Designation: C1868 – 18

Standard Practice for Ceramographic Preparation of UO_2 and Mixed Oxide $(U,Pu)O_2$ Pellets for Microstructural Analysis[1]

This standard is issued under the fixed designation C1868; 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 practice describes the procedure for preparing nuclear-grade uranium dioxide (UO_2) or mixed uranium-plutonium dioxide (MOX or $(U,Pu)O_2$)), sintered and non-irradiated pellets for subsequent microstructural analysis (hereafter referred to as ceramographic examination).

1.2 The ceramographic examination is performed to confirm that the microstructure of the sintered pellet is in compliance with the fuel specification, for example as defined in Specifications C776 and C833, as a function of the initial raw material properties and manufacturing process parameters.

1.3 The microstructure of a ceramic pellet includes: grain size, porosity size and distribution, and phase distribution for $(U,Pu)O_2$ pellets, that is, Pu-rich cluster size and distribution.[2]

1.4 The microstructural characteristics of the pellet are accessible after preparation which involves: sawing, mounting in a resin, surface polishing, and chemical etching, thermal etching, or both.

1.5 This practice describes the preparation processes mentioned in 1.4; it does not discuss the associated sampling practices (for example, Practice E105) or ceramographic examination methods (for example, the methods for determining average grain size are covered in Test Method E112).

1.6 Due to the radiotoxicity associated with these nuclear materials, all operations described in this practice should be performed in glovebox for $(U,Pu)O_2$ pellets and in a hood for UO_2 pellets.

1.7 The values stated in SI units are to be regarded as standard. No other units of measurement are included in this standard.

1.8 *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.*

1.9 *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:*[3]

C776 Specification for Sintered Uranium Dioxide Pellets for Light Water Reactors

C833 Specification for Sintered (Uranium-Plutonium) Dioxide Pellets for Light Water Reactors

C859 Terminology Relating to Nuclear Materials

D1193 Specification for Reagent Water

E105 Practice for Probability Sampling of Materials

E112 Test Methods for Determining Average Grain Size

3. Terminology

3.1 Except as otherwise defined herein, definitions of terms are as given in Terminology C859.

3.2 *Definitions of Terms Specific to This Standard:*

3.2.1 *grain*—single crystal; region of space occupied by a continuous crystal lattice.

3.2.2 *microstructure*—structure of a material as observed from a magnified view in the range from 0.1 to 100 μm involving properties such as grains, grain boundaries, pores, micro-cracks, and phases distribution of the sintered pellet.

3.2.3 *MOX*—mixed oxide, that is, a blend of uranium and plutonium dioxides.

3.2.4 *porosity*—amount of pores (voids) in an object expressed as percentage of the total volume.

3.2.5 *sintered pellet*—densified ceramic compact after heat treatment at elevated temperatures but below the melting point of the main component.

[1] This practice is under the jurisdiction of ASTM Committee C26 on Nuclear Fuel Cycle and is the direct responsibility of Subcommittee C26.05 on Methods of Test.

Current edition approved Jan. 1, 2018. Published January 2018. DOI: 10.1520/C1868-18.

[2] $(U,Pu)O_2$ fuel pellets are characterized by fissile Pu-rich zones dispersed in a fertile depleted UO_2 matrix.

[3] 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.

4. Summary of Practice

4.1 *Sample Sawing:*

4.1.1 This operation (also called sectioning) is typically performed by means of cutting machines equipped with diamond cut-off wheels.

4.1.2 The pellet is clamped in a specimen holder which is placed at the tip of an arm fitted with a counterweight which facilitates adjustment of the penetration force.

4.1.3 The sawing can be done longitudinally or radially. A longitudinal cut is preferable for most applications because it permits observation of the pellet structure both along the full axial length and across the full diameter. Both radial cut and longitudinal cross-sections together can give information about any preferential cracking or agglomerate deformation resulting from the pelletizing process, the sintering process, or both.

4.1.4 Water, emulsions, aqueous solutions, or low-viscosity mineral oils can be used as a coolant for the cut-off wheels and as a lubricant to minimize any potential mechanical damage of the sample.

