Electrochemical Properties of 
Cathode Materials LiNiO₂ 
and LiNiₓ₊₁Mₓ₋₁O₂ (M = Zn²⁺, Al³⁺ and Ti⁴⁺)

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Abstract: LiNiₓ-TiO₂ (y = 0.000, 0.012, 0.025, 0.050, 0.100 and 0.150) were synthesized by ball milling and the solid-state reaction method. The optimum condition to synthesize a cathode material LiNiₓ₀.₇₅Ti₀.ₒ₅O₂ is milling for 28 h and then preheating at 600°C for 16 h and finally calcining at 750°C for 30 h O₂ stream. LiNiₓ₀.₇₅Ti₀.₂₅O₂ (0 ≤ y ≤ 0.150) synthesized under this optimum condition have the α-NaFeO₂ structure of the rhombohedral system (space group, R ₃ m). The values of I₀₀₀/I₁₁₀ for y = 0.000, 0.012 and 0.025 are near 1.2, indicating these samples have the phase with the nearly stoichiometric composition. The samples with y = 0.012 and 0.025 show good splitting of 006 and 102 peaks and of 108 and 110 peaks, indicating the decrease in cation mixing. The particle size decreases as the value of y increases. All the samples except that with y = 0.000 exhibit relatively homogeneous particle size. The voltage vs. discharge capacity curves for y = 0.012 and y = 0.025 exhibit three distinct plateaus corresponding to phase transitions. Among LiNiₓ₀.₇₅TiO₂ (0 ≤ y = 0.150), LiNiₓ₀.₇₅Ti₀.₂₅O₂ has the largest first discharge capacity 154.8 mAh g⁻¹ and the relatively good cycling performance (77% at n = 10). For Ti in the composition LiNiₓ₀.₇₅Ti₀.₂₅O₂, Zn²⁺ and Al³⁺ were substituted. Electrochemical studies of LiNiₓ₀.₇₅Mₓ₋₁O₂ (M = Zn²⁺, Al³⁺ and Ti⁴⁺) show that the plateaus in the voltage vs. discharge capacity curve become more distinct and the voltage of the plateau corresponding to the phase transition from M to H increases as M varies from Zn²⁺ to Al³⁺ and then to Ti⁴⁺. LiNiₓ₀.₇₅Ti₁₋₂ₓO₂ has the largest first discharge capacity and a relatively good cycling performance compared with LiNiₓ₀.₇₅Mₓ₋₁O₂ (M = Zn²⁺ and Al³⁺). This sample has the largest value of c/a and the smallest particles. LiNiₓ₀.₇₅Alₓ₀.₂₅O₂ has the best cycling performance (98% at n = 10).

Keywords: LiNiO₂, substitution of Zn²⁺, Al³⁺ and Ti⁴⁺, ball milling, solid-state reaction method, discharge capacity, cycling performance

Introduction

The transition metal oxides such as LiCoO₂ (Ozawa, 1994; Alcantara et al., 1997; Peng et al., 1998), LiNiO₂ (Dahn et al., 1990, 1991; Marini et al., 1991; Ebner et al., 1994) and LiMn₂O₄ (Tarascon et al., 1991; Song and Ahn, 1998, Song et al., 1999; Ahn and Song, 2000) have been

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investigated as cathode materials for lithium secondary batteries. LiMn$_2$O$_4$ is very cheap and does not bring about environmental pollution, but its cycling performance is not good. LiCoO$_2$ has a large diffusivity and a high operating voltage and it can be easily prepared. However, it has a disadvantage that it contains an expensive element, Co. LiNiO$_2$ is a very promising cathode material since it has a large discharge capacity (Nishida et al., 1997) and is relatively excellent from the viewpoints of economics and environment. However, its preparation is very difficult as compared with LiCoO$_2$ and LiMn$_2$O$_4$.

