Designation: <del>B351/B351M - 08<sup>ε1</sup> B351/B351M - 13</del>

Used in USDOE-NE standards

# Standard Specification for Hot-Rolled and Cold-Finished Zirconium and Zirconium Alloy Bars, Rod, and Wire for Nuclear Application<sup>1</sup>

This standard is issued under the fixed designation B351/B351M; 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.

ε<sup>1</sup> NOTE—An editorial correction was made in paragraph 15.2.2.1 in June 2009.

# 1. Scope

- 1.1 This specification covers four grades of wrought zirconium and zirconium alloy bars, rod, and wire as follows:
- 1.1.1 R60001—Unalloyed grade,
- 1.1.2 R60802—Zirconium-Tin alloy (Zircaloy 2),
- 1.1.3 R60804—Zirconium-Tin alloy (Zircaloy 4), and
- 1.1.4 R60901—Zirconium-Niobium alloy.
- 1.2 Unless a single unit is used, for example corrosion mass gain in mg/dm<sup>2</sup>, the values stated in either inch-pound or SI units are to be regarded separately as standard. The values stated in each system are not exact equivalents; therefore each system must be used independently of the other. SI values cannot be mixed with inch-pound values.
- 1.3 The following precautionary caveat pertains only to the test method portions 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>

(https://standards.iteh.ai)

B350/B350M Specification for Zirconium and Zirconium Alloy Ingots for Nuclear Application E8/E8M Test Methods for Tension Testing of Metallic Materials

E21 Test Methods for Elevated Temperature Tension Tests of Metallic Materials

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

E114 Practice for Ultrasonic Pulse-Echo Straight-Beam Contact Testing

E214 Practice for Immersed Ultrasonic Testing by the Reflection Method Using Pulsed Longitudinal Waves (Withdrawn 2007)<sup>3</sup> G2/G2M Test Method for Corrosion Testing of Products of Zirconium, Hafnium, and Their Alloys in Water at 680°F (360°C) or in Steam at 750°F (400°C)

#### 3. Terminology

- 3.1 Definitions of Terms Specific to This Standard:
- 3.1.1 annealed, n—denotes material that exhibits a recrystallized grain structure.
- 3.2 Lot Definition:
- 3.2.1 *lot*, *n*—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 a 12 h period for final continuous anneal, or to a single furnace load for final batch anneal.
  - 3.3 Forms Definitions:
  - 3.3.1 bar, n:

<sup>&</sup>lt;sup>1</sup> This specification is under the jurisdiction of ASTM Committee B10 on Reactive and Refractory Metals and Alloys and is the direct responsibility of Subcommittee B10.02 on Zirconium and Hafnium.

Current edition approved April 1, 2008Oct. 1, 2013. Published April 2008November 2013. Originally approved in 1960. Last previous edition approved in 20072008 as 2007208 and 2007208 as 2007208 and 200720

<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>&</sup>lt;sup>3</sup> The last approved version of this historical standard is referenced on www.astm.org.



- 3.3.1.1 rounds, squares and hexagons,  $n=\frac{3}{8}$  in. [9.5 mm] and over in diameter or size.
- 3.3.1.2 flats, n— $\frac{1}{4}$  to 10 in. [6.4 to 250 mm] inclusive in width and  $\frac{1}{8}$  in. [3.2 mm] and over in thickness. Thickness  $\frac{1}{8}$  in. [3.2 mm] to under  $\frac{3}{16}$  in. [4.8 mm] can be cold-rolled strip as well as bar.
  - 3.3.2 rod, n—rounds in coils for subsequent reworking  $\frac{1}{4}$  to  $\frac{3}{4}$  in. [6.4 to 19 mm] in diameter.
- 3.3.3 wire, n—material less than 3/8 in. [9.5 mm] in diameter or size, in round, hexagonal, or octagonal cross section, furnished in coils or on spools or reels.

# 4. Ordering Information

- 4.1 Orders for materials under this specification shall include the following information as applicable:
- 4.1.1 Grade (see Section 1),
- 4.1.2 Dimensions and form (see 3.3),
- 4.1.3 Chemical analysis of elements not listed (see 6.1.3),
- 4.1.4 Product analysis (see 6.1.1.1),
- 4.1.5 Tensile test temperatures (7.1),
- 4.1.6 Material finish (Section 12),
- 4.1.7 Metallurgical condition (see 8.1 and 8.2),
- 4.1.8 Ultrasonic test standard hole size (see 9.2.2),
- 4.1.9 Additional ultrasonic tests (see 9.2.4),
- 4.1.10 Workmanship standards and methods of inspection (Section 13),
- 4.1.11 Product marking (Section 19), and
- 4.1.12 Packaging and package marking (Section 20).
- 4.2 In addition to the data specified in 4.1, the following options and points of agreement between the manufacturer and the purchaser should be specified on the purchase order as required:
  - 4.2.1 Tolerances (Section 11),
  - 4.2.2 Workmanship standards (Section 13),
  - 4.2.3 Special tests (Section 9),
  - 4.2.4 Inspection (Section 16),
  - 4.2.5 Corrosion visual standards (9.1.2.19.1.2), and
  - 4.2.6 Oxygen limits (see footnote A, Table 1).

