Electrochemical Extraction of Cerium by Forming Ce-Zn Alloys in LiCl-KCl Eutectic on W and Liquid Zn Electrodes

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The electrochemical formation of Ce-Zn alloys in LiCl-KCl eutectic was systematically investigated by cyclic voltammetry (CV), square wave voltammetry (SWV) and open circuit chronopotentiometry (OCP). Due to the decrease of Ce activity in the metal phase of formed Ce-Zn alloys, the deposition potential of Ce(III) at a liquid Zn electrode was found to be 0.64 V more positive than that on an inert W electrode. The co-reduction behaviors of Ce(III) and Zn(II) ions were also studied in LiCl-KCl eutectic. Signals associated with six and nine Ce,Zn x intermetallic compounds were detected by CV and OCP techniques, respectively. In addition, potentiostatic electrolyses were carried out to extract Ce at a liquid Zn electrode in LiCl-KCl-NeCl 3 melt and on a W electrode in LiCl-KCl-NeCl 3-ZnCl 2 melt, respectively. Deposited samples were analyzed by X-ray diffraction (XRD) and scanning electron microscopy (SEM) coupled with energy dispersive X-ray spectroscopy (EDS). The results showed that intermetallic compound CeZn 11 was prepared on both the inert W electrode and liquid Zn electrode.

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Pyrochemical reprocessing of spent nuclear fuels is now considered to be one of the most promising options in nuclear fuel cycles due to their outstanding properties such as high thermal and radiation stability which allows a shorter cooling time of fuels before reprocessing, compactness, high actinide content and inherent proliferation resistance.1–4 The aim of the traditional pyrochemical processing technology is to achieve a group-selective separation of all actinides (Ans) for recycling or transmutation, generating the minimum of ultimate waste flows with an acceptable decontamination of fission products. A typical process in pyrochemical reprocessing is the electrorefining,5–10 in which lanthanides (Lns) and Ans in spent fuels are dissolved into molten chloride salt by anodic dissolution. Subsequently, Ans (e.g. U) are selectively deposited on the stainless steel cathode or liquid cadmium cathode with respect to their different redox potentials in melt. In contrast, most fission products (FPs) still remain in the electrolyte.6 Actually, Lns are the major and most awkward FPs and are difficult to be separated from Ans due to their similar chemical properties. On the other hand, Lns are well known to possess large neutron capture cross sections, which can spoil the neutron economy of reactor cores in the transmutation operation. Therefore, quite challenging, it is still imperative to efficiently separate Ans from Lns.

In recent years, liquid-liquid reductive extraction has attracted considerable interest as a promising technique for the separation of minor actinides (MAs) from Lns.11–13 During a typical separation process, the solute cations in molten salt are recovered into the liquid metal phase via direct electrolysis or reductive extraction, due to the low activity coefficients of their corresponding metal phases in liquid metals. Since the operation of liquid metals plays an important role in the pyrochemical reprocessing, it is of crucial importance to understand the electrochemical behaviors of MAs and Lns at liquid cathodes (i.e. Bi, Cd and Al) for further optimization of the separation process.16–24

In this work, cerium, a typical Ln element, was chosen as the object of study because of its potential use in a wide range of scientific fields. For example, it can act as a surrogate for exploring the electrochemistry of uranium in molten salt due to their comparable ionic radii and electrochemical potentials.25 Additionally, liquid zinc has been regarded as a considerable material for the extraction of Ans and Lns from molten chloride salt.26,27 Herein, we used zinc as the assistant material for cerium extraction in light of its low melting point (692 K), which makes the extraction possible at relatively lower temperatures.28 To the best of our knowledge, previous investigations about the redox behaviors and formation mechanisms of Ce-Zn alloys associated with electroextraction were very rare. Therefore, the major objective of this work is to study the electrochemical behaviors of Ce(III) at a liquid Zn electrode and the co-reduction behaviors of Ce(III) and Zn(II) ions on a W electrode in LiCl-KCl molten salt. Moreover, potentiostatic electrolyses were also conducted to examine the feasibility of zinc-assisted extraction of Ce from LiCl-KCl melt at a relatively lower temperature.

