Actinides Separation from Lanthanides Using a Liquid Ga Electrode in LiCl-KCl Melts

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A novel pyroprocess is proposed to recover actinides from spent nuclear fuels, in which a liquid Ga is used as the key electrode material to achieve a high actinide/lanthanide separation efficiency. To evaluate the proposed pyroprocess, it is necessary to clarify the separation performance in the system of LiCl-KCl/liquid Ga electrode. In this study, an electrochemical actinides recovery was conducted by galvanostatic electrolysis of the liquid Ga electrode in LiCl-KCl melts containing both actinides (U, Pu and Am) and lanthanides (Ce, Nd, La and Gd) chlorides at 773 K. The obtained Ga alloy after the electrolysis was analyzed quantitatively to calculate the separation factors of Pu and Am based on Ce. The calculated separation factors in LiCl-KCl/liquid Ga electrode system were more than 10 times higher than those in LiCl-KCl/liquid Cd system.

1. Introduction

Pyroprocess which utilizes molten salts and liquid metals as the reaction media has been developed as one of the promising processes for reprocessing various kinds of spent nuclear fuels, such as metal alloys, oxides, nitrides and so on [1, 2]. Furthermore, the pyroprocess is expected to be applied in the concept of partitioning and transmutation [3]. In this concept, long half life nuclides such as TRUs (Pu, Np, Am and Cm) are recovered from spent nuclear fuels and high level liquid waste (HLLW) to be transmuted to stable or short half life nuclides. Thus, the reduction of an environmental burden of spent nuclear fuels is expected to be accomplished, which is a crucial challenge in the world. Since spent nuclear fuels and HLLW contain fission products as well, such as lanthanides, alkali metals, alkaline earth metals and noble metals, a recovery of TRUs and a separation from the fission products must be achieved at the same time in this concept. It is especially important to separate TRUs from lanthanides because chemical properties of lanthanides are similar with those of actinides.

In the previous study [4], the authors proposed a novel pyroprocess which can be applied in the partitioning and transmu-

tation concept, where a liquid Ga metal was used as the key electrode material in molten chlorides. As shown in Figure 1, the proposed pyroprocess consists of two steps; actinide/lanthanide separation step and actinide recovery step. At actinide/lanthanide separation step, a metallic basket loading spent nuclear fuels is immersed in LiCl-KCl melts and is used as the anode. Then, the dissolution of actinides (An) contained in the fuels proceeds at the anode,

$$An \rightarrow An^{3+} + 3e^- \qquad (1)$$

The fission products of lanthanides, alkalis and alkaline earths dissolve at the same time to form their cations and accumulate in the melts, while noble metal fission products remain in the anode basket to be separated from actinides. At the cathode, actinides are selectively deposited into a liquid Ga to form An-Ga alloy,

$$An^{3+} + 3e^- \rightarrow An_{\text{ in Ga}} \qquad (2)$$
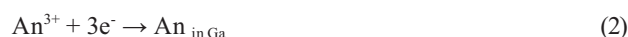
Then, actinides are recovered from An-Ga alloy at the actinide recovery step. Here, the recovery of actinides from An-Ga alloy by the distillation of the Ga is not reasonable as an indus-
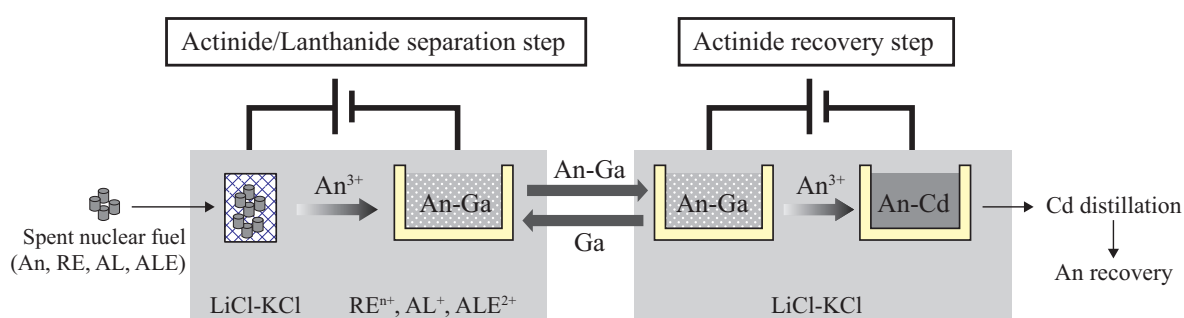

