Effect of Choices of Positive Electrode Material, Electrolyte, Upper Cut-Off Voltage and Testing Temperature on the Life Time of Lithium-Ion Cells

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The impact of the choices of positive electrode material, coating types, electrolyte (varied electrolyte additive combinations), upper cutoff voltage and testing temperature on the lifetime of various NMC/graphite cells were systematically studied in this work. Ultra-high precision coulometry (UHPC) and long term cycling tests between 3–4.1, 4.2 and 4.3 V were conducted using pouch type cells (~250 mAh). Cells with ~90% capacity retention between 3–4.3 V after ~2500 cycles (~1.8 years of testing) at 20°C, and cells with ~81% capacity retention between 3–4.3 V after ~2200 cycles at 40°C were developed. The correlation between UHPC and long term cycling data was also explored. The major outcome of this work is that NMC622/graphite cells, with appropriate electrolyte additives, can outperform NMC532/graphite cells, demonstrating that cells with NMC grades having higher nickel content need not have shorter lifetime.

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tap density, particle size distribution, etc. Coatings “A” and “B” were applied to these materials. Coating “A” is a typical aluminum oxide based coating whereas coating “B” is a coating based on aluminum and fluoroine. Cells containing these materials are called NMC532A, NMC622A, NMC532B and NMC622B cells, respectively. The positive electrode materials produced by Umicore were shipped to LiFUN Technology in China for pouch cell assembly.

**Pouch cells.**—402035 size machine-made NMC/natural graphite (NG) pouch cells (nominal 240 mAh to 4.4 V for NMC532 and nominal 220 mAh for NMC622) were obtained dry (no electrolyte) from LiFUN Technology (Ximma Industry Zone, Golden Dragon Road, Tianyuan District, Zhuzhou City, Hunan Province, PRC, 412000)). For simplicity the cells will be labelled after the positive electrode materials as described above. The negative electrodes in these cells each had a loading of 13.2 mg/cm² and were calendared to a density of 1.55 to 1.6 g/cm³. The negative electrodes were made from natural graphite (grade BTR-918, from BTR, China) and had compositions of 95.2% active material, 2% carbon black with the remainder being carboxylmethyl cellulose (CMC)/styrene butadiene (SBR) binder. The positive electrode loading was 21.1 mg/cm² for NMC532 and 19.3 mg/cm² for NMC622. All the positive electrodes were calendared to 3.3 g/cm³. The positive electrodes were 96% by weight active material with 2% conducting carbon additive and 2% by weight PVDF binder. The NMC532/graphite cells had a larger nominal capacity (240 mAh) than the NMC622 cells (220 mAh) because their electrodes were slightly longer and led to a slightly thicker jelly roll.

Based on this design, the cells could be charged at low rate to 4.5 V without unwanted lithium plating. The dry cells were vacuum sealed and placed in a heated vacuum oven at 100 °C long term cycling results for the cells with 2VC electrolyte, respectively. For each cycle, the fractional charge end point capacity slippage per hour (Fract. Slip./h), and the fractional fade per hour (Fract. Fade/h) as a function of upper cutoff voltage. The results for NMC532A, NMC532B, NMC622A and NMC622B cells are shown in subpanels 1 - 4, respectively. The red diamonds with a solid line, blue crosses with a solid line and green circles with a solid line show the results for cells with PES211, VC211 and 2VC electrolyte, respectively. For each cycle, CIE/h = (1 − CE)/t, [1] Fract.Slip./h = slope(Ch.End.Cap/Qd1/t) and, [2] Fract.Fade/h = slope(Qd1/Qa1/t), [3] where t is the time of each charge-discharge cycle, Qa1 is the first discharge capacity and the results from the 16th cycle have been plotted. The slope was calculated from the data in Figure 1 using the 12th to 16th cycle are very similar, which is ~0.9985, for the four types of cells regardless of the upper cutoff voltage. However, Figure 1 shows that the charge end point capacity slippage increased with increasing upper cutoff voltage for each type of cells. The UHPC results for cells with 2VC, VC211 and PES211 electrolytes were summarized.

