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BNWL-58

C-74-

FISSION PRODUCT DISTRIBUTION
IN IRRADIATED UO_2

MARCH 1965

ROUTE TO	REMARKS	EDITION	FILE NO.
<i>R. H. Hilliard</i>	<i>30655 201-T</i>		<i>DEC 29 1965</i>

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For The

U. S. ATOMIC ENERGY COMMISSION Under Contract No. AT(45-1)-1830

PRINTED BY/ FOR THE U. S. ATOMIC ENERGY COMMISSION

BNWL-58
UC-25, Metals, Ceramics,
and Materials

FISSION PRODUCT DISTRIBUTION
IN IRRADIATED UO_2

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March 1965

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Printed in USA. Price \$2.00. Available from the Clearinghouse for Federal
Scientific and Technical Information, National Bureau of Standards
U. S. Department of Commerce, Springfield, Virginia

ABSTRACT

The gross fission product relocation which occurs during high temperature irradiation of UO_2 fuel elements has been studied using autoradiography, microdrilling and radiochemical analysis. The fission product distribution is related to the characteristic microstructures of the UO_2 formed during irradiation. Annular bands of high fission fragment concentration are observed symmetrically about the thermal center with depletion in the large columnar grains. The distribution and concentration varies from fission product to fission product. Fission products concentrate in bands associated with metallic uranium inclusions that are formed during irradiation. Plutonium also migrates during irradiation.

FISSION PRODUCT DISTRIBUTION IN IRRADIATED UO_2

INTRODUCTION

The migration of fission products and plutonium in uranium dioxide fuel elements during irradiation at high temperatures has significant importance in burnup analyses, fuel element reactivity and fuel behavior. Earlier experiments⁽¹⁻⁸⁾ have shown that several fission products formed during irradiation exhibit extensive migration during high temperature irradiation. The purpose of this paper is to describe further refinements of experimental techniques and to discuss more precise data of the fission product distribution in irradiated UO_2 .*

Two complimentary techniques are used to study the distribution of fission products and plutonium: (1) autoradiographs are obtained using high resolution, low sensitivity photographic emulsion (see Appendix A), (2) microsamples from UO_2 fuel elements are obtained by precision microdrilling (Appendix B) and are analyzed by radiochemical analysis (Appendix C) for long half-life fission products.

The radiochemical results of samples from microdrilling show unusual resolution and reproducibility. The radiochemical results substantiate the data from the autoradiographs confirming areas of gross depletion and concentration. The autoradiographs provide significantly better resolution. However, they cannot distinguish between the different fission fragments. The data from both autoradiographs and radiochemical analysis are essential for complete fission product distribution analysis.

* The conclusions of this report apply only to UO_2 fuel elements. Similar data have been obtained for UO_2 containing PuO_2 and ThO_2 , and PuO_2 in ZrO_2 , MgO . These data have been reported in the Ceramics Research and Development Operation Quarterly Reports HW-76300, HW-76301, HW-76302, HW-76303, HW-76304, and HW-76305.

SUMMARY

Gross fission product relocation occurs during high temperature irradiation of UO_2 fuel elements. The distribution varies from fuel element to fuel element with major differences occurring with high and low exposure and with high and low heat generation rates.

The fission product distribution is related to the characteristic microstructures of the UO_2 formed during irradiation. Annular bands of high fission product concentration are observed symmetrically about the thermal center. A depletion of fission products is generally observed in the dense, pore-free region of the large columnar grains of UO_2 . In many instances, a concentration of radioactivity is observed near the thermal center and in the small columnar grains.

The distribution and concentration varies from fission product to fission product. Cesium and ruthenium show the greatest tendency to migrate while Zr-Nb and Ce-Pr shows the least. For example, variation in the cesium concentration across the fuel element radius varies as much as 1000 times.

Fission products concentrate in bands associated with metallic uranium inclusions that are formed at high temperatures during irradiation. This high concentration of fission fragments in the uranium could account for the increased stability of the uranium metal at the higher neutron exposures.

Plutonium also migrates during irradiation. The distribution of plutonium departs from the calculated distribution (assuming no relocation), with a higher than theoretical concentration near the thermal center and a minimum in the large columnar grains.

