# Electrolysis in Chloride Molten Salts for Sustainable Critical Metals Production and Recovery

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## Abstract:

Neodymium (Nd) and many other rare–earth metals are critical to enabling current and future applications such as electric vehicles, renewable energy harvesting, and consumer electronics. Presently, Nd metal, a key component of permanent magnets that enable these technologies, is produced via high temperature electrolysis of neodymium oxide in a molten fluoride electrolyte. This process is unattractive because it not only emits $CO_2$ at a graphite anode but is also energy–inefficient and generates perfluorocarbon (PFC) gases. In this article, we outline a chloride–based molten salt electrolysis (MSE) approach that offers a much safer, lower energy alternative to the conventional oxy–fluoride process. In chloride MSE, $Nd_2O_3$ is first chlorinated to $NdCl_3$ non–carbothermically. Then, $NdCl_3$ is electrolyzed in moderate–temperature chloride eutectic melts to produce high–purity Nd metal and chlorine gas, which can be recycled in the earlier chlorination step. The chloride MSE process is sustainable, highly energy–efficient and yields Nd metal at an energy consumption of 2.79 kWh/kg but with no direct $CO_2$ or PFC emissions. The current status of this process, unaddressed questions, and prospects for further development are briefly reviewed.

## Oxy–fluoride vs. Chloride Molten Salt Electrolysis

Rare earth elements (REEs) are 'critical' materials for many existing and emerging technologies. Electrified transportation, wind power generation, magnetic data storage and even defense equipment rely on neodymium (Nd), praseodymium (Pr), and dysprosium (Dy) as key components of permanent magnets that enable these technologies. Demand for REEs has drastically increased over the past few decades and is predicted to continue to grow even further. Currently, the processing and refining of REEs involve environmentally hazardous and energy–intensive steps restricting their practice primarily to countries with less stringent environmental regulations. But heavy dependence on a single source for critical materials access poses serious supply chain risks. Developing innovative yet sustainable methods for refining critical metals is crucial for mitigating the environmental impacts caused by currently practiced REE processing methods.[1]

REE production involves many steps, i.e., mining, ore beneficiation, solvent extraction and purification as shown in **Fig. 1**. These yield rare–earth oxides, which are converted into metal via electrolysis. Here, we focus on neodymium (Nd) metal production although the overall approach to metal production is similar for other rare–earths. The state–of–the–art process for Nd production uses a $Nd_2O_3$–containing $NdF_3$–LiF molten salt electrolyte (at temperatures exceeding 1050 $^oC$) and a consumable carbon anode.[2] This oxy–fluoride process converts purified $Nd_2O_3$ and the carbon to Nd metal (at the cathode) and generates $CO_2$ (at the anode) – see **Fig. 1**. However, the low solubility of $Nd_2O_3$ implies transport limitations on oxygen species at the anode, resulting in an appreciable amount of perfluorocarbon (PFC) gas emissions in addition to $CO_2$. These greenhouse gases ($CO_2$, PFCs) make the conventional oxy–fluoride molten salt electrolysis (MSE) process undesirable. Additionally, the consumable carbon anode necessitates a 'batch' process, requiring frequent heating and cooling cycles, rendering it very energy–intensive (>8 kWh/kg, accounting for all energy inputs).[3] Despite these disadvantages, the state–of–the–art molten salt electrolysis (MSE) process does present some key advantages: molten salts offer excellent ionic conductivities and high diffusivities, and high–temperature operation provides fast reaction kinetics – these are essential features for process scale–up, for lowering overpotentials and energy consumption, and for achieving high electrolysis rates.[2,4]