Figure 2 shows a summary of the UHPC results, where Figures 2a, 2b and 2c show the coulombic inefficiency per hour (CIE/h), the charge end point capacity slippage per hour (Fract. Slip/h), and the fractional fade per hour (Fract. Fade/h) as a function of upper cutoff voltage. The results for NMC532A, NMC532B, NMC622A and NMC622B cells are shown in subpanels 1 - 4, respectively. The general trend is that the cells with 2VC and VC211 had higher CIE/h, Fract. Slip/h and Fract. Fade/h compared to PES211 electrolytes. The black crosses, red circles and blue diamonds show the results for cells with 2VC, VC211 and PES211 electrolytes, respectively. For each cycle, CIE/h = (1 − CE)/t, [1] Fract.Slip./h = slope(Ch.End.Cap/Qd1/t) and, [2] Fract.Fade/h = slope(Qd1/Qa1/t), [3] where t is the time of each charge-discharge cycle, Qa1 is the first discharge capacity and the results from the 16th cycle have been plotted. The slope was calculated from the data in Figure 1 using the 12th to 16th cycle. We expect the CIE, fractional slippage and fractional fade to be related by: CIE/h = Fract.Slip./h + Fract.Fade/h, [4] as discussed by Harlow et al. and Smith et al. Figures 2a and 2b show that the CIE/h and Fract. Slip./h increased with increasing upper cutoff voltage, respectively, while Figure 2c shows that the Fract. Fade/h decreased in general when a particular cell type and particular electrolyte are considered. Additionally, Figure 2a shows that cells with 2VC had the highest (worst) CIE/h for all cell types at all voltages. Figures 2a1 and 2a2 show that NMC532A and NMC532B cells with VC211 electrolyte had slightly lower CIE/h than the same cells with PES211 electrolyte, respectively, with the same upper cutoff voltages. Figures 2a3 and 2a4 show that NMC622A and NMC622B cells, respectively, with VC211 electrolyte had higher CIE/h than the same cells with PES211 electrolyte at 4.1 V upper cutoff voltage, however, they were similar at 4.2 and 4.3 V upper cutoff voltages. Figure 2b shows that Fract. Slip./h decreased in the order of cells with 2VC, VC211 and PES211 electrolytes when the same cell type was compared.

The coulombic efficiency of cells was measured using the ultra-high precision chargers at Dalhousie University described in Reference 29. The cells after formation were tested at C/20 for 16 cycles between 3–4.1, 4.2 and 4.3 V. The cells were placed at 40 °C long term cycling. —The cells were tested for long term cycling with a C/3 rate between 3–4.1, 4.2 and 4.3 V. One cycle at a C/20 rate was included every 50 cycles. The cells were held at the top of charge until the current reached C/20 during each cycle. The tests were performed at 20 or 40 ± 0.1 °C in temperature-controlled boxes. Neware (Shenzhen, China) chargers were used for these tests.

Results and Discussion

Figures 1a–1d show the UHPC results for NMC532A, NMC532B, NMC622A and NMC622B cells, respectively, with VC211 electrolyte. In each panel the coulombic efficiency (CE), charge end point capacity (Cha. End. Pt. Cap.), discharge capacity (Qd) and ΔV (difference between average charge and average discharge voltages) are shown. For readers unfamiliar with charge end point capacity, this is the value of the cell capacity at the top of charge tracked cumulatively during the test. The charge end point has been arbitrarily set to zero capacity at cycle 2 for all cells. The charge end point capacity slippage, to be discussed later, (units = mAh/cycle) is the slope of the charge end point versus cycle number curve. The fractional charge end point slippage per hour (units = h⁻¹) is the charge end point capacity slippage divided by the cell capacity (mAh) and divided by the time of one cycle (h/cycle). The black crosses, red circles and blue diamonds show the results for cells measured with an upper cutoff voltage of 4.1, 4.2 and 4.3 V, respectively. Figure 1 shows that the CEs at the 16th cycle are very similar, which is ~0.9985, for the four types of cells regardless of the upper cutoff voltage. However, Figure 1 shows that the charge end point capacity slippage increased with increasing upper cutoff voltage for each type of cells. The UHPC results for cells with 2VC, VC211 and PES211 electrolytes were summarized.

**Ultra-high precision coulometry (UHPC).**—The coulombic efficiency of cells was measured using the ultra-high precision chargers at Dalhousie University described in Reference 29. The cells after formation were tested at C/20 for 16 cycles between 3–4.1, 4.2 and 4.3 V. The cells were placed at 40 ± 0.1 °C in temperature-controlled boxes during the tests.

