

# Standard Specification for Niobium-Titanium Alloy Billets, Bar, and Rod for Superconducting Applications<sup>1</sup>

This standard is issued under the fixed designation B884; 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 ( $\varepsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

- 1.1 This specification covers niobium-titanium alloy billets, bars, and rods, at 46 to 48 % titanium. This material is used in the manufacture of wire for superconducting applications.
- 1.2 The values stated in inch-pound units are to be regarded as standard. The values given in parentheses are mathematical conversions to SI units that are provided for information only and are not considered standard.
- 1.3 The following precautionary caveat pertains only to the test methods portion, Section 14, of this specification: *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

#### 2. Referenced Documents

- 2.1 ASTM Standards:<sup>2</sup>
- E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
- E92 Test Method for Vickers Hardness of Metallic Materials (Withdrawn 2010)<sup>3</sup>
- E112 Test Methods for Determining Average Grain Size
- E165 Practice for Liquid Penetrant Examination for General Industry
- E214 Practice for Immersed Ultrasonic Testing by the Reflection Method Using Pulsed Longitudinal Waves (Withdrawn 2007)<sup>3</sup>
- E384 Test Method for Knoop and Vickers Hardness of Materials

E2626 Guide for Spectrometric Analysis of Reactive and Refractory Metals

2.2 ANSI Standard:

ANSI B46-1 Surface Texture<sup>4</sup>

2.3 ASNT Standard:

ASNT SNT-TC-1A Personnel Qualification and Certification in Nondestructive Testing<sup>5</sup>

### 3. Terminology

- 3.1 Definitions of Terms Specific to This Standard:
- 3.1.1 *rod*, *n*—material greater than 0.5 in. (13 mm) and less than 2.5 in (60 mm) in. diameter.
- 3.1.2 *bar*, *n*—material greater than or equal to 2.5 in (60 mm) and less than 6 in. (150 mm) in diameter.
- 3.1.3 *billet*, *n*—material greater than or equal to 6 in. (150 mm) in diameter.
- 3.1.4 *lot*, *n*—a lot shall consist of material of the same size, shape, condition and finish produced from the same ingot or powder blend by the same reduction schedule and the same heat treatment parameters. Unless otherwise agreed between manufacturer and purchaser, a lot shall be limited to the product of an 8th period for final continuous anneal, or to a single furnace load for final batch anneal.

### 4. Ordering Information

- 4.1 Purchase orders for material under this specification should include:
  - 4.1.1 ASTM designation and year of issue,
  - 4.1.2 Quantity in weight, number of pieces, and dimensions,
- 4.1.3 Grain size limit for diameters greater than 7.75 in. (see 7.2 and Table 1),
  - 4.1.4 Surface texture, if required (see 10.3),
  - 4.1.5 Annealing condition, if different from 7.1,
- 4.1.6 Permissible variations in diameter and length (see 9.1 and 9.2),
- 4.1.7 Sampling and analytical methods, if required (see 11.3),

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<sup>&</sup>lt;sup>2</sup> 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.

 $<sup>^{3}\,\</sup>mbox{The last approved version of this historical standard is referenced on www.astm.org.$ 

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

<sup>&</sup>lt;sup>5</sup> Available from American Society for Nondestructive Testing (ASNT), P.O. Box 28518, 1711 Arlingate Ln., Columbus, OH 43228-0518, http://www.asnt.org.

**TABLE 1 Grain Size Requirements** 

Rod, Bar, and Billet Diameter in. (mm)	Grain Size Number (weighted average)
0.5 to 2 incl (13 to 50 incl) 2 excl to 4.5 incl (50 excl to 115 incl) 4 excl to 6 incl (115 excl to 150 incl)	4.5 or finer 2.5 or finer 1.5 or finer
6 excl to 7.75 incl (150 excl to 200 incl) Greater than 7.75 (200)	1.0 or finer To be set in purchase order

**TABLE 2 Chemical Requirements** 

Element	Ingot Maximum Limit (ppm)	
Aluminum	100	
Carbon	200	
Chromium	100	
Copper	100	
Hydrogen	45	
Iron	200	
Nickel	100	
Nitrogen	150	
Oxygen	1000	
Silicon	100	
Tantalum	2500	
Titanium	46 to 48 %	

**TABLE 3 Permissible Variations in Diameters** 

Diameter in. (mm)	Tolerance, plus or minus, in. (mm)
0.51 to 1.0 (13.1 to 25)	0.010 (0.25)
1.1 to 4.0 (25.1 to 100)	0.015 (0.4)
Over 4.0 (100)	0.020 (0.5)

- 4.1.8 Inspection requirements (see Section 15),
- 4.1.9 Certification and report needs (see Section 17), and
- 4.1.10 Additions to the specification and supplementary requirements, as required.

