

**Experimental Observations on
The Role of The Cadmium Pool in Mark-IV ER**

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ABSTRACT

Argonne National Laboratory (ANL) has developed and demonstrated an electrometallurgical process for the Department of Energy (DOE) to treat sodium bonded spent nuclear fuel. One of the key steps in the demonstration was electrorefining the spent fuel in a molten LiCl-KCl- UCl_3 /liquid cadmium system using a pilot scale electrorefiner (Mark-IV ER). This article summarizes experimental observations and engineering aspects for the roles of the liquid cadmium during electrorefining spent fuel in the Mark-IV ER. It was found that the liquid cadmium pool acted as an intermediate electrode during the electrorefining process. The cadmium level was gradually decreased due to its high vapor pressure and vaporization rate at the ER operational temperature. The low cadmium level caused the anode assembly to electrically short with the ER vessel hardware, which resulted in the difficulties to determine the endpoint of uranium dissolution from the anode baskets and reducing the current efficiency. A reflux cadmium vapor trap was installed and has successfully prevented the cadmium level from decreasing.

1. INTRODUCTION

The need for treatment of spent nuclear fuel has increased substantially during the last decade. Several methods for treatment of spent nuclear fuel are being investigated throughout the nuclear industry [1-8]. Argonne National Laboratory (ANL) has developed and demonstrated an electrometallurgical process for the Department of Energy (DOE) to treat sodium bonded spent nuclear fuel. The demonstration of the process has been performed on irradiated fuel elements from the Experimental Breeder Reactor (EBR-II) [9]. One of the key steps in the demonstration is electrorefining the spent fuel in a molten LiCl-KCl- UCl_3 /liquid cadmium system using a pilot scale electrorefiner (Mark-IV ER). The Mark-IV ER is located in the Fuel Conditioning Facility at the ANL-West site in Idaho.

This report summarizes experimental observations on roles of the liquid cadmium pool during electrorefining spent EBR-II fuel in the ER, which include: the use of the cadmium pool as an indicating electrode for uranium concentration, the effect of cadmium vapor trap operation on cadmium level, cadmium sample analysis results, and cadmium pool acting as an

intermediate electrode during a direct transport process in the ER. The results are important for understanding the electrochemical processing of the spent nuclear fuel, since the Department of Energy (DOE) is determining whether to apply this technology to treat the remaining EBR-II and/or other spent nuclear fuels [9].

1.1. Equipment Description

The major components of the Mark-IV electrorefiner (ER) are illustrated in Fig. 1. The ER vessel is made of steel (2.25 Cr-1 Mo) with an inside diameter of 1.0 m and height of 1.0 m. The vessel contains an approximately 10 cm bottom layer of molten cadmium and an approximately 32 cm top layer of molten LiCl-KCl eutectic containing approximately 10 wt% of UCl_3 . The ER operating temperature is typically 500 °C; however some experiments were conducted at 450 °C. A cadmium stirrer, rotating at 18-22 rpm, is used to continually mix the cadmium. The salt is stirred/mixed during the electrorefining process by the rotating cathode and anode assemblies. Four ports (25.4 cm diameter) on the top of the ER are available for inserting anode and cathode assemblies. Inside the ER vessel, steel side shapers are mounted on the inside walls adjacent to the cathode ports. The side shapers constrain the growth of the rotating uranium deposit to a maximum diameter of approximately 25 cm, ensuring the deposit can be easily removed. There are also two beryllia bottom shapers mounted on the inside bottom of the ER in the cathode ports. The function of the bottom shapers is to prevent the growth of the uranium deposit from contacting the cadmium pool. Both the side and bottom shapers can dislodge dendrites from the cathode deposit as it rotates during the electrorefining process.

1.2 Process Description

The spent driver fuel consists of uranium, zirconium, bond sodium, and fission products, all contained within a stainless steel cladding [10]. Prior to electrorefining, the fuel pins are removed from their respective assembly, chopped into 0.64 cm long segments, and placed into rectangular, perforated, fuel dissolution baskets (FDBs) [11]. Four FDBs are then placed in a cruciform arrangement, loaded onto an electrode assembly, and inserted into the electrolyte of the ER as the anode. Each set of four FDBs contains the segments from two chopped spent fuel assemblies (122 fuel pins). Two spent

