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POSTIRRADIATION EXAMINATION OF THORIA-URANIA FUEL RODS

— Quarterly Progress Report No. 5 —

June - August 1968

RESEARCH & DEVELOPMENT
DIVISION

NUCLEAR DEVELOPMENT
CENTER

Lynchburg, Virginia

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— Quarterly Progress Report No. 5 —

June - August 1968

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CONTENTS

	Page
1. INTRODUCTION	1
2. SUMMARY	2
3. ISOTOPIC ANALYSIS OF THORIA-URANIA FUEL	3
3.1. Experimental Measurements	3
3.2. Calculated Isotopic Abundances	3
4. METALLURGICAL ANALYSES	7
4.1. Profilometer Measurements	7
4.2. Metallographic Examination	9
4.3. Tensile Testing of the Borated Cladding	9
4.4. Examination of the Zircaloy Fuel Can	11

List of Tables

Table

1. Measured Isotopic Composition of Uranium Samples From Peak Burnup Region of Rods	5
2. Measured Isotopic Composition of Plutonium Samples From Peak Burnup Region of Rods	5
3. Comparison of Experimental and Calculated Uranium Isotopic Compositions	6
4. Comparison of Experimental and Calculated Plutonium Isotopic Compositions	6
5. Location of Profilometer Specimens	7
6. Tensile Testing of Stainless Steel Cladding	8
7. Tensile Testing of Zircaloy-2 Can	11
8. Effect of Irradiation on the Room-Temperature Tensile Properties of Zircaloy-2	12

List of Figures

Figure	Page
1. As-Polished Appearance of Fuel From Rod 105B - Center-of-Fuel to Cladding Traverse	13
2. As-Polished Appearance of Fuel From Rod 105E - Center-of-Fuel to Cladding Traverse	14
3. Tensile Specimen for Stainless Steel Cladding	15
4. Tensile Grips for Stainless Steel Cladding	16
5. Tensile Tests of Unirradiated Stainless Steel Cladding - Yield Strength After Annealing for 1 Hour at 1832 F	17
6. Tensile Tests of Unirradiated Stainless Steel Cladding - Tensile Strength After Annealing for 1 Hour at 1832 F	18
7. Tensile Tests of Unirradiated Stainless Steel Cladding - Uniform Elongation After Annealing for 1 Hour at 1832 F	19
8. Tensile Tests of Unirradiated Stainless Steel Cladding - Total Elongation After Annealing for 1 Hour at 1832 F	20
9. Tensile Tests of Unirradiated Stainless Steel Cladding - Yield Strength Before Annealing	21
10. Tensile Tests of Unirradiated Stainless Steel Cladding - Tensile Strength Before Annealing	22
11. Tensile Tests of Unirradiated Stainless Steel Cladding - Uniform Elongation Before Annealing	23
12. Tensile Tests of Unirradiated Stainless Steel Cladding - Total Elongation Before Annealing	24

1. INTRODUCTION

The objective of this program is to perform isotopic and metallurgical analyses on thoria-urania fuel, stainless steel cladding, and a Zircaloy fuel can taken from Core A of the Consolidated Edison Indian Point Reactor Number 1. The isotopic composition of the several actinide and fission product elements in the fuel will be determined. The fuel and the cladding will be examined metallographically. Specimens of the stainless steel cladding and the Zircaloy can (channel) will be tensile-tested under several conditions.

This report, the fifth in a series of quarterly progress reports on the program, covers the period from June 1 through August 31, 1968. The previous reports issued are as follows:

- BAW-3809-1 Progress Report No. 1
March - August 1967
- BAW-3809-2 Progress Report No. 2
September - November 1967
- BAW-3809-3 Progress Report No. 3
December 1967 - February 1968
- BAW-3809-4 Progress Report No. 4
March - May 1968

2. SUMMARY

Mass spectrometric determinations of the isotopic compositions of uranium and plutonium have been completed. Data for the peak burn-up zone of several rods are compared with the calculated abundances.

Profilometer measurements of the fuel cladding, metallographic examinations of the fuel and the cladding, and preparation of the tensile specimens were completed. Tensile testing of the unirradiated stainless steel specimens was completed.

