

Short communication

Improvement of structural and electrochemical properties of AlF₃-coated Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ cathode materials on high voltage region

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Abstract

The particle surface of Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ was modified by AlF₃ as a new coating material to improve the electrochemical properties in the high cutoff voltage of 4.5 V. The AlF₃-coated Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ showed no difference in the bulk structure compared with the pristine one and the uniform AlF₃ coating layers whose thickness is of about 10 nm covered Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ particles, as confirmed by a transmission electron microscopy. The AlF₃ coating on Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ particles improved the overall electrochemical properties such as the cyclability, rate capability and thermal stability compared with those of the pristine Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂. Such enhancements were attributed to the presence of the stable AlF₃ layer which acts as the interfacial stabilizer on the surface of Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂.
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1. Introduction

Li-ion batteries with high energy density and power capability has become an important power sources for portable electronic devices, such as cellular phones and laptop computers and, more recently, hybrid electric vehicles (HEV) [1,2]. Most of commercialized Li-ion batteries use LiCoO₂ as cathode materials. However, the smaller capacity and high cost of LiCoO₂ has led Li-ion batteries to lower energy density and high cost. Thus, extensive research has been carried out over past 10 years to find alternative cathode materials that offer low cost, longer life and higher capacity. Recently, Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂, one of Li[Ni_xCo_{1-2x}Mn_x]O₂ system, has known to be one of the most promising alternative materials to LiCoO₂, since their electrochemical and safety characteristics are comparable or superior to LiCoO₂ [3–5].

In order to increase the reversible capacity of Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂, the upper cutoff voltage should be increased more than 4.5 V. As expected, the increased upper cutoff voltage resulted in an increase in the specific discharge capacity. However, the enhanced discharge capacity was accompanied by unstable cycling performance and capacity fading was proportional to upper cutoff voltage. One way to improve the electrochemical performance at high voltage is to substitute fluorine for oxygen to stabilize the layered host structure, which is very effective for improving cycling performance of the cathode materials at high voltage [4–6].

As an alternative, thin layer coatings of cathode materials with metal oxides (Al₂O₃, ZrO₂, ZnO, MgO, etc.) and metal phosphate (AlPO₄) are substantially effective approaches for enhancing the electrochemical performances [7–12]. Although the reason for the improvement effect is not completely understood yet, the coating technology is being used for commercialized batteries. Cho et al. [8] reported that the enhanced cycling performance at high voltage was due to the fact that coating cathode materials by ZrO₂ layer could effectively suppress the lattice distortion and thereby phase transitions. Recently,

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Miyashiro et al. [9] reported that the improved electrochemical performance of the LiCoO_2 in the higher voltage region (>4.2 V) is ascribed to the stabilization of the interface between the cathode and electrolyte. Our group [9,13] also reported that the excellent cyclability of the ZnO-coated $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ and LiMn_2O_4 electrodes is ascribed to significantly reduced Mn dissolution, in which ZnO is worked as HF getter in the electrolyte. Recently, we also reported that the AlF_3 was very promising coating material for improvement of electrochemical performance of cathode materials [14,15]. We proposed that the AlF_3 coating layer would act as an interfacial stabilizer, as confirmed by an electrochemical impedance spectroscopy. In this work, we study the effects of AlF_3 coating on the structural and thermal, electrochemical properties of $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ at a high cutoff voltage.

2. Experimental

$\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ powders were synthesized by the coprecipitation method. Details of the preparation method are described in our previous paper [5]. The stoichiometric amounts of $\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, $\text{CoSO}_4 \cdot 7\text{H}_2\text{O}$ and $\text{MnSO}_4 \cdot \text{H}_2\text{O}$ were used as the starting materials. Such obtained $[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}](\text{OH})_2$ compound was thoroughly mixed with appropriate $\text{LiOH} \cdot \text{H}_2\text{O}$ and heated at 950°C for 15 h in air.

To prepare AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$, ammonium fluoride (Aldrich) and aluminum nitrate nonahydrate (Aldrich) was separately dissolved in distilled water. After the prepared $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ 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 $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ 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 $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ powders were heated at 400°C for 5 h in the nitrogen flowing.

Powder X-ray diffraction (XRD, Rint-2000, Rigaku, Japan) measurements using $\text{Cu K}\alpha$ radiation were employed to identify the crystalline phase of the synthesized materials. From the XRD patterns, lattice parameters were calculated by a least-squares method. The surface of the AlF_3 -coated powder was also observed using transmission electron microscopy (TEM, JEOL 2010).