#### 5. Materials and Manufacture

- 5.1 Materials covered by this specification shall be made from ingots which are produced by vacuum, plasma-arc or electron beam furnace melting, or a combination of these methods. All melting is to be carried out in furnaces usually used for reactive metals.
- 5.2 The products covered by this specification are formed with conventional forging, swaging, rolling, extruding, and drawing equipment normally available in metal working plants.

## 6. Chemical Composition

- 6.1 The Nb-Ti alloy ingots, billets, and rods covered by this specification shall conform to the chemical composition limits shown in Table 2.
- 6.2 The manufacturer's ingot analysis shall be considered the chemical analysis for the products supplied to this specification, except for the interstitials carbon, oxygen, nitrogen, and hydrogen. When specified in the purchase order, the analysis for these interstitials shall be measured on product.

### 7. Physical Properties

- 7.1 Unless otherwise specified in the purchase order, the material will be supplied in the annealed state.
- 7.2 The grain size of finished billets or rods shall meet the limits in Table 1 (see 14.3).
- 7.3 The product shall be free of cracks, laminations, inclusions, voids, and other ruptures with size larger than 3 % of the product diameter or 0.096 in. (2.5 mm) equivalent diameter, whichever is smaller. This characteristic shall be measured by ultrasonic testing (see 14.5).

### 8. Mechanical Properties

8.1 Hardness testing will be performed on each lot of finished product and the average of three readings shall be less than 170 DPH (see 14.2).

#### 9. Permissible Variations in Dimensions

- 9.1 Permissible variations in diameters for finished product shall be as specified in Table 3, unless otherwise agreed to between manufacturer and purchaser.
- 9.2 Permissible variations in length for finished product shall be as specified in the purchase order.

### 10. Workmanship, Finish, and Appearance

- 10.1 *Surface Condition*—The finished material shall be free of visually detectable cracks, seams, slivers, blisters, laps, gouges, and other injurious imperfections.
- 10.2 Liquid Penetrant Examination—The surfaces of billet and bar shall be examined using liquid penetrant inspection methods (see 14.4). The following indications are unacceptable:
  - 10.2.1 Cracks,
  - 10.2.2 Linear indications,
- 10.2.3 Rounded indications with dimensions exceeding 0.03 in. (0.8 mm), and
- 10.2.4 For sidewall surfaces only, rounded indications that are separated by less than 0.03 in. (0.8 mm) edge to edge.
- 10.3 *Surface Finish*—Surface finish shall be as specified in the purchase order (see 14.6).
- 10.4 Surface Preparation—The finished surface shall be pickled and rinsed in water. Removal of liquid penetrant test materials after pickling shall be by rinsing or additional pickling.
- 10.5 Cleanliness—Materials shall be clean to the extent that no contamination is visible to the unaided eye, corrected for 20/20 vision, when viewed under an illumination of at least 100 foot candles (1100 lux) on the surface being tested.

#### 11. Sampling

- 11.1 *Ingots*—Samples for ingot chemical analyses shall be taken on the ingot sidewall at least at three positions along the ingot including the middle and to within 5 in. (125 mm) of each end.
- 11.2 *Product*—Samples for chemical and mechanical testing shall be taken from the finished material after all metallurgical



processing to determine conformity to this specification. The samples may be taken prior to final inspection and minor surface conditioning by abrasion and pickling, and shall be representative of the finished product.

11.3 Care shall be exercised to ensure that the sample selected for testing is representative of the material and that it is not contaminated by the sampling procedure. If there is any questions relating to the sampling technique or the analysis thereof, the methods of sampling and analysis shall be as agreed upon between the purchaser and the manufacturer.

### 12. Number of Tests and Retests

- 12.1 *Initial Tests*—Each product sample shall be tested once for each product test requirement.
- 12.2 *Invalid Tests*—If any sample or test is found to be contaminated or improperly done, the result may be invalidated and a new test done to replace the original.
- 12.3 Retests—If a test result does not meet the specification or is questionable, retests may be performed on twice the number of samples originally tested. Both retest values must conform to the specification. All three values will be reported on the certification. The retest values shall be marked with an "R." Alternatively, each piece in the lot may be tested and deviant pieces rejected or reworked.
- 12.4 *Rework*—Product not meeting this specification may be reworked to meet this specification.

### 13. Significance of Numerical Limits

13.1 For the purpose of determining compliance with the specified limits for requirements of the properties listed in this specification, an observed value or a calculated value shall be rounded as indicated in accordance with the rounding method of Practice E29.

