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HOT-CELL EVALUATION OF THE GRIND-LEACH PROCESS. II. RECOVERY OF URANIUM AND THORIUM FROM IRRADIATED PYROLYTIC-CARBON-COATED SOL-GEL THORIA-URANIA PARTICLES

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OAK RIDGE NATIONAL LABORATORY
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ABSTRACT

Duplex-coated sol-gel ThO_2 and $(Th,U)O_2$ particles that had been irradiated to burnups of 15,000 and 17,000 Mwd/ton (Th + U) and had decayed for 10 and 19 months were ground to -100 mesh to ensure fracture of the kernels and the pyrolytic-carbon coatings, and then leached in boiling fluoride-catalyzed nitric acid to dissolve the thorium, uranium, and fission products. Recoveries of at least 99.6% of the uranium and 99.9% of the thorium were realized, and more than 86% of the gamma emitters were dissolved. These results contrast markedly with the uranium and thorium recoveries — as low as 95.4 and 93.1% respectively — that were obtained in earlier grind-leach studies with laminar-coated $(Th,U)C_2$ particles in a graphite matrix. The grind-leach process may, therefore, be applicable to HTGR fuels containing coated sol-gel oxide particles. However, the process may be of marginal value since the leached residue must be stored as high-level radioactive waste.

1. INTRODUCTION

Two principal methods have been proposed for the recovery of thorium and uranium from High Temperature Gas-Cooled Reactor (HTGR) fuel elements, which contain pyrolytic-carbon-coated $(Th,U)C_2$ or $(Th,U)O_2$ particles. When either fuel is processed to recover the thorium and uranium, the heavy metals must be separated from much larger quantities of pyrolytic carbon (PyC) and structural graphite. The proposed burn-leach process involves burning the carbon and graphite in air or oxygen to produce an "ash" of ThO_2-U_3O_8 and fission product oxides. This ash is dissolved in fluoride-catalyzed nitric acid, and the composition of the resulting solution is adjusted to that of a suitable feed for a conventional solvent extraction system. The proposed grind-leach process involves crushing the graphite matrix and fuel particles to expose the thorium- and uranium-containing kernels to the leaching action of nitric acid. The leach solution, after being filtered to remove the carbon, is processed by solvent extraction methods.

The hot-cell evaluation of the burn-leach process with irradiated prototype Peach Bottom (Th,U)C_2 fuel compacts that were supplied by General Atomic (a Division of the General Dynamics Corporation) indicated that the process had no major disadvantages.[1] Fission product volatility and losses of thorium and uranium to the leach residue were satisfactorily low, and the process appeared to be economically sound. However, in the hot-cell evaluation of the grind-leach process with Peach Bottom compacts and with experimental AVR fuel spheres,[2] the leached residues contained excessive amounts of thorium (6.9% of the total), uranium (4.5%), and fission products (up to 35%). These results completely contradicted those of the "cold" grind-leach experiments (i.e., with unirradiated prototype fuels), in which recoveries of the heavy metals were greater than 99.7%, and emphasized the importance of irradiation effects, such as thermal diffusion and fission product recoil, on subsequent processing of the fuel.

Investigations of irradiation effects in HTGR fuels began at ORNL several years ago. Early in 1962 the migration of uranium into the pyrolytic-carbon coatings of UC_2 particles was noted.[3] Later that year, Bomar and McClung reported a diffusion band of uranium in the laminar coatings of UC_2 particles after irradiation.[4] They succeeded in duplicating this effect by cyclically heating unirradiated particles from the same batch between 200 and 1160°C over a 2904-hr period.[5] Uranium from UC_2 particles migrated completely through an 84-μ laminar pyrolytic-carbon coating, probably along the lines of defects, in 94 hr at 1600°C.[6] Osborne <u>et al.</u>[7] found that all of the laminar coatings of particles from this batch had failed upon irradiation, and that the uranium had migrated out of the graphite matrix (that held the particles) into the surrounding graphite can. They also found that 30% of the fission product ^{137}Cs had migrated into the can.[7] Toner <u>et al</u>. reported that 45 to 52% of columnar pyrolytic-carbon coatings on UC_2 particles had cracked upon irradiation and that the UC_2 had reacted with the coatings.[8] In another experiment, the irradiation of