Electrochemical properties of the prepared powder were evaluated with coin type cell (2032). Cells were constructed with the cathode and the lithium metal anode separated by the porous polypropylene (PP) film. The used electrolyte was a 1:1 mixture of ethylene carbonate (EC) and diethyl carbonate (DEC) containing 1 M LiPF_6 by volume (Cheil Industries Inc., Korea). The cathodes were fabricated with mixture of 84 wt% of the prepared powder, 8 wt% of carbon black and 8 wt% of polyvinylidene difluoride (PVDF) binder on aluminum current collector. The cell assembly was carried out in argon-filled glove box. The cells were charged and discharged in the voltage range of 3.0–4.5 V at a constant current density of 90 mA g^{-1} . The long cycle-life tests were performed in a laminated-type full cell wrapped with an Al pouch. Mesocarbon microbeads (MCMC, Osaka gas) were

used as the anode. The fabrication of the cell was done in a dry room. The cells were charged and discharged between 3.0 and 4.4 V by applying a constant current of 1C-rate (180 mA g^{-1}).

The area specific impedance (ASI) was measured at a 10C pulse current rate using the HPPC procedure in accordance with the Partnership for a New Generation of Vehicles (PNGV) Battery Test Manual using a laminated-type full cell of capacity of 50 mAh wrapped with an Al pouch [16]. HPPC test consists of a series of pulse profile that contains pulse discharge (10C/10 s), rest step (32 s) and regenerative pulse charge (8C/10 s) followed by 1C-discharge for 10% of calculated capacity obtained from the formation cycles. Each series of steps are followed by a 1 h rest step to achieve cell equilibrium before subsequent pulse profile. The ASI values were calculated by $\Delta V/I$ (ΔV was difference of voltage between the start and the end at each discharge and regenerative pulse step; “ I ” was pulse current value). Differential scanning calorimetry (DSC) experiment was carried out for cathode material by fully charging the cells to 4.5 V versus Li. Measurements were carried out with differential scanning calorimeter (NETZSCH-TA4, Germany) at a temperature scan rate of 5°C min^{-1} in the temperature range of 100– 350°C .

3. Results and discussions

Fig. 1 shows XRD patterns of the pristine and AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ powders. All the powders were confirmed to be well-defined hexagonal $\alpha\text{-NaFeO}_2$ structure with space group of $R\bar{3}m$. No impurity peaks corresponding to Al-related compounds were observed from the XRD pattern. The lattice constants of the pristine and AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ powders were $a = 2.8615(2) \text{ \AA}$, $c = 14.2188(1) \text{ \AA}$ and $a = 2.8621(3) \text{ \AA}$, $c = 14.220(1) \text{ \AA}$, respectively. The two powders showed very similar lattice constants, suggesting that the AlF_3 did not affect the host structure.

Fig. 2 exhibits TEM images of the AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$. As expected, it is clearly observed that a nanoscale- AlF_3 layer having its thickness of 10 nm was coated homogeneously on the surface of $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$. The uniform contrast of the AlF_3 film indicates that the AlF_3 coating layer is thin and it would have amorphous phase and/or lower

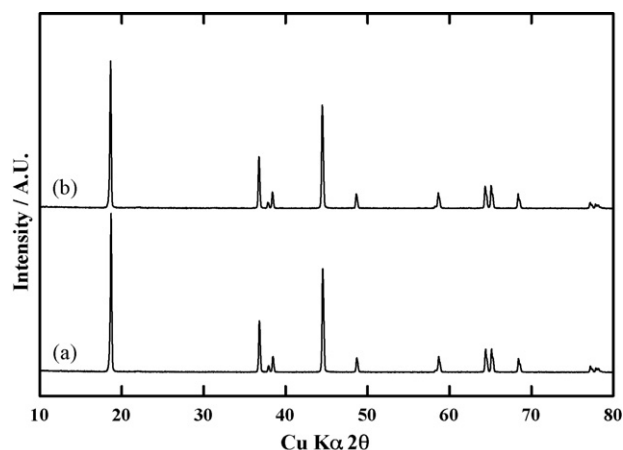


Fig. 1. XRD patterns of (a) pristine and (b) AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$.

