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# Effects of ZnO coating on electrochemical performance and thermal stability of LiCoO<sub>2</sub> as cathode material for lithium-ion batteries

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#### ABSTRACT

ZnO-coated LiCoO<sub>2</sub> particles are prepared by plasma-enhanced chemical vapour deposition (PE-CVD) in a coating range from 0.08 to 0.49 wt.%, and examined using field emission-scanning electron microscopy (FE-SEM), X-ray diffraction (XRD), and atomic absorption spectroscopy (AAS), with particular focus on surface characteristics. From charge-discharge cycling tests in the range of 3.0–4.5 V, the ZnO coating has little effect on the discharge capacity in the first few cycles, but the coating effectively improves the capacity retention after prolonged cycling. In the experimental range studied, the optimum amount of ZnO coating which maximizes the capacity retention is found to be 0.21 wt.%. An excessive amount of ZnO coating causes a decrease in both cyclic performance and thermal stability. The possible reasons for enhanced cycleability and thermal stability afforded by the ZnO coating are discussed from the viewpoint of the surface morphology of the bare and coated LiCoO<sub>2</sub> particles and their impedance spectra.

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### 1. Introduction

LiCoO<sub>2</sub> has been commercially used as the dominant cathode material in most lithium-ion batteries on account of its favourable electrochemical attributes, e.g., good capacity retention, favourable rate capability, and high structural reversibility below 4.2 V<sub>Li/Li+</sub>. Layered  $Li_x CoO_2$  cathode materials are typically charged up to 4.2 V (0.5 < x < 1) because further increases in the charge cut-off voltage result in structural damage and severe capacity decay [1,2]. It has been reported that when the surface of LiCoO<sub>2</sub> is coated with oxides such as Al<sub>2</sub>O<sub>3</sub> [3–6], MgO [6], ZrO [7,8], SiO<sub>2</sub> [8], CeO<sub>2</sub> [9], ZnO [10,11], and LiMn<sub>2</sub>O<sub>4</sub> [12], the coatings effectively prevent direct contact with the electrolyte solution, suppress phase transitions, improve the structural stability, and decrease the disorder of the cations in the crystal sites. As a result, a marked improvement in electrochemical performance is achieved in terms of reversible capacity, coulombic efficiency on the first cycle, cyclic behaviour, and rate capability. Although considerable effort has been made to explain the precise mechanism underlying the capacity fading observed at high charge potentials and the effect of a surface coating, further investigations are still required.

This study reports on the enhancement of both cyclic performance and thermal stability of LiCoO2 cathodes that are surface-modified by a ZnO coating. In an earlier work, Sun et al. [13] indicated the positive effects of ZnO coating on the capacity retention of spinel-phase cathodes. They suggested that a small amount of ZnO coating can play an important role in improving the cycling behaviour of the Mn-based cathode materials by significantly reducing the HF content or removing HF from the electrolyte. In the case of LiCoO<sub>2</sub> materials, Fang et al. [11] discussed the reasons for the improved cycleability of LiCoO<sub>2</sub> surface-modified by a ZnO coating in the context of its microstructural evolution and the variation in impedance spectra before and after cycling. They suggested that the surface compositional and microstructural differences between bare and coated LiCoO<sub>2</sub> materials can strongly affect their electrochemical performance. They observed a non-uniform distribution of ZnO nanoparticles on some specific planes, rather than a uniform dispersion on the LiCoO<sub>2</sub> surfaces. Nevertheless, a part of their explanation, particularly about the impedance spectra of the bare and ZnO-coated LiCoO<sub>2</sub> electrodes obtained before/after cycling, seems somewhat controversial. From a comparison of the size of the semicircles in the Nyquist plot of both cycled electrodes (Fig. 7 in [11]), it was recognized that the diameters of the highfrequency and low-frequency semicircles of their both bare and ZnO-coated LiCoO<sub>2</sub> electrodes is reduced after 30 cycles. In particular, a remarkable decrease in the low-frequency semicircle was observed with the ZnO-coated electrode. By contrast, most of the

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Fig. 1. Schematic diagram of rf-plasma coating system.

studies related to the impedance analysis for LiCoO<sub>2</sub> coated with metal oxides [14–16] have generally reported that either high or low frequency or both resistances of uncoated and coated electrodes is increased with cycling, and a surface-coated electrode undergoes relatively small increases in cell impedance. This is opposite to the findings of Fang et al. [11]. A clearer explanation on the reduced resistances, especially the charge-transfer resistance of both bare and ZnO-coated electrodes after cycling, should have been given in their work.

