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EFFECTIVE THERMAL CONDUCTIVITY OF $\text{PuO}_2\text{-UO}_2$ AT HIGH BURNUPS

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U.S. ATOMIC ENERGY COMMISSION
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 $\text{PuO}_2 - \text{UO}_2$ AT HIGH BURNUPS

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ABSTRACT

The integral conductivity of $(\text{Pu}_{0.2}\text{U}_{0.8})\text{O}_{2.00}$ from 1200°C to the temperature of melting was determined by re-irradiating a fuel pin at a peak power of 31 kW/ft following a steady-state irradiation to a burnup of 75,000 MWd/Te in the reflector of the Materials Test Reactor (MTR). The final high power irradiation was carried out in the VH-4, "hydraulic rabbit," facility of the MTR.

The $\int kd\theta$ calculated from the fuel surface temperature (1200°C) to melting was 42.8 ± 6.4 W/cm. Comparison of this value with data on the $\int kd\theta$ to melting of $(\text{Pu}_{0.2}\text{U}_{0.8})\text{O}_{2.00}$ irradiated to low burnups showed that within the experimental error of the experiments no significant change in the $\int_{1200}^{T_m} kd\theta$ of $(\text{Pu}_{0.2}\text{U}_{0.8})\text{O}_{2.00}$ occurred at a burnup of 75,000 MWd/Te.

1. SUMMARY

The effect of burnup on the integral conductivity to melting of mixed oxide fuel, $(\text{Pu}_{0.2}\text{U}_{0.8})\text{O}_{2.00}$, was determined by irradiating a fuel pin in the reflector of the MTR to a burnup of 75,000 MWd/Te at a power which did not result in fuel melting. After reaching a fuel burnup of 75,000 MWd/Te the fuel pin was removed, gamma scanned, and neutron radiographed to ensure that it had not failed. The fuel pin was then reirradiated in the VH-4, "hydraulic rabbit," facility of the Materials Test Reactor (MTR) in Idaho Falls, Idaho, at a power of 31 kW/ft for 5 hours.

Post-irradiation examination of the fuel pin showed that:

1. Fuel melting occurred to 58% of the fuel diameter.
2. Metallic fission products in the melted region of the fuel agglomerated into a large ingot at the bottom of the fuel column.
3. The maximum diametral strain in the cladding was 1.32%.

The $\int_{T_s}^{T_m} kd\theta$ calculated from the extent of melting observed in the fuel was 42.8 ± 6.4 W/cm. Comparison of this value with $\int kd\theta$ values obtained from low burnup mixed oxide irradiations showed that within the experimental error of the measurements the irradiation did not affect the conductivity integral.

2. INTRODUCTION

An assessment of the effects of burnup on the physical properties of the mixed oxides of plutonium and uranium is necessary to determine the conditions under which reactors fueled with these oxides can be operated safely. Studies of parameters which can alter the physical properties of the mixed oxides of plutonium and uranium during irradiation are being carried out as part of the *Fast Ceramic Reactor (FCR) Program*.

The experiment described in this report was conducted to determine the effects of irradiation to high burnup on the effective thermal conductivity of $(\text{Pu}_{0.2}\text{U}_{0.8})\text{O}_{2.00}$.^{*} This experiment was designed to complement a series of earlier irradiation experiments¹ which determined the effect of stoichiometry and density on the effective thermal conductivity of $(\text{Pu}_{0.2}\text{U}_{0.8})\text{O}_{2.00}$ fuel at low burnups. As noted in reference 1 a large number of integral to melt experiments have been run on UO_2 at low burnups. Efforts to measure UO_2 or mixed oxide conductivity at high burnups, however, have been limited to experiments conducted at BAPD on UO_2 using in-fuel thermocouples at temperatures below 1000°C .² These tests were conducted at too low a temperature to be applicable to LMFBFR's where typical operating temperatures are in the range of 1000°C to 2800°C . No comparable data on the effect of burnup or thermal conductivity of mixed oxides have been developed. The experiment described in this report was conducted to establish whether any significant change in conductivity occurred in mixed oxide fuel at high burnups.

In the present experiment two identical mixed oxide fuel pins were encapsulated and irradiated in the Materials Test Reactor (MTR) in Idaho Falls, Idaho. The capsules were irradiated to burnups of approximately 80,000 MWd/Te at powers of approximately 19 kW/ft. One capsule was removed from the reactor and examined to serve as a control. The remaining capsule was reirradiated in the VH-4, "hydraulic rabbit," facility of the MTR. The irradiation in the "hydraulic rabbit" was carried out at a linear power sufficient to produce center melting in the fuel, which was required to calculate the integrated thermal conductivity to the melting point of the fuel, $\int_{1200^\circ\text{C}}^{T_m} k d\theta$.

* Effective thermal conductivity refers to the integral conductivity to melting or the linear power required to produce incipient melting.

3. FUEL PIN AND CAPSULE DESIGN

The fuel pin and capsule design used for the irradiations are shown in Figures 3-1 and 3-2, respectively.

The fuel pin gas plenum was designed to safely accommodate the release of 100% of the fission gas generated in the fuel at burnups up to 100,000 MWd/Te. The required gas plenum volume was provided by increasing the diameter at the top of the fuel pin. This was necessitated by the length-to-diameter ratio limits required for capsules to be irradiated in the VH-4, "hydraulic rabbit," facility of the MTR. Experiments irradiated in this facility are "shot" into the reactor core by the hydraulic pressure of the coolant water flowing in the facility tube. Since the facility tube has a constant diameter and contains a 90-degree bend only experiments with less than a certain length-to-diameter ratio can be irradiated in the VH-4 facility.

