

# MICROSCOPIC DETERMINATION OF THE PuO<sub>2</sub> GRAIN SIZE AND PORE SIZE DISTRIBUTION OF MOX PELLETS WITH AN IMAGE ANALYSIS SYSTEM

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## Abstract

The industrial way to obtain the Pu distribution in a MOX pellet is by Image Analysis. The PuO<sub>2</sub> grains are made visible by alpha-autoradiography. Along with the Pu distribution the pore structure is an item which is examined, the latter is determined on the unetched sample. After the visualization of the sample structure, the sample is evaluated with an Image Analysis System. Each image is enhanced and a distinction is made between the objects to be measured and the matrix. The relevant parameters are then analyzed. When the overall particle distribution is wanted, all identified particles are measured and classified in size groups, based on a logarithmic scale. The possible conversion of two-dimensional diameters to three-dimensional diameters is accomplished by application of the Saltykov algorithm. When a single object is of interest, the object is selected interactively, and the result is reported to the user.

## 1. INTRODUCTION

MOX (Mixed OXide) is a nuclear fuel that contains depleted uranium oxide (usually 95 to 93%), a by-product from the enrichment of uranium and plutonium oxide (usually 5 to 7%), a product from nuclear reactors. One of the most important items in a MOX pellet specification is the Pu distribution in the pellet. Along with the Pu distribution the pore structure is an item which has to be examined. The industrial way to obtain this information is by Image Analysis.

## 2. VISUALIZATION

### 2.1. Pu distribution

To make the PuO<sub>2</sub> grains visible a replica technique (alpha-autoradiography) is used: the sample (donor) affects the structure of another surface (receptor). For alpha-autoradiography, contact is made between the polished ceramographic sample and a cellulose nitrate film during a given period. The film is sensitive to alpha particles, emitted by the plutonium and americium, resulting in a change of transparency of the film.

### 2.2. Pore size distribution

The pore structure is determined directly on the unetched sample.

## 3. EVALUATION

### 3.1. Configuration

After the visualization of the sample structure, the sample is evaluated with an Image Analysis System. A CCD-camera projects microscopic images to the Image Analysis System via a frame grabber. For each item to be evaluated, an image is acquired.

## 3.2. The algorithm

### 3.2.1. Enhancement of the region edges

Since blurred edges (transitions) can occur in an image due to the bandwidth restrictions of electronic and optical systems, edge transitions between a light and a dark region extend over several pixels with descending (ascending) grey values. This can lead to a so-called halo effect (rings around a region). To avoid this effect, a function is applied to sharpen the region edges.

### 3.2.2. Generation of binary regions

In order to be able to measure the regions, segmentation is used to generate binary regions.

#### 3.2.2.1. *Pu* distribution

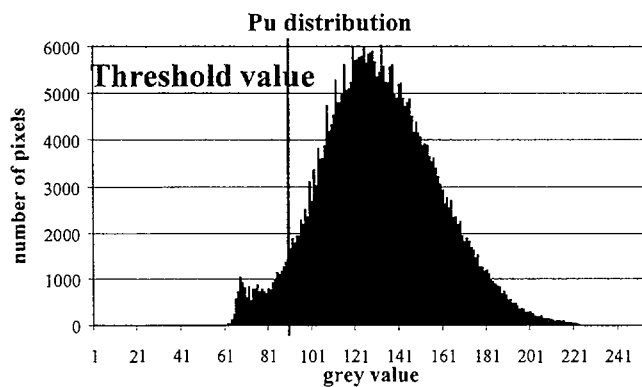


FIG. 1. Grey value distribution before segmentation

First, the number of regions is calculated with a fixed threshold value, determined by experience (Figure 1.). If the number of regions is larger or smaller than a predetermined maximum or minimum, the threshold value is changed automatically until this condition is fulfilled. Afterwards, the operator has still the possibility to adjust this value. Fig. 2 shows an example of an image before segmentation and Fig. 3 the same image after segmentation.