Moderate– or high–temperature chloride–based molten salts present an alternative medium to conventional oxy–fluoride melts because they also possess exceptional transport and kinetics properties, while allowing for more energy–efficient and sustainable metal refining.[4] A comparison between the chloride–based MSE process and the state–of–the–art oxy–fluoride process is depicted in **Fig 1**. In the chloride MSE route, $Nd_2O_3$ is first converted into $NdCl_3$ via non–carbothermic reaction with HCl or $Cl_2$ gas. This is uniquely possible for rare–earths because their chlorides are thermodynamically more stable than their oxides.[2] The resulting chloride salt is then used as a feedstock for molten salt electrolysis. The medium for electrolysis can be a variety of chloride eutectic melts, e.g., LiCl–KCl (59:41 mole.% ratio), which are readily available, benign and provide a wide electrochemical potential operating window for electrolysis.[4] When operated at moderate temperatures, electrolysis yields solid Nd metal. $Cl_2$ gas, emitted at the anode, can be sold as it is a valuable co–product, or can be recycled into the upstream chlorination step. This process offers numerous advantages compared to the conventional route: (i) First and foremost, the chloride MSE route does not generate direct greenhouse gas ($CO_2$, PFCs) emissions, and thus is fully sustainable; (ii) $Cl_2$ evolution offers the possibility of using a dimensionally stable, catalytic anode which not only lowers anode overpotentials and thus the cell voltage and energy consumption, but also provides better process control including continuous processing[5-7]; (iii) Reactor design and electrolyte optimization can enable higher current efficiencies (>80%), thus enhancing overall process efficiency above and beyond the conventional oxy–fluoride process. These aspects are discussed in further detail below.

