



Designation: C 1076 – 97

Standard Specification for Nuclear Grade Hafnium Oxide Pellets¹

This standard is issued under the fixed designation C 1076; 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 approval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This specification applies to pellets of stabilized cubic hafnium oxide used in nuclear reactors.

1.2 The values stated in SI units are to be regarded as the standard. The values given in parentheses are for information only.

2. Referenced Documents

2.1 ASTM Standards:

- C 559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles²
- C 859 Terminology Relating to Nuclear Materials³
- C 1098 Specification for Nuclear Grade Hafnium Oxide Powder³
- E 105 Practice for Probability Sampling of Materials⁴

2.2 ANSI Standard:

ANSI/ASME NQA-1 Quality Assurance Program Requirements for Nuclear Facility Applications⁵

2.3 U.S. Government Document:

Code of Federal Regulations, Title 10, Part 50—Energy (10CFR 50) Domestic Licensing of Production and Utilization Facilities⁶

3. Terminology

3.1 *Definitions*—Terms shall be defined in accordance with Terminology C 859 except for the following:

3.1.1 *buyer*—organization issuing the purchase order.

3.1.2 *pellet*—fabricated geometric shape of stabilized cubic hafnium oxide having a chemical composition as described in Section 4.

3.1.3 *pellet lot*—the pellets produced from one hafnium oxide powder lot using one set of process parameters. Pellet lot size shall be agreed upon between the seller and the buyer.

3.1.4 *phase transformation*—rearrangement of the atomic ordering of a crystalline lattice as a material is cycled through a critical transformation or inversion temperature. The change from one crystalline phase to another may be accompanied by a volume change that could lead to cracks or defects in articles fabricated from such materials.^{7,8}

3.1.5 *powder lot*—a specified quantity of hafnium oxide powder with stabilizing additive, blended together such that samples taken in accordance with Section 7 can be considered as representative of the entire quantity.

3.1.6 *seller*—hafnium oxide pellet supplier.

3.1.7 *stabilizing additive*—a material which, when present in sufficient concentration in the subject material exhibiting the phase transformation, produces a stabilized crystalline phase that does not undergo a transformation or inversion at any temperature within the expected fabrication or usage regime of the manufactured pellet. The potentially deleterious volume change is therefore avoided.

4. Chemical Composition

4.1 The starting hafnium oxide powder shall be in accordance with Specification C 1098.

4.2 A stabilizing additive shall be utilized with the hafnium oxide. The recommended stabilizing additive is yttrium oxide (Y_2O_3). The typical concentration range is 7 to 10 weight % Y_2O_3 in the finished product. References to other stabilizing additives (such as calcium oxide (CaO) and magnesium oxide (MgO)) may be found in the literature and these additives may be used if agreed upon by the buyer and seller. It is cautioned, however, that the buyer should be aware of potential destabilization during thermal cycling when MgO or CaO is used.

4.3 Use analytical chemistry methods as agreed upon between the buyer and the seller.

4.4 The hafnium concentration, in grams of hafnium per unit volume or grams of hafnium per unit length, may be specified by the buyer. In specifying the allowable range in hafnium concentration, the buyer should consider the following:

¹ This specification is under the jurisdiction of ASTM Committee C-26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.03 on Neutron Absorber Materials Specifications.

Current edition approved May 10, 1997. Published May 1998. Originally published as C 1076 – 87. Last previous edition C 1076 – 87.

² *Annual Book of ASTM Standards*, Vol 15.01.

³ *Annual Book of ASTM Standards*, Vol 12.01.

⁴ *Annual Book of ASTM Standards*, Vol 14.02.

⁵ Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.

⁶ Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.

⁷ Curtis, C. E., Doney, L. M., and Johnson, J. R., "Some Properties of Hafnium-Oxide, Hafnium-Silicate, Calcium-Hafnate, and Hafnium-Carbide," *Journal of American Ceramics Society*, Vol 37, 1954, pp. 458–465.