Experimental

Chemicals and melt preparation.— All chemicals were handled in a glove box under a U grade (less than 2 ppm O 2 and H 2 O ) argon atmosphere. Anhydrous KC1, LiCl, NH 4 Cl, ZnCl 2 and CeO 2 (Alfa Aesar, AR grade) were used for the preparation of the electrolyte. The chloride mixture (LiCl : KC1 = 58.8 : 41.2 mol%, analytical-grade) was at first dried under vacuum for more than 24 h at 473 K to minimize the amount of adsorbed water, and then melted in an alumina crucible placed in a quartz cell inside an electric furnace. The electrolyte was melted under a dry argon atmosphere and the working temperature was measured in the melt with a thermocouple protected by an alumina tube. The anhydrous CeCl 3 was prepared from CeO 2 and NH 4 Cl heated at 623 K for 2.5 h in a vacuum furnace.29 The obtained CeCl 3 was suitable for electrochemical investigations, as evidenced by the CV characterization (Fig. 1a).

The concentrations of Ce(III) and Zn(II) cations in the melt were determined using an inductively coupled plasma atomic emission spectrometer (ICP-AES, Perkin Elmer NEXION 300D). The salt samples were directly taken from the melt before each experiment, and each sample was dissolved in ultrapure water containing 3–5 mol L 1 HNO 3 for further analysis.

Apparatus and electrodes.— All electrochemical measurements were performed using an Autolab PGSTAT 302N potentiostatic/galvanostatic controlled with the Nova 1.9 software package from Metrohm. The reference electrode (RE) consisted of a silver wire (1 mm diameter, Alfa, 99.99% purity) dipped into the solution of AgCl (1 wt%) in LiCl-KCl molten salt, fixed in a quartz tube. All potentials

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were referred to this Ag/Ag⁺ couple. A spectral pure graphite rod (6 mm diameter) served as the counter electrode. The working electrode was a tungsten wire (0.5 mm diameter, Alfa, 99.99% purity) or liquid zinc placed in an alumina crucible (2.0 cm in height, 1.5 cm in diameter). A tungsten wire placed in an alumina sleeve was immersed into the liquid Zn as the electric lead. The lower end of the W working electrode was polished thoroughly using SiC paper, and then cleaned in ethanol using ultrasound. Before each measurement, the working electrode was cleaned by galvanostatic anodic polarization. The active electrode surface area was determined after each experiment by measuring the immersion depth of the electrode in melt salts.

Preparation and characterization of Ce-Zn alloy.— Ce-Zn alloy samples were prepared on inert W electrodes or liquid Zn electrodes by potentiostatic electrolysis at a cathodic potential previously defined by CV and OCP at 723 K. After the electrolysis, samples were removed from the bath and washed with ethylene glycol (Sinopharm, 99.8%), which scarcely reacts with Ce-Zn alloys, to remove salts and finally stored inside the glove box until further analyses. X-ray diffraction (XRD) (Bruker, D8 Advance) technique was employed to identify the formation of Ce-Zn alloys. Scanning electron microscopy (SEM) (Hitachi S-4800 and JEOL JSM 6360 LV) with energy dispersive spectrometry (EDS) (Hitachi S-4800) was employed to analyze the surface morphologies and the compositions of micro-zone chemicals.

Results and Discussion

Electrochemical behavior of Ce(III) on a W electrode.— Fig. 1a shows the typical cyclic voltammograms (CVs) in the absence (dotted curve) and presence (solid curve) of CeCl₃ (8.34 × 10⁻⁵ mol cm⁻³) in LiCl-KCl melt on an inert W electrode. The dotted curve recorded in purified LiCl-KCl melt salt has no additional signals except for the redox signal of lithium within the electrochemical window. In addition, the residual current is less than 1 mA at the potential range of −1.0 V to approximate −2.4 V vs. Ag/Ag⁺. The absence of any other redox couples in this region confirms that the prepared melt is free of oxides, hydroxides and other impurities and is of acceptable quality for subsequent electrochemical study of CeCl₃. After the addition of CeCl₃, the electroactive Ce(III) can be reduced via a one-step process before the electrodeposition of lithium, with the characteristic shape for the formation of a new phase. Since no alloy phase exists in the Ce-W system, the cathodic current corresponds to the formation of Ce metal deposition while the anodic peak E’ corresponds to the dissolution of Ce metal.

Subsequently, a liquid Zn electrode was employed as the working electrode to study the electrochemical behaviors of Ce(III) cations in LiCl-KCl melt. Unlike the inert W electrode, the liquid Zn metal can interact with Ce metal during reduction to form CeₓZnᵧ intermetallic compounds. Therefore, CVs recorded at the liquid Zn electrode are expectedly different to those recorded on inert W electrodes. Curves a and b in Fig. 2 designate the CVs before and after the addition of Ce(III) ions, respectively. In both cases, the anodic peak at about −0.8 V corresponds to the dissolution of Zn electrode. In addition, the current starts to increase negatively at about −1.2 V in the cathodic direction. However, the curves associated with neat LiCl-KCl melt and LiCl-KCl-CeCl₃ eutectic are significantly different, e.g., curve a shows that only Li reduction occurred at the liquid Zn electrode, whilst curve b suggests that the reduction of Ce(III) and Li⁺ should take place together.