### 5. Materials and Manufacture

- 5.1 Material covered by this specification shall be made from ingots that conform to Specification B350/B350M and that are produced by vacuum or plasma arc melting, vacuum electron-beam melting, a combination of these three methods or other melting processes conventionally used for reactive metals. All processes to be done in furnaces usually used for reactive metals.
- 5.2 The various mill products covered by this specification shall be formed with the conventional extrusion, forging, or rolling equipment normally found in primary ferrous and nonferrous plants.

#### 6. Chemical Composition

- 6.1 The grades of zirconium and zirconium alloy metal covered by this specification shall conform to the chemical composition requirements prescribed in Table 1.
- 6.1.1 The elements listed in Table 1 are intentional alloy additions or elements that are inherent to the manufacture of sponge, ingot or mill product.
- 6.1.1.1 Elements other than those listed in Table 1 are deemed to be capable of occurring in the grades listed in Table 1 by and only by way of unregulated or unanalyzed scrap additions to the ingot melt. Therefore, product analysis for elements not listed in Table 1 shall not be required unless specified in the purchase order and shall be considered to be in excess of the intent of this specification.
  - 6.1.2 Elements intentionally added to the melt must be identified, analyzed, and reported in the chemical analysis.
- 6.1.3 When agreed upon by producer and purchaser and requested by the purchaser in his written purchase order, chemical analysis shall be completed for specific residual elements not listed in this specification.
- 6.2 The manufacturer's ingot analysis shall be considered the chemical analysis, except for hydrogen, oxygen, and nitrogen content, which shall be determined on the finished product. Alternatively, the manufacturer may sample an intermediate or final size during processing with the same frequency and in the same positions relative to the ingot, as specified in Specification B350/B350M, to determine the composition, except for hydrogen, oxygen, and nitrogen, which shall be determined on the finished product.
  - 6.3 Check Analysis:

#### **TABLE 1 Chemical Requirements**

Flament	Composition, Weight %				
Element	UNS R60001	UNS R60802	UNS R60804	UNS R60901	
Tin		1.20-1.70	1.20-1.70		
Iron		0.07-0.20	0.18-0.24		
Chromium		0.05-0.15	0.07-0.13		
Nickel		0.03-0.08			
Niobium (columbium)				2.40-2.80	
Oxygen	A	Α	Α	0.09-0.15	
Iron + chromium + nickel		0.18-0.38			
Iron + chromium			0.28-0.37		
	Maximum Impurities	s, Weight %			
Aluminum	0.0075	0.0075	0.0075	0.0075	
Boron	0.00005	0.00005	0.00005	0.00005	
Cadmium	0.00005	0.00005	0.00005	0.00005	
Calcium		0.0030	0.0030		
Carbon	0.027	0.027	0.027	0.027	
Chromium	0.020			0.020	
Cobalt	0.0020	0.0020	0.0020	0.0020	
Copper	0.0050	0.0050	0.0050	0.0050	
Hafnium	0.010	0.010	0.010	0.010	
Hydrogen	0.0025	0.0025	0.0025	0.0025	
Iron	0.150			0.150	
Magnesium	0.0020	0.0020	0.0020	0.0020	
Manganese	0.0050	0.0050	0.0050	0.0050	
Molybdenum	0.0050	0.0050	0.0050	0.0050	
Nickel	0.0070		0.0070	0.0070	
Niobium		0.0100	0.0100		
Nitrogen	0.0080	0.0080	0.0080	0.0080	
Phosphorus				0.0020	
Silicon	0.0120	0.0120	0.0120	0.0120	
Tin	0.0050			0.010	
Tungsten	0.010	0.010	0.010	0.010	
Titanium	0.0050	0.0050	0.0050	0.0050	
Uranium (total)	0.00035	0.00035	0.00035	0.00035	

A When so specified in the purchase order, oxygen shall be determined and reported. Maximum, minimum, or both, permissible values should be specified in the purchase order.

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- 6.3.1 Check analysis is an analysis made by the purchaser or the manufacturer of the metal after it has been processed into finished mill forms, and is either for the purpose of verifying the composition of a heat or lot or to determine variations in the composition within a heat or lot.
  - 6.3.2 Check analysis limits shall be as specified in Table 2.
- 6.3.3 Check analysis tolerances do not broaden the specified heat analysis requirements but cover variations between laboratories in the measurement of chemical content.
  - 6.3.4 The manufacturer shall not ship material that is outside the limits specified in Table 1 for the applicable grade.

#### 7. Mechanical Properties

- 7.1 The material, as represented by the test specimens, shall conform to the tensile properties prescribed in Table 3 when tested at ambient temperature. Elevated temperature properties shall be used to determine compliance only when specified in the purchase order.
  - 7.2 Requirements for mechanical properties do not apply to wire.

