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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 — Préconisations relatives à la préparation céramographique pour examen de la microstructure

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Foreword

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The committee responsible for this document is ISO/TC 85, *Nuclear energy, nuclear technologies, and radiological protection*, Subcommittee SC 5, *Nuclear installations, processes and technologies*.

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

1 Scope

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

The examinations are performed before and after thermal treatment or chemical 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 but alpha-autoradiography can also be used. The first technique avoids the tendency for autoradiography to exaggerate the size of plutonium-rich clusters due to the distance the alpha particles travel away from the source.

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:

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

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

4 Principle

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

— specimen polishing, after embedding or not the specimen in a specific resin;

— thermal treatment or chemical etching to reveal the specimen microstructure.

5 Description

The procedure comprises the following operations:

— cutting the specimen;

— resin embedding;

— rough polishing;

— final polishing;

— thermal treatment or chemical etching.

The resin embedding operation may precede the specimen cutting operation. Resin embedding may be skipped when a mechanical device is used to hold the specimen during cutting and polishing. Thus, it is preferable to cut the pellet before embedding it.

6 Equipment

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

6.1 Diamond-impregnated metallography disk cutting machine with a cooling system.

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

6.3 Resin preparation equipment (spoons, spatulas, PVC containers, mould).

6.4 Labware for etching.

6.5 Ultrasonic specimen cleaning tank (optional).

6.6 Optical microscope or binocular capable of at least ×10.

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

6.8 Engraving pen.

7 Reagents and resin

7.1 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.

7.2 Acid aqueous solution for the chemical etching (e.g. chrome oxide and hydrofluoric acid).

7.3 Demineralised water, complying with ISO 3696[1].

7.4 Alcohol.

8 Operating procedure

8.1 Specimen cutting

The pellet is cut using a diamond-impregnated metallography disk-cutting machine with a cooling system, along the selected axis (longitudinal or axial).

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

8.2 Resin embedding

Prior to rough and final polishing, the cut specimen is embedded in resin. Therefore, the half pellet 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 pellet Pu content, is then poured inside the mould.

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

Then, the identification number of the pellet sample can be graved with the engraver pen on the resin.

The mould should be cleaned in order to reuse it.

8.3 Rough polishing

The resin-embedded specimens shall 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 is described below:

— P180 (82 µm) abrasive disk (for specimens embedded in resin after cutting): time as necessary to reach the surface flush with the resin;

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

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

— P1200 (15 µm) abrasive disk: 30 s to 1 min;

Rough polishing is performed on a polishing machine with an air pressure of 100 kPa to 150 kPa, and at a rotation speed of 70 r/min to 270 r/min.

The specimen is thoroughly cleaned in alcohol between each step and after completion of the rough polishing operation. The cleaning may be done in an ultrasonic tank.

8.4 Final polishing

An example is described below.

The polishing operation is performed using a series of 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 air pressure of 100 kPa to 200 kPa.

The specimen is thoroughly cleaned in alcohol and dried in air after polishing. The cleaning may be done in an ultrasonic tank. The polished surfaces should be mirror finished and free of scratches.

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

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

— porosity measurements, and

— overall ceramographic examinations to reveal cracking.

9 Structure development

9.1 General

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

9.2 Development by thermal treatment

After cutting, the sample is not embedded, but placed into a mechanical device. Then, the half pellet 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.

Rough polishing

Rough polishing is performed on a polishing machine with an air pressure of 100 kPa to 150 kPa, and at a rotation speed of 70 r/min to 270 r/min.

— P180 (82 µm) abrasive disk (for specimens embedded in resin after cutting): time as necessary to reach the surface flush with the resin

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

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

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

The specimen is thoroughly cleaned (ultrasonically or not) in alcohol between each step and after completion of the rough polishing operation.

Final polishing (see 8.4)

Furnace sequence

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

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

— heating rate: 283 °C/h;

— fixed temperature level: 1 600 °C;

— duration: 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.

9.3 Development by chemical etching

The specimens are immersed for 30 s to 1 min 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 pellet composition (Pu content, etc.) and the results obtained.

Examples:

— room-temperature etching;

— distilled water: 96 ml;

— chromium oxide Cr_2O_3: 20 g;

— mass fraction of 40 % hydrofluoric acid: 48 ml;

— etching time: 25 s to 1 min;

— hot etching: 70 °C:

— distilled water: 24 ml;

— saturated ammonium difluoride solution: 1 ml;

— sulfuric acid (s.g. 1,84): 1 ml;

— etching time: 1 min to 5 min.

9.4 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 pellet composition (Pu content, etc.) and according to the results obtained.