Figure 1. The concept of the proposed pyroprocess to recover actinides separating from fission products in LiCl-KCl melts[4]. An: actinides, RE: rare earths, AL: alkali metals, ALE: alkaline earth metals.

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trial process because of the very high boiling point of Ga (2673 K). Thus, electrochemical de-alloying reaction is applied at the actinide recovery step. When the formed An-Ga alloy electrode is used as the anode, actinides in An-Ga alloy are anodically dissolved,

$$An_{\text{in Ga}} \rightarrow An^{3+} + 3e^- \tag{3}$$

A liquid Cd cathode is used to deposit actinides as An-Cd alloys,

$$An^{3+} + 3e^- \rightarrow An_{\text{in Cd}} \tag{4}$$

The liquid Cd cathode has been conventionally used for a group recovery of actinides at the pyroprocess [1, 2]. By distilling Cd of the An-Cd alloy, the actinides are finally recovered [5]. The liquid Ga after the actinides recovery step is used again as the cathode material at the actinide/lanthanide separation step. As repeating the two steps, the concentrations of the fission products, such as lanthanides, alkalis and alkaline earths, increase in the melts at the actinide/lanthanide separation step. Thus, a part of the melts is periodically treated by zeolite to remove the fission products [6]: The fission products are adsorbed and/or occluded in the structure of zeolite.

In order to evaluate the actinide/lanthanide separation efficiency of the proposed pyroprocess, it is necessary to investigate the electrochemical deposition of actinides and lanthanides at a liquid Ga electrode. However, the studies on the electrochemical behavior of a liquid Ga electrode in LiCl-KCl melts were limited. Thus, in the previous study, the authors first measured the following basic properties of a liquid Ga electrode in LiCl-KCl melts [4];

  (1) the electrochemical window of the liquid Ga electrode,
  (2) the potentials for binary alloy formation at 773 K; U-Ga ($U^{3+} + 3e^- \rightarrow U_{\text{in Ga}}$, 1.55 V(vs. Li^+/Li)), Pu-Ga ($Pu^{3+} + 3e^- \rightarrow Pu_{\text{in Ga}}$, 1.4 V), Ce-Ga ($Ce^{3+} + 3e^- \rightarrow Ce_{\text{in Ga}}$, 1.3 V), Nd-Ga ($Nd^{3+} + 3e^- \rightarrow Nd_{\text{in Ga}}$, 1.3 V) and Li-Ga ($Li^+ + e^- \rightarrow Li_{\text{in Ga}}$, 1.1 V),
  (3) the current efficiency (~100 %) for the binary alloys formation and their de-alloying and
  (4) the activity coefficient of U, Pu, Ce and Li in liquid Ga.

In the present study, based on the above basic properties, two runs of the electrochemical actinides recovery were conducted using the liquid Ga electrode in the LiCl-KCl melts containing both actinides and lanthanides chlorides at 773 K. Then, the separation factors, which are inevitable to evaluate the proposed pyroprocess quantitatively, were calculated. Here, the distribution factor of element M (DF_M) is defined by the following equation,

$$DF_M = (X_{\text{M in Ga}})/(X_{\text{M in melts}}) \tag{5}$$

where $X_{\text{M in melts}}$ and $X_{\text{M in Ga}}$ represent the concentration of element M in the LiCl-KCl melts and in the liquid Ga, respectively. The separation factor of element M based on Ce (SF_M)

is defined by the ratio of DF_M to DF_{Ce},

$$SF_M = (X_{\text{Ce in melts}})/(X_{\text{M in melts}})/(X_{\text{Ce in Ga}}) \times (X_{\text{M in Ga}}) \tag{6}$$

The experiments of the equilibrium distribution of actinides and lanthanides between the LiCl-KCl melts and liquid Ga were also performed to calculate the separation factors at the equilibrium state which were compared with those obtained from the electrochemical actinides recovery experiments.

