The challenges, solutions and development of high energy Ni-rich NCM/NCA LiB cathode materials

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Abstract. In the last three decades, the successful application of lithium ion batteries (LIB) for consumer electronics has laid solid foundation for the rapid development of large format batteries for electric vehicles (EV) and energy storage systems (ESS). Up to now, in most of the commercial LIB, carbon material, e.g. graphite is used as anode material, while the cathode material changes from spinel LiMn₂O₄ (LMO), olivine LiFePO₄ (LFP), to layer-structured material LiNiₓ₋₀.₅Co₀.₅Mn₀.₅O₂, and LiNiₓ₋₀.₅Co₀.₅Al₀.₅O₂ (NCA), in order to get increased gravimetric and volumetric energy density. By combining the merits of the high capacity of lithium nickel oxide (LiNiO₂), with the good rate capability of lithium cobalt oxide (LiCoO₂), and the thermal stability and low cost of lithium manganese oxide (LiMn₂O₄), lithium nickel cobalt manganese oxide (LiNiₓ₋₀.₅Co₀.₅Mn₀.₅O₂, NCM) enjoys outstandingly comprehensive advantages, and turns to be the major cathode material for lithium ion batteries. One way to increase the energy density of NCM/NCA materials is to increase the Ni content and thus lowering the Co/Mn(Al) content, another way to get high energy density is to increase the charging cut-off voltage. High energy NCM/NCA materials are confronted more challenging issues, like degraded cycle life, severe swelling upon thermal storage, and safety problems. This paper will address such issues, and put forward some feasible solutions.

1. Introduction
In recent years, Lithium ion batteries are playing increasingly important roles in energy storage and conversion and largely used for consumer electronics, electric vehicle industry and energy storage system. Sorts of electrode materials, electrolyte systems and cell packages have been developed to meet the increasing demands of these applications. Cathode material is one of the most critical factors determining the energy density and cost of lithium-ion batteries. Therefore, developing new cathode materials with high capacity and good safety is indispensable for high performance lithium-ion batteries[1-3].

With the development and commercial application of electric vehicles, the extended driving range requires high energy of lithium ion battery. Up to now, in most of the commercial LIB, carbon material, e.g. graphite is used as anode material, while the cathode material changes from spinel LiMn₂O₄ (LMO), olivine LiFePO₄ (LFP), to layer-structured material LiNiₓ₋₀.₅Co₀.₅Mn₀.₅O₂, and LiNiₓ₋₀.₅Co₀.₅Al₀.₅O₂ (NCA), which deliver increased gravimetric and volumetric energy density, due to the high specific electrochemical capacity and high density of NCM or NCA materials.

Of course, electric vehicles prefer long moving range, like 300 to 500km, even longer distance for one charge, for high energy battery system, thus high energy density cathode materials, practically, Ni-rich NCM materials with high packing density are preferred. Based on LiNi₁/₃Co₁/₃Mn₁/₃O₂ (NCM111),
LiNi$_{0.5}$Co$_{0.2}$Mn$_{0.3}$O$_2$ (NCM523), LiNi$_{0.6}$Co$_{0.2}$Mn$_{0.2}$O$_2$ (NCM622), LiNi$_{0.8}$Co$_{0.1}$Mn$_{0.1}$O$_2$ (NCM811), the energy density of lithium ion cells cycled in the cut-off voltages of 4.2V-3.0V can be achieved at 180, 210, 230, and 280 Wh/kg.

LiNi$_{1-x-y}$Co$_x$Mn$_y$O$_2$ is essentially a solid solution of LiNiO$_2$-LiCoO$_2$-LiMnO$_2$. With the change of the relative ratio of $x$ and $y$, the property changes, generally corresponded to the separate component compound. The higher the Ni content, the higher is the capacity and more instability, the higher cobalt content means higher rate capability and higher cost, while the higher Mn content means better thermal stability and lower cost. So the most important way to increase the energy density is to increase the Ni content and thus lowering the Co/Mn content. With increasing Ni content in LiNi$_{1-x-y}$Co$_x$Mn$_y$O$_2$, the charging capacity of NCM111, NCM523, NCM622, NCM811 increases from 175, to 186, 198, 213 mAh.g$^{-1}$, respectively [4], while the cost decreases with the lowered Co content.