### 14. Test Methods

- 14.1 Analytical methods for chemical composition shall be in accordance with industry or manufacturer's standards. Guide E2626 may be used as a guide for chemical analysis techniques.
- 14.2 Hardness testing of product shall be according to Test Methods E92 or E384.
  - 14.3 Measure grain size according to Test Methods E112.
- 14.4 Perform liquid penetrant examination in accordance with Test Method E165.
- 14.5 Perform ultrasonic testing in accordance with Annex A1 for material greater than 2.0 in. (50 mm) in diameter and Practice E214 for material equal to or less than 2.0 in. (50 mm) in diameter.
- 14.6 Measure the surface finish when required by purchase order, in accordance with ANSI B46-1.

### 15. Inspection

15.1 In addition to the above specified inspections, the manufacturer shall inspect final product for dimensions and

identification. Other inspections shall be as agreed upon between purchaser and the manufacturer and included in the purchase order.

15.2 If so specified on the purchase order, the purchaser or his representative may witness the testing and inspection of the material at the place of manufacture. In such cases, the purchases shall state in the purchase order which tests are to be witness. The manufacturer shall give ample notice to the purchaser as to the time and place of the designated test. If the purchaser's representative is not present at the agreed upon time for the testing, and if no new date is agreed upon, the manufacturer shall consider the requirement for purchaser's inspection at the place of manufacture to be waived. When the inspector representing the purchaser does appear at the appointed place and time, the manufacturer shall afford all reasonable facilities to see that the material is being furnished in accordance with this specification. This inspection shall be conducted so as not to interfere unnecessarily with production operations.

### 16. Rejection and Rehearing

- 16.1 Material that does not conform to this specification or the purchase order may be rejected. The manufacturer may elect to repair the material or request a waiver from the customer.
- 16.2 In the event of a disagreement between the manufacturer and the purchaser concerning material compliance with the purchase order, a mutually acceptable referee may perform the tests in question. The referee's results shall be used in determining compliance.

### 17. Certification

17.1 The manufacturer shall furnish the purchaser with a certificate that the material was manufactured, sampled, tested and inspected in accordance with this specification and has been found to meet the requirements. The certificate shall include a report of the test results.

#### 18. Product Marking

18.1 Each billet, rod, bundle, or box shall be marked or tagged legibly and conspicuously with the number of this specification, type, temper, lot number, manufacturer's identification, nominal size, and the gross, net, and tar weights. If marking fluids are used, they shall be of such a nature as to be easily removed with cleaning solutions. The markings or their removal shall have no deleterious effect upon the material or its performance. The characters shall be sufficiently stable to withstand ordinary handling.

# 19. Packaging and Package Marking

- 19.1 All material shall be packed in such a manner as to ensure safe delivery to its destination.
  - 19.2 The box identification shall include the following:
  - 19.2.1 ASTM designation and alloy (NbTi),
  - 19.2.2 Purchase order number,
  - 19.2.3 Lot number,
  - 19.2.4 Number of pieces,



19.2.5 Manufacturer's name,

19.2.6 Gross, tare, and net weights, and

19.2.7 Size.

### 20. Keywords

20.1 niobium; niobium alloy; niobium-titanium alloy; superconductor; titanium; titanium alloy

#### **ANNEX**

(Mandatory Information)

#### A1. ULTRASONIC EXAMINATION OF NB-TI SUPERCONDUCTOR QUALITY MATERIAL - ROD, BAR, AND BILLET

### A1.1 Scope

### A1.1.1 Application:

A1.1.1.1 This annex establishes the minimum requirements for the ultrasonic inspection of cylindrical Nb-Ti alloy rod, bar, and billet intended for superconductor applications.

A1.1.1.2 Intermediate sizes of material are inspected using the longitudinal wave mode only. Smaller diameters (generally those under 1.5 in. (38 mm) are inspected using both shear and longitudinal wave modes.

### A1.1.2 *Methodology:*

A1.1.2.1 Ultrasonic waves are transmitted into the material under test. Waveforms reflected from surface and internal discontinuities are displayed on a CRT in the form of an A-Scan. The amplitude of the reflection from the area of the waveform of interest is recorded on a strip chart or equivalent device.

A1.1.2.2 Indications detected are evaluated on the CRT screen and/or recording. After comparison with the amplitudes of calibration reflectors, acceptability of the material is determined according to the appropriate accept/reject criteria. Rejectable areas are noted on each piece, and the inspection results for each piece tabulated on the inspection report.

