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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 ~~tubing~~tubes containing nominally ~~54.5 %~~54.5- to ~~57.0 %~~ 57.0-weight percent nickel and used for the manufacture of medical devices and surgical implants.

1.2 Requirements are for mill product, measuring ~~6~~5.50 to ~~130~~94.0 mm ~~(0.24~~[0.218 to ~~5.12 in.) diameter or thickness, in its annealed condition.~~3.70 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 ~~the~~ standard. The values ~~given in inch-pound units are for information only.~~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:*[2]

E4 Practices for Force Verification of Testing Machines
~~E8~~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 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 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
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
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
~~F2082~~F2633 ~~Test Method for Determination of Transformation Temperature of~~ Specification for Wrought Seamless Nickel-Titanium Shape Memory ~~Alloys by Bend and Free Recovery~~Alloy Tube for Medical Devices and Surgical Implants
IEEE/ASTM SI 10 American National Standard for Metric Practice

[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**

2.2 ~~*ASQ Standard:*~~*Other Standards:*
ASQ C1 General Requirements for a Quality Program[3]
ISO 9001 Quality Management Systems—Requirements[4]

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.

3.3 *Definitions:*
3.3.1 *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 ~~with~~ 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 ~~*Tubing*—~~*Tube*—Hollow cylindrical ~~shapes.~~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:*~~ *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:*~~ *Form*—bar, plate, or ~~tubing~~tube (see Section 4).
5.1.4 ~~*Condition*~~ *Condition*—(see ~~Sections 6.3~~3.3.1 ~~and 10.1~~).
5.1.5 *Mechanical* ~~*Properties,*~~ *Properties*—if applicable for special conditions (see Section 10).
5.1.6 *Surface* ~~*Condition*~~ *Condition*—(see Sections 6.4).
5.1.7 *Applicable Dimensions*, including diameter, thickness, width, and length (exact, random, multiples) or print number.
5.1.8 *Special* ~~*Tests,*~~ *Tests*—for example, chemical analysis on the finished mill product.
5.1.9 *Special* ~~*Requirements*~~ *Requirements*—(see ~~section~~Section ~~11~~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 ~~Bar, plate, and tubing~~ The product shall be supplied as ~~hot finished or cold finished and annealed or heat treated as~~ specified in the purchase order.

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

7. Chemical Composition Requirements

7.1 The heat 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 ~~mill~~ 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 ~~*Analytical Methods—*~~*Product Analysis:* ~~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~~

[3] Available from American Society for Quality (ASQ), 600 N. Plankinton Ave., Milwaukee, WI ~~53203.~~53203.Available from American Society for Quality (ASQ), 600 N. Plankinton Ave., Milwaukee, WI 53203, http://www.asq.org.
[4] 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
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 plus Oxygen, maximum	0.050
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.

~~shall be measured by combustion according to Test Methods E1019 or E1941. 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.~~

7.2.1 Product analysis limits shall be as specified in Table 2. Product analysis tolerances do not broaden the specification heat 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 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 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 E1019 or E1941. 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.

~~7.3 The titanium content of these alloys shall be determined by difference and need not be analyzed.~~

~~7.4 Product analysis limits shall be as specified in Table 2. Product analysis tolerances do not broaden the specification heat 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.~~

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 ~~must~~shall be used to ~~assure~~ensure the alloy formulation in terms of transformation temperature.

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
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.

8.2 ~~Alloy~~ 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, ~~Test Method F2082~~or as measured in accordance with another appropriate thermomechanical test method.

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 When measured in accordance with Test Method F2004 for transformation temperature by thermal analysis, the A_s shall be uniform to within the process capability of ±10°C on the purchased product or as agreed upon by the customer and supplier.~~

~~8.4 Transformation temperature parameters are normally specified in the wrought product in the annealed condition as defined in F2005. Other conditions for the certification of alloy transformation temperature shall be considered a special requirement.~~

9. Metallurgical Structure

9.1 *Microstructure:*

9.1.1 Microstructure shall be evaluated only in the hot-worked condition, prior to any cold processing. 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.

9.1.2 ~~Product~~ For all product evaluated as stated in 9.1.1, product shall have an average grain size number (G) of 4 or larger as measured by Test Method E112.

9.2 *Microcleanliness:*

9.2.1 Porosity and nonmetallic inclusions shall be evaluted only in the hot-worked condition, prior to any cold processing. 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.

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 ~~For all mill products, 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.). 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. Porosity and nonmetallic inclusions shall be evaluated in mill product at a section size not larger than 94.0 mm (3.70 in.) and not smaller than 6.3 mm (0.25 in.) in diameter, thickness, width, height, wall thickness, and so forth.~~ 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 number and location of samples in the product, the sample preparation, the number of fields of view and the measurement technique.

10. Mechanical Property Requirements

10.1 ~~Samples from the final product, annealed so that the material reaches a minimum temperature of 800°C for a minimum time of 15 min followed by rapid cooling by water quenching, gas quenching, or air cooling, shall conform to the mechanical properties found in~~Finished product shall be tensile tested in the fully annealed condition. Tensile testing shall be conducted in accordance with Test Methods ~~Table 3~~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 2—Annealed product should be tested at 5 to 10°C above A_f.

TABLE 3 Tolerance Requirements

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