Designation: F2633 – 13

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

This standard is issued under the fixed designation F2633; 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 requirements for wrought nickel-titanium shape memory alloy tube, nominally 54.5 to 57.0 mass/mass (weight) % nickel, in the superelastic condition, used for the manufacture of medical devices and surgical implants. Material shall conform to the applicable requirements of Specification F2063. This specification addresses those product variables that differentiate drawn medical grade tube from the raw material and mill product forms covered in Specification F2063.

1.2 This specification applies to tube with 10 mm (0.4 in.) and smaller nominal outside diameter and 2 mm (0.08 in.) and thinner nominal wall thickness.

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

2. Referenced Documents

2.1 *ASTM Standards:*[2]

A632 Specification for Seamless and Welded Austenitic Stainless Steel Tubing (Small-Diameter) for General Service

F2004 Test Method for Transformation Temperature of Nickel-Titanium Alloys by Thermal Analysis

F2005 Terminology for Nickel-Titanium Shape Memory Alloys

F2063 Specification for Wrought Nickel-Titanium Shape Memory Alloys for Medical Devices and Surgical Implants

F2082 Test Method for Determination of Transformation Temperature of Nickel-Titanium Shape Memory Alloys by Bend and Free Recovery

F2516 Test Method for Tension Testing of Nickel-Titanium Superelastic Materials

2.2 *ISO Standard:*[3]

ISO 9001 Quality Management Systems—Requirements

ISO 13485 Quality Management Standard for Medical Devices

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.2 See Terminology F2005 for the definition of terms used in this specification that are specific to nickel-titanium alloys.

3.3 *individual wall thickness measurement, n*—any one of the wall thickness measurements taken around the circumference on any one transverse cross-section of a single sample of the tube.

3.4 *lot, n*—the total quantity of product produced from the same melt heat under the same conditions, at essentially the same time.

3.4.1 *Discussion*—For purposes of this specification, conversion from bar to tubular form by extrusion, gundrilling, or other method is included within the scope of this definition.

3.5 *lot average concentricity, n*—the arithmetic average of the sample concentricities measured on a statistically representative number of samples from the lot.

3.6 *lot average wall thickness, n*—the grand average of the sample average wall thicknesses measured on a statistically representative number of samples from the lot.

3.7 *nominal outside diameter (OD), n*—the outside diameter specified on the purchaser's order or engineering drawing without regard to tolerance.

3.8 *nominal wall thickness, n*—the wall thickness specified by the purchaser's order or engineering drawing without regard to tolerance.

3.9 *sample average wall thickness, n*—the arithmetic average of all individual wall thickness measurements measured on a single sample.

[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 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**

3.10 *sample concentricity, n*—two times the offset between the centers of the two circles representing the outside diameter (OD) and the inside diameter (ID) of the tube.

3.10.1 *Discussion*—For purposes of this specification, the sample minimum wall and the sample maximum wall measured on any one transverse cross section of a single sample shall be used to calculate concentricity. Also, for purposes of this specification, sample concentricity shall be expressed as a percent and shall be calculated using the following equation:

$$\text{Sample concentricity percent} = 2 \times \left[\frac{A - B}{A + B}\right] \times 100$$

where:

A = sample maximum wall, and
B = sample minimum wall.

3.11 *sample maximum wall thickness, n*—the largest individual wall thickness measurement taken around the circumference on any one transverse cross section of a single sample of tube.

3.11.1 *Discussion*—In practice, the sample maximum wall thickness may be the largest of no less than four individual wall thickness measurements taken at uniformly spaced locations around the circumference of a single sample of the tube.

3.12 *sample minimum wall thickness, n*—the smallest individual wall thickness measurement taken around the circumference on any one transverse cross section of a single sample of tube.

3.12.1 *Discussion*—In practice, the sample minimum wall thickness may be the smallest of no less than four individual wall thickness measurements taken at uniformly spaced locations around the circumference of a single sample of the tube.

4. Ordering Information

4.1 Inquiries and orders for material under this specification may include the following information:

4.1.1 Quantity (total length or number of pieces),
4.1.2 This ASTM specification and date of issue,
4.1.3 Condition (see 5.2),
4.1.4 Active Austenite Finish Temperature (see 7.3),
4.1.5 Mechanical Properties (see Table 1),
4.1.6 Surface Finish (see 5.3),
4.1.7 Nominal Dimensions including either OD and ID, OD and wall, or ID and wall, or engineering drawing number,
4.1.8 Length (exact or random, see 9.4),
4.1.9 Dimensional Tolerances (see Table 2 and Table 3),
4.1.10 Certification requirements (see Section 11), and
4.1.11 Special requirements or supplementary requirements, if any.