3. ISOTOPIC ANALYSIS OF THORIA-URANIA FUEL

3.1. Experimental Measurements

Samples of the thoria-urania fuel from 13 fuel rods are being analyzed to determine the isotopic content of several actinide and fission product elements. Sample selection considerations and sample locations were described previously.¹ The samples were dissolved in a 13 M nitric-0.025M hydrofluoric acid mixture.² A sequential separation scheme was used to obtain uranium, plutonium, americium-curium, neodymium, promethium, and samarium.^{1,3}

This procedure employs an initial di-(2-ethylhexyl) phosphoric acid (D2EHP) extraction column to retain Pu(IV) and U(VI) from the 1 M HNO₃ influent. Uranyl ion is subsequently eluted from this column with 6 M HCl, and plutonium is then reduced and eluted as Pu(III) with HCl-HI. Some cross-contamination between the uranium and the plutonium fractions has been observed. This level of contamination is ordinarily of little consequence, but is a serious matter in these samples because of the high ²³⁸Pu content and the large ²³⁸U/²³⁸Pu ratio.

The possible isobaric interference from ²³⁸U was eliminated by performing an additional anion-exchange separation. The plutonium fraction in 8 M HNO₃ was loaded onto a column of Dowex 1-X2 (200-400 mesh, nitrate form). Any uranium contaminant was removed with 3 M HNO₃, after which plutonium was eluted with 0.6 M HNO₃.

The isotopic compositions of the uranium and plutonium fractions were determined mass-spectrometrically. The data for several of the rods are shown in Tables 1 and 2. All of these samples were taken from the peak burnup region of the rods.

3.2. Calculated Isotopic Abundances

The uranium and plutonium isotopic compositions were calculated for comparison with the experimental results. A brief description of

the reactor core and the location of the rods is presented in a previous report.¹ Radial power peaking factors for the fuel bundles had been calculated at core mid-life.⁴ For this study, it was necessary to generate peaking factors for the rods within the bundles. The rod peaking factors must reflect the softened spectrum seen by the rods on the outer row of the element adjacent to the water gap between elements. The rod peaking factors were calculated with the PDQ code⁵, and isotopic compositions were then calculated with the LIFE code.⁶

The LIFE code does not calculate ^{238}Pu . In order to obtain the ^{238}Pu content, it was assumed that all ^{230}Pu was produced from ^{235}U via ^{236}U and ^{237}Np . The cross sections and the neutron spectra were collapsed into one group, and a one-group calculation of ^{238}Pu was made.

The calculated and the measured isotopic compositions are compared in Table 3 for uranium and in Table 4 for plutonium. The uranium data are normalized to a value of 1.00 for ^{238}U , and plutonium data are normalized to a value of 1.00 for ^{239}Pu .

Table 1. Measured Isotopic Composition of Uranium
Samples From Peak Burnup Region of Rods

<u>Rod</u>	<u>^{233}U</u>	<u>^{234}U</u>	<u>^{235}U</u>	<u>^{236}U</u>	<u>^{238}U</u>
Preirradiation	0.0	1.0	93.3	0.3	5.4
20A	49.8	6.2	24.2	14.1	5.8
61A	43.1	4.9	33.7	12.5	5.8
62A	45.5	5.1	30.0	13.5	5.9
66A	47.4	5.7	27.3	13.8	5.8
105B	34.2	3.8	41.2	14.2	6.6
105C	11.6	1.3	74.7	6.2	6.2
105D	8.2	1.3	79.0	5.6	5.9
105E	6.5	1.2	81.1	5.1	6.1
106A	11.6	1.2	76.9	4.4	5.8
106B	11.4	1.2	78.3	4.4	5.7

Table 2. Measured Isotopic Composition of Plutonium
Samples From Peak Burnup Region of Rods

<u>Rod</u>	<u>^{238}Pu</u>	<u>^{239}Pu</u>	<u>^{240}Pu</u>	<u>^{241}Pu</u>	<u>^{242}Pu</u>
20A	40.5	34.0	11.7	9.5	4.3
61A	34.9	39.3	12.6	9.5	3.7
62A	36.8	36.5	12.7	10.2	3.8
66A	38.5	35.6	12.0	9.8	4.1
105C	6.9	76.0	10.6	6.0	0.5
105D	5.7	79.2	9.4	5.4	0.3
105E	5.1	80.6	9.0	5.0	0.3