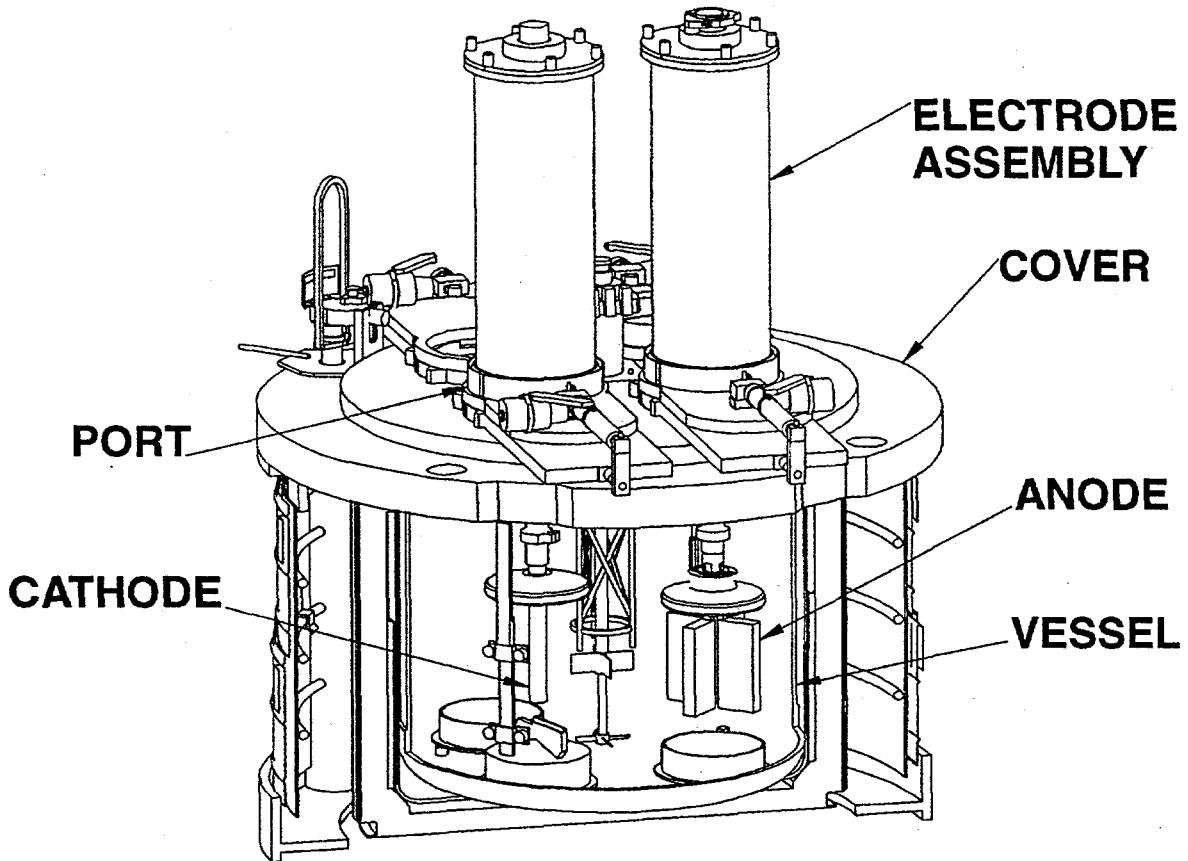


Figure 1. Schematic of FCF Mk-IV Electrorefiner. The front two ports are occupied. Support rings for the bottom scrapers are also shown. The salt mixer has been removed; only the cadmium mixer (thin blades) exists at present.

fuel assemblies contain nominally 8.1 kg uranium, 1.1 kg zirconium, and other fuel components [10].

The cathode is a 6.67 cm diameter, mild steel mandrel, with an active length of approximately 23 cm in the electrolyte. The three different electrorefining configurations employed are listed in Table 1.

Table 1. Cell Configurations Used in the Mark IV Electrorefiner.

Cell Configurations		ANODE	
		FDBs	Cadmium Pool
CATHODE	Steel Mandrel	Direct Transport (DT)	Deposition (DP)
	Cadmium Pool	Anodic Dissolution (AD)	Trivial

The primary purpose of electrorefining is to separate uranium from the other fuel components. Once the FDBs loaded with chopped fuel segments are inserted in the molten salt electrolyte, the bond sodium and active metal fission products chemically react and displace UCl_3 from the molten salt. Uranium in the fuel segment is electrochemically dissolved from the FDBs and deposited onto the cathode. Zirconium and noble metal fission products are ideally retained in the cladding hulls. The uranium metal dislodged by the side and bottom scrapers during the electrorefining dissolves in the cadmium pool and can be recovered using the deposition configuration.