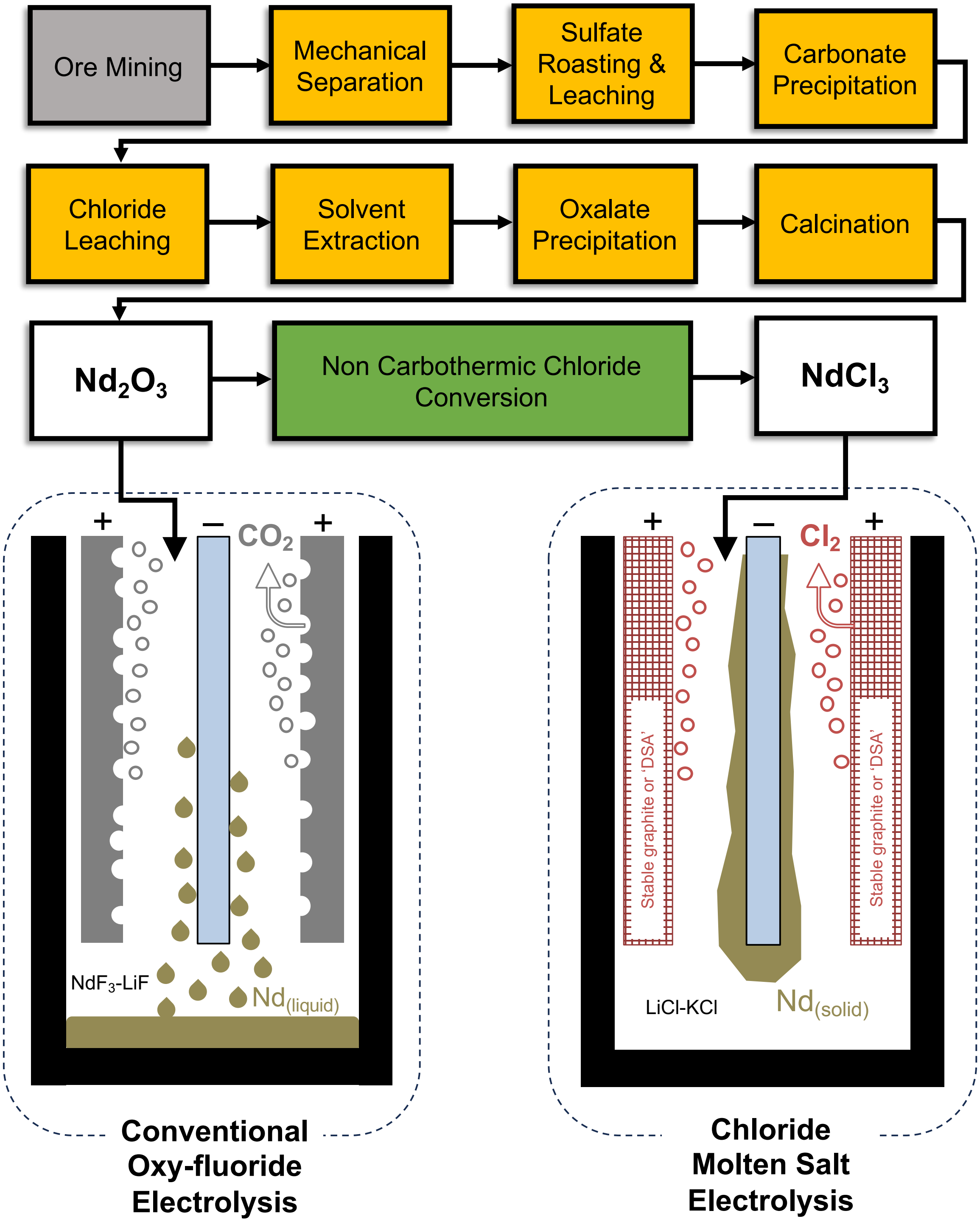

**Figure 1.** A process flow diagram and associated reactor schematics for both the conventional oxy–fluoride process and an alternative chloride–based electrolysis process. Whereas the chloride process requires first converting $Nd_2O_3$ to $NdCl_3$, it offers the benefits of eliminating $CO_2$ and PFC emissions, improved process control due to a more stable inter–electrode spacing provided by the non–consumable nature of the anode, and improved energy–efficiency on account of the higher current efficiency and lower cell voltage when using a catalytic DSA anode.

Specific energy consumption, i.e., the amount of energy required to produce a given mass of Nd metal (kWh/kg-Nd), is a metric that considers thermodynamics, overvoltage penalties associated with irreversibility factor during electrolysis, as well as inefficiencies from parasitic side reactions.[2] For the chloride MSE process, the thermodynamic potential for electrolysis of $NdCl_3$ (2.7 V) is higher than the oxy–fluoride process because $NdCl_3$ is more stable than $Nd_2O_3$. However, the chloride MSE process has significantly lower electrolyte resistance, improved species transport, and potentially offers fast kinetics at both the anode and the cathode. This allows the chloride process to operate at cell voltages that are at parity with or lower than the oxy–fluoride process. Considering the higher cathodic coulombic efficiency (discussed below) in chloride MSE, this process can enable much lower specific energies creating a more sustainable process by lowering the energy consumed as well as by eliminating direct greenhouse gas production.[2] A comparison of the relevant process parameters is provided in **Table 1**.

**Table 1.** Comparison of key process metrics between the conventional oxy–fluoride electrolysis and the chloride MSE alternative for Nd metal production.

| **Process Metric** | **Oxy–fluoride MSE** | **Chloride MSE** |
|---|---|---|
| Thermodynamic Potential | 2.5 V | 2.7 V |
| Cell Voltage | 4.0 V | < 4.0 V |
| Coulombic Efficiency | < 60% | > 80% |
| Specific Energy (kWh/kg) | 8.0* | < 2.79 |
| Direct $CO_2$ emissions | 0.2 kg-$CO_2$ / kg-Nd | none |

*electrical energy consumption is 3.7 kWh/kg; however, the need for heating batch reactors elevates the net energy consumption for the oxy–fluoride process. Chloride MSE opens up avenues for continuous processing, which could eliminate heating costs.

