Designation: F 2063 – 00

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, wire, 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 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:*

E 8 Test Methods for Tension Testing of Metallic Materials[2]

E 112 Test Method for Determining Average Grain Size[2]

E 120 Test Method for Chemical Analysis of Titanium and Titanium Alloys[3]

E 1019 Test Method for Determination of Carbon, Sulfur, Nitrogen and Oxygen in Steel and in Iron, Nickel, and Cobalt Alloys[4]

E 1097 Guide for Direct Current Plasma Emission Spectrometry Analysis[4]

E 1172 Practice for Describing and Specifying a Wavelength-Dispersive X-Ray Spectrometer[4]

E 1245 Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis[2]

E 1409 Test Method for Determination of Oxygen in Titanium and Titanium Alloys by the Inert Gas Fusion Technique[4]

E 1447 Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by the Inert Gas Fusion Thermal Conductivity Method[4]

E 1479 Practice for Describing and Specifying Inductively-Coupled Plasma Optical Emission Spectrometers[4]

F 2004 Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis[5]

F 2005 Terminology for Nickel-Titanium Shape Memory Alloys[5]

F 2082 Method for the Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery[5]

2.2 *ASQ Standard:*

C1 General Requirements for a Quality Program[6]

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 E4: General Terminology

4. Product Classification

4.1 *bar*—Round bars and flats from 5 mm (0.196 inches) to 130 mm (5.1 inches) in diameter or thickness (other sizes or shapes by special order).

4.2 *plate*—Any product 5 mm (0.196 inches) thick and over and 250 mm (9.8 inches) wide and over, with width equal to or greater than five times the thickness.

4.3 *strip*—Any product under 5 mm (0.196 inches) thick and under 600 mm (23.6 inches) wide.

4.4 *sheet*—Any product under 5 mm (0.196 inches) thick and 600 mm (23.6 inches) or more wide.

4.5 *wire*—Rounds less than 5 mm (0.196 inches) in diameter.

4.6 *tubing*—Hollow cylindrical shapes up to 50 mm (1.96 inches) in outside 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, strip, sheet, wire, 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).

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[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] *Annual Book of ASTM Standards*, Vol 03.01.

[3] *Annual Book of ASTM Standards*, Vol 03.05.

[4] *Annual Book of ASTM Standards*, Vol 03.06.

[5] *Annual Book of ASTM Standards*, Vol 13.01.

[6] Available from the American Society for Quality, 611 East Wisconsin Ave., Milwaukee, WI 53203.

5.1.6 *Surface Condition* (see Sections 6.4).

5.1.7 *Applicable Dimensions*, including diameter, thickness, width, spool size, coil diameter, 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, tubing, wire, and flat rolled products 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 or an equivalent method. Carbon shall be measured by combustion according to Test Method E 1019. Hydrogen shall be measured by inert gas fusion or vacuum hot extraction according to Test Methods E 120 or 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.

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.

TABLE 1 Chemical Requirements

Element	Weight Percent
Nickel	54.5 to 57.0
Carbon, maximum	0.070
Cobalt, maximum	0.050
Copper, maximum	0.010
Chromium, maximum	0.010
Hydrogen, maximum	0.005
Iron, maximum	0.050
Niobium, maximum	0.025
Oxygen, maximum	0.050
Titanium	Balance

TABLE 2 Product Analysis Tolerance[A]

Element	Tolerance Under the Minimum or Over the Maximum, (wt. %)
Nickel	0.2
Element	**Tolerance Over the Maximum, (wt. %)**
Carbon	0.002
Cobalt	0.001
Copper	0.001
Chromium	0.001
Hydrogen	0.0005
Iron	0.01
Niobium	0.004
Oxygen	0.004

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

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

8.3 When measured in accordance with Method F 2004 for transformation temperature by thermal analysis, the A_s shall be uniform to within $\pm 10°C$ 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 Annealed product 6 mm (0.236 in.) to 50 mm (1.96 in.) in section shall have a grain size of ASTM 4 or finer as measured by Test Method E 112.

9.2 *Non-metallic Inclusions and Porosity*:

9.2.1 For all mill products, porosity and nonmetallic inclusions such as $Ti_4Ni_2O_x$ and TiC particles shall be no larger than 12.5 µm (0.0005 in.). Furthermore, porosity and nonmetallic inclusions shall not constitute more than 1.0 % (area percent) of the structure as viewed at 400X to 500X 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 (¼ in.) in any direction except for the mill product length (that is, diameter, thickness, width, height,

wall thickness, etc.). Measurements shall be made in accordance with Practice E 1245 or an equivalent method, except that the plane-of-polish shall be either transverse or parallel to the working direction as agreed upon by the customer and the supplier. 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 as specified in F 2005 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. 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.

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)	10
Over 50	551 (79.9 KSI)	5

[A] Tested at ambient temperature of 20 to 25C (68 to 77°F).
[B] 4D indicates 4 times diameter.

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 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 affect 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 C, Co, Cu, H, Fe, Nb and O 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 for over 10 years. References are as follows:

Castleman, L. S., and Motzkin, S. M., "Biocompatability of Nitinol," *Biocompatability of Clinical Implant Materials*, Williams, D. F. (ed.), CRC Press, Boca Raton, Fl, 1981.

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

Oshida, Y., and Miyazaki, S., "Corrosion and Biocompatability of Shape Memory Alloys," *Corrosion Engineering*, Vol 40, 1991, pp. 1009–1025.

Shabalovskaya, S. A., "On the Nature of the Biocompatability and Medical Applications NiTi Shape Memory and Superelastic Alloys," *Bio-Medical Materials and Engineering*, Vol 6, No 4, 1996, pp. 267–289.

Ryhanen, J., et al., "The Biocompatability and Corrosion Behavior of Nitinol in Human Oseto- and Fibroblast Cell Cultures," in *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. 407–410.

Armitage, D. A., et al., "Haemocompatability of Surface Modified NiTi," *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. 411–416.

Villermaux, F. et al., "Cytocompatability of Niti Shape Memory Alloy Biomaterials," *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. 417–422.

Trepanier, C. et al., "In Vivo Biocompatability Study of NiTi Stents," *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. 423–428.

Ryhanen, J., "Biocompatibility Evaluation of Nickel-Titanium Shape Memory Alloy," ACTA UNIVERSITATIS OULUENSIS, *D Medica*, 518, Oulu University Press, April, 1999.

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.