In order to avoid the deposition effect of Li in curve b, the CV of LiCl-KCl-CeCl₃ (curve b) was subtracted from the CV of neat LiCl-KCl melt (curve a) and the result was shown as curve c in Fig. 2. The reduction peak A in curve c at about −1.41 V corresponds to the reduction of Ce(III) ions at the liquid Zn electrode. The result reveals that the reduction of Ce(III) at the liquid Zn electrode is about 0.64 V more positive than that on an inert W electrode through the formation of CeₓZnᵧ intermetallic compounds, due to the lower activity coefficient of cerium at the liquid Zn cathode. In this way, the equilibrium potential of the Ce(III)/CeₓZnᵧ system can be described by the following equation:

\[
E_{\text{Ce(III)/CeₓZnᵧ}} = E_{\text{Ce(III)/Ce}} - \frac{RT}{nF} \ln \left( \frac{[\text{Ce}]^{3+}}{[\text{Ce}]^{4+}} \right)
\]  

where \(E_{\text{Ce(III)/Ce}}\) denotes the equilibrium potential of pure Ce element, \(T\) is the absolute temperature in K, \(F\) represents the Faraday constant, and \(n\) is the number of electrons exchanged in the Ce(III)/Ce redox reaction.
Electrochemical behavior of Ce(III) in LiCl-KCl-ZnCl₂ melt on the W electrode.— In order to get more information about the alloy formation between electrode materials and electroactive elements in LiCl-KCl molten salt, experiments were then conducted to explore the co-reduction behaviors of Ce(III) and Zn(II) cations on the W electrode. Fig. 3a displays the comparison of the CVs of LiCl-KCl melt (dotted curve) and LiCl-KCl-ZnCl₂ (0.4 × 10⁻³ mol cm⁻³)-CeCl₃ (8.08 × 10⁻³ mol cm⁻³) melt (solid curve) on a W electrode with the scan rate of 0.1 Vs⁻¹ at 723 K. As can be seen from Fig. 3a, when both Ce(III) and Zn(II) cations are present in melt, a co-reduction process is expected to occur. Similar to the “underpotential deposition” at the liquid Zn electrode (red curve in Fig. 2), the co-reduction of Ce(III) and Zn(II) cations also allows the deposition of Ce(III) to occur at more positive potentials, which is nominated as the so-called “depolarization effect”. However, the curve obtained on the inert W electrode is significantly different from that at the liquid Zn electrode. In order to confirm the redox couples of the oxidation peaks, the cathodic inversion potentials of the CVs were adjusted (see Fig. 3b and 3c). The redox peaks A/A’ at about −0.83 V vs. Ag/Ag⁺ are related with the reduction and dissolution of Zn at the beginning of cathodic scan. The cathodic peak B can be observed at −1.31 V when the reverse potential is −1.4 V. Surprisingly, two corresponding anodic peaks B’ and B” appear, which could be due to the formation of different kinds of CeₓZnᵧ intermetallic compounds caused by intermetallic diffusion. When the reversed potential is more negative than −1.6 V, a new cathodic peak H can be observed. This might be due to the dissolution of a kind of Ce-Zn alloy generated by the diffusion of deposited Ce metal into the Zn-coated electrode, which has been previously deposited on the surface of the W electrode. The cathodic peak C at −1.77 V and its corresponding anodic peak C’ at −1.59 V can be observed when the inversion potential is about −1.85 V. Meanwhile, the large peak detachment suggests that the processes of CeₓZnᵧ formation are kinetically irreversible. Two anodic peaks D’ and D” were detected at −1.75 and 1.72 V, respectively. However, there is only one new cathodic peak D detected when the inversion potential was set to be −2.0 V. Unlike peak B, the cathodic peak D is broadened, which indicates that the formation potentials of the two intermetallic compounds are too close to be distinguished. The cathodic peak E at about −2.02 V should be associated with the formation of Ce metal, and the corresponding anodic peak E’ appears at about −1.85 V. Compared to the electrochemical behaviors of Ce(III) cations at the liquid Zn electrode, the underpotential deposition of Li was not observed even though the cathodic potential attained −2.2 V (Fig. 3b). The reason could be that the surface of the W electrode has been coated with a layer of Ce-Zn alloys, and hence no free Zn metal is present on the surface. Actually, through literature searching, similar behaviors have been observed in the preparation of Ln-Al alloys. This phenomenon indicates that the cathodic wave in Fig. 2 (curve b) at about −1.4 V should correspond to the formation of Ce-Zn and Li-Zn alloys.