TABLE 2 Permissible Variation in OD or ID Dimensions

Nominal OD or ID in mm (in.)	Permissible Variation from Nominal in mm (in.)
Less than or equal to 1.5 (0.059)	±0.025 (0.001)
1.5 to 6.5 (0.059 to 0.256) incl	±0.050 (0.002)
6.5 to 10 (0.256 to 1.2) incl	±0.075 (0.003)

TABLE 3 Permissible Variation in Wall Thickness Dimensions

Nominal Wall Thickness	Permissible Wall Thickness Variation
Less than 0.2 mm (0.008 in.)	±0.025 mm (0.001 in.) from nominal wall thickness
Greater than or equal to 0.2 mm (0.008 in.)	±12 % of nominal wall thickness[A]

[A]The ±12 % tolerance for a particular nominal wall thickness may be converted to a decimal by multiplying 0.12 times the nominal wall thickness and rounding to the appropriate decimal place.

5. Materials and Manufacture

5.1 *Method of Manufacture:*

5.1.1 Seamless tube shall be made from bar, hollow bar, or tube raw material forms that meet the requirements of Specification F2063.

5.1.2 Seamless tube shall be made by a process in which the tube periphery is continuous at all stages of the process.

5.2 *Condition*—Tube shall be furnished in the superelastic condition or as agreed upon between purchaser and supplier.

5.3 *Surface Finish:*

5.3.1 The tube outer surface shall be furnished, as specified by the purchaser, with a uniform, adherent oxide of any color, chemically etched, ground, or mechanically polished finish. Other finishes may be agreed upon between purchaser and supplier.

5.3.2 The tube inner surface shall be furnished, as specified by the purchaser, with a uniform, adherent oxide of any color, chemically etched, or mechanically conditioned finish.

5.3.3 Inner and outer surface roughness and the method of measurement shall be as agreed upon between the purchaser and supplier.

6. Chemical Composition

6.1 Seamless tube shall be made from bar, hollow bar, or tube raw material forms that meet the chemical composition requirements of Specification F2063.

NOTE 1—Non-metallic inclusions shall be rated and reported on the starting bar, hollow bar, or tube raw material forms by the raw material supplier per Specification F2063. Alternate non-metallic inclusion requirements and rating methods may be agreed upon between purchaser and supplier.

TABLE 1 Mechanical Properties

	UTS	Uniform Elongation	Upper Plateau Strength	Lower Plateau Strength	Residual Elongation
Condition	Min MPa (ksi)	Min % in 50 mm (2 in.) or 4D	Min MPa (ksi) at 3 % strain	Min MPa (ksi) at 2.5 % strain	Max % after 6 % strain
Superelastic[A]	1000 (145)	10	380 (55)	[B]	0.3 %

[A]Superelastic properties are measured per Test Method F2516 at room temperature.
[B]Lower plateau strength value may be measured and reported for information only.

6.2 Hydrogen content shall be analyzed and reported when required by the purchaser. The analysis shall be made on the finished tube.

7. Transformation Temperature

7.1 The raw material manufacturer shall measure the ingot transformation temperature of the raw material on fully annealed samples per Test Method F2004 as required in Specification F2063. The raw material supplier shall report the ingot austenite finish (ingot A_f) transformation temperature as well as any of ingot Martensite finish (M_f), Martensite peak (M_p), Martensite start (M_s), Austenite start (A_s), or Austenite peak A_p transformation temperatures as required by the raw material purchaser. Each ingot transformation temperature reported will be clearly labeled by transformation type.

7.2 The ingot A_f and any other appropriate ingot transformation temperature reported by the raw material supplier may be reported on the finished tube certification as the ingot transformation temperature when required by the finish tube purchaser. Each ingot transformation temperature reported will be clearly labeled by the transformation type.

7.3 The Austenite finish temperature of the finished tube (active A_f) with outer diameter in the range of 0.3 to 3.0 mm (0.012 to 0.12 in.) shall be measured by the supplier on representative full round tube sample(s) using the bend and free recovery method described in Test Method F2082 when required by the purchaser.

7.4 Alternate transformation temperatures and alternate measurement methods may be agreed upon between purchaser and supplier.