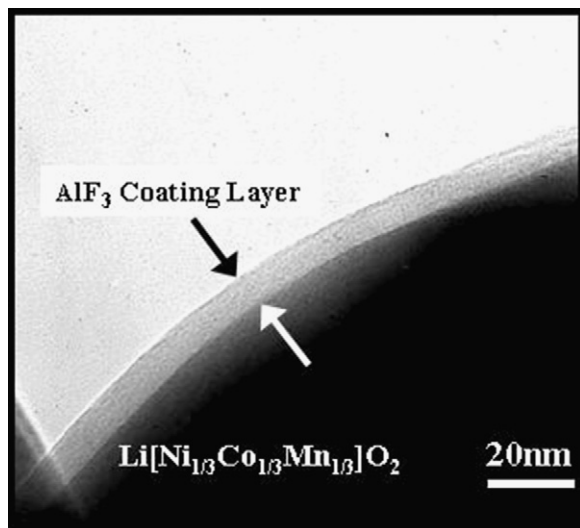


Fig. 2. The bright-field TEM images of AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$.

crystallinity owing to the heat treatment at lower temperature of about 400°C [15].

Fig. 3 shows the voltage versus capacity profiles of Li/pristine and AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cells. The cells were firstly activated by applying a current density of $0.2C$ rate (36 mA g^{-1}) for two cycles and then cycled at $0.5C$ rate (90 mA g^{-1}) between 3.0 and 4.5 V versus Li/Li^+ . Both cells had

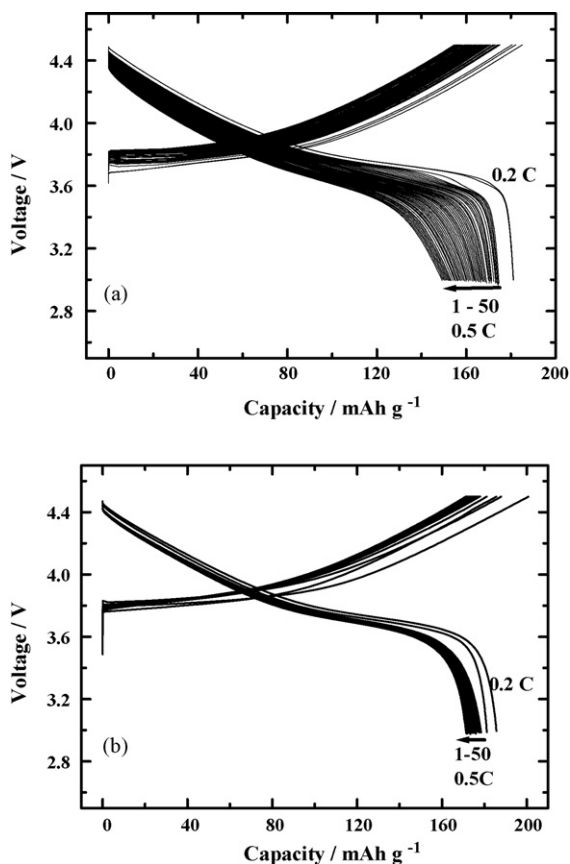


Fig. 3. Continuous charge–discharge curves of (a) Li/pristine and (b) Li/ AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$.

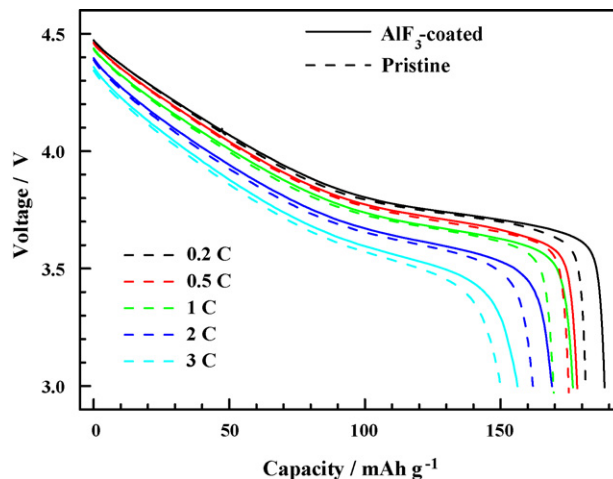


Fig. 4. Rate capability of Li/pristine and AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cells at various C -rate.

stable and smooth charge/discharge curves even on the higher cut-off voltage of 4.5 V. Observing the discharge curves at the end of discharge, one can clearly see the smooth voltage variation for the AlF_3 -coated samples, but an abrupt voltage dropping was seen for pristine $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$. The initial discharge capacity of the AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ is 178 mAh g^{-1} , as compared to that of pristine one having 175 mAh g^{-1} . However, the cycling behavior of the AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ remarkably enhanced showing capacity retention of 96% after 50 cycles, while the pristine electrode showed a gradual decrease in capacity, leading to a capacity retention of only 85%.