2. Experimental

All experiments were carried out in an Ar glove box with a purification system. Concentrations of impurities were kept to be low in the glove box (oxygen < 20 ppm, water < 10 ppm). A eutectic mixture of LiCl-KCl (58.8:41.2 mol%, 99.9 % purity, m.p. 625 K) containing lanthanide chlorides and/or actinide chlorides were loaded in an alumina crucible (SSA-S, Nikkato corp.) and melted at 773 K. Ga metal (99.9999 % purity) was also loaded at the bottom of the crucible in the case of the equilibrium distribution experiments. The weights of the LiCl-KCl melts and Ga metal used at the equilibrium distribution experiments (RUN1 and 2) and the electrochemical actinides recovery (RUN3 and 4) are summarized in Table 1. Table 2 lists the initial concentration of actinides and lanthanides in the LiCl-KCl melts. As seen in Figure 2, for the electrochemical measurements, the working electrodes were a Ta wire (1 mm diameter, 99.95 % purity) electrode and a liquid Ga electrode. The liquid Ga electrode consisted of Ga metal (99.9999 % purity) loaded in an alumina crucible (SSA-S, inner diameter 13 mm, Nikkato corp.) with the lead of a Ta wire which was covered by an alumina insulator not to contact with the LiCl-KCl melts. The reference electrode was Ag^+/Ag reference electrode: LiCl-KCl-1wt%AgCl and a Ag wire (1 mm diameter, 99.99% purity) as the lead were loaded in a Pyrex tube with a thin bottom wall. All potentials presented were calibrated to the potential of Li metal deposited electrochemically on a Ta wire in the LiCl-KCl melts, which was -2.405 V vs. Ag^+/Ag. A liquid Cd-0.1wt%Li alloy was used as the counter electrode material where a following reaction was expected to occur,
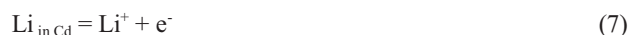
$$Li_{\text{in Cd}} = Li^+ + e^- \tag{7}$$

TABLE 1: Weights of the LiCl-KCl melts and Ga metal used at each RUN

	LiCl-KCl melts /g	Ga metal /g
RUN1	65.01	198.99
RUN2	20.569	25.072
RUN3	202.76	12.004
RUN4	202.76	12.243

TABLE 2: The initial concentrations of actinides (U, Pu and Am) and lanthanides (Ce, Nd, Gd, La, Pr and Eu) in the LiCl-KCl melts in mol% at each RUN