The presence of coolant water introduced into UO_2 fuel element during irradiation from cladding failures results in gross fission product migration in localized areas near the rupture.

RESULTS AND DISCUSSION

Gross fission product relocation occurs in UO_2 fuel elements irradiated with high central temperatures. The distribution varies from fuel element to fuel element and is related to the characteristic microstructures of the UO_2

formed during irradiation (Figure 1).^{*} Only in fuel elements which exhibit grain growth are the fission products observed to migrate.

Annular bands of high fission product concentration or fission product depletion are observed symmetrically about the thermal center. The number of bands varies and depends upon the irradiation conditions. For example, a fuel element irradiated for a relatively short period of time has multiple bands with various fission product concentrations (Figures 1 and 2). A depletion is observed in the dense porosity-free region of the columnar grains with a fission product concentration near the thermal center. The concentration is associated with the high porosity in the columnar grains. A narrow band of high fission product concentration is found in the small columnar grains immediately outside the depletion band and outside the high density, porosity-free large columnar grains.

Another example of low exposure fuel is shown in Figures 3 and 4. This fuel element differs from the previous fuel element (Figures 1 and 2) in that the heat generation rate increased with increased exposure. The converse was true in the previous element. The same general bands of varying fission product concentrations were observed but with some variations. The depleted band in the large columnar grain regions is much wider. A very high fission product concentration band corresponds to a narrow high porosity UO_2 region adjacent to the central cavity. In addition, a concentration band of lesser intensity extends into the columnar grains and is related to the presence of uranium metal⁽⁹⁾ and not to the presence of porosity.

In contrast, the distribution in high exposure UO_2 is significantly different, both in relation to the fission product distribution and to the UO_2 microstructure (Figures 5-7). (The heat generation rate of the high exposure UO_2 decreased with increased exposure.) The long narrow columnar grains have one intense band of fission product concentration (Figures 5 and 7). The remainder of the UO_2 has a smaller and uniform concentration of fission products.

^{*} See Appendix D for irradiation summary for all fuel elements and capsules.

Autoradiograph

Macrophotograph

0.10 in.

FIGURE 1

Autoradiograph of Irradiated, Vibrationally Compacted UO_2 Fuel Element (GEH-4-49) Showing Fission Fragment Distribution Relative to Structural Changes in UO_2 . (Halves are mirror images.)

FIGURE 2

Fission Product Distribution and Related Microstructures of Irradiated UO₂ Fuel Capsule (GEH-4-49). (Vibrationally compacted, fused UO₂ irradiated to 8×10^{18} fissions/cm³)

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FIGURE 3

Radiochemical Analyses of Microdrilled UO₂ Sample (APED-A-597)
 Showing Variation in Fission Product Distribution. (See also Figure 4)
 Neg. 0641965-3

FIGURE 4

Fission Product Distribution and Related Microstructures of Irradiated Pelleted UO₂ Fuel Element (APED-A-597). (Sintered UO₂ irradiated to 5×10^{19} fissions/cm³).

Neg. 0642485-1

FIGURE 5

Macrophotograph and Autoradiograph of Swaged UO_2 Fuel Element (GEH-14-237) Irradiated to 6×10^{20} Fissions/cm³ Showing Concentration of Fission Products and Metallic Uranium Inclusions in Same Positions of Columnar Grains.

As-Polished
(75X)

FIGURE 6

Concentration of Metallic Uranium Inclusions in Columnar Grains
of Swaged UO₂ Fuel Capsule (GEH-14-237) Irradiated to 6×10^{20}
Fissions/cm³.

Neg. 0641019

FIGURE 7

Fission Product Distribution and Related UO₂ Microstructures of Irradiated UO₂ Fuel Capsule (GEH-14-327). (Swaged UO₂ Irradiated to 6×10^{20} fissions/cm³)

Fission products tend to concentrate in bands associated with a metallic second phase that is formed at the high temperatures during irradiation.⁽⁹⁾ The second phase has been identified by X-ray diffraction as β -uranium⁽⁹⁾ which at high exposure is alloyed with fission fragments.⁽¹⁰⁾

Autoradiographs and radiochemical analyses^(5, 6, 9) show a fission product concentration in areas of UO_2 fuel element which correspond to areas of high uranium concentration (Figures 5-7). It cannot be determined from the autoradiographs if the fission products are alloyed with the uranium or are associated with the UO_2 surrounding the inclusions. The increased resistance of the inclusions to chemical etching in the higher exposure UO_2 suggests that the fission products have alloyed with the uranium. A higher concentration of etch pits in the UO_2 immediately surrounding the inclusions⁽⁹⁾ could also indicate an increased concentration of fission fragments in the UO_2 .