The objective of this study is to explore the role of ZnO coating in the capacity retention of LiCoO<sub>2</sub> during high-voltage cycling and the thermal stability of the charged LiCoO<sub>2</sub> electrode. The investigation focuses on variation of the morphology with coating amount and its effects on electrochemical performance. The effects of the ZnO coating are also discussed in relation to the impedance spectra of bare and coated LiCoO<sub>2</sub> on cycling. In addition to the electrochemical aspects, the thermal decomposition behaviour of the charged LiCoO<sub>2</sub> is examined as a function of the coating amount using differential scanning calorimetry (DSC) and thermogravimetric analysis (TGA). The optimized amount of ZnO coating on LiCoO<sub>2</sub> required to improve the capacity retention and achieve higher thermal stability is also investigated.

## 2. Experimental

Commercial LiCoO<sub>2</sub> powder with an average particle size of 8.2  $\mu$ m (Nippon Chemical Industrial Co.) was used as the raw material for the ZnO coating. ZnO-coated LiCoO<sub>2</sub> was prepared by plasma-enhanced chemical vapour deposition (PE-CVD) at room temperature. Diethyl zinc (C<sub>2</sub>H<sub>5</sub>)<sub>2</sub>Zn) was used as a zinc precursor. The LiCoO<sub>2</sub> particles were mixed homogeneously using a rake-shaped mixer in a reaction chamber during deposition of ZnO. Fig. 1 shows a schematic diagram of the PE-CVD apparatus. The deposition of the ZnO on the LiCoO<sub>2</sub> surface was carried out at a rf-plasma power of 200 W. Diethyl zinc and oxygen gas were fed into the CVD reactor at flow rates of 1 and 3 sccm, respectively. The base pressure was  $2.0 \times 10^{-5}$  Torr and the working pressure was maintained at  $8.0 \times 10^{-2}$  Torr. Details of the coating conditions are listed

in Table 1. The ZnO coated-LiCoO<sub>2</sub> was then characterized by X-ray diffraction (XRD), field emission-scanning electron microscopy (FE-SEM), atomic absorption spectroscopy (AAS), and BET surface area analysis. Powder XRD (Rigaku, D/MAX-2500) using CuKα radiation was used to identify the crystalline phase of both bare and coated LiCoO<sub>2</sub>. The surface morphology of the LiCoO<sub>2</sub> powders before and after coating was observed using FE-SEM (Hitachi Co., S-4200). The amount of ZnO deposited on the LiCoO<sub>2</sub> powders as a function of the coating time was analyzed by AAS (Thermo Electron Co., Solaar M Series). For the AAS analysis, the bare and ZnO-coated LiCoO<sub>2</sub> powders were immersed in 1 M LiPF<sub>6</sub> in ethylene carbonate (EC)-dimethyl carbonate (DMC)-ethyl-methyl carbonate (EMC) (1:1:1 by volume %) electrolyte for one week. Then, the amount of Zn<sup>2+</sup> dissolved in the electrolyte was determined quantitatively. The specific surface area of the bare and ZnO-coated LiCoO<sub>2</sub> was calculated with a BET surface area analyzer (Micromeritics, ASAP-2010) that used N<sub>2</sub> gas adsorption.

