Designation: F2063 – 12

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.] 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.

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 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
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:*

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.

[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.

Current edition approved Dec. 1, 2012. Published January 2013. Originally approved in 2000. Last previous edition approved in 2005 as F2063 – 05. DOI: 10.1520/D2063–12.

[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.

[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.

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

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

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 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).

5.1.5 *Mechanical Properties*—if applicable for special conditions (see Section 10).

5.1.6 *Surface 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*—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 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.

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.

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:*

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.

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.

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.

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 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.

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 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 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 Finished 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 2—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

10.2 Other special mechanical tests shall be as specified on the purchase order.

TABLE 3 Tolerance Requirements

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

TABLE 4 Annealed Mechanical Properties[A]

Diameter or Distance Between Parallel Sides, mm	Tensile Strength MPa, Minimum	Elongation in 50.0 mm [1.97 in.] or 4 D, % Minimum[B]
Up to 50.0 [1.97 in.]	551 (79.9 KSI)	15
Over 50.0 [1.97 in.]	551 (79.9 KSI)	10

[A] Tested at ambient temperature of 20.0 to 24.0°C [68.0 to 75.2°F].
[B] 4D indicates 4 times diameter.

11. Units of Measure

11.1 *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.

11.1.1 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.

11.1.2 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.

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

11.2 *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. IEEE/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.

12. Significance of Numerical Limits

12.1 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.

13. Special Requirements

13.1 Size variation and out-of-round tolerance shall be specified in the purchase order.

13.2 Special transformation temperature requirements in terms of product form, test location or heat treatment shall be specified on the purchase order.

13.3 Surface roughness shall be specified on the purchase order.

14. Certification

14.1 The supplier shall provide at the time of shipment a certification that the material was manufactured and tested in accordance with this specification. The certification shall include a summary of the test results for chemical composition, transformation temperature, metallurgical structure, direction of metallurgical structural analysis, and mechanical properties as agreed upon by the customer and supplier (see Sections 7, 8, 9, and 10).

15. Quality Program

15.1 The supplier shall maintain a quality program such as defined in Requirements ASQ C1 or ISO 9001 or similar quality program.

16. Keywords

16.1 cardiac devices; metals; NiTi; TiNi; nitinol; nickel-titanium alloys; titanium-nickel alloys; orthopaedic medical devices; vascular devices; shape memory alloys; stents; superelastic alloys; surgical implants

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 The purpose of this specification is to characterize the chemical, physical, thermomechanical and metallurgical properties of wrought nominally 54.5 to 57.0 % nickel-titanium alloys to be used in the manufacture of medical devices and surgical implants.

X1.2 The purchaser's choice of shape memory alloy transformation temperature and mechanical properties is dependent upon the design and application of the medical device.

X1.3 Thermo-mechanical process history, particularly cold work and heat treatment, affects the transformation temperature and other physical and mechanical properties of nickel-titanium shape memory alloys. The annealed condition stipulated in Sections 8.2.3 and 10.1 are for the test samples only. Finished product may be purchased in the hot-worked, hot-worked and cold-finished, or hot-worked and cold-worked, with or without a final heat treatment.

X1.4 Ingot chemical analysis can be affected by subsequent thermo-mechanical and chemical processing. For example, pickling can result in hydrogen pick up. Therefore, hydrogen is specified for the finished mill product (see Section 7.2).

X1.5 The nickel-titanium alloys covered by this standard are commonly called nitinol alloys. Nitinol is not a single alloy, it is a family of alloys each designated by a transformation temperature measured under controlled conditions and after a specified thermo-mechanical history.

X1.6 Transformation temperature uniformity refers to the range of As measured on an alloy formulation tested by a single laboratory working to Test Method F2004.

X1.7 The elements carbon, cobalt, copper, hydrogen, iron, niobium, and oxygen are residual elements in these alloys (see Table 1). They are controlled to special limits in order to ensure good shape memory physical and mechanical properties. The

product analysis tolerance limits are based upon the analytical capabilities that have been demonstrated for these compositions.

X1.8 *Units of Measure*:

X1.8.1 *ASTM Policy*—ASTM is promoting the use of rationalized IEEE/ASTM SI 10 (metric) units in their standards. The F12.04 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. Harmonization with corresponding ISO documents should be considered when assigning the SI values.

X2. BIOCOMPATIBILITY

X2.1 The material compositions covered by this specification have been employed successfully in human implants, exhibiting a well-characterized level of local biological response since 1972. References are as follows:

Castleman, L. S., et al., "Biocompatability of Nitinol Alloy as an Implant Material," *J. Biomedical Materials Research*, Vol 10, 1976, pp. 695–731.

Ryhanen, J., et al., "Biocompatability of Nickel Titanium Shape Memory Metal and its Corrosion Behavior in Human Cell Cultures," *J. Biomedical Materials Research*, Vol. 35, 1997, pp. 451–457.

Trigwell, S. and Selvaduray, G., "Effects of Surface Finish on the Corrosion of NiTi Alloy for Biomedical Applications," *SMST-97 Proceedings of the Second International Conference on Shape Memory and Superelastic Technologies*, Pelton, A et al., (eds.), SMST, Santa Clara, CA, 1997, pp. 383–388.

Wever, D.J., et al., "Cytotoxic, Allergic and Genotoxic Activity of a Nickel-Titanium Alloy," *Biomaterials*, Vol. 18, No. 16, 1997, pp. 1115–1120.

Trepanier, C. et al., "Effect of the Modification of the Oxide Layer on NiTi Stent Corrosion Resistance," *J. Biomedical Materials Research*, Vol. 43, 1998, pp. 433–440.

Ryhanen, J., "Biocompatibility of Nitinol," *Minimally Invasive Therapy and Allied Technology*, Vol. 9, No. 2, 2000, pp. 99-105.

Venugopalan, R. and Trepanier, C., "Assessing the Corrosion Behavior of Nitinol for Minimally Invasive Device Design," *Minimally Invasive Therapy and Allied Technology*, Vol. 9, No. 2, 2000, pp. 67-73.

Thierry, B., et al., "Nitinol versus Stainless Steel Stents: Acute Thrombogenicity Study in an Ex-Vivo Porcine Model," *Biomaterials*, Vol. 23, 2002, pp. 2997-3005.

Zhu, L., et al., "Oxidation of Nitinol and its Effect on Corrosion Resistance," S. Shrivastava, Proceedings from the Materials & Processes for Medical Devices Conference, 8-10 Sept. 2003, Anaheim, CA, ASM International, 2004, pp. 156–161.

X2.2 No known surgical implant has ever been shown to be completely free of adverse reaction in the human body. Long term clinical experience in the use of the materials referred to in this specification, however, has shown that an acceptable level of biological response can be expected, if the material is used in an appropriate application.

SUMMARY OF CHANGES

Committee F04 has identified the location of selected changes to this standard since the last issue (F2063 – 05) that may impact the use of this standard. (Approved Dec. 1, 2012.)

(1) Defined mill product.
(2) Changed Scope to allow units for either SI or inch-pound to be standard. Added Section 11, Units of Measure.
(3) Added ISO 9001.
(4) Added IEEE/ASTM SI 10 to Section 2.
(5) Revised to conform to different sections of the Ti template wording.
(6) Fixed unit conversion errors.
(7) Added Section 12, Significance of Numerical Limits.
(8) Restricted microstructure and inclusion assessment to hot-worked product, prior to any cold processing.
(9) Added in utilization of the new superelastic nitinol tensile test method for superelastic product.