Square wave voltammetry (SWV) measurement was also conducted in order to further elucidate the co-reduction process of Ce(III) and Zn(II) cations in LiCl-KCl melt. Fig. 4 gives the comparison of the SWVs of LiCl-KCl-ZnCl₂ (0.4 × 10⁻³ mol cm⁻³)-CeCl₃ (8.08 × 10⁻³ mol cm⁻³) system (black curve) and LiCl-KCl-CeCl₃ (8.34 × 10⁻³ mol cm⁻³) system (red curve) on W electrodes at 723 K. The red curve shows a large cathodic signal corresponding to the formation of Ce metal (peak E). After the addition of ZnCl₂, three new obvious reduction peaks were observed at −0.84 (peak A), −1.31 (peak B), and −1.93 V (peak D) vs. Ag/Ag⁺, respectively, which correspond to the formation of pure Zn metal and two different Ce-Zn alloys, respectively. The shoulder peak C, which is hardly distinguishable from peak D, might be attributed to the formation of another Ce-Zn alloy. The similar phenomenon was also observed in the corresponding CVs (see Fig. 3).

As a suitable technique for the study of potential alloy formation and dissolution, open circuit chronopotentiometry (OCP) technique was employed to investigate the mechanism of formation and dissolution of alloys and to identify the maximum number of formed alloys. At first, thin layered samples of alloys were prepared by cathodic deposition of Ce(III) at the Zn-coated W electrode for a short period of time in melt. Then, the potentiostatic control was disconnected and a transient curve of the open circuit potential was collected.

Figure 3. (a) Comparison of the CVs of LiCl-KCl melt (dotted curve) and LiCl-KCl-ZnCl₂ (0.4 × 10⁻³ mol cm⁻³)-CeCl₃ (8.08 × 10⁻³ mol cm⁻³) melts (solid curve) on a W electrode at 723 K. Scan rate: 0.1 mVs⁻¹. (b) and (c) CVs in LiCl-KCl-ZnCl₂ (0.4 × 10⁻³ mol cm⁻³)-CeCl₃ (8.08 × 10⁻³ mol cm⁻³) melts on W electrode at different inversion potentials at 723 K. Scan rate: 0.1 Vs⁻¹.

Figure 4. Square wave voltammograms of LiCl-KCl-ZnCl₂ (0.4 × 10⁻³ mol cm⁻³)-CeCl₃ (8.08 × 10⁻³ mol cm⁻³) melt on W electrodes at 723 K with a square wave period of 0.025 s and an amplitude of 0.05 V. Scan rate: 0.1 Vs⁻¹.
metallic compounds, the formation potential of intermetallic compound CeZn11 (plateau 1) is close to that of pure Zn metal, which suggests that intermetallic compound CeZn11 should be more thermodynamically stable than intermetallic compound CeCd11.

Several series of OCP measurements were carried out at the temperatures of 723, 773 and 823 K using a Zn-coated electrode. At 723 K, all plateaus were clearly detected on the Zn-coated W electrode. Nevertheless, the plateaus became much less distinguishable when the working temperature was higher than 823 K. This phenomenon indicates the higher stabilities of CeZn11 intermetallic compounds at lower working temperatures and the faster diffusion kinetics of deposited Ce metal in the Ce-Zn alloy layer at higher temperatures. Actually, similar behaviors have been observed in the preparation of Np-Al alloys on an active Al electrode.

Preparation and characterization of the Ce-Zn alloys.—In order to examine the formation mechanisms of Ce-Zn alloys and to establish the relationship between the composition of deposits and the equilibrium deposition potential, potentiostatic electrolyses were subsequently carried out to prepare alloy samples on the W and liquid Zn electrodes, respectively. After the electrolyses, samples were washed with ethylene glycol (Aldrich 99.8%) and stored inside the glove box for further analyses.

