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Short communication

AlF_3-coated $LiCoO_2$ and $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ blend composite cathode for lithium ion batteries

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ABSTRACT

Surface modifications of electrode materials can improve the electrochemical and thermal properties of cathodes for use in lithium batteries. In this study, AlF_3-coated $LiCoO_2$ and AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ cathode materials are blended, as both have the same crystal structure and exhibit similar electrochemical properties. The composite electrodes exhibit high discharge capacities of 180–188 $mAh\,g^{-1}$ in a voltage range of 3.0–4.5 V at room temperature. The capacity retention of the composite electrode is greater than 95% of the initial capacity after 50 cycles. The thermal stability of these composite electrodes is greatly improved because of the superior thermal stability of AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$. The blended AlF_3-coated $LiCoO_2$ and AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ electrode shows two exothermic peaks, one at 227 °C from AlF_3-coated $LiCoO_2$ and another at 277 °C from AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$, accompanied by significantly reduced exothermic heat generation.

1. Introduction

Current portable electronic equipments, such as smart phones and laptop computers, are much more user-friendly due to improved technology and increased personal convenience. Electronic circuits are becoming smaller and thinner; however, display panels have become larger, requiring more electricity than that used in conventional panels. Therefore, the style change of portable electronics has caused a great demand for small batteries with high capacity. $LiCoO_2$ is still the most widely used commercial cathode material for lithium ion batteries because of its easy synthesis, high electronic conductivity, excellent cycling performance, and reasonable rate capability. Increasing the operation voltage to greater than 4.2 V easily increases the discharge capacity of $LiCoO_2$. However, capacity retention of the $LiCoO_2$ above 4.2 V is not always satisfactory because of structural instability [1–3], which results in increased cell impedance [4–6]. To overcome this problem, many studies have been conducted to modify the $LiCoO_2$ particle surface with a metal oxide [7–9] or fluorine compounds [10,11]. The results demonstrated promising increases in the discharge capacity reversibility upon cycling; however, the resulting thermal properties remained unimproved due to the intrinsic structural instability of the parent $LiCoO_2$ at a highly delithiated state, $Li_{1-\delta}CoO_2$ ($\delta \geq 0.6$).

$Li[(Ni_{1/2}Mn_{1/2})_{1-x}Co_x]O_2$ ($x = 0–1/3$) is also an attractive cathode material with high capacity and good thermal stability [12]. $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ is an especially attractive cathode material because of its high capacity, safety, and low cost, although it exhibits a lower rate capability than that of $LiCoO_2$ due to a lower electronic conductivity. The upper cut-off voltage limit is increased with reversible capacity when $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ is coated with AlF_3 [13,14]. As a result, AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ electrodes show improved cycling performance and thermal stability [14].

In this work, AlF_3-coated $LiCoO_2$ and $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ cathode materials were blended to avoid their individual drawbacks. Though modification with AlF_3 on the surface of $LiCoO_2$ exhibited good performance above a high cut-off voltage, thermal stability, which is the most important requirement, was insufficient. Thus, blending of AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ with the AlF_3-coated $LiCoO_2$ material may be a good solution for improving the thermal stability of AlF_3-coated $LiCoO_2$ for use in lithium ion batteries. Furthermore, both materials have the same crystal structure and similar electrochemical capacities at the same cut-off voltage. Here, we report the electrochemistry and thermal properties of the blended AlF_3-coated $LiCoO_2$ and AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$.

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2. Experimental

To prepare AlF_3-coated $LiCoO_2$, ammonium fluoride (Aldrich) and aluminum nitrate nonahydrate (Aldrich) were separately dissolved in distilled water. Commercial $LiCoO_2$ (Nippon Chemical Co.) powders were immersed into the aluminum nitrate nonahydrate aqueous solution, and the ammonium fluoride solution was slowly added to the solution. The amount of AlF_3 in the solution was 0.5 mol% of the $LiCoO_2$ powders. The cathode powder solution was constantly stirred at 80 °C for 5 h, followed by washing with distilled water. The obtained $LiCoO_2$ powders were heated at 400 °C for 5 h in flowing nitrogen atmosphere [10,11]. Spherical $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ was synthesized via co-precipitation [15]. To prepare AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$, ammonium fluoride (Aldrich) and aluminum nitrate nonahydrate (Aldrich) were separately dissolved in distilled water. After the prepared $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ powders were immersed into the aluminum nitrate nonahydrate solution, the ammonium fluoride solution was slowly added to the solution. The amount of AlF_3 in the solution was 0.25 mol% of the $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ powders. The solution containing the active material was constantly stirred at 80 °C for 5 h, accompanied by a slow evaporation of solvent. The obtained $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ powders were heated at 400 °C for 5 h in the flowing nitrogen [14]. The composite cathode materials were prepared via mechanical blending of the AlF_3-coated $LiCoO_2$ and $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$.

