Research Article

Current Status of Pyroprocessing Development at KAERI

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Pyroprocessing technology has been actively developed at Korea Atomic Energy Research Institute (KAERI) to meet the necessity of addressing spent fuel management issue. This technology has advantages over aqueous process such as less proliferation risk, treatment of spent fuel with relatively high heat and radioactivity, and compact equipments. This paper describes the pyroprocessing technology development at KAERI from head-end process to waste treatment. The unit process with various scales has been tested to produce the design data associated with scale-up. Pyroprocess integrated inactive demonstration facility (PRIDE) was constructed at KAERI and it began test operation in 2012. The purpose of PRIDE is to test the process regarding unit process performance, remote operation of equipments, integration of unit processes, scale-up of process, process monitoring, argon environment system operation, and safeguards-related activities. The test of PRIDE will be promising for further pyroprocessing technology development.

1. Introduction

Pyroprocessing treatment can reduced the volume, radioactivity, and heat load of the light water reactor (LWR) spent fuels [1]. In addition, pyroprocessing based on the group recovery of transuranic element (TRU) can provide metal fuels for the sodium-cooled fast reactor while keeping higher intrinsic proliferation resistance. Therefore, successful development of pyroprocessing can save disposal space, reduce the radiotoxicity of spent fuels, and increase uranium utilization efficiency.

Korea Atomic Energy Research Institute (KAERI) has been developing pyroprocessing since 1997. The concept development, bench scale testing, and demonstration of laboratory scale key unit process had been carried out since 2006. From 2007 to 2011, the focus moved to the design and construction of engineering-scale integrated system. Pyroprocess integrated inactive demonstration facility (PRIDE) was constructed in 2011 and it began test operation in 2012.

Process flow diagram consists of head-end processes (decladding, voloxidation, oxide feed preparation), electrochemical processes (electrolytic reduction, electrefining, electrowinning), and waste treatment processes as shown in Figure 1.

2. Unit Process of Pyroprocessing

2.1. Head-End Process. The head-end process in pyroprocessing is to convert spent fuel assembly into a suitable feed material which is supplied to the electrolytic reduction process.

The first step of head-end process is to disassemble spent pressurized water reactor (PWR) fuel assembly and to extract fuel rods followed by cutting fuel rods of about 4 m into rod-cuts in proper size for decladding process. Disassembling, extraction, and cutting operations of actual spent PWR fuel assembly have been conducted in a laboratory scale for postirradiation examination (PIE) and “Direct Use of PWR spent fuel In CANDU reactors” (DUPIC) experiment in postirradiation examination facility (PIEF).

In order to enhance the decladding efficiency for high burn-up spent fuel, the oxidative decladding efficiency...
without rotation mode was tested. The decladding efficiency was close to 100% for spent fuel with a burn-up less than 40,000 MWd/tU when the oxidative decladding with rod-cuts less than 30 mm was performed at 500°C for 10 h. On the other hand, spent fuel with a burn-up higher than 50,000 MWd/tU showed the low decladding efficiency of about 15% due to small fuel-to-clad gap and low oxidation rate. An improved decladding efficiency higher than 99% for the high burn-up spent fuel required an oxidation time longer than 20 h at 700°C. Therefore, oxidative decladding equipment with rotational device would be required to reduce an oxidative decladding time and obtain the very high decladding efficiency.

Feed material forms such as granule and porous pellet were considered to enhance the current efficiency of electrochemical processes. The particles of U₃O₈ powder were bonded together during thermal pretreatment and transformed into aggregates above 1000°C. In 2009, a laboratory scale rotary voloxidizer (max. 1.5 kg U₃O₈ powder/batch) as shown in Figure 2 was designed and manufactured to investigate particle size controllability for U₃O₈ powder. The fabrication characteristics of granule from 200 g U₃O₈ powder was investigated under the conditions of thermal pretreatment temperature of 1150 to 1200°C in argon atmosphere, treatment time of 5 to 15 h, and rotational velocity of 1 to 3 rpm using a rotary voloxidizer. Recovery rate of granule higher than 1 mm with UO₂₊ₓ (0.25 < x < 0.67) composition (Figure 2) significantly increased with increasing temperature, rotation velocity, and time of up to 10 h. Typical recovery rate of granule showed about 89% (>1 mm) and 98% (>0.5 mm). The UO₂ granule with density of 4.38 g/cm³ (40% theoretical density (TD)) was fabricated by reduction of UO₂₋ₓ at 1000°C for 5 h in 4% H₂-Ar atmosphere. The porous UO₂ pellets with density of 6.78–7.67 g/cm³ (60–70% TD) were fabricated by traditional sequential process of compaction and pretreatment (sintering) using the U₃O₈ powder obtained by oxidation of UO₂ pellets.

