Designation: F1813 – 13

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

This standard is issued under the fixed designation F1813; 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 chemical, mechanical, and metallurgical requirements for wrought titanium-12molybdenum-6zirconium-2iron alloy mill products to be used in the manufacture of surgical implants.[2]

1.2 The values stated in either SI units or inch-pound units are to be regarded separately as standard. The values stated in each system may not be exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in non-conformance with the standard.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

E8/E8M Test Methods for Tension Testing of Metallic Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E112 Test Methods for Determining Average Grain Size

E1409 Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Technique

E1447 Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by Inert Gas Fusion Thermal Conductivity/Infrared Detection Method

E1941 Test Method for Determination of Carbon in Refractory and Reactive Metals and Their Alloys by Combustion Analysis

E2371 Test Method for Analysis of Titanium and Titanium Alloys by Atomic Emission Plasma Spectrometry (Withdrawn 2013)[4]

E2626 Guide for Spectrometric Analysis of Reactive and Refractory Metals

F67 Specification for Unalloyed Titanium, for Surgical Implant Applications (UNS R50250, UNS R50400, UNS R50550, UNS R50700)

F748 Practice for Selecting Generic Biological Test Methods for Materials and Devices

IEEE/ASTM SI 10 American National Standard for Metric Practice

2.2 *Aerospace Materials Specification:*[5]

AMS 2249 Chemical Check Analysis Limits, Titanium and Titanium Alloys

AMS 2631 Ultrasonic Inspection—Titanium and Titanium Alloy Bar, Billet and Plate

AMS 2380 Approval and Control of Premium-Quality Titanium Alloys

2.3 *ISO Standards:*[6]

ISO 6982 Metallic Materials Tensile Testing at Ambient Temperature

ISO 9001 Quality Management Standard

3. Terminology

3.1 *Definitions of 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.

3.1.2 *hot work, n*—any mechanical deformation process performed above the recrystallization temperature.

3.1.3 *lot, n*—the total number of mill products produced from the same melt heat under the same conditions at essentially the same time.

[1] This specification is under the jurisdiction of ASTM Committee F04 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.12 on Metallurgical Materials.

Current edition approved March 1, 2013. Published April 2013. Originally approved in 1997. Last previous edition approved in 2006 as F1813 – 06. DOI: 10.1520/F1813-13.

[2] FDA 510K application number K903630.

[3] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[4] The last approved version of this historical standard is referenced on www.astm.org.

[5] Available from Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001, http://www.sae.org.

[6] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

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

3.1.4 *solution anneal, v*—to heat treat in order to remove precipitates.

4. Product Classification

4.1 *Bar*—Rounds, flats or other shapes from 0.188 in. [4.76 mm] to 4.0 in. [102 mm] in diameter or thickness. (Other sizes and shapes by special order.)

4.2 *Forging Bar*—Bar as described in 4.1 used in the production of forgings. This product may be furnished in the hot worked condition.

4.3 *Wire*—Rounds, flats, or other shapes less than 0.188 in. [4.76 mm] in diameter or thickness.

5. Ordering Information

5.1 Include with inquiries and orders for material under this specification the following information:

5.1.1 Quantity,

5.1.2 ASTM designation and date of issue,

5.1.3 Form (strip, sheet, plate, bar, forging bar or wire),

5.1.4 Condition (see 6.2),

5.1.5 Mechanical properties (if applicable for special conditions),

5.1.6 Finish (see 6.1),

5.1.7 Applicable dimensions including size, thickness, width, length or drawing number,

5.1.8 Special tests, (if any), and

5.1.9 Other requirements.

6. Materials and Manufacture

6.1 *Finish*—The mill product may be supplied as specified by the purchaser with a descaled or pickled, abrasive blasted, chemically milled, ground, machined, peeled, or polished finish. On billets, bars, plates and forgings, it is permissible to remove minor surface imperfections by grinding if the resultant area meets the dimensional and surface finish requirements of this specification.

6.2 *Condition*—Material shall be furnished in the solution annealed or hot worked condition.

7. Chemical Requirements

7.1 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. The supplier shall not ship material with chemistry outside the requirements specified in Table 1.

7.1.1 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:*

7.2.1 Product analysis tolerances do not broaden the specified heat analysis requirements but cover variations in the measurement of chemical content between laboratories. The product analysis tolerances shall conform to the product tolerances in Table 2.

7.2.2 The product analysis is either for the purpose of verifying the composition of a heat or manufacturing lot or to determine variations in the composition within the heat.