The morphologies and distributions of the prepared powders were also observed using scanning electron microscopy (SEM, JSM-6340F, JEOL). The surface of the AlF_3-coated powder was also observed using transmission electron microscopy (TEM, JEOL 2010). Chemical compositions of the final products were analyzed with atomic absorption spectroscopy (AAS, Vario 6, Analyticjena).

The coin cells were composed of the prepared powder as a cathode, lithium foil as an anode, and an electrolyte with 1 M $LiPF_6$ in ethylene carbonate and diethyl carbonate (1:1 in volume, PANAX ETEC Co., Korea). Micro-porous polypropylene separator was used in those cells. The cathodes were fabricated with a mixture of 85 wt% prepared powder, 7.5 wt% carbon black and 7.5 wt% polyvinylidene difluoride (PVDF) binder on an aluminum current collector with the thickness of 20 μm. Then, the prepared electrode was roll-pressed and subsequently dried overnight at 110 °C in a vacuum oven to remove residual solvent. The loading density and thickness of cathode active materials on an aluminum current collector were about 7–8 mg cm^{-2} and 30 μm, respectively. The cell assembly was performed in an argon-filled glove box. Charge–discharge tests were performed with a R2032 coin type cell at a constant current density of 90 mA g^{-1} (0.5*C*-rate) in a voltage range of 3.0–4.5 V at 30 °C.

For the differential scanning calorimetry (DSC) experiment, the blended $LiCoO_2$ and $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ electrode was fully charged to 4.5 V and opened in a dry room. The prepared electrode (5–6 mg) was loaded into a stainless-steel sealed pan with a gold-plated copper seal. The measurements were performed in a differential scanning calorimeter 200 PC (NETZS, Germany) using a temperature scan rate of 1 °C min^{-1}. The weight was constant in all cases, indicating that there were no leaks during the experiments.

3. Results and discussion

Fig. 1 shows SEM images of AlF_3-coated $LiCoO_2$, AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$, and blended AlF_3-coated $LiCoO_2$ and AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ (blending ratio; AlF_3 coated $LiCoO_2$:AlF_3 coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ = 5:5). The coating medium, AlF_3 nano-particles, was observed on the surfaces of active materials, as shown in Fig. 1a and b. Blended AlF_3-coated $LiCoO_2$ and $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ particles are shown in Fig. 1c, indicating homogeneous distribution of the mixed particles. Fig. 2 exhibits TEM images of AlF_3-coated $LiCoO_2$ and $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ particles. Some agglomerations are found in the SEM image (Fig. 1a and b), which ranged from 50 to 100 nm. However, almost all coated particles showed AlF_3 coating layers having thickness of about 10 nm and coating the surface of $LiCoO_2$ and $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$. TEM images confirm that the smoothly formed AlF_3 coating layers are thin and uniform.

