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Standard Specification for Wrought Titanium-12 Molybdenum-6 Zirconium-2 Iron Alloy For for Surgical Implant Applications (UNS R58120)[1]

This standard is issued under the fixed designation F 1813; 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.

ε[1] NOTE—Footnote 1 was editorially revised June 2000.

1. Scope*

1.1 This specification covers the chemical, mechanical, and metallurgical requirements for wrought Ti-12 Mo-6 Zr-2 Fe alloy

[1] This specification is under the jurisdiction of ASTM Committee F-4 F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F0.4.12 on Metallurgical Materials.

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*A Summary of Changes section appears at the end of this standard.

to titanium – 12 molybdenum – 6 zirconium – 2 iron alloy to be used in the manufacture of surgical implants.[2] .

1.2 The values stated in inch-pound units are to be regarded as the standard. The metric SI equivalents given in parentheses are provided for information only.

2. Referenced Documents

2.1 *ASTM Standards:*

B 348 Specification for Titanium and Titanium Alloy Bars and Billets[3]

E 8 Test Methods for Tension Testing of Metallic Materials[3]

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[2] Wang, K., Gustavson, L., Dumbleton, J., "The Characterization of Ti-12Mo-6Zr-2Fe - A New Biocompatible Alloy Developed for Surgical Implants", *Beta Titanium Alloys in the 1990's*, Proceedings of a Symposium on Beta Titanium Alloys held at the 1993 Annual TMS meeting in Denver, Colorado, February 22–24, 1993.

[2] FDA 510K application number K903630.

[3] *Annual Book of ASTM Standards*, Vol 02.04. 03.01.

E 112 Test Methods for Determining Average Grain Size[3]

E 120 Test Methods for Chemical Analysis of Titanium and Titanium Alloys[4]

E 1124409 Test Methods for the Determination of Oxyg Avern in Titagnium and Titanium Alloys by the G Inert Gas Fusion S Technizque[5]

E 120 Test Methods for Chemical Analysis of Titanium and Titanium Alloys[4]

E 1409 Test 1447 Test Method for the Determination of Oxygen in Titanium and Titanium Alloys by the Inert Gas Fusion Technique[4]

E 1447 Test Method for the Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity Method[5]

F 748 Practice for Selecting Generic Biological Test Methods for Materials and Devices[6]

F 750981 Practice for Evaluating Material Extracts by Systematic Injection in the Mouse Assessment of Compatibility of Biomaterials for Surgical Implants with Respect to Effect of Materials on Muscle and Bone[6]

F 895 Test Method 1408 Practice for Agar Diffusion Cell Culture Subcutaneous Screening Test for Cytotoxicity[6]

F 981 Practice for Assessment of Compatibility of Biomaterials (Non-Porous) for Surgical Implants with Respect to Effect of Implant Materials on Muscle and Bone[6]

2.2 *Aerospace Materials Specification:*

AMS 2249 Chemical Check Analysis Limits, Titanium and Titanium Alloys[7]

[4] *Annual Book of ASTM Standards*, Vol 03.015.

[5] *Annual Book of ASTM Standards*, Vol 03.056.

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

[7] Available from the American Society of Automotive E;ngineers, 400 Commonwealth Drive, Dr., Warrendale, PA 15096-0001.

2.3 *ISO Standards:*
ISO 6982 Metallic Materials Tensile Testing at Ambient Temperature[8]
2.4 *American Society for Quality Standard:*
ASQ C1 Specification of General Requirements for a Quality Program[9]

3. ~~Ordering Information~~

~~3.1 Inquiries and orders for material under this specification shall include the following information:~~
~~3.1.1 Quantity (weight or number~~Terminology
3.1 *Definitions ~~of pieces),~~*
~~3.1.2 ASTM Designation,~~
~~3.1.3 Dimensions,~~
~~3.1.4 Condition,~~
~~3.1.5 Finish (see 4.2), and~~
~~3.1.6 Special requirements.~~ Terms Specific to This Standard:
3.1.1 *beta transus, n*—the minimum temperature at which the alpha plus beta phase can transform to 100 % beta phase.

4. ~~Materials and Manufacture~~

~~4.1 The titanium mill products covered in this specification are normally formed with the conventional forging, rolling~~Product Classification
4.1 *Bar*—Rounds or ~~other metal processing equipment found~~ flats from 3⁄16 in. (4.76 mm) to 4 in. (101.60 mm) in ~~primary ferrous~~ diameter or thickness (other sizes and ~~nonferrous plants. The ingot metal for such mill operations is usually multiple melted in arc furnaces (including furnaces such as plasma arc and electron beam) of a type conventionally used for reactive metals.~~ shapes by special order).
4.2 ~~*Finish*—Annealed material may be furnished to the implant manufacturer as descaled~~Wire—Rounds or ~~pickled, sandblasted, machined, ground,~~ flats less than 3⁄16 in. (4.76 mm) in diameter or ~~combinations of these operations.~~ thickness.

