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PLANT-SCALE ANODIC DISSOLUTION OF
UNIRRADIATED IFR FUEL PINS*

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ABSTRACT

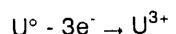
Anodic dissolution is a major operation in the pyrometallurgical process for recycling spent metal fuels from the Integral Fast Reactor (IFR), an advanced reactor design developed at Argonne National Laboratory. This process involves electrorefining the heavy metals (uranium and plutonium) from chopped, steel-clad fuel segments. The heavy metals are electrotransported from anodic dissolution baskets to solid and liquid cathodes in a molten salt electrolyte ($\text{LiCl}-\text{KCl}$) at 500°C . Uranium is recovered on a solid cathode mandrel, while a uranium-plutonium mixture is recovered in a liquid cadmium cathode. The anode configuration consists of four baskets mounted on an anode shaft. These baskets provide parallel circuits in the electrolyte and salt flow through the chopped fuel bed as the baskets are rotated. The baskets for the engineering-scale tests were sized to contain up to 2.5 kg of heavy metal. Anodic dissolution of 10 kg batches of chopped, steel-clad simulated fuel (U-10% Zr and U-Zr-Fs alloy) was demonstrated.

I. INTRODUCTION

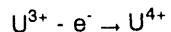
The Integral Fast Reactor (IFR) is an advanced reactor concept proposed and developed by Argonne National Laboratory. The reactor¹ employs a metallic fuel, an alloy of U, Pu, and Zr that is clad with stainless steel. A key element in the IFR concept is the pyrometallurgical process, which processes the spent fuels²⁻⁴ by electrorefining with a molten salt electrolyte ($\text{LiCl}-\text{KCl}-\text{UCl}_3/\text{PuCl}_3$) at 500°C . Spent-fuel pins are chopped and put in an anodic basket for dissolution in the electrorefiner. Essentially pure uranium is electrotransported to a solid cathode; a mixed U-Pu product is electrotransported to a liquid cadmium cathode.

In the anodic dissolution step, sufficient voltage is used to oxidize the heavy metal pins while leaving the

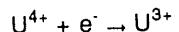
stainless steel cladding intact. Normal oxidation is expressed by



The second oxidation,



occurs at the surfaces of the metal baskets and cladding hulls. The reaction



occurs at the cathode, but results in no fuel dissolution unless the ions can come into contact with the fuel in the anode. When the U^{4+} ion comes in contact with the fuel in the anode,



occurs with resultant fuel dissolution.

After dissolution of the spent fuel, fission products and actinides distribute between the molten salt and liquid cadmium phases in the electrorefiner. The active fission products (such as alkali metals, alkaline earth metals, and rare earth metals) preferentially enter the salt phase and form chlorides. Noble metals prefer the cadmium phase. The actinides, depending on the oxidation state of the system, distribute to both the salt and cadmium phases.

Two methods of fuel dissolution in an electrorefiner were investigated: (1) direct dissolution in the cadmium anode of the cell and (2) anodic dissolution in the cell electrolyte. Previous studies of direct dissolution have been reported.⁵ This process requires a second step to electrotransport the heavy metal from the cadmium anode to a cathode. Another disadvantage of direct dissolution is that when fuel is dissolved from the stainless steel cladding (type D-9), nickel may be leached from the stainless steel surface. Nickel is rather

soluble in cadmium (5 wt % at 500°C), and leaching from austenitic stainless steel has been shown. The presence of dissolved nickel greatly reduces the solubility of uranium in cadmium. For instance, if nickel is added to a cadmium solution to make 0.35 wt % nickel in cadmium (at 500°C), then the uranium solubility is reduced from 2.34 to about 1.0 wt %. The dissolution of fuel in cadmium is dependent on the concentration of the driving force, which is the saturation concentration of uranium minus the uranium concentration at the dissolving fuel surface. When the saturation concentration is lowered, (e.g., by the presence of nickel), the available driving force for dissolution is lowered, as a result, dissolution time is increased. In anodic dissolution, whereby the heavy metal in the anodic dissolution baskets is electrotransported directly to the cathode, the dissolution rate is not dependent on solubility effects. Consequently, this method was chosen.