The capsule, Figure 3-2, consisted of an outer stainless steel containment can, an aluminum thermal dam, the fuel pin, and an expansion bellows.

The bellows eliminated the necessity of leaving a void at the top of the capsule to provide for expansion of the NaK used to provide a thermal bond between the fuel pin and the capsule. The expansion volume is provided by compressing the bellows. This capsule design enabled the capsules to be handled in any attitude during transportation and reactor loading without the possibility of gas bubbles being trapped in the NaK-filled heat transfer annulus. Entrapment of a bubble in the annulus next to the fuel pin could result in failure of the fuel pin due to overheating of the cladding as a result of the decreased heat transfer.

Detailed fuel fabrication parameters for each fuel pin are listed in Tables 3-1 and 3-2.

Table 3-1
ISOTOPIC ANALYSIS OF FUEL

Fuel Pin	Batch	Pu Isotopes (atom %)				U-235
		239	240	241	242	
E1E	B-107	91.10	8.20	0.70	0.04	24.50
E1F	B-105	90.98	8.25	0.72	0.04	23.20

Table 3-2
FUEL PIN FABRICATION DATA

Fuel Pin	E1E	E1F
Powder Source - Batch No.	B-107	B-105
Ratio Pu/Pu+U, %	19.70	20.00
Fuel Diameter, in.		
Range	0.2162 0.2167	0.2162 -0.2172
Average	0.2164	0.2167
Fuel Length, in.	1.879	1.856
Fuel Weight, g	11.738	11.707
Fuel Density, % T.D.		
Range	93.4-95.0	93.5-95.0
Average	94.7	94.6
Fuel O/M, Nominal	2.000	1.999
Cladding Diameter, in.		
i.d., nominal	0.220	0.220
o.d., nominal	0.250	0.250
Diametral Gap, in.		
Range	0.0038-0.0033	0.0038 0.0028
Average	0.0036	0.0033
Smeared Density, % T.D.	90.9	91.8
Total Weight of Fuel Pin, g	36.7	36.3
Cladding Material	347 SS	347 SS

Figure 3-2. Capsule Design for E1E and E1F Irradiations

4. IRRADIATION FACILITIES AND HISTORY

The two capsules, designated E1E and E1F, were irradiated in the MTR reflector to fuel burnups of 86,900 MWd/Te and 74,900 MWd/Te, respectively. The capsules were then removed from the reactor and were neutron radiographed and gamma scanned to establish that the fuel pins had not failed. After neutron radiographs and gamma scans were taken capsule E1E was selected to be destructively examined and capsule E1F was returned to the MTR for further irradiation in the VH-4, "hydraulic rabbit," facility. The positions which the capsules occupied in the MTR are indicated in Figure 4-1.

Prior to the 5-hour high power irradiation of capsule E1F, Co-A1 and Ni flux wires were irradiated in the VH-4 facility to determine the unperturbed flux to which the capsule would be exposed. The unperturbed fluxes determined from the flux wire analyses are listed in Table 4-1. Flux wires were also attached to the outside of each capsule during the irradiation in the MTR reflector. Flux values determined from the analysis of these wires are also listed in Table 4-1. Insertion and discharge dates for each capsule are listed in Table 4-2. A plot of the MTR operating power history during the time the capsules were in the reactor is shown in Figure 4-2.

Table 4-1
FLUX VALUES DETERMINED FROM FLUX WIRE ANALYSIS

Capsule	Flux (n/cm ² - sec)	
	E1E	E1F
MTR Reflector		
Thermal	5.5×10^{13}	4.9×10^{13}
Fast	1.2×10^{13}	1.1×10^{13}
MTR VH-4		
Thermal	-----	1.03×10^{14}
Fast	-----	3.3×10^{12}

Table 4-2
CAPSULE INSERTION AND REMOVAL DATES

Capsule	Position	Insertion	Discharge
E1E	Reflector	8-17-65	8-8-66
E1F	Reflector	8-17-65	8-8-66
	VH-4	3-13-65 (1400 hr)	3-13-65 (1930 hr)

Figure 4-1. MTR Reactor Tank Arrangement

4-3/4-4

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Figure 4-2. Operating Power History of Materials Test Reactor During Irradiation of Capsules E1E and E1F

5. POST-IRRADIATION EXAMINATION

The complete post-irradiation examination conducted at the General Electric Radioactive Materials Laboratory at Vallejos, California, consisted of:

1. Gamma scan and neutronradiography of capsules and fuel pins.
2. Radiochemical and flux wire analysis for fuel burnup.
3. Diametral and length measurements on the fuel pins.
4. Fission gas collection and analysis.
5. Sectioning and metallographic examination of the fuel pins.

5.1 GAMMA SCAN AND NEUTRONRADIOGRAPHY

The gross activity gamma scans and neutronradiographs of each capsule after removal from the reflector of the MTR are shown in Figures 5-1 through 5-3. No unusual peaks were observed which would indicate gross fuel relocation or gamma activity outside of the fuel cladding. The neutronradiographs did not show any gross fuel relocation or indications of cladding failure. Based on the gamma scans and neutron radiographs of the capsules it was concluded that both capsules had operated without failure. Capsule E1E was selected for destructive examination and capsule E1F was returned to the MTR for reirradiation in the VH-4 facility at powers sufficient to produce center fuel melting.