5. Ordering Information

5.1 Include with inquiries and orders for material under this specification shall include the following information:
5.1.1 Quantity,
5.1.2 ASTM designation and date of issue,
5.1.3 Form (bar or wire),
5.1.4 Condition (see 6.3),
5.1.5 Mechanical properties (if applicable for special conditions),
5.1.6 Finish (see 6.2),
5.1.7 Applicable dimension including size, thickness, width, or drawing number,
5.1.8 Special tests, if any,
5.1.9 Other requirements.

6. Materials and Manufacture

6.1 The various titanium mill products covered in this specification normally are formed with the conventional forging and rolling equipment found in primary ferrous and nonferrous plants. The alloy is usually multiple melted in arc furnaces (including furnaces such as plasma arc and electron beam) of a type conventionally used for reactive metals.
6.2 *Finish*—The mill product may be furnished to the implant manufacturer as descaled or pickled, sandblasted, chemically milled, ground, machined, peeled, polished, or combinations of these operations.
6.3 *Condition*—Material shall be furnished in the annealed or as rolled condition.

7. Chemical Requirements

57.1 The heat analysis shall conform to the chemical composition of Table 1. Ingot analysis may be used for reporting all chemical requirements, except hydrogen. Samples for hydrogen shall be taken from the finished mill product. Supplier shall not ship material with chemistry outside the requirements specified in Table 1.
57.1.1 Requirements for the major and minor elemental constituents are listed in Table 1. Also listed are important residual elements. Analysis for elements not listed in Table 1, is not required to verify compliance with this specification.
7.2 *Product Analysis*—Product analysis tolerances do not broaden the specified heat analysis requirements but cover variations between laboratories in the measurement of chemical content. The manufacturer shall not ship material that is outside the limits specified in Table 1. ~~P~~ The product analysis ~~limits~~ tolerances shall ~~be as specified~~ conform to the product tolerances in Table 2.

~~[8] Available from American Society for Quality; 161 West Wisconsin Avenue, Milwaukee, WI 53203.~~
[8] American National Standards Institute, 25 W. 43rd St., 4th Floor, New York, NY 10036.
~~[9] FDA 510K application number K903630, submitted by Howmedica, Inc., Rutherford, NJ.~~
[9] Available from the American Society for Quality, 600 N. Plankinton Ave., Milwaukee, WI 53203.

TABLE 1 Chemical Requirements

Element	Composition, ~~Weight~~ %, Mass/Mass	
	Min~~imum~~	Max~~imum~~
Molybdenum	10.0	13.0
Zirconium	5.0	7.0
Iron	1.5	2.5
~~Oxygen~~	~~0.08~~	~~0.28~~
Oxygen	0.008	0.28
Nitrogen	—	0.05
Carbon	—	0.05
Hydroge~~n~~	—	0.020
~~Titanium~~	~~Balance~~	~~Balance~~
Titanium[A]	balance	balance

[A]The percentage of titanium is determined by difference and need not be determined or certified.

TABLE 2 ~~Permissible Variation in Product Analysis (W~~ Toleight ~~%)~~rances[A]

Element	Tolerance Under the Minimum or Over the Maximum Limit[B]
Molybdenum	0.25
Zirconium over 4 to 6 % inclusive	0.20
Zirconium over 6 to 10 % inclusive	0.30
Iron	0.20
Oxygen up to 0.2 %	0.02
Oxygen over 0.2 %	0.03
Nitrogen	0.02
Carbon	0.002
Hydrogen	0.0002

[A]Refer to AMS 2249.
[B]Under the minimum limit not applicable for elements in which only a maximum percentage is indicated.

5~~7~~.2.1 The product analysis is either for the purpose of verifying the composition of a heat or lot or to determine variations in the composition within the heat.

5~~7~~.2.2 Acceptance or rejection of a heat or manufacturing lot of material may be made by the purchaser on the basis of this product analysis.

7.3 For reference purposes, use Test Methods E 120, E 1409, and E 1447 or other analytical methods agreed upon ~~by~~ between purchaser and ~~supplier shall be used.~~

~~5.4 Samples~~ supplier.