Engineering-scale rotary voloxidizer for feed form fabrication was installed at air cell in PRIDE facility in July 2012. Feed form fabrication technology will be demonstrated using simulated fuel pellet from 2013.

An off-gas treatment system for trapping fission products released from oxide feed fabrication process is very important for protecting release of nuclides to environment. In the middle of 1990s, research on the trapping of cesium released from the “Oxidation, REduction of enriched OXide fuel” (OREOX) and sintering processes has started in fabricating DUPIC fuel [2]. Off-gas treatment system for trapping both volatile fission gases such as H-3 and I in OREOX process and Cs in sintering process has been established at “DUPIC Fuel Development Facility” (DFDF) as shown in Figure 3(a). It was demonstrated that released Cs from sintering process was completely trapped by fly ash filters. Figure 3(b) shows that approximately 100% of each cesium isotope released was trapped by using fly ash filter.

2.2. Electrolytic Reduction. The electrolytic reduction process based on the electrochemical reduction in a LiCl-Li₂O electrolyte has been developed for the volume reduction of PWR spent fuels and for the supply of metal feeds for the electrorefining process. During the electrolytic reduction process, high heat load fission products dissolve into the chloride molten salt resulting in the decrease of heat load of spent fuels. Additionally, the metallization of oxides to more
dense metals enables the volume reduction of spent fuels. The metal products produced by the electrolytic reduction process can be directly transferred to the electrorefining process as feed materials. One of the concerning points is the concomitant transfer of residual salts in the metal products to the electrorefining process. The residual salts from electrolytic reduction process, LiCl-Li₂O, would cause the formation UO₂ and the breaking of the eutectic composition in the electrorefining salt, LiCl-KCl. To address this incompatibility of salts between electrolytic reduction and electrorefining, electrolytic reduction cathode processing was devised for the removal of residual salts in the metal products of the electrolytic reduction process as shown in Figure 4.

2.2.1. Development of Electrolytic Reduction Process. With the progress of electrolytic reduction reaction, the oxide ion from the oxide spent fuel contained in the cathode basket is evolved as gas on the anode leaving the metal spent fuel in the cathode basket, and alkali and alkali earth elements are dissolved into the molten salt. Lanthanides except Eu and metallic fission products remain in the cathode. Consequently, electrolytic reduction process can supply metal feed to electrorefining process and separate selectively the high heat load fission products such as Sr and Ba from the spent fuel.

The construction of a newly designed laboratory scale electrolytic reduction system (20 kg UO₂/batch) was completed at KAERI in 2009 and inactive demonstration has been performed showing high reduction yields up to 99.5%. In 2011, based on the laboratory scale demonstration results, the electrolytic reducer for PRIDE was designed and fabricated to treat 50 kg-U per day, which includes a cathode basket with a capacity of 50 kg-U loading and 6 anodes (Figure 5).

2.2.2. Development of Electrolytic Reduction Cathode Processor. After the electrolytic reduction process, the residual salt in the cathode basket amounts to about 20 wt% of the metal products. The cathode process for the removal of residual salts in the cathode basket of the electrolytic reducer can ease the burden of the electrorefining process. The recovered salt in the cathode processor will be treated in the waste salt regeneration process and then recycled to the electrolytic reduction process. The operation of cathode processor is based on the vaporization under vacuum condition.

In 2010, a laboratory scale cathode processor (4 kg LiCl/batch) was installed. This equipment is characterized
2.3. Electrorefining Process. Electrorefining, an electrochemical process to recover pure uranium from a metallic or oxide spent fuel, is an essential process in pyroprocessing, because it handles the uranium which is about 93% of spent fuel. During the electrorefining, the reduced metal from forgoing process, which is composed of uranium, TRU, and rare earth element (RE), is dissolved into a LiCl-KCl eutectic salt, whereas only uranium is deposited as a pure dendritic form on a solid cathode. The used salt containing the accumulated TRU and RE is transferred to electrowinning process.