Tests with single, uranium-zirconium fuel pins demonstrated that uranium can be anodically dissolved. In one such test, a single clad pin weighing 10.1 g was attached to a mild steel cylinder, placed in a horizontal position with both faces exposed, and held there by two set screws. The results from the test were as follows:

1. The 10.1-g pin was completely dissolved. Neither the mild steel cladding nor the mild steel cylinder, which held the clad pin, was attacked.
2. The calculated coulombic efficiency, based on the weight loss (7.4 g) and the total current (A-h) passed, was 50%.
3. The cell voltage during the entire operation at 150 mA (~300 mA/cm²) was ~0.95 V (IR included).

Tests were run in which multiple pins were held loosely in a single, cylindrical anode basket and were anodically dissolved. The anodic dissolution basket for one of these tests was a Type 304 stainless steel beaker 8.4 cm in diameter with 0.32-cm-dia. holes over the top 5.1 cm of the beaker. Steel clad uranium-10 wt % zirconium alloy (0.50-cm nominal dia., 1.809 kg uranium) chopped into 0.74-cm-long pieces was loaded into the anodic baskets. All of the uranium was dissolved with an average current of about 100 A and a coulombic efficiency of 44%. These results showed the need for better electrolyte flow through the chopped-fuel pin bed, and for higher dissolution currents to meet the uranium dissolution rate required in the IFR processing of spent fuel.

II. DESIGN OF ANODIC DISSOLVER

Improved electrolyte flow through the chopped-fuel pin bed and a higher uranium dissolution rate (higher current operation) can be achieved with a prismatic-basket design that consists of multiple baskets hanging

from a common anode shaft. The anodic dissolution basket and arrangement shown in Figs. 1 and 2, respectively, meet the following design requirements:

1. Parallel baskets that contain the clad, chopped fuel pins are attached to a central bus bar above the electrolyte surface.
2. Design of the prism-shaped baskets and design of the bus current collector (including current collection at the shaft that supports the baskets) incorporates open parallel faces to allow good circulation of electrolyte through the pin bed and sufficient current collector to reduce resistive voltage losses. The baskets can be rotated and positioned to promote chemical attack of the uranium in the pins by the reduction of U⁴⁺, which is believed to form electrochemically on the basket surface. By this design, the anodic dissolution device would capitalize on electrochemical, as well as chemical, dissolution of the heavy metal.
3. The anodic dissolution device was designed so that it can be raised above the electrolyte after the dissolution operation and rotated at about 250 rpm. This spin-drying operation was expected to remove nearly all of the salt from the baskets, after which the cladding hulls should be easily removed from them. Thus, the anodic dissolution device should be reusable, and waste handling associated with the cladding should be easier without concern for salt and the basket hardware.

Figure 1 is a detailed schematic of the anodic dissolution baskets designed to be used in tests to demonstrate anodic dissolution of about 10 kg of chopped, clad U-10 wt % Zr pins. A picture of the assembled anodic dissolution device that includes three of the four baskets used in the tests is shown in Fig. 2. Two of the baskets contained a center current collector and two baskets did not. These baskets were constructed of Type 304 stainless steel.

III. EXPERIMENTAL RESULTS

A. Materials Testing.

Type 316 stainless steel, Ni-200, and molybdenum were tested as potential materials for the anode basket. The experimental procedure was as follows: the metal was weighed, attached to an extension rod, and lowered into a salt phase containing LiCl-KCl eutectic with ~5 wt % UCl₃. The metal was anodized at selected currents for periods up to 7 1/2 hours. At the end of each time period, the test specimen was removed, disconnected from the extension rod, and weighed. This procedure was repeated several times until either a preset cutoff voltage was reached or a

measurable weight loss was obtained. During the entire testing period, salt and cadmium phases were mixed with a separate stirrer to minimize mass transfer limitations. The ohmic drop during operation was measured by placing the cell on open circuit for 10 sec every 10 minutes during anodization. The operating temperature was in the range of 500-510°C.

