Designation: F67 – 13 (Reapproved 2017)

Standard Specification for Unalloyed Titanium, for Surgical Implant Applications (UNS R50250, UNS R50400, UNS R50550, UNS R50700)[1]

This standard is issued under the fixed designation F67; 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, mechanical, and metallurgical requirements for four grades of unalloyed titanium strip, sheet, plate, bar, billet, forging, and wire used for the manufacture of surgical implants.

1.2 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.

1.3 *This international standard was developed in accordance with internationally recognized principles on standardization established in the Decision on Principles for the Development of International Standards, Guides and Recommendations issued by the World Trade Organization Technical Barriers to Trade (TBT) Committee.*

2. Referenced Documents

2.1 *ASTM Standards:*[2]

B265 Specification for Titanium and Titanium Alloy Strip, Sheet, and Plate

B348 Specification for Titanium and Titanium Alloy Bars and Billets

B381 Specification for Titanium and Titanium Alloy Forgings

E8/E8M Test Methods for Tension Testing of Metallic Materials

E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications

E290 Test Methods for Bend Testing of Material for Ductility

E1409 Test Method for Determination of Oxygen and Nitrogen in Titanium and Titanium Alloys by Inert Gas Fusion

E1447 Test Method for Determination of Hydrogen in Titanium and Titanium Alloys by Inert Gas Fusion Thermal Conductivity/Infrared Detection Method

E1941 Test Method for Determination of Carbon in Refractory and Reactive Metals and Their Alloys by Combustion Analysis

E2371 Test Method for Analysis of Titanium and Titanium Alloys by Direct Current Plasma and Inductively Coupled Plasma Atomic Emission Spectrometry (Performance-Based Test Methodology)

E2626 Guide for Spectrometric Analysis of Reactive and Refractory Metals

F981 Practice for Assessment of Compatibility of Biomaterials for Surgical Implants with Respect to Effect of Materials on Muscle and Insertion into Bone

IEEE/ASTM SI 10 American National Standard for Metric Practice

2.2 *Aerospace Material Specification:*[3]

AMS 2249 Chemical Check Analysis Limits, Titanium and Titanium Alloys

AMS 2380 Approval and Control of Premium Quality Titanium Alloys

AMS 2631 Ultrasonic Inspection Titanium Alloy Bar, Billet and Plate

2.3 *ISO Standards:*

ISO 5832-2 Implants for Surgery—Metallic Materials—Unalloyed Titanium[4]

ISO 6892 Metallic Materials—Tensile Testing at Ambient Temperature[4]

ISO 9001 Quality Management Systems[4]

3. Terminology

3.1 *Definitions of Terms Specific to This Standard:*

3.1.1 *lot, n*—the total number of mill products produced from the same melt heat under the same conditions at essentially the same time.

[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 March 1, 2017. Published April 2017. Originally approved in 1966. Last previous edition approved in 2013 as F67 – 13. DOI: 10.1520/F0067-13R17.

[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 Society of Automotive Engineers (SAE), 400 Commonwealth Dr., Warrendale, PA 15096-0001.

[4] 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

3.1.2 *cold work, n*—any mechanical deformation process performed below the recrystallization temperature which results in strain hardening of the material.

3.1.3 *hot work, n*—any mechanical deformation process performed above the recrystallization temperature.

3.1.4 *alpha case, n*—oxygen, nitrogen or carbon-enriched, alpha-stabilized surface which results from elevated temperature exposure.

4. Product Classification

4.1 Product classifications are consistent with Specifications B265, B348, and B381.

4.1.1 *Strip*—Any product 0.188 in. (4.76 mm) and under in thickness and less than 24 in. (610 mm) wide.

4.1.2 *Sheet*—Any product 0.188 in. (4.76 mm) and under in thickness and 24 in. (610 mm) or more in width.

4.1.3 *Plate*—Any product 0.188 in. (4.76 mm) thick and over and 10 in. (254 mm) wide and over, with widths greater than five times thickness. Plate up to 4 in. (102 mm), thick inclusive is covered by this specification.

4.1.4 *Bar*—Rounds, flats, or other shapes from 0.188 in. (4.76 mm) to 4 in. (102 mm) in diameter or thickness. (Other sizes and shapes by special order.)

