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PYROMETALLURGICAL PROCESSING OF INTEGRAL FAST REACTOR METAL FUELS

J. E. Battles, W. E. Miller, and E. C. Gay
Argonne National Laboratory
9700 South Cass Avenue
Argonne, IL 60439-4837 U.S.A.

Phone: 708/972-4383
FAX: 708/972-5246

ABSTRACT

The pyrometallurgical process for recycling spent metal fuels from the Integral Fast Reactor is now in an advanced state of development. This process involves electrorefining spent fuel with a cadmium anode, solid and liquid cathodes, and a molten salt electrolyte (LiCl-KCl) at 500°C. The initial process feasibility and flowsheet verification studies have been conducted in a laboratory-scale electrorefiner. Based on these studies, a dual cathode approach has been adopted, where uranium is recovered on a solid cathode mandrel and uranium-plutonium is recovered in a liquid cadmium cathode. Consolidation and purification (salt and cadmium removal) of uranium and uranium-plutonium products from the electrorefiner have been successful. The process is being developed with the aid of an engineering-scale electrorefiner, which has been successfully operated for more than three years. In this electrorefiner, uranium has been electrotransported from the cadmium anode to a solid cathode in 10 kg quantities. Also, anodic dissolution of 10 kg batches of chopped, simulated fuel (U-10% Zr) has been demonstrated. Development of the liquid cadmium cathode for recovering uranium-plutonium is under way.

INTRODUCTION

The Integral Fast Reactor (IFR) is an advanced reactor concept proposed by, and under development at, Argonne National Laboratory (ANL). Its distinguishing features are that it is a sodium-cooled, pool-type reactor [1] (i.e., all the major components, reactor core, pumps, and heat exchangers are in a large sodium-filled pot); it employs a metallic fuel (an alloy of U, Pu, and Zr clad with a stainless steel-type alloy); and it has an integral fuel cycle (discharged core and blanket materials are processed and refabricated in an on-site facility). The advantages of this concept are an exceptionally high degree of passive safety, resulting from use of a metallic fuel with a sodium coolant, and competitive economics, resulting from low costs for reactor construction and fuel recycle.

A key element in the IFR concept is the pyrometallurgical process, which employs electrorefining with a cadmium anode and a molten salt electrolyte (LiCl-KCl-U/$PuCl_3$) at 500°C, for processing spent metal fuels [2,3]. The IFR pyroprocess for recovery of pure uranium (for blanket element fabrication) and a U-Pu mixture (for driver elements) is based on the ability of a solid cathode to remove only pure uranium, even in the presence of much more $PuCl_3$ than UCl_3 in the molten chloride electrolyte salt. A simplified schematic of the electrorefining apparatus for processing the IFR fuel and blanket material is given in Fig. 1. Spent fuel pins are chopped and put in a basket for dissolution in the electrorefiner (ER) at 500°C. Cadmium dichloride ($CdCl_2$) is then added to oxidize alkali, alkaline earth, and most rare earth metals to their chlorides, which become a part of the molten chloride electrolyte. Essentially pure uranium is electrotransported to the solid cathode; a mixed U-Pu product is electrotransported to a liquid cadmium cathode (not shown in Fig. 1).

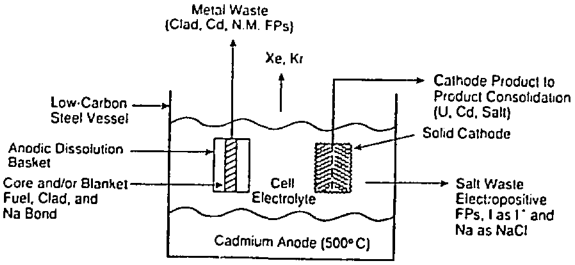

Fig. 1. Schematic Representation of Electrorefiner Operation with Anodic Dissolution of Fuel and Electrotransport to a Solid Cathode. (The abbreviation N.M. FPs represents noble metal fission products.)

These cathodes are removed from the ER cell and retorted to vaporize the cadmium and any occluded salt and to consolidate the product by

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melting. Noble metals, which do not electrotransport, fall into the cadmium pool; they eventually are removed by mechanical means and by distillation of the pool. The salt is freed of lanthanides and actinides by reduction and returned to the process.

