Asian Nuclear Prospects 2012

Development of Transport Technologies for High-temperature Fluid in Pyrometallurgical Reprocessing

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Abstract

The development of the engineering technology necessary for pyrometallurgical reprocessing is a key issue for industrial realization. The development of high-temperature transport technologies for molten salt and liquid cadmium is crucial for pyrometallurgical processing; however, there have been very few transport studies on high-temperature fluids. In CRIEPI, the transport technologies of molten salt and liquid cadmium have been developed for several types of transports methods. Based on the results of transport tests, engineering scale process equipments connected each other with the transport technology have been developed, and installed in the Ar atmosphere glove box for integrated tests.

Pyrometallurgical Reprocessing; Electrorefiner; Counterflow-Extraction Equipment; Zeolite Column; High-Temperature Fluid, Transport

1. Introduction

Pyrometallurgical reprocessing has been actively developed as part of the advanced nuclear fuel cycle by many countries, because it has several advantages such as producing less environmentally harmful nuclear waste (minor actinide recycling), improved economic efficiency, and intrinsic proliferation-resistance[1]. After March 11th 2011, the nuclear reactor in Fukushima Daiichi nuclear power plants had accidents of melt-down by the huge earthquake and the tsunami. Large amounts of

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debris were generated in the reactors and then were still remained. The debris will be taken from the reactor in the future, however, it will be difficult to dispose from the debris to the stable material by the present reprocessing (PUREX). The pyrometallurgical reprocessing might be adaptable to separate actinides from the debris, because it has a high performance by the combination of the electrorefing and the electroreduction in the molten salt. The pyrometallurgical reprocessing has large potentials of minor actinide recycle, disposal of debris, and compact plant. Central Research Institute of Electric Power Industry (CRIEPI) has been studying the pyrometallurgical reprocessing of metallic fuels since 1987 and has carried out joint studies with various organizations such as ANL, INL, JRC-ITU, and JAEA. The feasibility of pyrometallurgical reprocessing has been demonstrated through many laboratory-scale experiments. [2] [3] Development of the engineering technologies required for pyrometallurgical reprocessing is a key issue for its industrial realization. The processing equipment should have the functions of easy maintenance, reliability, criticality safety, and enable the handling of products. In pyrometallurgical reprocessing, products have so far been cooled before being transported to the next process. The cooling and heating times of high-temperature fluids should increase with the increased amount of fluid in engineering-scale equipment. If a high-temperature fluid can be directly transported to the next process, the material-handling time can be cut, irrespective of the equipment size. Although large-scale transport technology for high-temperature fluids has been developed for use in molten salt reactors [4] and sodium cooled fast reactors [5], small-scale transport technology suitable for pyrometallurgical reprocessing has not yet been developed. In this study, the transport characteristics of molten salt and liquid cadmium were evaluated using several types of transports technologies; gravitation, centrifugal pumps, a reciprocating pump, a suction pump, and gas pressure transport.

2. Transport technologies of high-temperature fluids in pyrometallurgical reprocessing

Pyrometallurgical reprocessing g mainly consists of electrorefining, cathode processing, and injection casting, followed by salt treatment (waste management), as shown in Fig. 1. Electrorefining is carried out to dissolve spent fuel and to recover the actinides on the cathodes. Uranium metal and uranium-plutonium-cadmium alloy are deposited on the cathodes. These deposits are entrained with salt and cadmium (Cd), respectively. In cathode processing, the salt or Cd is separated from the deposit by distillation. Injection casting produces the metallic fuel by casting a uranium-plutonium-zirconium alloy. The salt treatment system separates the fission products (FPs) from the molten salt in the electrorefiner. The removed FPs are occluded with zeolite and are solidified with a glass matrix. In pyrometallurgical reprocessing, the technologies for the transport of high-temperature fluids are applied to the handling of materials in the electrorefining, the cathode processing, and the salt treatment system (the reductive extraction and the zeolite column). The molten salt with FPs is transported from the electrorefining to the zeolite column through the reductive extraction. Then the decontaminated molten salt is transported from the zeolite column to the electrorefining by the back extraction. As another example of molten salt transport, the molten salt distilled from the solid cathode is transported from the condenser in the cathode processing to the electrorefining. The liquid-Cd- actinide alloy is transported from the Cd cathode to the distillation crucible of the cathode processing, and after distillation, the pure Cd is transported back to the Cd cathode. As another example of liquid Cd transport, a Cd-Li alloy is employed as an extraction solvent in the reductive extraction, and the molten salt and Cd alloy flow in opposite directions while contact with each other.