It is known that Li$_{1+x}$Ni$_{1-x}$O$_2$ forms rather than stoichiometric LiNiO$_2$ during preparation. This phenomenon is called cation mixing. Excess nickel occupies the Li sites, destroying the ideally layered structure and preventing lithium ions from easy movement for intercalation and deintercalation during cycling. This results in a small discharge capacity and a poor cycling performance. To solve the problem of cation mixing, Co$^{2+}$, Al$^{3+}$, Mn$^{2+}$ and Ti$^{4+}$ ions were substituted for lithium ion in LiNiO$_2$ (Gao et al., 1998; Brousseley, 1990; Caurant et al., 1996). According to Amine et al. (2002) and Gao et al. (1998), the substitution of Ti for Ni resulted in a large discharge capacity and a good cycling performance.

In this study, the optimum conditions to synthesize LiNiO$_2$ were investigated and LiNi$_{1-y}$M$_y$O$_2$ (M = Zn$^{2+}$, Al$^{3+}$ and Ti$^{4+}$, y = 0.005, 0.01, 0.025, 0.05 and 0.1) were synthesized under the optimum conditions for the synthesis of LiNiO$_2$. In addition their electrochemical properties were measured.

Materials and Methods

LiOH·H$_2$O (Aldrich Co., purity 98%), Ni(OH)$_2$ (Aldrich Co., purity 99%), ZnO (Aldrich Co., purity 99.9%), Al(OH)$_3$ (High Purity Chemical Laboratory Co., purity 99.99%) and TiO$_2$ (ananase)(Aldrich Co., purity 99%) were used as starting materials in order to synthesize LiNi$_{1-y}$M$_y$O$_2$ by ball milling and solid-state reaction method.

The experimental procedure of this work is given schematically in Fig. 1. Mixtures of starting materials in the compositions LiNi$_{1-y}$M$_y$O$_2$ were mixed by ball milling in acetone and dried in a drying oven. They were then preheated at 600°C for 16 h in air and palletized. This pellet was heated in a heating rate 50°C h$^{-1}$ and calcined at 750, 800 and 850°C for 30 h in O$_2$ stream. Then it was cooled in a cooling rate 100°C h$^{-1}$.

The phase identification of the synthesized samples was carried out by X-ray Diffraction (XRD) analysis using Cu-K$_\alpha$ radiation. The X-ray diffractometer was Rigaku III/A type. The scanning rate was 4°/min and the scanning range of diffraction angle (2θ) was 10° < 2θ < 80°. The morphologies of the samples were observed using a field emission scanning electron microscope (FE-SEM). The particle size distributions and the specific surface areas of the samples were analyzed by a particle size analyzer (Malvern Instruments).

The electrochemical cells consisted of LiNi$_{1-y}$M$_y$O$_2$ as a positive electrode, Li foil as a negative electrode and electrolyte [Purelyte (Samsung General Chemicals Co., Ltd.)] prepared by solvating 1M LiPF$_6$ in a 1:1(volume ratio) mixture of Ethylene Carbonate (EC) and diethyl carbonate (DEC). A Whatman glass-fiber was used as the separator. The cells were assembled in an argon-filled dry box. To fabricate the positive electrode, 85wt.% synthesized oxide, 10wt.% acetylene black and 5wt.% Polyvinylidene Fluoride (PVDF) binder solved in 1-Methyl-2-pyrrolidinone were mixed. By introducing Li metal, Whatman glass-fiber, positive electrode and the electrolyte, the
cell was assembled in a glove box filled with argon. All the electrochemical tests were performed at room temperature with a potentiostatic/galvanostatic system. The cells were cycled at a current density of 193 μA cm⁻² corresponding to 0.1 C rate between 2.8 and 4.3V.

**Results and Discussion**

The X-ray diffraction (XRD) patterns of LiNi₀.₉₇₅Ti₀.₀₂₅O₂ powders preheated at 600°C for 16 h after milling for various times had peaks corresponding to the LiNiO₂ phase and peaks for Li₂CO₃ phase. The peaks for the LiNi₀.₉₇₅Ti₀.₀₂₅O₂ powder milled for 28 h showed the highest intensities, indicating that the phases developed well. Therefore we chose 28 h for the ball milling time.

Figure 2 shows the XRD patterns of LiNi₀.₉₇₅Ti₀.₀₂₅O₂ powders calcined at 750, 800 and 850°C for 30 h in O₂ stream (after milling 28 h and preheating at 600°C for 16 h). They exhibit only the peaks corresponding to the LiNiO₂ phase. The intensities of the peaks increases and the splitting of 108 and 110 peaks become distinct as the calcining temperature rises.