UC_2 particles having duplex pyrolytic-carbon coatings caused penetration of the inner laminar layer, with spearhead attacks. However, the outer columnar coatings of all but one or two particles were intact. The primary aim of these and other investigations[9-17] was to determine the integrity of the particles and coatings with regard to the retention of fission gases and fission products that might contaminate the gas coolant and/or reactor structure. In general, it was concluded that the effectiveness of the coatings on UC_2 and $(Th,U)C_2$ particles was in the order: triplex[11] > duplex > single laminar.[8,10] Finally, a SiC layer was incorporated into a triplex coating for maximum fission product retention up to about 2000°C.[17] (Fuel for the O.E.C.D. Dragon Project HTGR is of the last type.)

In 1965 the first postirradiation examinations of pyrolytic-carbon-coated sol-gel ThO_2 and $(Th,U)O_2$ particles were made at ORNL.[14] They indicated that the oxide kernels were superior to carbide kernels with respect to migration and release of fission products since there was no diffusion of the heavy metals from the former into the pyrolytic-carbon coatings. Further studies with the oxide-coated particles confirmed these conclusions.[15-17] Since the pyrolytic-carbon-coated sol-gel oxide particles may be used in future loadings of the AVR and as a "TARGET"-type fuel for the Public Service Company of Colorado reactor, being designed by General Atomic,[18] applicability of the grind-leach process to the treatment of pyrolytic-carbon-coated sol-gel oxide particle fuels was investigated.

D. R. Cuneo and H. E. Robertson, Reactor Chemistry Division, generously furnished the irradiated coated particles. O. L. Kirkland carried out the hot-cell manipulations. V. C. A. Vaughen made numerous suggestions concerning the experimental techniques. The groups of R. E. Eby, C. E. Lamb, and E. I. Wyatt, Analytical Chemistry Division, performed the analytical services.

2. EXPERIMENTAL

2.1 Fuel and Irradiation Data

The fuel used in these studies was irradiated in the Oak Ridge Research Reactor (ORR), in the form of loose particles in the upper, vented compartments of the Gas Cooled Reactor Program Loop 1, Experiments 1-14 and 1-15. The fuel in Experiment 1-14 consisted of a mixture of coated sol-gel ThO_2--8% UO_2 and sol-gel ThO_2 particles, both produced at ORNL. The fuel in Experiment 1-15 was composed of a mixture of pyrolytic-carbon-coated sol-gel ThO_2 and $(Th,U)O_2$, and $(Th,U)C_2$ particles (Table 1). The sol-gel oxide kernels for this experiment were prepared at ORNL, and the carbide kernels were obtained from General Atomic; all were coated by General Atomic.[14] Irradiation conditions are described in Table 2.

The burnup of the fuel used in Experiment 1-14 was estimated to be 2.7 at. % of the heavy metals; that is, about 44% of the ^{235}U was depleted by fission and capture.[14] Postirradiation examination of 134 of the Experiment 1-14 particles revealed no failures of, or damage to, the pyrolytic-carbon coatings.[15] [In another experiment, the inner coatings of 121 of the particles from the same batch (OR-205) showed vertical cracks, some of which extended to the interface with the outer coating; in general, however, the particles remained intact.][12]

The depletion of ^{235}U in the fuel in Experiment 1-15 was estimated to be about 27.5%. After irradiation the particles were roughly classified by Cuneo and Robertson into two size ranges, + 420 μ and -420 μ, by screening with a 40-mesh sieve. All of the $(Th,U)O_2$ particles (~542 μ) were retained by the screen, and most of the ThO_2 and $(Th,U)C_2$ particles (~400 μ) passed through; only nine of the small particles were found with 114 of the large particles after the sieving. Metallographic examination of 38 of the Batch GA-321-2 $(Th,U)C_2$ particles, 88 of the GA-324 ThO_2, and 114 of the GA-323 $(Th,U)O_2$ particles revealed no obvious or potential coating failures.[16] A Fibrous Filter Analyzer[19] in the loop coolant gas stream indicated that only gaseous fission products, primarily xenon and krypton, were released from the upper compartment during the irradiation.[16]

Table 1. Pyrolytic-Carbon-Coated Particle Mixtures[a] Used in GCR-ORR Loop 1 Experiments 14 and 15

