Li₃PO₄ Coated Li[Ni₀.75Co₀.1Mn₀.15]O₂ Cathode for All-Solid-State Batteries Based on Sulfide Electrolyte

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ABSTRACT
Surface coating of cathodes is an essential process for all-solid-state batteries (ASSBs) based on sulfide electrolytes as it efficiently suppresses interfacial reactions between oxide cathodes and sulfide electrolytes. Based on computational calculations, Li₃PO₄ has been suggested as a promising coating material because of its higher stability with sulfides and its optimal ionic conductivity. However, it has hardly been applied to the coating of ASSBs due to the absence of a suitable coating process, including the selection of source material that is compatible with ASSBs. In this study, polyphosphoric acid (PPA) and (NH₄)₂HPO₄ were used as source materials for preparing a Li₃PO₄ coating for ASSBs, and the properties of the coating layer and coated cathodes were compared. The Li₃PO₄ layer fabricated using the (NH₄)₂HPO₄ source was rough and inhomogeneous, which is not suitable for the protection of the cathodes. Moreover, the water-based coating solution with the (NH₄)₂HPO₄ source can deteriorate the electrochemical performance of high-Ni cathodes that are vulnerable to water. In contrast, when an alcohol-based solvent was used, the PPA source enabled the formation of a thin and homogeneous coating layer on the cathode surface. As a consequence, the ASSBs containing the Li₃PO₄-coated cathode prepared by the PPA source exhibited significantly enhanced discharge and rate capabilities compared to ASSBs containing a pristine cathode or Li₃PO₄-coated cathode prepared by the (NH₄)₂HPO source.

Keywords: Surface Modification, All-Solid-State Batteries, Cathode, Coating, Lithium Battery

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1. Introduction
All-solid-state batteries (ASSBs) based on nonflammable inorganic solid electrolytes have gained significant attention [1-5]. The safety issues associated with lithium-ion batteries (LIBs) are expected to be remarkably improved by excluding the use of flammable liquid electrolytes. Moreover, ASSBs can be used over a wider temperature range than LIBs because the stability of the solid electrolytes for a change in temperature is much better than that of the organic liquid electrolytes. This leads to the possibility of removing battery management systems (BMSs) and cooling packages from battery packs. Therefore, it is considered that the energy density per battery pack could be enhanced by using ASSBs compared to that using LIBs, although the weight per cell increases due to the use of heavy solid electrolytes.

Several solid electrolyte candidates have been explored till date for the commercialization of ASSBs. Lithium thiophosphate, a sulfide electrolyte, is recognized as a promising electrolyte owing to its high ionic conductivity and favorable mechanical performance. However, sulfide electrolytes cannot maintain electrochemical stability in the voltage range for charging/discharging and cause undesirable side reactions with oxide cathodes due to their high reactivity [1-7]. Therefore, the interfacial layer attributed to side reactions and sulfide decomposition is formed at the cathode/electrolyte interface, which hinders the efficient movement of electrons and lithium ions during cycling [1-7]. This instability at the cathode/electrolyte interface is a critical issue that limits the commercialization of ASSBs based on sulfide electrolytes [1-7].
To stabilize the cathode/electrolyte interface, surface coating has been applied to the cathodes of ASSBs [8-15]. Stable coating materials can alleviate side reactions by preventing direct contact between the sulfide and oxide cathodes. Several oxides, such as LiNbO$_3$ [8,9], LiTaO$_3$ [10-12], Li$_2$ZrO$_3$ [13,14], and Li$_4$Ti$_5$O$_{12}$ [15] have been used as coating materials because of their good chemical stability with sulfides and suitable ionic conductivity. In addition, several phosphates, such as Li$_3$PO$_4$, have been suggested as efficient coating materials based on numerical computations [2,3,16]. Phosphates also have good chemical stability against sulfides and avoid anion exchange between the cathode and the electrolyte. They also possess suitable ionic conductivities (~10$^{-7}$ S·cm$^{-1}$), relatively high oxidation limits (~4.22 V), and low costs. Nevertheless, phosphate-based materials have rarely been applied as coatings, and even when used, their performance is not comparable to that of conventional oxide coatings [17]. The present study hypothesized that the inconsistency between the computational calculation and actual experimental results is attributed to the surface morphology of the coating layer and cathode degradation during the coating process.