2.3.1. High-Throughput Electrorefining System. The high-throughput electrorefining system is installed recently in PRIDE. The uranium dendrite is deposited and drawn from the electrorefiner, distilled for salt-removal in salt distiller, and then the salt removed dendrite is consolidated for storage or future use. Uranium trichloride (UCl$_3$) is prepared separately and supplied to the electrorefiner. The used salt in an ingot form containing U, TRU, and REs is transported to the next process, electrowinning.

2.3.2. Electrorefiner. In order to increase a throughput of electrorefiner, KAERI has developed the advanced
high-throughput electrorefiner by employing a graphite cathode. The self-scraping mechanism of graphite cathode increases the efficiency of the electrorefiner due to an elimination of the stripping step [3]. In addition, continuous operation of the advanced electrorefiner can be realized by adding a bucket-type deposit transfer system. The 20 kgU/batch throughput can be achieved when 40 kg of simulated fuel pellets are loaded into anode basket and the UCl$_3$ concentration is more than 8%. Recently, the high-throughput electrorefiner was manufactured and installed at PRIDE, which is shown in Figure 7. The electrorefiner for PRIDE was designed to treat 50 kg-U per day, which includes 25 graphite cathodes and 4 cathode baskets with a capacity of 100 kg-U loading.

2.3.3. Salt Distiller. The dendritic uranium deposit is drawn out with about 25 wt% of salt in it from the electrorefiner (Figure 8). To remove the salt for following consolidation process, a vacuum evaporation has been developed in laboratory scale to verify the performance. The engineering-scale salt distiller for PRIDE, which has a capacity of 50 kg-U/day, was installed. The salt distiller comprises a distillation tower, a cooler, and a vacuum system. Optimum operation conditions were obtained from the lab-scale experiment at 820°C with a capacity of 20 kg-U/batch, proving that the salt recovery yield was about 97 wt%.

2.3.4. Melting Furnace for Consolidating. The engineering-scale melting furnace for a continuous operation to increase its capacity has been developed. Since the uranium product has a form of dendrite that is difficult to melt by direct heating, a supplemental charge method was developed. The uranium dendrite is gradually added into preexisting molten uranium and tilted toward the molds. The performance of the engineering-scale melting furnace for PRIDE, as shown in Figure 9, was preliminarily evaluated by a melting test of Cu with a capacity of 50 kg-U/batch [4]. An induction heating system with a nonwater cooling coil in an argon gas ambient was also developed to remove the risk from a reaction of the U-dendrite with water in hot cell. The performance of this nonwater cooling coil was confirmed by a melting test of a copper [5]. The temperatures of the melting chamber and the heating coils were maintained below 250°C and 600°C, respectively, during the heating test up to 1050°C.

2.3.5. Uranium Chlorinator. UCl$_3$ plays the role of electrolyte ions to initiate the reaction. It also removes unreduced lanthanide oxides in the anode basket of the electrorefiner and stabilizes the initial cell voltage between electrodes in the electrorefining reactor. The engineering-scale UCl$_3$ fabrication apparatus consists of a chlorine gas generator, a uranium chlorinator, a Cd distiller, a pelletizer, and an off-gas and dry gas scrubber. The product UCl$_3$ in LiCl-KCl is transferred to the Cd distiller to remove Cd in UCl$_3$ and then transferred to a pelletizer to form a pellet which could be supplied easily to electrorefiner. The engineering-scale uranium chlorinator with a capacity of 30 kg-U/batch, as shown in Figure 10, was installed recently for PRIDE.

2.3.6. Molten Salt Transport System. As the electrorefining operation proceeded, TRU and RE are accumulated in
2.4. Electrowinning System. Electrowinning process is a step of pyroprocessing for the simultaneous recovery of U and TRUs from the remaining salt after electrorefining process using a liquid cadmium cathode (LCC), imposing higher nuclear nonproliferation. The electrowinning process developed at KAERI consists of three steps (Figure 12): (i) LCC electrowinning for simultaneous recovery of U and TRU using an LCC, (ii) residual actinide recovery (RAR) process for recovery of the residual low-concentration U/TRU in the salt after the LCC electrowinning process, and (iii) Cd distillation of the cathode product (i.e., Cd ingot including the U/TRU deposits) produced from the LCC electrowinning and the RAR processes. The essential technologies that include the development of effective LCC structure [7], RAR methods [8], analytical technologies [9], and computational model of electrowinning cell [10] have been developed to enhance the operation efficiency of electrowinning process. In order to demonstrate the developed technologies, the engineering-scale electrowinning system of PRIDE was established.