Figure 3 shows the FE-SEM micrographs of LiNi₀.₉₇₅Ti₀.₀₂₅O₂ powders calcined at 750, 800 and 850°C for 30 h in O₂ stream (after milling 28 h and preheating at 600°C for 16 h). The
Fig. 2: XRD patterns of LiNi$_{0.575}$Ti$_{0.425}$O$_2$ powder calcined at 750, 800 and 850°C for 30 h in O$_2$ stream.

(a) 750°C, (b) 800°C, (c) 850°C for 30 h in O$_2$ stream.

Fig. 3: FE-SEM micrographs of the LiNi$_{0.575}$Ti$_{0.425}$O$_2$ powder calcined at (a) 750°C, (b) 800°C and (c) 850°C for 30 h in O$_2$ stream.

Fig. 4: Variations of discharge capacity at 0.1C with the number of cycles for LiNi$_{0.575}$Ti$_{0.425}$O$_2$ calcined at 750°C, 800°C and 850°C.
Table 1: Lattice parameters and intensity ratio of 003 and 104 peaks, $I_{003}/I_{104}$, for LN$\text{Ni}_{x}$Ti$\text{O}_{y}$ (0 ≤ y ≤ 1.50).

<table>
<thead>
<tr>
<th>y in LN$\text{Ni}<em>{x}$Ti$\text{O}</em>{y}$ (0 ≤ y ≤ 1.50)</th>
<th>$a_{003}$ (Å)</th>
<th>$c_{104}$ (Å)</th>
<th>c/a</th>
<th>$I_{003}/I_{104}$</th>
</tr>
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<tbody>
<tr>
<td>0.000</td>
<td>2.873</td>
<td>14.225</td>
<td>4.951</td>
<td>1.155</td>
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<td>0.012</td>
<td>2.875</td>
<td>14.195</td>
<td>4.937</td>
<td>1.258</td>
</tr>
<tr>
<td>0.025</td>
<td>2.876</td>
<td>14.194</td>
<td>4.935</td>
<td>1.133</td>
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<tr>
<td>0.050</td>
<td>2.877</td>
<td>14.182</td>
<td>4.930</td>
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<td>14.153</td>
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</tr>
<tr>
<td>0.150</td>
<td>2.884</td>
<td>14.154</td>
<td>4.907</td>
<td>0.949</td>
</tr>
</tbody>
</table>

Particle size of the sample increases as the calcining temperature rises. The sample calcined at 750°C has the most homogeneous particle size.

Figure 4 shows the variations of discharge capacity at 0.1 C rate with the number of cycles (n) for LN$\text{Ni}_{0.75}$Ti$0.025$O$_2$ powders calcined at 750°C, 800°C and 850°C for 30 h in O$_2$ stream (after milling 28 h and preheating at 600°C for 16 h). The sample calcined at 750°C has the largest first discharge capacity and the similar cycling performance compared with the samples calcined at 800°C and 850°C. Therefore, we chose 750°C for the calcining temperature. LN$\text{Ni}_{1.27}$Ti$0.025$O$_2$ powder calcined at 750°C for 30 h in O$_2$ stream (after milling 28 h and preheating at 600°C for 16 h) showed relatively strong intensities of peaks in XRD pattern and had the smallest particles and the most homogeneous particle size compared with the samples calcined at 800°C and 850°C.

The XRD patterns of LN$\text{Ni}_{y}$Ti$\text{O}_{z}$ powders calcined at 750°C for 30 h in O$_2$ stream (after milling 28 h and preheating at 600°C for 16 h) were analyzed to have the α-NaFeO$_2$ structure of the rhombohedral system (space group, R3m).

