Experimental Observations for Anode Optimization of Oxide Reduction Equipment

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The electrochemical behavior was investigated during the electrolysis of nickel oxide in LiCl-Li2O salt mixture at 650°C by changing several components. The focus of this work is to improve anode design and shroud design to increase current densities. The tested components were ceramic anode shroud porosity, porosity size, anode geometry, anode material, and metallic porous anode shroud. The goal of these experiments was to optimize and improve the reduction process. The highest contributors to higher current densities were anode shroud porosity and anode geometry.

Keywords: Oxide reduction, Electrolysis, Molten Salt, Process optimization, Nickel oxide

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1. Introduction

Pyroprocessing is one option for processing spent nuclear fuel released from light water reactors and faster reactors. In the case of spent nuclear fuel from light water reactor, the fuel is in form of an oxide, then the fuel can be chemically and/or electrochemically reduced in a molten salt mixture of LiCl-Li$_2$O at 650°C [1-3]. Several small-scale studies for uranium oxide reduction have been reviewed at Korea Atomic Energy Research Institute for the purpose of initiating larger scale non-radiological metal oxide reduction technology. In this paper, nickel oxide was chosen to be reduced (eq. 1) in a cathode basket and platinum was used as the anode material to evolve oxygen gas (eq. 2) during the reduction process.

\[
\text{Cathode: } \text{NiO} + 2e^- \rightarrow \text{Ni} + \text{O}^{2-} \quad (1)
\]

\[
\text{Anode: } 2\text{O}^{2-} \rightarrow \text{O}_2 + 4e^- \quad (2)
\]

An important aspect of the electrolysis process is that as the cathode potential becomes more negative, the reduction of the metal oxide occurs before lithium reduction in the electrolyte [4-8]. The applied cell potential of this work was maintained at 2.3 V versus a nickel/nickel oxide reference electrode. There exists a potential where NiO reduction may occur, but lithium metal is not being deposited onto the cathode. The goal is to produce nickel metal by optimizing or not limiting the current at the anode.

A small amount of Li$_2$O (1wt%) is an important factor which aides in the transport of the O$^{2-}$ ion through the salt [9-15]. Fig. 1 represents a schematic depiction of the cathode basket reducing the nickel oxide and the flow of the oxygen ion towards the anode where oxygen bubbles form. It is important to track the oxygen movement due to the likelihood of corrosion from structural materials [16-20].

The objective of this work is to optimize the anode where O$_2$ bubbles are formed by experimenting with different anode configurations, materials, and shrouds.

2. Experimental

The oxide reduction cell vessel contained LiCl and Li$_2$O salt mixture at 650°C. The cell is housed in an argon-atmosphere glovebox. Historically, two anode assemblies were used. Each assembly had an upper and lower portion. The upper portion, which is kept out of the salt, consists of baffles within a metallic shroud to trap salt vapors as an
argon sweep gas was used to aid in oxygen gas removal (Fig. 2(a)). The lower portion of the anode assembly had a solid ceramic shroud along with the platinum anode. The platinum anode was held in place by attaching a platinum rod which goes through the upper assembly where electrical connections can be made. The ceramic shroud is 330 mm long with a wall thickness of 3.175 mm, having an outer diameter of 76.5 mm. The cathode assembly has a similar setup involving an upper portion and lower portion. The upper portion had stainless steel baffles and metallic rods insulated with ceramic to contact the nickel oxide. The lower portion contained a stainless-steel mesh basket, containing the nickel oxide (Fig. 2(b)). The basket is 53.9 mm thick, 54 mm long, and 156 mm tall. The Poroplate 5-layer mesh had a 38-micron opening. The basket attaches to the upper assembly by bolts through the upper assembly.