20 and 40 °C long term cycling.—The cells were tested for long term cycling with a C/3 rate between 3–4.1, 4.2 and 4.3 V. One cycle at a C/20 rate was included every 50 cycles. The cells were held at the top of charge until the current reached C/20 during each cycle. The tests were performed at 20 or 40 ± 0.1 °C in temperature-controlled boxes. Neware (Shenzhen, China) chargers were used for these tests.
Figure 1. The UHPC results for NMC532A (a), NMC532B (b), NMC622A (c) and NMC622B cells (d), respectively, with VC211 electrolyte. In each panel the coulombic efficiency (CE), charge end point capacity (Cha. End. Pt. Cap), discharge capacity (Qd) and ΔV (differences between average charge and average discharge voltages) are shown.

4.1, 4.2 and 4.3 V, respectively, while Figures 3a2–3c2 show the normalized capacity as a function of cycle number and Figures 3a3–3c3 show ΔV as a function of cycle number for the corresponding cells. The blue diamonds, green squares, black crosses and red circles show the results for NMC622A, NMC622B, NMC532A and NMC532B cells, respectively. Figures 4 and 5 show the corresponding results for cells with PES211 and VC211 electrolytes, respectively. Figures 6, 7 and 8 show the corresponding results for cells with 2VC, PES211 and VC211 electrolytes, respectively, tested at 40°C while the other conditions remained the same.

Figures 3–5 show that NMC532A and NMC532B cells show worse capacity fade and more rapid impedance growth (as monitored by ΔV) than NMC622A and NMC622B cells under the same conditions when the same electrolyte was used at 20°C. Additionally, NMC622A and NMC622B cells with VC211 electrolyte show the best capacity retention and ΔV control at all voltages. Figure 5a2 shows that NMC622A and NMC622B cells have capacity retentions of ~92% and ~93%, respectively, after ~2500 cycles (~1.7 years of testing) between 3 - 4.1 V, while Figure 5b2 shows ~88% and ~91% capacity retentions for NMC622A and NMC622B cells, respectively, tested between 3 - 4.2 V and Figure 5c2 shows ~87% and ~90% capacity retentions for the corresponding cells tested between 3–4.3 V after ~2500 cycles.

Figures 6–8 show that the capacity fade accelerated when the temperature increased from 20 to 40°C while the other conditions remained the same. Figures 6–8 show that NMC622A and NMC622B cells with the VC211 electrolyte had the best capacity retentions and ΔV control at all voltages at 40°C. Figure 8a2 shows NMC622A and NMC622B cells with VC211 electrolyte had capacity retentions of ~85% and 84%, respectively, after ~2500 cycles between 3–4.1 V, whereas Figure 8b2 shows capacity retentions of ~81% and 79%...
for the corresponding cells tested between 3–4.2 V for ~2500 cycles and Figure 8c2 show capacity retentions of ~79% and ~81% for the corresponding cells tested between 3–4.3 V for ~2200 cycles.

It is interesting that Figure 2 showed almost the same coulombic inefficiency for the NMC622A cells independent of testing voltage and almost the same coulombic inefficiency for the NMC622B cells independent of testing voltage when VC211 electrolyte was used in tests at 40°C. Figures 8a2, 8b2 and 8c2 show that the capacity retentions of the NMC622A and NMC622B cells were almost the same, independent of upper cutoff potential, for the 40°C tests when VC211 electrolyte was used, in agreement with the UHPC results. UHPC results, collected at C/20, do not capture the effects of impedance growth on capacity loss well. In the case of these cells, the impedance growth is small as shown in Figures 8a3, 8b3 and 8c3 which is why we believe the predictions from the UHPC experiments hold true. The NMC622A cell with VC211 tested to 4.3 V (see Figures 8c2 and 8c3) shows an increase in impedance growth and in increase in capacity loss near 2000 cycles.