### A1.2 Requirements

### A1.2.1 Equipment:

A1.2.1.1 *Electronic Apparatus*—The ultrasonic instrument shall be capable of generating, receiving, and amplifying high-frequency electrical pulses at frequencies and energy levels required to resolve the applicable calibration reflectors and perform a meaningful inspection.

A1.2.1.2 *Immersion Search Units*—Focused, immersion transducers shall be used for shear and longitudinal wave modes of inspection. The minimum (nominal) transducer frequency should be 2.25 MHz, although lower frequencies may be used if required.

A1.2.1.3 *Mechanical Apparatus*—The material to be inspected and the search unit assembly shall have automatic rotation and translation relative to each other, so that a helical scan of the material will be performed. Water shall be used as the couplant and may be treated with rust inhibitors, softeners, and wetting agents or heated to a sufficient temperature, or both, to reduce the formation of air bubbles.

A1.2.2 *Personnel*—Personnel performing ultrasonic inspections using this procedure shall be qualified and certified in accordance with the latest revision of ASNT SNT-TC-1A.

#### A1.3 Standardization

A1.3.1 Surface Condition of Test Material—The sound beam entry surface of the material under test shall be free of scale, dirt, or other foreign materials which could prevent the material from being tested at the required sensitivity.

### A1.3.2 Reference Standards:

A1.3.2.1 Fabrication—A reference standard containing reference reflectors shall be fabricated from material of the same nominal diameter, surface finish, heat treatment, and alloy (or acoustically similar material) as the material to be inspected. Standards of different diameter may be used provided the required thickness of material is covered by this specification. The attenuation of the standard back reflection should be within 25 % of the material to be inspected, or an appropriate attenuation correction should be utilized (this correction can include the use of a zoned, or stepped, test where successive depth regions are inspected in successive scans). The material used for the ultrasonic reference standard shall be free of internal reflectors that may interfere with or be confused with the reference reflectors.

A1.3.2.2 *Reference Reflectors*—The reference standard shall contain reference reflectors as described below.

A1.3.2.3 Longitudinal Wave Examination—For material 1.5 in. (38 mm) in diameter or less, the standard shall contain four radially drilled flat-bottomed holes (FBH) at depths given in Table A1.1. For material greater than 1.5 in. (38 mm) in diameter, the reference standard shall contain a minimum of two holes drilled at depths as given in Table A1.1. Dimensions of the FBHs are given in Table A1.1.

A1.3.2.4 *Circumferential Shear Wave*—For standardization of the circumferential shear wave examination, the standard shall contain a notch parallel to the longitudinal axis. Notch dimensions are given in Table A1.1.

A1.3.2.5 Axial Shear Wave—For standardization of the axial shear wave examination, the standard shall contain a notch transverse to the longitudinal axis. As an alternate method, standardization of the axial shear wave examination may use a hole drilled from the end, located a distance of 25 % of the diameter ( $\pm 0.005$  in. (.13 mm)) from the center of the bar. Hole and notch dimensions are given in Table A1.1.

A1.3.2.6 Other Configurations—Other standard configurations may be used as required by the individual contract. All standards shall be permanently identified and shall have an accompanying drawing.

### A1.3.3 Standardization of Apparatus:

**TABLE A1.1 Recommended Reference Reflector Specifications** 

Note 1—All dimensions given in inches.

Material Diameter	FBH Diameter, max	FBH Depths	End Hole Depth and Diameter	Notch Dimensions
$0.5 \leq D \leq 0.65$	0.014	0.15 D (±0.010) 0.30 D (±0.010) 0.50 D (±0.010) 0.60 D (±0.010)	0.50 0.013 diameter	0.03D deep 0.03D wide 0.125 long (maximums)
$0.65 < D \le 1.5$	0.03D or 0.032	0.15 D (±0.010) 0.30 D (±0.010) 0.50 D (±0.010) 0.60 D (±0.010)	0.50 0.03D or 0.032	0.03D or 0.032 deep 0.03D or 0.032 wide 0.125 long (maximums)
$1.5 < D \le 4.0$	0.03D or 0.064	0.90 <i>D</i> or ( <i>D</i> -0.125) and < 0.50 <i>D</i> or > 0.50 <i>D</i> and 0.10 <i>D</i> (±0.010)	N/A	N/A
$4.0 < D \le 8.0$	0.096	0.90 <i>D</i> or ( <i>D</i> -0.125) and < 0.50 <i>D</i> or > 0.50 <i>D</i> and 0.10 <i>D</i> (±0.010)	N/A	N/A
D > 8.0	0.127	0.90 <i>D</i> or ( <i>D</i> -0.125) and < 0.50 <i>D</i> or > 0.50 <i>D</i> and 0.10 <i>D</i> (±0.010)	N/A	N/A

A1.3.3.1 Reference Amplitudes—The equipment shall be standardized using the appropriate reference standard. There shall be a separate transducer and instrument channel used for each of the longitudinal, axial shear, and circumferential shear wave examinations. The sensitivity of the system must be set to obtain a pulse height on the instrument screen and chart recording of greater than 50 % of full scale for each reference reflector.