2.4.1. LCC Electrowinning Process. The electrowinning technique using an LCC is a key step for a nonproliferation because TRU could be codeposited with uranium in liquid cadmium. However, the U ion was known to be deposited in the shape of dendrite on the surface of the liquid cathode [11]. This U dendrite hinders a codeposition of the U and TRU elements. Therefore, the inhibition of the growth of uranium dendrites in the LCC has been considered a critical technique for the electrowinning process. KAERI has developed a mesh-type LCC assembly (i.e., mesh agitator with LCC) to prevent U deposits from growing into dendrites, resulting in the increase of U recovery [7]. The mesh agitator pushes the U deposits generated on the LCC surface into the liquid Cd. Then U deposits sink into the liquid Cd and are gathered at the bottom of the LCC crucible. The performance of the mesh agitator was evaluated in lab-scale LCC experiments. From this experiment, 14 wt% U/Cd was successfully collected without the growth of dendrite out of the LCC crucible.

An engineering-scale PRIDE LCC electrolytic system was designed and constructed based on the lab-scale LCC experiments and the computational simulation results of heat transfer and current distribution [12]. The LCC electrowinning equipment installed in PRIDE and its recovery capacity of heavy metal is 1 kg heavy metal (HM)/batch (Figure 13).

2.4.2. Residual Actinides Recovery Process. RAR process has been developed by combining the electrolysis using an LCC to collect most the residual actinides in a spent salt phase and an oxidation of a part of the rare earth fission products codeposited onto an LCC by a CdCl₂ oxidant [13]. The same electrowinning equipment using an LCC for a recovery of the fuel material can be used for RAR operation. Therefore, the RAR process has promising merits, such as compact equipment and a simple process to operate in a hot cell [14]. The PRIDE RAR processing equipment was designed and installed in the argon cell of the PRIDE.

2.5. Waste Treatment Process

2.5.1. Metal Waste Treatment Process. Metal wastes generated from the head-end process of spent fuel are mainly composed of cladding hulls and structural parts. A trace of actinides, such as U, Pu, Am, and Cm, and fission products (FPs)
penetrate into the inner layer of zircaloy cladding hulls. Structural parts made of various stainless steels, Inconel, and zircalloys are also activated, resulting in the generation of Co-60 by neutrons created during a fuel irradiation process in the reactor. Table 1 shows the amount of metal wastes by volume and mass before and after the treatment. It was assumed that the stacking density of the cladding hull was 980 kg/m³ and actual density of a cladding hull ingot after induction melting was 80% of theoretical density. In addition, the volume reduction of the fuel hardware by 500 ton compaction was assumed to be 1/8.

Two major streams of the metal waste treatment for storage or disposal are the compaction and melting processes. The melting process has a higher volume reduction factor compared to the compaction method while it is a high temperature process that accompanies higher energy and cost. Zr recovery from zircaloy cladding hulls correspond to an alternative technology that can reduce the amount of a high level waste and reuse expensive Zr metal through a recycling process.

2.5.2. Waste Salt Treatment Process. The waste salt treatment process is divided into two parts: LiCl waste salt at 650°C and eutectic (LiCl-KCl) waste salt treatment at around 700°C. In LiCl waste salt treatment process, Groups I and II fission products involved in LiCl waste salt are removed by using a melt crystallization process and then the remaining residual LiCl salts containing high content of Groups I and II fission products, such as Cs, Sr, and Ba, are immobilized by mixing with an inorganic matrix, Si-Al-P (SAP) complex, and sintering at 1,200°C, to the ceramic waste forms. Rare earth fission products bearing eutectic waste salt from the RAR process is treated by successive rare earth removal processes: a combination of oxygen sparging, layer separation, and vacuum distillation/condensation process. Finally, the remaining rare earth oxides (or oxchlorides) are fabricated into the durable ceramic waste forms by mixing with zinc titanate (ZIT) matrix and sintering at 1,200°C. During each waste salt treatment process, pure LiCl and eutectic (LiCl-KCl) salts were recovered for reuse with a high regeneration ratio. Figure 14 shows a brief process diagram of the waste salt treatment process currently being developed by KAERI.