Besides increasing the Ni content, another way to get high energy density is to increase the charging cut-off voltage. As an example of NCM523, the discharging capacity increases from 162.8 to 185.3, 196.9 and 208.1 mAh.g$^{-1}$ when the upper charging cut-off voltage is increased from 4.25 to 4.40, 4.50 and 4.60V (figure 4), meanwhile, the cost of unit energy decreases correspondingly. During the charging process, NCM materials undergo several phase transitions: H1 $\rightarrow$ M $\rightarrow$ H2 $\rightarrow$ H3. H1, H2 and H3 represent three hexagonal phases and M a monoclinic one. Compared with H1 and H2, H3 phase is in deep de-lithiated state, which is an extremely instable in structure. As shown in figure3, the redox onset of the H2$\rightarrow$H3 phase transition of NMC811 starts at >4.0 V while those of NMC111 and NMC622 commence at >4.4 V. So raising upper cut-off voltage is generally applied to low nickel NCM materials [5].

2. The challenges and the solutions of the Ni-rich NCM materials

2.1. Particle cracking of NCM materials upon repeated charge/discharge

The charging and discharging capacity of NCM materials increases with the Ni content in the same cut-off voltages, therefore, the Ni-rich NCM materials are involved in higher state of charge (SOC) with respect to the theoretical capacity, more phase transition and larger volume change during lithium
insertion/extraction, which will further cause cracks inside the secondary particle along the grain boundary, at the triple junction where three primary particles touch. In such a way, the particle cracking happens upon repeated charge/discharge, the structural stability deteriorates. As is mentioned in section 2, the capacity and thus SOC increases with the increased charging cut-off voltage, the crack becomes more seriously, as illustrated in figure 4.

Figure 4. Schematic illustration for the micro-crack growth and deterioration of LiNi0.76Co0.14Al0.10O2 particle upon cycling (a) [6], cross-sectional SEM images of NCA95 cathodes after 100 cycles and variation in charge transfer resistance of NCA95 at different SOC [7]

The particle crack develops upon repeated cycling, especially at high rate, or large DOD (figure 4 [6,7]). Particle cracking is supposed to be an additional but dominant failure mode of the agglomerated NCM materials, compared to the conventional single crystal material, e.g. lithium cobalt oxide, which is extensively used as cathode material in the LIB of consumer electronics.

Particle cracks cause loss of inner particle connectivity and thus increased resistance, and therefor the degradation of performance. Besides, electrolyte can penetrate along the crack into the particle, causing the increase of the interface area of NCM/electrolyte, and relative shortage of the liquid electrolyte, dry area inside the lithium cell may appear, and capacity may thus fade more quickly even drop sharply. In the other hand, the increase of the interface area of NCM/electrolyte may bring about the thermal instability and the risk of thermal runaway.

One solution to address the particle cracking is to make the NCM particle with high strength, resisting against the pressure during electrode pressing, and the internal stress from the repeated volume change of primary particle upon cycling. Of course, the first step is to prepare precursor with enhanced strength, by tuning the coprecipitation conditions and even the inner configuration of the reactor. As an example of precursor of good strength in figure 5, the particle of radial texture (a) enjoys high strength, while the other one with random texture (b) gives low strength. NCM cathode material made from the high strength precursor generally has better cycling stability. To a much extent, the particle crack can be alleviated, although not totally avoided in such a way.