For molten salt transport, the transport capacities among the above processes were evaluated in a Feasibility study based on a commercialized fast reactor cycle system. [6] 67 dm^3 of molten salt with FPs must be transported from the electrorefining to the reductive extraction every 6 days. If the molten salt is transported to the reductive extraction within 1 hour, the flow rate can be calculated to be approximately 1 dm^3/min. In the reductive extraction, if the molten salt can be treated within 19 hours, the flow rate will be approximately 0.06 dm^3/min. In the zeolite column, if 56 dm^3 of the treated salt in the reductive extraction can be treated in 5 batches and within 1 hour, the flow rate will be approximately 0.6 dm^3/min.

In the electrorefining, about 150 kg of the solid cathode deposit is produced each day and the amount of adhered salt has been evaluated to be 20% of the deposit. If the distilled salt can be transported from the cathode processing to the electrorefining within 1 hour, the flow rate will be approximately 0.3 dm^3/min. For liquid Cd transport, the flow rate and the head between the electrorefining and the cathode processing were evaluated in the same feasibility study. [6] The Cd cathode recovers 5.6 kg of actinides every day. The concentration of actinides must be less than the saturation limit in order to transport the Cd-actinide alloy in the liquid phase. The required amount of Cd would be 35 dm^3 since the actinide has a concentration of 2 wt% in Cd. If this alloy is transported to the cathode processing within 1 hour, the flow rate will be approximately 0.6 dm^3/min. In the reductive extraction, the flow rate of the Cd alloy was evaluated to be the same as that of the molten salt.
The head of the transport was estimated to be within 3 m on the basis of the height of all equipments.
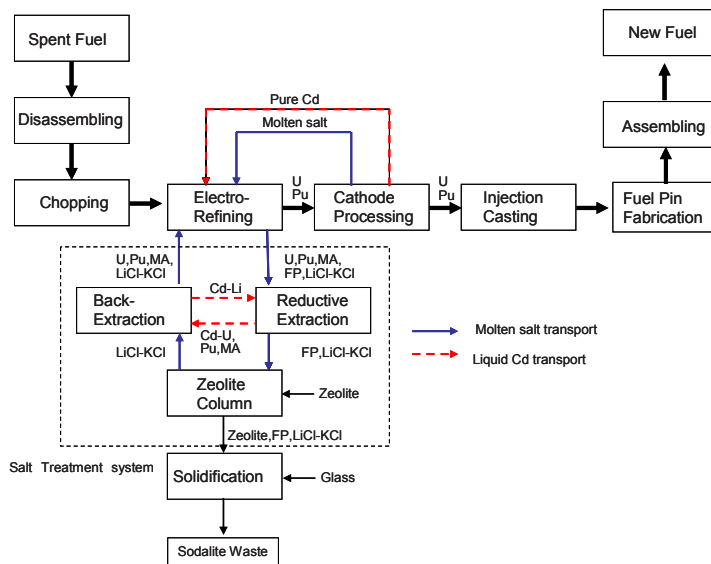

Fig.1 Flow diagram of pyrometallurgical reprocessing.

3. Review of high-temperature fluid transport technologies

The transport technologies of high temperature fluid reviewed and the transport methods were selected for these transport conditions. Several transport methods, such as gravity and pump (non machinery and machinery), are applicable for transporting high-temperature fluids. Gravitational transport was considered as favorable methods requiring little maintenance, because it requires no moving parts and it is adaptable transportation method among the processes. A suction pump, gas pressure transport and the electromagnetic pump were classified to non machinery pumps, as shown in Table 1. These transports were considered as favorable methods requiring little maintenance, because they require no moving parts. However, the electromagnetic pump was not applicable on the above transport conditions, and suction pump and gas pressure transport were considered as the applicable transport of molten salt and liquid Cd. In the suction pump, the theoretical maximum head was calculated to be < 1.3 m on the liquid Cd and < 6.5 m on the molten salt, respectively. The machinery pump is favorable for controlling the flow rate and head, and the centrifugal pump and the reciprocating pump were classified to machinery pump, as shown in Table 1. The transport capacity of molten salt was reported to be from 4 to 5.5 dm^3/min against a 1.5 m head [4]. The molten salt transport experiment was carried out by the reciprocating pump [7]. The performance of the reciprocating pump was enough unproven to transport the high temperature fluids, however it has a favorable feature that the fluid is transported at a constant and

low flow rate. The performances of the centrifugal pump were satisfactory developed by the molten salt reactor and the capacity of the pump can be optimized by the new design for the pyrometallurgical reprocessing. The molten salt and the liquid Cd have been studied to be the applicability of the favorable transport methods, such as the gravitation transport, the centrifugal pumps, the reciprocating pump, the suction pump, and the gas pressure transport.

Table 1 Comparison of the pump-based transport methods for high-temperature fluids.

