International
Standard

ISO 22765

Nuclear fuel technology — Sintered $(U,Pu)O_2$ pellets — Guidance for ceramographic preparation for microstructure examination

Technologie du combustible nucléaire — Pastilles (U,Pu) O_2 frittées — Recommandations relatives à la préparation céramographique pour examen de la microstructure

Second edition
2025-01

Reference number
ISO 22765:2025(en)

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Published in Switzerland

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Foreword

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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,* in collaboration with the European Committee for Standardization (CEN) Technical Committee CEN/TC 430, *Nuclear energy, nuclear technologies, and radiological protection,* in accordance with the Agreement on technical cooperation between ISO and CEN (Vienna Agreement).

This second edition cancels and replaces the first edition (ISO 22765:2016), which has been technically revised.

The main changes are as follows:

- in Clause 1, additional comments about equipment and preparation;
- in Clause 4, addition of required steps according to analyses to be carried out;
- in Clauses 7 and 8, update of the parameters used for polishing.

A list of all parts in the ISO 22765 series can be found on the ISO website.

Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

Nuclear fuel technology — Sintered (U,Pu)O_2 pellets — Guidance for ceramographic preparation for microstructure examination

1 Scope

This document is applied to fuel fabrication. It describes the ceramographic procedure used to prepare sintered (U,Pu)O_2 pellets for qualitative and quantitative examination of the (U,Pu)O_2 pellet microstructure.

The examinations are performed

a) before any treatment or any etching, and

b) after thermal treatment or after chemical or ion etching.

They allow

- observation of any cracks, intra- and intergranular pores or inclusions, and
- measurement of the grain size, porosity and plutonium homogeneity distribution.

The mean grain diameter is measured by one of the classic methods: counting (intercept method), comparison with standard grids or typical images, etc.[2]. The measurement of individual grain sizes requires uniform development of the microstructure over the entire specimen.

The plutonium cluster and pore distribution and localization are generally analysed by automatic image analysis systems. The plutonium distribution is usually revealed by chemical etching or by alpha autoradiography. A scanning electron microscope (SEM) or a microprobe can also be used. In this case an additional preparation can be needed depending on the equipment used. This preparation is not in the scope of this standard.

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 terminology databases for use in standardization at the following addresses:

- ISO Online browsing platform: available at https://www.iso.org/obp
- IEC Electropedia: available at https://www.electropedia.org/

4 Principle

The ceramographic preparation of (U,Pu)O_2 pellets involves two steps:

- polishing, after embedding or not the specimen;
- thermal treatment or chemical etching or ion etching to reveal the specimen microstructure.

The operating procedure comprises the following operations:

- Step 1 – cutting the specimen;
- Step 2 – resin embedding;
- Step 3 – rough polishing;
- Step 4 – fine polishing;
- Step 5 – a treatment or etching to develop grain boundaries.

The decision to cut the specimen (step 1) mainly depends on the analysis to be performed and on laboratory products and scrap management. If necessary, cutting the specimen could be replaced by rough polishing.

The resin embedding operation (step 2) can precede the specimen cutting operation which will be done in this case with a wire saw. Resin embedding may be skipped when a mechanical device is used to hold the specimen during polishing.

Rough polishing (step 3) or fine polishing (step 4) can be sufficient for indirect analyses such as alpha autoradiography with film or scintillator.

Mirror finish requires fine polishing (step 4). This surface aspect is required for direct analysis on the specimen with optical microscopes or scanning electron microscope (grain, crack, and porosity observation, ...).

If a treatment or etching has already been performed on the specimen, an additional fine polishing (step 4) is required to obtain a mirror finish.

A treatment or etching (step 5) is required for grain size analysis, after a fine polishing resulting in a mirror finish.

5 Apparatus

The equipment should be installed in an environment capable of monitoring specimen containment throughout the operating sequence.

5.1 Diamond-impregnated metallography disk cutting machine, with a cooling system or wire saw.

5.2 Automatic polishing machine, preferably with a system for exerting constant pressure on the test specimen**.**

5.3 Resin preparation equipment, e.g. spoons, spatulas, PVC containers, mould.

5.4 Labware for **etching.**

5.5 Ultrasonic specimen cleaning tank (optional)**.**

5.6 Optical microscope or binocular, capable of at least ×10.

5.7 Programmable furnace, able to reach a temperature of about 1 600 °C under argon gas sweeping.

5.8 Engraving pen.

6 Reagents and resin

6.1 Resin to be determined by the user, for example:

- liquid epoxy resin mixed with a suitable proportion of activator from the same manufacturer, curing generally occurs within 24 h at room temperature;
- polyester resin; curing occurs in 1 h.

6.2 Acid aqueous solution for the chemical etching, e.g. chrome oxide and hydrofluoric acid.

6.3 Demineralised water, according to ISO 3696.

6.4 Ethanol.

7 Operating procedure

7.1 Specimen cutting

The specimen is cut using a diamond-impregnated metallography disk-cutting machine with a cooling system, parallel to the selected axis (longitudinal or axial). For an on-axis observation of the specimen, an off-axis cutting is necessary to take into account the thickness of the material removed during polishing.

After cutting, the specimen is cleaned in ethanol or demineralised water. An ultrasonic cleaning tank may be used.