A key component to the oxide reduction process is the anode shroud. The shroud allows the produced oxygen to be directed to the off-gas system where the oxygen concentration is measured and exhausted outside of the glovebox. Additionally, it protects the anode surface from non-soluble lithium metal which may be formed at the cathode. Four different anode shrouds were tested: solid ceramic (Fig. 3(a)), porous ceramic (Fig. 3(b)), large porous ceramic (Fig. 3(c)), and porous metallic stainless steel 316 (Fig. 3(d)); the ceramic shrouds were fabricated from MgO partially-stabilized zirconia. The solid ceramic does not allow a direct path of the oxygen ion movement to the anode during the electrochemical reduction process; thus, a porous ceramic shroud should yield insight towards facilitating the diffusion of the oxygen ion to the anode surface, resulting in higher current densities. The porous metal shroud was constructed to incorporate the idea of direct oxygen ion path with a rapid manufacturing turn around.

To investigate the oxygen ion movement during the reduction process, holes were incorporated into the anode shrouds. Fig. 4 illustrates the path of the ion; the red arrows represent how the ion must move down and around a solid anode shroud before contact with the anode where the two ion gains four electrons and is converted to oxygen gas. Higher oxide reduction efficiency can be expected with a higher flux through the shroud pores, which leads to an increase in operating throughput.

Anode geometry and composition were also investigated. A change in geometry could simplify the manufacturing process along with providing increased structural integrity. The possible change in composition could hinder corrosion of the anode, increasing its lifetime [21-24]. Theoretically, platinum can be damaged if the cell potential is higher than 3.2 V but this work will limit the potential to be 2.3 V
and should not damage the platinum anodes. The original wagon wheel design was constructed by platinum (99.95%, Johnson Matthey) as shown in Fig. 5(a). The geometry was redesigned to a planar type (114.3 × 47.63 × 5 mm, 99.95%, Johnson Matthey) for easy processability and better structural integrity. The material of the planar-type anode was platinum (Fig. 5(b), 99.95%, Johnson Matthey), ruthenium (Fig. 5(c), 99.9%, ESPI Metals), and iridium (Fig. 5(d), 99.95%, Johnson Matthey).

All experiments were performed in an inert glovebox (less than 5 ppm H₂O and less than 150 ppm O₂) in a furnace containing 65 kg of LiCl and 650 g of Li₂O at a temperature of 650°C. For all tests, the cathode was loaded with NiO pellets (American Elements, dia. 8 mm, length 6 mm, 99.9%). Either a dual or single anode setup was used and shall be noted when appropriate.

Table 1. Elemental composition of the salt before the first nickel oxide reduction experiment

<table>
<thead>
<tr>
<th>Sample</th>
<th>Cr (ppm)</th>
<th>Fe (ppm)</th>
<th>Ti (ppm)</th>
<th>Mo (ppm)</th>
<th>Sample</th>
<th>Cr (ppm)</th>
<th>Fe (ppm)</th>
<th>Ti (ppm)</th>
<th>Mo (ppm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Premix A</td>
<td>0.73</td>
<td>349.77</td>
<td>2,798.51</td>
<td>64.25</td>
<td>Mixing A</td>
<td>0.81</td>
<td>342.52</td>
<td>3,003.04</td>
<td>66.28</td>
</tr>
<tr>
<td>Premix B</td>
<td>0.82</td>
<td>365.84</td>
<td>3,015.8</td>
<td>68.17</td>
<td>Mixing B</td>
<td>0.61</td>
<td>341.15</td>
<td>2,927.03</td>
<td>65.95</td>
</tr>
<tr>
<td>Premix C</td>
<td>1.59</td>
<td>353.66</td>
<td>3,025.51</td>
<td>67.57</td>
<td>Mixing C</td>
<td>0.61</td>
<td>337.86</td>
<td>2,867.47</td>
<td>64.87</td>
</tr>
<tr>
<td>Average</td>
<td>1.05</td>
<td>356.42</td>
<td>2,946.61</td>
<td>66.67</td>
<td>Average</td>
<td>0.68</td>
<td>340.51</td>
<td>2,932.51</td>
<td>65.7</td>
</tr>
<tr>
<td>St. Dev.</td>
<td>0.38</td>
<td>6.84</td>
<td>104.8</td>
<td>1.72</td>
<td>St. Dev.</td>
<td>0.1</td>
<td>1.96</td>
<td>55.48</td>
<td>0.6</td>
</tr>
</tbody>
</table>