The chemistry of the pyroprocess is based on the relative ease of oxidation of the elements that make up the metal fuel. This was determined from the free energies of formation of chlorides of these elements. Our calculations indicated that alkali and alkaline earth metals are readily oxidized into the salt, and less easily oxidized (noble) elements remain as metals. The amount of oxidant can be adjusted so that the actinides and rare earths will mostly be chlorides. The elements subject to electrotransport are U, Pu, Np, Am, Cm, and Zr. Thus, oxidation effects most of the separation of actinides from fission products.

A process model based on thermodynamic data has been developed for predicting the electrotransport of uranium, plutonium, and rare earth fission products [4]. The model also predicts the compositions of the solid cathode and liquid cadmium cathode. A series of experiments was conducted for model verification. The results of these experiments indicated that little or no rare earths are deposited with uranium; the uranium deposit is essentially free of plutonium and other actinides until the uranium inventory reaches a low level; the minor actinides (Np, Am, Cm) follow plutonium; and minor quantities of zirconium are electrotransported. The agreement between this observed behavior and that predicted by the model was very good.

Process development is continuing at the Argonne-Illinois site and experiments performed with an engineering-scale electrorefiner are discussed below. Demonstration of the process with spent metal fuel will be conducted in the fuel cycle facility attached to the EBR-II reactor at the Argonne-Idaho site. This facility was built in the 1960's and used at that time in a demonstration of the closed fuel cycle with uranium-alloy metal fuel [5].

EXPERIMENTAL EQUIPMENT

An engineering-scale electrorefining facility (10 kg of uranium on a single solid cathode) was constructed to demonstrate the electrolytic transport of uranium at plant-scale levels and to measure the dissolution rate of clad segments of uranium-zirconium alloy. The electrorefiner assembly is enclosed by a glovebox (with ovens, transfer locks, wells, and lifting systems), which has a system for controlling gas pressure, temperature, and gas purity. The major components are briefly described below.

Electrorefiner Assembly

The electrorefiner assembly consists of a crucible, crucible insert and cover, electrolyte stirrer assembly, cadmium stirrer assembly, cathode assembly, and anodic dissolver assembly. The crucible is fabricated from low-carbon steel. The crucible insert consists of a 0.32-cm-thick perforated steel cylinder and a 18-cm-thick bottom piece made with four holes. Four scraper/baffle assemblies are mounted inside the crucible insert in the salt space to limit the diameter of the uranium deposit and to promote mixing of the molten material. The crucible cover is constructed of 5-cm-thick low-carbon steel. This cover has many holes of various sizes for suspending components into the crucible. Four large holes are used as mounting positions for a cathode, cadmium stirrer assembly, electrolyte stirrer assembly, and anodic dissolver assembly.

Enclosure System

The enclosure assembly is 4.9 m wide x 2.4 m deep x 3.7 m high and is supported 1.5 m off the floor so that the electrorefiner crucible flange can rest on top of a similar flange on the enclosure floor. Windows (1.0-cm-thick laminated safety glass) with standard glove rings and gloves are situated at appropriate locations on the walls and ceiling of the enclosure to allow for experimental apparatus operation, maintenance, and materials handling.

Two separate mechanical lifting systems are provided inside the enclosure. The primary system is an electrically powered bridge crane with 1993-kg capacity. A backup lifting system (manually operated jib crane with 453-kg capacity) is housed in the electrorefiner area to allow unloading of the bridge crane hook should the primary system become inoperative.

Environmental Control System

The environmental control system consists of a main purification and pressure control subsystem, a backup pressure control subsystem, and a temperature control subsystem. Levels of oxygen, nitrogen, and moisture are controlled to less than 15 ppm each. Oxygen and moisture in the enclosure are continuously monitored with an electrochemical micro-fuel sensor and a hygrometer, respectively. Nitrogen is monitored on demand with a gas chromatograph. The temperature control subsystem consists of an auxiliary argon circulation system equipped with a blower and a water-cooled heat exchanger to minimize the temperature variations.

EXPERIMENTAL RESULTS

Anodic Dissolution

Small-scale laboratory tests were conducted with individual clad fuel (unirradiated) segments to determine the advantages of anodic dissolution vs. direct dissolution. The results showed that both methods were suitable, and that anodic dissolution provided a higher dissolution rate. The voltage used was sufficient to oxidize the heavy metal pin but leave the cladding shell intact. Based on the laboratory tests, anodic dissolution was chosen for scale-up to plant scale (10 kg batch size).