Based on the results obtained in Fig. 2, potentiostatic electrolysis experiments were first conducted at a liquid Zn electrode at the potential of $-1.4 \, \text{V vs. Ag/Ag}^+$ for 5.0 h in LiCl-KCl-CeCl3 (8.08 × 10^{-5} mol cm^{-3}) melt at 723 K. According to the discussion above, both Li and Ce have been reduced on the surface of the liquid Zn electrode at the deposition potential of $-1.4 \, \text{V}$. Normally, metallic Li-containing alloys are active to ethylene glycol. However, here we found that the deposit obtained was stable in ethylene glycol and even in water, which indicated that there were no Li-containing alloys formed in the electrolysis process. The absence of Li-containing alloys during potentiostatic electrolysis might be caused by the low current densities (less than $-10 \, \text{mA cm}^{-2}$). In other words, the reduction of Li can be hindered at low current densities. As shown in Fig. 6a, the presence of both Zn and CeZn11 phases is identified. It reveals that the deposited Ce exists in the form of the most thermodynamically stable intermetallic compound CeZn11 at the liquid Zn electrode. Fig. 6b displays the SEM image of the cross-section of the deposit and two obviously different morphologies (zones A and B) can be observed. The EDS result of zone A (Fig. 6c) shows the presence of salt, which was difficult to be removed during the sample washing treatment. The EDS result of zone B is given in Fig. 6d, and the molar atom percentage ratio of Zn to Ce is roughly 15.2, which is higher than 11. The high content of Zn might be caused by the liquid Zn electrode itself.

In addition, potentiostatic electrolysis experiments were also conducted in LiCl-KCl-CeCl3 (3.35 × 10^{-5} mol cm^{-3})-CeCl3 (7.99 × 10^{-5} mol cm^{-3}) melt using a W wire with a diameter of 1.0 mm as the working electrode at the potential of $-1.4 \, \text{V}$. The electrolysis lasted for 2.5 h at 723 K. It should be noted that, it was difficult for the formed alloys to be adhered on the surface of the W electrode at the experimental temperature. Therefore, we put an alumina crucible (2.0 cm in height, 1.5 cm in diameter) under the W electrode and the formed alloys could drop into the crucible during the electrolysis. Fig. 7a presents the XRD pattern of the sample, from which the phases of Zn and CeZn11 were detected. Generally, based on previous results, it is possible to obtain different kinds of CeZn11 intermetallic compounds with different compositions of Zn and Ce on the cathode. However, similar to the reduction of Ce(III) ions at the liquid Zn electrode, only the most thermodynamically stable intermetallic compound CeZn11 was obtained. The reason might be that the formed CeZn1 intermetallic and metallic Zn phases could interdiffuse each other to form a new Zn-rich CeZn1 intermetallic phase. Therefore, only the intermetallic phase CeZn11 was found by XRD analysis. As shown in Fig. 7b, two different morphologies were observed on the surface of Ce-Zn
alloys. Representative EDS results of zones A and B are displayed in Fig. 7c and Fig. 7d, respectively. The molar atom percentage ratio of Zn to Ce for zone A is 16.8 (>11), and the reason might be that zone A is composed of the mixture of CeZn_{11} and Zn phases. However, the molar atom percentage ratio of Zn to Ce for zone B is 5.7 (<11), and the reason might be that zone B is composed of the mixture of CeZn_{11} and other alloy phases. In addition, the presence of KCl in both zones may be caused by the residual salt, which was coated by liquid Zn and difficult to be removed during the sample washing treatment.

Conclusions

The electrochemical behaviors of Ce(III) ions on an inert W electrode and a liquid Zn electrode were first studied using CV, SWV...
and CP techniques. The reduction of Ce(III) to Ce(0) occurs at about −2.05 V vs. Ag/AgCl on the inert W electrode, whereas the reduction of Ce(III) ions at the liquid Zn electrode occurs at about −1.41 V, generating a disparity of 0.64 V. Then, the co-reduction behaviors of Zn(II) with Ce(III) cations on the W electrode were also studied using CV, SWV and OCP techniques. The results showed that nine Ce,Zn$_2$ intermetallic compounds could be detected in the LiCl-KCl-CeCl$_3$-ZnCl$_2$ system. Potentiostatic electrolysis was employed to prepare alloys at the active liquid Zn electrode and the inert W electrode. XRD analysis of the deposits revealed that only the most thermodynamically stable intermetallic compound CeZn$_{11}$ could be obtained on both the W and liquid Zn electrode. This work also manifested the feasibility of Ce extraction from molten salt via the reduction of Ce(III) cations at a liquid Zn electrode or the co-reduction of Zn(II) and Ce(III) cations on a W electrode.

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