To address this issue, we manufactured Li$_3$PO$_4$ coated cathodes using two types of source materials, namely, polyphosphoric acid (PPA) and (NH$_4$)$_2$HPO$_4$, and compared their physical morphologies and electrochemical performances. (NH$_4$)$_2$HPO$_4$ is generally used as a source material for Li$_3$PO$_4$ coating of cathodes [18-20]. Li$_3$PO$_4$ coated cathodes prepared using (NH$_4$)$_2$HPO$_4$ have shown enhanced electrochemical performance compared to uncoated cathodes in LIBs, presenting a positive coating effect. However, (NH$_4$)$_2$HPO$_4$ requires water as a solvent for preparing the coating solutions because it is not soluble in alcoholic solvents. Water-based solutions are likely to damage the surfaces of high-Ni cathodes, which are vulnerable to water. Moreover, a slightly thick and non-uniform coating layer, which is not suitable for ASSBs, has been observed because of the low wettability of water against the cathode surface [18-20]. In contrast, PPA is highly soluble in alcoholic solvents. Therefore, it is expected that coating solutions based on alcohol and PPA do not damage the performance of high-Ni cathodes, even after prolonged contact. Moreover, alcohol-based solutions are advantageous for forming thin and homogeneous coating layers owing to the superior wettability of alcohols with oxide cathodes compared to water-based solutions [21-23]. In this study, a high-Ni Li[Ni$_{0.75}$Co$_{0.1}$Mn$_{0.15}$O$_2$ cathode was used as the pristine cathode. The characteristics of Li$_3$PO$_4$ coated cathode according to the source materials for coating were observed and compared to those of the pristine cathode in detail through various experimental techniques such as scanning electron microscopy (SEM), transmission electron microscopy (TEM), and X-ray photoelectron spectroscopy (XPS).

2. Experimental

We used a commercial cathode (Li[Ni$_{0.75}$Co$_{0.1}$Mn$_{0.15}$O$_2$) as a pristine cathode. To prepare the Li$_3$PO$_4$ coating solutions, PPA (Aldrich) and (NH$_4$)$_2$HPO$_4$ (Aldrich) were dissolved in reagent alcohol (99.9%, Aldrich) and deionized water, respectively, and CH$_3$CH$_2$OLi (Aldrich) was used as the lithium source. The amount of coating source was adjusted to 0.01, 0.03, and 0.05 wt.% based on the weight of the pristine powder. Subsequently, the pristine powder was mixed with the coating solutions and stirred at 70°C until the solvent evaporated. The remaining powder was completely dried in a vacuum oven at 90°C and heat treated at 400°C for 1 h in an oxygen atmosphere. The obtained powder was observed using SEM (JSM-7610F PLUS) and TEM (JEOL JEM-2100F) to compare the surface morphologies.

The electrochemical performances of the pristine and coated cathodes were measured using all-solid-state cells. We manufactured the composite cathode by mixing the cathode powder (pristine or coated), sulfide solid electrolyte (argyrodite (Li$_3$PS$_4$Cl)), Jeong Kwan Co.) and a conductive material (Super-C) at a weight ratio of 70:30:2. As the separating layer, the solid electrolyte (0.2 g) was compressed under a pressure of 30 MPa and a composite cathode was formed on the side of the separating layer. The anode layer composed of Li-In was pelletized at the opposite site and the total assembly was compressed again (30 MPa). The 2032-coin cells containing the total assemblies were subjected to a galvanostatic cycling in the voltage range of 1.88–3.73 V considering the voltage drop of the Li-In anode. We then performed electrochemical impedance spectroscopy (EIS) measurements using an electrochemical workstation (AMETEK, Versa STAT 3) with an AC voltage of
10 mV amplitude applied over the frequency range of 0.01 Hz to 500 kHz.

The composite electrodes were cycled 30 times and collected from all-solid-state cells in a dry box. Subsequently, they were moved to an XPS (Thermo Scientific K-Alpha plus) instrument using a vacuum transfer vessel to observe the undesirable side reactions between the cathodes and sulfide electrodes during cycling. Prior to XPS analysis, the electrodes were etched to ~100 nm using Ar sputtering to remove contamination from the surface.