2. EXPERIMENTAL OBSERVATIONS ON ROLES OF CADMIUM POOL IN MARK-IV ER OPERATIONS

2.1. Use of the Cadmium Pool as an Indicating Electrode for Dissolved Uranium Metal

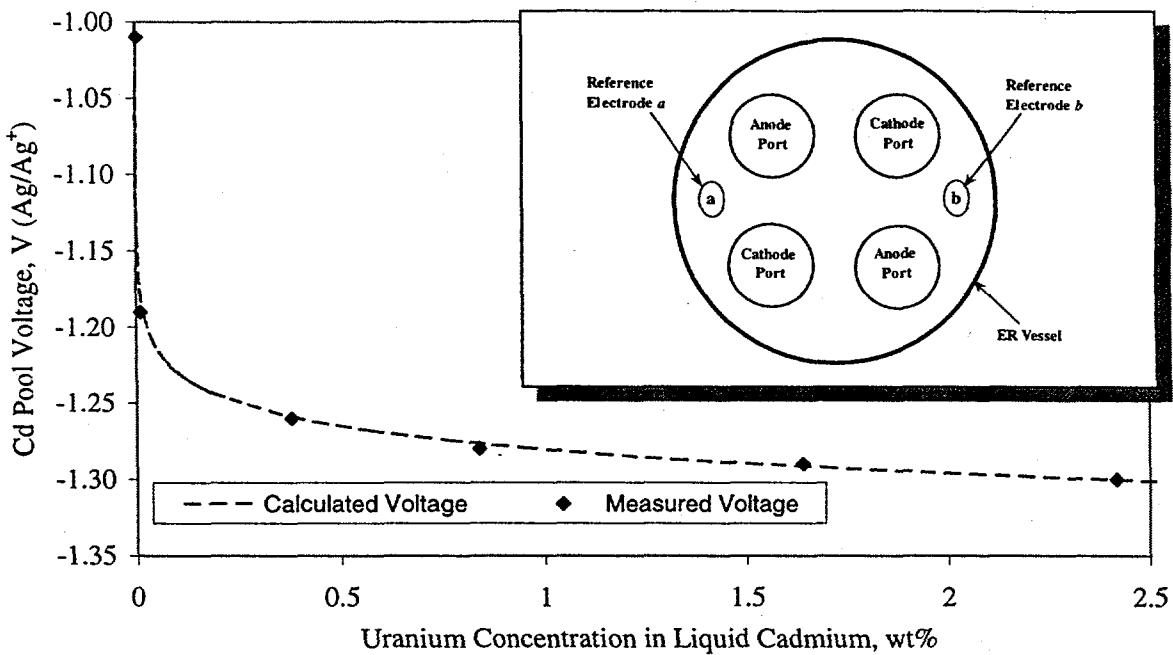


Figure 2. Response of Cadmium Pool as an indicator electrode for dissolved uranium. Inset: schematic of plan view for the Mark-IV Electrorefiner.

There are two Ag/AgCl reference electrodes installed in the ER salt phase as shown in the inset of Fig. 2.

The voltage differences between the reference electrodes and the ER vessel or anode/cathode can be measured during the electrorefining processes. The measured voltage difference between the reference electrodes and ER vessel represents the voltage difference between the reference electrodes and cadmium/salt interface. Since the concentration of UCl₃ in the eutectic is kept relatively constant at approximately 10 wt%, the variations in the reference electrode readings actually reflect the variations of uranium concentration in the molten cadmium. The standard Nernst Equation can be used to calculate the reference electrode voltage versus uranium concentration in the molten cadmium.

In the ER system, the predominant electrode reaction at cadmium/salt interface at equilibrium is:



By the Nernst Equation, the measured reference electrode reading represents:

$$\Delta V_{Cd-ref} = E_{U^{3+}/U}^0 + \frac{RT}{3F} \ln \frac{a_{U^{3+}}}{a_{U_{(Cd)}}} - (E_{Ag^+/Ag}^0 + \frac{RT}{F} \ln a_{Ag^+}) \quad (2)$$

$$\Delta V_{Cd-ref} = (E_{U^{3+}/U}^0 - E_{Ag^+/Ag}^0 - \frac{RT}{F} \ln a_{Ag^+} + \frac{RT}{3F} \ln a_{U^{3+}}) - \frac{RT}{3F} \ln a_{U_{(Cd)}} \quad (3)$$

where $a_{U^{3+}}$ is the U³⁺ activity in the salt phase, a_{Ag^+} the Ag⁺ activity in the reference electrode, and $a_{U_{(Cd)}}$ the uranium activity in the liquid cadmium. Since the items in the parentheses are constant at a given temperature, equation (3) can be re-written as:

$$\Delta V_{Cd-ref} = C - \frac{RT}{3F} \ln a_{U_{(Cd)}} \quad (4)$$

where C is constant at the ER operational temperature.

The solubility of uranium metal in cadmium at 500°C is 1.124×10^{-2} mole percent [12]. The activity of uranium in the molten cadmium at saturation is unity [12]. Therefore, the constant C can be determined experimentally by measuring the reference electrode voltage when the cadmium pool is saturated with uranium. The measured value of C at uranium saturation is -1.30 V.

When the uranium concentration in the molten cadmium is below saturation, the reference electrode reading will be:

$$\Delta V_{Cd-ref} = -1.3 - \frac{RT}{3F} \ln a_{U_{(Cd)}} \quad (5)$$

The ΔV_{Cd-ref} determined by equation (5) has been used as the equilibrium voltage or open circuit voltage at the salt/cadmium interface for the electrorefiner. Fig. 2 shows the measured ΔV_{Cd-ref} versus the uranium concentration in the

cadmium pool. The ΔV_{Cd-ref} calculated by the Equation 5 is also plotted in the figure for comparison. It can be seen that the measured ΔV_{Cd-ref} agrees well with the predicted values.

2.2. Cadmium Levels in Mark-IV ER

The Mark-IV ER was loaded with 431 kg of cadmium in December 1994. The measured cadmium level after the loading was 9.86 centimeters. Fig. 3 provides a plot of the measured and predicted cadmium levels in the Mark-IV electrorefiner.