Fig. 4 shows the discharge voltage versus capacity profiles of Li/pristine and AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cells at various C -rate between 3.0 and 4.5 V. The cells were charged galvanostatically with a current density of 36 mA g^{-1} ($0.2C$ -rate) before each discharge test and then discharged at current densities from 36 ($0.2C$ -rate) to 540 mA g^{-1} ($3C$ -rate). Similar to cycling performance, the AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ showed an enhanced discharge capacity compared to pristine one at various currents.

In order to investigate the long-term cycling performance, a carbon electrode using MCMB was employed as the anode. Laminated-type lithium-ion cell using an Al pouch with a capacity of 50 mAh were assembled. The fabricated batteries using pristine and AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cathode were charged and discharged for 800 cycles at $1C$ -rate between 3.0 and 4.4 V and the result was shown in Fig. 5. The C /pristine $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cell showed a gradual capacity fading with cycling and it has a capacity retention of only 59% after 820 cycles. It showed a slow decrease in the discharge voltage as much as 0.14 V at the 820th cycle shown in Fig. 5a. On the other hand, the AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cell showed a better cycling performance and has capacity retention of 81% after 820 cycles in Fig. 5b and c, even at higher cutoff voltage to 4.4 V. The voltage decay during cycling is quite lower compared with that of the C /pristine $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cell in Fig. 5a.

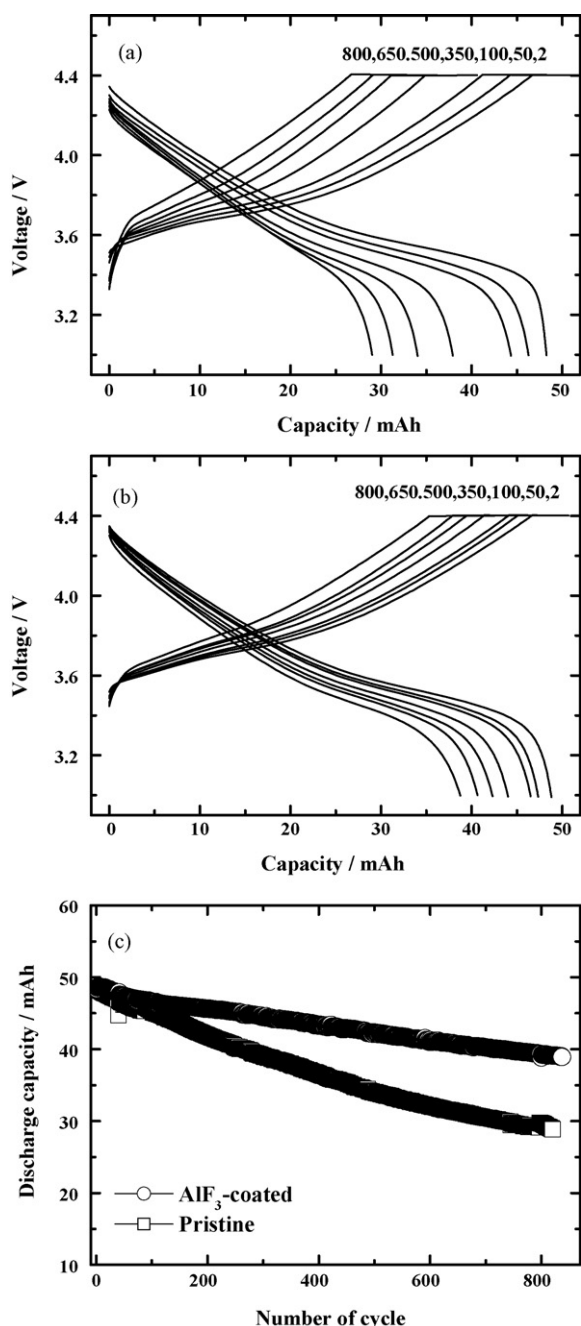


Fig. 5. (a) Continuous charge–discharge curves of C/pristine $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cell, (b) continuous charge–discharge curves of C/ AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cell and (c) corresponding discharge capacity vs. cycling number.

