Standard Specification for Niobium and Niobium Alloy Seamless and Welded Tubes

This standard is issued under the fixed designation B394; 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 (´) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification covers wrought niobium and niobium alloy seamless and welded tubes as follows:

1.1.1 R04200-Type 1—Reactor grade unalloyed niobium,
1.1.2 R04210-Type 2—Commercial grade unalloyed niobium,
1.1.3 R04251-Type 3—Reactor grade niobium alloy containing 1% zirconium, and
1.1.4 R04261-Type 4—Commercial grade niobium alloy containing 1% zirconium.

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 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:

B391 Specification for Niobium and Niobium Alloy Ingots
E8 Test Methods for Tension Testing of Metallic Materials
E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications
E2626 Guide for Spectrometric Analysis of Reactive and Refractory Metals

3. Terminology

3.1 Definitions of Terms Specific to This Standard:

3.1.1 Lot, n—a lot shall consist of all material produced from the same ingot at one time, with the same cross section, processed with the same nominal metallurgical parameters and heat treated at the same conditions.

4. Ordering Information

4.1 Orders for materials under this specification shall include the following information as applicable:

4.1.1 Type and grade (Section 1),
4.1.2 ASTM designation and year of issue,
4.1.3 Welding (Section 5),
4.1.4 Quantity in weight, number of pieces, and dimensions,
4.1.5 Chemistry (6.3),
4.1.6 Temper designation (Section 8),
4.1.7 Permissible variations in length and quantity or weight (9.2, 9.4, and Table 1H),
4.1.8 Quality and finish (10.4),
4.1.9 Sampling (11.2),
4.1.10 Hydrostatic or pneumatic test (14.2),
4.1.11 Inspection (Section 15),
4.1.12 Required reports (Section 17), and
4.1.13 Additions to the specification and supplementary requirements, as required.

5. Materials and Manufacture

5.1 Material covered by this specification shall be made from ingots that conform to Specification B391 and that are produced by vacuum or plasma arc melting, vacuum electron-beam melting, or a combination of these three methods.

5.2 Seamless tubes may be made by any seamless method that will yield a product meeting the requirements of this specification, such as, but not limited to, extrusion of billets with subsequent cold working by drawing, swaging, or pilgering, with intermediate anneals, until the final dimensions are reached.

5.3 Welded tubing shall be made from flat-rolled products by an automatic or semiautomatic welding process with no addition of filler metal in the welding operation. Other methods of welding, such as the addition of filler metal or hand welding,
may be employed if approved by the purchaser and tested by methods agreed upon between the manufacturer and the purchaser. The manufacturer must use proper precautions to prevent contamination during welding.

6. Chemical Requirements

6.1 The niobium and niobium alloy ingots and billets for conversion to finished products covered by this specification shall conform to the requirements for chemical composition as prescribed in Table 2.

<p>| TABLE 1 Permissible Variations in Diameter and Wall Thickness Measured at any Location |</p>
<table>
<thead>
<tr>
<th>Nominal Outside Diameter, in. (mm)*</th>
<th>Variation in Outside Diameter, Over and Under, in. (mm)*</th>
<th>Variation in Inside Diameter, Over and Under, in. (mm)*</th>
<th>Variation in Wall Thickness, Over and Under, %C,D</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.187 to 0.625 (4.7 to 15.9), excl</td>
<td>0.004 (0.010)</td>
<td>0.004 (0.010)</td>
<td>10</td>
</tr>
<tr>
<td>0.625 to 1.000 (15.9 to 25.4), excl</td>
<td>0.005 (0.13)</td>
<td>0.005 (0.13)</td>
<td>10</td>
</tr>
<tr>
<td>1.000 to 2.000 (25.4 to 50.8), excl</td>
<td>0.0075 (0.19)</td>
<td>0.0075 (0.19)</td>
<td>10</td>
</tr>
<tr>
<td>2.000 to 3.000 (50.8 to 76.2), excl</td>
<td>0.010 (0.25)</td>
<td>0.010 (0.25)</td>
<td>10</td>
</tr>
<tr>
<td>3.000 to 4.000 (76.2 to 101.6), excl</td>
<td>0.0125 (0.32)</td>
<td>0.0125 (0.32)</td>
<td>10</td>
</tr>
</tbody>
</table>

*These tolerances are applicable to only two dimensions, such as outside diameter and wall, or inside diameter and wall, or outside diameter and inside diameter.