In the fuel dissolution step, uranium and plutonium metals are oxidized at the anode (fuel segments in perforated, prismatic anode baskets), and uranium/plutonium chlorides are simultaneously reduced at the cathode (either a metal mandrel or a container vessel with cadmium). (The Cl^- balance is maintained.) Table I shows the oxidation-reduction reactions for uranium, the bulk of the fuel. For the anode processes, reaction (1) is the normal oxidation of the fuel. The second reaction is a parasitic reaction which oxidizes U^{3+} to U^{4+}. This reaction occurs at the metal basket surfaces and on the metal surface of the cladding hulls. (A corresponding reaction of U^{4+} to U^{3+}, reaction (5), occurs at the cathode.) This reaction results in no fuel dissolution unless the U^{4+} ion is brought into contact with the fuel matrix in the anode. If the U^{4+} ion comes in contact with fuel metal in the anode, then anode reaction (3) occurs with resultant fuel dissolution. This was an important consideration in designing the anode baskets with a prismatic shape. Cylindrical baskets gave very low current efficiency, since the U^{4+} ions formed on the outside shell of the basket could not be brought into contact with fuel and were reduced parasitically, reaction (5), at the cathode. With four prismatic baskets arranged on an anode shaft in the shape of a plus (+), each basket follows in the wake of another basket when the anode shaft is rotated, and the U^{4+} rich salt is continuously contacted with fuel as the salt flows through the openings in the basket shell.

TABLE I. Anodic Dissolution Reactions

Anode		
(1)	$U^\circ - 3e \longrightarrow U^{3+}$	(USEFUL)
(2)	$U^{3+} - e \longrightarrow U^{4+}$	(PARASITIC)
(3)	$3U^{4+} + U^\circ \longrightarrow 4U^{3+}$	(USEFUL)
Cathode		
(4)	$U^{3+} + 3e \longrightarrow U^0$	(USEFUL)
(5)	$U^{4+} + e \longrightarrow U^{3+}$	(PARASITIC)

In the anodic dissolution tests with the engineering-scale electrorefiner, the cathode was the cadmium pool in the bottom of the electrorefiner vessel, and four anode baskets mounted on the anode shaft gave parallel circuits in the electrolyte. The simulated fuel pins were U-10 wt% Zr in stainless steel cladding. The cell voltage was limited to -1.3 volt (to prevent corrosion of the cladding and basket materials). The anode baskets were used in over 10 runs to dissolve 10 kg of uranium and showed little visual evidence of corrosion. In these tests, ten kilogram batches of clad heavy metal pins were completely dissolved in 24-30 hours. The current efficiency for this anodic process was greater than 50%.

An important feature of the anode design is the capability for removing residual electrolyte (after dissolution) from the empty cladding hulls in the anode baskets. After dissolution is complete, the anode shaft is raised to lift the baskets out of the electrolyte, and then the basket assembly is rotated at a high rate. The centrifugal force produced in the assembly strips residual liquid electrolyte from the baskets. In a stripping experiment, one of the four baskets and its contained cladding hulls were water washed to remove residual salt after the spinning operation. About 15 g of salt was found along with 0.02 g of fines. The heavy metal content of the salt is 3.2 wt%, which means that the heavy metal content of residue in the four baskets is about 2 g (<0.02% of process input). The cladding hulls, essentially salt free, are easily removed from the anode baskets, which are then reused.

The anodic dissolution of simulated fuel in the engineering-scale electrorefiner is now a routine operation. However, it was not certain whether our results were applicable to the dissolution of irradiated fuels. For this reason, experiments* with sodium-bonded irradiated fuel (both anodic dissolution and electrotransport to solid cathodes) were conducted using a small electrorefining cell in the Hot Fuel Examination Facility at EBR-II. Of particular importance is the quantity of uranium, plutonium, and fission products remaining with the cladding hulls at the completion of the anodic dissolution operation. These materials were quantified for 10 cladding hulls resulting from 10 anodic dissolution tests of single segments (0.64-cm long) of irradiated fuel from EBR-II. Binary fuel, U-10 wt% Zr, was used in the first six tests and ternary fuel, U-19 wt% Pu-10 wt% Zr, in the last four tests. For these examinations, electrolyte-coated anodes containing the fuel remnants (cladding and any remaining fuel) were treated with deionized water to dissolve the salt coating (in a plant-scale process this coating will be removed by centrifugal force, as described above). The fuel remnants were then dissolved in acid (including the cladding) and the solutions analyzed.