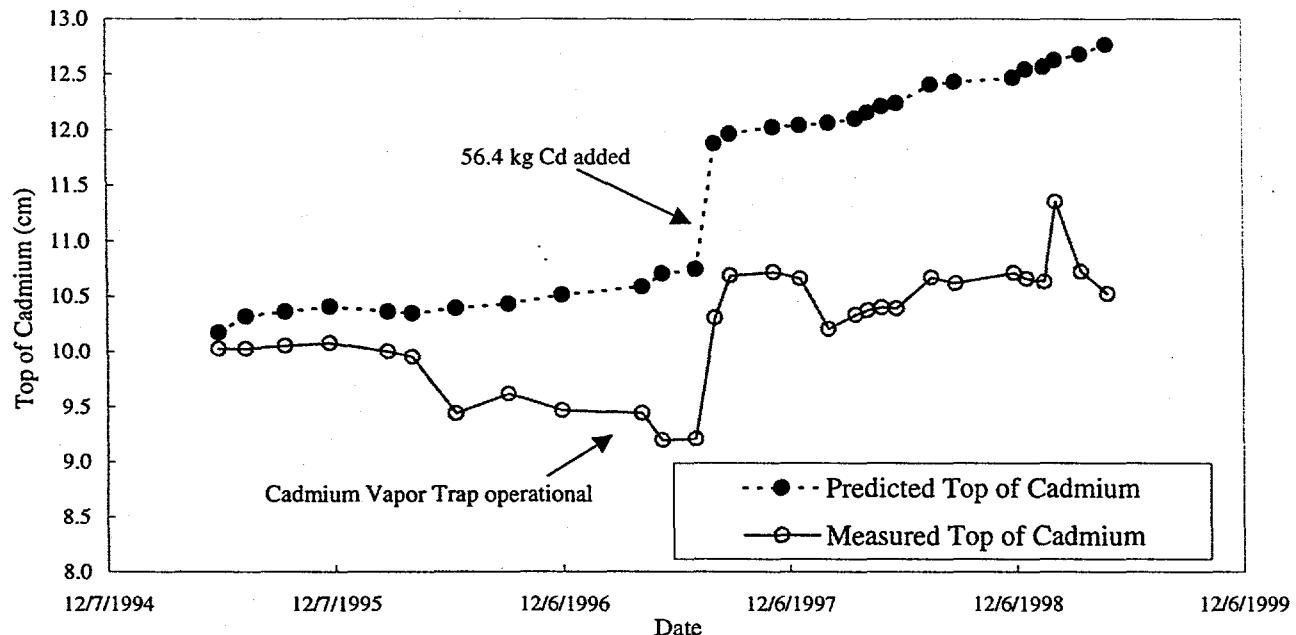


Figure 3. Measured and Predicted Cadmium Levels in Mark-IV Electrorefiner

Predicted cadmium levels were determined from the predicted cadmium mass, the predicted cadmium density, and the volume calibration equations [13]. The predicted cadmium mass was from the electrorefiner inventory, the measured electrorefiner input and output streams, and electrorefiner process modeling.

In Fig. 3, the measured cadmium level was relatively constant at 10 centimeters until June 1996, when it dropped approximately 5 millimeters (about a 27 kg loss of material in the cadmium pool). The predicted cadmium level did not show a similar decrease in level. In fact, the measured cadmium levels, though fluctuating, have been constantly lower than the predicted values. In July 1997, 56 kg of cadmium was added to the ER to raise the cadmium level (Fig. 3). The effect of the cadmium level on the electrorefining process is discussed in the Section 3.

2.3. Effect of Cadmium Vapor Trap Operations on Cadmium Level in ER

It is believed that one of the major causes for the observed decreases in cadmium level is the high vapor pressure and high vaporization rate of cadmium at 500°C, and the relatively cool temperatures above the molten salt. Cadmium

vapor could migrate through the salt phase [14] and deposit on the metal surfaces in the ER cover gas space, which made it difficult to disassemble components. To solve the problem, a reflux vapor trap (RVT) was also designed and installed to the ER to mitigate the cadmium vapor deposition on cool surfaces in the electrorefiner. The detailed RVT operations have been reported elsewhere [15]. It can be seen in Fig. 3 that although the measured cadmium level varies, the subsequent measurements after installing the cadmium vapor trap show that the level has stabilized compared to its initial decline.

At present, no conclusions have been drawn for the drop in the measured cadmium level between December 1997 and June 1998, for which the predicted cadmium level did not show a similar change. The sharp increase in the measured cadmium level between December 1998 and June 1999 was probably a level measurement error.

3. EXPERIMENTAL OBSERVATIONS ON ROLES OF CADMIUM POOL DURING ELECTROREFINING PROCESS IN THE ER

3.1. Cadmium Pool as Secondary Electrode for Uranium Recovery

Considerable experience gained during electrolytic deposition of metals from high temperature molten salts shows that cathode deposits have a basically dendritic or powdery structure. Dendritic growth of the deposits has been observed by several investigators during the study of electrorefining of uranium [16-18]. Therefore, one of the major purposes of keeping a cadmium layer in the ER is to collect the uranium dendrites which are scraped/dropped from the cathode during electrorefining. The dropped uranium will dissolve in the cadmium pool and can be recovered using the deposition configuration of Table 1. The experimental results and

statistical analysis for the deposition runs performed in the Mark-IV ER will be published in a separate report.