The cycling results shown in Figures 3–8 were summarized. The average capacity fade per 100 cycles and the average ΔV growth per 100 cycles were calculated by dividing the total capacity fade and the total ΔV growth, respectively, by the total cycle number and then multiplying by 100. Figure 9 shows a summary of results for cells tested at 20°C. Figures 9a1–9a4 show the average fade per 100 cycles for NMC532A, NMC532B, NMC622A and NMC622B, respectively, as a function of upper cutoff voltage, while Figures 9b1–9b4 show the average ΔV growth per 100 cycles as a function of upper cutoff voltage for the same cells. Figure 9 shows that the average fade rate and average ΔV growth rate increased with increasing upper cutoff voltage. Figure 9 shows that cells with VC211 showed the smallest fade rate and ΔV growth rate of all cell types when the upper cutoff voltage was the same. Additionally, Figure 9 shows that NMC622A and NMC622B with VC211 electrolyte had the smallest fade rate and ΔV growth rate at upper cutoff voltages of 4.1 and 4.2 V, while NMC622B cells had a smaller fade rate and ΔV growth rate than NMC622A cells at 4.3 V with VC211 electrolyte. Figure 10 shows the corresponding summary for cells tested at 40°C. Figure 10 shows similar trends in that the average fade rate and ΔV growth rate increased with increasing upper cutoff voltage when the cell type and electrolyte were fixed. Additionally, NMC622A and NMC622B cells showed a smaller average fade rate and smaller ΔV growth rate than NMC532A and NMC532B cells when the same electrolyte and upper cutoff voltage were compared. NMC622A and NMC622B cells with VC211 electrolyte showed the smallest average fade rate and ΔV growth rate at all upper cutoff voltages, while NMC622B cells with VC211 electrolyte showed slightly better performance than the corresponding NMC622A cells at the upper cutoff voltage of 4.3 V.

Figures 11a1, 11b1 and 11c1 show the correlation between the average capacity fade per 100 cycles and CIE/h, Fract. Fade/h and Fract. Fade/h, respectively, measured from UHPC for NMC622A cells tested at 40°C, while Figures 11a2–11c2 show the corresponding correlation for NMC622B cells. Figure 12 shows the correlation between the average ΔV growth per 100 cycles and the CIE/h, Fract. Slip./h and Fract. Fade/h for the corresponding cells. Cells with 2VC, PES211 and VC211 electrolytes are shown in green circles, red diamonds and blue crosses, respectively. The upper cutoff voltage is shown beside each data point. Figures 11 and 12 show that average fade rate and ΔV growth rate, respectively, increased with increasing CIE/h and Fract. Slip./h when the cell type and electrolyte were fixed. This demonstrates the value of the short-term UHPC measurements in directing the search for electrolytes that yield long lifetime. However, the correlation is not as strong, with some outliers, when all the electrolytes were included for each cell type. Some cases of interest are discussed below.

Figures 11 and 12 clearly show that cells with 2VC have higher CIE and higher charge end point capacity slippage, as determined by short-term UHPC measurements, than the corresponding cells (same cell type and same upper cutoff potential) with PES211 or VC211. Figures 11 and 12 also clearly show that cells with 2VC have higher
Figure 3. The 20°C cycling results for the cells with 2VC electrolyte. Discharge capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a1), 4.2 (b1) and 4.3 V (c1), respectively. The normalized capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a2), 4.2 (b2) and 4.3 V (c2), respectively. ΔV as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a3), 4.2 (b3) and 4.3 V (c3), respectively. In most cases results for two pair cells are shown.

Figure 4. The 20°C cycling results for the cells with PES211 electrolyte. Discharge capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a1), 4.2 (b1) and 4.3 V (c1), respectively. The normalized capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a2), 4.2 (b2) and 4.3 V (c2), respectively. ΔV as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a3), 4.2 (b3) and 4.3 V (c3), respectively. In most cases results for two pair cells are shown.
Figure 5. The 20°C cycling results for the cells with VC211 electrolyte. Discharge capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a1), 4.2 (b1) and 4.3 V (c1), respectively. The normalized capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a2), 4.2 (b2) and 4.3 V (c2), respectively. ΔV as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a3), 4.2 (b3) and 4.3 V (c3), respectively. In most cases results for two pair cells are shown.

Figure 6. The 40°C cycling results for the cells with 2VC electrolyte. Discharge capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a1), 4.2 (b1) and 4.3 V (c1), respectively. The normalized capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a2), 4.2 (b2) and 4.3 V (c2), respectively. ΔV as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a3), 4.2 (b3) and 4.3 V (c3), respectively. In most cases results for two pair cells are shown.
Figure 7. The 40°C cycling results for the cells with PES211 electrolyte. Discharge capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a1), 4.2 (b1) and 4.3 V (c1), respectively. The normalized capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a2), 4.2 (b2) and 4.3 V (c2), respectively. ∆V as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a3), 4.2 (b3) and 4.3 V (c3), respectively. In most cases results for two pair cells are shown.