Table 2. Salt samples used to determine the concentration of lithium oxide in the salt

<table>
<thead>
<tr>
<th>Sample</th>
<th>wt% Li₂O</th>
<th>Sample</th>
<th>wt% Li₂O</th>
</tr>
</thead>
<tbody>
<tr>
<td>Premix A</td>
<td>1.05</td>
<td>Mixing A</td>
<td>1.06</td>
</tr>
<tr>
<td>Premix B</td>
<td>1.06</td>
<td>Mixing B</td>
<td>1.06</td>
</tr>
<tr>
<td>Premix C</td>
<td>1.06</td>
<td>Mixing C</td>
<td>1.06</td>
</tr>
<tr>
<td>Average</td>
<td>1.06</td>
<td>Average</td>
<td>1.06</td>
</tr>
<tr>
<td>St. Dev.</td>
<td>0.005</td>
<td>St. Dev.</td>
<td>N/A</td>
</tr>
</tbody>
</table>

3. Results and Discussion

Before the first nickel oxide reduction experiment, several salt samples were taken and analyzed using ICP-MS. Three dip samples were taken before a mixing procedure (Premix A–C). The salt was mixed using mixers for an hour and a half, and three addition salt samples were taken (Mixing A–C) during the mixing. Table 1 presents the impurities in the molten salt, including chromium, iron, titanium, and molybdenum. The chromium and iron are most likely appearing from the vessel walls and other stainless-steel pieces, while titanium and molybdenum are present from previous experiments.

The concentration of lithium oxide was also determined via titration before the first electrochemical nickel oxide experiment. Three samples were taken before the mixing procedure, along with three samples after the mixing process. Table 2 illustrates the concentration of lithium oxide to be around 1.06 weight percent, indicating a minimal difference between premixing and post mixing.
3.1 Porous Versus Solid Ceramic Shroud

An investigation was performed to compare the effectiveness of having a porous anode shroud. The cathode was loaded with NiO, and two platinum wagon wheel anodes were used. A constant 2.3 V cell voltage was applied between the cathode and each anode. Anodes 1 and 2 denote the current being measured for power supply 1 and 5, the plus indicates a positive current, or the current on the anodes. The anodes and anode shrouds were immersed 51 mm into the salt. Fig. 6(a) reveals the measured anode current on Anode 1, and Fig. 6(b) shows the current on Anode 2. On both anodes, it can be clearly observed that the current increase by approximately 15% with the porous anode shroud.

While comparing the solid and porous ceramic shrouds, oxygen concentration was measured in the off-gas system directly from the anodes, as well as in the furnace headspace. A fear with using a porous shroud is oxygen leaking into the head space (underneath the heat shields) resulting in a high degree of corrosion of the furnace components. Before each reduction experiment, a calibration of the oxygen sensors was performed to ensure accurate values. Fig. 7(a) is a plot of the oxygen coming from the anodes, showing higher oxygen levels using the porous shroud with the higher observed current. Fig. 7(b) reveals there was no

Fig. 6. (a) Electrical response of solid vs. porous ceramic shroud with a two anode set up (Anode 1), (b) Electrical response of solid vs. porous ceramic shroud with a two anode set up (Anode 2).

Fig. 7. (a) Oxygen concentration from the anodes, (b) Collected gases from the head space in the furnace showing no increase in oxygen.
increase in oxygen concentration in the head space.

Porosity size was studied to determine if an increase in porosity would yield a higher measured current. Again, a single cathode loaded with NiO was used along with two platinum wagon wheel anodes, and two large porous ceramic shrouds (recall the diameter of these holes are 3.175 mm, Fig. 3(c)). Fig. 8(a) presents the collected data when applying a cell potential of 2.3 V between the cathode and each anode. Both anodes and anode shrouds were inserted 51 mm into the salt. Comparing to the applied current at approximately 30 A in Fig. 6(a), use of the shroud with the larger holes resulted in a current of approximately 26.5 A. A depletion of NiO from the basket may have also caused a lower current response. More importantly oxygen was detected in the head space inside the furnace, suggesting the large hole ceramic shroud allowed the evolved oxygen gas to escape (Fig. 8(b)) under this structure and conditions. It should be noted some of the holes were above the salt level leading to a possible escape of the oxygen. This was validated by observing the readout of the glovebox oxygen sensor increasing.