The experimental results are summarized in Table 1. The results indicate that only molybdenum lost no weight before reaching the preset voltage cutoff (1.98 V). No weight loss was found for Type 316 stainless steel below a 1.3 V cutoff. The results indicate that iron, nickel, austenitic stainless steel, molybdenum, and iron-molybdenum alloys are good candidate materials for the anode baskets.

Figure 1. Schematic of Anodic Dissolution Baskets for Engineering-Scale Electrorefiner (CMT/IFR Drawing No. 1003)

Figure 2. Photograph of Anodic Dissolution Basket Assembly (Three of four baskets are shown) (ANL Neg. No. 5634 Frame 6)

Table 1. Summary of Results for Anodization Experiments

Material	Current, mA	Current Density, ^a mA/cm ²	Test Time, h	Measured Wt. Loss, ^b mg	Voltage Range During Test, V
Ni-200 ^c	50	39	5	0	-1.26 to -1.46
	200	159	4	74	-1.21 to -1.46
	400	312	7	445	-1.28 to -1.55
Mo ^d	100	450	6-1/2	0	-0.8 to -1.98
	50	20	7-1/2	0	-0.95 to -1.17
	300	120	1/2	N/M ^e	-1.23 to -1.98
Type 316 SS ^f	100	20	6	0	-1.15 to -1.21
	200	40	4-2/3	0	-1.14 to -1.34
	400	80	7	7	-1.37 to -1.53
	500	100	6	400	-1.25 to -1.44

^aCurrent density is calculated from depth in salt layer and current used.

^bWeight loss could be due to either M(Ni, Mo, Fe, Cr) + UCl₃ \rightarrow MCl_x + U or from UCl₄ + M \rightarrow MCl_x + U. We cannot tell from our results.

^cRod was ~4-in.-long, 1/16-in.-dia.

^dHollow tube ~3-1/2-in. long, 1/8-in. dia.

^eN/M = not measured -- no visible attack.

^fSolid rod ~4-in. long, 1/4-in. dia.

III. ENGINEERING-SCALE DEMONSTRATION

An engineering-scale electrorefining facility⁴ (that can handle deposits as large as 10 kg of uranium on a single solid cathode is shown in Fig. 3. This facility was constructed to demonstrate the electrolytic transport of uranium at plant-scale levels and to measure the dissolution rate of clad segments of unirradiated uranium fuel pins. In the anodic dissolution tests with this electrorefiner, the cathode was either the cadmium pool in the bottom of the electrorefiner vessel, a solid mandrel cathode, or a liquid cadmium cathode. Pins of steel-clad unirradiated U-10% Zr and U-Zr-Fs alloy (84.7% U-10.4% Zr-2.4% Mo-2.0% Ru-0.3% Rh-0.2% Pd) were chopped into segments and dissolved using the anodic dissolution baskets.

Fig. 3. Electrorefining Cell

A. Anodic Dissolution of Uranium-Zirconium Alloy.

Approximately 200 kg of depleted uranium has been anodically dissolved in the engineering-scale electrorefiner. Results of two of the tests (AD-8 and AD-9) with chopped, steel-clad depleted uranium - 10 wt % zirconium alloy are described below. These results are typical of all of the tests with depleted uranium and uranium-zirconium alloy.

Table 2 shows the fuel loaded in the anodic dissolution baskets at the start of Runs AD-8 and AD-9. The 12.003 kg and 14.519 kg of U-Zr alloy and cladding used in Runs AD-8 and AD-9, respectively, contained 9.182 kg and 11.108 kg uranium, respectively. These chopped fuel segments occupied 75 to 88% of the full height of each basket. Perforated plate covers were attached to the baskets to prevent spillage of pin segments during the spin-drying operation, which is used at the end of the anodic dissolution test to remove salt from the baskets. Typical operating conditions for the anodic dissolution tests are given in Table 3.