Figure 8. The 40°C cycling results for the cells with VC211 electrolyte. Discharge capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a1), 4.2 (b1) and 4.3 V (c1), respectively. The normalized capacity as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a2), 4.2 (b2) and 4.3 V (c2), respectively. The ∆V as a function of cycle number for cells tested with upper cutoff voltages of 4.1 (a3), 4.2 (b3) and 4.3 V (c3), respectively. In most cases results for two pair cells are shown.
capacity fade rates and higher impedance growth rates than cells with VC211 as could be expected from the UHPC results. Figure 11 shows that cells with PES211 have higher capacity fade rates than cells with VC211 which would not be expected based on the UHPC results which show lower CIE and lower charge end point capacity slippage for PES211 cells than VC211 cells. However, as stated earlier, the UHPC measurements made at low rates (C/20) do not capture the effects of long term impedance growth in cells and its impact on capacity retention. Figure 12 shows that cells with PES211 have larger impedance growth rates than corresponding cells with VC211. It is for this reason that cells with PES211 show larger capacity fade than cells with VC211, in our opinion. This points to differences in the parasitic reactions occurring in VC211 and PES211 cells. Even though the rates of parasitic reactions are lower in PES211 cells compared to VC211 cells, apparently their impact on impedance growth is larger.

Conclusions

This work studied the impact of the choices of positive electrode material, different electrode material coatings, electrolyte (with various electrolyte additive combinations), upper cutoff voltage and testing temperature on the lifetime of NMC/graphite cells. The positive electrode materials studied here included: NMC532A, NMC532B, NMC622A and NMC622B. The “A” type coating was based on Al2O3
Figure 11. The correlation between the average capacity fade per 100 cycles and CIE/h (a1), Fract. Slip./h (b1) and Fract. Fade./h (c1), respectively, measured from UHPC for NMC622A cells tested at 40°C. The correlation between the average capacity fade per 100 cycles and CIE/h (a2), Fract. Slip./h (b2) and Fract. Fade./h (c2), respectively, measured from UHPC for NMC622B cells tested at 40°C. In most cases results for two pair cells are shown.

Figure 12. The correlation between the average ΔV growth per 100 cycles and CIE/h (a1), Fract. Slip./h (b1) and Fract. Fade./h (c1), respectively, measured from UHPC for NMC622A cells tested at 40°C. The correlation between the average ΔV growth per 100 cycles and CIE/h (a2), Fract. Slip./h (b2) and Fract. Fade./h (c2), respectively, measured from UHPC for NMC622B cells tested at 40°C. In most cases results for two pair cells are shown.
and the “B” type coating was based on aluminum and fluorine. The electrolytes studied included: 2VC, PES211 and VC211. The cells were tested at both 20 and 40 °C between 3–4.1, 4.2 and 4.3 V. It was shown that cells with VC211 electrolyte had the best performance in most cases, and that NMC622A and NMC622B cells showed better performance than NMC532A and NMC532B cells when the same electrolyte was used under the same testing conditions. NMC532A and NMC532B cells showed worse capacity fade and ΔV growth than NMC622A and NMC622B cells under the same conditions when the same electrolyte was used. Additionally, NMC622A and NMC622B cells with VC211 electrolyte showed the best capacity retention and ΔV control at all voltages. NMC622A and NMC622B cells had capacity retentions of ~92% and ~93%, respectively, between 3–4.1 V, ~88% and ~91%, respectively, between 3–4.2 V and ~87% and ~90%, respectively, between 3–4.3 V after ~2500 cycles and ~1.7 years of testing at 20 °C. At 40 °C, NMC622A and NMC622B cells with the VC211 electrolyte had capacity retentions of ~85% and 84%, respectively, between 3–4.1 V, ~81% and 79%, respectively, between 3–4.2 V after ~2500 cycles and ~79% and ~81%, respectively, between 3–4.3 V after ~2200 cycles.

The correlations between the long term cycling results and the UHPC measurements during the early life of the cells were explored. Correlations between UHPC and long term cycling results were generally good. However, cells with PES211 had larger capacity fade rates than expected based on the UHPC results. This was caused by larger than expected impedance growth in these cells. The experiments reported here dispel the myth that cells with NMC grades with higher Ni content generally have shorter lifetimes. Here it is definitively shown that NMC622 grades with the same particle sizes and coatings as NMC532 grades used in cells of the same design and the same electrolytes have longer lifetime.

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