Designation: F 1713 – 96

Standard Specification for Wrought Titanium-13Niobium-13Zirconium Alloy for Surgical Implant Applications[1]

This standard is issued under the fixed designation F 1713; 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 titanium13niobium-13zirconium alloy to be used in the manufacture of surgical implants **(1)**.[2]

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

2. Referenced Documents

2.1 *ASTM Standards:*

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

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

E 1409 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[4]

F 748 Practice for Selecting Generic Biological Test Methods for Materials and Devices[5]

F 981 Practice for Assessment of Compatibility of Biomaterials for Surgical Implants with Respect to Effect of Materials on Muscle and Bone[5]

F 1472 Specification for Wrought TI-6AI-4V Alloy for Surgical Implant Applications[5]

2.2 *Other Standards:*

ASOC C1 Specification of General Requirements for a Quality Control Program[6]

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

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *capability aged, n*—the condition of the material that is obtained if, following solution treatment, a sample of the mill product is subjected to an aging treatment such as given below, for certification testing.

3.1.1.1 Ramp up from room temperature to 923 ± 25°F (495 ± 14°C) over 2.5 ± 1 h.

3.1.1.2 Age for 6 ± 0.25 h at 923 ± 25°F (495 ± 14°C).

3.1.1.3 Remove from furnace and air cool to room temperature.

3.1.2 *solution treated, n*—the condition of the material that is obtained if, following the final hot-working or cold-working operation, the mill product is rapidly quenched, for example, by water quenching, from a temperature above 1112°F (600°C).

3.1.3 *unannealed, n*—the condition of the material that is obtained after the normal hot-working or cold-working operation used for fabrication of the mill product. There are no subsequent heat treatment requirements.

4. Product Classification

4.1 *Strip*—Any product under 0.1875 in. (4.75 mm) in thickness and under 24 in. (610 mm) wide.

4.2 *Sheet*—Any product under 0.1875 in. (4.75 mm) in thickness and 24 in. (610 mm) or more in width.

4.3 *Plate*—Any product 0.1875 in. (4.75 mm) thick and over and 10 in. (254 mm) wide and over, with widths greater than five times thickness. Plate up to 4 in. (101.60 mm), thick inclusive is covered by this specification.

4.4 *Bar*—Rounds or flats from 3⁄16 in. (4.75 mm) to 4 in. (101.60 mm) in diameter or thickness. (Other sizes and shapes by special order.)

4.5 *Wire*—Rounds less than 3⁄16 in. (75 mm) in diameter.

5. Ordering Information

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

5.1.1 Quantity (weight or number of pieces),

5.1.2 Applicable ASTM designation,

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

5.1.4 Condition (see Section 3 and 6.1),

5.1.5 Mechanical properties (if applicable, for special conditions),

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[1] This specification is under the jurisdiction of ASTM Committee F-4 on Medical and Surgical Materials and Devices and is the direct responsibility of Subcommittee F04.12 on Metallurgical Materials.

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[2] The boldface numbers in parentheses refer to the list of references at the end of the text.

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

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

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

[6] Available from the American Society for Quality Control, 161 W. Wisconsin Ave., Milwaukee, WI 53203.

[7] Available from the Society of Automotive Engineers, 400 Commonwealth Drive, Warrendale, PA 15096.

5.1.6 Finish (see 6.2),

5.1.7 Applicable dimensions including size, thickness, width, or print number,

5.1.8 Special tests, and

5.1.9 Special 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 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.

6.2 *Finish*—The mill product may be furnished to the implant manufacturer as descaled or pickled, sandblasted, ground, machined, or combinations of these operations.

7. Chemical Requirements

7.1 Ensure that the heat analysis conforms to the chemical composition of Table 1. Ingot analysis may be used for reporting all chemical requirements, except hydrogen. Take samples for hydrogen from the finished mill product.

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 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. Ensure that the product analysis tolerances conform to the check tolerances of Table 2.

7.3 For referee purposes, use Methods E 120 or other analytical methods agreed upon between the purchaser and the supplier.

7.4 Ensure that the samples for chemical analysis 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. In cutting samples for analysis, therefore, the operation should be carried out insofar as possible in a dust-free atmosphere. Chips should be clean and sharp. Samples for analysis should be stored in suitable containers.

8. Mechanical Requirements

8.1 Ensure that the material supplied under this specification conforms to the mechanical property requirements given in Table 3.

8.2 Specimens for tension tests shall be machined and tested in accordance with Test Methods E 8. 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 be increased so as to produce fracture in approximately one additional minute.

8.3 *Number of Tests*—Perform a minimum of two tension tests 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 not meet the specified requirements, test two additional test pieces representative of the same lot in the same manner. The lot will be considered in compliance only if both additional test pieces meet the specified requirements. If a specimen fails outside the gage, the test is null in accordance with Methods E 8, and a retest shall be performed.

TABLE 1 Chemical Requirements

Element	Composition, %
Nitrogen, max	0.05
Carbon, max	0.08
Hydrogen, max	0.012[A]
Iron, max	0.25
Oxygen, max	0.15
Niobium	12.5–14.0
Zirconium	12.5–14.0
Titanium[B]	balance

[A] Material 0.032 in. (0.813 mm) and under may have hydrogen content up to 0.015 %.

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

TABLE 2 Product Analysis Tolerance[A]

Element	Tolerance Under the Minimum or Over the Maximum Limit[B]
Nitrogen	0.02
Carbon	0.02
Hydrogen	0.0020
Iron	0.10
Oxygen	0.02
Niobium	0.30
Zirconium	0.40

[A] Refer to AMS 2249.