4.2 *Sample Mounting:*

4.2.1 This operation (also called embedding) is typically performed by immersing the sectioned pellet in a resin which is subsequently hardened by polymerization so as to obtain a block that is easier to handle during the polishing process.

4.2.2 While this operation can be performed in a hot mounting machine under hot compression (150 to 190°C), it is preferable to perform manually in a glovebox (cold mounting) since the associated mounting media include epoxies, polyester resins (thermosets), or acrylates (thermoplastics) which all cure exothermically when mixed with hardeners and catalysts.

4.2.3 In the case of highly porous pellets (with large closed pores or significant open porosity), the exposed pores create potential initiation points for surface damage during polishing caused by pull-outs. Because of this, it is preferable to perform sample mounting in a vacuum mounting chamber by sample impregnation with high fluidity resin.[4]

4.3 *Sample Polishing:*

4.3.1 Rough and fine polishing are the mechanical abrasion processes performed on the sample in order to obtain a flat, scratch-free surface with minimum mechanical damage (with the end goal of obtaining a mirror finish).

4.3.2 The rough polishing process consists of flattening the sample surface and removing the saw marks by the application of pressure with a coarse-grit medium (for example, large abrasive particles) bonded to adhesive paper or a metal disc.

4.3.3 The fine polishing process consists of removing the scratches created by the rough polishing process by the application of pressure with a free, fine-grit medium (for example, a loose collection of small particles such as diamond or alumina) incorporated into a paste or suspension spread onto a cloth.

4.3.4 Each of the polishing processes uses a fluid which acts both as a lubricant and as a cooling agent.

4.3.5 Polishing step involves successive stages using increasingly fine grit medium (decreasing in particle size). Each stage reduces the scratch sizes generated during the previous stage.

4.3.6 The fine polished surface should be free of scratches when viewed by microscope (mirror finish).

4.4 *Sample Surface Etching:*

4.4.1 Etching is the process to reveal and delineate grain boundaries and other microstructural features that are not visible on the ground and polished surface of the sample.

4.4.2 The polished surface is typically etched by either chemical or thermal methods for the purpose of performing ceramographic examination as indicated in 1.3.

4.4.3 Chemical etching consists of submerging the sample in an acid solution (etchant) that will preferentially attack or create color-specific phases with different chemical potentials. The advantages of this method are that it is simple and fast and can reveal phase distribution; the disadvantage is that it involves management of contaminated acid wastes.

4.4.4 Thermal treatment consists of heating the polished sample in a furnace to reveal the surface features by promoting the diffusional, material transport mechanisms (such as surface diffusion, volume diffusion, and evaporation-condensation). The advantage of this method is that it provides high resolution; the disadvantages are that it is time consuming, requires a furnace rated for nuclear material (for example, in a glovebox), and cannot reveal phase distribution.

4.5 *Sample Cleaning:*

4.5.1 The sample should be cleaned (for example, in an ultrasonic cleaner) after sawing step and each polishing stage.

5. Significance and Use

5.1 The ceramographic examination of the nuclear fuel pellet is mandatory to ensure that the microstructural characteristics are in compliance with the fuel specifications relative to performance in reactor, particularly concerning thermo-mechanical behavior and fission gas release.

5.2 This practice is applicable for sintered UO_2 pellets with any ^{235}U concentration and $(U,Pu)O_2$ pellets containing up to 15 weight % PuO_2 with less than 10 % porosity.

6. Apparatus and Materials

6.1 *Cutting Machine,* for sawing the sample; power-driven diamond wheel with adjustable speed and force; protective transparent hood; cooling system for the cutting device and sample; adjustable specimen holder for radial or longitudinal pellet sectioning (and possibly embedded pellet).

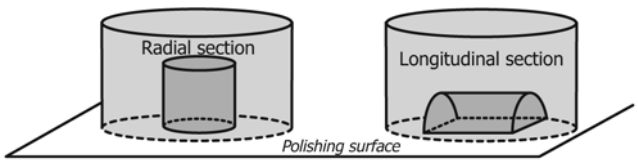

FIG. 1 Mounted Sample (Sectioned Pellet) for Ceramographic Examination

[4] When the sample is very brittle (that is, it cracks easily) or highly porous, it is recommended that it be mounted by vacuum impregnation before the sawing step.