Experiment No.	1 - 14		1 - 15		
Particle Type	ThO_2	$(Th,U)O_2$	ThO_2	$(Th,U)O_2$	$(Th,U)C_2$
Coating	Duplex[b]	Duplex[b]	BISO[c]	BISO[c]	BISO[c]
Particle Batch No.	OR-205	OR-206	GA-324	GA-323	GA-321-2
Wt, g	10.55	53.41	38.66	25.00	6.30
Wt, %	16.52	83.48	55.30	35.60	9.10
Th wt, g	3.57	19.65	19.02	13.60	1.12
U wt, g	-	1.65	-	1.08	1.08
^{235}U wt, g	-	1.54	-	1.00	1.00
Core Diameter, μ	243 ± 13	206 ± 20	233 ± 9	343 ± 23	181 ± 20
Coating Thickness, μ	128 ± 14	100 ± 5	89 ± 3	100 ± 6	97 ± 5
Particle Diameter, μ	500 ± 28	406 ± 27	412 ± 11	542 ± 23	375 ± 25

[a] Irradiated as loose particles in the upper, vented compartments of the Gas Cooled Reactor Program Loop 1.

[b] Inner coating of laminar PyC; outer coating of columnar PyC (ORNL).

[c] Inner coating of porous PyC; outer coating of isotropic PyC (GA).

Table 2. Irradiation Data for GCR-ORR Loop Experiments[12-16]

Experiment No.	1 - 14	1 - 15
Position in Reactor	ORR-B1	ORR-B1
Date Inserted	July 30, 1964	July 2, 1965
Date Removed	May 2, 1965	April 1, 1966
Total Hours at Power	5183	4920
Thermal Flux	4.9×10^{13}	3.4×10^{13}
Capsule Temperature, °C		
Center	1288-1370	1400 (max)
Sleeve	982-1066	1010 (max)
Outer Tube	593-732	455 (max)

Cuneo reported,[18] on the basis of small (~ 100-mg) samples, the following isotopic analyses (expressed as %) for the particles:

		Irradiated	
	Unirradiated	Loop 1-14	Loop 1-15
^{233}U	0.0	14.94	12.52
^{234}U	1.006	1.59	1.58
^{235}U	91.77	68.58	66.03[a]
^{236}U	0.485	7.40	8.07
^{238}U	6.74	7.49	11.80[a]

[a] *Apparently contaminated by natural uranium.*

2.2 Equipment and Procedures

Techniques that had been developed during previous $(Th,U)C_2$ grind-leach studies[2] were used in the grind-leach experiments reported here. The fuel particles were fractured in a modified Waring food blender and then passed through a 100-mesh (149-μ) sieve to ensure the fracture of all the coatings and kernels. The Loop 1-14 sample was riffled to obtain two approximately equal portions. The +420-μ- and -420-μ-diam particle samples from Loop 1-15 were ground and sieved separately. Each portion of fuel was leached three times (total contact time, 9 hr) in boiling 13 M HNO_3--0.05 M HF--0.1 $Al(NO_3)_3$, and washed with water several times after each leach. In order to minimize cross-contamination from external sources, the leaching and washing were done in special Erlenmeyer flasks that were equipped with reflux condensers and M-porosity fritted-glass filters. The final wash from the last leach was sampled separately to determine whether washing was complete. The leached carbon residues were finally dried with acetone, weighed, and poured directly into clean sample bottles. Aliquots of the residue were burned in oxygen, and the ash was dissolved in a minimal volume of fluoride-catalyzed nitric acid in order to determine the quantities of unleached thorium and uranium with the highest degree of accuracy possible. The solution resulting from the "wet combustion" of the carbon in a concentrated sulfuric-nitric acid mixture was analyzed for fission products. The amounts of thorium and uranium in the liquid samples were determined by colorimetric, coulometric, or fluorometric methods. Radiochemical analyses were made by multichannel gamma spectrometry, followed by computer analysis of each spectrum.[20]