Table 2. Uranium Loading in Anodic Dissolution Experiments

Run No.	Basket No. ^a	Height of Chopped Pin Bed, cm	Initial Weight, kg	
			U-10 wt % Zr Alloy and Cladding ^b	Uranium
AD-8	3	19.7	3.004	2.298
	4	17.8	3.004	2.298
	1	19.7	3.004	2.298
	2	18.1	2.991 (12.003) ^c	2.288 (9.182) ^c
AD-9	1	22.2	3.452	2.641
	2	22.2	2.682	2.817
	3	22.2	3.704	2.833
	4	22.2	3.681 (14.519) ^c	2.817 (11.108) ^c

^aBaskets 1 and 3 contained a center current collector.

^bThe diameter of the uranium-zirconium rods was nominally 0.196 in. and the rods were clad with 316 stainless steel.

^cTotal weight of the above material in baskets 1, 2, 3, and 4.

Table 3. Operating Conditions for the Anodic Dissolution Tests

Temperature	500°C
LiCl-KCl- UCl_3 Electrolyte	(1 to 2 mol % UCl_3)
Anodic Dissolver Rotation Speed	20 to 75 rpm
Salt Mixer Rotation Speed	150 rpm
Cadmium Mixer Rotation Speed	50 to 100 rpm
Dissolution Current	200 to 300 A
Cutoff Voltage	1.0 to 1.3 V

The uranium dissolution rates for Runs AD-8 and AD-9 are shown in Table 4. No attack on the stainless steel cladding and baskets was found after these runs. All of the U-Zr alloy was removed from the baskets in these runs. In Run AD-8, a dissolution rate of 0.5 kg U/h was sustained for the initial 32% of the uranium that was dissolved, 0.4 U/h for the next 46%, and 0.2 kg U/h or less for the final 22% of the uranium that was dissolved. In Run AD-9, a dissolution rate of 0.4 kg U/h was sustained for the initial 96% of the uranium that was dissolved. The overall anodic efficiency (ratio of the measured weight of uranium dissolved to the theoretical weight) that would be dissolved based on the ampere-hours used during the dissolution) was about 50%.

After dissolution of the uranium, the baskets were raised above the salt and rotated at about 250 rpm. This spin-drying operation removed nearly all of the salt from the baskets. The cladding hulls from one of the baskets were picked at random, removed from the glovebox, and washed with deionized water. This solution was filtered, and the filtrate and small amounts of fines on the filter were analyzed for uranium. Results are shown in Table 5. The amount of material retained by four baskets was about 61 g. The amount of uranium in this material was 2.0 g, which is only 0.02% of the fuel weight fed to the basket as feed material. The cladding hulls were easily removed from the baskets. Voltage and current measurements were also made to determine the effect of the number of parallel baskets on the amount of current that could be passed through the cell. Figure 4 shows that the apparent cell resistance obtained during electrochemical transfer of uranium from the anodic dissolution basket to the cadmium pool decreased from 16 mΩ to 6 mΩ as the number of parallel baskets was increased from 1 to 4. This result is consistent with earlier polarization measurements made using multiple parallel rods.

Figure 4. Effect of Parallel Anodes on the Apparent Cell Resistance

Table 4. Results from Anodic Dissolution Tests

Run No.	Chopped-Pin Bed Depth, cm	Dissolution Period, h	wt % of U-10 Zr		Uranium Dissolution Rate, kg/h	Anodic Efficiency, %
			Beginning	End		
AD-8	17.8-19.7	6	100	67.7	0.5	69
		11	67.7	21.6	0.4	51
		7	21.6	7.5	0.2	55
		7	7.5	0	0.1 ^a	50
AD-9	22.2	26.75	100	3.6	0.4	52
		2.2	3.6	0	0.2 ^a	50

^aThe dissolution rate may have been higher than the value shown, which is the lowest possible rate of dissolution.