An increase in fuel length was observed in both the gamma scans and neutronradiographs. The pre- and post-irradiation fuel lengths are listed in Table 5-1.

Table 5-1
PREIRRADIATION AND POSTIRRADIATION FUEL LENGTH MEASUREMENTS
OF CAPSULES E1E and E1F AFTER
IRRADIATION IN THE MTR REFLECTOR

Capsule	E1E	E1F
Preirradiation Length (in.)	1.879	1.856
Postirradiation Length (in.)		
Gamma Scan	1.962	1.933
Neutron Radiography	1.950	1.920
$\Delta V/V$ (Based on γ -Scans)*	0.0442	0.0416

* This value neglects any radial densification change and does not include the volume of the central void.

Assuming that the cladding diameter remained constant, the changes in fuel length measured correspond to a $\Delta V/V$ of 4.4% and 4.2% for capsules E1E and E1F, respectively. A $\Delta V/V$ of this magnitude is approximately 0.2% $\Delta V/V$ per 10^{20} fis/cc. It should be noted that the $\Delta V/V$ calculated is an apparent swelling rate not an absolute swelling rate since the fuel was not initially 100% dense and the neutron radiographs show that a center void was present in the fuel when it was removed from the reactor.

The gamma scan and neutronradiograph of capsule E1F taken following the irradiation of the capsule in the VH-4, "hydraulic rabbit," facility are shown in Figures 5-4 and 5-5. The gamma scan indicated a shift in activity toward the bottom of the fuel pin. The most noticeable difference between the gamma scans obtained before and after irradiation in the VH-4 facility (Figures 5-2 and 5-4) was the decrease in ZrNb-95 and La-140 activity at the top of the fuel column and the large increase in the activity of Ru at the bottom of the fuel column. As discussed in subsection 5.5 the ruthenium activity at the bottom of the fuel column was found to be due to a large ingot of fission products formed at the bottom of the fuel column.

POSITION	ISOTOPES OBSERVED	Zr Nb95 (relative)	La 140 (relative)	POSITION	ISOTOPES OBSERVED	ZrNb 95 (relative)	La-140 (relative)
1	Cr 51. Co 60			8	RuRh 103, ZrNb 95, La-140	100	4.8
2	Cr 51. Co 60			9	RuRh 103, ZrNb 95, La-140	130	5.0
2a	Cr 51. Co 60			10	RuRh 103, ZrNb 95, La 140	93	4.5
3	Cr 51. Co 60			11	RuRh 103, ZrNb 95, La 140	130	5.5
4	Cr 51. Co 60			12	RuRh 103, ZrNb 95, La-140	82	4.2
5	Cr 51. Co 60			13	RuRh-103, ZrNb-95, La-140	120	4.8
6	RuRh-103(tr), ZrNb 95, Co60, La-140	7.5	0.35	14	RuRh-103, ZrNb-95, Co-60, La-140	6.5	0.4
7	ZrNb-95, LaMO (RuRh is low)	170	6.0	15	Cr 51. Co 60		

Figure 5-1. Gamma Scan, Capsule E1E after Steady-State Irradiation

Figure 5-2. Gamma Scan, Capsule E1F after Steady-State Irradiation

CAPSULE E1E

CAPSULE E1F

Figure 5-3. Neutronradiographs of Capsules E1E and E1F

Position	Isotopes Observed	Comments
1	Co-60	
2	Co-60	
3	Co-60	
4	Co-60	
5	Cs-137, ZrNb-95, Co-60	
6	Ru-106, Cs-137, ZrNb-95, Co-60, La-140, Pr-144	
7	Ru-106, Cs-137, ZrNb-95, Co-60, La-140, Pr-144	Similar to Position 6 except Cs-137 is lower.
8	Ru-106, Cs-137, ZrNb-95, Co-60, La-140, Pr-144	Fission products twice the intensity of Position 7.
9	Ru-106, Cs-137*, ZrNb-95, Co-60, La-140, Pr-144	Similar to Position 8 except Cs-137 is lower.
10	Ru-106, Cs-137*, ZrNb-95, Co-60, La-140, Pr-144	Similar to Position 9.
11	Ru-106, ZrNb-95, Co-60	Ru-106 is high; twice the intensity relative to Position 10. ZrNb-95 is low; 1/4 as intense as Position 10.
12	Co-60	Trace of fission products.

* trace

Figure 5-4. Gamma Scan of Capsule E1F Following Irradiation in VH-4 Facility

Figure 5-5. Neutronradiograph of Capsule E1F Following High Power Irradiation in VH-4 Facility

5.2 BURNUP DETERMINATION

The burnup of the fuel was determined from mass spectrometer measurements of the neodymium present in the fuel and the depletion of plutonium and uranium (heavy elements) in the fuel.³ Burnup values were also calculated from the flux wire analyses discussed in Section 4. The calculated burnup values are listed in Table 5-2.

The neodymium values are considered to be the most accurate numbers and were used for all calculations requiring burnup values.

Table 5-2
COMPARISON OF FUEL BURNUP DETERMINED BY DIFFERENT METHODS

Capsule	Burnup (MWd/Te)	
	E1E	E1F
Flux Wire Analysis	92,200 ± 13,800	82,850 ± 12,400
Neodymium *	86,900 ± 2,600	75,000 ± 2,300
Heavy Element **	82,700 ± 2,500	73,100 ± 2,200

* Based on Nd-148.