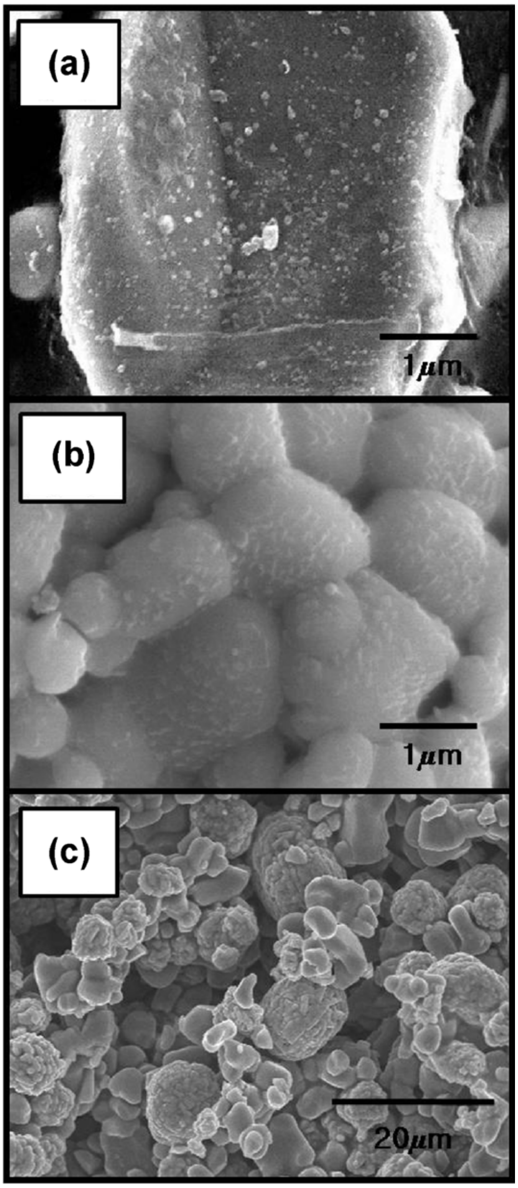

Fig. 1. SEM images of (a) AlF_3-coated $LiCoO_2$, (b) AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ and (c) blended AlF_3-coated $LiCoO_2$ and AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ (blending ratio = 5:5).

Fig. 3 depicts the initial charge and discharge curves of the blended AlF_3-coated $LiCoO_2$ and AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ electrodes with various blending ratios at a current of 36 mA g^{-1} (0.2*C*-rate) in a voltage range of 3–4.5 V. The AlF_3-coated $LiCoO_2$ delivered a discharge capacity of approximately 188 mAh g^{-1}, a slightly greater capacity than that of the AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ electrode that showed approximately 180 mAh g^{-1} at the same cut-off voltage. As a result, it is expected that the discharge capacity for the blended electrodes can range from 180 to 188 mAh g^{-1}, depending on the blend ratio of

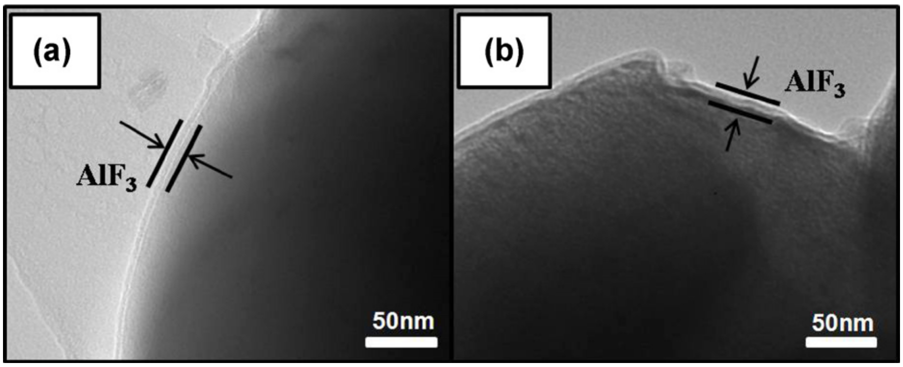

Fig. 2. TEM images of (a) AlF_3-coated $LiCoO_2$ and (b) AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$.

the AlF_3-coated $LiCoO_2$ and AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$. All blended electrodes exhibited the first discharge capacity in a range of 180–188 mAh g^{-1}. The addition of AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ in the AlF_3-coated $LiCoO_2$ resulted in a slightly decreased discharge capacity. Additionally, the AlF_3-coated $LiCoO_2$ electrode exhibited a higher operation voltage across the whole range compared to that of the AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$, ascribed to the intrinsic characteristics of the redox potential for $LiCoO_2$. For example, the initial charge potential was decreased to 3.68 V from 3.9 V by increasing the AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ portion. The resulting potential plateau stemming from the AlF_3-coated $LiCoO_2$ gradually decreased around 3.9 V. In other words, the addition of AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ decreases the total achievable energy density.

As shown in Fig. 3, the voltage profile was different when active materials were blended at various ratios. Charge and discharge behaviors became clear in their derivative forms. Fig. 4 shows the differential capacities ($dQ\,dV^{-1}$) versus the cell voltages of these electrodes. The AlF_3-coated $LiCoO_2$ cell exhibited a sharp oxidation peak at 3.92 V during the charging state. The corresponding reduction peak appeared at 3.9 V. AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ exhibited oxidation and reduction peaks at 3.7–3.76 V and 3.73 V, respectively. Oxidation peaks of the blended cathodes were observed at 3.7–3.76 V and 3.94 V during charging due to the blending of different materials. These values showed that each of the materials could react in electrochemical oxidation and reduction. Reduction peaks for the blended electrodes were observed at 3.72 V and 3.87 V. The incorporation of more AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ produced smaller peaks near 3.94 V during charge and near 3.87 V during discharge, which coincides with the data illustrated in Fig. 3. The blend composite cathode materials did not suppress the structural change induced by the phase transition; however, it did contribute to the electrochemical reaction.