3.2. Cadmium Pool Acts as an Intermediate Electrode During Direct Transport Process

When using the direct transport configuration (Table 1) in the ER, a dc current was applied between the anode (FDBs) and cathode (steel mandrel). It, however, has been noticed that the differential voltage readings of the two reference electrodes appeared to behave independently for direct transport (Fig. 4).

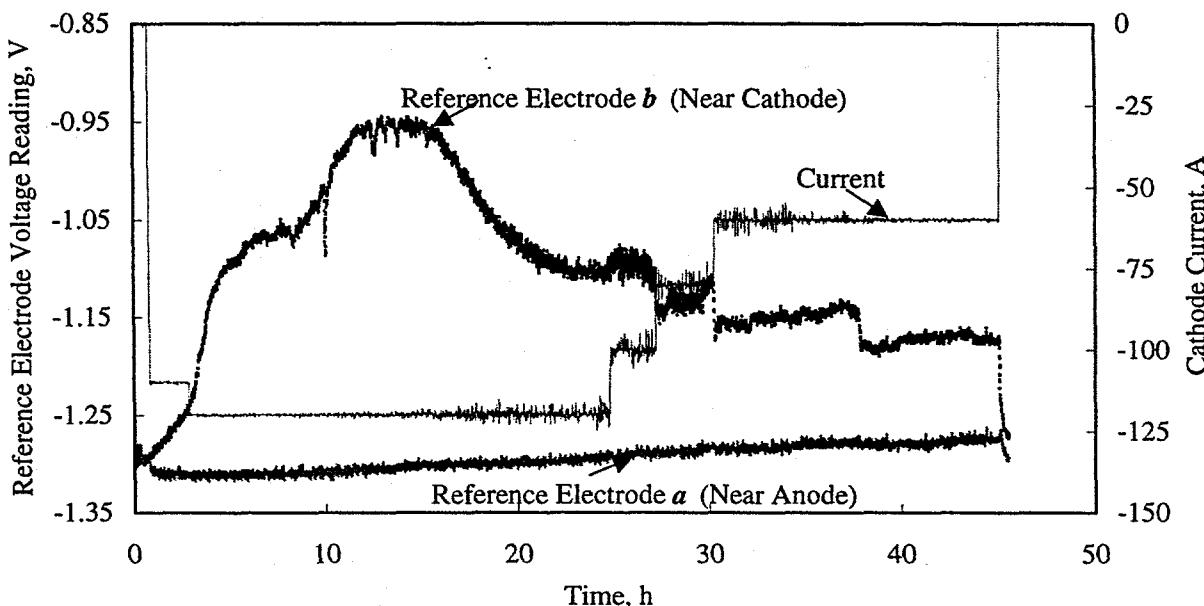


Figure 4. Reference electrode voltage readings and cell current during electrorefining process in Mark-IV Electrorefiner

As mentioned above, the measured voltages for the reference electrodes represent the voltage difference between the reference electrode and the salt/cadmium interface. Variations in the voltage readings while using the direct transport (DT) configuration (Fig. 4) indicated that the interface was not at an equilibrium state when a dc current was applied between the anode and the cathode. There must be an electrical field between the anode/cadmium and cathode/cadmium during the DT process. Based on the reference electrode voltages shown in Fig. 4, a conceptual electrical field distribution model was developed. A schematic of the model is given in Fig. 5.

Using the (DT) configuration in the ER as shown in Fig. 5, only a portion of the applied dc current would pass through

the molten salt medium, directly from the anode to cathode. The balance of the current would go through another path, the cadmium pool, with the cadmium pool acting as an intermediate electrode. This resulted from the cadmium pool always having an intermediate voltage between the FDBs and the cathode when the direct transport configuration was used. The current passing through the cadmium pool resulted in reduction (U^{+3} to U) at the salt/cadmium interface adjacent to the anode baskets, and in oxidation (U to U^{+3}) at the salt/cadmium interface adjacent to the cathode mandrel. Thus the net *electrochemical* transfer of uranium metal into or out of the cadmium pool was zero, which was typical behavior for intermediate electrodes. (Some uranium metal was physically transferred to the cadmium pool by the action of the scrapers on the growing uranium deposit).

