



# Standard Specification for Zirconium and Zirconium Alloy Ingots for Nuclear Application<sup>1</sup>

This standard is issued under the fixed designation B350/B350M; 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 ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

## 1. Scope

1.1 This specification covers vacuum-melted zirconium and zirconium alloy ingots for nuclear application.

1.2 The values stated in either inch-pound units or SI units are to be regarded separately as standard. Within the text, the SI units are shown in brackets. The values stated in each system are not exact equivalents; therefore, each system shall be used independently of the other. Combining values from the two systems may result in nonconformance with the specification.

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, health, and environmental practices and determine the applicability of regulatory limitations prior to use.*

1.4 *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:*<sup>2</sup>

[E29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications](#)

[E114 Practice for Ultrasonic Pulse-Echo Straight-Beam Contact Testing](#)

[E2626 Guide for Spectrometric Analysis of Reactive and](#)

<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.

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<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

[Refractory Metals](#) (Withdrawn 2017)<sup>3</sup>

## 3. Terminology

3.1 *Lot Definitions:*

3.1.1 *ingot, n*—a quantity of metal cast into a shape suitable for subsequent processing to various mill products.

## 4. Classification

4.1 Ingots are furnished in five grades as follows:

4.1.1 R60001 Unalloyed Zirconium,

4.1.2 R60802 Zirconium-Tin Alloy,

4.1.3 R60804 Zirconium-Tin Alloy,

4.1.4 R60901 Zirconium-Niobium Alloy, and

4.1.5 R60904 Zirconium-Niobium Alloy.

## 5. Ordering Information

5.1 Orders for material under this specification should include the following information as required to describe adequately the desired material:

5.1.1 Quantity in weight or pieces,

5.1.2 Name of material,

5.1.3 Grade ([Table 1](#)),

5.1.4 Size (diameter, length, or weight), in the unit system regarded as standard (inch-pound or SI), and

5.1.5 ASTM designation and year of issue.

NOTE 1—A typical ordering description is as follows: two each zirconium ingots, Grade R60001, 12 in. diameter by 1000 lb each, ASTM Specification: B350/B350M – 01.

5.2 In addition to the data specified in [5.1](#), the following options and points of agreement between the manufacturer and the purchaser should be specified in the purchase order if required:

5.2.1 Inspection ([Section 12](#)), and

5.2.2 Oxygen analysis requirements ([Table 1](#)).

## 6. Materials and Manufacture

6.1 Materials covered by this specification shall be produced by multiple vacuum arc melting, or electron beam melting, or other melting processes conventionally used for

<sup>3</sup> The last approved version of this historical standard is referenced on [www.astm.org](http://www.astm.org).

**TABLE 1 Chemical Requirements**

Element	Composition, Weight %				
	UNS R60001	UNS R60802	UNS R60804	UNS R60901	UNS R60904
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	2.50–2.80
Oxygen	<sup>A</sup>	<sup>A</sup>	<sup>A</sup>	0.09–0.15	<sup>A</sup>
Iron + chromium + nickel	...	0.18–0.38	...	...	...
Iron + chromium	...	...	0.28–0.37	...	...
Maximum Impurities, Weight %					
Aluminum	0.0075	0.0075	0.0075	0.0075	0.0075
Boron	0.00005	0.00005	0.00005	0.00005	0.00005
Cadmium	0.00005	0.00005	0.00005	0.00005	0.00005
Calcium	...	0.0030	0.0030	...	...
Carbon	0.027	0.027	0.027	0.027	0.027
Chromium	0.020	...	...	0.020	0.020
Cobalt	0.0020	0.0020	0.0020	0.0020	0.0020
Copper	0.0050	0.0050	0.0050	0.0050	0.0050
Hafnium	0.010	0.010	0.010	0.010	0.010
Hydrogen	0.0025	0.0025	0.0025	0.0025	0.0010
Iron	0.150	...	...	0.150	0.150
Magnesium	0.0020	0.0020	0.0020	0.0020	0.0020
Manganese	0.0050	0.0050	0.0050	0.0050	0.0050
Molybdenum	0.0050	0.0050	0.0050	0.0050	0.0050
Nickel	0.0070	...	0.0070	0.0070	0.0070
Niobium	...	0.0100	0.0100	...	...
Nitrogen	0.0080	0.0080	0.0080	0.0080	0.0080
Phosphorus	...	...	...	0.0020	0.0020
Silicon	0.0120	0.0120	0.0120	0.0120	0.012
Tin	0.0050	...	...	0.010	0.010
Tungsten	0.010	0.010	0.010	0.010	0.010
Titanium	0.0050	0.0050	0.0050	0.0050	0.0050
Uranium (total)	0.00035	0.00035	0.00035	0.00035	0.00035

<sup>A</sup> 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.

reactive metals; all melting is to be carried out in furnaces usually used for reactive metals.

## 7. Condition

7.1 Unless otherwise specified, ingots shall be conditioned by machining or grinding or both to remove surface and subsurface defects detrimental to subsequent fabrication.

7.2 After conditioning has been completed, no abrupt changes in diameter or local depression that will impair subsequent fabrication shall be permitted. The difference between the maximum and minimum radii of the conditioned ingot shall not exceed 20 % of the maximum radius. Lands, grooves, and local depressions shall be blended to a maximum angle of 30° to the axis of the ingot. Each end of the ingot shall be chamfered or radiused. The minimum chamfer or radius shall be ½ in. [12 mm].

