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Standard Specification for Wrought Nickel-Titanium Shape Memory Alloys for Medical Devices and Surgical Implants[1]

This standard is issued under the fixed designation F2063; 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, physical, mechanical, and metallurgical requirements for wrought nickel-titanium bar, flat rolled products, and tubes containing nominally 54.5- to 57.0-weight percent nickel and used for the manufacture of medical devices and surgical implants.

1.2 Requirements are for mill product, measuring 5.50 to 94.0 mm [0.218 to 3.70 in.] in diameter or thickness. Mill product is not intended to have the final shape, final surface finish, or final properties of the medical device, implant, or their components. Finished NiTi cold-worked tube should be considered under Specification F2633.

1.3 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.

1.4 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards*:[2]

E4 Practices for Force Verification of Testing Machines
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
E1019 Test Methods for Determination of Carbon, Sulfur, Nitrogen, and Oxygen in Steel, Iron, Nickel, and Cobalt Alloys by Various Combustion and Inert Gas Fusion Techniques
E1097 Guide for Determination of Various Elements by Direct Current Plasma Atomic Emission Spectrometry
E1172 Practice for Describing and Specifying a Wavelength Dispersive X-Ray Spectrometer
E1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis
E1409 Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by Inert Gas Fusion
E1447 Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by Inert Gas Fusion Thermal Conductivity/Infrared Detection Method
E1479 Practice for Describing and Specifying Inductively Coupled Plasma Atomic Emission Spectrometers
E1941 Test Method for Determination of Carbon in Refractory and Reactive Metals and Their Alloys by Combustion Analysis
E2465 Test Method for Analysis of Ni-Base Alloys by Wavelength Dispersive X-Ray Fluorescence Spectrometry
F1710 Test Method for Trace Metallic Impurities in Electronic Grade Titanium by High Mass-Resolution Glow Discharge Mass Spectrometer
F2004 Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis
F2005 Terminology for Nickel-Titanium Shape Memory Alloys

[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] 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.

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

F2082 Test Method for Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery
F2633 Specification for Wrought Seamless Nickel-Titanium Shape Memory Alloy Tube for Medical Devices and Surgical Implants
IEEE/ASTM SI 10 American National Standard for Metric Practice

2.2 ~~*Other Standards:*~~*ISO Standard:*
~~ASQ C1 General Requirements for a Quality Program~~[33]
ISO 9001 Quality Management Systems—Requirements

3. Terminology

3.1 The terminology describing the physical and thermal properties of these alloys shall be as defined in Terminology F2005.

3.2 See also Practice E4~~: General Terminology.~~ (General Terminology).

3.3 *Definitions:*

3.3.1 *ingot, n*—quantity of metal cast into a shape suitable for subsequent processing to various mill products.

3.3.2 *mill product, n*—any finished or semi-finished product from a mill. Product may be straight or coiled. Product types include hot-worked, hot-worked and cold-finished, and hot-worked and cold-worked, with or without a final heat treatment.

~~NOTE 1—Mill product is not intended to have the final shape, final surface finish, or final properties of the medical device, implant, or their components.~~

4. Product Classification

4.1 *Bar*—Round bars and flats from 5.50 to 94.0 mm [0.218 to 3.70 in.] in diameter or thickness (other sizes or shapes by special order).

4.2 *Plate*—Any product 5.50 up to 94.0 mm [0.218 to 3.70 in.] in thickness, with a width equal to or greater than five times the thickness.

4.3 *Tube*—Hollow cylindrical shapes from 5.50 up to 94.0 mm [0.218 to 3.70 in.] in outer diameter.

5. Ordering Information

5.1 Inquiries and orders for material under this specification shall include the following information:
5.1.1 *Quantity*—weight, length, or number of pieces.
5.1.2 *Alloy formulation,* in terms of transformation temperature parameter (see Section 8).
5.1.3 *Form*—bar, plate, or tube (see Section 4).
5.1.4 *Condition*—(see ~~3.3.1~~3.3).
5.1.5 *Mechanical Properties*—if applicable for special conditions (see Section 10).
5.1.6 *Surface Condition*—(see ~~Sections~~Section 6.4).
5.1.7 *Applicable Dimensions,* including diameter, thickness, width, and length (exact, random, multiples) or print number.
5.1.8 *Special Tests*—for example, chemical analysis on the finished mill product.
5.1.9 *Special Requirements*—(see Section 13).