## Molten Salt Electrolysis in Chloride Melts – Fundamental and Practical Considerations

Here, a few key considerations in designing efficient rare–earth electrowinning processes, especially as they related to chloride MSE, are reviewed. A key aspect of process design in MSE of rare–earths, including Nd, is their multivalency, i.e., stability of multiple oxidation states in a halide medium. For Nd, $Nd^{3+}$ and $Nd^{2+}$ are both stable.[4] Thus, during electrolysis, $NdCl_3$ is first converted to $Nd^{2+}$ (very fast step [1] shown in Fig. 2), followed by reduction of $Nd^{2+}$ to Nd metal (relatively slow step [2] in Fig. 2). $Nd^{2+}$ is susceptible to diffusion away from the cathode, which represents an efficiency loss. To quantify this loss, an efficiency loss factor $\mathcal{L}$ may be defined as the ratio of the rate of $Nd^{2+}$ loss through diffusion to the rate of $Nd^{2+}$ reduction to metal. As shown in Fig. 2 (right), $\mathcal{L}$ is proportional to the $Nd^{2+}$ diffusion–limited mass transfer coefficient ($\mathcal{D}_{2+}/\delta$) and its concentration ($C_{2+}$), and inversely propositional to the exchange current density ($i_0$) and overpotential ($\eta$) for Nd deposition. Thus, to minimize $Nd^{2+}$ loss, the electrolysis process must be operated at very large overpotentials (as large as possible without triggering parasitic reactions such as LiCl or KCl electrolysis) – this ensures fast reduction of $Nd^{2+}$ to metal, thereby preventing $Nd^{2+}$ loss to the electrolyte. Also, strategies to employ porous separators or diffusion barriers are effective in preserving the $Nd^{2+}$ near the cathode and avoiding $Nd^{2+}$ oxidation back to $Nd^{3+}$ at the anode. Finally, temperature has a key role to play since increasing temperature increases $i_0$ and thus lowers the loss factor.

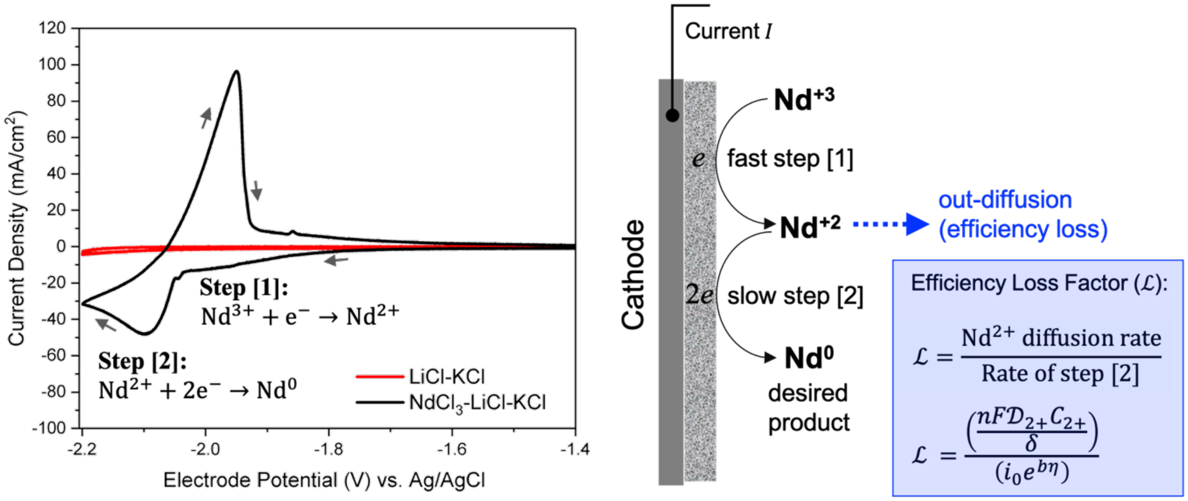

**Figure 2.** (left) Cyclic voltammogram (CV, at 100 mV/s) gathered on a tungsten working electrode immersed in molten LiCl–KCl eutectic electrolyte at 475 °C in the presence (*black curve*) and in the absence (*red curve*) of $NdCl_3$.[4] CV showing reduction of $NdCl_3$, first from $Nd^{3+}$ to $Nd^{2+}$, followed by reduction to $Nd^0$ (metal). (right) The intermediate $Nd^{2+}$ is prone to out–diffusion, which contributes to efficiency loss. Efficiency loss can be minimized by lowering the loss factor $\mathcal{L}$, which is defined as the ratio of the $Nd^{2+}$ diffusion rate to the Nd deposition rate via step [2].