** Pu+U

5.3 DIAMETER AND LENGTH MEASUREMENTS

Pre- and post-irradiation measurements of the diameter along the length of the fuel column are shown in Figures 5-6 and 5-7 for fuel pins E1E and E1F, respectively. Little change in diameter was observed to have occurred in fuel pin E1E, except for one region near the bottom of the fuel column. In this region diametral increases up to 0.002 inch were observed. Shown in Figure 5-8 are profilometer traces of the diameter of fuel pin E1E at discrete intervals along the pin. The maximum ovality was found to occur near the interface between the fuel and the bottom UO₂ insulator pellet. The axes of ovality were observed to shift abruptly 90 degrees in orientation just below the center of the fuel column.

The diameter of the E1F pin increased a maximum of 0.0033 inch at a point near the center of the bottom half of the fuel column. Since no increase in the cladding diameter was observed for fuel pin E1E the increase in diameter of the E1F fuel pin is believed to be caused primarily by the change in fuel volume resulting from the melting and thermal expansion of the fuel.

No change in fuel pin length was measurable for either fuel pin E1E or E1F.

5.4 FISSION GAS ANALYSIS

The percentage release of fission gas from fuel in the E1E and E1F pins was calculated from (1) the measured Kr-85 content of the gas released from each fuel pin, (2) the calculated volume of fission gas formed, and (3) the measured volume of gas released from each fuel pin. The calculated percent gas release values are listed in Table 5-3.

The fractional release values determined from the Kr-85 analysis are believed to be more accurate than values determined from the volume calculation because the relative magnitude of the values determined from the Kr-85 analysis were what would be expected based on the difference in burnup of the fuel in the two pins.

5.5 METALLOGRAPHIC EXAMINATION

The sectioning diagrams for each fuel pin are shown in Figures 5-9 and 5-10 along with photomicrographs of the as cut transverse sections. As can be seen in the macrographs a central void was present in each fuel pin which increased in diameter uniformly from the bottom to top of the fuel column.

Figure 5-6. Profilometer Measurements, Fuel Pin E1E

Figure 5-7. Pre- and Post-Irradiation Cladding Profiles of E1F Fuel Pin

Figure 5-8. Polar Profilometer Measurements, Capsule E1E

Figure 5-9. Sectioning Diagram of Capsule E1E Showing "As Cut" Transverse Sections

Figure 5-10. Sectioning Diagram of Capsule E1F Showing "As Cut" Transverse Sections

Table 5-3
PERCENT FISSION GAS RELEASE

Fuel Pin	E1E	E1F
Kr-85 Produced (atoms)	3.82×10^{18}	3.74×10^{18}
Kr-85 Released (atoms)	3.63×10^{18}	3.2×10^{18}
% Kr-85 Released	95.0	85.5
Calculated Volume of Fission Gas Formed (cc)	28.9	24.8
Calculated Volume of Fission Gas Released (cc)	24.2	23.7
% Gas Released Based on Gas Volumes	83.7	95.6
Burnup, MWd/Te	86,900	75,000

5.5.1 Examination of E1E Fuel

A composite photograph showing one radius of a cross section taken near the center of the fuel column of E1E is shown in Figure 5-11. The cross section shown in Figure 5-11 was prepared from the as-cut section identified as No. 38301 in Figure 5-9.

The most striking characteristic, which is not typical of low burnup mixed oxide fuel, was the presence of a ring of fine porosity around the fuel periphery adjacent to the cladding. The porosity extended inward from the outer radius of the fuel to a depth of ~ 0.012 inch. The fuel grain size in this region ranged in diameter from 1 to 3μ . Pores in this region of the fuel ranged in diameter from 3 to 5μ and pore densities were found to be as high as 20,000 pores/mm². Grain boundaries in this region of the fuel were readily attacked by a light etch (10% oxalic acid, electrolytic-6V for 2 sec).

Within the porous region, larger equiaxed grains which extended from ~ 0.012 inch to ~ 0.024 inch from the cladding i.d. were observed. A third region, ~ 0.020 inch wide, in which a transition from equiaxed to elongated grains occurred was observed inside the equiaxed grain region. Inside this region columnar grains extended to the central void.

The fuel-cladding gap, originally 0.003 inch (diametral), was observed to be completely closed. Part of the gap was filled with a grey phase ~ 0.002 inch thick, which was not observed to adhere preferentially to either the fuel or clad. Microprobe examination of similar material in the E1F fuel (see subsection 5.4.4) showed that it contained Fe, Ni, and Cr (cladding constituents); Pd and Mo were also detected. Cs was not detected, but because the sample was prepared in water it is believed it was removed prior to analysis.

Cladding attack was not observed in any of the micrographs.

5.5.2 Electron Microscopy of E1E Fuel

Replicas of the irradiated E1E fuel and an unirradiated control sample were prepared and examined with an electron microscope. Optical and electron micrographs of the unirradiated fuel are shown in Figure 5-12. Electron micrographs from the peripheral, equiaxed grain, and columnar grain regions of the E1E fuel are shown in Figure 5-13.

The photomicrograph of the unirradiated fuel (Figure 5-12) shows that the as-fabricated fuel consisted of equiaxed grains 20 to 40μ in diameter. Grey and white inclusions* were also observed in the fuel. A second phase was observed in the grain boundaries, especially at triple points. An electron micrograph of the second phase is shown in Figure 5-12. This phase stood out in relief indicating that it was either harder or more resistant to etching than the fuel matrix.

* Microprobe examination of similar phases in unirradiated fuel prepared by the same process as the E1E fuel has shown the white inclusions are stainless steel and the grey phase contains $Al(Al_2O_3)$, probably picked up in processing.