Type		Fluid	Flow rate	Head
Non machinery pump	Suction pump	Cd	-	< 1.3m*
		Salt	-	< 6.5 m*
	Gas pressure transport	Cd	-	-
		Salt	-	-
	Electromagnetic pump	Liquid Metal [5]	< 227 dm3/min	1.7 MPa
		Salt	-	-
Machinery pump	Centrifugal pump	Cd	-	-
		Salt [4]	4-5.5 dm^3/min	1.5 m
	Reciprocating pump	Cd	-	-
		Salt [7]	14-2 dm^3/min	0.02-0.07 MPa

*The value was calculated from the vacuum pressure and density.

4. Development of molten salt transport technologies

4.1 Gravitation transport

Molten salt was transported down 435 mm from the supply tank to the recovery tank by gravity. The transport tube had a 10.1 mm inside diameter. The rate of molten salt transport by the gravitation was well controlled between 0.5 and 6 dm^3/min by adjusting the orifice valve opening, as shown in Fig. 2. [8]
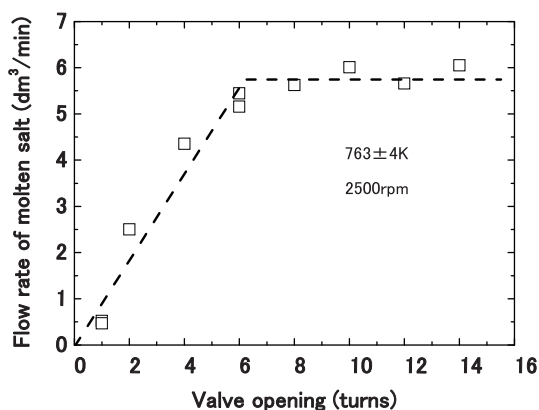

Fig. 2 Relationship between the molten salt flow rate controlled by gravity and amount of valve opening [8].

When the valve opening was six turns, the value of the flow rate was scattered within ±3%. The flow rate of molten salt exhibited good reproducibility at the same valve opening over several repeated experiments. Also, the function of the orifice valve was maintained during 89 transport experiments.
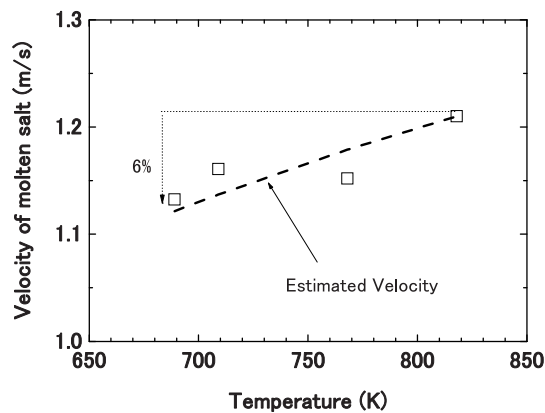

Fig. 3 Relationship between the molten salt velocity and temperature. (valve opening: 6 turns)

The relationship between the velocity of the molten salt and the temperature is shown in Fig. 3. [8] The molten salt velocity decreased by approximately 6% when temperature decreased. The molten salt temperature has little effect on the flow rate at operating temperatures from 698 to 818 K during gravitation transport. The broken line in Fig. 3 is the velocity estimated by extrapolating the relationship between kinetic viscosity and temperature. [8] This line was in agreement with the relationship between the experimentally obtained velocity and the temperature. The velocity of the molten salt can thus be estimated from the relationship between the kinetic viscosity and temperature.

4.2   Centrifugal pump

Molten salt was transported from the supply tank to the recovery tank by a centrifugal pump (Sanwa Hydrotech Co., Osaka). [8] The performance of the pump was measured for heads from 0.96 to 1.48 m. The flow rate is plotted against the rotation speed of the centrifugal pump for heads of 0.96 m and 1.48 m in Fig. 4.
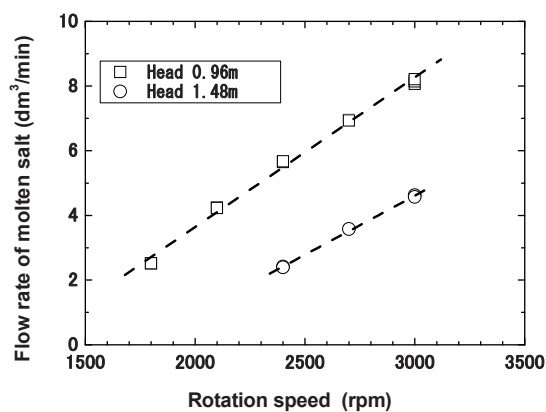

Fig. 4 Relationship between the average flow rate and the rotation speed of the centrifugal pump [8].