4.1.5 *Forging bar*—Bar as described in 4.1.4 used in the production of forgings. This product may be furnished in the hot worked condition.

4.1.6 *Billet*—A solid semi-finished section hot rolled or forged from an ingot, with a cross sectional area greater than 16 $in.^2$ (10 322 mm^2) whose width is less than 5 times its thickness.

4.1.7 *Forging*—Any product of work on metal formed to a desired shape by impact or pressure in hammers, forging machines, upset presses, or related forming equipment.

4.1.8 *Wire*—Rounds, flats or other shapes less than 0.188 in. (4.76 mm) in diameter or thickness.

4.1.9 *Other*—Other forms and shapes, including tubing, may be provided by agreement between purchaser and supplier.

5. Ordering Information

5.1 Inquiries and orders for material under this specification shall include the following information:

5.1.1 Quantity (weight or number of pieces),

5.1.2 Grade (1, 2, 3, or 4),

5.1.3 ASTM designation and date of issue,

5.1.3.1 Units to be certified – SI or inch-pound,

TABLE 2 Product Analysis Tolerances[A]

Element	Limit or Maximum of Specified Range %, (mass/mass)		Tolerance Under the Minimum or Over the Maximum Limit[B]
Nitrogen	up to	0.05	0.02
Carbon		0.10	0.02
Hydrogen	up to	0.015	0.0020
Iron	up to	0.25	0.10
Iron	over	0.25	0.15
Oxygen	up to	0.20	0.02
Oxygen	over	0.20	0.03

[A] Refer to AMS 2249.

[B] Under minimum limit not applicable for elements where only a maximum percentage is indicated.

5.1.4 Form (sheet, strip, plate, bar, billet, forging, wire, or other forms),

5.1.5 Condition (see 6.1),

5.1.6 Mechanical properties (if applicable, for special conditions),

5.1.7 Finish (see 6.2),

5.1.8 Applicable dimensions and tolerances including size, thickness, width, and length (exact, random, multiples) or drawing number,

5.1.9 Special tests (if any), and

5.1.10 Other requirements.

6. Manufacture

6.1 *Condition*—Material shall be furnished in the hot-worked, cold-worked, forged, annealed, or stress-relieved condition.

6.2 *Finish*—The mill product may be furnished as descaled or pickled, abrasive blasted, chemically milled, ground, machined, peeled, polished, or as specified by the purchaser. On billets, bars, plates, and forgings, it is permissible to remove minor surface imperfections by spot grinding if such grinding does not reduce the dimension below the minimum permitted by the dimensional tolerance ordered.

7. Chemical Composition

7.1 The heat analysis shall conform to the chemical composition of Table 1. Ingot analysis may be used for reporting all chemical requirements, except hydrogen. Samples for hydrogen shall be taken from the finished mill product. Supplier shall not ship material with chemistry outside the requirements specified in Table 1 for the applicable grade.

TABLE 1 Chemical Requirements

Element	Composition[A], % (mass/mass)			
	Grade 1 UNS R50250	Grade 2 UNS R50400	Grade 3 UNS R50550	Grade 4 UNS R50700
Nitrogen, max	0.03	0.03	0.05	0.05
Carbon, max	0.08	0.08	0.08	0.08
Hydrogen, max[B]	0.015	0.015	0.015	0.015
Iron, max	0.20	0.30	0.30	0.50
Oxygen, max	0.18	0.25	0.35	0.40
Titanium	balance	balance	balance	balance

[A] Forgings are designated Grade F-1, F-2, F-3, or F-4 respectively. Forging compositions are as specified in Table 1.

[B] Maximum hydrogen content for billet is 0.0100 wt%.