8. Mechanical Properties

8.1 Tensile testing of superelastic material shall be performed per Test Method F2516 using full round tube specimens with no reduced center section. Mechanical properties are listed in Table 1 for the superelastic condition tested at room temperature. Alternate test methods, conditions, test temperatures, and mechanical properties may be agreed upon between purchaser and supplier.

9. Permissible Variation in Dimensions

9.1 *OD or ID:*

9.1.1 Permissible variations of OD or ID are listed in Table 2. Alternate tolerances may be agreed upon between purchaser and supplier.

9.1.2 OD may be measured by hand micrometer, dial indicator, linear variable displacement transducer (LVDT), coordinate measuring machine (CMM), or laser micrometer or other non-contact method.

9.2 *Wall Thickness:*

9.2.1 The wall thickness variation throughout the lot shall be as shown in Table 3, or as agreed upon between purchaser and supplier. All individual wall thickness readings shall fall within the indicated range.

9.2.2 Wall thickness measurement may be made directly with a hand micrometer, dial indicator, LVDT, or CMM. Alternately, wall thickness may be measured optically on a transverse metallographic cross section, or by some other appropriate method. The method of wall thickness measurement shall be agreed upon between purchaser and supplier.

9.3 *Concentricity:*

9.3.1 Concentricity shall be measured and reported when specified by the purchaser.

9.3.1.1 For wall thickness of 0.2 mm (0.008 in.) or greater and OD/W ratio of 15 or less, all sample concentricities shall be less than or equal to 10 %. For tube with nominal wall thickness less than 0.2 mm (0.008 in.) or OD/W ratio greater than 15, concentricity shall be agreed upon between purchaser and supplier.

9.3.2 This concentricity requirement does not apply to tube specified as OD and ID unless agreed upon between purchaser and supplier.

9.3.3 Alternate means of calculating and reporting concentricity may be agreed upon between purchaser and supplier.

9.4 *Length:*

9.4.1 For exact length orders, nominal length and length variation shall be agreed upon between purchaser and supplier.

9.4.2 For random length orders, a maximum and minimum length shall be agreed upon between purchaser and supplier.

9.5 *Straightness:*

9.5.1 Straightness shall be measured and reported when specified by the purchaser.

9.5.2 The deviation from straightness shall not exceed 0.50 mm per 300 mm (0.020 in. per 12 in.) of tube length.

9.5.3 Alternate straightness requirements and measurement methods may be agreed upon between purchaser and supplier.

10. Permissible Outer and Inner Surface Imperfections

10.1 Outer and inner surface imperfections on tubes with nominal wall thickness greater than 0.2 mm (0.008 in.) shall be no deeper than 10 % of the nominal wall thickness. Imperfection depths on thinner wall thickness may be agreed upon by purchaser and supplier.

10.2 The method of detecting and measuring surface imperfections shall be agreed upon between the purchaser and supplier.

11. Certification

11.1 The supplier shall provide certification that the material meets the requirements of this specification. A report of the test results shall be furnished at the time of shipment.

11.2 Certification of raw material compliance to chemistry (except for hydrogen), non-metallic inclusion and ingot transformation temperature requirements may be provided as evidence of compliance of the finished tube to the requirements of this specification.

12. Quality Program Requirements

12.1 The supplier shall maintain a quality program such as defined in ISO 9001 or ISO 13485.

13. Keywords

13.1 medical devices; metals (for surgical implant); nickel-titanium alloys; niti; nitinol; shape memory alloy; stents; superelastic alloys; surgical implants; tube

SUPPLEMENTARY REQUIREMENTS

The following supplementary requirements shall apply only when specified by the purchaser in the inquiry, contract, or order.

S1. Inner Diameters Surface Cleanliness

S1.1 Tube with ID size of greater than or equal to 1.5 mm (0.060 in.) shall meet the requirements of Specification A632 Supplement 3 for cleanliness. The method for inspecting inside surface cleanliness for smaller ID sizes shall be agreed upon between purchaser and supplier. This supplementary requirement does not apply to the ID oxide condition.

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 The primary reason for this specification is to establish a tubular product standard specification for wrought, seamless nickel titanium shape memory alloy tube used to fabricate stents and other medical devices.

X1.2 Nickel titanium alloy tube made to this specification is typically intended to perform a super elastic function. The relationship between the operating temperature of the device, and the transformation temperature of the material from which the device is made will determine how the device will perform. The device design and the specification of material transformation temperature to ensure effective super elastic performance is beyond the scope of this specification. In this specification, the use of the term superelastic is a convenient way to characterize the finished tube's room temperature mechanical behavior. This characterization is not sufficient for the specification of material or design of a device to achieve superelastic performance.

