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Standard Specification for Wrought Zirconium-2.5Niobium Alloy for Surgical Implant Applications (UNS R60901)[1]

This standard is issued under the fixed designation F2384; 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 zirconium-2.5niobium alloy to be used in the manufacture of surgical implants (**1**).[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]

B550/B550M Specification for Zirconium and Zirconium Alloy Bar and Wire

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

E1552 Test Method for Determining Hafnium in Zirconium and Zirconium Alloys By Direct Current Plasma—Atomic Emission Spectrometry

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

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

SI 10 American National Standard for Use of the International System of Units (SI): The Modern Metric System

2.2 *ISO Standard:*[4]

ISO 6892 Metallic Materials Tensile Testing at Ambient Temperature

ISO 9001 Quality Management Systems—Requirements

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *annealed, adj*—material that exhibits a recrystalized grain structure.

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

4. Product Classification

4.1 *bar*—rounds, flats or shapes from 4.76 to 101.60 mm [0.1875 to 4 in.] in diameter or thickness (other sizes and shapes by special order).

4.2 *wire*—rounds or flats less than 4.76 mm [0.1875 in.] 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 Units to be certified—SI or inch-pound,

5.1.4 Grade (if applicable),

5.1.5 Form (bar, or wire),

5.1.6 Condition (see 6.3),

5.1.7 Mechanical properties (if applicable for special conditions),

5.1.8 Finish (see 6.2),

5.1.9 Applicable dimension including size, thickness, width, or drawing number,

5.1.10 Special tests, if any, and

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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] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036.

5.1.11 Other requirements.

6. Materials and Manufacture

6.1 Materials covered by this specification shall be produced by multiple vacuum melting in arc furnaces, electron beam melting, or other melting processes conventionally used for reactive metals.

6.2 *Finish*—The mill product may be furnished to the implant manufacturer as descaled or pickled, abrasively blasted, chemically milled, ground, machined, peeled, polished, or as specified by the purchaser.

6.3 *Condition*—Barstock shall be furnished in the annealed condition unless otherwise specified.

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, oxygen, and nitrogen. Samples for hydrogen, oxygen and nitrogen shall be taken from the finished mill product. The supplier shall not ship material with chemistry outside the requirements specified in Table 1. Guide E2626 may be used as a guide for chemical analysis techniques.

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 between laboratories in the measurement of chemical content. 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.

7.3 For referee purposes, use Test Method E1552 and E1941 or other analytical methods, as agreed upon between the purchaser and the supplier.

TABLE 1 Chemical Requirements

Element	Composition % mass/mass	
	min	max
Niobium	2.40	2.80
Oxygen	0.09	0.13
Carbon	...	0.027
Chromium	...	0.020
Hafnium	...	0.010
Hydrogen	...	0.0025
Iron	...	0.15
Nitrogen	...	0.0080
Tin	...	0.0050
Zirconium	balance[A]	balance[A]

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

TABLE 2 Product Analysis Tolerances

Alloying Element	Permissible Variation from the Specified Range, % mass/mass
Niobium	0.050
Oxygen	0.020
Carbon	0.002
Chromium	0.002
Hafnium	0.002
Hydrogen	0.0005
Iron	0.002
Nitrogen	0.0016
Tin	0.001

7.4 The samples for chemical analysis shall be representative of the material being tested. The utmost care must be used in sampling zirconium 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.

8.2 Specimens for tension tests shall be machined from bar in the longitudinal direction and tested in accordance with Test Methods E8/E8M. 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*—Perform a minimum of two tension tests from each lot (see 3.1.2). 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.

8.4 Tension test results for which any specimen fractures outside the gage length shall be considered acceptable, if both the elongation and reduction of area meets the minimum requirements specified. Refer to Test Methods E8/E8M, sections 7.11.4 and 7.11.5. If either the elongation or reduction of area is less than the minimum requirement, discard the test and

TABLE 3 Mechanical Properties[A]

Condition	Tensile Strength, min, MPa [psi]	Yield Strength (0.2 % offset), min, MPa [psi]	Elongation[B] in 2 in. or 4D or 4W, min, %
Annealed	450 [65 000]	310 [45 000]	15

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

retest. Retest one specimen for each specimen that did not meet the minimum requirements.

9. Significance of Numerical Limits

9.1 The following applies to all specified limits in this specification: For purposes of determining conformance with these specifications, an observed value 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.

10. Units of Measure

10.1 *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.

10.1.1 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.

10.1.2 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 product certification

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

10.2 *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. 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.

11. Special Requirements

11.1 The microstructure shall be a fine dispersion of the alpha and beta phases resulting from processing in the alpha plus beta field. There shall be no coarse, elongated alpha platelets. The average grain size of forgings shall be ASTM No. 8 or finer when tested in accordance with Test Methods E112.

12. Certification

12.1 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.

13. Quality Program Requirements

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

14. Keywords

14.1 metals (for surgical implants); orthopaedic medical devices; zirconium alloys; zirconium 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 zirconium-2.5niobium alloy to be used in the manufacture of surgical implants.

X1.2 ISO standards are listed for reference only. Use of an ISO standard in addition to or instead of a preferred ASTM standard may be agreed upon between the purchaser and supplier.

X1.3 This zirconium alloy is based on Specification B550/B550M, Grade R60705, and has been used extensively in the chemical industry since the 1970's.

X1.4 *Units of Measure*:

X1.4.1 *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 will be written with SI as the primary units.

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 since January 1996 (**2-4**). 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 zirconium 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 (**1,5**). 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) Davidson, J. A., Asgian, C. M., Mishra, A. K., and Kovacs, P., "Zirconia (ZrO_2)-coated Zirconium-2.5Nb Alloy for Prosthetic Knee Bearing Applications," *Bioceramics 5*, T. Yamamuro, T. Kokubo, and T. Nakamura (eds.), Kobunshi Kankokia, Kyoto, Japan, 1992, pp. 389-401.

(2) FDA 510(k) No. K914878.

(3) FDA 501(k) No. K934353.

(4) FDA 501(k) No. K962557.

(5) 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," *J. Appl. Biomater*., 4 (4), 1993, pp. 331-339.