3. Results and Discussion

The performance of the coated cathodes depends highly on the coating thickness. For this reason, before comparing the effect of source materials, we conducted a preliminary experiment to determine the required amount of coating. Fig. 1a shows the initial discharge capacity of Li₃PO₄ coated Li[Ni₀.75Co₀.1Mn₀.15]O₂ cathode prepared using PPA as the source material with three different amounts of coating viz. 0.01, 0.03, and 0.05 wt.% of the cathode weight. We fixed the current density to 17 mA·g⁻¹ and the voltage range to 3.73–1.88 V, considering the voltage drop due to the use of a Li-In anode. While the discharge capacity of the pristine cathode was 171.6 mAh·g⁻¹, that of the Li₃PO₄ coated cathode increased slightly to 173.5 (0.01 wt.%), 182.0 (0.03 wt.%), and 174.3 mAh·g⁻¹ (0.05 wt.%), according to the coating amount. The difference in the measured values was insignificant at a low current density (17 mA·g⁻¹). However, with increase in current density, the effect of the Li₃PO₄ coating on the discharge capacity of the cathodes was distinctly observed. Fig. 1b shows the discharge capacities of the pristine and Li₃PO₄ coated cathodes at current densities of 17, 34, 51, and 85 mA·g⁻¹. At high current densities (34, 51, and 85 mA·g⁻¹), the 0.01 and 0.03 wt.% coated cathode shows much higher discharge capacity compared to that of the pristine cathode. However, when the amount of coating is increased to 0.05 wt.%, the capacity at high current densities rapidly decreased as compared to that of the pristine cathode. This could be an effect of 0.05 wt.%
coating, which exceeded the appropriate thickness.

Although Li$_3$PO$_4$ has a suitable ionic conductivity ($\sim 10^{-7}$ S·cm$^{-1}$), which is much lower than that of general solid electrolytes ($\sim 10^{-2}$–$10^{-4}$ S·cm$^{-1}$). Therefore, if the coating layer is too thick, the movement of Li ions during cycling would be hindered. Moreover, considering the extremely low electronic conductivity of Li$_3$PO$_4$, it is necessary to control the thickness of the coating layer so that electrons can escape from the cathode and be transferred to conductive carbon. Based on the electrochemical performance shown in Fig. 1, the optimum amount of Li$_3$PO$_4$ coating was determined to be 0.03 wt.%, and thus, we used this value for the subsequent experiments.

To compare the morphologies of the Li$_3$PO$_4$ coatings based on the effect of the source materials, we observed the surfaces of the coated cathodes using SEM and STEM. For convenience, the 0.03 wt.% Li$_3$PO$_4$ coated samples using (NH$_4$)$_2$HPO$_4$ and PPA sources are denoted as N-sample and P-sample, respectively. In the SEM images shown in Fig. 2, the surface of the pristine sample is clear and smooth (Fig. 2a). In contrast, the surface of the N-sample was covered with rough particles, which were expected to form Li$_3$PO$_4$ (Fig. 2b), indicating that the formed coating layer was close to the powder type rather than the uniform film type. The P-sample also presented a coating layer; however, the surface was relatively smooth, and the quantity of attached particle-like shapes was much smaller than that of the N-sample (Fig. 2c).

The TEM images of the pristine and two coated samples are also compared to observe the uniformity and thickness of the coating layer in more detail, as shown in Fig. 3. Generally, the surfaces of high-Ni cathodes contain Li residues, such as Li$_2$CO$_3$ and LiOH, which are formed during the synthesis and storage processes. To remove these particles and observe the Li$_3$PO$_4$ coating layer more clearly, we washed the cathode powders before TEM analysis. As shown in Fig. 3a, the pristine sample exhibited a smooth surface without a special surface layer. However, the N-sample showed a rough and relatively thick coating layer (Fig. 3b). Notably, the morphol-
ogy of the coating layer was distinctly changed by the PPA source. As demonstrated in Fig. 3c, the coating layer of the P-sample was thin and homogeneous compared to that of the N-sample. This coating shape is expected to be advantageous for homogeneous protection against reactive sulfide electrolytes and smooth movement of lithium ions during cycling.