**TABLE 2 Permissible Variations in Product Analysis** 

Alloying Elements	Permissible Variation from the Specified Range (Table 1), %		
Tin	0.050		
Iron	0.020		
Chromium	0.010		
Nickel	0.010		
Iron + chromium	0.020		
Iron + chromium + nickel	0.020		
Niobium	0.050		
Oxygen	0.020		
Each Impurity Element	20 ppm or 20 % of the specified limit, whichever is smaller		

TABLE 3 Mechanical Properties of Annealed Material, Tested in the Longitudinal Direction

Grade	Condition	Temperature	Tensile Strength, min ksi [MPa]	Yield Strength (0.2 % offset), min ksi [MPa]	Elongation in 2 in. or 50 mm, min % <sup>A</sup>
R60001	annealed	RT <sup>B</sup>	42 [290]	20 [140]	18
R60802	annealed	RT	60 [415]	35 [240]	14
R60802	annealed	600°F [316°C]	31 [215]	15 [105]	24
R60804	annealed	RT	60 [415]	35 [240]	14
R60804	annealed	600°F [316°C]	31 [215]	15 [105]	24
R60901	cold worked	RT	74 [510]	50 [345]	10
R60901	annealed	RT	65 [450]	45 [310]	15

<sup>&</sup>lt;sup>A</sup> When a sub-size specimen is used, the gage length shall be as specified in Test Methods E8/E8M for that specimen.

#### 8. Metallurgical Properties

- 8.1 Unless otherwise stated in the purchase order, the cold worked and annealed materials supplied under these specifications shall be in the fully annealed condition, that is, at least 90 % recrystallized. The percent recrystallization of hot worked and annealed material shall be as agreed upon between the purchaser and manufacturer at the time of purchase.
- 8.2 Grade R60901 in sizes under 1 in. [25 mm] in minimum dimension furnished under this specification shall be in the cold-worked condition unless otherwise specified. Sizes 1 in. [25 mm] and over in minimum dimension shall be furnished in the annealed condition unless otherwise specified.
- 8.3 Other conditions, such as cold-worked or stress-relieved, can be specified as agreed upon between the purchaser and the manufacturer at the time of purchase.

# 9. Special Requirements

- 9.1 *Corrosion Properties:*
- 9.1.1 The product shall be corrosion resistant when tested in accordance with 15.2.4 and shall meet the criterion in 9.1.2.
- 9.1.2 Acceptance Criterion:
- 9.1.2.1 *Grades R60802 and R60804*—All coupons thus tested shall exhibit a continuous, black, lustrous oxide film and be free of white or brown corrosion product in excess of standards previously agreed upon between manufacturer and purchaser. Coupons shall exhibit a weight gain of not more than 22 mg/dm² in a 72-h test or 38 mg/dm² in a 336-h test.
- 9.1.2.2 *Grade R60901*—All coupons shall exhibit a continuous, uniform, dark gray oxide film. Coupons shall exhibit a weight gain of not more than 35 mg/dm<sup>2</sup> in a 72-h test or 60 mg/dm<sup>2</sup> in a 336-h test.
  - 9.2 Ultrasonic Inspection:
- 9.2.1 In lieu of the ultrasonic test of the ingot as specified in Specification B350/B350M, the manufacturer may alternatively perform ultrasonic inspection at an intermediate size in accordance with Practices E114 and E214, with the approval of the purchaser.
- 9.2.2 Unless otherwise specified by the purchaser, the reference standard shall consist of a 0.06 in. [1.5 mm] flat bottom hole drilled perpendicular to the longitudinal product axis to a depth of 0.5 in. [13 mm].
  - 9.2.3 Any defect reflections greater than the indication from the reference standard should be rejected.
- 9.2.4 Additional tests may be specified in the purchase order. The test method and standards shall be agreed upon in advance between manufacturer and purchaser.

#### 10. Significance of Numerical Limits

10.1 For the purpose of determining compliance with the specified limits for requirements of the properties listed in the following table, an observed value or a calculated value shall be rounded as indicated in accordance with the rounding methods of Practice E29.

Property
Chemical composition, and tolerances
(when expressed as decimals)
Tensile strength and yield strength
Elongation

Rounded Unit for Observed or Calculated Value nearest unit in the last right-hand place of figures of the specified limit nearest 1000 psi [10 MPa] nearest 1 %

# 11. Permissible Variations in Dimensions

- 11.1 Table 4, Permissible variations in sectional dimensions for hot-worked bars in rounds and squares.
- 11.2 Table 5, Permissible variations in hot-rolled flat bars or bars sheared from plate.

<sup>&</sup>lt;sup>B</sup> "RT" represents room temperature; Note 4 in Test Methods E8/E8M indicates that RT shall be considered to be 50 to 100°F [10 to 38°C] unless otherwise specified. Paragraph 9.4.4 in Test Methods E21 states that for the duration of the test, the difference between the indicated temperature and the nominal test temperature is not to exceed ±5°F [3°C] for tests at 1800°F [1000°C] and lower, and ±10°F [6°C] for tests at higher temperatures.