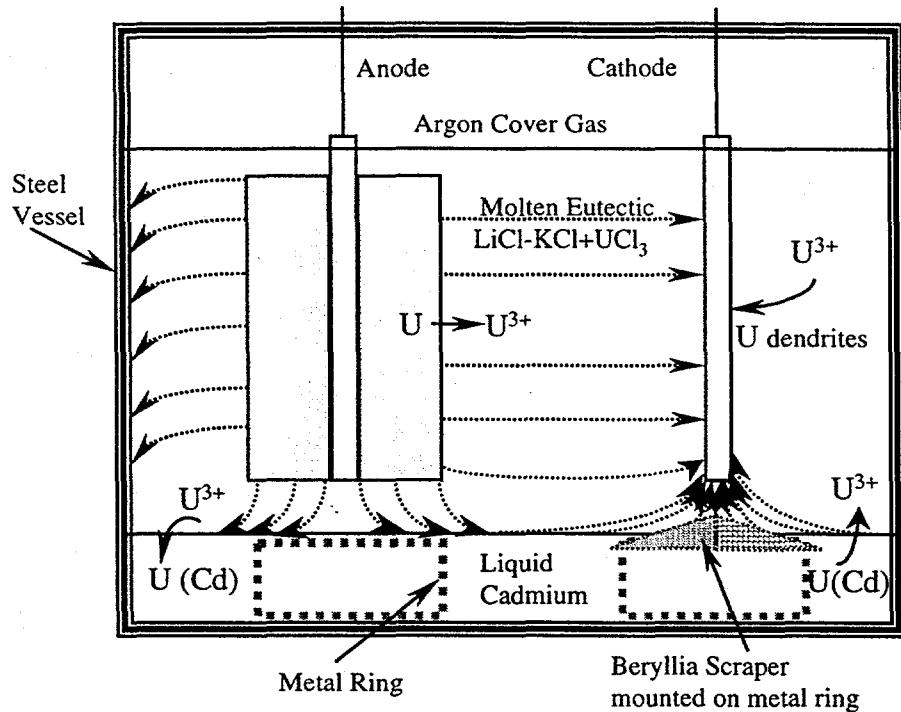


Figure 5. Schematic of current distribution and action of cadmium pool as an intermediate electrode. Arrows with dashed lines indicate primary current distribution. Uranium oxidation occurs in the chopped fuel and at the liquid cadmium/molten salt interface local to the cathode mandrel. Uranium reduction occurs at the cathode mandrel and at the liquid cadmium/molten salt interface local to the fuel dissolution baskets.

The measurements with the two reference electrodes and modeling results for the primary current distribution show that cadmium pool acts as an intermediate electrode. When electrorefining with the DT configuration began, the voltage difference between the cadmium pool and reference electrode *a* (close to the anode) became more negative. This was an indication that U^{3+} was reduced to uranium metal and dissolved into the cadmium at the salt/cadmium interface near the anode baskets. Conversely, the voltage difference between the cadmium pool and reference electrode *b* (close to the cathode) became less negative. This was an indication that uranium was being oxidized from the cadmium pool in the vicinity of the cathode.

The voltage difference between the cadmium pool and reference electrode *b* (close to the cathode) reached an apex value of -0.95 V after approximately 14 hours, and then gradually became more negative (Fig. 4). Two factors could contribute to this observed behavior. For one, the electrode area of the growing uranium deposit was increasing. As a result, the extent of concentration polarization for uranium oxidation from the cadmium pool would be less as the area of the cadmium pool in the vicinity of the deposit also increased. Furthermore, the collection efficiency for the uranium deposit was less than 100%. Uranium dendrites that were dislodged would dissolve, with some time delay, in the cadmium pool. As the dendrites dissolved and the local

uranium concentration in the cadmium pool increased, the voltage difference between the cadmium pool and reference electrode *b* became more negative.

The primary current distribution for the DT configuration in the ER has been simulated using the FIDAP [19] computation code. The simulation results also showed that the cadmium pool acted as an intermediate electrode. A comparable result has also been reported by T. Kobayashi et al. [7]. By numerically simulating the electrochemical processes in a LiCl-KCl liquid cadmium system, which had a nonconductive ceramic partition between the anode and cathode salt regions, they showed that the cadmium pool could be used as an intermediate electrode.

3.3. Formation of Electrical Shorting Path Between Anode Baskets and ER Vessel Resulting from Decreasing Cadmium Level

As noted above, the cadmium level decreased during the period from June 1996 to July 1997. The decrease in the cadmium level caused a problem for the ER operations, namely, formation of an electrical shorting path between anode baskets and ER vessel hardware. Fig. 6 shows schematically how the electrical shorting path developed.

As indicated in Fig. 6, there is a metal ring beneath each port in the ER. The cathode port rings have beryllia bottom

scrapers on its top while anode port rings have a bare metal surface. The height of the metal rings is 9.84 cm from the inside bottom of the vessel. If the cadmium level is below 9.84 cm, the surface of the anode ring will be exposed to the salt phase. When a dc current is applied from the anode to the cathode, the potential of the cadmium pool is lower than that of the anode baskets. Uranium will deposit on the exposed surface of the ring beneath the anode baskets. The deposit will gradually accumulate and finally reach the FDBs (the vertical distance between the top of the metal ring and bottom of the FDBs is 3 inches), which forms an electrical shorting path between the anode baskets and the ER vessel.

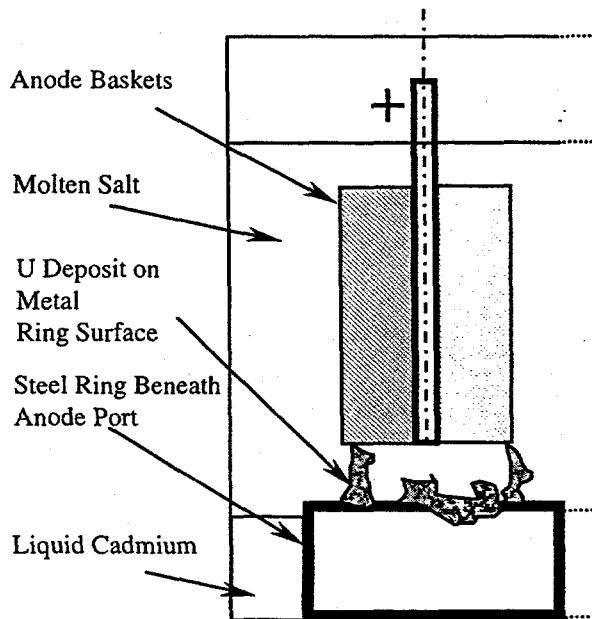


Fig. 6. Schematic of electrical shorting between FDBs and ER vessel.