The average flow rate of the molten salt was controlled between 2.5 and 8 dm³/min by changing the rotation speed in the case of a 0.96 m head. When the rotation speed was 3000 rpm, the value of the flow rate was scattered within ±1%. The flow rate of the molten salt exhibited good reproducibility at the same rotation speed over several repeated experiments. In the case of a head of 1.48 m, the molten salt was not able to flow at a rotation speed of 2100 rpm, owing to the large pressure loss. The average flow rate of molten salt was controlled between 2.5 and 4.6 dm³/min at rotation speeds from 2400 to 3000 rpm at a head of 1.48 m.

4.3 Gas pressure

Molten salt was transported from the salt reservoir to the crucible through the zeolite column by gas pressure. [10] The velocity of molten salt was increased with increasing argon gas pressure, as shown in Fig. 5. When the velocities of molten salt and water through a 30 cm column were compared as shown in Fig. 5, the velocity of the molten salt was found to almost be in agreement with that of water at an argon pressure of 0.3 MPa. This was considered to be because the kinetic viscosity of the molten salt (v=1.4 μm^2/s at 773 K) was the almost same as that of water (v=1.0 μm^2/s at 293 K). Also, the velocity of molten salt through a 10 cm column became larger than that of molten salt through the 30 cm column at an argon gas pressure of 0.3 MPa, as shown in Fig. 5. The slope of the relationship between the velocity and pressure for the 30 cm column was smaller than that for the 10 cm column, because the pressure loss of the 30 cm column was larger than that of the 10 cm column. The flow rate of the molten salt through the column was well controlled by adjusting the gas pressure.
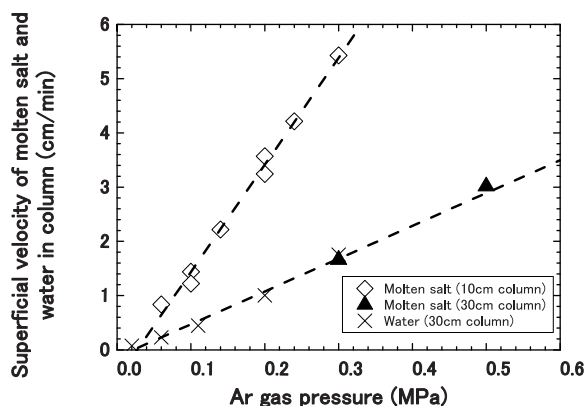

Fig. 5 Relationship between superficial velocities of molten salt and water passing through column filled with zeolite 4A powder and argon gas pressure [10].

4.4 Reciprocating pump

A reciprocating pump for the transport in the case of low flow rate (<1 dm³/min) and a variable pump head (<3 m) have been developed for application to reductive extraction and the electrorefining. Before the transport of molten salt, water and liquid gallium (Ga) were transported by a reciprocating pump at approximately 323 K. The relationship between the flow rate and the reciprocating speed of reciprocating pump is shown in Fig.6. The flow rate of the water increased with the increasing reciprocating speed, and the flow rate of liquid Ga similarly increased up to a reciprocating speed of 30 cycle/min; however, the flow rate became lower than the predicted flow rate at 60 cycle/min. This was considered to be due to the driving of the piston being delayed by more than 0.5 s because the viscosity of Ga (1.9 mNs/m²) was larger than that of water (0.55 mNs/m²). If the reciprocating pump normally operates at 60 cycle/min, the driving pressure should be larger than 0.4 MPa. The molten salt at a

temperature of approximately 773 K was transported from the supply tank with the upper tank of a 2.5 m head by the same type of the reciprocating pump. The flow rate was controlled between 0.094 and 0.175 dm^3/min by adjusting the reciprocating speed, as shown in Fig.6. When the reciprocating speed was 15 cycle/min, the value of the flow rate was scattered within ±1%. The flow rate in molten salt exhibited good reproducibility at the same reciprocating speed over several repeated experiments.
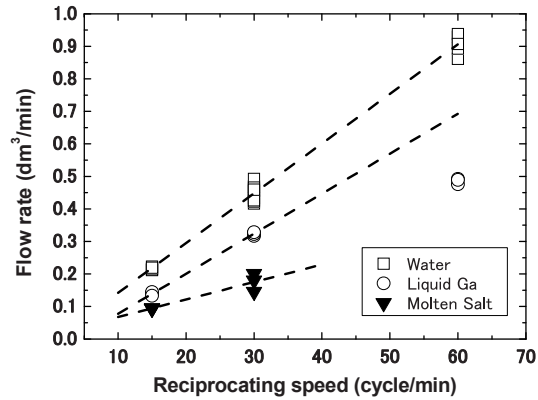
Fig. 6 Relationship between the flow rate and the reciprocating speed of the reciprocating pump.