⁸ Brown, L. M., and Madiyasni, K. S., "Characterization of Alkoxy-Derived Yttria-Stabilized Hafnia," *Journal of American Ceramics Society*, Vol 53, 1970, pp. 590–594.

- 4.4.1 Variations in chemical composition,
 - 4.4.2 Pellet bulk density,
 - 4.4.3 Type and concentration of stabilizing additive, and
 - 4.4.4 Pellet dimensions and dimensional tolerances.
- 4.5 The impurity concentration, excluding zirconia and the stabilizing additive, shall not exceed 0.5 weight %. Individual element limits are specified in Table 1. The buyer may specify additional limits for any other elements not listed in Table 1.
- 4.6 The zirconium concentration shall not exceed 4.5 weight % on the basis of Zr/(Zr + Hf).
- 4.7 The moisture concentration limit is included in the total hydrogen limit (see Table 1).

5. Physical Requirements

5.1 Physical Dimensions:

5.1.1 Dimensional requirements shall be in accordance with applicable drawings and purchase order documents.

5.1.2 Pellet dimensions shall be measured to ensure compliance with the buyer's requirements. Sampling plans to meet the acceptance criteria shall be agreed upon between the buyer and the seller to ensure that the pellets represented by the sample are within the required tolerance.

5.2 Density:

5.2.1 Pellet density limits shall be specified by the buyer. The incorporation of a stabilizing additive will introduce a change in the theoretical density of the fabricated pellets and should be taken into account. The method of establishing the theoretical density value shall be mutually agreed upon between the buyer and the seller.

5.2.2 The method of density measurement shall be Test Method C 559 or an alternative method submitted by the seller for approval by the buyer. Sampling plans to meet the acceptance criteria shall be agreed upon between the buyer and the seller. The method of density measurement and the method of compliance with 5.2.1 shall be submitted by the seller to the buyer for approval.

5.3 *Mechanical Properties*—Required mechanical properties and test methods shall be mutually agreed upon between the buyer and the seller. It is recommended that the plan of mechanical testing to verify pellet integrity include compressive testing at 69 MPa (10 000 psi).

5.4 *Phase Stabilization*—A crushed sample of the fired product shall be tested by powder X-ray diffraction. The diffraction pattern must show that the required cubic crystalline phase has been developed in the product. The criteria for

satisfactory development of the cubic phase shall be mutually agreed upon between the buyer and the seller.

5.5 *Visual Appearance*—Visual examination shall be conducted on finished pellets in accordance with Section 7 on Sampling. The seller and the buyer shall agree on visual standards as representing the requirements of 5.5.1, 5.5.2, and 5.5.3. These standards shall be used as acceptance standards for the visual examination of the pellets. In the event of a dispute, the method of defect measurement shall be submitted by the seller for approval by the buyer. Maximum permissible defects are defined as follows:

5.5.1 *End Chips*—Pellet-end surface shall not be chipped beyond 10 % of the end-face surface area and no chip shall exceed 1.02 mm (0.040 in.) in depth.

5.5.2 *Circumferential Chips*—Pellet circumferential surfaces shall not be chipped beyond 5 % of the circumferential surface area. No single chip shall exceed a depth of 1.02 mm (0.040 in.).

5.5.3 *Cracks*—Cracks not exceeding 90° of circumference in length are acceptable provided the other requirements of the specifications are met.

5.5.4 Fissures and other defects shall be evaluated with respect to the criteria of 5.5.1, 5.5.2, and 5.5.3.

6. Cleanliness

6.1 The finished pellets shall be handled in a manner to avoid contamination by grinding fluids and dust, cleaning agents, and organic materials such as plastics and paper used in packaging. Cleaning solutions, if used, shall be free of halides or non-volatile additives and shall be removed from the pellets prior to sampling and packaging.