### 3.2 Ceramic Versus Metal Anode Shroud

The metal shroud performance was compared to the porous 1.58 mm diameter ceramic shroud, both tests uti-
lized a clean platinum anode plate. Fig. 9 represents the overlay of the data from both experiments, showing a similar performance. There were also no elevated oxygen levels in the head space nor glovebox.

After the experiments with the metallic anode shroud, the assembly was removed from the furnace. The first observation was made on the component which connects the metallic anode shroud to the ceramic coupler. There appeared to be a film growth and discoloration (Fig. 10). The inside of the metal shroud and coupler was also documented to investigate any changes. There appeared to be an amorphous growth on the metal section (Fig. 11). The ceramic couple had discoloration but no damage or flaking. This growth can be attributed to the condensation of fine particulates or corrosion. There was enough material between the metal shroud and ceramic coupler to glue the two components together.

3.3 Anode Geometry and Anode Orientation

An investigation was performed to determine the effects of anode geometry. The platinum wagon wheel has higher surface area (10,832 mm²), whereas the platinum plate has a surface area of 5,974 mm² (when 50.8 mm of the plate and wagon wheel are inserted into the salt). Current was measured when applying a cell potential of 2.3 V. It is important to note, only one anode and one anode small hole, porous, ceramic shroud was used (recall in the previous tests two anodes and two anode shrouds were used). Once the currents were obtained, current densities were calculated for proper comparisons (Fig. 12). As shown in Fig. 12, there is clearly a large difference in current density and it can be concluded that the platinum plate optimizes the oxide reduction process under these conditions.

Oxygen concentration was also collected during the anode geometry investigation. Theoretically, at higher current densities there should be more oxygen generation. However, there does not appear to be a significantly higher oxygen concentration (Fig. 13(a)) with the Pt plate anode when compared with the wagon wheel. This could be attributed to the oxygen sensors and their associated issues. Oxygen was also monitored in the head space or the gas phase outside the porous ceramic anode shroud. Though the platinum plate has higher overall oxygen concentration, there is no increase in oxygen concentration as the process progresses (Fig. 13(b)). From the presented data there does not appear to be oxygen in the head space and the difference in values can be attributed to the fluctuations in the oxygen sensors.

Due to the planar geometry not being identical on all axes, it is important to determine the effect of anode orientation towards the cathode. Ideally, the optimal orientation of the anode is to maximize the surface area pointing towards the cathode. Fig. 14 is a visual representation of
different anode orientations with respect to the cathode basket.

To test the orientation of the anode plate, 2.3 V was applied between the anode and cathode, while a small hole (1.58 mm diameter) porous ceramic shroud was used. The anode and shroud were inserted 51 mm into the salt. The current was recorded for approximately 100 minutes, then the plate was rotated by 45-degrees. Fig. 15 shows the resultant current over the three different orientations. The first illustration in the above figure denotes the 0-degree mark, followed by the second to represent 45-degree, and finally the last orientation to be 90-degree. The plot suggests there does not seem to be a preference on anode orientation due to the increasing current. The oxygen concentration is reported as volume percent at a flowrate of 1 L min⁻¹.

Throughout the anode orientation experiment, the oxygen was recorded from the anode along with the head space gas. Fig. 16(a) is a plot of the oxygen concentration throughout the orientation experiment. This figure agrees with the above figure suggesting an increase in current also increases the oxygen produced. From these figures it is clear the anode plate may have been turned too early and
equilibrium was not established. Fig. 16(b) again confirms the lack of oxygen in the head space of the furnace during the experiment.