Table 3. Comparison of Experimental and Calculated Uranium Isotopic Compositions

(All Values Normalized to $^{238}\text{U} = 1.00$)

Rod		^{233}U	^{234}U	^{235}U	^{236}U	^{238}U
20A	Experimental	8.65	1.08	4.19	2.45	1.00
	Calculated	8.07	0.80	6.13	2.23	1.00
61A	Experimental	7.40	0.84	5.80	2.14	1.00
	Calculated	8.07	0.80	6.13	2.23	1.00
62A	Experimental	7.64	0.86	5.05	2.26	1.00
	Calculated	7.84	0.75	6.45	2.17	1.00
66A	Experimental	8.15	0.98	4.70	2.38	1.00
	Calculated	7.84	0.75	6.45	2.17	1.00
105B	Experimental	5.22	0.57	6.37	2.18	1.00
	Calculated	5.10	0.49	7.96	1.96	1.00
105C	Experimental	1.87	0.21	12.1	1.00	1.00
	Calculated	2.48	0.26	13.3	1.03	1.00
105D	Experimental	1.40	0.22	13.4	0.96	1.00
	Calculated	1.54	0.22	13.8	0.89	1.00
105E	Experimental	1.07	0.20	13.3	0.85	1.00
	Calculated	1.33	0.21	13.9	0.90	1.00
106A	Experimental	2.02	0.21	13.4	0.77	1.00
	Calculated	2.30	0.23	14.1	0.67	1.00
106B	Experimental	2.02	0.22	13.8	0.78	1.00
	Calculated	1.63	0.22	14.3	0.65	1.00

Table 4. Comparison of Experimental and Calculated Plutonium Isotopic Compositions

(All Values Normalized to $^{239}\text{Pu} = 1.00$)

Rod		^{238}Pu	^{239}Pu	^{240}Pu	^{241}Pu	^{242}Pu
20A	Experimental	1.09	1.00	0.35	0.28	0.13
	Calculated	0.84	1.00	0.39	0.27	0.11
61A	Experimental	0.89	1.00	0.32	0.24	0.10
	Calculated	0.84	1.00	0.39	0.27	0.11
62A	Experimental	1.01	1.00	0.35	0.28	0.11
	Calculated	0.76	1.00	0.39	0.26	0.09
66A	Experimental	1.08	1.00	0.34	0.28	0.11
	Calculated	0.76	1.00	0.39	0.26	0.09
105C	Experimental		1.00	0.14	0.077	0.005
	Calculated	*	1.00	0.13	0.071	0.005
105D	Experimental		1.00	0.12	0.068	0.004
	Calculated	*	1.00	0.12	0.059	0.003
105E	Experimental		1.00	0.11	0.062	0.003
	Calculated	*	1.00	0.12	0.064	0.004

*Not calculated.

4. METALLURGICAL ANALYSES

Personnel assigned to the Metallurgical Analyses Task of this project will conduct detailed postirradiation examinations on several components from the first core of the Indian Point Reactor: ThO_2 - UO_2 fuels, borated type-304 stainless steel cladding, and a Zircaloy-2 fuel element can.

Efforts completed during this report period were profilometer measurements of the fuel cladding, metallographic examinations of the fuel and the cladding, and preparation of the tensile specimens. Tensile testing of the stainless steel and the Zircaloy specimens was initiated.

4.1. Profilometer Measurements

Profilometer measurements of sections from rods 61A and 105D₁ have been completed. The locations of specimens removed from all of the rods are shown in Table 5. No ridging at the pellet interfaces and no increases in rod diameter were observed in rods 61A and 105D₁. These results are consistent with those reported previously.⁷

Table 5. Location of Profilometer Specimens

Rod number	Estimated peak rod burnup, GWd/T	Specimen location		
		High	Average	Low
61A	24	1	1	1
105A	22	1	1	1
105C	15	1	0	0
105D ₁	17	1	0	0
106A	6	1	0	1

Table 6. Tensile Testing of Stainless Steel Cladding

Fluence	Test temperature, F										
	70		600		750	900	1050	1200		1300	1450
	BA	AA	BA	AA	AA	AA	AA	BA	AA	AA	AA
Peak	2	2	2	2	0	2	2	2	2	2	0
Average	2	2	2	2	0	0	0	2	2	0	0
Minimum	2	2	2	2	0	2	2	2	2	2	0
As-received	2	2	2	2	0	0	0	2	2	0	0
Aged 3000 hr	2	2	2	2	2	2	2	2	2	2	2
Aged 6000 hr	2	2	2	2	0	0	0	2	2	2	0

BA = before annealing
AA = after annealing

4.2. Metallographic Examination

Twenty-one specimens were examined metallographically to determine the extent of microstructural changes in the fuel, cladding, and Kannigen braze resulting from operation in the reactor.