7. Sampling

7.1 Sampling plans to meet the acceptance criteria and inspection and measurement procedures that describe the method of compliance with this specification shall be approved by the buyer. The degree of sampling varies with the application and for this reason should be specified on the purchase order. Practice E 105 is referenced as a guide.

7.2 Each powder and pellet sample taken shall be sufficient to perform the following in the event they are necessary or desired by the buyer:

- 7.2.1 Quality verification tests,
- 7.2.2 Acceptance tests,
- 7.2.3 Referee tests, in the event these become necessary, and
- 7.2.4 Retention of archive sample by the seller.

7.3 Archive samples shall be retained by the seller for a period of time specified by the buyer and delivered to the buyer upon request.

8. Inspection and Certification

8.1 The seller shall inspect the material covered by this specification and shall furnish the buyer with certificates of test showing the results of testing and inspection performed on each pellet lot prior to shipment. The seller shall certify that each pellet lot is in compliance with the provisions of this specification.

TABLE 1 Impurity Concentration Limits

Element	Maximum Concentration Limit (µg/g Pellet)
B	400
C	1 000
Gd	200
Gd + Sm + Eu + Dy	500
Co	100
Si	2 000
Th	400
F	30
F + Cl + Br + I	100
H (total hydrogen from all sources)	2



9. Rejection and Rehearing

9.1 Unless the buyer and the seller agree otherwise, rejection and acceptance shall be on a pellet lot basis.

9.2 Pellet lots that fail to conform to the requirements of this specification may be rejected by the buyer. The seller may petition the buyer to waive selected requirements for identified out-of-specification lots. Decision to grant such waiver belongs to the buyer. The seller may also apply any remedy to bring rejected lots into specification providing the seller can demonstrate to the buyer that such remedy does not impair the function or preclude the certification of the rejected material.

9.3 In the event of disagreement over the results of analysis, samples shall be submitted to a mutually selected referee for resolution.

10. Packaging and Shipping

10.1 The pellets shall be packaged in sealed containers to prevent loss or damage, or both, of material and contamination from airborne or container materials. The exact size and type of packaging shall be as mutually agreed upon between the buyer and seller.

10.2 Each container shall be clearly marked with the following:

- 10.2.1 Hafnium oxide pellets plus stabilizing additive,
- 10.2.2 Purchase order number,
- 10.2.3 Gross, tare, and net weight,
- 10.2.4 Lot number, and
- 10.2.5 Name of pellet manufacturer.

11. Quality Assurance

11.1 Quality assurance requirements shall be agreed upon between the buyer and the seller when specified in the purchase order. Code of Federal Regulations, Title 10, Part 50 (Appendix B) and ANSI/ASME NQA-1 are referenced as guides.

12. Keywords

12.1 CaO; hafnium oxide; MgO; stabilizer; stabilizing; additive; thermal cycling test; Y₂O₃

The American Society for Testing and Materials takes no position respecting the validity of any patent rights asserted in connection with any item mentioned in this standard. Users of this standard are expressly advised that determination of the validity of any such patent rights, and the risk of infringement of such rights, are entirely their own responsibility.

This standard is subject to revision at any time by the responsible technical committee and must be reviewed every five years and if not revised, either reapproved or withdrawn. Your comments are invited either for revision of this standard or for additional standards and should be addressed to ASTM Headquarters. Your comments will receive careful consideration at a meeting of the responsible technical committee, which you may attend. If you feel that your comments have not received a fair hearing you should make your views known to the ASTM Committee on Standards, at the address shown below.

This standard is copyrighted by ASTM, 100 Barr Harbor Drive, PO Box C700, West Conshohocken, PA 19428-2959, United States. Individual reprints (single or multiple copies) of this standard may be obtained by contacting ASTM at the above address or at 610-832-9585 (phone), 610-832-9555 (fax), or service@astm.org (e-mail); or through the ASTM website (www.astm.org).