TABLE 3 Mechanical Requirements: Annealed-Bar, Billet, Forgings, and Other Forms[A]

Grade	Tensile Strength, min		Yield Strength, 0.2 % Offset, min		Elongation[B] in 4*D*, min, %	Reduction of Area, min, %[C]
	ksi	MPa	ksi	MPa		
1	35	240	25	170	24	30
2	50	345	40	275	20	30
3	65	450	55	380	18	30
4	80	550	70	483	15	25

[A] These properties apply to forgings having a maximum cross section area not greater than 3 in.^{2} (1935 mm^2). Mechanical properties of forgings having greater cross sections shall be negotiated between the manufacturer and the purchaser.
[B] Elongation of material 0.063 in. (1.6 mm) or greater in diameter (*D*) or width (*W*) shall be measured using a gauge length of 2 in. or 4*D* or 4*W*. The gauge length must be reported with the test results. The method for determining elongation of material under 0.063 in. (1.6 mm) in diameter or thickness may be negotiated. Alternatively, a gauge length corresponding to ISO 6892 may be used when agreed upon between supplier and purchaser. (5.65 times the square root of *So*, where *So* is the original cross sectional area.)
[C] Reduction of area not required for tubing.

7.1.1 Requirements for the major and minor elemental constituents are listed in Table 1. Also listed are important residual elements. 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 tolerances do not broaden the specified heat analysis requirements but cover variations in the measurement of chemical content between laboratories. The product analysis tolerances shall conform to the product tolerances in Table 2.

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 analyses outside the tolerance limits allowed in Table 2 are cause for rejection of the product. A referee analysis may be used if agreed upon by supplier and purchaser.

7.2.4 For referee purposes, use Test Methods E2371, E2626, E1409, E1447, E1941 or other analytical methods agreed upon between the purchaser and supplier.

7.3 Samples for chemical analysis shall be representative of the material being tested. The utmost care must be used in sampling titanium for chemical analysis because of its affinity for elements such as oxygen, nitrogen, and hydrogen. In cutting samples for analysis, therefore, the operation should be carried out insofar as possible in a dust-free atmosphere. Cutting tools should be clean and sharp. Samples for analysis should be stored in suitable containers.

8. Mechanical Requirements

8.1 The material supplied under this specification shall conform to the mechanical property requirements in Table 3, Table 4 and Table 5. Grades may be ordered in the cold worked condition to higher minimum tensile strength but a minimum 10 % elongation in 4*D* or 2 in. (50 mm) must be met except for wire. See footnotes in Table 5. Alternate properties may be agreed upon between the purchaser and supplier.

8.2 Specimens for tension tests shall be prepared and tested in accordance with Test Methods E8/E8M. Tensile properties shall be determined using a strain rate of 0.003 to 0.007 in/in/min (mm/mm/min) through yield and then the crosshead speed may be increased so as to produce fracture in approximately one additional minute. Tensile tests results for which any specimen fractures outside the gage length shall be considered valid, if both the elongation and reduction of area meet the minimum requirements specified. Refer to sections 7.11.4 and 7.12.5 of Test Methods E8/E8M. If either the elongation or reduction of area is less than the minimum requirement, invalidate the specimen and retest. Retest one specimen for each specimen that did not meet the minimum requirements.

8.2.1 *Bar, Forging Bar, Shapes, and Wire*—Test according to Test Methods E8/E8M. Should any test specimen not meet the specified requirements, test two additional test pieces representative of the same lot, in the same manner, for each failed test specimen. The lot will be considered in compliance only if all additional test pieces meet the specified requirements

8.2.2 Tension testing of wire shall be conducted in accordance with Test Methods E8/E8M Tension testing shall be conducted using the appropriate gauge length, shown in Table 5, for the size wire being tested. For wire sizes ≥0.0625 in. (≥1.59 mm) (4*D* gauge length) a strain rate of 0.003 to 0.007 in./in. (mm/mm)/min through the specified yield strength shall be used and then the crosshead speed shall be increased so as to produce fracture in approximately one additional minute. For wire diameters less than 0.0625 in. (1.59 mm) a crosshead speed of 0.5 to 1.0 in./min (12.7 to 25.4 mm/min) shall be used. Once yielding has begun, the crosshead speed may be increased to a maximum of 3.0 in./min (76.2 mm/min).

8.2.3 Sheet, Strip, and Plate—Test according to Test Methods E8/E8M. Tests in the transverse direction need be made only on product from which a specimen not less than 8.0 in. (200 mm) in length for sheet and 2.50 in. (64 mm) in length for plate can be taken. Should any of these test specimens not meet the specified requirements, test two additional test pieces representative of the same lot, in the same manner, for each failed test specimen. The lot will be considered in compliance only if all additional test pieces meet the specified requirements.

