaminants that might adversely affect either the biocompatibility or the finished coatings or the ability to bond the coating properly during manufacturing. The method in 6.2 (Practices B 215) is commonly used for relatively coarse spherical powders used to fabricate sintered porous coatings. Other types of powders may require different methods for cleanliness characterization. The development and implementation of such methods are the responsibility of the implant manufacturer.

X2. BIOCOMPATIBILITY

X2.1 The biocompatibility of metallic implants is a direct function of their composition. The alloy compositions covered by this specification have been used in the wrought form for surgical implants and have been used successfully in human implant applications in contact with soft tissue and bone for over a decade. Because of the well characterized level of biological response exhibited by these alloys, they have been used as a control material in Practice F 981.

X2.2 No known surgical implant material has ever been shown to be completely free from adverse reactions in the human body. Long-term clinical experience of the use of the material referred to in this specification, however, has shown that an acceptable level of biological response can be expected, if the material is used in appropriate applications.

SUMMARY OF CHANGES

(*1*) Added 6.2 concerning particle shape, eliminated Section 3 and renumbered document, and added X1.1 in which powder shape and application is addressed.

(*2*) Editorial corrections have been made to meet terminology and formatting guidelines established for implant material standards.