Recently, our group [17] reported that the thin Al_2O_3 coating layer not only protected the cathode surface against HF attack in electrolyte but also worked as an HF scavenger, as confirmed by ToF-SIMS. These effects made it possible to maintain the morphology of original particle shape of active material during extensive cycling, which greatly improved battery performance. However, the Al_2O_3 coating layer was converted to Al-O-F and Al-F fragments after extensive cycling, suggesting that the formation of Al-O-F could be in an intermediate stage to be transformed to Al-F bond. It is speculated that the amphoteric

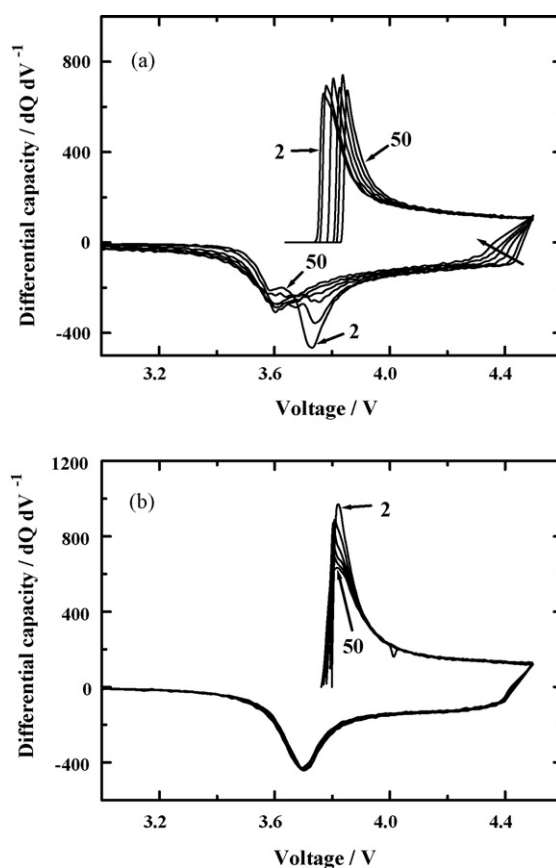


Fig. 6. Differential capacity vs. voltage (dQ/dV^{-1}) curves of (a) Li/pristine and (b) Li/ AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cells at the voltage range of 3.0–4.5 V during 50 cycles.

Al_2O_3 coating layer would not be endurable from HF attack in the electrolyte after extended cycling. Therefore, some of the Al_2O_3 coating layer could be break-up and the remaining HF in the electrolyte would easily attack the active particles, resulting in capacity loss with cycling. However, AlF_3 is very resistant to HF solution and it, thereby, is reasonable to consider that the AlF_3 -coated cathode materials significantly improve electrochemical performances for long-term cycling.

Fig. 6 shows differential capacity (dQ/dV^{-1}) versus voltage profiles of Li/pristine and AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cells at the 2nd, 10th, 20th, 30th, 40th and 50th cycle. The cells were cycled between 3.0 and 4.5 V with a current density of 90 mA g^{-1} . At the first cycle, the Li/pristine $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cell redox peaks at around 3.77 V on charging and 3.73 V on discharging. As the electrochemical cycling proceeded, however, the redox peaks changed to a more polarized state and then shifted to 3.85 and 3.64 V at the 50th cycle. In addition, lowering of the initial discharge voltage at around 4.4 V (indicated with arrow) suggests the increase in the internal resistance during cycling, which is well accordance with discharge voltage drop at the initial state shown in Figs. 3a and 5a. Surprisingly, the redox peaks for the AlF_3 -coated $\text{Li}[\text{Ni}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}]\text{O}_2$ cells with cycling remained almost unchanged even after extensive cycling though the oxidation peak height decreased.