Another effective way is to prepare single crystal type NCM materials, whose particle is strong enough to resist against physical press or internal stress. The single crystal particle still remains its integrity after long term cycles. Generally, single crystal NCM material enjoys some unique attributes: 1) enhanced strength, alleviating particle crack; 2) increased pellet density, increasing the volumetric energy density; 3) lowered surface area, reducing the side reaction of NCM cathode with electrolyte; 4) enhanced connectivity intra- and inter- particles, in favor of electronic, ionic, and thermal conductivity of the electrode. Consequently, the single crystal NCM material enjoys better performance than conventional agglomerated counterpart, in terms of cycling stability, thermal storage, safety as well as volumetric energy density. Table 1 gives a comparison of the performance of single crystal NCM based LIB with the conventional NCM agglomerates.
The preparation of single crystal NCM materials can be achieved by adjusting the precursor process [8], increasing Li/Me and sintering temperature [9], or adding fusing agents for lowering sintering temperatures [10].

2.2. Structural and chemical instabilities (related to performance deterioration upon cycling and thermal storage)

The charging capacity increases with Ni content in LiNi\(_{1-x-y}\)Co\(_x\)Mn\(_y\)O\(_2\), while the chemical stability deteriorates. The Ni rich NCM material with higher charging capacity involve the transition metal of higher oxidation state, which tends to release active oxygen, causing side reaction of the cathode with electrolyte. There will be more serious side redox reaction between cathode and electrolyte during thermal storage and cycling at elevated temperature. In general, higher Ni content NCM material suffers from worse chemical stability and thermal stability (related to safety issue of lithium ion cells). Subsequently, Ni-rich NCM material-based lithium cell are confronted big challenges in cycling, thermal storage, and safety.

Moreover, Ni-rich material behaves less stable when exposed in air, releasing lithium species impurities on the surface of the particles (this topic will be discussed in the following section).

The performance of NCM material depends fundamentally on the precursor Ni\(_{1-x-y}\)Co\(_x\)Mn\(_y\)(OH)\(_2\). Precursor is the predominant factor to be concerned for addressing the structural and chemical instability. Well-crystallized precursor with enhanced texture, less surface defects, and lower impurities is naturally advantageous for preparing NCM materials. In the industrial practice, the continuous stirred tank reactor (CSTR) or batch reactor are adopted to mass produce precursor material. Generally, the
particle size distribution of precursor prepared by CSTR is wide, and that by batch mode is narrow (figure 6).

Figure 6. SEM of precursors prepared by CSTR (a), batch process (b) and their particle size distribution (c)

Sintering is also quite critical for the performance of NCM materials, over-sintering or less-sintering will deliver poor electrochemical performance. In the case of NCM materials prepared from CSTR precursor with wide particle size distribution, small particles are generally over-sintered with bigger primary grain size, while large particle less-sintered with smaller grain size. In other words, large particles and small ones should be sintered at different temperature for optimized performance. In a sense, precursor produced in batch mode with fairly good consistency is preferred for NCM materials with respect to electrochemical performance.

Doping is a common way for modification of functional materials. LiNi_{1-x-y}Co_xMn_yO_2 itself is a mutually doped solid solution consisting of component LiNiO_2, LiCoO_2 and LiMnO_2, enjoying better electrochemical and thermal stability than the pristine LiNiO_2. Besides, several other elements of ppm level are chosen for doping to further stabilize the structure of the NCM materials, including Mg, Al, Fe, Ti, Zr, Cr, Y, Ga, etc.[11-12]. The mechanism for improved electrochemical performance of NCM material by doping might be: (1) introducing electrochemically inactive elements into the host structure, (2) preventing the undesired phase transition from the layered structure to the rock-salt like one, and (3) promoting the lithium ion transport due to increased lithium slab distance by the dopants. For example, Al doping improves cycling stability and thermal storage of the NCM cathode material, but at the loss of capacity and rate capability. Ti doping is helpful for improved cycleability and rate capability of NCM material. Proper substitution of Cr for Mn and Y/Al for Ni can improve the cycle performance and rate capability of the materials, due to the increased lattice parameter c of the doped materials, beneficial to the diffusion of lithium ions. Most of the research on anion doping is to replace partial O with F[13], Cl[14] and S. The strong electron absorption of F enhances the structural stability of the material, and the strong bond between Li-F also has a promoting effect on the exreaction and insertion of lithium ions. The co-doping of Mg and F reduces the cation mixing, and improves the cycling performance and capacity of the material.