To determine if the uranium inclusions contained a higher fission product concentration, irradiated UO_2 fuel elements were leached with 3N HCl in an attempt to selectively dissolve the uranium from the UO_2 . The leach solution was analyzed radiochemically and compared with the average fission product concentration in the UO_2 . Preliminary data of two fuel elements show a higher than average concentration of Cs^{136} and Sr^{90} in the leach solution (Table I).

TABLE I
RADIOCHEMICAL ANALYSES OF IRRADIATED UO_2 FUEL ELEMENTS

Fuel Element	Fission Fragment	Fission Fragment Leach Solution, mg U*	Concentration UO_2	Concentration Difference mg U*	Increased Concentration in Leach Solution
GEH-14-178 ⁽¹⁾	Sr^{90}	1.8×10^6	1.7×10^5	1.6×10^6	9.4 X
	Ru^{106}	5.8×10^5	7.2×10^5	-1.4×10^5	-0.2 X
	Cs^{137}	2.9×10^7	5.7×10^4	2.9×10^7	500 X
GEH-14-237 ⁽²⁾	Sr^{90}	1.3×10^6	1.3×10^5	1.2×10^6	9.2 X
	Ru^{106}	2.6×10^5	4.9×10^5	-2.3×10^5	-0.5 X
	Cs^{137}	4.1×10^7	4.5×10^4	4.1×10^7	911 X

* Disintegrations/min

These results do not agree with microprobe analysis of similar metallic phases from AERE, Harwell which indicate the presence of ruthenium and molybdenum in some inclusions and of cerium and barium in others.⁽¹⁰⁾ The high cesium and strontium may have been leached from the UO_2 -cladding region or the thermal cavity instead of from the uranium inclusions. However, results suggest some fission products tend to accumulate in the uranium. The presence of these elements could account for the increased chemical resistance at higher exposures and the stabilization of the uranium in the beta phase.⁽⁹⁾

The distribution and concentration in the same element varies from nuclide to nuclide. Cs^{137} and Ru^{106} show the greatest tendency to migrate. The Cs^{137} concentration, for example, varies as much as 1000 times across the fuel element radius (Table II). Ce^{144} - Pr^{144} and Zr^{95} - Nb^{95} exhibit the least tendency to migrate.

TABLE II
FISSION FRAGMENT MINIMUM CONCENTRATION
(APED-A-597)

Fission Fragment	Radius of Minimum Fission Fragment Concentration, cm	Ratio of Maximum and Minimum Fission Fragment Concentration
Ce-Pr^{144}	0.36	1.4
Cs^{137}	0.30	300
Ru^{106}	0.34	100
Zr-Nb^{95}	0.39	1.5
Sr^{90}	0.38	6
Pu	0.40	1.4

A closer analysis of our most reproducible data (Figure 7) shows the location of the minimum in the fission product concentration to vary between nuclides (Table II).

The greater the refractory nature of the fission products, the farther the minimum is from the thermal center. The criterion for refractoriness is the thermodynamic stability of the fission fragment oxides.

Plutonium also migrates during irradiation. In fuel elements examined, the distribution of plutonium departs from a calculated distribution, assuming no relocation. A higher than theoretical concentration is measured near the thermal center.

Localized redistribution is enhanced in fuel elements which rupture during irradiation. Autoradiographs of two fuel elements irradiated for short periods after a break developed in the cladding, revealed an enlargement of the depletion near the rupture (Figures 8 and 9). The localized depletion enlarged both toward and away from the thermal center. An alteration of the symmetry of the porosity in the large columnar grains in this same area also occurred. The outer edge of the porosity moved toward the thermal center.