A1.3.3.2 *Coverage*—Sufficient transducer overlap must be maintained to ensure 100 % coverage of the material under test

A1.3.3.3 *Standardization*—Standardization shall be accomplished as follows:

A1.3.3.4 The longitudinal wave transducer shall be positioned normal to the surface of the reference standard, and oriented to obtain maximum signal response from the flatbottomed holes. Defect alarm gates shall be positioned between the front and back surface reflections so that the signal from each FBH is within the gate.

A1.3.3.5 The circumferential shear wave transducer shall be positioned to maximize the signal response from the longitudinal notch or the end-drilled hole. A defect alarm gate shall be positioned to include this signal.

A1.3.3.6 The axial shear wave transducer shall be set up to maximize the shear wave signal response from the transverse notch. A defect alarm gate shall be positioned to include this signal.

A1.3.3.7 The sensitivity of the system must be set to obtain a pulse height on the instrument screen and chart recording of greater than 50% of full scale for each calibration standard defect.

A1.3.3.8 *Verification*—The reference standard must be scanned and the reference reflectors detected at the beginning

and end of each lot, at intervals not exceeding one hour, and at the beginning and end of each separate chart recording when more than one chart recording is used to describe a lot. If the standard is not reproduced with responses from the artificial defects within 80 percent of the initial amplitude, all product inspected since the last acceptable reproduction of the reference standard must be reinspected.

### **A1.4 Product Inspection**

A1.4.1 Material with diameters from 0.5 to 1.5 in. (13 to 38 mm) (inclusive) shall be inspected using both longitudinal and shear wave modes. Material with diameter greater than 1.5 in. (38 mm) requires use of the longitudinal mode only. All product inspection shall be under the same conditions as those used at calibration (feed rate, system rotational speed, gain settings, etc.).

A1.4.1.1 Water Path—The water path during testing shall be within  $\pm 10\%$  of the water path used at standardization.

A1.4.2 Scanning and Index—The product shall be inspected by a continuous and overlapping scan indexed in a direction parallel to the longitudinal axis of the piece. The scanning speed and index of the search unit must be the same as that at which standardization is performed.

A1.4.3 System Adjustment—When production material has a different diameter than the calibration standard, adjustment of the gate length and transducer vertical position are permitted. No adjustments affecting test sensitivity or resolution are permitted during the sequence of calibration, test of production material, and subsequent rerun of the reference standard.

A1.4.4 Multiple Scans of Attenuative Materials—Larger diameter material with high attenuation properties may require a multiple-step, or "zoned" inspection if the longer metal path

reference reflectors can only be detected at a gain setting which causes saturation of those reflectors at a shorter metal path. In this event, the material shall be scanned in zones corresponding to the depths where acceptable signals are obtained. This requires the use of additional flat-bottomed holes in the standard to ensure adequate depth coverage. The zones shall be contiguous so that complete coverage is achieved, with the reference reflector in the linking the zones being displayed on both scans. The use of and description of the zones shall be noted on the test report.

### **A1.5** Interpretation of Results

A1.5.1 *Acceptance*—Material inspected in accordance with this test procedure shall be acceptable if no indications are present that exceed the following:

A1.5.1.1 *Shear Wave Mode*—Material inspected in the shear wave mode shall be rejected if defect indications are found to exceed 80 % of the amplitude established at calibration.

A1.5.1.2 Longitudinal Wave Mode—Material inspected in the longitudinal mode shall be rejected if defect indications are found to exceed the smallest amplitude established at calibration.

A1.5.2 Surface Indications—Material rejected above where defect indications are clearly associated with surface defects may be accepted after repair and retesting for acceptability per this procedure.

A1.5.3 Defect Removal After Inspection—Material rejected above may be accepted if defective areas are clearly and permanently marked on the material, and removed in subsequent trimming operations.

# A1.6 Test Report

A1.6.1 At minimum, record the following data on the test report. Maintain test reports in accordance with contract requirements:

A1.6.1.1 *Material Identification*—Heat number, size, lot number, etc.,

A1.6.2 Specific written test procedure,

A1.6.3 Type of test, that is, longitudinal wave, circumferential shear, etc.,

A1.6.4 Reference standard identification, and

A1.6.5 Number of acceptable and rejectable pieces.

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