Surface coating is another effective way to improve the stability of Ni-rich cathode materials. Conventionally, one or several metal oxides or phosphates of nano-sized particles are dispersed onto the surface, followed by heat treatment, such as Al_2O_3, TiO_2, CeO_2, Y_2O_3, ZrO_2 and MPO_4 (M = Al, Fe, Y)[15-21]. The mechanism of the coating for the improvement of the materials still remains controversy. The effect of surface coating includes: (1) partial isolation of the oxidizing cathode from electrolyte solution; (2) scavenging the erosive species of HF in electrolyte from attacking cathode materials; (3) consuming the surface lithium impurities. (4) surface doping enhancement due to the diffusion of coating element into the thin surface layer with a high concentration.

2.3. Surface alkali impurities
Layer structured cathode materials generally has surface impurities, i.e. Li_2CO_3 and LiOH, the higher is the Ni content, the more is the lithium impurities, Ni-rich NCM/NCA > Ni-low NCM> LCO. These impurities may cause gelation of the slurry during electrode coating process, result in gassing due to the decomposition of Li_2CO_3 at high voltage, and also are severely detrimental to cell performance in terms of both capacity retention and rate capability, especially at elevated temperature.
The surface lithium impurities consist of two kinds of sources, residual lithium and extractual lithium, respectively. The former comes from the residue of lithium in the sintering process, and the latter results from extraction of lithium from the matrix of particles, due to the chemical instability of the Ni-rich NCM/NCA.

When NCM or NCA materials are prepared, extra lithium source is added to compensate for the loss of lithium during sintering, thus after sintering, there still exists some lithium residue on the surface, in the form of Li$_2$O at high temperature. The Li$_2$O will transform to LiOH and Li$_2$CO$_3$ when reacting with H$_2$O and CO$_2$ in the air. This part of surface impurities can be defined as “residual lithium impurities”. The residual lithium impurities are a kind of enrichment of the unreacted lithium on the surface, is related to the initial Li/Me ratio, temperature profile, atmosphere, the precursor and lithium source used.

The other source of surface lithium impurities is the extraction of lithium species from the matrix of the particle, and further conversion to LiOH and Li$_2$CO$_3$, in series to the diffusion of lithium from the center to the surface layer. In a sense, LiMeO$_2$ can be taken as Li$_2$O.Me$_2$O$_3$, in the existence of moisture and CO$_2$, Li$_2$O is continuously extracted out of the particle, and converted to LiOH and finally to Li$_2$CO$_3$, this part of lithium impurities can be defined as “extractual lithium impurities” when exposed to air, the surface residual LiOH reacts with CO$_2$ preferentially to form Li$_2$CO$_3$, LiOH content decreases quickly in several hours to a lower level, while Li$_2$O is being extracted out, and further converted to Li$_2$CO$_3$, even dominating the increase of the lithium impurities (figure 7). The more Li$_2$O is extracted, the less chemical stability is the NCM material. Generally, the higher Ni content NCM is covered with higher content of lithium impurities; Single crystal NCM has lower initial lithium impurities and is comparatively stable; Precursor from different process also gives NCM of different chemical stability even with the same Ni/Co/Mn composition. One way to restrict the lithium species extraction is to control the content of CO$_2$ and moisture in the atmosphere where NCM is processed. Another way is to wash NCM powders in pure water to remove the surface impurities, but the Li$_2$O in host structure may also be rinsed away, which degrades the cycling stability especially at high temperature.