3. RESULTS AND DISCUSSION

Table 3 shows the results from one of two duplicate experiments in which we leached -100-mesh $(Th,U)O_2$ particle fragments (from Loop 1-14) that had been irradiated and had been allowed to decay for 19 months. The residues contained an average of 1.97% of the gross gamma emitters, 3.33% of the ^{137}Cs, 1.05% of the ^{106}Ru, 1.06% of the ^{144}Ce, 0.25% of the uranium, and 0.04% of the thorium. Our

Table 3. Results Obtained by Leaching Ground Pyrolytic-Carbon-Coated $(Th,U)O_2$ Particles with Fluoride-Catalyzed Nitric Acid[a]

		Uranium		Thorium		Gross Gamma	
Sample Description	Amount (ml or g)	mg	% of Total Found	mg	% of Total Found	counts/min	% of Total Found
Fuel Sample	13.4						
1st Leach + Washes	154	363.8	99.35	4509.8	99.66	34.8×10^{10}	96.14
2nd Leach + Washes	115	1.28	0.35	13.80	0.30	0.48×10^{10}	1.32
3rd Leach + Washes	94	0.01	<0.01	0.91	0.02	0.09×10^{10}	0.25
Final Wash	25	<0.01	<0.01	<0.01	<0.01	$<10^{6}$	<0.01
Residue	7.0	1.07	0.29	0.68	0.02	0.83×10^{10}	2.29

[a] Irradiated to a burnup of ~ 17,500 Mwd/metric ton (Th + U); decayed 19 months.

uranium mass spectroscopic analyses for the entire 29.5 g of particles showed 16.83% ^{233}U, 1.83% ^{234}U, 66.08% ^{235}U, 8.81% ^{236}U, and 6.45% ^{238}U. Cuneo calculated from our data that the burnup of heavy metal atoms was 3.04% (2.88% from fission of ^{235}U and 0.16% from ^{233}U). This value is higher than the burnup (2.7%) that was calculated on the basis of Cuneo's analyses.[21] The Th:U ratio was 12.4:1, compared with 11.9:1 prior to irradiation. We calculated the burnup (by ^{137}Cs counting) to be about 17,500 Mwd/ton (Th + U).

The 540-μ-diam (Th,U)O_2 and the 400-μ-diam (Th,U)C_2 and ThO_2 particles (from Loop 1-15), which had decayed 10 months, were leached separately. The carbon residue from the (Th,U)O_2 particles contained 0.04% of the uranium, 0.07% of the thorium, and 14.1% of the gamma emitters (Table 4). The larger amounts of fission products in the leached residue of Loop 1-15, compared with those in the 1-14 residue, were primarily due to the shorter decay period and the resulting higher concentration of the relatively insoluble ^{95}Nb. (It was interesting to note that the ^{95}Zr:^{95}Nb atom ratio was 12:1 in the leach solution and 1:8 in the residue.) The mass spectrometric analysis for the uranium in the solution from the first leach of the mixed oxide particles showed 13.67% ^{233}U, 1.68% ^{234}U, 69.51% ^{235}U, 8.48% ^{236}U, and 6.65% ^{238}U. We calculated the burnup (by ^{137}Cs counting) to be about 15,000 Mwd/metric ton (Th + U).

The smaller-diameter (~ 400-μ-diam) particles from Loop 1-15, a mixture of ThO_2 and (Th,U)C_2, were used to test a proposed method for processing thorium breeder reactor fuels. The finely ground particles were first leached with a nitric acid solution in an effort to selectively dissolve the reactive (Th,U)C_2 away from the nitric acid-insoluble ThO_2, that is, to dissolve the fissile fuel away from the fertile blanket. The exposed ThO_2 was then leached twice with nitric acid containing fluoride. Essentially all the uranium and thorium was recovered in the three leaches; only 0.4% and 0.1%, respectively, remained in the pyrolytic-carbon residue (Table 5). The first leach apparently did preferentially dissolve the (Th,U)C_2, since 426 mg of thorium and 535 mg of uranium were recovered out of a calculated 445 and 427 mg, respectively, in the sample. The Th:U ratio was 0.80, compared with the preirradiation ratio of 1.04.