5. Development of liquid cadmium transport technologies

5.1 Gravitation transport

Liquid Cd was transported 280 mm between the supply tank and recovery tank by gravity. The flow rate of liquid Cd transport was reported to be well controlled between 0.8 and 2.6 dm^3/min by adjusting the Swagelok valve opening, as shown in Fig. 7. [11] When the valve opening was two turns, the value of the flow rate was scattered within ±8%. The scatter for liquid Cd was the larger than that for molten salt, because the Swagelok valve was a ball valve and had poorer controllability than the orifice valve used for molten salt transport. The broken line in Fig. 7 shows the flow rate calculated using the flow coefficient, which was in good agreement with the experimental data. The temperature of liquid Cd has little effect on the flow rate at an operating temperature from 698 to 756 K during gravitation transport.
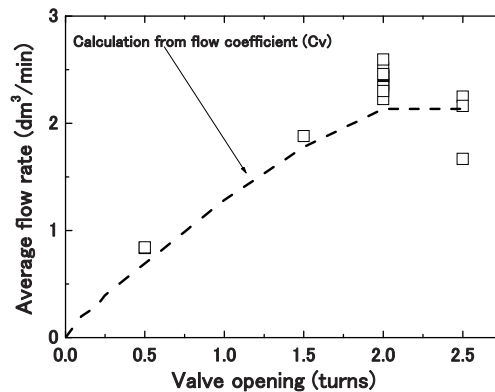
Fig. 7 Relationship between the liquid Cd flow rate controlled by gravity and amount of valve opening [11].

5.2   Centrifugal pump

Liquid Cd was transported from the supply tank to the recovery tank against a 1.63 m head using a centrifugal pump (Sanwa Hydrotech Co., Osaka). [11] The average flow rate is plotted against the rotation speed of the centrifugal pump and the valve opening in Fig. 8. The average flow rate of the liquid Cd was controlled between 1.0 and 2.5 dm³/min by varying the rotation speed with the valve opened by 2.5 turns. When the rotation speed was 3000 rpm and 2.5 turns, the value of the flow rate was spattered within ±8%. The variation of liquid Cd was larger than that of molten salt, because in the transport of Cd the valve was opened after starting the pump. The flow rate of liquid Cd exhibited satisfactory reproducibility at the same rotation speed and the same valve opening over several repeated experiments. The temperature of liquid Cd had a negligible effect for transport by the centrifugal pump in the operating temperature range from 683 to 755 K.
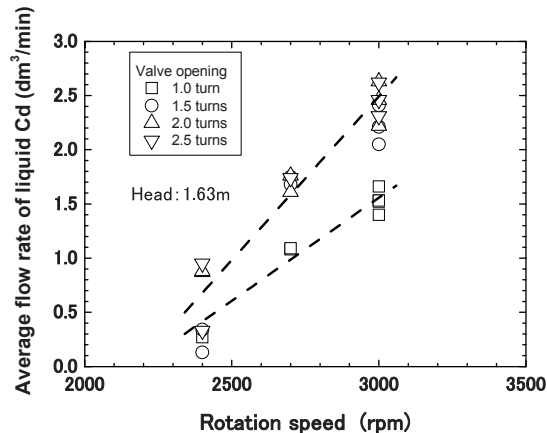

Fig. 8. Relationship between the average flow rate and the rotation speed of the centrifugal pump [11].

5.3   Suction pump

The theoretical maximum head of liquid Cd at 773 K was calculated to be approximately 1.3 m in the case of using a suction pump. Thus, it would be difficult for the liquid Cd to be transported by more than 1 m in the vertical direction, because the transport tube suffers a pressure loss. Liquid Cd was pumped upward from a crucible that was 0.93 m below the vacuum tank using a suction pump. [11] Figure 9 shows the relationship between the amount of Cd in the crucible and the amount of Cd at 730 K transported by the suction pump. The slope in Fig. 9 was 0.55 for all positions along the suction tube (8 - 43 mm), where the position is the distance from the bottom of the crucible to the inlet of the suction tube. The amount of Cd transported was well controlled by adjusting the amount of Cd in the crucible and the position of the suction tube, as shown in Fig.9. When the position of the suction tube was 23 mm, the variation of the amount of Cd transported was scattered within ±2%. The relationship between the amount of the liquid Cd transported and the amount of Cd in the crucible exhibited good reproducibility at each position of the suction tube over several transport experiments. The flow rate varied from 0.8 to 3.5 dm³/min, even though the valve was opened as quickly as possible to minimize the pressure loss of the valve. For the transport of liquid Cd using the suction pump, it is difficult to control the flow rate by adjusting the valve.
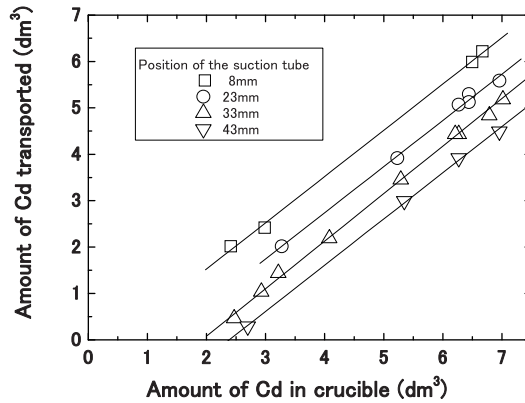
Fig. 9 Relationship between the amount of Cd in the crucible and the amount of Cd at 730 K transported by the suction pump. [11]