The electrochemical performances of the pristine, N-, and P-samples were characterized using all-solid-state cells to observe the difference in the coating effect according to the type of source material. Fig. 4a shows a comparison of the discharge capacities of the samples at current densities of 17, 34, 51, and 85 mA·g⁻¹. The capacity and rate capability of the P-sample were superior to those of the pristine sample. However, the performance of the N-sample was much inferior to that of the pristine sample and the P-sample. Fig. 4b-d show the initial charge-discharge profiles of the pristine and Li₃PO₄-coated samples at current densities of 17, 34, and 85 mA·g⁻¹. The values obtained in Fig. 4 are listed in Table 1. The effect of surface coating was more clearly observed in the capacities at higher current densities. The capacity retentions of the pristine sample at 51 and 85 mA·g⁻¹, defined as the percentage of retained capacity at 51 and 85 mA·g⁻¹ compared to that at 17 mA·g⁻¹, were only 45.6 and 24.7%, respectively. However, the capacity reduction of the P-sample at high current densities is much smaller than that of the pristine sample, indicating that the rate capability is improved by the Li₃PO₄ coating. The capacity retentions at 51 and 85 mA·g⁻¹ are increased to 69.6 and 40.3%, respectively. Numerically simulated studies [2,3] have shown that when oxide cathodes are in contact with sulfide electrolytes, anion exchange between S²⁻ (in the electrolyte) and O²⁻ (in the cathode) occurs strongly because the P-O bond energy is much higher than the P-S bond energy in sulfides. This leads to the formation of an interfacial layer composed of PO₄³⁻, which deteriorates the electrochemical performance of the cells. However, the fact that, phosphates contain the same anion (O²⁻) as the oxide cathode and the same cation (P⁵⁺) as the sulfide electrolyte, helps avoid anion exchange. Moreover, the strong orbital hybridization between P and O in phosphates enables higher chemical stability against interfacial reac-

![Image](image-url)
tions. The enhanced electrochemical performance of the P-sample, as depicted in Fig. 4, confirms the simulation results. In contrast, the discharge capacities of the N-sample were just 135.6 (17 mA·g⁻¹), 65.0 (34 mA·g⁻¹), 18.3 mAh·g⁻¹ (85 mA·g⁻¹), which were significantly lower than those of the pristine sample. This result is unusual because (NH₄)₂HPO₄ is generally used for Li₃PO₄ cathode coatings, and the coated cathodes have shown enhanced electrochemical performance in cells using liquid electrolytes [18,24]. First, it is suspected that the water-based solvent for the (NH₄)₂HPO₄ source damages the high-Ni cathodes. Considering the vulnerability of high-Ni cathodes to water, it is possible that contact with a water-based solution deteriorates their electrochemical performance. It could also be that the rough coating layer is not suitable for protection against interfacial reactions related to sulfide electrolytes. A thick and inhomogeneous coating layer can also hinder the movement of Li ions during cycling.

The difference in the coating layer owing to the source material was also clearly identified through impedance analysis. Fig. 5 shows the Nyquist plots of the pristine, N-, and P-samples after one cycle. A distorted semicircle is attributed to several factors, such as grain boundary resistance due to the sulfide electrolyte and interfacial resistances at the cathode/electrolyte interface and at the anode/electrolyte interface [25-27]. Because the grain boundary resistance and interfacial resistance at the anode/electrolyte interface are expected to be almost constant, the difference in the semicircle is mostly dependent on the interfacial resistance at the cathode/electrolyte interface. We clearly observe that the semicircle size of the N-sample is significantly larger than those of other samples, which indicates that the fabrication process of Li₃-

![Figure 5](image.png)

**Fig. 5.** Nyquist plot of the all-solid-state cells containing pristine, N-, and P-samples. The panel on the right is an enlarged part of the region bounded by the red square on the left.

