INTERNATIONAL

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Standard Specification for Nuclear Grade Hafnium Oxide Pellets[1]

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 reapproval. 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:*[2]

C 559 Test Method for Bulk Density by Physical Measurements of Manufactured Carbon and Graphite Articles

C 859 Terminology Relating to Nuclear Materials

C 1098 Specification for Nuclear-Grade Hafnium Oxide Powder

E 105 Practice for Probability Sampling ~~of~~Of Materials

2.2 *ANSI Standard:*[3]

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

2.3 *U.S. Government Document:*[4]

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

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.[5, 6]

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.

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

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[2] For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Sevice at service@astm.org. For *Annual Book of ASTM Standards*volume information, refer to the standard's Document Summary page on the ASTM website.

~~[3] Available from American National Standards Institute, 11 W. 42nd St., 13th Floor, New York, NY 10036.~~

[3] Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.org.

~~[4] Available from Superintendent of Documents, U.S. Government Printing Office, Washington, DC 20402.~~

[4] Available from U.S. Government Printing Office Superintendent of Documents, 732 N. Capitol St., NW, Mail Stop: SDE, Washington, DC 20401, http://www.access.gpo.gov.

[5] 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.

[6] 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. 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:

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.~~ shall be approved by the buyer prior to use.

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~~compression 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 phace 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~~ on Sampling. The method of defect measurement shall be approved by the buyer prior to use 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.

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