8.3 For sheet and strip, perform at least one bend test according to Test Methods E290 from each lot in both the longitudinal and transverse directions. Tests in the transverse direction need be made only on product from which a specimen not less than 8.0 in. (200 mm) in length for sheet and 2.50 in. (64 mm) in length for plate can be taken. The bend test specimen shall withstand being bent cold through an angle of 105° without fracture on the outside of the bent portion. The bend shall be made around a mandrel which has a diameter equal to that shown in Table 4 for the applicable grade.

9. Units of Measure

9.1 *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

TABLE 4 Mechanical Requirements: Annealed-Sheet, Strip, and Plate

Grade	Tensile Strength,[A] min		Yield Strength,[A] (0.2 % Offset)				Elongation[B] in 2 in. or 50 mm, min,%	Bend Test Mandrel Diameter[C]	
	ksi	MPa	min		max			Under 0.070 in. (1.8 mm) in Thickness	0.070 to 0.187 in. (1.8 to 4.75 mm) in Thickness
			ksi	MPa	ksi	MPa			
1	35	240	25	170	45	310	24	3*T*	4*T*
2	50	345	40	275	65	450	20	4*T*	5*T*
3	65	450	55	380	80	550	18	4*T*	5*T*
4	80	550	70	483	95	655	15	5*T*	6*T*

[A] Minimum and maximum limits apply to tests taken both longitudinal and transverse to the direction of rolling. Mechanical properties for conditions other than annealed or plate thickness over 1 in. (25 mm) may be established by agreement between the manufacturer and the purchaser.
[B] Elongation of material 0.063 in. (1.6 mm) or greater in diameter (*D*) or width (*W*) shall be measured using a gauge length of 2 in. or 4*D* or 4*W*. The gauge length must be reported with the test results. The method for determining elongation of material under 0.063 in. (1.6 mm) in diameter or thickness may be negotiated. Alternatively, a gauge length corresponding to ISO 6892 may be used when agreed upon between supplier and purchaser. (5.65 times the square root of *So*, where *So* is the original cross sectional area.)
[C] *T* equals the thickness of the bend test specimen; refer to Test Methods E290. Bend tests are not applicable to material over 0.1875 in. (4.75 mm) in thickness.

TABLE 5 Mechanical Requirements: Annealed Wire[A]

Diameter, in. (mm)	Grade	Ultimate Tensile Strength, min, ksi (MPa)	Yield Strength, (0.2% offset) min, ksi (MPa)	Elongation[B] min, %	Reduction in Area, min, %
≥0.125 (≥3.18)	1	35 (240)	25 (170)	24	30
	2	50 (345)	40 (275)	20	30
	3	65 (450)	55 (380)	18	30
	4	80 (550)	70 (483)	15	25
<0.125 to 0.062 inclusive (<3.18 to 1.58 inclusive)	1	35 (240)	25 (170)	15	. . .
	2	50 (345)	40 (275)	12	. . .
	3	65 (450)	55 (380)	10	. . .
	4	80 (550)	70 (483)	8	. . .
<0.062 to 0.020 inclusive (<1.58 to 0.51 inclusive)	1	35 (240)	. . .	12	. . .
	2	50 (345)	. . .	10	. . .
	3	65 (450)	. . .	8	. . .
	4	80 (550)	. . .	6	. . .
<0.020 to 0.005 inclusive (<0.51 to 0.13 inclusive)	1	35 (240)	. . .	10	. . .
	2	50 (345)	. . .	8	. . .
	3	65 (450)	. . .	6	. . .
	4	80 (550)	. . .	4	. . .

[A] Mechanical properties for cold worked condition shall be established by agreement between the supplier and the purchaser.
[B] Elongation of material 0.063 in. (1.6 mm) or greater in diameter (*D*) or width (*W*) shall be measured using a gauge length of 2 in. or 4*D* or 4*W*. The gauge length must be reported with the test results. The method for determining elongation of material under 0.063 in. (1.6 mm) in diameter or thickness may be negotiated, or a 10 in. gauge length may be used. Alternatively, a gauge length corresponding to ISO 6892 may be used when agreed upon between supplier and purchaser. (5.65 times the square root of *So*, where *So* is the original cross sectional area.)