**Table 1.** Electrochemical properties of the pristine, N-, and P-samples at different current densities.

<table>
<thead>
<tr>
<th>Current Density</th>
<th>Pristine</th>
<th>N-sample</th>
<th>P-sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pristine</td>
<td>Discharge capacity (mAh·g⁻¹)</td>
<td>171.6 (η = 76.5)</td>
<td>135.6 (η = 68.4)</td>
</tr>
<tr>
<td>N-sample</td>
<td>Capacity retention (%)</td>
<td>100.0</td>
<td>100.0</td>
</tr>
<tr>
<td>P-sample</td>
<td>Capacity retention (%)</td>
<td>100.0</td>
<td>100.0</td>
</tr>
<tr>
<td>17 mA·g⁻¹</td>
<td>128.7 (η = 89.4)</td>
<td>65.0 (η = 84.9)</td>
<td>150.8 (η = 95.4)</td>
</tr>
<tr>
<td>34 mA·g⁻¹</td>
<td>78.4 (η = 79.0)</td>
<td>41.5 (η = 87.0)</td>
<td>126.6 (η = 94.6)</td>
</tr>
<tr>
<td>51 mA·g⁻¹</td>
<td>42.4 (η = 69.0)</td>
<td>18.3 (η = 76.2)</td>
<td>73.4 (η = 74.3)</td>
</tr>
<tr>
<td>85 mA·g⁻¹</td>
<td>24.7 (η = 69.0)</td>
<td>13.5 (η = 76.2)</td>
<td>40.3 (η = 74.3)</td>
</tr>
</tbody>
</table>
PO₄ coating by the (NH₄)₂HPO₄ source has increased the impedance value of the Li[Ni₀.75Co₀.1Mn₀.15]O₂ cathode. In contrast, as shown on the right side of Fig. 5, the semicircle of the P-sample is smaller than that of the pristine sample, indicating that the Li₃PO₄ coating by the PPA source reduces the impedance value of the cells. The thin and uniformly coated Li₃PO₄ layer can efficiently decrease the interfacial resistance at the cathode/electrolyte interface, this explains the enhanced discharge capacity and rate capability of the P-sample, as observed in Fig. 4.

Fig. 6 shows the cyclic performance of the samples at a current density of 34 mA·g⁻¹. Although the discharge capacity of the N-sample was too low, the pristine and P-samples showed stable discharge capacities of ~130-140 mAh·g⁻¹ and ~150-160 mAh·g⁻¹, respectively, during 30 cycles. The cycled pristine and P-samples were collected for XPS analysis to study the change in the cathode/electrolyte interface caused by the appropriately coated Li₃PO₄ layer. The S 2p XPS spectrum of the pristine sample before testing is shown in Fig. 7a. Two sharp main peaks at ~161.6 and ~162.7 eV marked in sky-blue show S 2p½ and S 2p½ components of the non-bridging sulfur (S⁻) in the electrolyte [11,26]. The small purple and red peaks located on the left side of the main peaks are associated with S-S (~163.5 and ~164.6 eV) and P-[S]ₓ-P type (~163.1 and ~164.2 eV) bonds, respectively, which are due to the decomposition of sulfide electrolytes [28,29]. The peaks shown in green on the right side (~160.3 and ~161.4 eV) represent the transition-metal sulfides (or lithium sulfide) derived from the side reactions at cathode/electrolyte interfaces [1,2]. The transition-metal sulfides (such as CoS, NiS, and MnS) are formed by the diffusion of transition metals into the electrolyte and that of S into the cathode. The right panels of Fig. 7 are the enlarged images for peaks from transition-metal sulfides.

In the XPS profiles of the pristine sample after 30 cycles, the intensity of the main sky-blue peaks is slightly reduced, as shown in Fig. 7b. Instead, the green peaks related to the transition-metal sulfides (or lithium sulfide) are distinctly increased, indicating that the side reactions actively occurred during cycling. The formation of metal sulfides is accelerated in the charged state because the high oxidation states of the transition metals (Co⁴⁺ and Ni⁴⁺) are highly reactive with S²⁻ [2,30]. Interestingly, the green peaks in the XPS spectrum of the P-sample are much smaller than those in the XPS profile of the pristine sample, as is evident from Fig. 7c. This indicates that the side reactions concerning the formation of transition-metal sulfides are significantly reduced by the Li₃PO₄ coating.