Electrowinning of solid Nd sponge at the cathode is advantageous because, unlike conventional oxy–fluoride processing, the product Nd stays adhered to the cathode surface and thus continually experiences an externally imposed cathodic potential. This avoids the possibility of a spontaneous comproportionation reaction between Nd and the electrolyte via: $Nd + Nd^{3+} \rightarrow 2Nd^{2+}$. This reaction is thermodynamically favored[4] when an externally unpolarized Nd droplet contacts the electrolyte as is the case in the oxy–fluoride process (**Fig. 1**). However, producing solid Nd via chloride MSE comes with its own set of challenges. Since metal deposition in moderate– and high–temperature melts take advantage of the intrinsically high exchange current densities in these media, spongy or porous deposits are typically obtained.[2] As shown in **Fig. 3**, high purity (spot purity >92 at.%) deposits are easily obtained via chloride MSE; however, FIB–lift out followed by SEM/EDS reveals porosity in the deposited Nd metal. Electrolyte salts, e.g., LiCl and KCl, get entrapped in the Nd pores during electrolysis. This necessitates post–processing such as vacuum distillation or arc re–melting to purify the Nd so that it is suitable for application in NdFeB permanent magnet fabrication. Recent developments in the use of electrolyte additives are noteworthy in this context[8] – additives can enable more compact and thus ultra–high purity electrowon metal that circumvents the need for post–purification, thereby maintaining the energy requirements at a minimal value during metalmaking.

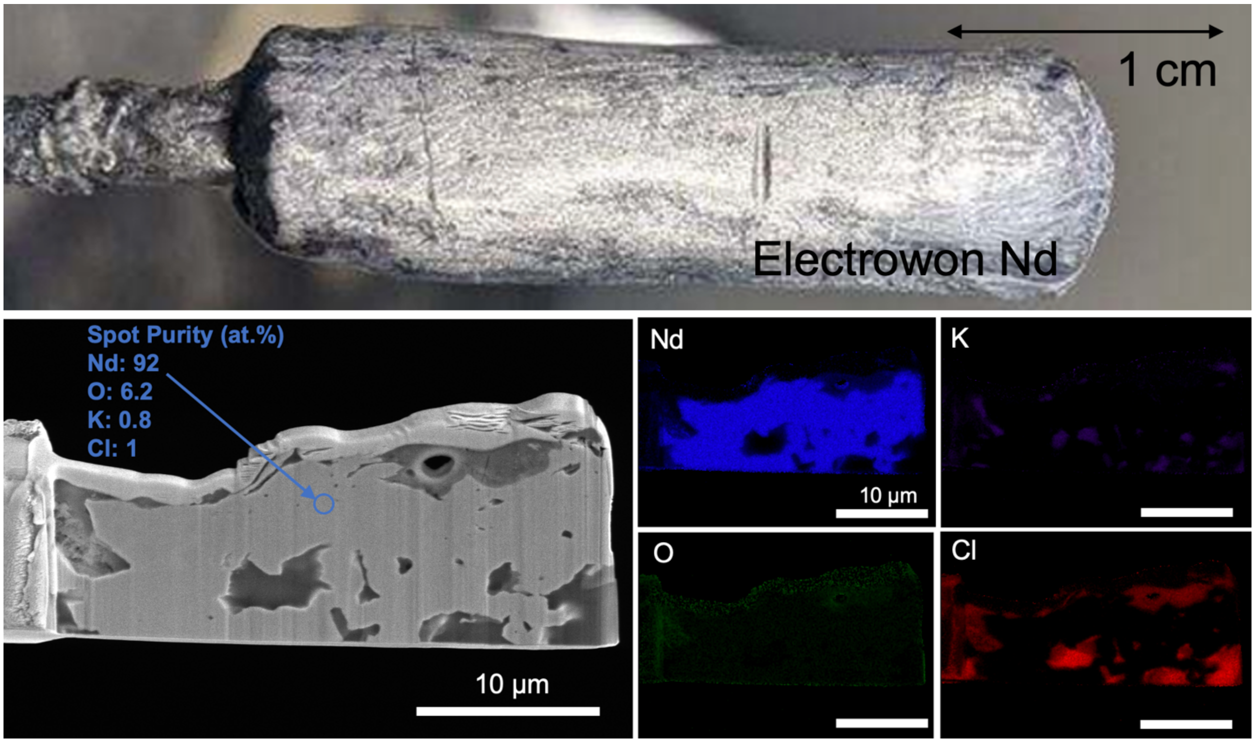

**Figure 3.** A Nd deposit produced by electrolysis at 200 mA/cm$^2$ in a 1.65 M $NdCl_3$ containing LiCl–KCl electrolyte at 500 °C (top). FIB lift out followed by SEM/EDS shows high–purity (92 at.%) Nd regions but also shows entrapped salt (likely LiCl) due to the porous nature of the Nd deposits.