X1.3 This specification covers the superelastic condition. Alternate conditions for nitinol medical tube include annealed, cold-worked, and shape memory.

X1.4 When measuring wall thickness, the tube to be measured shall be sufficiently prepared to eliminate any burr or other material that may interfere with accurate mechanical or optical measurement. This preparation can be done by end-finishing procedures such as reaming and deburring, Electro Chemical Machining (ECM) cutting, or Electro Discharge Machining (EDM) wire cutting (for direct micrometer, dial indicator, LVDT, and CMM measurement), or metallographic mounting, grinding, and polishing (for optical measurement). Metallographic preparation for optical measurement shall be performed in such a way as to maximize sample edge retention and minimize sample deviation from the perpendicular. Micrometers used for wall measurement shall have a pin diameter or effective anvil diameter less than the minimum tube ID size. Dial indicators, Linear Variable Displacement Transducers (LVDTs), and Coordinate Measuring Machines (CMMs) shall have a precision consistent with the required wall thickness tolerance.

X1.5 *Bias and Precision* —The choice of wall thickness measurement technique is critical for a tube with very thin walls. Once a method is agreed upon between purchaser and supplier, the bias and precision and gauge repeatability and reproducibility of the method should be evaluated to ensure the method is accurate and repeatable. In the absence of bias and precision data, no test method shall be used to reject a tube with measured wall thickness within 0.005 mm (0.0002 in.) of the specification limits.

X1.6 In the event that OD, ID and wall thickness are specified on the purchase order, the supplier and purchaser shall resolve which two of these shall apply.

X1.7 Concentricity may be used in conjunction with OD, ID, or wall tolerances to better define the allowable variation in wall thickness. For example, when a tube is specified as OD and ID, when agreed upon by supplier and purchaser, concentricity may be used to limit wall variation within the larger range allowed by a comparison of the upper and lower OD and ID tolerance limits. In this application, concentricity shall not be interpreted as a wall thickness specification requiring resolution per X1.6.

X1.8 Straightness of nitinol tube may be measured by a number of methods. These include measuring the gap between the tube and a straight edge (gap must not exceed some specified value), rolling the tube down a surface plate with a

specified angle (tube must roll freely), rolling the tube on an angled surface plate under a bridge with a specified gap (tube must roll under the bridge), and measuring the vertical gap on a surface plate (gap must not exceed some specified value). Each method will require an acceptance criterion that may have a value different than that specified for other methods.

X1.9 Surface imperfections may be detected by a number of methods. This includes visual inspection methods. The suspect defect may be metallographically prepared and measured using a measuring reticule on a light microscope. This method is particularly suited for measuring long continuous imperfections such as "scratches" or "draw lines." Other methods that may be used to evaluate surface imperfection depth include removal of some amount of material from the surface containing the imperfection (to see if the imperfection is still visible at a specified material removal), or use of a *Z* axis measuring microscope to directly measure the depth. These same methods may be applied to ID defects by carefully splitting or exposing the tube ID for inspection using wire EDM, diamond saw, fine abrasive saw, or grinding to remove some portion of the tube.

X1.10 Non-metallic inclusions are created in the melting and casting steps of the process used to manufacture nitinol raw materials. The raw material producer rates and reports inclusion content per the requirements outlined in Specification F2063. The tube supplier further processes the raw material to fabricate a finished tube. The tube supplier's process does not create non-metallic inclusions. However, the morphology of the pre-existing non-metallic inclusions will be changed as the metal is deformed to produce the reduced finish tube cross section. To date, no formal, industry-accepted correlation between raw material non-metallic inclusion size, distribution and density (morphology), and finished tube inclusion morphology has been established. Although the inclusions in the final tube are not specified by this specification, they are controlled by the requirements of Specification F2063.

X2. BIOCOMPATIBILITTY

X2.1 Biocompatibility of nickel-titanium shape memory alloys is addressed in Specification F2063.

SUMMARY OF CHANGES

Committee F04 has identified the location of selected changes to this standard since the last issue (F2633– 07) that may impact the use of this standard. (Approved October 1, 2013.)

(1) Wording has been changed to conform to the ASTM F04.12 template language.

(2) Section 7 has been changed to allow alternate ingot transformation types while still requiring the original ingot A_f.