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Nuclear fuel technology — Guidelines for ceramographic preparation of UO_2 sintered pellets for microstructure examination

Technologie du combustible nucléaire — Lignes directrices pour la préparation céramographique de pastilles UO_2 frittées pour l'examen de la microstructure

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Foreword

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The procedures used to develop this document and those intended for its further maintenance are described in the ISO/IEC Directives, Part 1. In particular the different approval criteria needed for the different types of ISO documents should be noted. This document was drafted in accordance with the editorial rules of the ISO/IEC Directives, Part 2 (see www.iso.org/directives).

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This document was prepared by Technical Committee ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 5, *Nuclear installations, processes and technologies*.

This second edition cancels and replaces the first edition (ISO 16793:2005), which has been technically revised.

Nuclear fuel technology — Guidelines for ceramographic preparation of UO_2 sintered pellets for microstructure examination

1 Scope

This document describes the ceramographic preparation of uranium dioxide (UO_2) sintered pellets for qualitative and quantitative microstructure examinations.

These examinations can be carried out before and after thermal or chemical etching.

They enable

— observations of fissures, inter- or intra-granular pores and inclusions, and

— measurement of pore and grain size and measurement of pore and grain size distributions.

The measurement of average grain size can be carried out using a classical counting method as described in ISO 2624 or ASTM E112[3], i.e. intercept procedure, comparison with standard grids or reference photographs.

The measurement of pore-size distributions is usually carried out by an automatic image analyser. If the grain-size distributions are also measured with an image analyser, it is recommended that thermal etching be used to reveal the grain structure uniformly throughout the whole sample.

2 Normative references

There are no normative references in this document.

3 Terms and definitions

No terms and definitions are listed in this document.

ISO and IEC maintain terminological databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at http://www.electropedia.org/

4 Principle

Ceramographic preparation of UO_2 sintered pellets is carried out in two main stages.

a) Sample polishing: the sample can either be mounted in resin, or held in an appropriate mechanical gripping device (an example is shown in Figure 1).

b) Sample etching: the microstructure can be revealed by chemical or thermal etching. Thermal etching cannot be done on a mounted sample.

5 Procedures

5.1 General

Ceramographic samples can be prepared using three different procedures: the sample can be unmounted (5.2), mounted without subsequent dismounting (5.3), or mounted with subsequent dismounting (5.4).

5.2 Sample preparation without mounting

Prepare samples using the following stages:

— Sectioning (8.1);

— Grinding (8.3);

— Polishing (8.4);

— Chemical (9.2) or thermal (9.3) etching.

5.3 Sample preparation with mounting, not needing subsequent dismounting

Prepare samples using the following stages:

— Sectioning (8.1);

— Mounting with any suitable resin (8.2);

— Grinding (8.3);

— Polishing (8.4);

— Chemical etching (9.2).

5.4 Sample preparation with mounting, needing subsequent dismounting

Prepare samples using the following stages:

— Sectioning (8.1);

— Mounting with polyester or acrylic resin (8.2);

— Grinding (8.3);

— Polishing (8.4);

— Dismounting (8.5);

— Chemical (9.2) or thermal (9.3) etching.

6 Apparatus

6.1 Metallographic sectioning machine, with a diamond wheel and a water-inlet system.

6.2 Manual polishing machine, or automatic polishing machine with a force system capable of maintaining a constant pressure on the samples, recommended between $0{,}8 \times 10^5$ Pa and $1{,}0 \times 10^5$ Pa (see Figure 2).

6.3 Ultrasonic cleaning bath.

6.4 Vaccum chamber, with a roughing pump for impregnation.

6.5 Heater, capable of heating up to 80 °C for dismounting.

6.6 Thermal etching furnace, working under an oxidizing atmosphere of carbon dioxide (CO_2). Both the sample and the temperature-measurement system shall be in the isothermal zone of the furnace.

6.7 Mechanical gripping device, for sample preparation without mounting.

6.8 Optical microscope.

7 Reagents and consumables

7.1 Castable mounting compounds, (two or three components) with a polyester-, epoxy- or acrylic-based composition.

7.2 Abrasive papers, with grits between P60 (269 µm) and P2500 (8,4 µm).

7.3 Impregnation resin, like liquid epoxy, polyester or acrylic resin mixed with hardener.

7.4 Alcohol, industrial grade.

7.5 Diamond polishing suspensions, or pastes with diamond size between 0,25 µm and 15 µm.

7.6 Nylon, silk, wool or synthetic polishing cloths, woven or non-woven.

7.7 Aqueous polishing suspensions of alumina or silica, with particle sizes between 0,05 µm and 1 µm.

7.8 Hydrogen peroxide, 30 % by mass, density 1,11 kg/l.

WARNING — Hydrogen peroxide is corrosive and oxidizing. Avoid exposure by contact with the skin or eyes. Use suitable personal protective equipment (including suitable gloves, face shield or safety spectacles, etc.) when working with hydrogen peroxide.

7.9 Sulfuric acid, 95 % by mass or more, density 1,84 kg/l.

WARNING — Concentrated sulfuric acid is corrosive and causes burns. Avoid exposure by contact with the skin or eyes. Use suitable ventilation (e.g., fume hood) and personal protective equipment (e.g. suitable gloves, face shield or safety spectacles) when working with concentrated sulfuric acid.

