Supplementary Information

A New Approach for the Preparation of Variable Valent Rare Earth Alloys from Nano Rare Earth Oxide at Low Temperature in Molten Salt

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Supplementary Fig. 1 shows the CVs obtained on molybdenum electrodes \( (S = 0.322 \text{cm}^2) \) at various scan rates and cathodic limits with the addition of 2 wt. % AlCl\(_3\) and 1wt. % nano-Sm\(_2\)O\(_3\), nano-Yb\(_2\)O\(_3\), and nano-Eu\(_2\)O\(_3\) in LiCl-KCl melts at 480 °C, respectively. The peak currents increase with the increase of scan rate. From Fig. 1a, since the deposition of Al is close to the reduction one of the Sm(III) to Sm(II), the reduction/oxidation peaks \((E_1/E_1')\) prior to Al redox couple \((A/A')\) could be related to the redox reaction of Sm(III)/Sm(II) or the formation and oxidation of Al-Mo alloy, or their combination. In order to figure out who is responsible for the peaks \(E_1/E_1'\).

We carried out the CVs obtained on molybdenum electrodes at various scan rates in KCl-LiCl-AlCl\(_3\)-Yb\(_2\)O\(_3\) melts (see Fig. 1b), due to a larger potential difference between deposition potential of Al and reduction one of the Yb(III) to Yb(II). The redox peaks \((E_2/E_2')\) occurring at about -0.5 V correspond to the reduction/oxidation of Yb(III)/Yb(II). The shoulder \(F'\) prior to Al oxidation peak is likely to be associated with the oxidation Al-Mo alloy deposited. To further investigate the formation of Al-Mo alloy, the CVs at various scan rates in KCl-LiCl-AlCl\(_3\)-Eu\(_2\)O\(_3\) melts with a narrow potential window were performed (see Fig. 1c). One couple of redox current waves prior to Al redox peaks truly exists. Sometimes its cathodic current is easy to be hidden by a larger-scaled cathodic current of Al reduction and it is hard to be discovered. Oppositely, a CV with a wider potential window in KCl-LiCl-AlCl\(_3\)-Eu\(_2\)O\(_3\) melts was conducted (Fig. 1d). The cathodic and anodic limits of the electrochemical window correspond to the reduction of Li\(^+\) and the oxidation of chloride ions, respectively. The redox peaks \((E_3/E_3')\) occurring at about 0 V correspond to the reduction/oxidation of Eu(III)/Eu(II). No formation or oxidation current of Al-Mo alloy is observed in the potential range of +1.0 to -1.5 V. We interpreted that as due to the small formation current of Al-Mo alloy. The discussion of Al-Mo alloy formation peaks can be available as supplementary information.
(b) Yb$_2$O$_3$

(c) Eu$_2$O$_3$
Supplementary Fig. 1 CVs obtained on molybdenum electrodes ($S = 0.322\text{cm}^2$) at various scan rates and cathodic limits with the addition of 2 wt. % AlCl$_3$ and 1wt. % (a) nano-Sm$_2$O$_3$ (b) nano-Yb$_2$O$_3$ (c,d) nano-Eu$_2$O$_3$ in LiCl-KCl melts at 480 °C.