Results for the U-Pu-Zr fuel (expressed as the percentage of initial material or isotope of the material which remained in the remnant after anodic dissolution) are as follows: ^{95}Nb, 0.02; ^{95}Zr, 0.03; ^{106}Ru, 0.02; ^{137}Cs, 0.003; ^{144}Ce, 0.5; U, 0.03; and Pu, 0.005. The removal of some of the other fission products was slightly less complete. Similar results were obtained for the U-Zr fuel. Essentially all the fuel dissolves.

Electrotransport to Solid Cathode

A number of alternatives are available for heavy metal product collection at a solid cathode. In the IFR process, solid cathodes are used to collect uranium and exclude plutonium metal from the product. For this operation, uranium is electrotransported from the cadmium

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*R. W. Benedict, ANL-W, private communication.

pool in the electrorefiner to a solid cathode. Prior to this, uranium is electrotransported from anodic dissolution baskets to the cadmium pool, or it is added to the cadmium by direct dissolution. Alternatively, uranium can be electrotransported directly from the anodic dissolution baskets to a solid cathode. This latter mode of operation reduces overall processing time, since the dissolution and collection processes occur simultaneously.

Single Mandrel Cathode. A single cylindrical iron mandrel has been tested as a cathode. The uranium deposit collected on this cathode must be physically removed from the iron mandrel in a product recovery step. During formation of a deposit, the cathode shaft is rotated and scrapers (BeO and steel plates located in the electrolyte phase) limit the overall diameter and length of the deposit. Dendrites that grow beyond these limits are broken off by the scrapers, fall into the cadmium pool, and are recycled. The density of the dendritic deposit is increased as the electrotransport of uranium and the shaping operations are continued; however, the current efficiency is lowered because of the physical loss of uranium from the cathode.

Twenty-five single mandrel cathode runs were conducted to determine the process parameters for electrotransport of uranium to single mandrel cathodes. In all of the single mandrel cathode tests prior to Run 43, salt mixing was continued during the entire run. Under continuous salt mixing, the highest weight of uranium collected on the single mandrel cathode (without a beryllia plate attached to the lower end of the cathode) was 6.4 kg.

The operating conditions for Run 43 were as follows. (1) The single mandrel cathode was operated with salt mixing early in the run and no mixing during the latter half of the run. (2) To take full advantage of the uranium deposited on the wall of the electrorefiner after anodic dissolution, the single mandrel cathode was placed in the same position of the electrorefiner as was used for the baskets in the preceding anodic dissolution. (In most of the earlier single mandrel cathode tests, the uranium in the cadmium anode pool was added by direct dissolution.)

The electrodeposition time for Run 43 was 37 hours. During this run, the salt mixer (150 rpm) was turned off after 20 hours, the rotation speed of the cathode during the run was 20 rpm, the cadmium mixer was not used, and approximately 8224 Ah was passed through the cell. For most of the run, the cell voltage was less than 0.6 V, the current was between 200 A and 250 A, and the cell resistance was between 1 and 3 mΩ. The current and resistance profiles for the run are given in Fig. 2. Calculations indicate that currents of about 400 A could have been passed through the cell with a 0.8 V cutoff (the voltage cutoff limit for this run). This high current was not used because the cycler was limited to 250 A for sustained operation.

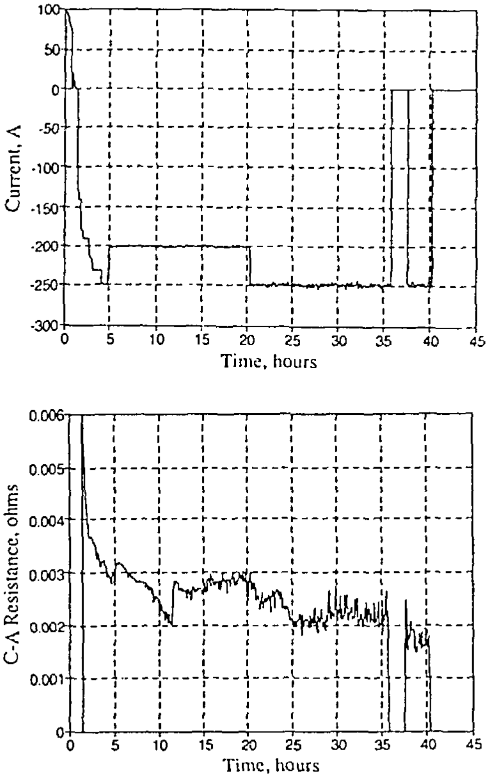

Fig. 2. Current and Resistance Profiles for Run 43

The weight of the cathode deposit collected during Run 43 was 11.8 kg. Results of chemical analysis of samples taken from the top, middle, and bottom of the deposit indicate that the amount of uranium in the deposit was greater than 9 kg. The deposit was uniform in shape and showed the highest density at the lower end of the deposit.