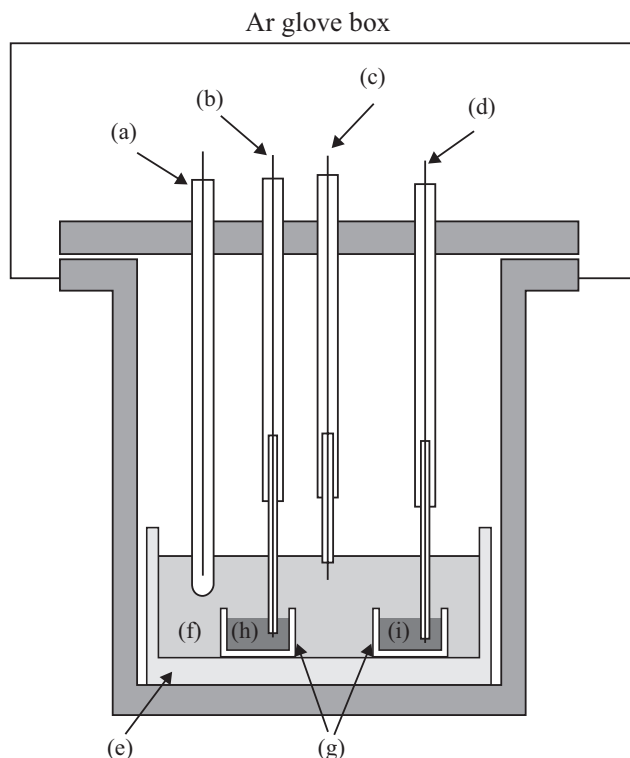|	U	Pu	Am	Ce	Nd	Gd	La	Pr	Eu
RUN1	6.21×10^{-1}	7.24×10^{-1}	2.54×10^{-2}	3.69×10^{-1}	3.67×10^{-1}	3.61×10^{-1}			
RUN2				9.78×10^{-2}	9.61×10^{-2}	1.02×10^{-1}	1.09×10^{-1}	8.44×10^{-2}	8.22×10^{-2}
RUN3	4.32×10^{-3}	2.25×10^{-1}	9.22×10^{-3}	7.34×10^{-2}	6.77×10^{-2}	6.82×10^{-2}	7.28×10^{-2}		
RUN4	3.82×10^{-3}	2.23×10^{-1}	8.25×10^{-3}	7.58×10^{-2}	6.97×10^{-2}	7.01×10^{-2}	7.57×10^{-2}		

Figure 2. Schematic drawing of experimental setup for electrochemical measurements. (a) Ag^+/Ag reference electrode, (b) liquid Ga electrode, (c) Ta wire electrode, (d) Cd-Li counter electrode, (e) alumina crucible, (f) LiCl-KCl melts, (g) small alumina crucible, (h) liquid Ga and (i) liquid Cd-Li.

The structure of the counter electrode was the same as the liquid Ga electrode. The electrochemical measurements were performed using a potentiostat/galvanostat (HZ5000, HOKUTO Denko Corp.). Parts of the LiCl-KCl melts and the liquid Ga alloys were sampled and analyzed by Induction coupled plasma–atomic emission spectroscopy (ICP–AES). A gamma analysis was conducted to measure Am quantitatively. All samples were dissolved in 1N HNO_3 and/or concentrated

HNO_3 before the quantitative analysis.

3. Results and discussion

3.1. Equilibrium distribution of actinides and lanthanides in LiCl-KCl/liquid Ga system. Two runs of equilibrium distribution experiments were carried out at 773 K. Initially, the LiCl-KCl melts contained lanthanides and/or actinides (see Table 2), while no actinides and lanthanides were contained in the liquid Ga phase at the bottom of the crucible. Ga-3.8wt%Li alloy was then added to the LiCl-KCl melts as the reductant to proceed the following reaction,

$$M^{3+} + 3Li_{\text{ in Ga}} = M_{\text{ in Ga}} + 3Li^+ \qquad \text{(M: An or Ln)} \qquad (8)$$

The added amounts of Ga-3.8wt%Li alloy at RUN1 and RUN2 corresponded to approximately 77 % of the initial amounts of actinides in the LiCl-KCl melts and to approximately 75 % of the initial amounts of lanthanides in the LiCl-KCl melts, respectively, to be extracted into the liquid Ga phase. The distribution between the LiCl-KCl melts and liquid Ga phase was considered to reach the equilibrium within 6 hours after the addition of the reductant. According to the quantitative analysis results of the LiCl-KCl melts and Ga, the distribution factors and the separation factors were calculated. The calculated values are listed in Table 3 and 4 together with the reported values [7, 8]. The distribution factor and the separation factor of U at RUN1 and those of Eu at RUN2 could not be obtained because the concentrations $X_{\text{U in melts}}$ and $X_{\text{Eu in Ga}}$ were below the detection limit of ICP-AES. The obtained separation factors of Pu and Am in LiCl-KCl/liquid Ga system agreed with those reported by Toda et al. and were confirmed to be around 10~30 times higher than those of the LiCl-KCl/liquid Cd system [7, 8]. It was also found that lanthanides distributed with the comparable separation factors between LiCl-KCl/liquid Ga and LiCl-KCl/liquid Cd systems [8].