The P 2p XPS spectra of the cycled pristine and P-
samples are shown in Fig. 8. The sharp main peaks at ~131.9 and ~132.7 eV marked in sky-blue are attributed to the P 2p\(3/2\) and 2p\(1/2\) components in the electrolyte. The yellow small peaks at ~130.5 and ~131.3 eV are associated with the reduced phosphorus species due to the decomposition of electrolyte [31]. On the other hand, the purple and red peaks can be assigned to transition-metal phosphate (~133.7 and ~134.5 eV) and Li\(_3\)PO\(_4\) (or P\(_2\)S\(_x\)) (~132.8 and ~133.6 eV), respectively. The detection of Li\(_3\)PO\(_4\) indicates the formation of PO\(_4^{3-}\) polyanions at the cathode/electrolyte interface via side reactions, which is energetically favorable, because the P-O bond energy is higher than the P-S bond energy in the electrolyte. The transition-metal phosphate is derived from the inter-diffusion between the transition-metal ions (in the cathode) and P ions (in the electrolyte) with respect to the side reactions. The figures on the right side show an enlarged view of this section.

Comparing the P 2p spectra of the pristine sample before (Fig. 8a) and after 30 cycles (Fig. 8b), it is clear that the red and purple peaks increased during cycling, confirming that the side reactions such as Li\(_3\)PO\(_4\) formation progressed during cycling. However, the intensity of these peaks in the XPS spectra of the P-sample (after 30 cycles) distinctly decreased, indicating the effect of the Li\(_3\)PO\(_4\) layer. Although the side reactions were not completely terminated, it is clear that Li\(_3\)PO\(_4\) is an efficient coating material for mitigating side reactions and enhancing stability at the cathode/sulfide electrolyte interface. The improved electrochemical performance and reduced impedance of the P-sample are attributed to the Li\(_3\)PO\(_4\) coating effect. However, considering the poor characteristics of the N-sample, it is also clear that such an effect could be obtained only when the coating is performed using an appropriate coating source and process.

The overall effect of the Li\(_3\)PO\(_4\) coating on the source material is shown in Fig. 9. The (NH\(_4\))\(_2\)HPO\(_4\) source forms a rough Li\(_3\)PO\(_4\) coating layer on the surface of the Li[Ni\(_{0.75}\)Co\(_{0.1}\)Mn\(_{0.15}\)]O\(_2\) cathode. The need to use a water-based solution that can damage the high-Ni cathode is also a disadvantage as an appropriate coating source. In contrast, the PPA source using an alcohol-based solution formed a thin and uniform Li\(_3\)PO\(_4\) coating layer without deteriorating the high-Ni cathode. As a result, the effect of the Li\(_3\)PO\(_4\) layer with the ability to suppress the side reactions could be sufficiently elicited, and the electrochemical properties of

![Fig. 8. P 2p XPS profiles of the (a) pristine sample before test, (b) pristine sample after 30 cycles, and (c) P-sample after 30 cycles.](image)

![Fig. 9. Schematic illustration of the effect of Li\(_3\)PO\(_4\) coating prepared using the PPA source compared with that prepared using the (NH\(_4\))\(_2\)HPO\(_4\) source.](image)
the all-solid-state cells containing a high-Ni cathode could be improved.

4. Conclusions

In this study, we applied a Li$_3$PO$_4$ surface coating to a Li[$\text{Ni}_{0.75}\text{Co}_{0.1}\text{Mn}_{0.15}$]O$_2$ cathode to stabilize the cathode/sulfide interface. The Li$_3$PO$_4$ coating prepared using the PPA source formed a thin homogeneous layer on the surface of the cathode. In contrast, the sample prepared using the (NH$_4$)$_2$HPO$_4$ source presented a rough and particle-like shape. The discharge capacity and rate capability of the all-solid-state cells based sulfide electrolyte are significantly enhanced by the introduction of Li$_3$PO$_4$ coating prepared using the PPA source. XPS analysis of the cycled pristine and Li$_3$PO$_4$ coated cathode (prepared by PPA source) clearly shows that the Li$_3$PO$_4$ layer with high stability has successfully reduced the interfacial reaction between the oxide cathode and the sulfide electrolyte during cycling. However, the Li$_3$PO$_4$-coated sample prepared using the (NH$_4$)$_2$HPO$_4$ source exhibited inferior electrochemical performance compared with that of the pristine sample. This result confirms that PPA is a suitable source for preparing Li$_3$PO$_4$ coatings for ASSB cathodes.

Acknowledgments

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References