2.4. Safety issue

From the thermodynamic point of view, the lithium ion battery itself is an unstable high energy system, consisting of highly oxidizing cathode, reductive anode, and flammable organic electrolyte. Efforts should be made to improve the safety systematically, in terms of materials, cells, modules, battery pack, car design, and even application.

There are many factors affecting the thermal stability of cathode materials. The first factor is the composition and the structure of the materials, as is well known, the thermal stability of LFP and LMO is better than NCM, and NCA. The Ni-low NCM materials are fairly stable compared to Ni-rich NCM counterparts. As is shown in figure 8, the heat flows of the charged NCM111, NCM523, NCM622, NCM811 are 512.5, 605.7, 721.4, 904.8 J.g$^{-1}$, and their DSC peak temperature are 306, 290, 264 and
232 °C, respectively\textsuperscript{[10]}. Accordingly, it is a possible way to improve the thermal stability of NCM with certain Ni content by increasing Mn content and lowering Co content, keeping the similar electrochemical capacity but improved thermal stability.

It will be helpful for improving thermal stability to properly increase the particle sizes and reduce the specific surface area of cathode material. Of course, single crystal materials naturally have advantage in stability due to decreased surface area and surface defects.

Suitable doping and coating can also be adopted to minimize the exothermic reaction of the delithiated cathode material, which triggers the “thermal runaway” of LIBs.

3. Typical application-oriented specifications of NCM/NCA cathode materials

Lithium ion batteries are extensively used in consumer electronics, electrical vehicles, and energy storage system. All these applications require long lifespan, good reliability and safety, although the target levels of the performance may be different from each other. Battery electric vehicle (BEV) prefer energy type batteries, hybrid electric vehicles (HEV) or 48V regeneration system need power type batteries, while plug-in hybrid electric vehicles (PHEV) need batteries with both mild energy density and power density. Accordingly, the energy type materials, high-rate type materials should be developed. Here are some examples of NCM523 based cathode materials for different application (Table 2 and Table 3). For automotive battery applications, doping or coating is the necessary modification method for improved reliability of cathode materials.

### Table 2. Agglomerate high Nickel materials (Ni≥60mol%) developed for various applications

| Product name | 6E  | 6E2  | 6E2X | 83E  | 88E  | NCA  |
|--------------|-----|------|------|------|------|------|
| Application  | Power Tool | BEV | HEV | BEV/PHEV | BEV | Power Tool |
| NCM (A) ratio | 60-20-20 | 65-15-20 | 65-15-20 | 83-11-06 | 88-09-03 | 89-09-02 |
| Product type | Unimodal | Unimodal | Unimodal | Bi-modal | Bi-modal | Bi-modal |
| Doping       | Yes | Yes | Yes | Yes | Yes | Yes |
| Coating      | Yes | Yes | Yes | Yes | Yes | Yes |
| D_{50}, \mu m | 10.5 | 7.3 | 4.7 | 9.7 | 11.1 | 14.1 |
| Tap density, g.cm\textsuperscript{-3} | 2.57 | 2.02 | 1.56 | 2.41 | 2.42 | 2.65 |
| Pellet density, g.cm\textsuperscript{-3} | 3.36 | 3.28 | 2.75 | 3.52 | 3.39 | 3.72 |
| Li\textsubscript{2}CO\textsubscript{3}, % | 0.19 | 0.18 | 0.16 | 0.20 | 0.24 | 0.19 |
| LiOH, % | 0.17 | 0.32 | 0.34 | 0.33 | 0.35 | 0.23 |
| 0.1C Ch. cap., mAh.g\textsuperscript{-1} | 201.7 | 207.2 | 207.8 | 234.7 | 237.5 | 240.9 |
| 0.1C Disch. cap., mAh.g\textsuperscript{-1} | 181.3 | 186.4 | 193.1 | 211.3 | 216.7 | 214.4 |
| 0.2C Disch. cap., mAh.g\textsuperscript{-1} | 178.9 | 183.4 | 185.6 | 205.9 | 211.7 | 211.2 |
| 0.5C Disch. cap., mAh.g\textsuperscript{-1} | 172.6 | 176.9 | 179.4 | 196.9 | 202.3 | 203.6 |
| 1.0C Disch. cap., mAh.g\textsuperscript{-1} | 167.1 | 171.8 | 174.2 | 190.4 | 195.7 | 198.0 |
| Cap. retention. (coin cell, 25°C, 1C/1C, 80th @4.4-3.0V), % | 92.8 | 94.8 | 90.3 | 97.1(@4.4-3.0V) | 94.2(@4.4-3.0V) | 94.4(@4.4-3.0V) |