*For applicable tolerances for very small tubes, less than 0.187 in. (4.9 mm) in outside diameter, or very thin wall tubes, less than 0.010 in. (0.25 mm), the producer should be consulted.

C When tubes as ordered require wall thicknesses % in. (19.05 mm) or over, or an inside diameter 60 % or less of the outside diameter, a wider variation in wall thickness is required. On such sizes, a variation in wall thickness of 12.5 % over and under will be permitted.

D Ovality measured at any cross section: For tubes with nominal wall thickness less than 3 % of the nominal outside diameter, the ovality tolerance is double the tolerance shown in the second and third columns.

6.2 The manufacturer’s ingot analysis shall be considered the chemical analysis for products supplied under this specification, except for interstitials as specified in 6.3. Alternately, an analysis of a representative sample of in process or final product from the same ingot may be substituted.

6.3 When requested by the purchaser at the time of purchase, the manufacturer shall furnish a report certifying the values of the interstitial elements (C, O, N, H) on end product as prescribed in Table 3 for each lot of material supplied. End product interstitial samples must be taken after all thermal and chemical processing.

6.4 Guide E2626 is recommended as a guide, where applicable.

7. Mechanical Requirements

7.1 The annealed materials supplied under this specification shall conform to the requirements for mechanical properties as specified in Table 4.

8. Temper Designations

8.1 Unless otherwise stated, the materials supplied under these specifications shall be in the fully annealed condition, that is, at least 90 % recrystallized.

<table>
<thead>
<tr>
<th>TABLE 2 Chemical Requirements</th>
</tr>
</thead>
<tbody>
<tr>
<td>Element</td>
</tr>
<tr>
<td>Carbon</td>
</tr>
<tr>
<td>Nitrogen</td>
</tr>
<tr>
<td>Oxygen</td>
</tr>
<tr>
<td>Hydrogen</td>
</tr>
<tr>
<td>Zirconium</td>
</tr>
<tr>
<td>Tantalum</td>
</tr>
<tr>
<td>Iron</td>
</tr>
<tr>
<td>Silicon</td>
</tr>
<tr>
<td>Tungsten</td>
</tr>
<tr>
<td>Nickel</td>
</tr>
<tr>
<td>Molybdenum</td>
</tr>
<tr>
<td>Hafnium</td>
</tr>
<tr>
<td>Titansium</td>
</tr>
<tr>
<td>When specified:</td>
</tr>
<tr>
<td>Boron</td>
</tr>
<tr>
<td>Aluminum</td>
</tr>
<tr>
<td>Beryllium</td>
</tr>
<tr>
<td>Chromium</td>
</tr>
<tr>
<td>Cobalt</td>
</tr>
</tbody>
</table>

8.2 Guide E2626 is recommended as a guide, where applicable.

TABLE 3 Additional Chemical Requirements for Finished Product (When Specified by Purchaser)

<table>
<thead>
<tr>
<th>Element</th>
<th>Type 1 (Reactor Grade Unalloyed Niobium)</th>
<th>Type 2 (Commercial Grade Unalloyed Niobium)</th>
<th>Type 3 (Reactor Grade Niobium—1 % Zirconium)</th>
<th>Type 4 (Commercial Grade Niobium—1 % Zirconium)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Oxygen</td>
<td>0.0250</td>
<td>0.0400</td>
<td>0.0250</td>
<td>0.0400</td>
</tr>
<tr>
<td>Carbon</td>
<td>0.0100</td>
<td>0.0150</td>
<td>0.0100</td>
<td>0.0150</td>
</tr>
<tr>
<td>Nitrogen</td>
<td>0.0100</td>
<td>0.0100</td>
<td>0.0100</td>
<td>0.0100</td>
</tr>
<tr>
<td>Hydrogen</td>
<td>0.0015</td>
<td>0.0015</td>
<td>0.0015</td>
<td>0.0015</td>
</tr>
</tbody>
</table>