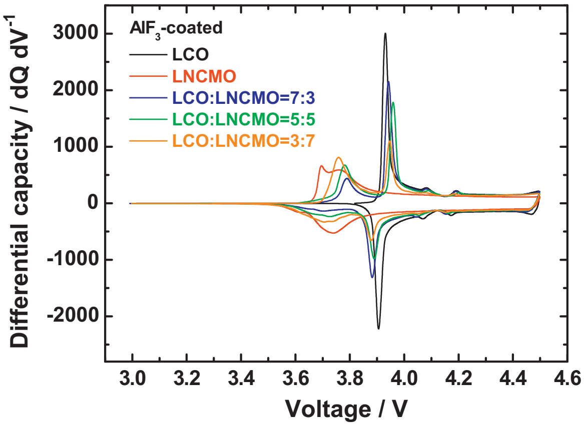

Fig. 4. Differential capacity versus voltage curves of blended AlF_3-coated $LiCoO_2$ and AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ with varied blending ratios.

Cycling performances of the blended electrodes are shown in Fig. 5. The cycling tests were performed by applying a constant current of 90 mA g^{-1} (0.5C-rate) at 30 °C. The capacity retention of AlF_3-coated $LiCoO_2$ after 50 cycles was 97.5%. However, the addition of the AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ resulted in gradual capacity fading and the retention decreased to 94–97%. A slight

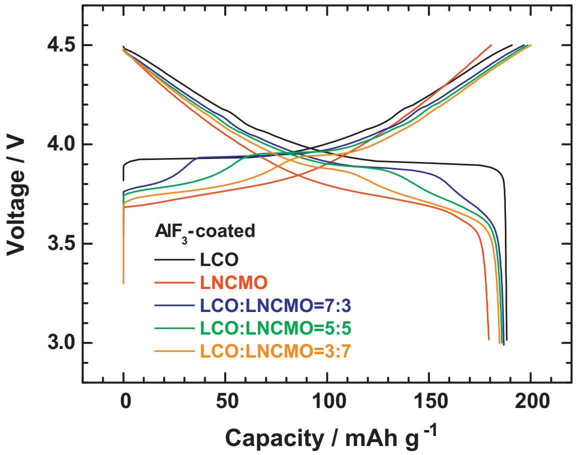

Fig. 3. Initial charge and discharge curves of blended AlF_3-coated $LiCoO_2$ and AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ with varied blending ratios.

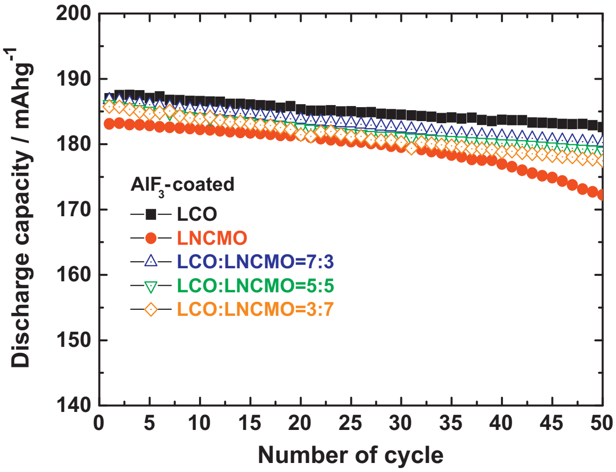

Fig. 5. Cycling performances of blended AlF_3-coated $LiCoO_2$ and AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ with varied blending ratios.

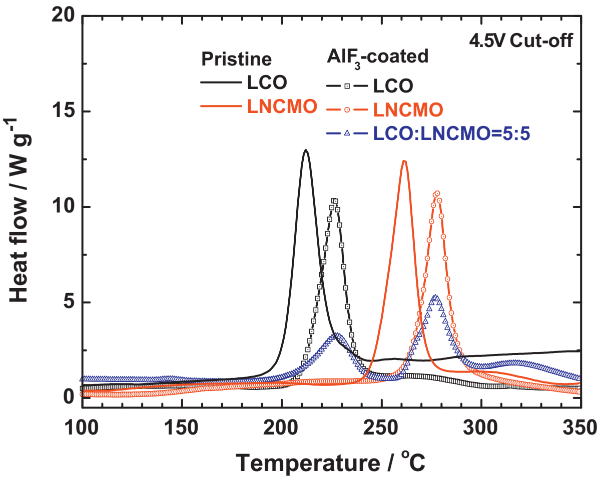

Fig. 6. Comparison of DSC traces of (a) $LiCoO_2$, (b) $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$, (c) AlF_3-coated $LiCoO_2$, (d) AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ and (e) blended AlF_3-coated $LiCoO_2$ and $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ (5:5) charged to 4.5 V.