6. Materials and Manufacture

6.1 The material shall be made from ingot made from nickel and titanium with no other intentional alloy additions.

6.2 The material shall be vacuum or inert atmosphere melted to control metallurgical cleanliness and alloy chemistry.

6.3 The product shall be supplied as specified in the purchase order.

6.4 The product surface condition may be oxidized, descaled, pickled, blasted, machined, ground, mechanically polished, or electropolished.

7. Chemical Composition Requirements

7.1 The ~~heat~~ingot analysis shall conform to the requirements of Table 1. Ingot analysis may be used for reporting all chemical requirements except hydrogen. Samples for hydrogen analysis shall be taken from the finished product (see Section 4) or as agreed upon between the customer and supplier. The supplier shall not ship material that is outside the limits specified in Table 1.

7.1.1 Requirements for major and minor elements are listed in Table 1. Important residual elements are also listed. Analysis for elements not listed in Table 1 is not required to verify compliance with this specification.

7.2 *Product Analysis:*

~~[3] Available from American Society for Quality (ASQ), 600 N. Plankinton Ave., Milwaukee, WI 53203.Available from American Society for Quality (ASQ), 600 N. Plankinton Ave., Milwaukee, WI 53203, http://www.asq.org.~~

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

TABLE 1 Chemical Composition Requirements

Element	% (mass/mass)
Nickel	54.5 to 57.0
~~Carbon, maximum~~	~~0.050~~
Carbon, maximum	0.040
Cobalt, maximum	0.050
Copper, maximum	0.010
Chromium, maximum	0.010
Hydrogen, maximum	0.005
Iron, maximum	0.050
Niobium, maximum	0.025
Nitrogen, maximum	0.005
~~Nitrogen plus Oxygen, maximum~~	~~0.050~~
Oxygen, maximum	0.040
Titanium[A]	Balance

[A] Approximately equal to the difference between 100 % and the sum percentage of the other specified elements. The percentage titanium content by difference is not required to be reported.

7.2.1 Product analysis limits shall be as specified in Table 2. Product analysis tolerances do not broaden the specification ~~heat~~ingot analysis requirements, but cover variation between laboratories in the measurement of chemical content. The manufacturer shall not ship material that is outside the limits specified in Table 1.

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

7.2.3 Acceptance or rejection of ~~a heat~~an ingot or manufacturing lot of material may be made by the purchaser on the basis of this product analysis. Product analysis may be conducted by a third party if agreed upon by the supplier and the purchaser.

7.2.4 Major elements shall be analyzed by direct current plasma spectrometry according to Guide E1097; atomic absorption, inductively coupled plasma spectrometry according to Practice E1479; X-ray spectrometer according to Practice E1172; glow discharge mass spectrometry according to Test Method F1710; or an equivalent method. Carbon shall be measured by combustion according to Test ~~Method~~MethodS E1019 or E1941 or wavelength dispersive X-ray fluorescence spectrometry according to Test Method E2465. Hydrogen shall be measured by inert gas fusion or vacuum hot extraction. Hydrogen shall be measured by inert gas fusion or vacuum hot extraction according to Test Method E1447. Nitrogen and oxygen shall be measured by inert gas fusion according to Test Method E1409.

7.2.5 The titanium content of these alloys shall be determined by difference and need not be analyzed.

8. Transformation Temperature

8.1 The nickel and titanium contents of nickel-titanium shape memory alloys cannot be measured to a precision required to guarantee shape memory or superelastic properties. Calorimetry or an equivalent thermomechanical test method shall be used to ensure the alloy formulation in terms of transformation temperature. Testing shall be performed on material in the fully annealed condition.

8.2 Product alloy formulation shall be specified in terms of the transformation temperature parameter(s) required by the purchase order. This parameter shall be one of the following: M_f, M_p, M_s, A_s, A_p, A_f as defined in Terminology F2005 and as measured on the product in accordance with Test Method F2004, or as measured in accordance with another appropriate

TABLE 2 Product Analysis Tolerance[A]

Element	Tolerance Under the Minimum Limit or Over the Maximum Limit, % (mass/mass)[B]
Carbon	0.002
Cobalt	0.001
Copper	0.001
Chromium	0.001
Hydrogen	0.0005
Iron	0.01
Nickel	0.2 under min; 0.2 over max
Niobium	0.004
~~Nitrogen~~	~~0.004~~
Nitrogen	0.001
Oxygen	0.004

[A] Product analysis tolerance limits are based on analytical capabilities that have been demonstrated for this composition.

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

thermomechanical test ~~method.~~method, such as Test Method F2082. Test Method F2004 shall be used to determine transformation temperatures unless otherwise agreed between the supplier and purchaser.