5.4 Reciprocating pump

Liquid Cd at approximately 773 K was transported from the supply tank to the upper vessel with a 1.5 m head by the same type of reciprocating pump for the molten salt transport. The relationship between the flow rate of Cd / Cd alloy and the reciprocating speed of reciprocating pump is shown in Fig.10. The flow rate was controlled between 0.2 and 0.8 dm^3/min by adjusting the reciprocating speed. The flow rate was controlled between 0.2 and 0.8 dm^3/min by adjusting the reciprocating speed. Both flow rates between Cd and Cd alloy similarly increased with the increasing reciprocating speed. In the general, the flow rate of Cd-Gd alloy became smaller than that of Cd transported by the centrifugal pump and the gravitation, because the viscosity of Cd-Gd alloy was larger than that of Cd. However, the flow rate of Cd alloy was almost same as that of Cd by the reciprocating pump, because the performance of the reciprocating pump is little influenced by the viscosity [5]. When the reciprocating speed was 30 cycle/min, the value of the flow rate was scattered within ±10%. The flow rate in liquid Cd exhibited good reproducibility at the same reciprocating speed over several repeated experiments.
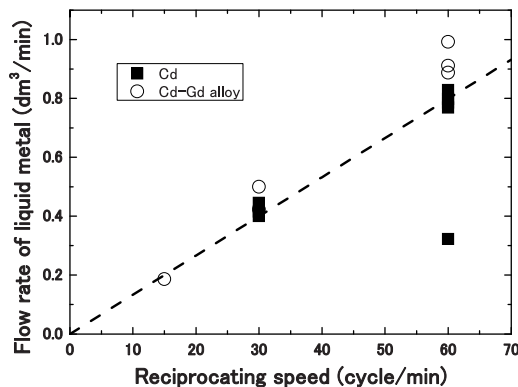

Fig. 10 Relationship between the flow rate of Cd/ Cd alloy and the reciprocating speed of the reciprocating pump.

6.   Applications of high-temperature fluid transport in pyrometallurgical reprocessing

6.1   Electrorefiner with high-temperature fluid transport system

An engineering-scale electrorefiner with a 780 mm diameter and a depth of 800 mm was designed. Approximately 150 kg of LiCl-KCl eutectic salt was loaded into the electrorefiner. Gadolinium (Gd) was used to simulate the actinides. The concentration of Gd in the molten salt was between 1 and 4 wt%. The amount of Gd metal in the anode was about 2 kg. The crucible of the Cd cathode had a 300 mm diameter and was 160 mm deep. The first electrorefiner with a high-temperature fluid transport system was fabricated in a large argon glove box, as shown in Fig.11. [12] The transport of Cd-Gd alloy from the Cd cathode crucible to the distillation crucible enabled a good material balance. The handling time between the electrorefiner and the cathode processor in this system was shortened to within 1 hour. These results suggest that the electrorefiner with the high-temperature fluid transport system can be applied to pyrometallurgical reprocessing. [13] The first electrorefiner with high-temperature fluid transport system had 5 tanks and Cd and Cd alloy was transported at five times in a batch. The first electrorefiner has a problem that the residue of liquid metal in the tank was larger than the amount of transported Cd alloy. In a transport batch, approximately 20% of Cd-Gd alloy in the Cd cathode crucible was transported to the distillation crucible.
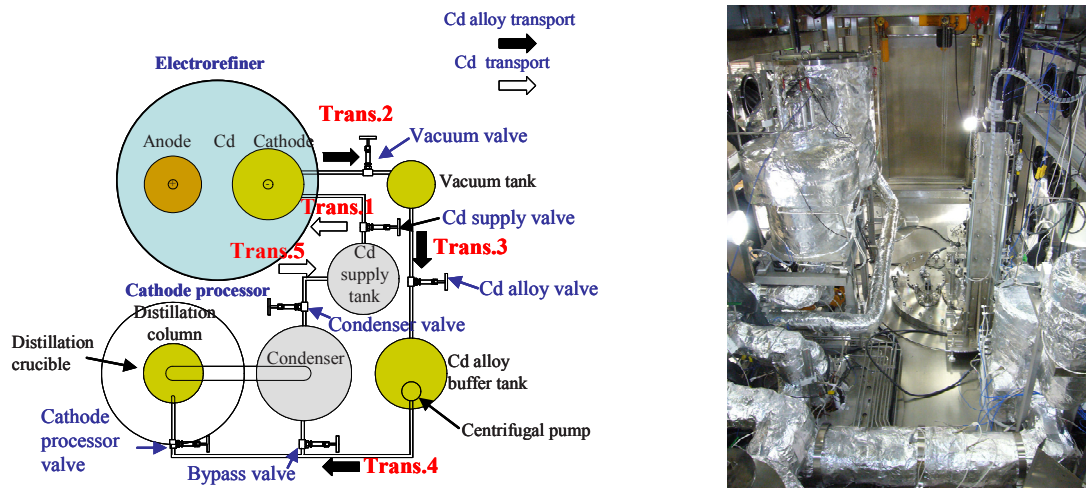
(a) Diagram of the electrorefiner system   (b) Photograph of the electrorefiner