NOTE: ALL PHOTOS REDUCED TO 53%
FOR REPRODUCTION

9603-33-37 100X

AS-POLISHED

9603-68 500X
EDGE OF CENTRAL VOID, TYPICAL
(ETCHED WITH 10% OXALIC ACID,
ELECTROLYTICALLY AT 6 V FOR
6 SEC)

9603-40 500X
GRAIN SIZE TRANSITION REGION,
TYPICAL (AS-POLISHED)

9603-39 500X
INNER EDGE OF HIGH POROSITY
REGION, TYPICAL (AS-POLISHED)

9603-38 500X
FUEL-CLADDING INTERFACE,
TYPICAL (AS-POLISHED)

Figure 5-11. Radial Cross Section of Fuel Pin E1E Taken Near Center of Fuel Column

PHOTOMICROGRAPH OF UNIRRADIATED FUEL,
ETCHED 10 MINUTES WITH $10\text{HNO}_3 - 20\%\text{H}_2\text{O}_2 - 70\%\text{H}_2\text{O}$ (500X)

ELECTRON MICROGRAPH OF ROSETTE STRUCTURE
IN UNIRRADIATED FUEL (4500X)

(REDUCED TO 75% FOR REPRODUCTION)

ELECTRON MICROGRAPH OF SECOND PHASE
OBSERVED IN GRAIN BOUNDARY TRIPLE
POINTS OF UNIRRADIATED FUEL (12,000X)

ELECTRON MICROGRAPH OF OUTER PERIPHERY
OF FUEL IRRADIATED IN CAPSULE E1E (4500X)

Figure 5-12. Photomicrographs of Unirradiated and Irradiated Fuel From Capsule E1E

Figure 5-13. Electron Micrographs—E1E Fuel

An electron micrograph of the rosette structure visible in the optical photomicrograph in Figure 5-12 is also shown in Figure 5-13. It is hypothesized that the rosettes result from small composition gradients in the fuel, or from grain boundary migration.

The electron micrographs of the irradiated fuel showed:

1. At the fuel periphery the grains appeared to be fractured and second-phase material or voids were observed at subgrain boundaries. A similar effect was observed by Padden⁴ in highly irradiated UO_2 .
2. In the equiaxed grain region of the fuel, voids and second-phase particles were observed in the grain boundaries. Smaller voids and second-phase particles were observed inside the grains. The second-phase particles in the grain boundaries were elongated in the direction of the grain boundaries while those occurring inside grains were equiaxed.
3. In the columnar grain region of the fuel second-phase particles were larger than those observed in the equiaxed grain region. Pores in this region of the fuel were elongated along the radial direction of the fuel.

A comparison of the grain size of the irradiated and unirradiated fuel showed that the grain size of the as-fabricated fuel ($20\text{--}40\mu$) was ten to twenty times greater than that of the irradiated fuel near the fuel periphery ($1\text{--}2\mu$). Even more surprising was the fact that the grain size in the unirradiated fuel was larger than that observed in the equiaxed region of the irradiated fuel.

5.5.3 Examination of E1F Fuel

A composite photograph showing one radius of a cross section taken near the center of the E1F fuel column is shown in Figure 5-14. The cross section shown in Figure 5-14 was prepared from the as-cut section identified as No. 38487 in Figure 5-10.

The fuel structure observed is typical of oxide fuel which has melted. A region of very high density fuel was observed to extend inward from the molten boundary. Inside the high density region a restructured region was observed which extended to the central void. As observed in the E1E fuel, the fuel adjacent to the cladding contained a large amount of porosity in a zone extending inward ~ 0.012 inch from the cladding.

Evidence of fuel melting* was observed to 58% of the fuel diameter.

In the unmelted regions of the fuel, the distribution and appearance of fission products was very similar to that observed in the E1E fuel. However, in fuel which had melted a noticeable decrease in fission product concentrations was observed (see Figures 5-11 and 5-14). As discussed below, the decrease in the concentration of the metallic fission products in the melted region of the fuel is explained by the formation of the large ingot at the bottom of the fuel column.

A longitudinal section from the E1F fuel pin showing the fuel- UO_2 insulator interface at the bottom of the fuel column is shown in Figure 5-15.** The white inclusion is an ingot of fission products which were highly beta-gamma active. Because of the interest in the fission product distributions in highly irradiated fuel this fuel section was examined with a microprobe. The results of the microprobe examination are presented in the following subsection.

5.5.4 Microprobe Examination of E1F Fuel

The areas numbered from one through eight in Figure 5-15 were examined with an electron-microprobe to determine the relative amounts of fission products and impurities present in these regions. Results of the examination of each area are discussed below.

5.5.4.1 Fission Product Ingot (Areas 1 and 2)

The ingot in the center of the sample was examined near its center (area 1) and near the edge (area 2). The fluorescent x-ray emissions from the ingot were scanned using both a LiF and a $\text{PET}^\dagger$ crystal for elements from atomic number 11

* Defined as the limit of porosity at the outside diameter of the high density region observed in the fuel.

** The grey area filled with porosity which appears in the lower right portion of Figure 5-15 is mounting resin. Grinding of the fuel was discontinued before all of the resin was removed to ensure that the ingot was not lost.