8.2.1 When measured in accordance with Test Method F2004 for transformation temperature by thermal analysis, the A_s shall be uniform on the purchased product to within the ranges in Table 3 or as agreed upon by the purchaser and supplier.

8.2.2 Table 3 tolerances are for A_s only. Tolerances for M_f, M_p, M_s, A_p, and A_f are as agreed upon by the purchaser and supplier.

8.2.3 Transformation temperature parameters are normally specified in the wrought product as defined in Terminology F2005. Other conditions for the certification of alloy transformation temperature shall be considered a special requirement.

8.3 Section size, location, evaluation method, and results shall be reported on the product certification report provided to the purchaser.

9. Metallurgical Structure

9.1 *Microstructure:*

9.1.1 Microstructure shall be evaluated only in the hot-worked ~~condition, prior to any cold processing.~~ or annealed condition. Such evaluations shall take place at a section size not larger than 94.0 mm [3.70 in.] and not smaller than 5.50 mm [0.218 in.] in diameter, thickness, width, height, wall thickness, or other maximum dimension. Evaluation may take place on in-process product that will be utilized to create the final product form. Condition, section size, location, and evaluation method shall be reported on the product certification report provided to the purchaser.

9.1.2 For all product evaluated as stated in 9.1.1, the product shall have an average grain size number (G) of 4 or larger as measured by Test Method E112. Results shall be reported on the product certification report provided to the purchaser.

9.2 *Microcleanliness:*

9.2.1 Porosity and nonmetallic inclusions shall be ~~evaluted~~evaluated only in the ~~hot-worked condition, prior to any cold processing.~~ hot-worked, or for cold-worked mill product in the annealed condition, or as agreed between the supplier and purchaser. Such evaluations shall take place at a section size not larger than 94.0 mm [3.70 in.] and not smaller than 5.50 mm [0.218 in.] in diameter, thickness, width, height, wall thickness, or other maximum dimension. Evaluation may take place on in-process product that will be utilized to create the final product form. Condition, section size, location, and evaluation method shall be reported on the product certification report provided to the purchaser.

9.2.2 For product with A_s less than or equal to 30°C, the maximum allowable dimension of porosity and nonmetallic inclusions such as $Ti_4Ni_2O_x$ and TiC particles shall be 39.0 µm [0.0015 in.]. The maximum dimension shall be the maximum length of all contiguous particles and voids, including particles separated by voids. Furthermore, porosity and nonmetallic inclusions shall not constitute more than 2.8 % (area percent) of the structure as viewed at 400× to 500× in any field of view.

9.2.3 For product with A_s greater than 30°C, the maximum allowable dimensions of porosity and nonmetallic inclusions such as $Ti_4Ni_2O_x$ and TiC particles shall be agreed upon by the purchaser and supplier

9.2.4 Measurements shall be made in accordance with Practice E1245 or an equivalent method with longitudinal samples parallel to the working direction. The supplier and purchaser shall agree upon the size of the product when sampled, the number and location of samples in the product, the sample preparation, the number of fields of view, and the measurement technique. These shall be reported on the product certification report provided to the purchaser.

10. Mechanical Property Requirements

10.1 ~~Finished~~Final product shall be tensile tested in the fully annealed condition. Tensile testing shall be conducted in accordance with Test Methods E8/E8M.

10.1.1 Tension test samples from the final product shall be annealed so that the material reaches a minimum temperature of 800°C [1470°F] for a minimum time of 15 min followed by rapid cooling by water quenching, gas quenching, or air cooling.

10.1.2 Tensile properties shall be determined using a strain rate of 0.003 to 0.1 mm/mm/min [in./in./min]. Tensile properties shall meet the requirements listed in Table 4 using the appropriate gauge length for the product size being tested.

NOTE 1—Annealed product should be tested at 5 to 10°C above A_f.

10.1.3 Specimens for tension tests from product above 50.0 mm [1.97 in.] in diameter or thickness may be taken from plate or strip rolled from the product. For product 50.0 mm [1.97 in.] or less in diameter or thickness, specimens shall be made from the product.

10.1.4 Tensile properties shall be measured in the longitudinal direction with respect to the final fabrication of the sample. Transverse tensile properties for wide flat products shall be as agreed upon between the customer and the ~~supplier~~supplier. Specimen size and condition shall be reported on the product certification report provided to the purchaser.

TABLE 3 ~~Tolerance Requirements~~Typical Tolerances

A_s (°C)	Tolerance Range (°C)
≥70	± 7
–50 < A_s < 70	± 10
≤ –50	± 15