Designation: F 2063 – 05

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

This standard is issued under the fixed designation F 2063; 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 containing nominally 54.5 % to 57.0 % nickel and used for the manufacture of medical devices and surgical implants.

1.2 Requirements are for mill product, measuring 6 to 130 mm (0.24 to 5.12 in.) diameter or thickness, in its annealed condition.

1.3 The values stated in SI units are to be regarded as the standard. The values given in inch-pound units are for information only.

2. Referenced Documents

2.1 *ASTM Standards:* [2]

E 4 Practices for Force Verification of Testing Machines
E 8 Test Methods for Tension Testing of Metallic Materials
E 112 Test Method for Determining Average Grain Size
E 1019 Test Method for Determination of Carbon, Sulfur, Nitrogen and Oxygen in Steel and in Iron, Nickel, and Cobalt Alloys
E 1097 Guide for Direct Current Plasma Emission Spectrometry Analysis
E 1172 Practice for Describing and Specifying a Wavelength-Dispersive X-Ray Spectrometer
E 1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis
E 1409 Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Technique
E 1447 Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity Method
E 1479 Practice for Describing and Specifying Inductively-Coupled Plasma Optical Emission Spectrometers
E 1941 Test Method for Determination of Carbon in Refractory and Reactive Metals and Their Alloys
F 1710 Test Method for Trace Metallic Impurities in Electronic Grade Titanium by High Mass-Resolution Glow Discharge Mass Spectrometer
F 2004 Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis
F 2005 Terminology for Nickel-Titanium Shape Memory Alloys
F 2082 Test Method for the Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery

2.2 *ASQ Standard:*

C1 General Requirements for a Quality Program[3]

3. Terminology

3.1 The terminology describing the physical and thermal properties of these alloys shall be as defined in Terminology F 2005.

3.2 See also Practice E 4: General Terminology.

4. Product Classification

4.1 *Bar*—Round bars and flats (other sizes or shapes by special order).

4.2 *Plate*—Any product with width equal to or greater than five times the thickness.

4.3 *Tubing*—Hollow cylindrical shapes.

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 tubing (see Section 4).

5.1.4 *Condition* (see Sections 6.3 and 10.1).

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

[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.

[3] Available from American Society for Quality (ASQ), 600 N. Plankinton Ave., Milwaukee, WI 53203.

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

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 11).

6. 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 shall be supplied as hot finished or cold finished and annealed or heat treated as specified in the purchase order.

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

7. Chemical Composition

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*—Major elements shall be analyzed by direct current plasma spectrometry according to Guide E 1097; atomic absorption, inductively coupled plasma spectrometry according to Practice E 1479; X-ray spectrometer according to Practice E 1172; glow discharge mass spectrometry according to Test Method F 1710; or an equivalent method. Carbon shall be measured by combustion according to Test Methods E 1019 or E 1941. Hydrogen shall be measured by inert gas fusion or vacuum hot extraction according to Test Method E 1447. Nitrogen and oxygen shall be measured by inert gas fusion according to Test Method E 1409.

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

TABLE 1 Chemical 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.

7.4 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 be used to assure the alloy formulation in terms of transformation temperature.

8.2 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 F 2005 and as measured in accordance with Test Method F 2004, Test Method F 2082 or as measured in accordance with another appropriate thermomechanical test method.

8.3 When measured in accordance with Test Method F 2004 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 F 2005. Other conditions for the certification of alloy transformation temperature shall be considered a special requirement.

9. Metallurgical Structure

9.1 *Microstructure*:

9.1.1 Product shall have an average grain size number (G) of 4 or larger as measured by Test Method E 112.

9.2 *Microcleanliness*:

9.2.1 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

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.

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 E 1245 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 Table 3.

10.2 Material may be ordered in the cold worked or heat treated condition to higher ultimate tensile strength and lower elongation or other physical and mechanical properties as agreed upon between the supplier and purchaser.

10.3 Specimens for product above 50 mm (1.96 in.) in diameter or thickness may be taken from plate or strip rolled from the product. For product 50 mm (1.96 in.) or less in diameter or thickness, specimens shall be made from the product.

10.4 Tensile properties shall be measured in the longitudinal direction with respect to the final fabrication of the sample.

TABLE 3 Annealed Mechanical Properties[A]

Diameter or Distance Between Parallel Sides, mm	Tensile Strength MPa, Minimum	Elongation in 50 mm (2 in.) or 4 D, % Minimum[B]
Up to 50 (1.96 in.)	551 (79.9 KSI)	15
Over 50	551 (79.9 KSI)	10

[A] Tested at ambient temperature of 20.0 to 24.0°C (68 to 75.2°F).
[B] 4D indicates 4 times diameter.

Transverse tensile properties for wide flat products shall be as agreed upon between the customer and the supplier.

10.5 Tensile testing shall be performed in accordance with Test Method E 8. Tensile properties shall be those listed in Table 3 using the appropriate gage length for the product size being tested.

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

11. Special Requirements

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

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

11.3 Surface roughness shall be specified on the purchase order.

12. Certification

12.1 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).

13. Quality Program

13.1 The supplier shall maintain a quality program such as defined in Requirements C1.

14. Keywords

14.1 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 8.4 and 10.1 are for the test samples only. Finished product is normally purchased in the cold worked or cold worked and heat treated condition.

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 F 2004.

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.

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 (F 2063 – 00) that may impact the use of this standard. (Approved Nov. 1, 2005.)

(*1*) Updated test methods in section 2.1 and 7.2.
(*2*) Renamed section 9.2.
(*3*) Updated product forms throughout the text.
(*4*) Altered allowable porosity, inclusion size, and percentage; restricted direction of porosity and inclusion size measurement in section 9.2.1.
(*5*) Reduced carbon content, established a maximum for the combination of oxygen and nitrogen in Table 1 and brought the table into compliance with the template wording.
(*6*) Added nitrogen to Table 2 and brought table into compliance with template wording.
(*7*) Increased tensile strengths in Table 3.
(*8*) Added date of first use in implant to X2.1 and updated Reference list.
(*9*) Added Summary of Changes section.