7.2.3 Acceptance or rejection of a heat or manufacturing lot of material may be made by the purchaser on the basis of this product analysis. Product analysis outside the tolerance limits allowed in Table 2 are cause for rejection of the product. A referee analysis may be used if agreed upon by the supplier and purchaser.

7.2.4 For referee purposes, use Test Methods E2371, E1409, E1941, E2626, and E1447 or other analytical methods agreed upon between the purchaser and the supplier.

7.3 Samples for chemical analysis shall be 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. In cutting samples for analysis, therefore, the operation should be carried out insofar as possible in a dust-free atmosphere. Cutting tools should be clean and sharp. Samples for analysis should be stored in suitable containers.

8. Mechanical Requirements

8.1 The material supplied under this specification shall conform to the mechanical property requirements in Table 3. Alternative properties may be agreed upon between the purchaser and supplier.

8.2 Specimens for tension tests shall be prepared and tested in accordance with Test Methods E8/E8M. Tensile properties shall be determined using a strain rate of 0.003 to 0.007

TABLE 1 Chemical Requirements

Element	Composition % mass/mass	
	Min	Max
Nitrogen	—	0.05
Carbon	—	0.05
Hydrogen	—	0.020
Iron	1.5	2.5
Oxygen	0.008	0.28
Molybdenum	10.0	13.0
Zirconium	5.0	7.0
Titanium	Balance[A]	

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

TABLE 2 Product Analysis Tolerances[A]

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

[A] Refer to AMS 2249.

[B] Under the minimum limit not applicable for elements where only a maximum percentage is indicated.

TABLE 3 Mechanical Properties—Bar and Wire

Condition[A]	Ultimate Tensile Strength min, psi [MPa]	Yield Strength (0.2 % offset), min, psi [MPa]	Elongation[B] in 2 in. [50 mm], 4*D* or 4*W* min %	Reduction of Area min, %
Solution annealed	135 000 [931.5]	130 000 [897]	12	30

[A] Mechanical properties for conditions other than those listed in this table may be established by agreement between the supplier and purchaser.
[B] Elongation of material 0.063 in. [1.6 mm] or greater in diameter (*D*) or width (*W*) shall be measured using a gauge length of 2 in. or 4*D* or 4*W*. The gauge length shall be reported with the test results. The method for determining elongation of material under 0.063 in. [1.6 mm] in diameter or thickness may be negotiated. Alternately, a gauge length corresponding to ISO 6892 may be used when agreed upon between supplier and purchaser (5.65 times the square root of *So*, where *So* is the original cross sectional area).

in./in./min [mm/mm/min] through yield and then the crosshead speed may be increased so as to produce fracture in approximately one additional minute.

8.2.1 *Bar, Forging Bar, and Wire*—Test according to Test Methods E8/E8M. Perform at least one tension test from each lot in the longitudinal direction.

8.2.2 Tensile tests results for which any specimen fractures outside the gauge length shall be considered valid, if both the elongation and reduction of area meet the minimum requirements specified. If either the elongation or reduction of area is less than the minimum requirement, invalidate the specimen and retest. Retest one specimen for each invalidated specimen.

8.2.3 Should any test specimen not meet the specified requirements (except as noted in 8.2.2), test two additional test pieces representative of the same lot, in the same manner, for each failed test specimen. The lot will be considered in compliance only if all additional test pieces meet the specified requirements.

9. Dimensions and Permissible Variation

9.1 *Units of Measure:*

9.1.1 *Selection*—This specification requires that the purchaser selects the units (SI or inch-pound) to be used for product certification. In the absence of a stated selection of units on the purchase order, this selection may be expressed by the purchaser in several alternate forms listed in order of precedence.

9.1.1.1 If the purchaser and supplier have a history of using specific units, these units shall continue to be certified until expressly changed by the purchaser.

9.1.1.2 In the absence of historic precedence, if the units used to define the product on the purchaser's PO, specification, and engineering drawing are consistent, these units shall be used by the supplier for the product certification.

9.1.1.3 If the purchaser's selection of units is unclear, the units of measure shall be agreed upon between the purchaser and supplier.

9.1.2 *Conversion of Units*—If the supplier's test equipment does not report in the selected units, the test equipment units may be converted to the selected units for certification purposes. Accurate arithmetic conversion and proper use of significant digits should be observed when performing this conversion. IEEE/ASTM SI 10 provides guidelines for the use of SI units. Annex A provides conversion tables and Annex B provides rules for conversion and significant digits.