A significant problem associated with the interpretation of the autoradiographs is the identification of the isotopes which expose the emulsion. The emulsion registers the complex radiation from many isotopes. Since these isotopes decay at different rates, the apparent distribution of fission products as determined by autoradiography, could change with time. Improved resolution is generally obtained when the autoradiographs are made soon after discharge when isotopes with relative short half-life contribute significantly to the total radiation of the fuel. This apparent variation with time could explain the cause of some discrepancies between the radiochemical analysis and autoradiographs, e. g. GEH-4-49 (Figures 1 and 2) where the time period between the autoradiography and the microdrilling was large.

The extensive migration of fission fragments appears complex, undoubtedly resulting from more than one mechanism. Six possible mechanisms are being considered to explain fission product migration:

- Thermal diffusion of fission products in the large temperature gradient
- Entrainment by voids and pores that diffuse along the thermal gradient
- High temperature sublimation, and subsequent condensation in outer portions of the fuel element
- Fission product diffusion through the UO_2 because of an electrical potential resulting from the thermal electrical properties of UO_2

FIGURE 8

Autoradiograph and Macrograph of Sintered UO_2 (Containing W Marker Wires) (GEH-4-72) Irradiated to Approximately 5×10^{16} Fissions/ cm^3 Showing Variation in Fission Product Distribution Resulting from Cladding Failure.

Neg. 06243237-4

FIGURE 9

Autoradiograph of Large Diameter, Vibrationally Compacted UO₂ Fuel Element (GEH-12-29) Showing Variations in Fission Product Distribution Resulting from Cladding Failure.

(Fuel element operated for less than 1 min at fuel power)

Neg. 0643237-3

- Fission fragments, which are soluble in UO_2 at high temperatures, precipitate selectively as the UO_2 temperatures decrease during reactor shutdown or fuel burnup. Simultaneous precipitation with uranium may also occur.
- Fission fragment migration as gaseous or volatile precursors of the measured nuclides.

ACKNOWLEDGEMENT

The author is indebted to the staff of Radiometallurgy Laboratory for the autoradiography and microdrilling and to P. O. Jackson for the radiochemical analysis. Fuel Element APED-A-597 was obtained through the cooperation of General Electric-APED personnel in a sample exchange program.

APPENDIX A

AUTORADIOGRAPHY

Autoradiographs of irradiated UO_2 are obtained from metallographic cross sections of irradiated UO_2 with (1) high resolution, low sensitive photographic plates,^(8, 11) and (2) photographic stripping films.⁽⁶⁾ Initially, autoradiographs were made using thin glass plates.⁽¹⁾ The darkening of the glass was used to determine relative fission product concentrations. Increased resolution is obtained using improved photographic emulsion techniques.⁽⁶⁾

A technique, originally developed at Vallecitos Atomic Laboratory,^(8, 11) involves the exposure of photographic plates with a high resolution, low sensitivity emulsion which is sensitive principally to beta radiation. The technique was modified at Battelle-Northwest (at that time, Hanford Laboratories). The emulsion side of the 2 1/4 x 4 in. glass plates are covered with a 0.001 cm thick, aluminized Milar sheet. The reverse side of the plate is masked with opaque paper and sealed with black tape. The aluminized coating of the Milar sheet prevents exposure by visible light and minimizes radioactive contamination.

Cross sections of irradiated UO_2 are mounted in resin, ground and polished to obtain a smooth, flat metallographic surface. These surfaces are placed face down on the Milar covered emulsion of the photographic plate. A lead weight is placed on the sample to insure the best contact between the sample and the plate.

Exposure times vary from a few seconds to days, depending upon the radioactivity of the UO_2 . After exposure the Milar and mask are removed and the plate is developed. The developed plate is enlarged approximately 5X using "Gravure" copy film. A contact or enlargement print is made from the negative into a final positive print, i.e., dark areas on the print represent areas of radioactivity from the irradiated sample.

Lantern slides are also used when the radioactivity is low and exposure times are longer. Because of the low radioactivity, the exposure is possible in a low level, light tight, glove box.

Highest resolution is obtained using stripping film.⁽⁶⁾ The method consists of floating the emulsion on the sample, exposing and developing while in contact with the sample. The emulsion is then transferred to a glass plate. Because of the difficulty in handling, this method has been applied only to relatively low exposure fuel systems.