SEM
Table 3. Single crystal high Nickel materials (Ni\geq60mol\%) developed for various applications

| Product name | 60SC | 65SC | 70SC | 83SC | 88SC |
|--------------|------|------|------|------|------|
| Application  | BEV  | BEV  | BEV  | BEV  | BEV  |
| NCM (A) ratio| 60-20-20 | 65-15-20 | 70-10-20 | 83-11-06 | 88-09-03 |
| Product type | Unimodal | Unimodal | Unimodal | Unimodal | Unimodal |
| Doping | Yes | Yes | Yes | Yes | Yes |
| Coating | Yes | Yes | Yes | Yes | Yes |
| D_{50}, \mu m | 3.8 | 3.6 | 4.3 | 3.9 | 4.0 |
| Tap density, g.cm^{-3} | 2.12 | 2.15 | 2.20 | 1.84 | 1.79 |
| Pellet density, g.cm^{-3} | 3.23 | 3.25 | 3.24 | 3.41 | 3.44 |
| Li_2CO_3, % | 0.11 | 0.15 | 0.16 | 0.25 | 0.26 |
| LiOH, % | 0.26 | 0.28 | 0.28 | 0.17 | 0.17 |
| 0.1C Ch. cap., mAh.g^{-1} | 202.4 | 207.9 | 213.3 | 228.8 | 238.1 |
| 0.1C Disch. cap., mAh.g^{-1} | 182.5 | 186.3 | 189.2 | 207.7 | 213.3 |
| 0.2C Disch. cap., mAh.g^{-1} | 178.8 | 183.6 | 185.7 | 203.3 | 208.5 |
| 0.5C Disch. cap., mAh.g^{-1} | 172.4 | 177.5 | 179.3 | 196.6 | 201.7 |
| 1.0C Disch. cap., mAh.g^{-1} | 167.2 | 172.4 | 174.2 | 191.4 | 196.5 |
| Cap. retention. (coin cell, 25\degree C, 1C/1C, 80th @4.5-3.0V), % | 94.0 | 93.5 | 93.0 | 95.1(@4.4-3.0V) | 92.9(@4.4-3.0V) |

4. Conclusion
Ni-based cathode materials have been developed and commercialized in last decade, and the application has extended from consumer electronics to electro-motility, and energy storage system, the cell configuration is diversified from 18650 cylindrical cells of <3.0Ah to large sized prismatic cells and pouch cells of 50Ah level. For small size cylindrical cells, like 18650 or 21700, NCM or NCA of Ni content close to 90% has been largely used, although there still remains the risk of safety issue to some extent, while for large size prismatic cells or pouch cells, NCM of Ni content less than 65% are being used because of more challenging issues, e.g. safety, swelling, even cycle life. NCM materials applied in high-voltage cell are initiated presently. All these applications are attributed to the understanding the relationship of composition-process-structure-performance, and the improvement in the comprehensive properties by optimizing the categories of technology including precursor, sintering, doping, coating, etc.

Obviously, efforts must be made to handle the challenging issues remained for better reliability, especially with respect to the reuse of battery retired from electric vehicles, therefore, cutting edge technologies are inevitably required.

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