TABLE 6 Round Wire Size Tolerances

Diameter, in. (mm)	Size Variation, in. (mm)	Out-of-Round,[A] in. (mm)
0.3125 to 0.125 inclusive (7.94 to 3.18 inclusive)	±0.002 (0.051)	0.002 (0.051)
<0.125 to 0.030 inclusive (<3.18 to 0.76 inclusive)	±0.001 (0.025)	0.001 (0.025)
<0.030 to 0.010 inclusive (<0.76 to 0.25 inclusive)	±0.0008 (0.020)	0.0008 (0.020)
<0.010 to 0.005 inclusive (<0.25 to 0.13 inclusive)	±0.0005 (0.013)	0.0005 (0.013)

[A] Out-of-Round is the difference between the maximum and minimum diameters of the wire measured at the same cross section.

purchase order (PO), this selection may be expressed by the purchaser in several alternate forms listed in order of precedence.

9.1.1 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.

9.1.2 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

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

9.2 *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.

10. Special Requirements

10.1 Alpha case is not permitted for products supplied with a machined, ground, or chemically milled surface finish. For other products, there will be no continuous layer of alpha case when viewed at 100 × magnification.

10.2 Size variation and out-of-round tolerances for round wire shall meet the requirements specified in Table 6.

10.3 Ultrasonic Inspection—All centerless ground or peeled and polished round bar ≥0.375 in. (9.5 mm) in nominal diameter shall be ultrasonically inspected at final diameter according to AMS 2631, Class A1. Equivalent test methods may be substituted when agreed upon by purchaser and supplier.

NOTE 1—AMS 2631 contains varying flat bottom hole (FBH) requirements based on melting grades per AMS 2380. Since the FBH requirement for class A1 is the same, regardless of the melting grade, it is not necessary to specify the melting grade.

11. Significance of Numerical Limits

11.1 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.

12. Certification

12.1 The supplier shall provide a certification that the material was tested in accordance with this specification and met all requirements. A report of the test results shall be furnished to the purchaser at the time of shipment.

12.2 Gauge length must be reported with elongation.

13. Quality Program Requirements

13.1 The supplier shall maintain a quality program such as defined in ISO 9001 or similar quality program.

14. Keywords

14.1 metals (for surgical implants); orthopaedic medical devices; titanium alloys; titanium/titanium alloy; titanium/titanium alloys (for surgical implants)

APPENDIXES

(Nonmandatory Information)

X1. RATIONALE

X1.1 The purpose of this specification is to characterize the chemical, mechanical, and metallurgical properties of wrought unalloyed titanium to be used in the manufacture of surgical implants.

X1.2 The choice of composition and mechanical properties is dependent upon the design and application of the medical device.

X1.3 ISO standards are listed for reference only. Although the ISO 5832-2 standard listed in 2.3 is similar to the corresponding ASTM standards, it is not identical. Use of the ISO standard instead of the preferred ASTM standards may be agreed upon between the purchaser and supplier.

X1.4 The various titanium mill products covered in this specification normally are formed with the conventional forging and rolling equipment found in primary ferrous and nonferrous plants. The material is usually multiple melted in arc furnaces (including furnaces such as plasma arc and electron beam) of a type used for reactive metals.

X1.5 The number of samples required in 9.1.2 and 9.2 are not intended to represent a statistically valid sample of the lot. The number of samples required represents a sampling plan with balanced cost and benefit that has served suppliers and purchasers in this market for over 10 years. More frequent sampling may be performed as agreed between supplier and purchaser.

X2. BIOCOMPATIBILTY

X2.1 The unalloyed titanium compositions covered by this specification have been employed successfully in human implant applications in contact with soft tissue and bone for over a decade. Due to the well characterized level of biological response exhibited by these unalloyed titanium materials, they have been used as control materials in Practice F981.

X2.2 No known surgical implant material has ever been shown to be completely free of adverse reactions in the human body. Long term clinical experience of 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 appropriate applications.

SUMMARY OF CHANGES

Committee F04 has identified the location of selected changes to this standard since the last issue (F67 – 12) that may impact the use of this standard. (Approved June 1, 2013.)

(1) Editorial corrections have been made in order to meet terminology and formatting guidelines established for implant material standards.

(2) Ultrasonic inspection requirements have been added to 10.3.