### 3.4 Platinum Versus Ruthenium Versus Iridium Anodes

The plate material was also tested and changed to ruthenium. The ruthenium plate was attached to a silver rod by utilizing a tac welding technique. Silver was chosen due to its high electrical conductivity and corrosion resistance from oxygen in the head space of the furnace. Fig. 17(a) is a photograph of the new ruthenium plate welded to a silver rod. Fig. 17(b) is a photograph of the plate after passing 117 Ah through the system, resulting in discoloration on the surface part immersed in the salt. To clean the surface of the anode for reuse, a 7-molar solution of HCl was used (Fig. 17(c)). After a 5-hour soak time the surface appeared to have less build up; however, the surface area used for oxide reduction after the rinse appeared to have a blue tint, this may be the initial formation of ruthenates.

After applying a cell potential of 2.3 V between the anode and cathode, the current at the anode displayed a similar decreasing trend (compared with other rectangular anode plates) when current versus time was observed (Fig. 18(a)). Fig. 18(b) presents the voltages on the anode and cathode when applying a 2.3 V of cell voltage. These voltage plots are useful to ensure the anode does not undergo dissolution.

Another piece of equipment studied was the anode shroud material. Recall from the previous experiments a MgO partially-stabilized zirconia ceramic was used (solid, 1.58 mm diameter small hole porosity, and 3.175 mm diameter large hole porosity). The ceramic shrouds are difficult to obtain and require a large amount of time to manufacture. Furthermore, there could be a limit on geometry during the manufacturing process. To avoid this potential problem, a stainless steel 316 anode shroud was created. Fig. 3(d) shows a white ceramic collar towards the top, used for electrical
isolation. In the same figure there is a stainless-steel mesh with an opening of 100-micron Poroplate to allow the diffusion of the oxygen ion to be enhanced. A platinum plate facing the cathode was used to test this metallic shroud.

The anode assembly with metallic shroud and platinum plate was inserted 51 mm into the salt. At this point the power supply was used and applied a cell voltage of 2.3 V between the cathode and anode. The anode plate was ensured to be facing the cathode. Fig. 19(a) represents the measured current approximately at 30 A. Fig. 19(b) reveals an increase in anode and cathode potential as the experiment continued. This effect could be attributed to a
film growth having a non-conductive surface.

While conducting experiments using various plates and anodes, the oxygen from the anode were also measured. In the previous experiment with a porous ceramic shroud with a platinum anode immersed by 50.8 mm, approximately 10% of oxygen was read at a flow rate of 1 L min⁻¹. Fig. 20 represents the measured oxygen resulting in approximately 9% oxygen. The head space gas was also monitored, indicating that there was no leak of oxygen out of the anode shroud and assembly.

The next anode material chosen for testing was iridium with metal shroud. This plate had the same dimensions as the platinum and ruthenium geometry. However, the surface properties may be of benefit during metal oxide reduction in this salt. The iridium anode was placed inside the metal anode shroud and lowered 107.95 mm into the salt, this is the maximum coverage to investigate possible highest currents. Again, a cell voltage of 2.3 V was applied between the anode and cathode. A total of 337.4 Ah was applied. Fig. 21(a) is a photograph of the new iridium plate attached to the silver rod. The experiment was terminated due to a decrease in current. Fig. 21(b) shows the iridium plate after the experiment with a surface film on the surface. A small chip on film towards the bottom right corner was found, which indicates that the film has a possibility of spalling off into the salt during the reduction experiment.

The currents on the anode and cathode were recorded during the experiment. Fig. 22(a) plots the current (the break in the current denotes restarting the power supply the following day). Fig. 22(b) shows a voltage plot as a function of time. Again, there is an increase in voltage
which could be a result from the surface film growth.

For a meaningful comparison, all individual experiments had their current densities calculated and overlaid in one plot as shown in Fig. 23(a). The letter following the immersion depth represents the shroud material (C for ceramic and M for metallic). Recall the Pt Wheel was inserted by 51 mm and had a solid ceramic shroud. This resulted in the poorest performance of the group, meaning the experiment had the lowest current density over time. The Pt plate inserted by 51 mm with the small hole porous shroud seems to have performed the best; however, this was also the shortest test. The ruthenium also had a high current density but quickly began to decrease. Additional investigations are needed into anode film growth and the effect upon current densities. Fig. 23(b) tracks the voltages from the different anode materials during the reduction experiments to follow the dissolution potentials.