Table 5. Salt and Solids Retained in Anodic Dissolution Baskets

No. of Baskets	Weight of Cladding Hulls, g		Weight of Indicated Component in Salt Washed from Cladding Hulls, g			
	Before Washing	After Washing	LiCl KCl- UCl ₃	Zr	Fe	U
1	503	488	15.2 ^a	0.06 0.008 ^b	0.05 0.01 ^b	0.002 ^b
4	2012 ^c	-	61 ^{c,d}	0.3 ^c	0.2 ^c	0.01 ^c

^aThis salt contained 0.5 g uranium.

^bWeight of element found in 0.02 g solid fines from washings.

^cEstimate based on measurements for one basket.

^dThis salt contained 2.0 g uranium.

B. Anodic Dissolution of Uranium-Zirconium-Fissium Alloy.

Uranium-zirconium alloy was used in the anodic dissolution tests described in the previous section of this paper. Two anodic dissolution tests (Runs 40A and 42) were completed with uranium-zirconium-fissium alloy. In Run 40A, uranium was electrotransported from the anodic baskets to the cadmium pool in the electrorefiner. This uranium was later electrotransported to a solid cathode. In Run 42, the uranium was electrotransported directly to a solid cathode from the anodic dissolution baskets.

1. Run 40A. The purpose of Run 40A was to determine the behavior of noble metals in uranium-zirconium-fissium alloy (U-Zr-Fs)^a during anodic dissolution of chopped, steel-clad pin segments of this alloy. The uranium in the pins was electrotransported to the cadmium pool.

The anodic dissolution baskets were loaded with 13.496 kg chopped, steel-clad U-Zr-Fs alloy rods (1.97 kg cladding, 9.85 kg uranium, 1.16 kg zirconium, and 0.51 kg fissium). The baskets were rotated in the electrorefiner at 75 rpm. The salt and cadmium mixers were turned off. After a dissolution time of 25.9 h (6621.5 Ah), the baskets were rotated at 250 rpm in the gas phase above the salt. After this spin-drying operation, the baskets were weighed to determine the quantity of uranium dissolved. The weight of material transferred from the anodic baskets during the dissolution period was 9.03 kg. Since the weight of uranium, zirconium, and fissium initially loaded in the baskets was 11.52 kg, some of the components of the U-Zr-Fs alloy remained in the cladding hulls in the baskets. This was confirmed by visual examination of the pin segments. Several pin segments were submitted for chemical analysis of the residue.

The average concentration of the material left in the four pin segments removed from the top and bottom of two of the anodic baskets is provided in Table 6. An estimate of the material remaining in the baskets (as a percent of the starting material) is given in Table 7. This data indicates that nearly all (99.7 wt %) of the uranium was dissolved from the pins. Most of the material retained in the pins was salt (68.4 wt %). Minor constituents of the material retained in the pins included molybdenum (9.0 wt %), ruthenium (6.8 wt %), zirconium (6.1 wt %), rhodium (1.0 wt %), and palladium (0.2 wt %).

Table 6. Chemical Analysis of Residue in Dissolved U-Zr-Fs Pin Segments from Run 40A

Material	Concentration, wt %
Uranium	5.9
Zirconium	6.1
Cadmium	0.4
Molybdenum	9.0
Iron	1.7
Ruthenium	6.8
Rhodium	1.0
Palladium	0.2
Salt	68.4

Table 7. Estimate of Material^a Remaining in Baskets After Anodic Dissolution Run 40A

Metal	Percentage of Starting Material, wt %
Uranium	0.3
Zirconium	13.0
Molybdenum	80.9
Ruthenium	74.0
Rhodium	77.4
Palladium	29.3

^aSalt concentration was 68.4 wt %. Assumed that pin segments analyzed are representative of all dissolved materials in basket.

2. Run 42. In the second anodic dissolution of U-Zr-Fs alloy, the results from Run 42 were essentially as described above for Run 40A. The residue remaining in the cladding hulls consisted of two parts, a metallic phase and a salt phase. Samples of the residue were taken for further analysis of the metallic part of the residue. The salt was separated from the metallic phase, which was then analyzed by XRD (X-ray diffraction) and SEM/EDX (scanning electron microscopy). XRD results are given in Table 8. SEM/EDX indicated that Zr, Mo, Ru, and Rh were possible present in the samples.