3.2. Electrochemical separation of actinides from lanthanides using a liquid Ga electrode. Cyclic voltammetry was performed in the LiCl-KCl melts containing both actinides (U, Pu, Am) and lanthanides (Ce, Nd, La, Gd) chlorides at 773 K to determine the electrochemical conditions for

TABLE 3: Distribution factors of actinides and lanthanides obtained at equilibrium distribution experiments at 773 K. $DF_M = (X_{\text{M in Ga}})/(X_{\text{M in melts}})$

	DF_{Pu}	DF_{Am}	DF_{Ce}	DF_{Nd}	DF_{Gd}	DF_{La}	DF_{Pr}
RUN1	7.3×10^1	2.3×10^1	1.9×10^{-1}	2.6×10^{-1}	7.0×10^{-2}		
RUN2			3.9	4.9	1.2	9.0×10^{-1}	5.2

TABLE 4: Equilibrium separation factors of actinides and lanthanides based on Ce at 773 K
$SF_M = (X_{\text{Ce in melts}})/(X_{\text{M in melts}})/(X_{\text{Ce in Ga}})\times(X_{\text{M in Ga}})$

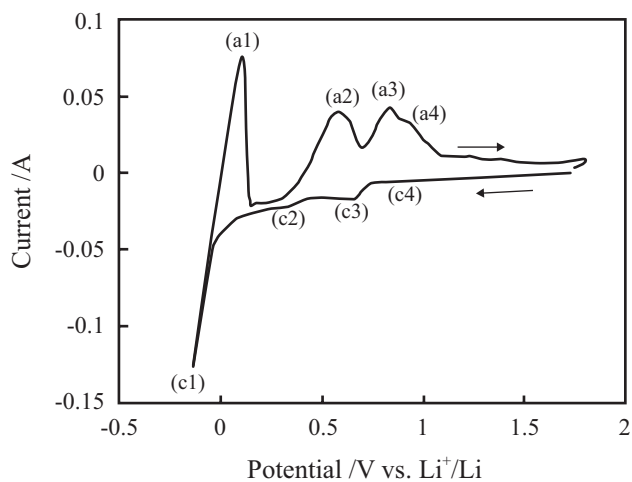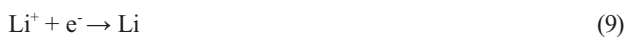| system		SF_U	SF_{Pu}	SF_{Am}	SF_{Ce}	SF_{Nd}	SF_{Gd}	SF_{La}	SF_{Pr}
LiCl-KCl / liquid Ga	RUN1	-	3.8×10^2	1.2×10^2		1.4	3.7×10^{-1}		
	RUN2					1.2	3.2×10^{-1}	2.3×10^{-1}	1.3
	Reported value[7]	3.0×10^4	2.60×10^2	1.12×10^2	1 (base)				
LiCl-KCl / liquid Cd	Reported value[8]	5.56×10^1	2.50×10^1			1.04	2.70×10^{-1}	3.70×10^{-1}	1.14
	Reported value[7]		1.3×10^1	7					

Figure 3. Cyclic voltammogram of a Ta wire electrode in LiCl-KCl melts containing actinides (U, Pu and Am) and lanthanides (Ce, Nd, La and Gd) chlorides at 773 K. Concentrations of U, Pu, Am, Ce, Nd, La and Gd in the melts were 5.99×10^{-3}, 2.32×10^{-1}, 9.64×10^{-3}, 7.42×10^{-2}, 7.02×10^{-2}, 7.24×10^{-2} and 6.83×10^{-2}, respectively, in mol%. Scan rate was 100 mV s^{-1}.