For an investigation of the electrochemical characteristics, half-cells were fabricated in a dry room with a maximum moisture content of less than 0.5%. The cathode consisted of 87 wt.% active materials, 8 wt.% acetylene black (AB) conductor, and 5 wt.% polyvinylidene fluoride (PVDF) binder. The mixed slurry was uniformly spread on a thin aluminum foil with a thickness of 15  $\mu$ m, followed by drying at 80°C for 1 h. The electrodes were cut into 2 × 2 cm pieces and then roll-pressed to increase the number of

Table 1	
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Coating conditions of ZnO on LiCoO<sub>2</sub> powder by PE-CVD.

Process parameter	Condition
RF power	200 W
Chamber temperature	25 °C
Base pressure	$2 \times 10^{-5}$ Torr
Working pressure	$8.0 \times 10^{-2}$ Torr
O <sub>2</sub> flow rate	3 sccm
DEZn flow rate	1 sccm
Ar flow rate	10 sccm
Bubble pressure	100 Torr
Chiller temperature	15°C
Coating time	15 min, 30 min, 1 h, 1.5 h

inter-particle contacts. The pressed electrodes were dried in a vacuum oven at 80°C for 12 h. Half-cells were then assembled with the bare or ZnO-coated LiCoO<sub>2</sub> as the working electrode, lithium metal foil as the counter electrode, 1 M LiPF<sub>6</sub>-EC:DMC:EMC (1:1:1 by volume) as the electrolyte, and a polypropylene-based film as the separator. All the assembled cells were charged and discharged galvanostatically over the voltage range,  $3.0-4.5 V_{Li/Li+}$  at a 1 C rate by means of a Maccor 4000 battery cycler. Electrochemical impedance spectroscopy (EIS) measurements were performed on cells in the fully discharged state using a Schlumberger model SI 1260 impedance/gain-phase analyzer connected to a Schlumberger model SI 1286 electrochemical interface. The amplitude of the a.c. signal was 5 mV over the frequency range of 1 MHz and 10 mHz.

To investigate the effect of the coating on the thermal stability of the charged cathodes, the thermal decomposition behaviour of both bare and ZnO-coated LiCoO<sub>2</sub> was examined by means of differential scanning calorimetry (DSC) and thermogravimetric (TG) analysis. For the DSC (DuPont instruments, DSC 2910) experiments, the fresh cells were charged to 4.5 V at a 0.1 C rate and held at that voltage for 24 h. After disassembling the cells in a dry room, approximately 5 mg of the cathode material containing the electrolyte was hermetically sealed in an aluminum DSC sample pan. All experiments were performed at a heating rate of  $5^{\circ}$ C min<sup>-1</sup>. Similarly, TGA (TA instruments, SDT 2960) was also carried out at a heating rate of  $5^{\circ}$ C min<sup>-1</sup> in an argon atmosphere.

#### 3. Results and discussion

The XRD patterns of bare  $LiCoO_2$  and ZnO-coated  $LiCoO_2$  powders are given in Fig. 2. The ZnO coating does not cause any noticeable change in the crystal structure of  $LiCoO_2$ . All the XRD



Fig. 2. XRD patterns of bare  $LiCoO_2$  and ZnO-coated  $LiCoO_2$  powders, showing a rhombohedral structure with R3-m space group.



Fig. 3. SEM images of bare and ZnO-coated LiCoO2 particles: (a) bare, (b) 0.08 wt.%, (c) 0.10 wt.%, (d) 0.21 wt.%, and (e) 0.49 wt.% ZnO-coated LiCoO2.



Fig. 4. BET surface area of bare and ZnO-coated  $\text{LiCoO}_2$  with different coating amounts.

patterns of samples coated with ZnO appear to be the same as that of bare  $LiCoO_2$ , due to the very small amount (less than 0.5 wt.%) of coating oxides relative to the  $LiCoO_2$  powder.

