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

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

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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

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

1 Scope

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

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

They enable

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

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

The measurement of average grain size may be carried out using a classical counting method as described in ISO 2624 or ASTM E112, 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 Principle

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

a) Sample polishing: The sample may 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 may be revealed by chemical or thermal etching. Thermal etching cannot be done on a mounted sample.

3 Procedures

3.1 General

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

3.2 Sample preparation without mounting

Preparation stages:

— sectioning;

— grinding;

— polishing;

— etching (chemical or thermal).

3.3 Sample preparation with mounting, not needing subsequent dismounting

Preparation stages:

— sectioning;

— mounting with any suitable resin;

— grinding;

— polishing;

— chemical etching.

3.4 Sample preparation with mounting, needing subsequent dismounting

Preparation stages:

— sectioning;

— mounting with polyester or acrylic resin;

— grinding;

— polishing;

— dismounting;

— chemical or thermal etching.

4 Essential apparatus

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

4.2 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).

4.3 Ultrasonic cleaning bath.

4.4 Device maintained under vacuum, with a roughing pump for impregnation.

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

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

4.7 Mechanical gripping device, for sample preparation without mounting.

4.8 Optical microscope.

5 Reagents and consumables

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

5.2 Abrasive discs, with grits between P60 (269 µm) and P2500 (8,4 µm).

5.3 Impregnation resin, with epoxy-based composition.

5.4 Alcohol, industrial grade.

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

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

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

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

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

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

5.11 Chromium (VI) oxide.

5.12 Carbon dioxide (CO_2) gas, purity ⩾ 99,995 %, or other oxidizing gas.

5.13 Water.

6 Methods

6.1 Sectioning

Pellet sectioning is carried out using a metallographic sectioning machine (4.1) 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 (5.13) or alcohol (5.4), with or without an ultrasonic bath (4.3).

For the grinding and polishing stages, the sectioned sample is either mounted in an appropriate resin (5.1) or placed in a mechanical gripping device (4.7) to hold the sample onto the polishing machine (an example of such a device is shown in Figures 1 and 2).

6.2 Mounting

The sectioned sample may be mounted in a resin (5.1).

For samples which do not need to be dismounted, a liquid epoxy, polyester or acrylic resin mixed with hardener 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.

6.3 Grinding

The mounted or mechanically held unmounted samples are ground on a polishing machine (4.2). The grinding is carried out in different stages as shown in the following example:

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

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

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

The samples are thoroughly cleaned with water (5.13) or alcohol (5.4), with or without an ultrasonic bath (4.3) after each stage.

6.4 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 (5.6) with diamond abrasives (5.5) 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 (5.7) with particle sizes between 1 µm and 0,05 µm on a fine and long-fibre cloth (5.6).

An automatic polishing machine (4.2) is usually used.

After polishing, the sample is thoroughly cleaned with alcohol (5.4) or water (5.13), with or without an ultrasonic bath (4.3).

The polished surface shall be free from scratches.

The polishing quality is controlled by observation with a microscope (4.8), at a magnification adequate for the subsequent examinations.

Just before polishing, the ground surface may be impregnated under vacuum with a liquid resin in order to fill up the pores.

6.5 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 must be kept below the decomposition temperature of the resin according to the supplier's data sheets (normally below 80 °C).

7 Etching

7.1 Introduction

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

7.2 Chemical etching

The most common reagents of the chemical etching solutions are water, hydrogen peroxide (5.8), sulfuric acid (5.9), hydrofluoric acid (5.10) and/or chromium (VI) oxide (5.11). 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 (5.13) 20 ml

— Hydrogen peroxide (5.8) 2 ml

— Sulfuric acid (5.9) 1 ml

The proportions can change slightly according to the material.

The sample is etched for 3 min to 6 min, 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.

7.3 Thermal etching

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

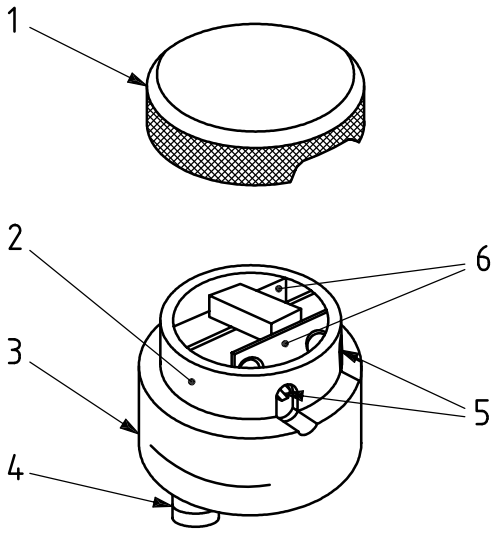
The conditions for thermal etching are as follows:

— gas: CO_2 or other oxidizing gas;

— heating rate: at least 100 °C/h;

— final temperature: between 1 300 °C and 1 450 °C, depending on the material;

— time at final temperature: 15 min or more, depending on the material.



Key

1	cover	4	adjustment screw
2	sliding part	5	tightening screw
3	fixed part	6	holding and positioning apparatus for the sample

Figure 1 — Example of mechanical gripping device of a pellet