^aFs stands for a mixture of noble metal fission products with the following approximate composition for U-Fs alloy: 95 wt % U, 2.5 wt % Mo, 2.0 wt % Ru, 0.26 wt % Rh, 0.19 wt % Pd, and 0.1 wt % Zr.

Table 8. X-ray Diffraction Analysis of Metallic Residue Remaining in Hulls after Anodic Dissolution in Run 42

Sample	Results	
1	X-Ray Major:	"Mo-Zr" type pattern, Ru (solid solution probable).
	Very minor:	ZrO_2 (fc cubic form)
	Trace:	possible Rh
2	X-Ray "Mo-Zr" type pattern and Ru (solid solution probable)	

C. Discussion of Results.

Previous studies of synthetic U-Fs and U-Pu-Fs alloys have shown that the noble metal fission products form several intermetallic phases, some of which contain U and Pu. The studies of U-Fs alloys revealed three intermetallic phases (in addition to the various U phases): U_2Ru , delta, and $ZrRu$. The delta phase was believed to be a Mo-U phase, probably MoU_2 . As the zirconium content of the alloy was increased above that of the fissium, the $ZrRu$ phase was formed in place of the U_2Ru phase.

Studies of U-Pu-Fs alloys revealed four intermetallic phases of interest: gamma prime, theta, U_2Ru , and $ZrRu$. Again, when the zirconium content was increased above that contained in fissium, the $ZrRu$ phase was formed in place of the U_2Ru phase. The gamma prime phase is apparently similar to the delta phase in the U-Fs system: it is a U-Pu-Mo phase. The theta phase is a U-Pu-Pd phase. In both studies, there was some indication that other constituents of the alloy may be soluble in the intermetallic phases.

IV. CONCLUSIONS

The following conclusions were drawn from the anodic dissolution tests:

1. The multiple-basket anodic dissolver can dissolve 10 kg of uranium-zirconium alloy (chopped, steel-clad, and unclad pin segments of U-10 wt % Zr) in about 27 hours.
2. All of the uranium-zirconium alloy can be removed from the baskets with an ampere-hour efficiency of about 50%.
3. The salt-removal operation with the anodic dissolution device removes nearly all of the salt from the baskets and cladding hulls after dissolution of uranium-zirconium alloy; thus, the cladding hulls are easily removed from the baskets and the baskets are reusable.

4. No corrosion occurred on either the 304 stainless steel baskets or the 316 stainless steel cladding hulls up to a cutoff voltage of 1.3 V.

5. The following anodic dissolution operating conditions were established when all of the uranium loaded in the anodic baskets was dissolved: 50% coulombic efficiency (664 Ah/kg uranium), stepwise decreasing current from 300 to 100 A, open-circuit voltage of 0.5 V, and an increase in the cell resistance from 4 to 9 m Ω .

The following conclusions were drawn from the anodic dissolution tests with uranium-zirconium-fissium alloy:

1. The residue left in the pin shells (cladding segments) after anodic dissolution is not the undissolved original pins. The residue is about 65 wt % salt plus noble metals and zirconium. The metallic portion is essentially free of heavy metal.
2. For the fissium alloy (5% used here), about 20% of the original Zr in the pin remains associated with noble metals Ru, Mo, and Rh in the metallic residue. The remainder of the Zr, along with the heavy metal, is removed during the anodic process.
3. Our estimate indicates that essentially all of the Ru, Mo, and Rh in the original pin is associated with Zr in the metallic part of the residue after anodic dissolution. We also estimate that only about 35% of the original palladium in the pin remains in the residue after dissolution, indicating that it may behave different from the other noble metals.
4. The electrochemical behavior of uranium was the same in U-Zr-Fs and U-10 wt % Zr in the anodic dissolution process.

V. ACKNOWLEDGMENTS

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