† Pentaerythritol

NOTE: ALL PHOTOS REDUCED TO 53%
FOR REPRODUCTION

9753-19-23 100X
AS-POLISHED

9753-27 500X
EDGE OF CENTRAL
VOID (AS-POLISHED)

9753-29 250X
VOIDS IN HIGH DENSITY
BAND (AS-POLISHED)

9753-32 500X
INNER EDGE OF REGION
CONTAINING LARGE PORES
(ETCHED 2 MIN WITH 5% H_2SO_4)

9753-16 500X
FUEL-CLADDING INTERFACE
(AS-POLISHED)

Figure 5-14. Radial Cross Section of Fuel Pin E1F Taken Near Center of Fuel Column

A140-02

FUEL- UO_2
INSULATOR
INTERFACE

Figure 5-15. Longitudinal Cross Section of E1F Fuel Taken at Fuel - UO_2 Insulator Interface Near Bottom of Fuel Column

(sodium) to atomic number 94 (plutonium). The center of the ingot was found to consist of Mo, Ru, Tc, Rh, and a trace of Pd. Using the calibration curves of Bramman, et al.,⁵ these elements were found to be present in the ratio of Mo: Ru: Tc: Rh: = 0.85: 7.5: 2.3: 1.0. The edge of the ingot was found to be of similar composition with the ratios of the elements being Mo: Ru: Tc: Rh: = 0.80: 7.5: 2.2: 1.0. The average composition of the ingot expressed in percentage is given in Table 5-4.

Table 5-4
AVERAGE COMPOSITION OF
FISSION PRODUCT INGOT

Constituent	Concentration, %
Molybdenum	7
Ruthenium	65
Technetium	19
Rhodium	9
Palladium	Trace

5.5.4.2 Comparison of Fission Product Concentrations Across the Fuel Radius (Areas 3, 4 and 5).

Fission product concentrations were determined for comparison in the fuel near: (1) the ingot (area 3), (2) the cladding (area 4), and (3) in the high density region of the fuel adjacent to the molten boundary (area 5). An area approximately 150 microns square was swept with the electron beam in each location and the fluorescent x-rays of two fission products, Zr and Ru, were counted. These two fission products were selected as being representative of the oxide-forming and metallic inclusion-forming fission products, respectively.

A marked depletion of both Zr and Ru was found in the melted region of the fuel. The high density region (area 5) contained only 38% and the region adjacent to the ingot (area 3) only 74% as much Zr as the unmelted region near the cladding (area 4). The presence of Ru could not be detected in either the high density region or the central region of the fuel although it was found to be in the unmelted region of the fuel in concentrations comparable to Zr.

Grain boundaries in the melted central region of the fuel were examined in detail. The specimen current image of a grain boundary which was examined is shown in Figure 5-16. X-ray image scans of the grain boundary for Ba, Si, and Zr are also shown in Figure 5-16. Both Ba and Si were present in significant amounts. Only trace amounts of Zr were detected in the grain boundary.

5.5.4.3 Fuel-Cladding Interface (Area 6)

The fuel-cladding interface was examined in the region of area 6 for evidence of fuel-cladding reaction. No reaction was observed in the optical micrographs (Figure 5-14). X-ray pulse images taken for Pd, Mo, U, and Fe, Ni, and Cr are shown in Figure 5-17. No Cs was detected, presumably because it had been removed during aqueous preparation of the sample. Pd, Mo, and U were detected in both the fuel and cladding. Of the major cladding constituents only Ni was observed to have migrated into the fuel in the x-ray pulse image scans. Analyses for Fe, Ni, and Cr were made at several spots in the cladding and into the fuel. The results of the analyses are plotted in Figure 5-18. The migration of Fe and Ni into the fuel was slightly greater than that determined for Cr.

5.5.4.4 Dendritic Structure in the Melted Regions of the Fuel (Areas 7 and 8)

The photomicrograph shown in Figure 5-19 shows one of the areas (see area 7) typically observed adjacent to voids in the high density region of the fuel which melted. The spherical light grey globules are fuel. The dark grey material surrounding the white globules were found to contain Ba and other fission products along with Fe and Si. The medium grey dendrites were found to contain Si.

Figure 5-20 shows a typical area where a metallic globule of fission products was observed adjacent to a void (see area 8). The white inclusion was found to contain 17% Mo, 40% Ru, 31% Tc, and 12% Rh. The dark grey phase seen bridging the neck of the void was found to contain Fe, Ba, Zr, Si, Sr, and Cr.

Based on the examination of areas 7 and 8 it appears that both metallic and nonmetallic impurities are rejected into whatever voids (or grain boundaries) are available within the molten fuel as it solidifies.

1200X

SPECIMEN CURRENT IMAGE PICTURE OF GRAIN BOUNDARY IN MELTED REGION NEAR FUEL CENTER

1200X

BARIUM $L\alpha$ X-RAY PULSE IMAGE OF GRAIN BOUNDARY IN MELTED REGION NEAR FUEL CENTER

1200X

SILICON $L\alpha$ X-RAY PULSE IMAGE OF GRAIN BOUNDARY IN MELTED REGION NEAR FUEL CENTER

1200X

ZIRCONIUM $L\alpha$ X-RAY PULSE IMAGE OF GRAIN BOUNDARY IN MELTED REGION NEAR FUEL CENTER

Figure 5-16. Analysis of Impurities in Grain Boundaries of Melted Fuel Near Center of E1F Fuel

565X

SPECIMEN CURRENT IMAGE OF FUEL-CLADDING INTERFACE. FUEL IS DE-
LINEATED. CLADDING IS IN LOWER
RIGHT HALF OF PHOTOGRAPH.