The success achieved in Run 43 has made the single steel mandrel cathode the primary solid cathode of choice. The simplicity, ruggedness, and ease of assembly of this cathode are advantages over the multipin cathode.

Multipin Cathode. Multiple rods of uranium-zirconium alloy (each rod about 0.5-cm dia) have been used as a multipin cathode. In this cathode, the uranium rods may be melt consolidated along with the dendritic deposit, and physical removal from the base cathode material is not required. Multipin cathode with steel rods were also tested.

Experiments were conducted to investigate the electrotransport of uranium to multipin U-Zr cathodes and multipin steel cathodes. The arrangement and number of rods that provide the highest uranium collection rate and that could be used to collect 10 kg of uranium were determined. Cathode deposits of 7 to 10 kg uranium were produced with both U-Zr and steel multipin cathodes. Rod arrangement patterns in the cathode were either in a circular or a plus (+) configuration. The number of rods was varied from 5 to 13. Runs were made under various stirring conditions in the cadmium anode pool and the electrolyte, including the condition of no stirring (other than that due to cathode rotation). Half the runs were made at one uranium concentration (3 wt%) in the electrolyte; in the other half, the uranium concentration was doubled. Collection efficiency (rate of collection) was not greatly affected by any of the variables except the stirring rate, with the highest rates being obtained when the salt mixer and anode cadmium mixer were at 0 rpm. Stirring causes dendrite loss (with subsequent recycle) from the cathode deposit. The most favorable characteristic for some of the multipin cathodes was the high current (150-200 A) that could be sustained throughout the runs. The concentration of uranium in the cathode products from these runs was between 75 and 90 wt%.

Direct Transport. In three runs (AD8-14, 32, and 42), uranium was electrotransported directly from the anodic dissolution baskets to the solid cathodes. The simultaneous dissolution and deposition have the potential for shorter processing time. The highest uranium collection efficiency (Run AD8-14, 45%) was achieved with the single solid mandrel cathode. The highest weight of uranium collected (Run 32, 7.8 kg) was achieved with the U-Zr multipin cathode. In future tests, electrode design changes will be made to increase the uranium collection efficiency and the weight of uranium collected on the solid cathode.

Electrotransport to Liquid Cadmium Cathode

A plutonium/uranium mixture is collected in a cadmium pool contained in a ceramic crucible suspended in the electrolyte phase. To keep reasonable size, it is necessary to collect an amount of plutonium in the cadmium pool in excess of the solubility of plutonium in cadmium (3.62 wt% at 500°C). When plutonium exceeds its solubility in liquid cadmium or liquid cadmium-uranium, it precipitates as $PuCd_6$, an intermetallic compound. In small-scale laboratory work (0.2 kg plutonium scale), cadmium cathodes which contain about 15 vol% plutonium solid have been produced. For a plant-scale electrorefiner, the 10 kg heavy metal fuel batch will contain about 2.0-2.5 kg plutonium. A cathode pool containing about 30 kg of cadmium is required for this plutonium collection (a 5 L ceramic crucible is required).

Electrochemical transport of kilogram quantities of uranium to a liquid cadmium cathode (LCC) is being studied in the engineering-scale electrorefiner. The cadmium-cathode electrode consists of a stirrer in a ceramic crucible which is suspended in the electrolyte phase in a manner similar to the anodic dissolver. The experiments conducted in the engineering-scale electrorefiner have used uranium to simulate the behavior of uranium-plutonium mixtures. Limitations in these experiments include: (1) one metal (uranium) is electrotransported rather than two metals simultaneously (uranium and plutonium), and (2) without plutonium, the effect of $PuCd_6$ on the process was not determined. On the other hand, the containment of uranium dendrites in the product ingot was a major concern and was sufficient reason for using uranium to simulate the behavior of uranium-plutonium mixtures.

Four liquid-cadmium cathode runs have been completed. The weight of uranium in the product from these runs was 3.0, 2.8, 4.4, and 3.0 kg, respectively. The cadmium concentration in the products (68-75 wt%) was about that needed to form $PuCd_6$ (74 wt% cadmium), if the uranium were replaced with plutonium. If the electrotransport characteristics of uranium-plutonium mixtures were similar to the electrotransport characteristics of uranium determined in these experiments, the LCC design and operating conditions used in these tests would be suitable for collecting at least 3 kg of uranium and plutonium. However, because of the low electrodeposition current (25-30 A), the process goal of a 24-h electrodeposition time was not achieved in runs LCC-1, -2, and -3.