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TABLE 4 Mechanical Properties for Material, Annealed Condition
(90 % Minimum Recrystallized)

<table>
<thead>
<tr>
<th>Grade</th>
<th>Ultimate Tensile Strength, min, psi (MPa)</th>
<th>Yield Strength (0.2 % offset), min, psi (MPa)</th>
<th>Elongation in 1-in. (25-mm) gage length, min, %</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type 1 – welded</td>
<td>18 000 (125)</td>
<td>10 500 (73)</td>
<td>25</td>
</tr>
<tr>
<td>Type 1 – seamless</td>
<td>18 000 (125)</td>
<td>8500 (59)</td>
<td>25</td>
</tr>
<tr>
<td>Type 2 – welded</td>
<td>18 000 (125)</td>
<td>10 500 (73)</td>
<td>25</td>
</tr>
<tr>
<td>Type 2 – seamless</td>
<td>18 000 (125)</td>
<td>8500 (59)</td>
<td>25</td>
</tr>
<tr>
<td>Type 3</td>
<td>28 000 (195)</td>
<td>18 000 (125)</td>
<td>20</td>
</tr>
<tr>
<td>Type 4</td>
<td>28 000 (195)</td>
<td>18 000 (125)</td>
<td>20</td>
</tr>
</tbody>
</table>

8.2 Other temper designations, such as cold-worked temper or stress-relieved temper, can be specified as agreed upon between the purchaser and the manufacturer at the time of purchase.

9. Permissible Variations in Dimensions and Weight

9.1 Diameter and Wall Thickness—The permissible variations in diameter and wall thickness of the tubes shall not exceed those prescribed in Table 1.

9.2 Length—When tube is ordered cut to length, the useable length shall not be less than that specified, but a variation of +½ in. (3.18 mm) will be permitted for lengths up to 6 ft (1.8 m). For lengths over 6 ft, a variation of +½ in. (6.4 mm) will be permitted, unless otherwise specified at the time of purchase.

9.3 Straightness—The tube shall be free of bends or kinks, and the maximum bow shall not exceed values shown in Table 5.

9.4 Quantity or Weight—For orders requiring up to 100 ft (30.5 m) of finished tubing, the manufacturer may overship by 20 %. When the order is for quantities up to 1000 ft (305 m) or 1000 lb (453.6 kg), the manufacturer may overship by 10 %. The permissible overshipment shall be negotiated for orders larger than this quantity.

10. Quality and Finish

10.1 Finished niobium and niobium alloy tubes shall be free of injurious internal and external imperfections of a nature that will interfere with the purpose for which it was intended.

10.2 The finished tubes shall be visibly free of oxide, grease, oil, residual lubricants, and other extraneous materials.

10.3 The finished tubes shall be free of cracks, seams, slivers, burrs, blisters, and other injurious imperfections exceeding 10 % of the nominal wall thickness.

10.4 Methods of testing for these defects and standards of acceptability shall be as agreed upon between the manufacturer and the purchaser.

11. Sampling

11.1 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.

11.2 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 question 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 Two samples from each lot of tubes shall be tested for flare and tensile properties in the longitudinal direction.

12.2 When hydrostatic or pneumatic testing is required, all tubes in the lot shall be tested.

12.3 If end-product chemical tests are required (6.3), one chemical test shall be made from each lot of finished product.

12.4 Retests—If any sample or specimen exhibits obvious surface contamination or improper preparation, disqualifying it as a truly representative sample, it shall be discarded and a new sample or specimen substituted.