Figure 4. Cyclic voltammogram of a liquid Ga electrode in LiCl-KCl melts containing actinides (U, Pu and Am) and lanthanides (Ce, Nd, La and Gd) chlorides at 773 K. Concentrations of U, Pu, Am, Ce, Nd, La and Gd in the melts were 3.82×10^{-3}, 2.23×10^{-1}, 8.25×10^{-3}, 7.58×10^{-2}, 6.97×10^{-2}, 7.57×10^{-2} and 7.01×10^{-2}, respectively, in mol%. Scan rate was 10 mV s^{-1}.

actinides/lanthanides separation using a liquid Ga electrode. Figure 3 shows a cyclic voltammogram of a Ta wire electrode in the melts. Several cathodic current rises and their corresponding anodic peaks are observed in the cyclic voltammogram. The cathodic current rise, c1, and the anodic current peak, a1, observed at around 0 V (vs. Li$^+$/Li) were ascribed to Li metal deposition (reaction 9) and its dissolution (reaction 10), respectively,

$$Li^+ + e^- \rightarrow Li \tag{9}$$

$$Li \rightarrow Li^+ + e^- \tag{10}$$

The cathodic current increases from approximately 0.45 V (c2) and the corresponding anodic current peak is observed at around 0.6 V (a2). The former current increase corresponded to the lanthanide metal deposition (reaction 11) and the latter current peak to the reverse reaction (reaction 12), respectively [9, 10].

$$Ln^{3+} + 3e^- \rightarrow Ln \qquad (Ln: La, Ce, Gd) \tag{11}$$

$$Ln \rightarrow Ln^{3+} + 3e^- \tag{12}$$

The small cathodic current increases from approximately 0.74 V (c3) and 0.90 V (c4) were considered to be due to the deposition of Pu (reaction 13) and U (reaction 14) metals, respectively [11, 12].

$$Pu^{3+} + 3e^- \rightarrow Pu \tag{13}$$

$$U^{3+} + 3e^- \rightarrow U \tag{14}$$

The anodic current peak (a3) and current shoulder (a4) were considered to be ascribed to the dissolutions of Pu (reaction 15) and U (reaction 16) metals, respectively [11, 12].

$$Pu \rightarrow Pu^{3+} + 3e^- \tag{15}$$

$$U \rightarrow U^{3+} + 3e^- \tag{16}$$

Concerning Am and Nd, of which divalent cations are stable in the LiCl-KCl melts, complicated electrochemical behaviors have been proposed. According to the reports [13, 14], the cathodic current for Am (reaction 17) and Nd (reaction 18)
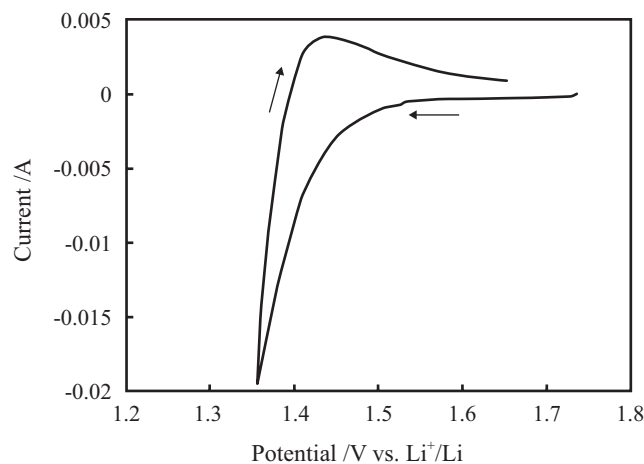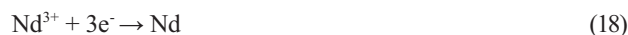
metals deposition seemed to overlap the cathodic current increases of c3 and c2, respectively,

$$Am^{2+} + 2e^- \rightarrow Am \tag{17}$$

$$Nd^{3+} + 3e^- \rightarrow Nd \tag{18}$$

The anodic current for the dissolution of Am, the reverse reaction of reaction 17, could be considered to overlap peak a3 [13]. The formations of their divalent cations were considered to proceed from the potential approximately 0.2 V more positive than their metal deposition potentials [13, 14].