During the quarter, an attempt was made to etch the $\text{ThO}_2\text{-UO}_2$ fuel to reveal the grain structure. Although a number of etching procedures were tried, none of the procedures proved to be satisfactory. Thus, as-polished specimens were used in the detailed examination of the $\text{ThO}_2\text{-UO}_2$ fuels. These examinations did not provide any information to supplement that reported in BAW-3809-4.⁷ Typical photomicrographs of the $\text{ThO}_2\text{-UO}_2$ fuels are shown in Figures 1 and 2. As shown in these figures, only very minor microstructural changes occurred during irradiation.

4.3. Tensile Testing of the Borated Cladding

The scope of the tensile testing program is shown in Table 6. As indicated in the table, some of the irradiated and some of the unirradiated specimens are to be annealed at 1832 F for 1 hour. The unirradiated specimens that are annealed will provide a base line for the irradiated specimens which will be annealed to remove the point defects.

Suitable testing grips and an extensometer have been developed. The details of the specimens and a photograph of the grips containing a specimen are shown in Figures 3 and 4, respectively. All of the tensile specimens have been prepared, and tensile testing of the unirradiated specimens has been completed. Three irradiated specimens were tensile-tested in a limited access area; however, excessive contamination of the area with ^{60}Co , presumably from the Kannigen braze, was encountered, so that all subsequent testing will be conducted in the hot cell. The necessary arrangements for transferring the tensile machine to the hot cells have been completed.

The results of the tensile tests conducted thus far are shown graphically in Figures 5 through 12. After annealing, the yield and tensile strengths (Figures 5 and 6) of as-received, 3000- and 6000-hr thermally aged specimens decrease as the temperature increases from 70 to 750 F. Little difference is observed in the yield strength of the specimens tested in the range from 750 to 1000 F. The yield strength, regardless of the

previous thermal history of the material, increased with increasing temperature from 1000 to 1300 F. The peak in the yield strength versus temperature curves is more pronounced for the 6000-hr thermally aged material than for the 3000-hr thermally aged or the as-received material. The normal softening trend, as indicated by the decrease in strength with increasing temperature, is regained above 1300 F, and at 1450 F (the maximum test temperature used in this program) the yield strength is approximately the same as that at 900 F. The ultimate tensile strength versus temperature curves for these materials do not show a peak. There is little difference in the tensile strength of material tested at temperatures from 750 to 1200 F.

The total elongation of the as-received, 3000- and 6000-hr thermally aged material (Figures 7 and 8) rapidly decreases as the test temperature increases from 70 to 600 F. At 600 F, the total elongation begins to increase with increasing temperature until a peak in the total elongation occurs at 850 F. The total elongation then decreases as the temperature increases from 850 to 1450 F.

Figures 9 through 12 represent the strength and ductility of as-received, 3000- and 6000-hr thermally aged materials that were not annealed. Since data for this material were obtained only at 70, 600, and 1200 F, insufficient data are available to draw curves similar to those in Figures 5 through 8.

The yield and tensile strengths of these materials decrease as the test temperature increases from 70 to 1200 F (the maximum test temperature used in testing this material). The total elongation decreases as the temperature increases from 70 to 600 F. From 600 to 1200 F, the total elongation increases with increasing temperature. It is interesting to note that the tensile strengths and total elongations of the as-received and thermally aged materials are identical at 1200 F.

From these results, it can be concluded that annealing (1 hr at 1832 F) of the unirradiated specimens before testing has essentially no effect on the strength or the ductility of the as-received or thermally aged cladding.