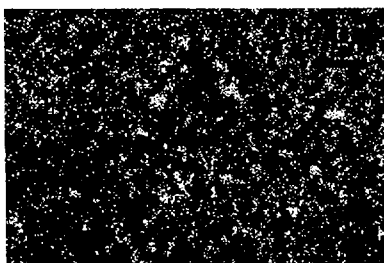


FIG. 2. Image of the *Pu* distribution before segmentation

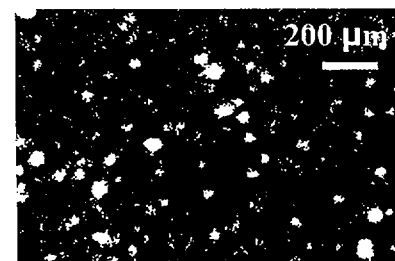


FIG. 3. Image of the same *Pu* distribution after segmentation

### 3.2.2.2. Pore size distribution

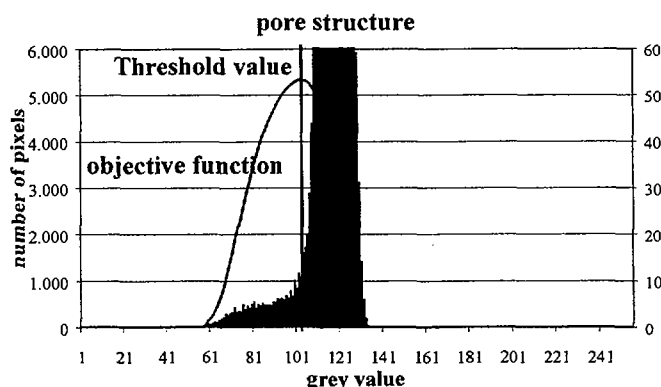


FIG. 4. Grey value distribution before segmentation

The threshold value is calculated by maximization of the interclass variance (Fig. 4): the image is partitioned into two connected sets (classes) of grey values. The objective is to separate the classes as much as possible and to regroup them as much as possible around their center. Here also, the operator has still the possibility to adjust this value after the calculation. Fig. 5 contains an image before and Fig. 6 the same image after segmentation.



FIG. 5. Image of the pore structure before segmentation

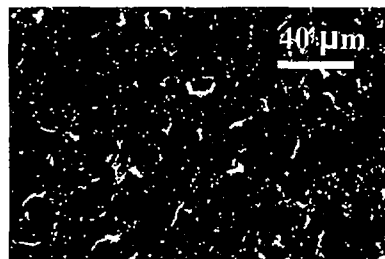


FIG. 6. Image of the pore structure after segmentation

### 3.2.3. Separation of connected regions (only for the Pu distribution)

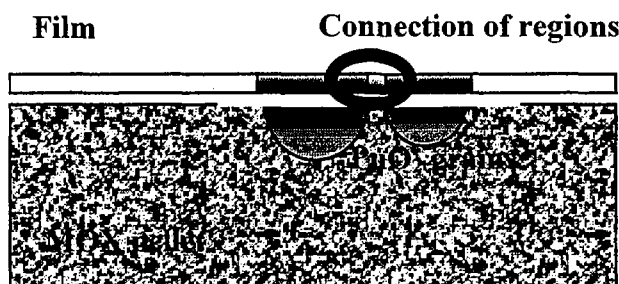


FIG. 7. Connection of regions

Since the areas on the film, which are affected by the plutonium and the americium, are slightly larger than the  $\text{PuO}_2$  grain sections, black regions from separate  $\text{PuO}_2$  grains may get connected, as shown in Figure 7.

To undo these connections, the regions are partitioned into different size groups and eroded and dilated a number of times, depending on their size.

### 3.2.4. Actual measurement

According to the settings of the measurement properties, automatic measurement of the regions is executed.

### 3.2.5. Extrapolation of the results to 3-D space

The Johnson-Saltykov method is used to determine the size distribution of particles in unit volume of an aggregate from a distribution of section areas.

This model assumes that:

- the 3-D particles are spherical in shape.
- each 3-D particle size distribution can be represented by a discontinuous distribution.