$$Am^{3+} + e^- \rightarrow Am^{2+} \tag{19}$$

$$Nd^{3+} + e^- \rightarrow Nd^{2+} \tag{20}$$

Cyclic voltammetry was also carried out using a liquid Ga electrode in the melts. As shown in Figure 4, a cathodic current starts to increase from around 1.5 V. As mentioned above, in our previous study [4], the potentials of binary Ga alloy formation for U-Ga, Pu-Ga, Ce-Ga, Nd-Ga and Li-Ga were found to proceed at lower potential than 1.55 V, 1.4 V, 1.3 V, 1.3 V and 1.1 V, respectively. Therefore, the cathodic current observed in Figure 4 corresponded to actinides-Ga alloy formation (reaction 2). Furthermore, the corresponding anodic peak at 1.43 V in Figure 4 was due to the de-alloying reaction (actinides dissolution from the alloy (reaction 3)). Our previous study also showed that the liquid Ga dissolution (reaction 21) proceeded at more positive potential than 1.85 V [4],

$$Ga \rightarrow Ga^{3+} + 3e^- \tag{21}$$

The large potential difference between the actinide dissolution and the Ga dissolution suggested that de-alloying at the second step of the proposed process (Figure 1) would proceed without any Ga loss by controlling the anodic potential below 1.85 V.

Two runs of actinides recovery were conducted using a liquid Ga electrode in the LiCl-KCl melts containing both actinides and lanthanides chlorides. The electrolysis condition was determined based on the results of cyclic voltammetry (Figure 4); galvanostatic electrolysis at -6 mA at RUN3 and -20 mA at RUN4 for group recovery of actinides with a limited lanthanides deposition. The electrolysis was stopped before the concentration of actinides in the liquid Ga reached
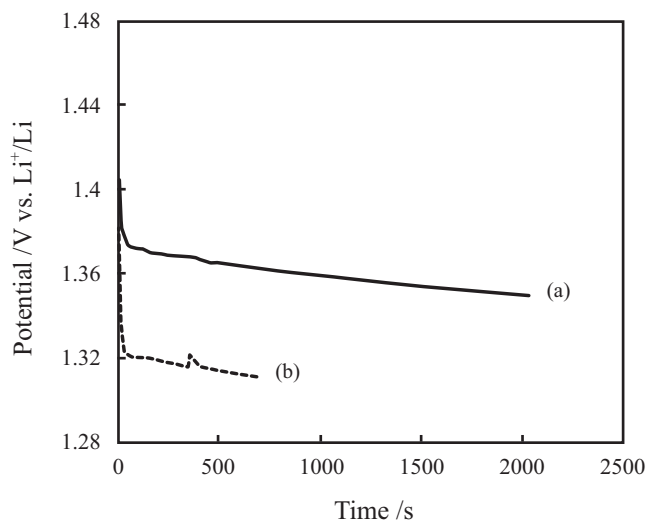
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Figure 5. The liquid Ga electrode potential developments during electrochemical actinides recovery experiments. (a): galvanostatic electrolysis using the liquid Ga electrode at -6 mA in LiCl-KCl melts at 773 K at RUN3. Concentrations of U, Pu, Am, Ce, Nd, La and Gd in the melts were 4.32×10^{-3}, 2.25×10^{-1}, 9.22×10^{-3}, 7.34×10^{-2}, 6.77×10^{-2}, 7.28×10^{-2} and 6.82×10^{-2}, respectively, in mol%. (b): galvanostatic electrolysis using the liquid Ga electrode at -20 mA in LiCl-KCl melts at 773 K at RUN4. Concentrations of U, Pu, Am, Ce, Nd, La and Gd in the melts were 3.82×10^{-3}, 2.23×10^{-1}, 8.25×10^{-3}, 7.58×10^{-2}, 6.97×10^{-2}, 7.57×10^{-2} and 7.01×10^{-2}, respectively, in mol%.