Table 1 shows lattice parameters a, c and intensity ratio of 003 and 104 peaks, $I_{003}/I_{104}$, for LN$\text{Ni}_{x}$Ti$\text{O}_{y}$ (0 ≤ y ≤ 1.50) calcined at 750°C for 30 h in O$_2$ stream. It is known that the intensity ratio of 003 and 104 peaks can be used to investigate the degree of cation (Li$^+$ and Ni$^{2+}$) mixing. When $I_{003}/I_{104}$ is smaller than 1.2, the cation mixing occurs. On the other hand, when $I_{003}/I_{104}$ is larger than 1.2, the composition of the sample is completely stoichiometric (Morales et al., 1990; Choi et al., 1996). The values of $I_{003}/I_{104}$ for y = 0.100 and 0.150 compositions are 1.0 and 0.949, respectively, while those for y = 0.000, 0.012 and 0.025 are near 1.2. This indicates that the LN$\text{Ni}_{x}$Ti$\text{O}_{y}$ (y = 0.012 and 0.025) samples have the nearly stoichiometric compositions. It is also known that the splitting of two peaks 006 and 102 or 108 and 110 indicates the decrease in cation mixing. The splitting of 006 and 102 peaks occurs well for the samples with y = 0, 0.012 and 0.025, but it does not occur for the samples with y = 0.1 and 0.15. The splitting of 108 and 110 peaks occurs well for the samples with y = 0.000, 0.012 and 0.025 and 0.050, but does not occur for the samples with y = 0.100 and 0.150.

The FE-SEM micrographs of the LN$\text{Ni}_{x}$Ti$\text{O}_{y}$ powders calcined at 750°C for 30 h in O$_2$ stream showed that the particle size decreased as the value of y increased. All the samples except that with y = 0.000 exhibited relatively homogeneous particle size.

Figure 5 shows the voltage vs. discharge capacity curves of the first cycle for LN$\text{Ni}_{x}$Ti$\text{O}_{y}$ (y = 0.000, 0.012, 0.025, 0.050, 0.100 and 0.150) calcined at 750°C for 30 h in O$_2$ stream. LN$\text{Ni}_{0.75}$Ti$0.025$O$_2$ has the largest first discharge capacity. The curves for y = 0.012 and y = 0.025 exhibit three distinct plateaus where phase transitions occur from hexagonal structure (H$_2$) to hexagonal structure (H$_3$), hexagonal structure (H$_2$) to monoclinic structure (M) and from monoclinic structure (M) to hexagonal structure (H$_3$).

Figure 6 shows the variations of discharge capacity at 0.1 C rate with the number of cycle (n) for LN$\text{Ni}_{x}$Ti$\text{O}_{y}$ calcined at 750°C for 30 h in O$_2$ stream. The cycling performance
Fig. 5: Voltage vs. discharge capacity curves of the first cycle for LiNi<sub>y</sub>Ti<sub>1-y</sub>O<sub>2</sub> (y = 0.000, 0.012, 0.025, 0.050, 0.100 and 0.150)

Fig. 6: Variations of discharge capacity with the number of cycles for LiNi<sub>y</sub>Ti<sub>1-y</sub>O<sub>2</sub> (a) y = 0.000 (b) y = 0.012 (c) y = 0.025 (d) y = 0.050 (e) y = 0.100 and (f) y = 0.150

(percentage of discharge capacity to the first discharge capacity) of LiNiO<sub>2</sub> at n = 10 is 60%. The cycling performance at n = 10 increases as the value of y increases. They are 63, 77 and 88%, respectively, for the samples with y = 0.012, 0.025 and 0.050. The samples with y = 0.100 and 0.150 have very small discharge capacities. When Ti is substituted for Ni in order to improve the cyclability of LiNiO<sub>2</sub>, the composition LiNi<sub>0.975</sub>Ti<sub>0.025</sub>O<sub>2</sub> has the largest first discharge capacity 154.8 mAh g<sup>-1</sup> and a relatively good cycling performance (77% at n = 10).

The above results show that the composition LiNi<sub>0.975</sub>Ti<sub>0.025</sub>O<sub>2</sub> has the best electrochemical properties among the compositions LiNi<sub>y</sub>Ti<sub>1-y</sub>O<sub>2</sub> (0 ≤ y ≤ 0.150). Instead of Ti in the compositions LiNi<sub>0.937</sub>Ti<sub>0.062</sub>Y<sub>0.062</sub>O<sub>2</sub> Zn (with atomic value +2) and Al (with atomic value +3) were substituted.
Table 2: Lattice parameters and intensity ratio of 003 and 104 peaks, $I_{003}/I_{104}$, LiNi$_{0.57}$M$_{0.43}$O$_2$ (M = Zn$^{2+}$, Al$^{3+}$ and Ti$^{4+}$) in LiNi$_{0.96+\delta}$M$_{0.04}$O$_2$ (M = Zn$^{2+}$, Al$^{3+}$ and Ti$^{4+}$)