7.4 Ensure that the samples for chemical analysis ~~shall be~~ are representative of the material being tested. The utmost care must be used in sampling titanium for chemical analysis because of its affinity for elements such as oxygen, nitrogen, and hydrogen. ~~Therefore, in~~ In cutting samples for ~~analysis any contamination~~ analysis, therefore, the operation should be ~~avoided. Chips should be clean, and sharp cutting~~ carried out insofar as possible in a dust-free atmosphere. Cutting tools should be ~~used.~~ clean and sharp. Samples for analysis should be stored in suitable containers.

~~5.5 Requirements for the major and minor elemental constituents are listed in Table 1. Also listed are important residual elements. Analysis for elements not listed in Table 1 is not required to verify compliance with this specification.~~

~~6.~~

8. Mechanical Requirements

6~~8~~.1 ~~M~~The material supplied under this specification shall conform to the mechanical property requirements ~~given~~ in Table 3.

~~6.2 Perform~~

8.2 Specimens for tension tests shall be machi~~n~~ged and tested in accordance with Test Methods E 8. ~~Determine tensile~~ Tensile properties shall be determined using a strain rate of 0.003 to 0.007 in./in./min (mm/mm/min) through ~~the specified~~ yield ~~strength,~~ and then the cross~~-~~head speed ~~shall~~ may be increased so as to produce fracture in approximately ~~one additional minute.~~

~~6.3~~ 1 min.

8.3 *Number of Tests*~~—A~~—Perform a minimum of two tension tests ~~shall be made~~ from each lot. A lot is defined as the total number of mill products produced under the same conditions at essentially the same time. Should either of the two test specimens

TABLE 3 Solution-Annealed Mechanical Properties of Ti-12Mo-6Zr-2Fe Bar and Wire

Cond Size, in. (mm)	Ultimate Tensile Strength, min, psi (MPa)	Yield Strength (0.2 %), min, (0.2 % Offset), min, psi (MPa)	Elongation[B,A] in 2 in. or 4*D* or 4*W* min, %	Reduction in of Area, min. (%)
Solution[C] All Annealed	135 000 (931.5) 135 000 (931.5)	130 000 (897) 130 000 (897)	12 12	30 30

[A] Di Elongation of material 0.1062 in. (21.5475 mm) t o 1½ in. (38.1 mm).
[B] Gar gre later ingth = 4 × diameter.
[C] S (*D*) or width (*W*) shall be measutred usiong a gage length of 2 in. or 4*D* or 4*W*. The gage lieng sthall must be reported with the test results. The method for determining edlongat 1450 ± 25°F (788 ± 14°C) fiolln owed byf materapid qal uench to bdelow 3r 0.0°F62 in. (.°C1.575 mm), for a in diappmeter opr thickness maty be negotimated. Alternately, as r gagequi length corresponding to ISO 6892 may be usetd when agraieed upon between supplizer and me purchanical pser (5.65 $\sqrt{So}$, where *So* is ty rhequ original cross-semenctsional area).

not meet the specified requirements, test two additional test pieces representative of the same lot shall be tested in the same manner. The lot shall will be deemed to comply considered in compliance only if both additional test pieces meet the specified requirements. If a sample specimen fails outside the gage section, gage, the sample test is void null in accordance with Test Method E 8 and additional sample shall be tested.

7. Microstructure

7.1 The microstructure shall be fully recrystallized single phase beta microstructure after solution annealing. There shall be no continuous alpha network at prior beta grain boundaries. There shall be no alpha precipitates.

7.2 Grain size of the solution annealed barstock shall be five or finer based upon Test Methods E 112.

7.3 Products supplied with E 8, and a machined or ground surface finish retest shall have no alpha case. For other products, there shall be no continuous layer of alpha case when examined at 100X.

7.4 The beta transus temperature shall be determined by a suitable method and reported on the material certification for each heat.

8. Quality Program Requirements

8.1 The producer shall maintain a quality program, for example, such as defined in ASQ C1. performed.

9. Certification

9.1 A certificationSpecial Requirements

9.1 The microstructure shall be provided by fully recrystallized single-phase beta microstructure after solution annealing. The grain size in the manufacturer of annealed condition shall be 5 or finer based upon Test Methods E 112.

9.2 Determine the material that the material was manufactured beta transus temperature for each heat by a suitable method and tested in accordance with this specification. A report of on the test results shall be furnished at material certification if required by the purchaser.

9.3 Alpha case is not permitted for products supplied with a machined, ground, or chemically milled surface finish. For other products, there will be no continuous layer of alpha case whipen examined at 100× magnification.