The morphology of bare and ZnO-coated LiCoO<sub>2</sub> particles with different coating amounts is presented in Fig. 3. A smooth and clean surface is observed in the case of the bare commercial LiCoO<sub>2</sub> in Fig. 3(a), while for all coated samples, several tens of nano-sized spherical oxide particles are uniformly dispersed on the LiCoO<sub>2</sub> surface, providing a coating with a diameter of about 20-30 nm, as shown in Fig. 3(b), (c) and (d). As the coating amount is increased to 0.21 wt.%, the ZnO particles increasingly cover the entire surface of LiCoO<sub>2</sub>. As shown in Fig. 3(e), however, severe agglomeration of ZnO nanoparticles is found on the surface of the 0.49 wt.% ZnO-coated LiCoO<sub>2</sub>, and the size distribution of this clustered ZnO is no longer uniform. The corresponding changes in the specific surface area of the bare and ZnO-coated LiCoO<sub>2</sub> as a function of the coating amount are given in Fig. 4. As expected, the BET surface area increases with increasing ZnO coating amount. The relatively small increase in the surface area at a ZnO coating amount of 0.49 wt.% might be caused by the existence of the agglomerated oxide particles, as shown in Fig. 3(e).

Fig. 5(a) shows the changes in the discharge capacities during repeated charge-discharge cycling for the bare and ZnO-coated LiCoO<sub>2</sub> with different coating amounts. The ZnO coating substantially improves the cyclic performance of LiCoO<sub>2</sub>. The results of the cycling test in the range of 3.0-4.5V reveal that both bare and ZnO-coated LiCoO<sub>2</sub> materials exhibit little difference in their discharge capacity during the first few cycles. In subsequent cycles, however, the bare LiCoO<sub>2</sub> abruptly degrades, whereas the ZnO-coated LiCoO<sub>2</sub> fades relatively slowly. In addition, different coating amounts cause different degrees of capacity retention upon cycling. Fig. 5(b) shows the capacity retention of the test cells after 30 cycles as a function of the concentration of deposited  $Zn^{2+}$ , where capacity retention is defined as the percentage of the discharge capacity that remains after the 30th cycle with respect to the discharge capacity at the first cycle. Compared with bare LiCoO<sub>2</sub> that displays a capacity retention of 37% after 30 cycles, the 0.21 wt.% ZnO-coated LiCoO<sub>2</sub> retained approximately 65% of its initial capacity. Further increases in the coating amount result in a decrease in cyclic stability, which might be due in part to the electrochemical inactivity resulting from the excess coating materials. It is believed that the presence of an excess amount of ZnO on the surface of the matrix particles may lead to a decrease in the particle-particle electronic conductivity and the formation of agglomerated ZnO particles, as observed on the surface of 0.49 wt.%



**Fig. 5.** (a) Cycling performance of bare and ZnO-coated LiCoO<sub>2</sub> with different coating amounts between 3 V and 4.5 V at 1 C rate, and (b) capacity retention of test cells after 30 cycles as function of deposited  $Zn^{2+}$  concentration.

ZnO-LiCoO<sub>2</sub>. This may inhibit lithium-ion transport through the interface between the surface layer and cathode particles.

Cobalt dissolution from a LiCoO<sub>2</sub> cathode during high-voltage cycling, which causes degradation of the LiCoO<sub>2</sub> structure, is widely held to be one of the major reasons for capacity fading at high cut-off voltages [17–20]. For a direct examination of the effect of the surface coating on Co dissolution from LiCoO<sub>2</sub> cathode, approximately 70 mg of the bare and 0.21 wt.% ZnO-coated LiCoO<sub>2</sub> powders, which were used in the cycling test (30 cycles at 1 C rate), were prepared and then immersed in a 1 M LiPF<sub>6</sub>-EC:DMC:EMC(1:1:1 by volume) electrolyte for 10 days at 80°C. As shown in Table 2, the amount of Co dissolved from the 0.21 wt.% ZnO-coated LiCoO<sub>2</sub> is less than 0.1 ppm, which is much lower than that from the bare LiCoO<sub>2</sub>, viz., 0.79 ppm. This indicates that the uniform dispersion of ZnO nanoparticles on the LiCoO<sub>2</sub> and electrolyte which, in turn, can reduce the dissolution of Co ions from LiCoO<sub>2</sub> into the electrolyte.