To circumvent efficiency loss due to multivalency, and to mitigate salt entrapment when making solid Nd metal, higher–temperature (>860 °C) chloride MSE may be practiced. Several chloride mixtures, e.g., $BaCl_2$–KCl above 650 °C, are stable liquids at very high temperatures. When electrolysis is performed in such melts containing $NdCl_3$ ($\approx$2.4 M) at 860 °C, solid Nd is produced as evidenced in the CV in **Fig. 4**. Here, the negative scan direction yields Nd electrodeposition at cathodic potentials, but the reverse scan (going towards positive potentials from –2.0 V to –0.4 V vs. Mo quasi–reference electrode) exhibits Nd stripping because the solid Nd product is adhered to the tungsten (W) working electrode. Electrolysis at even higher temperatures (1050 °C, i.e., above the Nd melting point) also yields Nd plating as expected, but the stripping peak during the reverse scan is largely absent. This indicates that liquid Nd formed during the negative scan does not adhere to the cathode surface because liquid Nd (density = 6.9 g/cm$^3$) settles at the bottom of the electrolyte (density $\leq$ 3 g/cm$^3$) and thus is detached from the working electrode. This experiment suggests the possibility of electrowinning liquid Nd metal as has been explored recently by several other investigators.[9] Liquid metal plating exhibits very high exchange current densities (1-10 A/cm$^2$) compared to solid metal plating ($\approx$0.1 A/cm$^2$),[10] thus liquid Nd plating can lower the efficiency loss factor $\mathcal{L}$ (**Fig. 2**) as long as comproportionation related losses can also be minimized. Finally, liquid metal plating opens the possibility for a truly 'continuous' process in which metal is continually electrowon and separated – this route is attractive for scale–up, process control and overall process techno–economics.

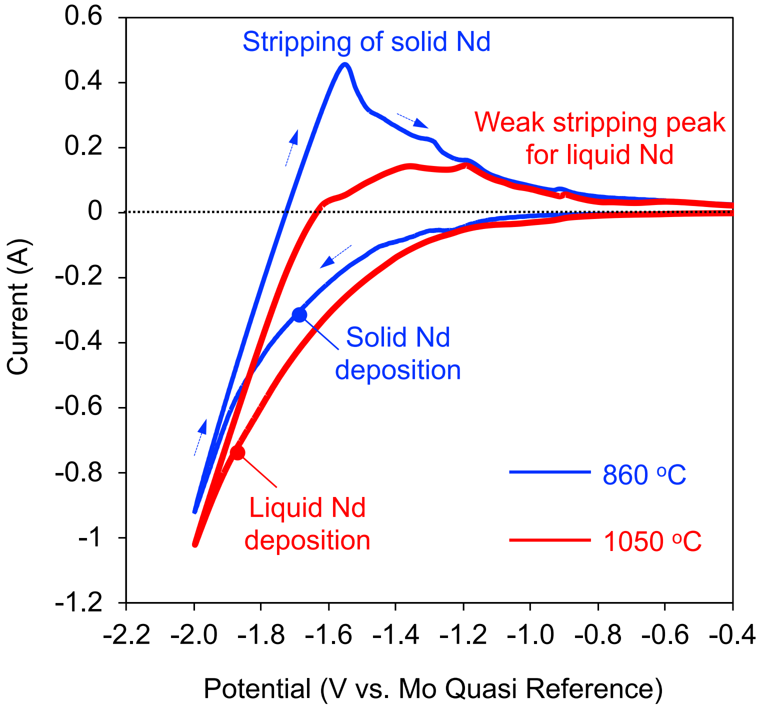

**Figure 4.** Cyclic voltammetry (200 mV/s) at a cylindrical W electrode in a $BaCl_2$–KCl molten salt containing 2.4 M $NdCl_3$. CV suggests that, at 860 °C (below the Nd metal melting point), solid Nd is formed during the cathodic scan direction. This Nd adheres to the W working electrode, and then undergoes oxidation during the reverse scan. However, at 1050 °C, liquid Nd is electrowon which separates (due to higher density than the electrolyte) from the W electrode, and thus is not re–oxidized during the reverse scan. This hints at the possibility of making liquid Nd via chloride MSE.