## 8. Chemical Requirements

8.1 The ingot shall conform to the requirements for chemical composition as prescribed in **Table 1**. Guide **E2626** may be used as a guide for chemical analysis techniques.

NOTE 2—Nuclear grade zirconium ingots produced from electrolytic starting material should be produced using methods such that the final ingot fluorine content is <1ppm to mitigate risk of poor breakaway performance. In order to achieve this target fluorine level, a quad melt process under vacuum is strongly recommended.

8.2 The ingot shall be sampled in sufficient places along the side wall so that the top sample is within 5 in. [125 mm] of the top face and the distance between samples or between the bottom face and a sample does not exceed one ingot diameter. A minimum of three samples per ingot is required.

8.3 These samples shall be analyzed for the alloying and impurity elements given in **Table 1**.

8.4 Analysis shall be made using the manufacturer's standard methods. In the event of disagreement as to the chemical composition of the metal, methods of chemical analysis for reference purposes shall be determined by a mutually acceptable laboratory.

8.5 *Product Check Analysis*—Product check analysis is an analysis made by or for the purchaser for the purpose of verifying the composition of the ingot. The check analysis tolerances reflect the variation between laboratories in the measurement of chemical composition. The permissible variation in the product check analysis from the specified range is as prescribed in **Table 2**.

## 9. Retest

9.1 If any sample or specimen exhibits obvious contamination, improper preparation, or flaws disqualifying it as a representative sample, it shall be discarded and a new sample or specimen substituted.

**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

9.2 If the results of any test are not in conformance with the requirements of this specification, the ingot may be retested at the option of the manufacturer. The manufacturer may scalp or crop the ingot to remove nonconforming material then resample remaining ingot position(s). The ingot shall be acceptable if all results of retests following the initial test(s) conform to this specification.

## 10. Significance of Numerical Limits

10.1 For purposes of determining compliance with the specified limits for requirements of the properties listed in Table 1 and Table 2, an observed value or a calculated value shall be rounded as indicated in accordance with the rounding method of Practice E29.

## 11. Ultrasonic Test

11.1 Ingots shall be inspected ultrasonically by the methods described in 11.1.1 and 11.1.2 unless otherwise agreed upon between the manufacturer and the purchaser.

11.1.1 *Standardization*—The test shall be conducted in accordance with Practice E114. The search unit shall be a longitudinal beam transducer, operated at a maximum frequency of 2¼ MHz using a suitable couplant with the piece being tested. The search unit shall be standardized on a test block, setting the back reflection equal to 100 %. The test block shall be of the same approximate diameter and surface condition as the ingot. Standardization is required before the inspection begins on the ingot.

11.1.2 Unless otherwise approved by the purchaser, the reference standard shall consist of an ingot section containing two 0.093-in. [2.4-mm] diameter holes, one of which shall be radially located at the approximate ingot center, the other at the approximate mid-radius position. These drilled holes shall terminate at a plane (normal to the longitudinal ingot axis) located 0.5 in. [12 mm] below the ingot end face. During calibration, the two reference holes should line up with the beam, with the mid-radius hole farthest from the search unit. Then the complete length of the ingot shall be inspected. Two traverses shall be made along the length of the cylindrical ingot surface 90 degrees apart. Additional traverses shall be made if the entire ingot is not adequately inspected by this procedure. Remove defect indications observed that exceed that of the reference standard. If the back reflection is between 50 and 95 %, increase the gain to bring back reflection up to 100 % and inspect the trace indication to observe any internal reflec-

tions greater than the indication from the reference standard. Again, remove any such defects. Following such amplitude adjustment and inspection, recalibrate the search unit on the test block. Note the position of all lesser defects and report them to the purchaser along with the ingot number.

## 12. Inspection

12.1 The manufacturer shall inspect the material covered by this specification prior to shipment. 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 tests. If the purchaser's representative is not present at the time agreed upon for the testing, the manufacturer shall consider the requirement for purchaser's inspection at place of manufacture to be waived.

12.2 The manufacturer shall afford the inspector representing the purchaser, without charge, all reasonable facilities to satisfy him that the material is being furnished in accordance with this specification. This inspection shall be conducted so as not to interfere unnecessarily with the operation of the works.

## 13. Rejection

13.1 Rejection for failure of the material to meet this specification shall be reported to the manufacturer within 60 calendar days from the receipt of the material by the purchaser. Unless otherwise specified, rejected material may be returned to the manufacturer at the manufacturer's expense, unless the purchaser receives, within three weeks of the notice of rejection, other instructions for disposition.

## 14. Referee

14.1 In the event of disagreement between the manufacturer and the purchaser on 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.

## 15. Certification

15.1 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. A report of the test results shall be furnished.

## 16. Packaging and Marking

16.1 Each ingot shall be metal die stamped on the top with the manufacturer's ingot number. Each box or skid shall be legibly and conspicuously marked or tagged with the following information:

- 16.1.1 Order or contract number,
- 16.1.2 Name of material,
- 16.1.3 Grade,
- 16.1.4 Ingot number,