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Standard Specification for Wrought Titanium-13Niobium-13Zirconium Alloy for Surgical Implant Applications (UNS R58130)[1]

This standard is issued under the fixed designation F1713; 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-13niobium-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 standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.

2. Referenced Documents

2.1 *ASTM Standards:*[3]

E8 Test Methods for Tension Testing of Metallic Materials

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

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E2371 Test Method for Analysis of Titanium and Titanium Alloys by Atomic Emission Plasma Spectrometry (Withdrawn 2013)[4]

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

F1472 Specification for Wrought Titanium-6Aluminum-4Vanadium Alloy for Surgical Implant Applications (UNS R56400)

2.2 *Aerospace Material Specification:*

AMS 2249 Chemical Check Analysis Limits, Titanium and Titanium Alloys[5]

2.3 *American Society for Quality (ASQ) Standard:*

ASQ C1 Specifications of General Requirements for a Quality Program[6]

2.4 *ISO Standards:*

ISO 5832-3 Implants for Surgery—Metallic Materials—Part 3: Wrought Titanium 6-Aluminium 4-Vanadium Alloy[7]

ISO 6892 Metallic Materials Tensile Testing at Ambient Temperature[7]

ISO 9001 Quality Management Systems—Requirements[7]

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 *capability-aged, adj*—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.

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

3.1.2.2 Remove from furnace and air cool to room temperature.

3.1.3 *cold work, n*—any mechanical deformation process performed below the recrystallization temperature which results in strain hardening of the material.

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

3.1.5 *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.

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

[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 Society for Quality (ASQ), 600 N. Plankinton Ave., Milwaukee, WI 53203, http://www.asq.org.

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

3.1.6 *solution-treated, adj*—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).

3.1.7 *unannealed, adj*—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 *Bar*—Rounds or flats from 0.188 in. (4.76 mm) to 4 in. (101.6 mm), inclusive, in diameter or thickness. (Other sizes and shapes by special order.)

4.2 *Wire*—Rounds or flats 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 (weight or number of pieces);

5.1.2 Applicable ASTM designation and year of issue;

5.1.3 Form (wire or bar, see Section 4);

5.1.4 Condition (see 6.2);

5.1.5 Mechanical properties (if applicable, for special conditions) (see 8.1);

5.1.6 Finish (see 6.1);

5.1.7 Applicable dimensions including size, diameter, thickness (for rectangular wire), or print 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 bars, 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 unannealed, solution-treated, or capability-aged condition, as specified in the purchase order. Conditions and mechanical properties other than those listed in Table 3 may be established by agreement between the supplier and the purchaser.

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.

TABLE 1 Chemical Requirements

Element	Composition (% mass/mass)
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 (% mass/mass)[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.

TABLE 3 Mechanical Properties[A,B]

Condition	Tensile Strength min, psi (MPa)	Yield Strength (0.2 % offset), min psi (MPa)	Elongation min, %[C]	Reduction of Area min, %[D]
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) inclusive diameter.
[B] Solution treated or unannealed material is not intended for use as a final product without subsequent hot working or heat treatment, or both.
[C] Limits apply to tests taken both longitudinal and transverse to the direction of rolling. Elongation of material 0.063 in. (1.575 mm) or greater in diameter (D) or thickness (T) shall be measured using a gage length of 2 in. or 4D or 4T. The gage length shall be reported with the test results. The method for determining elongation of material under 0.063 in. (1.575 mm) in diameter or thickness may be negotiated. Alternately, a gage length corresponding to ISO 6892 may be used when agreed upon between the supplier and purchaser. (5.65 square root S_o, where S_o is the original cross sectional area.)
[D] Applies to bar only.

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 analyses. Product analysis outside the tolerance limits allowed in Table 2 shall be cause for rejection of the product. A referee analysis may be used if agreed upon by supplier and purchaser.

7.2.4 For referee purposes, use Test Methods E1409, E1447, E1941, and E2371 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 shall 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 machined and tested in accordance with Test Methods E8. Tensile properties shall be determined using a strain rate of 0.003 to 0.007 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.

8.3 *Number of Tests:*

8.3.1 *Bar and Wire*—Perform at least one tension test from each lot in the longitudinal direction. Should the test result not meet the specified requirements, test two additional test pieces representative of the same lot, in the same manner, for each failed test piece. The lot shall be considered in compliance only if all additional test pieces meet the specified requirements.

8.3.2 Tensile tests results for which any specimen fractures outside the gage length shall be considered acceptable, 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, discard the test and retest. Retest one specimen for each specimen that did not meet the minimum requirements.

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 shall 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 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, when examined at 100×.

10. Significance of Numerical Limits

10.1 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.

11. Certification

11.1 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. Quality Program Requirements

12.1 The producer shall maintain a quality program, such as is defined in ASQ C1, ISO 9001, or similar.

13. Keywords

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

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 The purpose of this specification is to characterize the chemical, 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-6 aluminum-4 vanadium ELI alloy (**2**).

X1.5 ISO standards are listed for reference only. Although the ISO 5832-3 Standard listed in section 2.4 is 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 multiple melted in arc furnaces (including furnaces such as plasma arc and electron beam) of a type conventionally used for reactive metals.

X2. BIOCOMPATIBILITY

X2.1 The suitability of this material from a human implant perspective depends on the specific application. The biologic 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 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 F1472) 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 (**3-6**), 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 (**6-9**). 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).

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(2) Mishra, A. K., and Davidson, J. A., "Stress Corrosion Cracking Resistance of Ti-13Nb-13Zr," *Annals of Biomedical Engineering*, Vol 21, No. 1, 1993, p. 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.