4.4. Examination of the Zircaloy Fuel Can

An outline of the tensile program is presented in Table 7. Preparation of all of the tensile specimens has been completed, and an extensometer has been developed. Room-temperature tensile testing was initiated before the tensile machine was installed in the hot cells. The results of the room temperature tests are presented in Table 8. The specimens that have been tested have received approximately one-half (1.5×10^{20}) and three-quarters (2.2×10^{21}) of the peak fast fluence. From the limited data now available, no definite conclusions can be drawn.

Metallographic examination of the Zircaloy specimens was initiated but not completed during this quarter.

Table 7. Tensile Testing of Zircaloy-2 Can

<u>Fluence</u>	<u>Test temperature, F</u>					
	<u>70</u>		<u>525</u>		<u>750</u>	
	<u>L</u>	<u>T</u>	<u>L</u>	<u>T</u>	<u>L</u>	<u>T</u>
Minimum	3	3	3	3	3	3
1/2 peak	3	3	3	3	3	3
3/4 peak	3	3	3	3	3	3
Peak	3	3	3	3	3	3

L = longitudinal
T = transverse

Table 8. Effect of Irradiation on the Room-Temperature Tensile Properties of Zircaloy-2

(E > 1 MeV)	Direction of specimen	0.2% yield strength, psi	Ultimate tensile strength, psi	Uniform elongation, %	Total elongation, %
1.5×10^{21}	Longitudinal	97,400	104,000	1.13	1.88
1.5×10^{21}	Longitudinal	101,500	105,700	0.56	0.81
1.5×10^{21}	Longitudinal	101,200	108,600	0.99	5.35
1.5×10^{21}	Transverse	100,100	105,900	1.29	2.59
1.5×10^{21}	Transverse	95,300	109,500	1.19	3.57
1.5×10^{21}	Transverse	95,600	102,800	0.89	5.62
1.5×10^{21}	Transverse	102,200	109,300	0.67	7.66
1.5×10^{21}	Transverse	91,300	96,600	0.81	7.73
2.2×10^{21}	Longitudinal	101,800	109,000	0.72	4.15
2.2×10^{21}	Longitudinal	102,500	114,800	0.59	4.39
2.2×10^{21}	Longitudinal	109,400	113,800	0.88	5.35
2.2×10^{21}	Transverse	99,000	104,800	0.81	6.49
2.2×10^{21}	Transverse	94,900	104,400	0.58	7.30
2.2×10^{21}	Transverse	99,800	101,500	0.40	4.32

Note: Irradiation temperature was 525 F.

Figure 3. Tensile Specimen for Stainless Steel Cladding

Split Tubular Specimen

Dimensions in Inches

Figure 4. Tensile Grips for Stainless Steel Cladding

Figure 1. As-Polished Appearance
of Fuel From Rod 105B -
Center-of-Fuel to Cladding
Traverse

Center of Fuel

Estimated Burnup - 30 GWd/T
Original Magnification - 250X
Reduction in
Reproduction Process - 29%

Cladding

Figure 2. As-Polished Appearance
of Fuel From Rod 105E -
Center-of-Fuel to Cladding
Traverse

Center of Fuel

Estimated Burnup - 30 GWd/T
Original Magnification - 250X
Reduction in
Reproduction Process - 22%

Cladding

Figure 5. Tensile Tests of Unirradiated Stainless Steel Cladding - Yield Strength After Annealing for 1 Hour at 1832 F

Figure 6. Tensile Tests of Unirradiated Stainless Steel
Cladding - Tensile Strength After Annealing
for 1 Hour at 1832 F

Figure 7. Tensile Tests of Unirradiated Stainless Steel Cladding - Uniform Elongation After Annealing for 1 Hour at 1832 F

Figure 8. Tensile Tests of Unirradiated Stainless Steel
Cladding - Total Elongation After Annealing
for 1 Hour at 1832 F

Figure 9. Tensile Tests of Unirradiated Stainless Steel Cladding - Yield Strength Before Annealing

Figure 10. Tensile Tests of Unirradiated Stainless Steel
Cladding - Tensile Strength Before Annealing

Figure 11. Tensile Tests of Unirradiated Stainless Steel
Cladding - Uniform Elongation Before Annealing

Figure 12. Tensile Tests of Unirradiated Stainless Steel Cladding - Total Elongation Before Annealing

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