(1) LiCl Waste Salt Regeneration Process. For the reuse of LiCl waste salt, Groups I and II fission products have to be separated from the LiCl waste salt. To separate Groups I and II fission products such as Cs and Sr, chemical agents addition method and ion-exchange method were tested [15–18]. However, the results showed that simultaneous separation of Cs and Sr from the LiCl molten salt via various chemical agents or ion-exchange by zeolite was nearly unlikely [19]. To overcome these difficulties for the separation of both cesium and strontium, a layer crystallization process, which uses the solubility difference of impurities (FPs in this case) between a solid (=crystal) and a melt phase was introduced.

Layer crystallization is a very simple process, which uses cooled plates immersed in a melt for crystal formation, where a crystal grows as a compact crystalline layer on a cooling
Table 1: Composition of pretreatment process wastes (10 ton/MTU).

<table>
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<th>As generated</th>
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<th>After treatment</th>
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<tbody>
<tr>
<td></td>
<td>Mass (kg)</td>
<td>Composition (%)</td>
<td>Volume (m³)</td>
<td>Composition (%)</td>
</tr>
<tr>
<td>Cladding hulls</td>
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<td>70.3</td>
<td>2.50</td>
<td>35.7</td>
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<td>Fuel Hardware</td>
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<td>29.7</td>
<td>4.50</td>
<td>64.7</td>
</tr>
<tr>
<td>Sum</td>
<td>3.91</td>
<td>100</td>
<td>7.00</td>
<td>100</td>
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Electro-reduction process
- LiCl waste salt
- LiCl purification
- Concentrated LiCl waste
- Residual waste solidification
- Solidification system

Electro-refining/winning process
- LiCl-KCl waste salt
- LiCl-KCl purification
- Selective precipitation
- Distillator
- Layer crystallizer
- Solid LiCl salt detachment device
- Final wasteform (SAP and ZIT wasteform)

Figure 14: Schematic diagram of waste salt treatment process of KAERI.

Figure 15: Photo of PRIDE engineering-scale LiCl waste salt treatment apparatus.

Surface. With the laboratory-scale layer crystallization apparatus (maximum batch size: 4 kg/batch), about 80–90% of LiCl salt containing small amount impurities, Groups I and II fission products can be recovered and the remaining LiCl in the crystallization furnace with most of impurities is transferred to the immobilization process to fabricate the final waste form.

Figure 15 shows the PRIDE engineering-scale LiCl waste salt treatment apparatus of which the maximum capacity is 25 kg-LiCl/batch composed of a layer crystallization and a solid LiCl salt detachment device.

(2) Eutectic (LiCl-KCl) Waste Salt Regeneration Process. Rare earth chloride elements involved in LiCl-KCl eutectic waste salt were converted to their molten salt-insoluble precipitates in the oxidative precipitation step by an oxygen gas sparging method. After a full precipitation of these lanthanide precipitates, an eutectic waste salt is separated into two layers: an upper pure (or purified) salt layer and a lower precipitate layer. The upper pure salt layer can be mechanically separated from the precipitate layer, where the separated pure salt layer could be reused (1st pure salt recovery). Then, the adhering eutectic salt involved in the precipitate layer was separated and recovered in the distillation/condensation step (2nd pure salt recovery). Finally, all the remaining rare earth oxides or oxychlorides were fabricated as a final waste form in the immobilization step.

The PRIDE engineering-scale rare earth separation apparatus (maximum batch size: 25 kg-eutectic salt/batch) that consists of three devices, a rare earth precipitation, a solid eutectic salt detachment, and a layer separation, is shown in Figure 16.

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(3) Residual Waste Solidification Process. In order to treat waste salt for final disposal, a dechlorination approach was adapted, where metal chloride waste was dechlorinated and thermally stabilized by using the synthetic inorganic composite SAP composed of SiO$_2$, Al$_2$O$_3$, and P$_2$O$_5$, and its resultant product was sintered with a conventional borosilicate glass to obtain a monolithic wasteform.