Table 4. Results Obtained by Leaching[a] <100-Mesh PyC-Coated $(Th,U)O_2$ Particles[b]

	Distribution, % of Total		
	First Leach Solution	Second and Third Leach Solutions	Residue
Uranium	98.9	0.08	0.04
Thorium	99.7	0.23	0.07
Gross Gamma	74.2	11.7	14.1
^{95}Zr	94.5	-	5.5
^{95}Nb	46.1	27.4	26.5
^{106}Ru	86.2	9.1	4.7
^{137}Cs	92.6	0.30	7.1
^{144}Ce	91.9	0.17	8.0

[a] Boiling 13 M HNO_3--0.05 M HF--0.1 M $Al(NO_3)_3$; first leach was for 5 hr, and second and third leaches were for 2 hr each.

[b] Irradiated to a burnup of 15,000 Mwd/ton (Th + U); decayed 10 months.

Table 5. Leaching of Heavy Metals and Fission Products from <100-Mesh PyC-Coated ThO_2 and $(Th,U)C_2$ Particles[a]

	Distribution, % of Total		
	First Leach[b] Solution	Second and Third Leach[c] Solutions	Residue
Uranium	83.8	15.8	0.4
Thorium	6.0	94.0	0.1
Gross Gamma	28.3	10.4	61.3
^{95}Zr	0.3	40.0	59.7
^{95}Nb	0.01	3.4	96.6
^{106}Ru	0.01	33.0	67.1
^{137}Cs	0.1	81.1	18.8
^{144}Ce	0.2	79.1	20.8

[a] Irradiated to a burnup of 15,000 Mwd/ton (Th + U); decayed 10 months.

[b] 5 hr with 13 M HNO_3.

[c] Each leach (2 hr) with 13 M HNO_3--0.05 M HF--0.1 M $Al(NO_3)_3$.

The second and third leaches of the ThO_2 dissolved 6630 mg of thorium and 101 mg of uranium. As before, the residue contained a major fraction of the fission products - in this case, 61.3% of the gamma emitters (principally ^{95}Zr-Nb, ^{106}Ru, ^{137}Cs, ^{144}Ce, and ^{125}Sb). The higher retention, as compared with that obtained in tests with $(Th,U)O_2$ (Table 4), is probably due to migration of the $(Th,U)C_2$ particles into the PyC-coatings. This phenomenon was observed in earlier tests with coated carbide HTGR fuels.[2]

These few experiments indicate that the grind-leach process can be applied to the reprocessing of pyrolytic-carbon-coated sol-gel oxide fuel particles. From the reprocessing viewpoint, our data support the postirradiation metallographic and microradiographic studies and the fission product release (annealing) experiments that showed coated oxide particles to be superior to coated carbide particles.[16,17] In addition to the smaller losses of uranium and thorium, fission product retention in the residue from processing the oxide particles was about a factor of 3 lower than in the residue from the carbide particles. For example, the residue from the coated ThO_2 and $(Th,U)C_2$ particles contained (on the basis of specific activity per unit weight of residue) five times as much ruthenium, three times as much zirconium, and four times as much niobium as the residue from the coated $(Th,U)O_2$ particles. However, it contained only about one-half as much cesium and one-fourth as much cerium as the oxide residue. Apparently, in the former case, cesium and cerium migrated* through the pyrolytic carbon into the outer container during irradiation, and thus possibly were not present in the carbide-core particles in significant amounts prior to leaching. This hypothesis is confirmed by the fact that the oxide-core particles contained 6.8 times as much cesium and 6.4 times as much cerium per gram of (Th + U) as the carbide-core particles, whereas the ratios of the other fission products (with the exception of zirconium) were about 1. Recently reported data by Cuneo showed a significant

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*J. G. Morgan and his group at ORNL mechanically separated the outer shells of triplex-coated UC_2 particles, irradiated at 1370°C to 24 at. % burnup, and found that 14% of the total cesium, 17% of the cerium, and lesser amounts of other fission products had migrated to that location.[12]

quantity of $^{134,137}Cs$ in the graphite support sleeve that surrounded the Loop 1-15 particles.[21] The amount appeared to be a function of the temperature of the graphite sleeve.

Although the grind-leach process can be applied successfully to the treatment of HTGR fuels containing carbon-coated sol-gel oxide particles, the economics of the process may not be attractive owing to the large retention of fission products by the leached carbon residue. Even with long-decayed fuel, the residues would have to be treated as intermediate- to high-level solid wastes, and their storage would add significantly to the processing costs.[22]

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