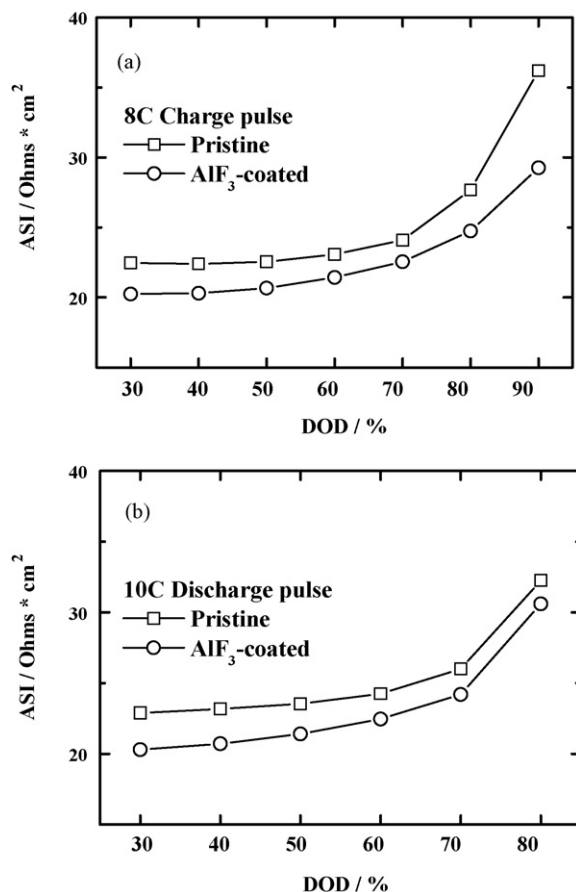


Fig. 7. Pulse power ASI as a function of DOD for a C/pristine and AlF₃-coated Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ cells. (a) Charge pulse and (b) discharge pulse.

In Fig. 7 is shown the area specific impedance (ASI) at the 10C-rate (1800 mA g⁻¹) discharge as a function of depth of discharge (DOD). The test was carried out on C/pristine and AlF₃-coated Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ cells using a 10C-rate pulse. As can be seen in Fig. 7, the AlF₃-coated Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ electrode exhibits much lower ASI values than those for the pristine Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂. Again, the improved ASI characteristics could be ascribed to the reduced interfacial impedance between cathode and electrolyte, which confirms that the coated AlF₃ layer on the surface of the Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ works as the interfacial stabilizer.

The thermal stability of cathode materials, especially the delithiated state, is of great interest related with battery safety. Fig. 8 represents the DSC data of the Li/the pristine and AlF₃-coated Li_{1-x}[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ cells charged to 4.5 V versus Li. For the pristine Li_{1-x}[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ electrode, the exothermic peak temperature was about 261.5 °C. Meanwhile, the AlF₃-coated Li_{1-x}[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ electrode showed an enhanced thermal behavior; the exothermal peak shifted to slightly higher temperature and the generated heat for the AlF₃-coated Li_{1-x}[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ electrode was somewhat reduced in Fig. 8. These results clearly show that the AlF₃-coated Li_{1-x}[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ cathode has better thermal stability characteristics than pristine one. The thin AlF₃ coating layer prevents the direct contact of the active cathode

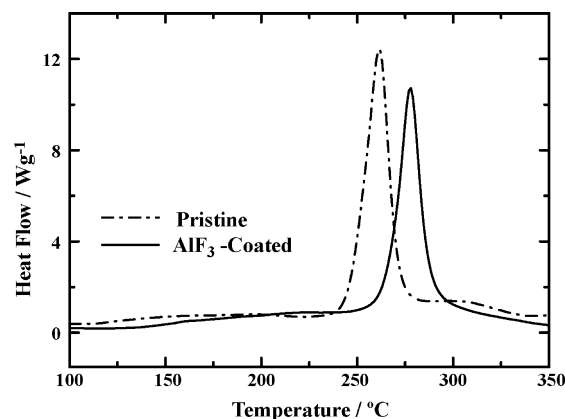


Fig. 8. DSC profiles of pristine Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ and AlF₃-coated Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ cells charged at state to 4.5 V.

materials with the liquid electrolyte, which could greatly reduce the decomposition of active material from HF attack, and thereby possibly suppress the oxygen release from the cathode particle.

4. Conclusions

The effects of AlF₃ coating on Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ at high cutoff voltage of 4.5 V cycling were investigated. The AlF₃-coated Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ showed enhanced cycling performance and rate capability compared to the pristine Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂. The AlF₃-coated Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ cell has capacity retention of 81% after 800 cycles in the full cell using carbon as the anode even at higher voltage limit to 4.4 V, while the pristine Li[Ni_{1/3}Co_{1/3}Mn_{1/3}]O₂ exhibited capacity retention of only 60%. From the differential capacity curves, the AlF₃ coating on cathode particle stabilized the interface between the cathode and electrolyte during electrochemical cycling. The improved electrochemical performance could be ascribed to the reduction of the structural change of active material from HF attack by a thin AlF₃ coating layer preventing the highly delithiated cathode from being directly contacted with liquid electrolyte.

Acknowledgement

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