The effect of ZnO coating on the thermal stability of charged cathodes was examined by comparing the DSC curves (Fig. 6) of

Table 2

AAS analysis of dissolved Co content of bare and ZnO-coated  $\rm LiCoO_2$  immersed in  $1\,M\,\rm LiPF_6-EC:DMC:EMC\,(1:1:1)$  electrolyte.

Sample	Co content dissolved in electrolyte (ppm)
Bare LiCoO <sub>2</sub>	0.79
0.21 wt.% ZnO-coated LiCoO <sub>2</sub>	<0.1

fully-charged bare and ZnO-coated LiCoO<sub>2</sub> active materials without removing the electrolyte. The extent of the exothermic reaction is much less and the onset temperature for thermal decomposition is much higher for all ZnO-coated LiCoO<sub>2</sub> materials than for bare LiCoO<sub>2</sub>. In particular, 0.21 wt.% ZnO coating on the surface of LiCoO<sub>2</sub> significantly delays the onset temperature of the exothermic reaction by at least 15°C, and its relative integrated area under the peak is smaller than a quarter of that of the bare material. In addition, the thermal behaviour of the coated materials as a function of the coating amount is fairly consistent with the tendency of the results obtained from the electrochemical cycling tests. Details of peak temperatures and amount of heat generated through the loss of oxygen for the bare and ZnO-coated LiCoO<sub>2</sub> are presented in Fig. 6(b).

Similar trends are also observed in the TGA results, as shown in Fig. 7(a). The weight-loss profile significantly varies with the coating amount. As is widely known, the large weight losses that occur at approximately 250 °C correspond to oxygen liberated from the degraded LiCoO<sub>2</sub> layered structure at high temperatures. Because there is a different amount of electrolyte solvent in each sample, different amounts of weight loss that are due to the volatile solvents are observed below 150 °C. For a more accurate comparison of the oxygen generation temperature for each sample, dM/dT (the temperature derivative of the sample weight) was plotted as a function of sample temperature. This procedure allows the weight-loss profile to be based on the same reference point above 150 °C. The



**Fig. 6.** (a) DSC profiles of bare and ZnO-coated  $LiCoO_2$  with different coating amounts, and (b) variation of peak temperatures and amount of heat generated through loss of oxygen for bare and ZnO-coated  $LiCoO_2$ . Cells were charged to 4.5 V at 0.1 C rate, and equilibrated at that voltage for 24 h.



**Fig. 7.** (a) TGA results for bare and ZnO-coated  $LiCoO_2$  with different coating amounts, and (b) derivative, dM/dT, versus temperature for the data sets shown in (a). Cells were charged to 4.5 V at 0.1 C rate, and equilibrated at that voltage for 24 h.

data in Fig. 7(b) clearly show that the release of oxygen from the 0.21 wt.% ZnO-coated LiCoO<sub>2</sub> sample occurs at a much higher temperature than that from the other materials. Both DSC and TGA/DTA data reveal that the ZnO coating is effective in retarding the decomposition reaction of LiCoO<sub>2</sub> with the electrolyte, and thereby leads to a decrease in the amount of heat generated, as well as an improvement in capacity retention during high-voltage cycling.

The role of the ZnO coating was further confirmed by impedance analysis. Fig. 8 shows the impedance spectra (Nyquist plots) of both bare and ZnO-coated electrodes measured after the 10th, 20th and 30th cycles. All the spectra consist of two well-defined semicircles, followed by a very short linear portion. In general, these impedance spectra exhibit a small, high-frequency semicircle, which is related to the resistance of the surface films that cover the LiCoO<sub>2</sub> particles in the electrolyte, and also a large, medium-to-low frequency semicircle, which is attributed to the charge-transfer resistance at the interface between the surface layer and the cathode particles, as discussed in previous studies [21,22]. The inclined line in the lower-frequency range represents the Warburg impedance, which is associated with lithium-ion diffusion in the LiCoO<sub>2</sub> particles. The diameters of the second semicircles, corresponding to the charge-transfer resistance, for both bare and ZnO-coated electrodes increase with increasing number of cycles. On the other hand, there is little change in the high-frequency semicircle for any of the electrodes during cycling. In particular, the bare LiCoO<sub>2</sub> electrode exhibits a drastic increase in charge-transfer resistance after