The voltage and current traces shown in Fig. 7c indicate that electrical shorting between the anode and the vessel was occurring during the electrorefining process. The evidence of the shorting is the fluctuations in the anode voltage trace. The anode voltage was measured relative to the Ag/AgCl reference electrodes. The open circuit potential was assumed to be zero. While shorting occurred, the FDBs and cadmium pool were parallel anodes, with uranium simultaneously being oxidized from both sources. The presence of the shorting path has made it difficult to interpret the endpoint of the electrorefining process, because the amount of charge passed to dissolve the uranium from the FDBs and charge lost to the

vessel as a result of shorting could not be individually measured.

As the anode baskets rotated in the ER salt phase, the electrical shorting path developed between baskets and vessel hardware should be momentary. At the moment when the FDBs touched the deposits on the metal ring beneath them, a sharp decrease occurred in the anode voltage curve, indicating the shorting path was "closed." Then the shorting path was eliminated mechanically by the rotating FDBs or burned off by the high current. After the removal of the shorting path, the anode voltage returned to normal, indicating the shorting path was "open." When the next shorting path built up due to uranium depositing near the FDBs, another sharp decrease in anode the voltage curve occurred, and so on.

The momentary electrical shorting mechanism is corroborated by the experimental results illustrated in Figs. 7a and 7b. Figure 7a provides the anode voltage trace for a period of 60 seconds when the shorting has taken place and the anode assembly was rotating at 5 rpm. Five major voltage transients occurred in the 60 seconds, which indicated there existed only one major contact point between the FDBs and vessel hardware per revolution. This was ascribed to eccentricity in the anode assembly such that only one of the four FDBs made contact with the buildup of material in the vessel. The minor transients in the voltage trace were non-recurring due to weaker contact between the anode and vessel, and could arise from contact from any of the other three FDBs.

In another experiment, the anode assembly was rotated at 25 rpm. Figure 7b provides the anode voltage trace for a period of 60 seconds when the shorting was present and the anode assembly was rotating at 25 rpm. Twenty-five voltage transients occurred in the 60 seconds, with some variation in the magnitude of the voltage transients. While the FDBs rotated at 25 rpm, the uranium dissolution rate might not be sufficiently fast enough to build a strong shorting path for every revolution. The increased mixing action of the anode assembly rotating at 25 rpm, as compared to 5 rpm, increased the probability of washing or breaking long dendritic growths from between the anode and vessel. Thus, the deposit on the metal ring should grow in a more dense fashion, causing fewer strong shorting events and more weak shorting events.

Alternatively, the momentary shorting was briefer on the measurement time scale, and the voltage transients did not approach 0 V as closely as was observed for the anode rotating at 5 rpm.

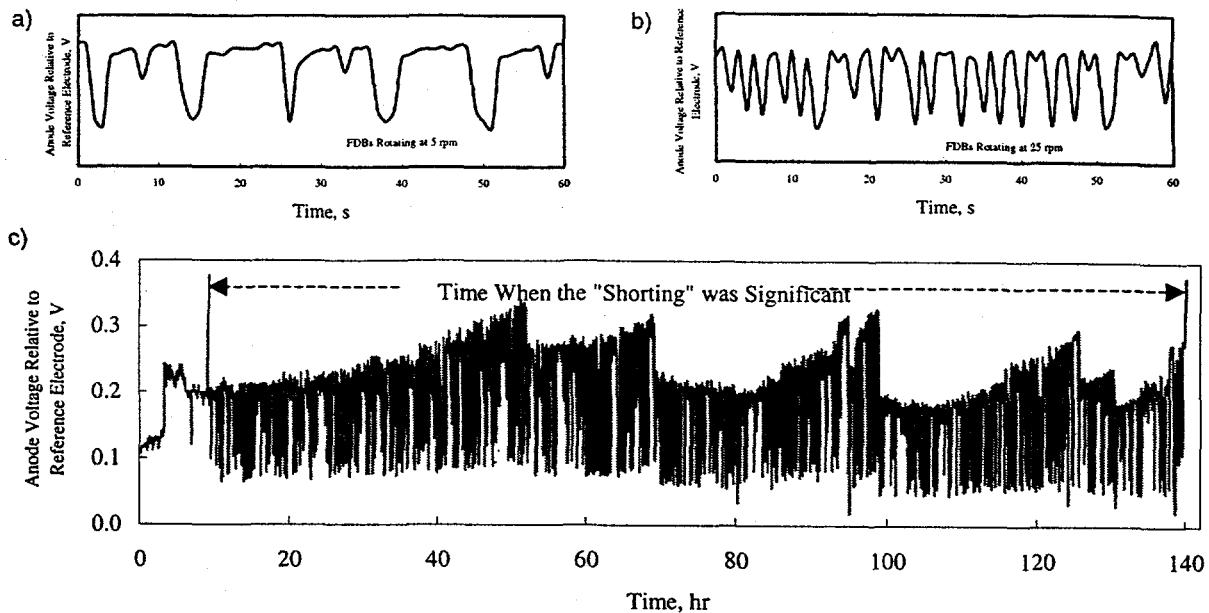


Figure 7. Anode voltage traces when anode baskets electrically shorted with ER vessel during electrorefining process.