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

9. Special Requirements

9.1 Ensure that the microstructure is martensitic with finely dispersed alpha or beta phases, or both. The alpha or beta phases, or both, may be too fine to be visible metallographically but must be present to ensure adequate strength. No continuous alpha network at prior beta grain boundaries will be present. The microstructure within the prior beta grain boundaries will be acicular. Perform metallographic evaluation in the aged condition.

9.2 Determine the beta transus temperature for each heat by a suitable method and reported on the materials certification, if required by the purchaser.

9.3 Products supplied with a machined or ground surface finish will have no alpha case. For other products, there will be

TABLE 3 Mechanical Properties[ABC]

Condition	Tensile Strength min, psi (MPa)	Yield Strength (0.2 % offset), min psi (MPa)	Elongation in 4d or 4w min, %[D]	Reduction of Area min, %[E]
Capability aged	125 000 (860)	105 000 (725)	8	15
Solution treated	80 000 (550)	50 000 (345)	15	30
Unannealed	80 000 (550)	50 000 (345)	8	15

[A] Up to 4 in. (101.60 mm) including thickness or diameter.

[B] Mechanical property requirements apply to both longitudinal and transverse directions in plate, sheet and strip only.

[C] Solution treated or unannealed material is not intended for use as a final product without subsequent hot working or heat treatment, or both.

[D] Measure elongation on material under 0.187 in. (4.75 mm) in thickness or diameter over a 2 in. gage length. Elongation on material under 0.032 in. (0.813 mm) in thickness may be obtained by negotiation.

[E] Applies to bar and plate only.

no continuous layer of alpha case, when examined at 100 ×.

10. Certification

10.1 The manufacturer will provide a certification of the material that the material was manufactured and tested in accordance with this specification. A report of test results will be furnished at the time of shipment.

11. Quality Program Requirements

11.1 The producer will maintain a quality program, such as is defined in Specification ASQC C1.

12. Keywords

12.1 metals (for surgical implants)–titanium alloys; orthopaedic medical devices—titanium/titanium alloy; titanium/titanium alloys (for surgical implants)

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 The purpose of this specification is to characterize the chemical, physical, mechanical, and metallurgical properties of wrought titanium-13niobium-13zirconium alloy to be used in the manufacture of surgical implants.

X1.2 The microstructural requirements contained in this specification represent the 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 standard grade titanium-6aluminum4vanadium alloy **(2)**.

X2. BIOCOMPATIBILITY

X2.1 The suitability of this material from a human implant perspective is dependent on the specific application. The biologic 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 June 1994. 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 titanium-6aluminum-4 vanadium alloy (see specification F 1472) 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 **(3,4,5,6)**, as listed as follows, comparing the biological response to that of a reference material, titanium-6aluminum-4vanadium alloy. These tests were conducted to support the usage of this material in surgical implant devices **(6,7,8,9)**. In all cases, the results indicated that this material was no more reactive with the environment than the reference material.

X2.3.1 L929 MEM-Cytotoxicity (Mouse Fibroblasts),

X2.3.2 Sensitization Assay (Kligman Maximization Study),

X2.3.3 Rabbit Pyrogen Test,

X2.3.4 Mammalian Mutagenicity Test (Rodent Bone Marrow Micronucleus Test),

X2.3.5 Rabbit Intramuscular Implantation Test,

X2.3.6 Rabbit Blood Hemolysis Test,

X2.3.7 Ames Mutagenicity Assay, and

X2.3.8 Systemic Toxicity and Irritation Test (USP XXII Biological Test).

REFERENCES

(**1**) Mishra, A.K., Davidson, J.A., Kovacs, P., and Poggie, R.A., "Ti13Nb-13Zr: A New Low Modulus, High Strength, Corrosion Resistant, Near Beta Alloy for Orthopaedic Implants," Beta Titanium Alloys in the 1990's, D. Eylon, R.R. Boyer, and D.A. Koss, eds., The Minerals, Metals and Materials Society, Warrendale, PA, 1993, pp. 61–72.

(**2**) Mishra, A.K., and Davidson, J. A., "Stress Corrosion Cracking Resistance of Ti-13Nb-13Zr," *Annals of Biomedical Engineering*, Vol 21(1), 1993, pp. 67.

(**3**) Mishra, A.K., Davidson, J.A., Kovacs, P., and Poggie, R.A., "Mechanical, Tribological and Electrochemical Behavior of Ti13Nb-13Zr–A New, Low Modulus Titanium Alloy for Orthopaedic Implants," Proc. 12th S. Biomedical Eng. Conf., New Orleans, LA, April 2–4, 1993, IEEE, Piscataway, NJ, 1993, pp. 13–15.

(**4**) Goodman, S.B., Davidson, J.A., Fornasier, V.L., and Mishra, A.K., "Histological Response to Cylinders of a Low Modulus Titanium Alloy (Ti-13Nb-13Zr) and a Wear Resistant Zirconium Alloy (Zr-2.5Nb) Implanted in the Rabbit Tibia," *Journal of Applied Biomaterials*, Vol 4, 1993, pp. 331–339.

(**5**) Kovacs, P., and Davidson, J.A., "The Electrochemical Behavior of a New Titanium Alloy with Superior Biocompatibility," Titanium '92: Science and Technology, F.H. Froes and I. Caplan, eds., The Minerals, Metals and Materials Society, Warrendale, PA, 1993, pp. 2705–2712.

(**6**) FDA 510(k) No. K914343.

(**7**) FDA 510(k) No. K930480.

(**8**) FDA 510(k) No. K936233.

(**9**) FDA 510(k) No. K943523.