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

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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.