7.2 Resin embedding

Prior to rough and fine polishing, the cut specimen is embedded in resin. Therefore, the specimen fragment to be analysed is put inside a mould with the cut face against the mould bottom.

The prepared resin, with potentially a colouring in order to distinguish the specimen Pu content, is then poured inside the mould.

After the required time (e.g. around 30 min to 60 min), the embedding specimen can be removed from the mould.

Then, the identification number of the specimen sample can be engraved with the engraver pen on the resin surface.

The mould should be cleaned in order to reuse it.

7.3 Rough polishing

The resin-embedded specimens should be, at first, rough-polished to eliminate any resin traces on the surface and ensure flatness. This may be done using a multi-stage process.

An example of polishing is described below, with:

- a P180 (82 µm) abrasive disk (for specimens embedded in resin after cutting): time as necessary to reach the surface flush with the resin;
- a P400 (35 µm) abrasive disk: 30 s to 60 s;
- a P800 (22 µm) abrasive disk: 30 s to 60 s;
- a P1200 (15 µm) abrasive disk: 30 s to 60 s;

Rough polishing is performed on a polishing machine with an individual air pressure-above 100 kPa, and at a rotation speed of 70 min^{-1} to 270 min^{-1}.

The specimen is thoroughly cleaned in ethanol or demineralised water between each step and after completion of the rough polishing operation; rough cleaning could lead to significant scratches at the next steps. The cleaning may be done in an ultrasonic tank.

At the end of this step, it is recommended to check the specimen flatness. During the process frequent measures of the embedded specimen may be used to control flatness.

It is also recommended to ensure the absence of facets, scratches, cracks and chips.

7.4 Fine polishing

An example is described below.

The polishing operation is performed using woven satin disks after injecting a diamond paste with a grain size of 1 µm to 6 µm. Polishing is carried out for about 20 min on a polishing machine with an individual air pressure above 100 kPa.

The specimen is thoroughly cleaned in ethanol or demineralised water and dried in air after polishing. The cleaning may be done in an ultrasonic tank. The polished surface should be mirror finished and free of scratches, cracks or chips from the preparation that may alter the analysis.

The polishing quality is checked by observing the specimen under a microscope or a binocular.

The specimens prepared in this way are suitable for examinations such as

- porosity measurements, and
- overall ceramographic examinations to reveal cracking.

8 Structure development

8.1 General

To determine the grain size, the structure should first be revealed by thermal treatment or chemical etching or ion etching. Chemical etching can also be used to reveal the distribution of plutonium clusters as an alternative to alpha-autoradiography.

8.2 Development by thermal treatment

After cutting, the sample is not embedded, but placed into a mechanical device. Then, the half specimen placed in the mechanical device is polished. The polishing operation prior to the thermal treatment may be done using a multi-stage process.

An example is described below:

a) **Rough polishing**

Rough polishing is performed on a polishing machine with an air pressure above 100 kPa and at a rotation speed of 70 min^{-1} to 270 min^{-1}, with:

- a P180 (82 µm) abrasive disk (for specimens embedded in resin after cutting): time as necessary to reach the surface flush with the resin.
- a P400 (35 µm) abrasive disk: 30 s to 60 s.
- a P800 (22 µm) abrasive disk: 30 s to 60 s.
- a P1200 (15 µm) abrasive disk: 30 s to 60 s.

The specimen is thoroughly cleaned (ultrasonically or not) in ethanol or demineralised water between each step and after completion of the rough polishing operation; rough cleaning could lead to significant scratches at the next steps.

b) **Fine polishing**

See 7.4.

c) **Furnace sequence**

The sample is introduced into the furnace after dismounting from the mechanical device.

Thermal treatment can be carried out under various conditions depending on product manufacturing conditions. An example is described below:

- heating rate: regular, between 5,5 h and 6 h;
- fixed temperature level: around 1 600 °C (around 100 °C under the sintering temperature);
- duration: around 6 h;
- scavenging with argon throughout the cycle.

The conditions are chosen to suit the furnace characteristics, the presence of other gases, gas flow and the duration of the cycle.

8.3 Development by chemical etching

The specimens are immersed for 30 s to 60 s in a reactive mixture, then rinsed in water and dried in air.

Chemical etching involves a large number of variables. The operator determines the nature of the reagent and the duration of the etching operation according to the specimen composition (Pu content, etc.) and the results obtained.

Examples

- room-temperature etching;
  - distilled water, H_2O: 96 ml;
  - chromium oxide, solid, Cr_2O_3: 20 g;
  - hydrofluoric acid, concentrated, HF, mass fraction of 400 g/kg: 48 ml;
  - etching time: 25 s to 60 s;
- hot etching: 70 °C;
  - distilled water, H_2O: 24 ml;
  - ammonium bifluoride solution, saturated, $NH_4{\cdot}HF_2$: 1 ml;
  - sulfuric acid, concentrated, H_2SO_4, mass fraction of 960 g/kg: 1 ml;
  - etching time: 1 min to 5 min.

8.4 Development by ion etching

The specimen is set in a chamber of the ion etching device. An example of the device is shown in Figure 1.

After evacuating air by rotary and diffusion pumps, argon gas is introduced into the chamber.

Argon ions are sputtered onto the surface of the specimen by ion gun for about 10 min. The operator determines the duration of the etching operation according to the specimen composition (Pu content, etc.) and according to the results obtained.