Fig. 8. Impedance spectra of bare  $LiCoO_2$  and ZnO-coated  $LiCoO_2$  electrodes obtained after (a) 10th, (b) 20th, and (c) 30th cycles.

prolonged cycling, while the ZnO-coated LiCoO<sub>2</sub> electrodes, particularly the 0.21 wt.% ZnO-coated LiCoO<sub>2</sub> electrode, display much smaller increase in the interfacial resistance compared with the bare LiCoO<sub>2</sub>. As shown in Fig. 8(a), however, the effect of the coating on cell impedance appears to be rather insignificant during the initial period of cycling, and even the 0.08 wt.% ZnO-LiCoO<sub>2</sub> is slightly more resistive than bare LiCoO<sub>2</sub> surface. These results imply that the ZnO coating plays a major role in reducing the cell impedance, in particular the charge-transfer resistance, during extended cycling tests, rather than affecting the initial impedance values of the LiCoO<sub>2</sub> electrode. Regarding the cell impedance, Chen et al. [23] suggested that the cathode-side impedance, especially the charge-transfer resistance, is the main contributor to the cell impedance. We conclude that the decrease in charge-transfer resistance through surface coating by ZnO, which is probably due to the suppressed dissolution of cations from the cathode, is one of the major reasons for the improved capacity retention of the LiCoO<sub>2</sub> electrode on cycling, as shown in Fig. 5.

#### 4. Conclusions

ZnO-coated LiCoO<sub>2</sub> particles have been prepared by the plasmaenhanced chemical vapour deposition method with various coating amounts. As the coating amount is increased to 0.21 wt.%, uniformly-distributed ZnO nanoparticles increasingly cover the LiCoO<sub>2</sub> surfaces without any change in particle size, and these coated oxides greatly reduced the amount of Co dissolution from LiCoO<sub>2</sub> into the electrolyte. At a coating amount of 0.49 wt.%, however, there is severe agglomeration between ZnO particles on the surface of LiCoO<sub>2</sub>. From cycling tests in the range of 3.0-4.5 V, the effect of the ZnO coating on the discharge capacity is rather insignificant during the first few cycles, but the coating effectively improves capacity retention after prolonged cycling. In the experimental range studied, the optimum amount of ZnO coating that maximizes capacity retention is 0.21 wt.%, which is guite consistent with the optimum range reported by Fang et al. [11], even though these authors used a different coating solution and procedure. On the other hand, an excessive amount of ZnO coating results in a decrease in both cyclic performance and thermal stability. It is concluded that the agglomerated ZnO coating oxide particles on the surface of 0.49 wt.% ZnO-LiCoO<sub>2</sub> can act as an obstacle to lithium intercalation through the interface between the surface layer and the cathode particles. Both DSC and TGA data reveal that the ZnO coating is effective in retarding the decomposition reaction of LiCoO<sub>2</sub> with the electrolyte, thereby leading to a decrease in the amount of heat generated. In addition, the variation of thermal behaviour with coating amount is fairly consistent with the results obtained from electrochemical cycling tests. The a.c. impedance measurements confirm that the ZnO coating on the LiCoO<sub>2</sub> particles substantially decreases the cell impedance, in particular the charge-transfer resistance, after prolonged cycling. In conclusion, Co dissolution from the LiCoO<sub>2</sub> cathode during high-voltage cycling and increase in charge-transfer resistance on cycling can be considered as the major reasons for capacity fading, especially during high-voltage cycling, and a ZnO coating on the LiCoO<sub>2</sub> surface by PE-CVD successfully mitigates these problems. Surface modification with ZnO also has beneficial effects on the thermal stability of charged LiCoO<sub>2</sub>.

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