\[
\begin{array}{cccccc}
M & a_{\text{L}} (\AA) & c_{\text{L}} (\AA) & c/a & I_{003}/I_{104} \\
Zn^{2+} & 2.875 & 14.150 & 4.921 & 1.379 \\
Al^{3+} & 2.870 & 14.151 & 4.930 & 1.400 \\
Ti^{4+} & 2.876 & 14.194 & 4.935 & 1.133 \\
\end{array}
\]

Fig. 7: XRD patterns of LiNi$_{0.57}$M$_{0.43}$O$_2$ (M = Zn$^{2+}$, Al$^{3+}$ and Ti$^{4+}$) powders after calcining at 750°C for 30 h in O$_2$ stream

Figure 7 shows the XRD patterns of LiNi$_{0.97}$M$_{0.03}$O$_2$ (M = Zn$^{2+}$, Al$^{3+}$ and Ti$^{4+}$) powders calcined at 750°C for 30 h in O$_2$ stream (after milling 28 h and preheating at 600°C for 16 h). All the samples were analyzed to have the α-Fe$_2$O$_3$ structure of the rhombohedral system (space group: R3m). The LiNi$_{0.97}$Al$_{0.03}$O$_2$ exhibits distinct splitting of 006 and 102 peaks and of 108 and 110 peaks, indicating very small amount of cation mixing. The LiNi$_{0.97}$Zn$_{0.03}$O$_2$ also shows relatively good splitting of 006 and 102 peaks and of 108 and 110 peaks.

Table 2 shows the lattice parameters and intensity ratio of 003 and 104 peaks, $I_{003}/I_{104}$, for LiNi$_{0.97}$M$_{0.03}$O$_2$ (M = Zn$^{2+}$, Al$^{3+}$ and Ti$^{4+}$). The values of $I_{003}/I_{104}$ for M = Zn$^{2+}$ and Al$^{3+}$ are 1.379 and 1.400, respectively. These values are quite large, indicating that the compositions of these samples are nearly stoichiometric.

Figure 8 shows the variations of hexagonal lattice parameters a, c and trigonal distortion c/a with M in LiNi$_{0.57}$M$_{0.43}$O$_2$ (M = Zn$^{2+}$, Al$^{3+}$ and Ti$^{4+}$). The value of c/a increases as M varies from Zn$^{2+}$ to Al$^{3+}$ and then Ti$^{4+}$. This indicates that two-dimensional layered structure develops better as M varies from Zn$^{2+}$ to Al$^{3+}$ and then to Ti$^{4+}$.

The FE-SEM micrographs of the LiNi$_{0.97}$M$_{0.03}$O$_2$ (M = Zn$^{2+}$, Al$^{3+}$ and Ti$^{4+}$) powders calcined at 750°C for 30 h in O$_2$ stream showed that the samples with M = Zn$^{2+}$ and Al$^{3+}$ had small particles and large particles. As M varies from Zn$^{2+}$ to Al$^{3+}$ and then to Ti$^{4+}$, the particles became smaller. LiNi$_{0.97}$Ti$_{0.03}$O$_2$ had particles with similar sizes.

Figure 9 shows the voltage vs. discharge capacity curves of the first cycle for LiNi$_{0.97}$M$_{0.03}$O$_2$ (M = Zn$^{2+}$, Al$^{3+}$ and Ti$^{4+}$). LiNi$_{0.97}$Ti$_{0.03}$O$_2$ has the curve showing the most distinct plateaus. The voltage of the plateau corresponding to the phase transition
Fig. 8. Variations of hexagonal lattice parameters a, c and trigonal distortion c/a with M in LiNi_{0.975}M_{0.025}O_{2} (M = Zn^{2+}, Al^{3+} and Ti^{4+})

Fig. 9. Voltage vs. discharge capacity curves of the first cycle for LiNi_{0.975}M_{0.025}O_{2} (M = Zn^{2+}, Al^{3+} and Ti^{4+})

from M to H, increases as M varies from Zn\(^{2+}\) to Al\(^{3+}\) and then to Ti\(^{4+}\). They are 3.68, 3.69 and 3.75V, respectively.