565X

URANIUM Ma X-RAY PULSE IMAGE OF
FUEL-CLADDING INTERFACE.

565X

CHROMIUM Ka X-RAY PULSE IMAGE OF
FUEL-CLADDING INTERFACE.

565X

IRON Ka X-RAY PULSE IMAGE OF
FUEL-CLADDING INTERFACE.

565X

NICKEL Ka X-RAY PULSE IMAGE OF
FUEL-CLADDING INTERFACE.

565X

PALLADIUM La PULSE IMAGE OF
FUEL-CLADDING INTERFACE.

565X

MOLYBDENUM La PULSE IMAGE OF
FUEL-CLADDING INTERFACE.

Figure 5-17. Analysis of Fission Products and Cladding Constituents at Fuel-Cladding
Interface of Fuel Pin E1F

Figure 5-18. Analysis of Cladding Constituents in Fuel and Cladding at Fuel-Cladding Interface in Fuel Pin E1F

A140-4

1000X

Figure 5-19. Photomicrograph of Dendritic Phase Containing Non-Metallic Fission Products Observed in Melted Region of E1F Fuel

A140-05

500X

Figure 5-20. Photomicrograph of Void Observed in Melted Region of E1F Fuel Containing both Metallic and Non-Metallic Fission Products

6. RESULTS AND DISCUSSION

6.1 $\int kd\theta$ DETERMINATION

Following irradiation of capsule E1F in the VH-4 facility the integral conductivity from the fuel surface to the melting point (solidus) was calculated using the following expression:

$$\int_{T_s}^{T_r} kd\theta = \frac{Q \times PSF(r)}{4\pi}$$

where

T_r = temperature of fuel at radius of interest, °C

T_s = fuel surface temperature, °C

Q = heat generation, watts/cm

r = fuel radius, cm

PSF (r) = power shape factor at radius of interest

Calculated operating powers for the E1E and E1F fuel pins during their irradiation in the MTR reflector and during the 5-hour irradiation of capsule E1F in the VH-4 facility are listed in Table 6-1. The linear power value calculated for the E1F capsule includes a correction for fuel depletion which occurred during the initial irradiation in the reflector of the MTR.

Based on the calculated operating power of 31.2 ± 4.7 kW/ft* the $\int_{T_s}^{T_m} kd\theta$ for fuel pin E1F was calculated to be 42.8 ± 6.4 W/cm**. Using a fuel-cladding heat transfer coefficient of 1700 Btu/h-ft² °F as determined from the data of Craig, et al.,⁶ the fuel surface temperature during the 5-hour irradiation was calculated to be 1200°C, therefore

$$\int_{1200}^{T_m} kd\theta = 42.8 \pm 6.4 \text{ W/cm}$$

6.2 COMPARISON OF HIGH AND LOW BURNUP $\int kd\theta$ VALUES

The $\int_{T_s=1200}^{T_m} kd\theta$ for two low burnup mixed oxide, $(\text{Pu}_{0.2}\text{U}_{0.8})\text{O}_{2.00}$, fuel pins (designated E1C-2 and E1D-2) reported by Craig and Baily¹ are listed in Table 6-2 for comparison with the data for fuel pin E1F. Comparison of these values shows that the $\int_{T_s=1200}^{T_m} kd\theta$ calculated for the E1F fuel pin is greater than the values determined at low burnups; if the experimental error is considered, however, there is no difference between the $\int_{T_s}^{T_m} kd\theta$ determined from both the high and low burnup irradiations.

6.3 EFFECT OF MELTING ON DISTRIBUTION OF FISSION PRODUCTS

Shown in Figure 6-1 are macrographs and corresponding alpha and beta-gamma autoradiographs of the transverse cross sections taken at the middle of fuel pins E1E and E1F. Listed in Table 6-3 are the measured diameters for the porosity limit (molten boundary), columnar grain growth, and equiaxed grain growth determined from the photomicrographs of the fuel sections. Examination of the alpha and beta-gamma autoradiographs of the fuel section of E1E shows that a decrease in both alpha and beta-gamma activity occurred uniformly from the fuel surface to its center in the sample that did not melt. In the alpha autoradiographs of the section from fuel pin E1F the variation in alpha activity from the fuel center to surface was less than observed in the E1E fuel pin. A large decrease in the beta-gamma activity occurred in the center of the E1F fuel as indicated by the dark region seen in the beta-gamma radiograph. As can be seen by comparing the diameter measured for the

* The accuracy of the power calculated was determined to be $\pm 15.0\%$. This is the only uncertainty accounted for in the integral conductivity calculation.

** Calculation is shown in Appendix A.

porosity limit and that of the dark area observed in the beta-gamma radiograph listed in Table 6-3 they correspond very closely. A similar correspondence was observed by Craig and Bailly¹ in the beta-gamma radiographs of low burnup mixed oxide fuels which had undergone center melting. In the low burnup irradiations an area of high beta-gamma activity was observed in the restructured region of the fuel surrounding the central void. Based on the observation of the large ingot of beta-gamma active fission products observed at the bottom of the E1F fuel column, it is concluded that at high fuel burnups the fission product concentration in the molten region of the fuel was much greater than the solubility limit for these species in the fuel and they precipitated into an ingot. The depletion of fission products in the melted region was verified by microprobe analysis (see subsection 5.4.4).