3.5 Anode Film Investigations

Recall a statement was made about observing film on the plate geometry design anodes. This next section shall describe the film on the platinum anode plate. After the platinum anode plate was removed from the furnace and allowed to cool, an attempt was made to mechanically scrape away the film; however, the scrape was unsuccessful. Having prior knowledge that 7 molar HCl solution removed the film observed on the platinum wagon wheel, a similar solution was created where the platinum rectangle anode was soaked in solution. Surprisingly, the film did not fully dissolve. The platinum rectangle anode was then rinsed with distilled water and mechanical means to remove the film was attempted. A thin putty knife was able to fit between the surface of the plate and the film. The film was removed successfully with minimal force. Fig. 24 is a photograph
after the HCl soak and partial scrape, showing the removal of the film. It should be noted that the surface of the platinum anode surface appears to be in good condition after removal of the film.

The characteristics of the film (after HCl rinse) was investigated including growth, magnetism, and resistivity. The film was measured to be approximately 1/2-mm. An interesting topic to be investigated would be the limiting condition of the growth or if spalling would occur and continue to grow. This flake was then pulverized and mixed before inserting a magnet. There appears to be a small fraction of constituents that adhered to the magnet (Fig. 25). The most likely elements to be magnetic would be iron, nickel, and cobalt (respectively). These elements could appear in the salt from corrosion processes from the stainless-steel components or Haynes. Electrical conductivity on the surface of the anode is critical for optimized oxide reduction. A Fluke multimeter was used to measure the conductivity of the film with the probes approximately 13 mm apart. Clearly the growth has high resistance effectively lowering the reaction rate of electrochemically reducing nickel oxide.

A similar approach was taken to the iridium film to compare this information to the platinum anode film. The characteristics of interest were growth thickness, magnetism, and resistivity. Figs. 26(a) and 26(b) are photographs of the anode surface before and after (respectively) the nickel oxide reduction. There appears to be a chip of the film missing, this could be attributed to the thermal shock while removing the anode from the molten salt. Further investigation is needed to determine if a higher temperature gradient could remove the growth.

The last metal to be utilized as an anode was ruthenium. Again, a similar comparison was performed to determine growth, magnetism, and resistivity. Figs. 27(a) and 27(b) are photographs before and after (respectively) nickel oxide reduction. Note the surface finish in Fig. 27(a), having a non-smooth appearance. This can be accredited to the difficulty in manufacturing a plate from ruthenium. The result would be a slightly higher surface area when compared to the platinum and iridium, but this was not quantified.
There was an interesting observation which occurred after the HCl soak. The active surface area had a blue hue. This may be an indicator for the formation of a ruthenium, oxygen, and lithium compound (ruthenates). Fig. 28 is a photograph of the ruthenium anode after the HCl soak showing a contrast between the active surface area and the area not in contact with the salt during the nickel oxide reduction.

According to the comparison, reduction performance was significantly different with different materials of anode. Ruthenium appeared to be the best candidate for nickel oxide reduction followed by platinum and/or iridium. Ruthenium’s performance was the longest per amp-hour before electrochemical fouling was experienced. Table 3 compiles all the measured values. It is important to note these values should be normalized for amp-hours utilized.

4. Conclusion

The oxide reduction process is a promising technology. The use of this process can aid in certain metal production in molten salts. During the reduction of nickel oxide in 65 kg LiCl and 1 kg of Li₂O at 650℃, the process was optimized by understanding the O²⁻ ion movement to the anode. Having a porous shroud aided in higher currents, therefore suggesting a more rapid reduction. However, too large holes seemed to be detrimental, since the oxygen gas was not captured and removed from the system. In either case, a slow immersion rate showed the ceramic did not break or crack. At 650℃ there is a high chance of oxygen corroding the stainless-steel vessel and other components. The largest optimization was changing anode geometries from a wagon wheel to planar with a porous anode shroud. Anode material was investigated and resulted in a non-conductive film growth when electrochemically reducing nickel oxide.

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Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper. The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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