10. Certification

10.1 The supplier shall provide a certification that the material was tested in accordance with this specification. A report of the test results shall be furnished to the purchaser at the time of shipment.

11. Quality Program Requirements

11.1 The producer shall maintain a quality program as defined in ASQ C1.

12. Keywords

102.1 metals (for surgical implants); orthopaedic medical devices; titanium alloys; titanium/titanium alloy; titanium/titanium titanium alloys (for surgical implants)

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 The purpose of this specification is to characterize the ~~composition~~ chemical, physical, mechanical, and metallurgical properties of wrought ~~annealed Titanium-12Molybdenum-6Zirconium-2Iron titanium~~ titanium-12 molybdenum-6 zirconium-2 iron alloy to ~~ensure consistency in the starting material~~ be used in the manufacture of ~~medical devices, in particular of~~ surgical implants.[2]

X1.2 The microstructural requirements contained in this ~~S~~ specification represent the current general consensus with respect to optimization of mechanical properties for implant applications.

X1.3 The minimum mechanical properties specified ~~assure~~ ensure a baseline of strength and ductility for the highly stressed devices for which ~~may be manufactured from~~ this alloy is typically used.

X1.4 The stress corrosion cracking resistance of this alloy is similar to that of ~~Titanium 6A1–4V~~ titanium-6 aluminum- 4 vanadium ELI alloy.[2]

X1.5 ISO standards are listed for reference only. Use of an ISO standard, in addition to or instead of a preferred ASTM standard, may be negotiated between the purchaser and supplier.

X2. BIOCOMPATIBILITY

X2.1 The suitability of this material from a human implant perspective is dependent on the specific application. The biological tests appropriate for the specific site, such as recommended in Practice F 748 should be used as a guideline. A summary of the in vitro and animal testing that has been performed as of the approval date of this specification is provided in X2.3.

X2.2 No known surgical implant material has ever been shown to be completely free of adverse reactions in the human body. The alloy composition covered by this specification, however, has been subjected to testing in laboratory animals, and has been used clinically since Feb. 6, 1998. The results of these studies indicate a well-characterized level of local biological response that is equal to or less than that produced by the reference material unalloyed titanium (see Specification F 67) that has a long history of successful clinical application in soft tissue and bone implants in humans.

X2.3 As of the time of the original approval of this specification, this titanium alloy material had a limited history of clinical use in humans. An extensive series of in- vitro and animal ~~tests~~ studies had been performed~~,~~ as ~~listed below,~~ follows, comparing the biological response to that of ~~the control material, Titanium 6Aluminum-4Vanadium ELI alloy.~~ a reference material. These tests were conducted to support the usage of this material in ~~specific~~ surgical implant devices. In all cases, the results indicated that this material was no more ~~bioreactive~~ reactive with the environment than ~~that of~~ the reference material.

X2.~~1~~3.1 Acute systemic toxicity by mouse ~~injection (See Practice F 750).~~
~~X2.1.2 Cytotoxicity~~ injection,[2]
X2.3.2 Cytotoxicity by agar ~~overlay (See Practice F 898).~~
~~X2.1.3 Hemolytic~~ overlay,[2]
X2.3.3 Hemolytic potential by direct exposure~~.~~,[2]
X2.~~1~~3.4 Dermal sensitization by guinea pig maximization~~.~~,[2]
X2.~~1~~3.5 Mutagenicity by the Ames test (saline and DMSO extracts)~~.~~,
X2.~~1~~3.6 Response to particulate debris (release of IL-1, IL-6 and PGE^2 in cell culture and ex-vivo histology ~~rabbits).~~

~~X2.2 The suitability of this material, from a biological performance perspective is dependent on the specific site. For new functional applications~~ rabbits).[2]

SUMMARY OF CHANGES

(*1*) Added 6.3 which allows for ~~this~~ material ~~it may be advisable~~ to ~~undertake some additional biologic tests appropriate for the specific site, such as recommended in Practice F 748.~~

~~X2.3 No known surgical implant material has ever been shown to~~ be ~~completely free of adverse reactions~~ supplied in the ~~human body. However, the alloy composition covered by this standard has~~ as rolled condition.

(*2*) Editorial corrections have been ~~subjected~~ made to ~~testing in laboratory animals. The results of these studies indicate a well characterized level of biological response that is equal to or less than that produced by the reference material Titanium-6A1-4V ELI alloy which has a long history of successful clinical application in soft tissue~~ meet terminology and ~~bone implants in humans.~~ formatting guidelines established for implant material standards.