6.4 EFFECT OF OPERATION WITH MOLTEN FUEL ON FUEL PIN PERFORMANCE

The E1F fuel pin operated for 5 hours with 58% of the fuel diameter molten after operating to a fuel burnup of 75,000 MWd/Te. The only effect which the high power irradiation was observed to have on the E1F fuel pin which might affect its performance was the diametral increase caused by thermal expansion of the fuel and the change in fuel volume which occurred on melting. The maximum diametral increase measured for the E1F fuel pin was 0.0033 inch which corresponds to a diametral strain of 1.32%.

Ductilities of fast ceramic reactor fuel claddings at high neutron fluences are presently unknown. However, the E1F irradiation indicates that if cladding ductility is sufficient to withstand diametral strains of 2% to 3%, then slow transients which result in significant fuel melting (up to 50% of the fuel diameter) will not have a catastrophic effect on fuel pins operating at peak linear powers expected in fast ceramic reactors at fuel burnups up to 75,000 MWd/Te.

Table 6-1
FUEL PIN OPERATING POWERS

Fuel Pin	E1E	E1F
Operating Power (kW/ft)		
Neodymium Burnup	18.5 ± 0.6	16.4 ± 0.5
Heavy Element Burnup	16.6 ± 0.5	16.0 ± 0.5
Flux Wire Analysis	20.7 ± 2.0	18.6 ± 1.7
End of Life (Physics Calculation)*	19.5 ± 2.9	17.4 ± 2.6
VH-4 Irradiation (Physics Calculation)*	-----	31.2 ± 4.7
Burnup, MWd/Te	86,900 ± 2,600	75,000 ± 2,300

* Takes into account decrease in power as a result of fissile atom depletion in the fuel.

Table 6-2
COMPARISON OF $\int_{1200}^{T_m} k d\theta$ VALUES

Fuel Pin	E1F	E1C-2	E1D-2
$\int_{1200}^{T_m} k d\theta$	42.8 $\pm$ 6.4	40.6 $\pm$ 6.1	36.8 $\pm$ 5.5
Linear Power, kW/ft	31.2	31.2	36.3
Temperature Clad o.d., °F	916	883	984
ΔT Clad, °F	195	174	200
Temperature Clad i.d., °F	1111	1057	1184
Surface Heat Flux, Btu/h-ft ²	1.83 X 10 ⁶	1.85 X 10 ⁶	2.15 X 10 ⁶
Gap Conductivity, Btu/h-ft ² - °F	1700	1850	2500
Temperature Fuel Surface, °C	1200	1125	1120
ϕ_c / ϕ_s , center to surface flux ratio	0.268	0.33	0.33
Burnup, MWd/Te	75,000		

Table 6-3
**DIAMETERS OF BOUNDARIES OBSERVED IN
PHOTOMICROGRAPHS AND AUTORADIOGRAPHS**

Fuel Pin	Diameter (in.)	
	E1E	E1F
Photomicrographs		
Porosity Limit (Melting)		0.58
Columnar Grain Growth	0.33	0.38
Equiaxed Grain Growth	0.40	0.39
Beta-Gamma Autoradiograph		
Dark Area		0.60

Figure 6-1. Macrographs, Alpha, and Beta Gamma Radiographs of Transverse Sections from Midplane of Pins E1E and E1F

7. CONCLUSIONS

Examination of high burnup mixed oxide fuel pins, one of which operated with center fuel melting during the last 5 hours of its irradiation, has shown that:

1. The $\int_{1200}^{T_m} kd\theta$ of stoichiometric mixed oxide, $(Pu_{0.2}U_{0.8})O_{2.00}$, after irradiation to a fuel burnup of 75,000 MWd/Te was calculated to be 42.8 ± 6.4 W/cm. Comparison of this experimentally determined value with experimental values for low burnup mixed oxide fuels¹ indicated no change in the $\int_{1200}^{T_m} kd\theta$ within the experimental error of the measurements.
2. Melting of high burnup mixed oxide fuel will result in the formation of ingots of the metallic fission products present in the melted region of the fuel. As a result of the precipitation of the metallic fission products from the molten region of the fuel the extent of melting in high burnup mixed oxide fuels may be determined from beta-gamma autoradiographs of fuel cross sections.
3. Mixed oxide fuel pins operating at peak linear powers expected in fast reactors with oxide fuel and at fuel burnup up to 75,000 MWd/Te could experience diametral strains of 2% to 3% for overpower operation resulting in melting up to 50% of the fuel diameter. The occurrence of cladding failure would depend on the remaining ductility of the cladding.

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APPENDIX A

CALCULATION OF $\int_{T_s}^{T_r} r \, kd\theta$

$$\int_{T_s}^{T_r} r \, kd\theta = \frac{Q \times \text{PSF}(r)}{4 \pi}$$

where

T_r = temperature of fuel at radius of interest, °C

T_s = fuel surface temperature, °C

Q = heat generation, W/cm

r = radius of fuel

$\text{PSF}(r)$ = power shape factor at radius of interest

During the irradiation in the VH-4 facility the power of capsule E1F was calculated to be 31.2 ± 4.7 kW/ft.

$$Q = (31.2 \pm 4.7 \text{ kW/ft}) (10^3 \text{ W/kW}) (1 \text{ in./2.54 cm}) (ft/12 \text{ in.})$$

$$= 1,023.6 \pm 154.2 \text{ W/cm}$$

$$\text{PSF}(r) = 0.525$$

$$\begin{aligned} \int_{T_s}^{T_r} r \, kd\theta &= \frac{(1,023.6 \pm 154.2 \text{ W/cm}) (0.525)}{4 \pi} \\ &= 42.8 \pm 6.4 \text{ W/cm} \end{aligned}$$

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