The rare earth precipitates from the eutectic waste salt treatment process must be immobilized to a chemically stable solid wasteform for long-term storage in a geological environment. To immobilize rare earth oxide/oxychlorides, the solidifying matrix, ZIT, composed of Zn$_2$TiO$_4$·CaHPO$_4$·SiO$_2$·B$_2$O$_3$ has been developed.

To realize the immobilization method, the lab-scale solidification process which consists of crushing, pulverizing, mixing/reacting and sintering equipment was used. Up to now, the unit equipments have been developed to obtain a proper performance and their processing conditions have been investigated to abstract the scale-up factors.

Table 2 shows the chemical durability of wasteforms and some physical properties for two wasteforms, SAP and ZIT wasteform. Leach-resistance of radionuclides was comparable to other radioactive wasteforms and the wasteforms had similar physiochemical properties, compared with silicate and phosphate glassy waste form [20].

3. Pyroprocess-Integrated Inactive Demonstration Facility

PRIDE has been constructed and operated as an engineering scale demonstration facility for pyroprocess studies [21]. The objective of PRIDE is to support integrated pyroprocessing demonstration and equipment development which are essential to realize the pyroprocessing. PRIDE is an integrated pyroprocessing system with unit reactor of 50 kgHM/batch capacity. It will use depleted uranium with surrogate materials to show integrated performance and scale-up issues of full-spectrum pyroprocessing technology.

PRIDE is a three-story building and it has a large-scale argon atmosphere cell (40.3 m length, 4.8 m width, 6.4 m height) at the second floor. The argon cell is equipped with cell operation equipments (small and large transfer systems, windows, cell lights, feed-through, etc.) and utilities. All process equipments are remotely operated and maintained by using remote handling systems such as 34 pairs of mechanical “Master Slave Manipulators” (MSMs), a “Bridge transported Dual arm Servo Manipulator” (BDSM), and an overhead crane [22]. Not only the remote handling systems but also all process equipments and devices for the use in the PRIDE were designed as an assembly of modules considering easy maintenance. The special interface between modules and equipments was devised to assemble and disassemble the modules.

The argon utility system of PRIDE was also developed for maintaining the cell atmosphere within the desired operating parameters (concentration of oxygen and moisture <50 parts per million (ppm), respectively, the pressure (~10 mmAq~100 mmAq) and the temperature (28~40°C)). The argon utility system consists of argon cell recirculation and cooling system, the purification system, and the relief system. The recirculation and cooling system controls the pressure and temperature of the cell within the above operation conditions, and the purification system removes oxygen and moisture from the cell.

Recently, major process equipments such as electrolytic reducer, cathode processor, electrorefiner, salt transfer system, salt distiller, LCC type electrowinner, residual actinide recovery apparatus, cadmium distiller, and waste molten salt treatment apparatus were installed in the PRIDE argon cell (Figure 17), and voloxidizer, salt waste form fabrication system, UCl$_3$ fabrication system, and uranium ingot melting furnace were installed at the first floor of PRIDE. The initial operation test of the argon system and evaluation of remote operability of cell equipment are being performed.

4. Conclusion

KAERI has developed an environment-friendly and proliferation resistant pyroprocessing for spent fuel treatment to recover useful materials such as uranium, plutonium, and reduce the volume and radiotoxicity of spent fuel. Through the last decade, R&D, some innovative technologies such as an advanced voloxidation, a high-capacity electrolytic reduction, a high-throughput electrorefining with a graphite cathode, a mesh-type liquid cadmium cathode electrowinning, and waste salt regeneration by crystallization method, have
been tested successfully. PRIDE facility has been constructed at KAERI and advanced technologies in engineering-scale are planned to be demonstrated in PRIDE. The PRIDE will verify the performance of equipments, interaction between units, remote operability, facility management system, and process monitoring. For the commercialization of pyroprocessing, knowledge of a scale-up is essential and PRIDE engineering-scale demonstrations compared to laboratory-scale tests which has been studied to date will give the scale-up information especially on the batch size effects and process yields affected by the equipment size. Therefore, the PRIDE engineering-scale tests will evaluate the technical feasibility, economic feasibility, and nonproliferation acceptability of pyroprocessing.

References