The electrodeposition time in Run LCC-4 (32.5 h) was significantly reduced from that of the earlier runs. The LCC used in Run LCC-4 consisted of an alumina crucible supported in a steel cage, which was attached to an elevation mechanism. An alumina-covered impeller was located above the crucible. The elevation mechanism was used to position the impeller at different heights in the crucible. The impeller shaft was used as an electrical probe to make contact with the cadmium in the crucible, and the impeller was used as a uranium dendrite breaker. Uranium dendrites were broken off by rotating the impeller at 20 to 270 rpm, and the dendrites were forced beneath the cadmium surface. This step was necessary to prevent dendrites from bridging between the cadmium in the crucible, the steel support rods, and the cage that supported the ceramic crucible. Since the support rods and cage were at anodic potential, uranium contact with this hardware could cause an electrical short circuit. The LCC equipment used in Run LCC-4 was the same as that used in Runs LCC-1, -2, and -3, except that alumina was substituted for BeO as the crucible material and the operating procedure was altered. Changes in the operating procedure were: different positioning of the impeller, use of reference electrodes to determine cathode voltage, incorporation of no-current-flow period into the duty cycle, and change in the distance from cathode to anode.

Data collected in this and other runs showed that a high level of cadmium in the collection crucible is beneficial. The crucibles are tapered and, as the level in the crucible increases, the area of the cadmium-salt interface increases. Also, as the cadmium level inside the crucible increases, the distance from the cathode to the anode decreases. Both of these effects reduce cell resistance and thereby allow increased transport rates at higher currents.

The uranium concentration in this product varied from 7.0 to 29.0 wt%; 7.0 to 12.0 wt% uranium was found in the lower half of the cadmium phase, and 15.0 to 29.0 wt% uranium in the upper half. There was a negligible amount of salt in the product. A small layer of salt covered the product and filled voids created by the dendrite breaker. This is an improvement over Runs LCC-1 to -3, which showed 1.7 to 4.8 wt% salt in the product. The concentration of cadmium in the product varied from 71.5 to 99.4 wt%. The weight of uranium collected in the cathode crucible, estimated from the chemical analysis of the product, agrees with the estimate based on product weight within the uncertainty of the chemical analysis. If the distribution of heavy metal in cadmium shown in this run can be obtained with this hardware during electrotransport of uranium-plutonium mixtures, a LCC product with 3 kg of heavy metal can be collected. Work is proceeding in optimizing operating conditions to further reduce processing time.

Product Consolidation

Both the plutonium-uranium products electrotransported to liquid cadmium cathodes and the uranium products electrodeposited on a solid mandrel cathode have to be processed to remove salt and cadmium and recover the uranium and plutonium for recycle. Tests have shown that high-purity uranium (<80 ppm Cd) can be obtained by retorting a uranium product deposited on a solid cathode that contained about 12 wt% salt and 2 wt% cadmium. Consolidation tests with up to about 7 kg of uranium dendrites have produced well-formed, high-purity uranium ingots. Tests have also shown that high-purity uranium-plutonium (<54 ppm Cd) can be obtained by retorting a uranium-plutonium product codeposited in a liquid cadmium cathode that contained about 68 wt% cadmium, 26.1 wt% uranium, and 5.7 wt% plutonium.

SUMMARY

A prismatic, multiple basket anodic dissolver has demonstrated that 10 kg of uranium (chopped pin segments of steel-clad uranium-zirconium alloy) can be dissolved in about 24 hours. The salt removal operation with the anodic dissolution device provides nearly salt-free baskets and cladding hulls. Cathode products that contain 9-10 kg of uranium were produced by electrodepositing uranium on a solid mandrel cathode in about 27 hours. The single teel mandrel cathode is the primary choice of solid cathode to recover pure uranium in the IFR process. Results from tests designed to electrotransport uranium to liquid cadmium cathodes show that 3 kg of uranium can be collected in about 33 hours. If the distribution of heavy metal in cadmium achieved in these runs can be obtained with this hardware during electrotransport of uranium-plutonium mixtures, a product with 3 kg of heavy metal can be collected, and this would meet the IFR processing needs for uranium-plutonium mixtures.

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