Figure 10 shows the variations of discharge capacity at 0.1C rate with the number of cycles (n) for LiNi_{0.975}M_{0.025}O_{2} (M = Zn\(^{2+}\), Al\(^{3+}\) and Ti\(^{4+}\)). LiNi\(_{0.975}\) has the largest first discharge capacity (154.8mAh\(^{-1}\)), followed in order by LiNi_{0.975}Al_{0.025}O_{2} (128.5mAh\(^{-1}\)) and LiNi_{0.975}Zn_{0.025}O_{2} (118.6mAh\(^{-1}\)). The cycling performances at n = 10 are 77\%, 98\% and 85\%, respectively, for LiNi_{0.975}M_{0.025}O_{2} (M = Zn\(^{2+}\), Al\(^{3+}\) and Ti\(^{4+}\)). These results agree well to the reported data. It is known that the substitution of a non-transition element Al improves cyclability (Ohzuku et al., 1995; Fey et al., 2003; Song and Lee, 2002) and the substitution of Zn improves cyclability a little but unsatisfactorily (Fey et al., 2003; 2002). LiNi_{0.975}Ti_{0.025}O_{2} has the largest first discharge capacity and a relatively good cycling performance.
Fig. 10: Variations of discharge capacity with the number of cycles for LiNi\textsubscript{0.975}M\textsubscript{0.025}O\textsubscript{2} (M = Zn\textsuperscript{2+}, Al\textsuperscript{3+} and Ti\textsuperscript{4+})

Conclusions

The optimum condition to synthesize a cathode material LiNi\textsubscript{0.975}Ti\textsubscript{0.025}O\textsubscript{2} is milling for 28 h and then preheating at 600°C for 16 h and finally calcining at 750°C for 30 h O\textsubscript{2} stream. LiNi\textsubscript{1-y}Ti\textsubscript{y}O\textsubscript{2} (0 ≤ y ≤ 0.150) synthesized under this optimum condition have the α-NaFeO\textsubscript{2} structure of the rhombohedral system (space group, R\text{3\text{m}}). The values of I\text{003}/I\text{110} for y = 0.000, 0.012 and 0.025 are near 1.2, indicating these samples have the phase with the nearly stoichiometric composition. The samples with y = 0, 0.012 and 0.025 show good splitting of 006 and 102 peaks and of 108 and 110 peaks, indicating the decrease in cation mixing. The particle size decreases as the value of y increases. All the samples except that with y = 0.000 exhibit relatively homogeneous particle size. The voltage vs. discharge capacity curves for y = 0.012 and y = 0.025 exhibit three distinct plateaus corresponding to phase transitions. Among LiNi\textsubscript{1-y}Ti\textsubscript{y}O\textsubscript{2} (0 = y = 0.150), LiNi\textsubscript{0.975}Ti\textsubscript{0.025}O\textsubscript{2} has the largest first discharge capacity 154.8 mAh g\textsuperscript{-1} and the relatively good cycling performance (77% at n = 10).

Electrochemical studies of LiNi\textsubscript{0.975}M\textsubscript{0.025}O\textsubscript{2} (M = Zn\textsuperscript{2+}, Al\textsuperscript{3+} and Ti\textsuperscript{4+}) show that the plateaus in the voltage vs. discharge capacity curve become more distinct and the voltage of the plateau corresponding to the phase transition from M to H\textsubscript{1} increases as M varies from Zn\textsuperscript{2+} to Al\textsuperscript{3+} and then to Ti\textsuperscript{4+}. LiNi\textsubscript{0.975}Ti\textsubscript{0.025}O\textsubscript{2} has the largest first discharge capacity and a relatively good cycling performance compared with LiNi\textsubscript{0.975}M\textsubscript{0.025}O\textsubscript{2} (M = Zn\textsuperscript{2+} and Al\textsuperscript{3+}). This sample has the largest value of \(\eta\) and the smallest particles. LiNi\textsubscript{0.975}Al\textsubscript{0.025}O\textsubscript{2} has the best cycling performance (98% at n = 10).

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