12.4.1 In case of a failure, retest two additional specimens. If both retest specimens conform to this specification, discard the original values and consider the material acceptable; otherwise, the lot shall be rejected or reworked and tested.

12.5 If the results of the final tube inspections are not in conformance with the requirements of this specification, the lot may be reworked at the option of the manufacturer. The lot shall be acceptable if results of all tests, after reworking, conform to this specification.

13. Significance of Numerical Limits

13.1 For the purposes 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 Tension Test—Conduct tension tests in accordance with Test Methods E8. Determine the yield strength by the 0.2 % offset method. Small size, 1-in. (25.4-mm) gage length specimens proportional to the standard specimen can be used. Determine tensile properties using a strain rate of 0.003 to 0.007 in./in./min (mm/mm/min) through the yield point. After the yield strength has been exceeded, increase the cross-head speed to approximately 0.05 in./in./min (mm/mm/min) to failure.

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14.2 Hydrostatic and Pneumatic Test—These tests are optional. Should the purchaser require either or both of these tests on finished tubular product, the requirement must be agreed upon at the time of purchase. The cost of this special provision will be negotiated between the purchaser and the manufacturer at the time of purchase. The procedure for the hydrostatic test is outlined in 14.2.1, and the procedure for the pneumatic test is outlined in 14.2.2. If both tests are specified by the purchaser, the hydrostatic test will be done first.

14.2.1 Hydrostatic Test—Each tube so tested shall withstand without showing bulges, leaks, or other defects, an internal hydrostatic pressure that will produce in the tube wall a stress of 75% of the minimum specified yield strength at room temperature. This pressure shall be determined by the following equation:

\[ P = \frac{2ST}{D} \]  

(1)

where:

- \( P \) = minimum hydrostatic pressure, psi (or MPa),
- \( S \) = allowable fiber stress of 75% of the minimum yield strength, psi (or MPa),
- \( t \) = average wall thickness of the tube, in. (or mm), and
- \( D \) = outside diameter of the tube, in. (or mm).

Maintain the test pressure for at least 10 s.

14.2.2 Pneumatic Test—Each tube so tested shall withstand an internal air pressure of 100 psi (.7 MPa) minimum for a minimum of 5 s without showing evidence of leakage. The test method used, for example, placing the tube under water or using the pressure differential method, shall permit easy visual detection of any leakage. Any evidence of leakage shall be cause for rejection of that tube.

14.3 Flare—A section of the finished tube shall be capable of being flared without cracking that is visible to the unaided eye. The flare shall be made with a tool having a 60° included angle until the outside diameter has been increased 15%.

15. Inspection

15.1 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 purchaser shall state in his purchase order which tests he desires to 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 does not present himself at the agreed-upon time for the testing, and if no new date is set, 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 him 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 fails to conform to the requirements of this specification may be rejected. Rejection should be reported to the producer or supplier promptly and in writing. In case of dissatisfaction with the results of the test, the producer or supplier may make claim for a rehearing.

16.2 In the event of disagreement between the manufacturer and the purchaser about the conformance of the material to the requirements of this specification or any special test specified by the purchaser, a mutually acceptable referee shall perform the tests in question. The results of the referee’s testing shall be used in determining conformance of the material to this specification.

17. Certification

17.1 A producer’s or supplier’s certification shall be furnished to the purchaser that the material was manufactured, sampled, tested, and inspected in accordance with this specification and has been found to meet the requirements. When specified in the purchase order or contract, a report of the test results shall be furnished.

18. Product Marking

18.1 Each bundle, box, or carton shall be marked or tagged legibly and conspicuously, at a minimum, with the heat number, alloy or type, manufacturer’s identification, nominal size, and the gross, net, and tare 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 when properly transported by any common carrier.

20. Keywords

20.1 niobium; niobium alloy; seamless tubes; seamless and welded tubes; welded tubes