## Challenges and Prospects for Future Development of Chloride MSE

While the chloride–based MSE route is a promising method for enabling clean and sustainable rare–earth metal production across the globe, there still exist several unresolved challenges with this technology. Chlorine handling, particularly at high temperatures is a key safety concern. High temperature chlorine corrosion is more aggressive due to the increased rate of transport of chlorine through passivating surface oxide films but also due to the volatility of metallic chlorides.[11] While enhanced chlorine corrosion poses a materials challenge for chlorine handling systems, it also restricts the usability of traditional anode materials. Conventional titanium–coated mixed metal oxide (MMO) dimensionally stable anodes (DSA) are common in lower temperature aqueous chlor–alkali processes; however, the sintered oxide coating has been observed to delaminate from the titanium substrate presumably due to chlorine penetration leading up to corrosive attack of the underlying titanium during high–temperature chloride MSE. Graphite itself can act as a chlorine–evolving anode but it typically exhibits large overpotentials for chlorine evolution than MMO and can itself get oxidized to $CO_2$ in the presence of trace water or oxygen during chloride MSE. New DSA materials or stacks need to be developed to address these issues of long–term anode stability while also lowering the anodic overpotentials during chloride MSE.[12]

Despite the many advantages of the moderate–temperature chloride MSE process, it remains a ‘batch’ process around 500 °C. The electrowon solid Nd needs to be collected by removing the cathode from the electrolyte melt at the end of a batch. While a cathode ‘swap’ could be performed while the cell is held at temperature to minimize process interruptions, a truly ‘continuous’ process is very desirable for scalability, cost and energy–efficiency. Work is underway in our laboratory to develop a novel reactor and process design to continuously electrowin liquid Nd metal at 1050 °C in a chloride melt. Using an inert yet catalytic anode material, a low volatility chloride electrolyte (e.g., $BaCl_2$–KCl or $BaCl_2$–$SrCl_2$), and a reactor design to periodically siphon the molten metal akin to industrial Al electrowinning, a continuous process is possible. A continuous process would greatly reduce the specific energy consumption by eliminating the heating–cooling cycles necessary in the conventional oxy–fluoride route.

