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Microradiography of
Pyrocarbon-Coated $^{238}\text{PuO}_2$ Microspheres
to Determine Coat Thickness

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R. A. Morris
R. D. Strong

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MICRORADIOGRAPHY OF PYROCARBON-COATED
 $^{238}\text{PuO}_2$ MICROSPHERES TO DETERMINE COAT THICKNESS

by

R. A. Morris and R. D. Strong

ABSTRACT

A method of microradiographing pyrocarbon-coated PuO_2 microspheres without contaminating the plate and the radiographic equipment is described. Resolution of the order of one micron has been obtained. The equipment and the technique used are discussed.

INTRODUCTION

As part of a study on the feasibility of coating $^{238}\text{PuO}_2$ microspheres with pyrocarbon, microradiography was chosen for rapid determination of coat thickness. This method previously had been used at Los Alamos Scientific Laboratory with $^{235}\text{U}_2\text{C}_3$ particles of similar size. Due to the extreme contamination hazard associated with plutonium, a new type of fixture was designed to contain the contaminants without an appreciable loss of radiographic resolution.

METHOD

Microradiography of coated microspheres is normally accomplished by using Kodak high resolution plates with the particles in direct contact with the emulsion. Since these plates have a very low sensitivity to x-radiation requiring long exposure, a target-to-film distance (TFD) of 12 in. and a target size of 1.5 mm are used to shorten the exposure. Normally this combination would result in unsatisfactory resolution, but the minute size of the objects makes the unsharpness negligible. The normal exposure parameters are 6 kvp at 15 mA for 10 min, using a Balteau Electric Corporation

5-50-kvp unit with a 10-mil beryllium window. A helium atmosphere is used to reduce atmospheric absorption. The geometrical relationships, shown in Fig. 1, of a typical setup using a 150- μ -diam particle with a 40- μ -thick coating indicate that if the particle is against the emulsion, the geometrical unsharpness is less than 1 micron, satisfactory for this use.

The $^{238}\text{PuO}_2$ contamination problem requires isolation of the particles from the outside atmosphere but close enough contact with the plate emulsion to ensure satisfactory resolution. A 0.25-mil-thick Mylar film was selected from available thin films for its low radiographic absorption, dimensional stability, strength, and availability. A film sample was tested for resistance to anticipated alpha radiation and heat; the film did not appear to deteriorate after periods of exposure much greater than normal. Uncoated particles discolored the Mylar film but did not penetrate it in three weeks' time. The theoretical unsharpness increases by only 0.05 μ and is still less than 1 μ . In actual practice the unsharpness is somewhat increased since the film does not lie perfectly flat.

Fig. 1. Geometrical Relationships in X-ray Setup

The small fixture shown in Fig. 2 was devised for transferring and radiographing particles without exposure to outside atmosphere. A 0.10-g sample of coated particles was loaded from a dry box into the glass bottle to keep the outside of the bottle from becoming contaminated. The bottle was then screwed into the fixture to which the Mylar film had previously been sealed onto both sides of the plate with double-sided tape. The fixture was positioned in a carrying case which held the bottle upright

Fig. 2. Particle Containment Fixture

during transportation. The case and fixture are shown in Fig. 3. When the micro-radiograph was to be made, the fixture was inverted and the particles passed from the bottle through a hole into the viewing port. The fixture was then placed on the plate as shown in Fig. 4. After radiographic exposure, the fixture was reinverted, allowing most of the particles to return to the bottle, and placed in the carrying case.

Fig. 3. Fixture and Carrying Case

Fig. 4. Fixture in Exposure Position

To compensate for the additional 0.5-mil thickness of Mylar film, the exposure was changed to 10 kvp at 15 mA for 10 min.

The plates were developed for 8 min in GE Supermix x-ray developer, which is more readily available than the Kodak D-19 developer recommended by the manufacturer. Kodak x-ray fixer was used. Tests indicated that the speed was increased somewhat using the x-ray developer without noticeably affecting either the contrast or the resolution of enlargements.

RESULTS

The developed plates were viewed through a Nikon Shadowgraph with a 100X lens. For a permanent record, Polaroid photographs of the enlarged image were made. Some of these enlargements are shown

in Fig. 5: Fig. 5a shows representative examples of the nominal 20- μ coating deposited in 4 hours (The dark spots, caused by alpha exposure of the plate by particles with very thin coatings, do not occur when the thickness is over 15 μ .); Fig. 5b, examples of the nominal 30- μ coating deposited in 6 hours; Fig. 5c, examples of the nominal 40- μ coating deposited in 8 hours. The resolution obtained by this method of microradiography is apparent from the figure.

CONCLUSION

Microradiographic evaluation of the coat thickness of pyrocarbon-coated $^{238}\text{PuO}_2$ microspheres has been demonstrated with the use of 0.25-mil-thick Mylar films to contain the particles and separate them from the microradiographic plate.

Fig. 5. 100X Enlargements of Typical Particle Microradiographs