decrease in the capacity retention was observed for the AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$, approximately 93% of its initial discharge capacity. Thus, it is likely that the addition of the AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ as well as that of AlF_3-coated $LiCoO_2$ did not retain the capacity; however, the capacity retention for the blended electrodes remained high, more than 94% during cycling. It is also notable that several blended systems have employed layered Ni-rich compositions like $Li[Ni_{0.8}Co_{0.15}Al_{0.05}]O_2$ and $LiMn_2O_4$ to improve the thermal properties, although the composite electrode sacrifices at least 25% of the initial electrochemical capacity of $Li[Ni_{0.8}Co_{0.15}Al_{0.05}]O_2$ [16,17]. In the present study, we showed that a blended electrode (for example, AlF_3-coated $LiCoO_2$:AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2 = 1:1$) delivered an initial discharge capacity of approximately 186 mAh g^{-1}, and the capacity was retained around 96% during cycling. The blending was directed to preserve the higher capacity of $LiCoO_2$ or $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$, unlike Ni-rich and spinel blending. Therefore, the blending of AlF_3-coated $LiCoO_2$ and AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ may be promising for their capacity and retention.

The thermal stability of cathode materials, especially at the delithiated state, is most related with battery safety. Fig. 6 compares DSC profiles of the electrochemically delithiated $Li_{1-\delta}CoO_2$, $Li_{1-\delta}[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$, AlF_3-coated $Li_{1-\delta}CoO_2$, AlF_3-coated $Li_{1-\delta}[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ and blended AlF_3-coated $Li_{1-\delta}CoO_2$ and $Li_{1-\delta}[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ electrodes to 4.5 V versus Li. First of all, for the pristine $LiCoO_2$ electrode, the exothermic peak temperature was approximately 212.0 °C, which generated 2785 J g^{-1} of exothermic heat. This result showed that the pristine $LiCoO_2$ at high voltage had no good thermal stability. Therefore the $LiCoO_2$ was coated with AlF_3, and the AlF_3-coated $LiCoO_2$ electrode had improved thermal stability, shifting the exothermic temperature approximately to 226.5 °C of the exothermic peak, which produced 2102 J g^{-1} of exothermic heat. This result implies that, although the nanolayer AlF_3 was applied to the surface of $LiCoO_2$, the thermal stability may not be sufficient to use high voltage cut-off with reliable safety [14]. Specifically, the main exothermic temperature was too low, compared to the pristine $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$. For the pristine $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ electrode, the exothermic peak temperature was about 261.5 °C which generated 1998 J g^{-1} of exothermic heat. Meanwhile, the AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ electrode showed an enhanced thermal behavior; the exothermal peak shifted to a much higher temperature of approximately 277 °C, and the resulting exothermic heat was mostly reduced relative to that of the AlF_3-coated $LiCoO_2$, 1738 J g^{-1} [18]. The most possible reason is due to the presence of the electrochemically inactive tetravalent Mn in the $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ compound. Furthermore, we previously reported that the insulating thin AlF_3 coating layer covers the active materials, and thereby possibly suppresses the oxygen release from the active particle [19]. The blended electrode exhibited two smaller exothermic peaks at 227 °C and 277 °C, generating 1665 J g^{-1} of exothermic heat. It is natural to think that the reduced amount of AlF_3-coated $LiCoO_2$ in the blended electrode simultaneously lowers the exothermic heat generation at 227 °C, though the resulting energy density is reduced as the content of AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ increases. Sacrificing the energy density somehow but improving the safety term is necessary to utilize the high capacity materials with reliable safety. These results clearly show that the blended electrode demonstrates further improved thermal stability characteristics compared to those of AlF_3-coated $LiCoO_2$ and $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ materials.

4. Conclusions

In this study, AlF_3-coated $LiCoO_2$ with high capacity, excellent capacity retention and improved thermal property was successfully achieved through blending with AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$. Since both materials have the same crystal structure and exhibit similar electrochemical performances, the blended electrodes delivered higher discharge capacities than did the AlF_3-coated $LiCoO_2$. Blending of the thermally stable AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2$ successfully reduced the exothermic heat generation of AlF_3-coated $LiCoO_2$. Therefore, AlF_3-coated $LiCoO_2$:AlF_3-coated $Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O_2 = 5:5$ provides high capacity with good retention, improves thermal property and addresses the demerits of AlF_3-coated $LiCoO_2$.

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