10. Special Requirements

10.1 The microstructure shall consist of a fully recrystallized beta phase structure. Primary alpha and alpha prime (also known as martensitic alpha) are not permitted in the microstructure when viewed at 100× magnification. The grain size in the annealed condition shall be 5 or finer based upon Test Methods E112.

10.2 Determine the beta transus temperature for each heat by a suitable method and report on the material certification if required by the purchaser.

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

10.4 All centerless ground or peeled and polished round bar ≥0.375 in. [9.5 mm] in nominal diameter shall be ultrasonically inspected at final diameter according to AMS 2631 Class A1. Equivalent test methods may be substituted when agreed upon by the purchaser and supplier

NOTE 1—AMS 2631 contains varying flat bottom hole (FBH) requirements based on melting grades per AMS 2380. Since the FBH requirement for Class A1 is the same regardless of the melting grade, it is not necessary to specify the melting grade.

11. Significance of Numerical Limits

11.1 The following applies to all specified numerical limits in this specification. To determine conformance to these limits, an observed or calculated value shall be rounded to the nearest unit in the last right hand digit used in expressing the specification limit, in accordance with the rounding method of Practice E29.

12. Certification

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

12.2 Gauge length shall be reported with elongation.

13. Quality Program Requirements

13.1 The supplier shall maintain a quality program such as defined in ISO 9001 or similar quality program.

14. Keywords

14.1 metals (for surgical implants); orthopaedic medical devices; titanium alloys; titanium alloys (for surgical implants)

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

NOTE X1.1—Choose the appropriate paragraphs for your alloy and product forms.

X1.1 The purpose of this specification is to characterize the chemical, mechanical, and metallurgical properties of wrought titanium-12molybdenum-6zirconium-2iron alloy to be used in the manufacture of surgical implants.

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

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

X1.4 The stress corrosion cracking resistance of this alloy is similar to that of titanium-6aluminum-4vanadium ELI alloy.

X1.5 ISO Standards are listed for reference only. Although standards listed in 2.3 are similar to the corresponding ASTM standards, they are not identical. Use of the ISO standard instead of the preferred ASTM standards may be agreed upon between the purchaser and supplier.

X1.6 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 material is usually multiple melted in arc furnaces (including furnaces such as plasma arc and electron beam) of a type conventionally used for reactive metals.

X1.7 *Units of Measure, ASTM Policy*—ASTM is promoting the use of rationalized SI (metric) units in their standards. The F04.12 Committee has modified this specification to facilitate the transition by the medical materials industry to SI between now and 2018. In the first phase of this transition, running to 2013, the specifications will be structured to allow the use of either SI or inch-pound units. The choice of primary units in each specification will be determined by the industry using the specification. The change to SI units during this period may be initiated by the purchaser through his purchase documentation. In the second phase of this transition, the specifications shall be written with SI as the primary units. Harmonization with corresponding ISO documents should be considered when assigning the SI values.

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 F748 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 for more than a decade (if appropriate). 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 F67) 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 studies had been performed as follows, comparing the biological response to that of a reference material. These tests were conducted to support the usage of this material in surgical implant devices In all cases, the results indicated that this material was no more reactive with the environment than the reference material.

X2.3.1 Acute systemic toxicity by mouse injection,[2]

X2.3.2 Cytotoxicity by agar overlay,[2]

X2.3.3 Hemolytic potential by direct exposure,[2]

X2.3.4 Dermal sensitization by guinea pig maximization,[2]

X2.3.5 Mutagenicity by the Amestest (saline and DMSO extracts),[2] and

X2.3.6 Response to particulate debris (release of IL-1, IL-6, and PGE_2 in cell culture and ex-vivo histology rabbits).[2]

SUMMARY OF CHANGES

Committee F04 has identified the location of selected changes to this standard since the last issue (F1813 – 06) that may impact the use of this standard.

(1) Editorial corrections have been made in order to meet terminology and formatting guidelines established for implant material standards within F04.12.
(2) Added F04.12 wording allowing independent SI and Inch Pound units to 1.2.
(3) Updated referenced documents.
(4) Added terminology.
(5) Deleted reference to ASQ C1 quality method.
(6) Changed language to "prepared" from "machined" for tension tests in 8.2.
(7) Added UT requirement in 10.4.
(8) Corrected the Product Analysis Tolerances for carbon and hydrogen.