Fig. 11 First electrorefiner with high-temperature fluid transport system. [13]

The new electrorefiner with high-temperature fluid transport system will be designed in order that a maximum residue of Cd alloy was minimized. On the basis of the high-temperature molten salt and liquid Cd transport experiments, the new high-temperature fluid transport system was redesigned, as shown in Fig. 12. This system has twice transport at supply of Cd and recovery of Cd alloy, and the residue of Cd alloy in the crucible was minimized by controlling the position of the reciprocating pump. Approximately 88 % of Cd-Gd alloy was transported to the distillation crucible, because Cd-Gd alloy was directly transported from the Cd cathode crucible to the distillation crucible. The flow rate of the Cd alloy was controlled between 0.1 and 0.9 dm^3/min by adjusting the reciprocating speed. The decreasing amount from the Cd crucible was in agreement with the increasing amount of the distillation crucible, when the Cd alloy was transported by the reciprocating pump in the new system. The flow of Cd was supplied between 0.5 and 3 dm^3/min by adjusting valve opening. These results was suggested that the new transport system was especially better controllability of the flow rate than first system. In addition, the

handling time between the electrorefiner and the cathode processor in the new system was shortened within 15 minute. The new high-temperature fluid transport system has better transport performance than the first transport system.
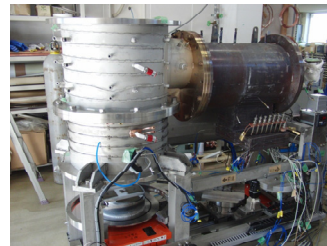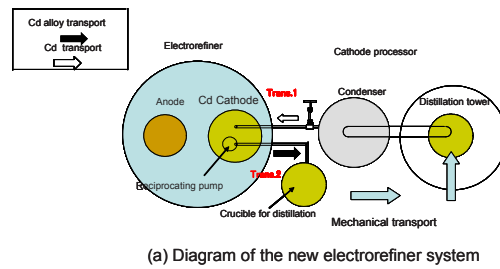

(a) Diagram of the new electrorefiner system

(b) Photograph of the electrorefiner   (c) Photograph of the cathode processor

Fig. 12 New electrorefiner with high-temperature fluid transport system.

6.2   Counter flow extraction

Counter flow reductive extraction equipment has been developed by the application of a feeding float unit. [14] [15] This system for transporting the molten salt and liquid Cd was designed so that these fluids could be transported at the low flow rate required. The practical extraction equipment will be designed to be large size by using the feeding float system, because volume of the float must be the same as that of the transported fluid.

(a) New feed system for Counter flow extraction   (b) Photograph of counter flow extraction
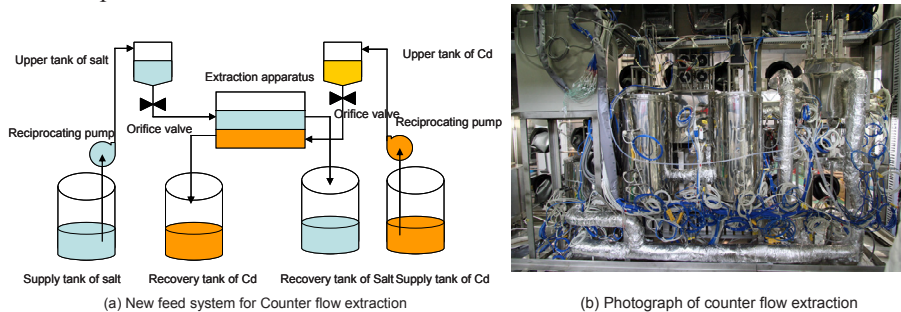
Fig. 13 The new feed system of molten salt and liquid Cd for use in reductive extraction equipment.