APPENDIX B

MICROSAMPLING

Scribing

Metallographic surfaces of irradiated UO_2 can be scribed with a hard tool point, and the UO_2 removed with an adhesive. The polished UO_2 cross section is scribed with a vibrating diamond point across a pre-determined diameter. The linear motion of the tool is controlled using a metal straight edge as a guide. The erupted UO_2 is removed from the surface with cellophane tape or "fax" film. The line of UO_2 particles adhering to the tape or plaxtic is cut into equal segments for radiochemical analyses.

The disadvantages of this method are (1) the small amounts of UO_2 obtained for analyses, and (2) the potential scatter or UO_2 during scribing.

Microdrilling

Recently, techniques have been developed for remote microdrilling of irradiated ceramic materials.⁽¹²⁾ Drills as small as 0.030 cm in diameter are accurately positioned (to ± 0.002 cm) at predetermined parts of the UO_2 system. Drillings are made at intervals of approximately 0.06 cm and from 0.02 to 0.05 cm deep.

During drilling, the UO_2 powder is collected through a vacuum tip onto a plastic filter disc. The UO_2 and filter are removed for radiochemical analyses. A new drill, filter, and vacuum tip are used for each drilling.

APPENDIX CRADIOCHEMICAL ANALYSES⁽¹³⁾

UO₂ samples received on cellophane tape are treated with a 50-50 mixture of benzene and ether and subjected to ultrasonic vibrations. The loose UO₂ particles are centrifuged from the organic phase. The organic phase is washed once with a mixture of 6N HCl and 0.1N HF and once with water to remove any extracted radionuclides.

UO₂ samples received on Fax film are separated with acetone. The loose UO₂ particles and organic phase are treated identically to that described in the preceding paragraph.

Initially the UO₂ was dissolved by boiling to dryness several times with a mixture of 6N HNO₃ and 0.1N HF and finally diluted with that mixture. More recently, a closed system employing a nitrogen protective atmosphere above the sample has been used during dissolution. The samples are first boiled in HCl and hydroxylamine hydrochloride followed with a 8N HNO₃ - 0.1N HF acid mixture. The initial treatment is found to prevent volatilization of ruthenium trioxide during dissolution of UO₂.

In order to minimize loss from aerosols spraying from the dissolution flask, off-gasses are passed through an acid trap followed by a caustic trap. Normal activity in the caustic trap is insignificant. The acid trap is combined with the sample after dissolution.

For samples which are analyzed a long time after discharge, Ce¹⁴⁴-Pr¹⁴⁴ and Zr⁹⁵-Nb⁹⁵ are determined by direct gamma energy analysis. Cs¹³⁷, Ru¹⁰⁶, and Sr⁹⁰ are determined after chemical separation. For samples analyzed shortly after discharge all nuclides, Ce¹⁴⁴-Pr¹⁴⁴, Ru¹⁰³,*

* Ru¹⁰³ results would be subject to the largest experimental error since Ru¹⁰³ is usually present in lesser amounts.

Ru^{106} , Zr^{95} - Nb^{95} , Cs^{137} and Sr^{90} are measured after chemical separations.

Uranium is determined fluorophotometrically or by direct weighting of UO_2 sample (approximately 1/2 mg). Total plutonium is determined by scintillation counting of plutonium.

APPENDIX DIRRADIATION SUMMARY OF UO₂ IRRADIATIONS

Irradiation Designation	APED-A-597	GEH-4-49	GEH-4-72	GEH-12-29	GEH-14-177	GEH-14-237
Test Facility	GETR	MTR	MTR	ETR	MTR	MTR
Irradiation Time (Equivalent Full Power Days)	--	14.7	0.75	-*	398	501
Estimated Heat Genera- tion (KW/cm)						
Initial	0.87	1.2	~1.3	~1.3	0.68	0.63
Final	1.23	1.1	-	-	0.27	0.19
Estimated Exposure (Fission/cm ³)	2 x 10 ¹⁹	8 x 10 ¹⁸	~5 x 10 ¹⁶	-*	4.7 x 10 ²⁰	6 x 10 ²⁰

* GEH-4-72 ruptured after 4 min at full power

** GEH-12-29 was irradiated for only a few minutes before cladding failure.

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