TABLE 5: Distribution factors of actinides and lanthanides obtained at electrochemical recovery of actinides at 773 K. $DF_M = (X_{M\ in\ Ga})/(X_{M\ in\ melts})$

	DF_U	DF_{Pu}	DF_{Am}
RUN3	1.7×10^{-1}	6.3×10^{-2}	2.6×10^{-2}
RUN4	2.2×10^{-1}	1.1×10^{-1}	6.9×10^{-2}

TABLE 6: Non-equilibrium separation factors of actinides based on Ce
$SF_M = (X_{Ce\ in\ melts})/(X_{M\ in\ melts})/(X_{Ce\ in\ Ga})\times(X_{M\ in\ Ga})$

	SF_U	SF_{Pu}	SF_{Am}	SF_{Ce}
RUN3	$>1.2\times10^{3}$	$>4.2\times10^{2}$	$>1.8\times10^{2}$	1
RUN4	$>4.5\times10^{2}$	$>2.2\times10^{2}$	$>1.4\times10^{2}$	(base)

Since $X_{Ce\ in\ Ga}$ was below detection limit of ICP-AES, SF_M was evaluated using the lower detection limit of Ce.

their solubility (0.034 mol%-U at 772 K [15], 0.17 mol%-Pu at 773 K [16]). The passed electrical charges at RUN3 and RUN4 were 12.1 C and 14.1 C, respectively. These values corresponded to 0.024 mol%-actinides and 0.028 mol%-actinides recovered in the liquid Ga at RUN3 and RUN4, respectively, under the assumption of 100 % current efficiency. As shown in Figure 5, the potential shifts gradually to the negative direction along with the electrolysis time indicating the concentration increase of actinides in the liquid Ga. It was noticed that during the electrolysis the potential of liquid Ga electrode was kept at higher than the lanthanide-Ga alloy formation potential; 1.3 V for Ce-Ga and Nd-Ga alloys as shown in our previous study [4]. Thus, actinides were expected to be recovered as actinides-Ga alloy more preferentially than the lanthanides-Ga alloy. The liquid Ga electrode was removed from the melts just after ending the electrolysis. Then, parts of Ga alloys were sampled and analyzed quantitatively to calculate the distribution factors and the separation factors. Table 5 and 6 list the calculated values. The separation factor of U was higher than that of Pu and Am, indicating the preferential recovery of U in liquid Ga among actinides. High separation factors of Pu and Am were achieved by using the liquid Ga electrode. These values were more than 10 times higher than the separation factors of the LiCl-KCl/liquid Cd system (see Table 4). It seemed that the separation factors of Pu and Am were close to those obtained from the equilibrium distribution experiments in the LiCl-KCl/liquid Ga system, while the separation factors of U were considered to be different between the electrochemical actinides recovery and equilibrium distribution; lower at non-equilibrium state (electrochemical actinides recovery experiment in this study) than at equilibrium state (equilibrium distribution experiment reported by Toda et al. [7]). The lower value at non-equilibrium state could be explained by the smaller amount of U in the liquid Ga electrode due to the limitation of diffusion of U in the LiCl-KCl melts or in the liquid Ga phase during the electrolysis. Further investigation is required to clarify the effect of the electrolysis conditions on the non-equilibrium separation factors.

4. Conclusion

Galvanostatic electrolyses were performed using a liquid Ga electrode in the LiCl-KCl metls containing both actinides and lanthanides chlorides at 773 K to recover actinides in the liquid Ga with limited amounts of lanthanides codeposition. The obtained results confirmed that the separation factors of Pu and Am based on Ce at non-equilibrium state were more than 10 times higher than those of the LiCl-KCl/liquid Cd system. Thus, liquid Ga was confirmed to be a promising electrode material to recover actinides. The proposed pyroprocess using a liquid Ga electrode was likely to have a higher TRUs (Pu and minor actinides)/lanthanides separation efficiency compared to the conventional pyroprocess where a liquid Cd electrode is used for the group recovery of actinides. As the future study, the separation factors of other minor actinides, Np and Cm, should be measured.

Acknowledgement

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