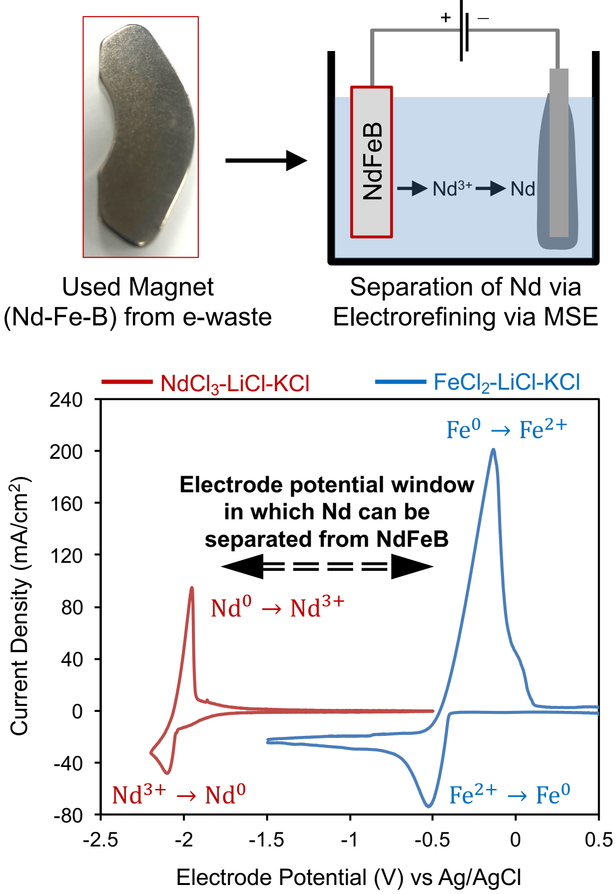

**Figure 5.** An exploratory concept for recovering Nd from used NdFeB magnets from electronic waste. Recovery is achieved via electrorefining in which Nd undergoes selective oxidation at the anode and is deposited as pure Nd metal at the cathode. Cyclic voltammetry of $NdCl_3$ and $FeCl_2$ containing LiCl–KCl melts confirms that selective Nd separation from NdFeB is feasible at anode potentials in the approximate range of –1.7 to –0.8 V vs. Ag/AgCl.

Implementation of chloride MSE requires a $NdCl_3$ feedstock. Producing a chloride feedstock first requires $Nd_2O_3$ production from ore via the established methods of ore processing (**Fig. 1**). In ore processing, a mixed rare–earth chloride stream undergoes treatment using an organic ligand such as oxalate to selectively precipitate $Nd^{3+}$. The oxalate is then calcined into $Nd_2O_3$, which releases $CO_2$. Targeting this chloride intermediate stream rather than a purified oxide could provide opportunities for upstream integration of chloride MSE, which would further reduce overall $CO_2$

emissions associated with primary Nd production. An even greener alternative feedstock is the large volume of used magnets in end–of–life devices such as hard disk drives and wind turbines. Additional reduction in $CO_2$ emissions can be achieved through recycling of the Nd in used magnets. It has been shown that oxide feedstocks derived from used magnets could provide a >50% reduction in greenhouse gas emissions compared to those produced from ore bodies.[13] Given that recycling also often involves an oxalate treatment followed by a calcination step, targeting the separation of Nd from NdFeB magnets into its chloride form ($NdCl_3$) for direct input into chloride MSE offers an opportunity to achieve nearly–complete circularity in rare–earths processing.

Note here that Nd separation from NdFeB may also be achieved electrolytically in an electrorefining cell (**Fig. 5**) with chloride molten salt as the electrolyte. Here, Nd from NdFeB is selectively oxidized to $Nd^{3+}$ (at the anode), which then undergoes reduction to pure Nd metal (at the cathode) – this has the potential to accomplish separation and purification in a single unit operation. Cyclic voltammetry of $NdCl_3$ and $FeCl_2$ containing melts confirms that selective separation of Nd can be achieved over a rather wide potential window (**Fig. 5**). However, a key challenge in realizing such separation is the slow solid–phase diffusivity of Nd out of the NdFeB anode. Utilizing micro–sized NdFeB particles with short diffusion lengths and high surface areas will be key to achieving industrially relevant rates of Nd separation and electrorefining. Specialized pulsing waveforms can be employed as well to maximize transport of Nd out of the solid particle and into $Nd^{3+}$ species while preserving selectivity.[14]

## Summary

Critical materials, such as rare–earth metals, are essential to numerous applications including clean energy; however, the present industrial practices for producing rare–earth metals involve environmentally–damaging and thus unsustainable chemical and electrochemical processes. For example, neodymium metal production using oxy–fluoride molten salt electrolysis is not only energy–inefficient but also generates potent greenhouses gases ($CO_2$, PFCs). An alternative moderate–temperature chloride–based molten salt electrolysis process can address these issues, providing energy–efficient and sustainable neodymium metal production. But more work is needed in advancing this process, i.e., developing catalytic yet stable chlorine–evolving anodes, 'continuous' metal recovery while enhancing its purity, and upstream or downstream (recycling) integration into the supply chain for minimizing the greenhouse gas emissions. The chloride MSE can be viewed as a platform technology. While it is being developed presently for rare–earth electrowinning, one can easily envision its broader application to rare–earth electrorefining and the electrolytic production of high–volume metals like Fe and Al. Presently, these high–volume metals industries account for nearly 10% of global greenhouse gas emissions. Thus, the chloride MSE process presents a huge opportunity in truly achieving sustainability if developed further for producing Fe, Al, Ti, Mg and other commodity metals.

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