7.10 Hydrofluoric acid, 38 % to 40 % by mass, density 1,13 kg/l.

WARNING — Hydrofluoric acid is a highly corrosive acid that can severely burn skin, eyes, and mucous membranes. Hydrofluoric acid differs from other acids because the fluoride ion readily penetrates the skin, causing destruction of deep tissue layers. Unlike other acids that are rapidly neutralized, hydrofluoric acid reactions with tissue may continue for days if left untreated. Familiarization and compliance with the Safety Data Sheet is essential.

7.11 Chromium (VI) oxide.

WARNING — Chromium (VI) dioxide is carcinogenic and a strong oxidizer. Use suitable ventilation (e.g., fume hood) and personal protective equipment (e.g. suitable gloves, face shield or safety spectacles). Familiarization and compliance with the Safety Data Sheet is essential.

7.12 Carbon dioxide (CO_2) gas, purity ≥99,995 %, or other oxidizing gas.

7.13 Demineralized water, according to ISO 3696.

8 Methods

8.1 Sectioning

Pellet sectioning is carried out using a metallographic sectioning machine (6.1) with a diamond wheel and a water-inlet system. The rotation speed of the wheel, the feed speed of the sample and the amount of water shall be selected carefully to avoid creating artefacts.

Pellets are usually sectioned such that the finally ground and polished surface is on the diameter of the pellet. Pellets may also be sectioned transversally.

The sectioned sample is cleaned with water (7.13) or alcohol (7.4), with or without an ultrasonic bath (6.3).

For the grinding and polishing stages, the sectioned sample is either mounted in an appropriate mounting compound (7.1) or placed in a mechanical gripping device (6.7) to hold the sample onto the polishing machine (an example of such a device is shown in Figures 1 and 2).

8.2 Mounting

The sectioned sample may be mounted in a mounting compound (7.1).

For samples which do not need to be dismounted, a liquid epoxy, polyester or acrylic resin mixed with hardener (7.3) can be used according to the supplier's instructions.

For samples requiring subsequent dismounting, a liquid polyester or acrylic resin mixed with hardener is used according to the supplier's instructions. These types of resin shrink more than other types during the hardening process, thus facilitating dismounting.

The polymerization is usually complete within 20 min to 24 h at ambient temperature, depending on the type of resin used.

8.3 Grinding

The mounted or mechanically held unmounted samples are ground on a polishing machine (6.2). The grinding is carried out in different stages as shown in the following example:

— abrasive paper (7.2) P180 (82 µm): about 2 min to 10 min of grinding is necessary for the sectioned resin-mounted samples, 30 s to 1 min is sufficient for unmounted samples;

— abrasive paper (7.2) P400 (35 µm): 30 s to 1 min;

— abrasive paper (7.2) P800 (22 µm): 30 s to 1 min;

— abrasive paper (7.2) P1200 (15 µm): 30 s to 1 min.

The samples are thoroughly cleaned with water (7.13) or alcohol (7.4), with or without an ultrasonic bath (6.3) after each stage.

8.4 Polishing

The polishing can include different stages, decreasing the roughness of the polishing cloth and the particle size of the abrasive.

The ground sample is polished on a short-fibre polishing cloth (7.6) with diamond polishing suspensions (7.5) of particle sizes between 15 µm and 0,25 µm, for about 20 min.

The polishing can be finished using aqueous suspensions of alumina or silica (7.7) with particle sizes between 1 µm and 0,05 µm on a fine and long-fibre cloth (7.6).

An automatic polishing machine (6.2) is usually used.

After polishing, the sample is thoroughly cleaned with alcohol (7.4) or water (7.13), with or without an ultrasonic bath (6.3).

The polished surface shall be free from scratches.

The polishing quality is controlled by observation with a microscope (6.8), at a magnification adequate for the subsequent examinations.

Just before polishing, the ground surface can be impregnated under vacuum (6.4) with a liquid resin (7.3) in order to fill up the pores.

8.5 Dismounting

Polyester or acrylic resins display significant shrinkage on hardening, making rapid dismounting of the sample at low temperature possible using a simple heater. For safety reasons, the temperature shall be kept below the decomposition temperature of the resin according to the supplier's data sheets (normally below 80 °C).

9 Etching

9.1 Introduction

The grain structure of the sample surface is highlighted by chemical or thermal etching.

9.2 Chemical etching

The most common reagents of the chemical etching solutions are water, hydrogen peroxide (7.8), sulfuric acid (7.9), hydrofluoric acid (7.10) and/or chromium (VI) oxide (7.11). The proportion of each reagent and the etching time depend on the experience of the laboratory and the material. As an example, the following etching solution can be used:

Water (7.13)	20 ml
Hydrogen peroxide (7.8)	2 ml
Sulfuric acid (7.9)	1 ml

The proportions can change slightly according to the material.

The sample is etched for 3 min to 6 min, and then rinsed with water and air dried. Vacuum drying may also be applied if samples must be kept clean for a longer period of time.

9.3 Thermal etching

Thermal etching is used for samples which are either unmounted or mounted in polyester or acrylic resin for subsequent dismounting.