The latter makes the calculation less time-consuming and less complex. There is neither a sequential dependence upon previous calculations of other particle sizes, so errors will not propagate through the calculation.

Saltykov sets the interval limits of both the sphere diameters and the section area diameters equally spaced on a logarithmic scale. This ensures that the finest subdivisions occur at small diameters, where they are needed. If necessary, the scale can be extended easily in the direction of either larger or smaller diameters.

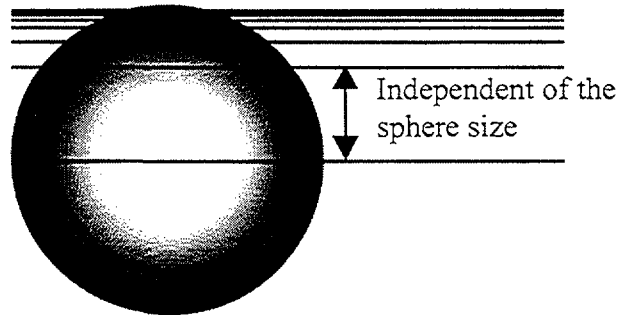


FIG. 8. The probability to cut a sphere between its maximum limits is independent of the sphere diameter

The probability to cut a sphere between two interval limits is independent of the diameter of the sphere (Fig. 8); consequently, the distribution of relative areas (for a monodispersed system of spheres) is independent of the absolute dimensions of the sphere. The calculation for a polydispersed system starts with the largest section area and ends with the smallest one.

Figs 9 and 10. show the result of the analysis of a typical MOX pellet.

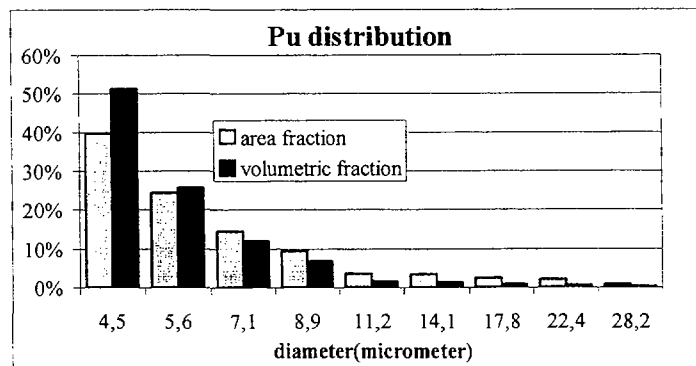


FIG. 9. Area and volumetric fraction of the PuO<sub>2</sub> grains

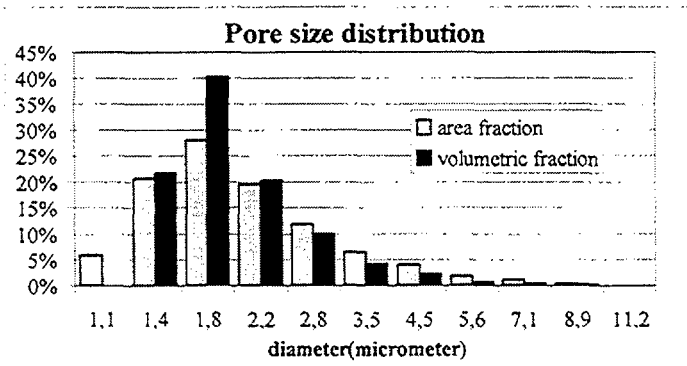


FIG. 10. Area and volumetric fraction of the pores

#### 4. CONCLUSIONS

- This way of determining the Pu distribution and pore structure makes statistical analysis possible, since large areas are studied. This makes the method more accurate and representative.
- Once the image is acquired, the automated part of the analysis is perfectly reproducible.
- Finally, the acquisition and the analysis of images are non-time consuming activities, so the results are rapidly available.

#### REFERENCES

- [1] KS 400 Imaging System Release 3.0 User's Guide, B40-615e (August 1998)
- [2] MICHEL COSTER et J.L. CHERMANT, Précis d'Analyse d'Images, ISBN 2-87682-020-X
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