So, on the basis of the high-temperature molten salt and liquid Cd transport experiments, the new high-temperature fluid transport system was redesigned, as shown in Fig. 13.In the system, molten salt and liquid Cd were pumped from the supply tank up to the upper tank by reciprocating pumps. Next the orifice valves of the upper tanks containing the molten salt and liquid Cd were opened to allow both liquids to flow into the extraction apparatus. The flow rate of the molten salt was controlled by the

opening of the orifice valve to be between 0.02 and 0.13 dm^3/min, and the flow rate of the liquid Cd was similarly controlled between 0.03 and 0.13 dm^3/min. These flow rates satisfied the requirement of a sufficiently slow speed and a continuous flow of the reductive extraction equipment.

This equipment was designed and constructed as a system combining the above transport system and the extraction apparatus.

6.3   Zeolite column system

On the basis of the previous described transport tests, the zeolite column system was newly designed and fabricated in an argon glove box. The system has a flow system through two columns basis on gas pressure transport, as shown in Fig.14. The flow rate can be controlled by the gas pressure. The flow rate was obtained from the measured weight of the drained molten salt and the level meter in the supply tank. The performance of the zeolite column system will be measured in a future study.
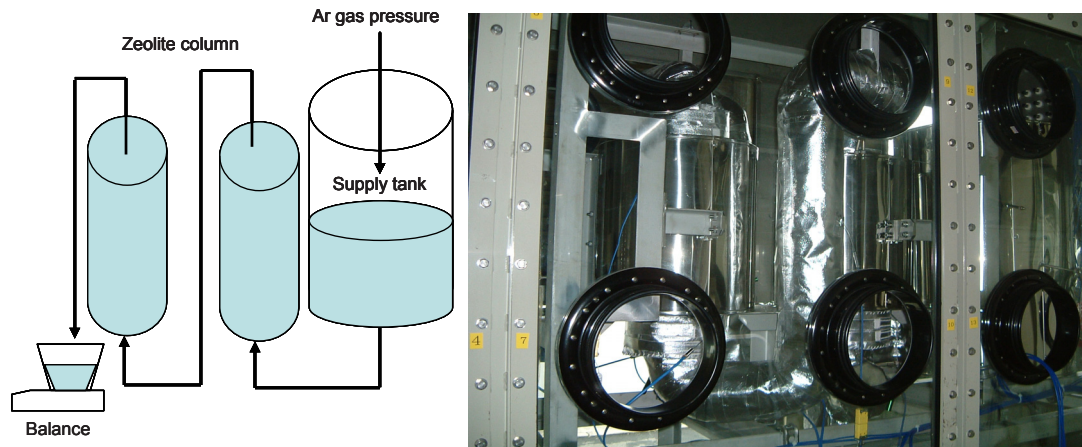
(a) Flow diagram of zeolite column system   (b) Photograph of zeolite column system
Fig. 14 Engineering-scale zeolite column system.

7.   Conclusion

The following transport behaviors of molten salt and liquid Cd were determined by various transportation experiments.

(1) In the gravitational transport of molten salt and liquid Cd, the flow rate was well controlled by adjusting the valve opening. The molten salt was transported at a flow rate from 0.5 to 6 dm^3/min, and the liquid Cd was transported at a flow rate from 0.8 to 2.6 dm^3/min. The flow rates of the fluids exhibited satisfactory reproducibility at the same valve opening.

(2) Gas pressure transport was an appropriate means of transport of the molten salt through the column. The pressure loss of the column increased with increasing column length, and a velocity of 3 cm/min in the case of using a 30 cm column was required at high gas pressure of 0.5 MPa. The velocity of the molten salt was the same as that of water, because the kinetic viscosity of molten salt is almost the same as that of water.

(3) A suction pump enabled good reproducibility of liquid Cd transport. The theoretical maximum head of liquid Cd was calculated to be approximately 1.3 m in the case of suction pump at 773 K. It is difficult to control the flow rate by adjusting the valve.

(4) A centrifugal pump can be applied to transport the molten salt and liquid Cd. The flow rate was

controlled by adjusting the rotation speed and the valve opening. Molten salt at a temperature of 773 K was transported at a flow rate from 2.5 to 4.6 dm^3/min at a 1.5 m head, and liquid Cd metal at 773 K was transported at a flow rate from 1.0 to 2.5 dm^3/min at a 1.6 m head.

(5) A reciprocating pump was suitable appropriate for the transport of molten salt and liquid Cd at a low flow rate. The flow rate of the molten salt was controlled between 0.094 and 0.175 dm^3/min by adjusting the reciprocating speed, and the flow rate of the liquid Cd was controlled between 0.2 and 0.8 dm^3/min by adjusting the reciprocating speed.

On the basis of the results of transport tests, we have developed interconnected engineering-scale process equipment (a electrorefiner, counterflow extraction, and a zeolite column system) as a new transport technology, which was installed in an argon glove box for integrated tests.

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