In order to solve the electrical shorting problem, 56 kg of cadmium metal was added to the ER in July 1997, which raised the cadmium level from 9.22 cm to 10.29 cm. This increased level ensured the metal ring located beneath the anode port was submerged in the cadmium. The voltage and current traces after the addition of cadmium are given in Fig. 8.

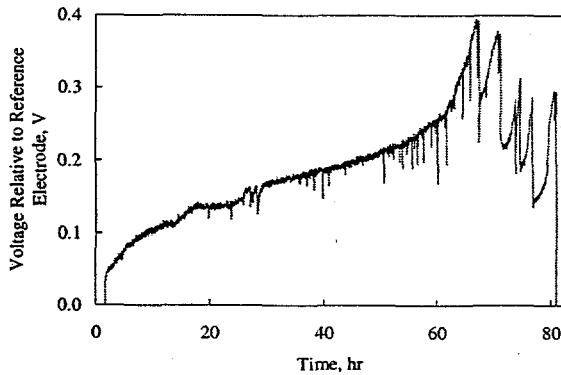


Figure 8. Anode voltage trace after the cadmium level was raised.

The clean anode voltage trace in Fig. 8 indicates that the shorting path between the FDBs and the metal ring, which had been protruding above the cadmium pool, was eliminated by raising the cadmium level.

4. Analytical Results of Cadmium Samples

Two types of samples, "filtered" and "dip", are taken from the cadmium pool. A filtered sample is taken by drawing molten cadmium through a tantalum frit into a tantalum tube. The filter isolates the liquid cadmium from particulates. A dip sample is taken by immersing a 1.27 cm diameter steel crucible into the molten cadmium. Uranium is determined by isotopic dilution mass spectrometry after removing the interfering transuranics. The plutonium content of the cadmium pool is determined by alpha counting. Zirconium and most other species are determined by ICP (inductively coupled plasma) analysis.

Table 2 shows the average weight fraction in the cadmium during spent fuel treatment of driver fuel. Comparing "filtered" and "dip" samples of like elements, the average weight fraction is lower in the filtered samples and the standard deviation is a smaller percentage of the average weight fraction. Since dip samples have no filtering mechanism, any insolubles and particulate will be collected in the samples. This is confirmed by sampling results in Table 2.

Because of the low solubility of zirconium in cadmium at 500 °C (0.21 wt%) and initial experiments with unirradiated feedstocks, the zirconium was saturated in the cadmium pool prior to irradiated fuel testing. Zirconium analysis of the filtered samples produced an average measured weight fraction of $2.33E-03 \pm 2.80E-04$, which bounds the zirconium solubility in cadmium. Zirconium analysis of dip samples show an average measured weight fraction about twice as

high as the filtered samples, illustrating the value of the filtered samples.

Table 2. Average Weight Fraction in Cd Pool During Spent Fuel Treatment

Element (Sample Type)	Average Weight Fraction in Cd Pool	Standard Deviation	Deviation over Average
Uranium (Filtered)	3.90E-03	1.79E-03	46.0%
Uranium (Dip)	4.98E-03	4.53E-03	91.1%
Plutonium (Filtered)	1.68E-05	1.23E-05	73.1%
Plutonium (Dip)	2.67E-05	3.18E-05	119.3%
Zirconium (Filtered)	2.33E-03	2.80E-04	12.0%
Zirconium (Dip)	4.59E-03	3.14E-03	68.4%
Ruthenium (Dip) ¹	1.90E-04	N/A ²	
Technetium (Dip) ¹	2.50E-04	N/A ²	

¹ None detected in filtered samples.

² Only one analysis above detection limit.

5. Conclusion

Several conclusions of practical interest can be drawn from the experimental observations:

- The cadmium pool acts as an intermediate electrode during the direct transport process in the Mark-IV electrorefiner.
- The cadmium level has gradually decreased due to its high vapor pressure and vaporization rate at the electrorefiner operational temperature.
- The low cadmium level has caused the anode baskets momentarily to touch the ER vessel hardware as the baskets were rotating, which generated a periodic electrical shorting path between the anode baskets and ER vessel during electrorefining operations.
- The reflux vapor trap has successfully prevented the cadmium level from declining. The primary function of the vapor trap was to mitigate cadmium formation on mechanical equipment.
- The existence of the periodic electrical shorting path made the process of uranium dissolution from the anode baskets less effective. However, a new anode assembly has been fabricated which incorporates ceramic scrapers

that terminate beyond the diameter of the fuel dissolution baskets. These scrapers are expected to preclude electrical shorting between the anode assembly and the vessel by mechanically removing any buildup as the anode rotates.

6. Acknowledgments

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