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Advantages of gas-phase coatings on NMC battery active materials

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Electrode-electrolyte interface optimization is key for enabling next generation lithium battery technology, as it greatly determines the battery lifetime and performance. An outline is given on the interface instabilities occurring with the most used cathode material $LiNi_xMn_yCo_zO_2$ *(NMC), and mitigation strategies using coating layers are highlighted. A superior coating approach is shown by Powall using gas-phase chemistry. This not only offers an effective industrial scale solution but enables cost reduction by making production overall more efficient.*

The role of single- and poly-crystalline cathode materials in batteries

The most abundantly used cathode materials for electric cars and high energy density batteries are layered metal oxides, especially $LiNi_xMn_yCo_zO_2$ (or NMC, where x+y+z=1). NMCs can store large amounts of lithium at a high voltage, which yields high power and energy output from the battery cell. The surfaces of NMC cathode materials are typically unstable during battery operation, which leads to battery failure. This paper highlights that atomic layer deposition (ALD) surface coatings can stabilize NMC surfaces, enabling long lasting performance of battery cells.

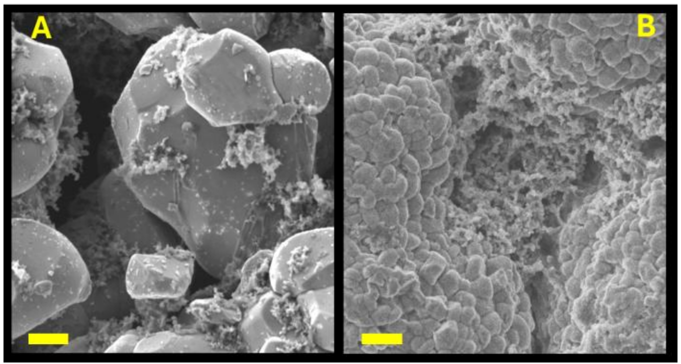

Figure 1 A) Polycrystalline NMC processed in an electrode. B) Single crystalline NMC processed in an electrode. The scale bar indicates 1 μm size.

The NMC cathode material class has many shapes, sizes and compositions. By varying the metal composition of the material, its capacity, operational voltage and stability can be tuned. The particle shape determines some key characteristics as well. Here, two classes of layered metal oxide morphologies can be distinguished. The first is single crystalline (SC) NMC, which has smooth, separate micrometer-sized crystals (Figure 1A). The second class is polycrystalline (PC) NMC, which consists of agglomerate particles formed from smaller crystallites varying from 200 to 600 nanometers (Figure 1B).

Both SC NMC and PC NMC have their own applications. SC NMC is used for long life application, while PC NMC can provide a higher power output and is, therefore, preferred for fast charging.

Nevertheless, both types of NMC suffer from unwanted surface reactivity towards the electrolyte. Coating of the NMC surface is a ubiquitous solution to prevent surface reactivity. The outer surface of NMCs is particularly unstable against electrolytes during high voltage operation. Nickel, when in its 4+ state, may migrate to the lithium-ion position in the crystal. When such event happens near the crystal surface, the migrated nickel may reform the crystal locally which leads to oxygen loss at the surface and reformation of the lattice to a resistive rock salt layer NiO. The oxygen coming from the crystal also causes oxidation of electrolyte at the interface. As a result, the surface forms a resistive interface of reaction products during operation. A logical solution is to apply a protective layer to prevent such surface reformation and electrolyte degradation. In fact, NMCs with protective surface coatings have been shown to exhibit improved high voltage stability and longer battery cycle life (>500 cycles). For SC NMCs some reports even indicate extremely long lifetime (>>1000 cycles) with almost no degradation occurring. The reason is that on SC NMCs the coating remains

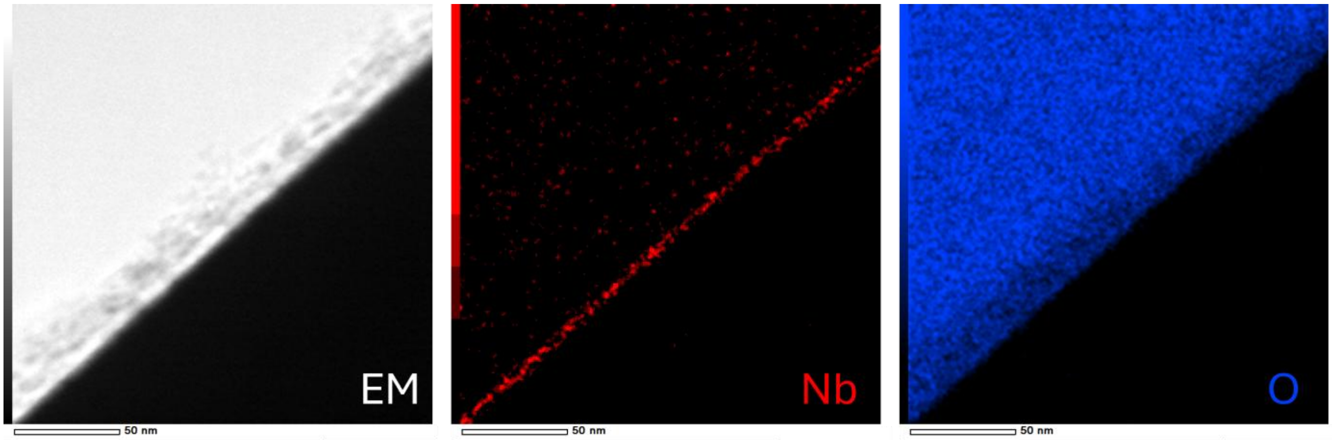

Figure 2 Nanolayered coatings applied by gas-phase coating show a conformal protective layer capable in preventing side reactivity increasing the durability of the materials. The deposition layer shown by TEM was applied at Powall in our continuous powder reactor capable of coating 100 kg/h throughput (named Operio).

as a conformal layer during the entire lifetime of the material, while polycrystalline materials may crack or break during their lifetime exposing new uncoated surfaces and creating new reactive sites.

However, bringing these advances from lab to mass-production scale is not trivial. This white paper highlights three examples of surface coatings which show a significant gain in performance. Such coatings are viable candidates for tonne-scale and cost-efficient production using Powall's continuous gas-phase nanocoating process.

Role of coatings to improve battery performance

Several modes of battery failure exist in NMC battery cells which are related to surface reactivity. For each failure mode, we provide examples of coatings that are effective in mitigating that specific failure and thereby improving battery cycle life.

Mitigating surface reformation

Metal oxide coatings like Al_2O_3, TiO_2 and ZrO_2 are known to reduce CAM particle surface reactivity, suppress lattice cation mixing and improve processability[1–3], but some studies have trouble reproducing the effect in Ni-rich PC NMCs[4]. Due to significant crack formation during cycling, a fresh surface of PC NMCs is continuously exposed which renders the "outer" coated surface less effective in mitigating unwanted reactivity.[5] One key development was the use of "grain-boundary" coatings, which is an interphase layer between the grains of primary particles.[6]

Typically, first the outer secondary particle is coated and then using annealing the coating is diffused into the grain boundary.

In contrast, SC materials show great benefit as in one step the coating continuously covers the entire interface during battery operation[7]. The coating improves surface lattice stability, prevents transition metal dissolution[8] and reduces lithium exchange during aqueous processing[9,10].

The gold standard in battery performance was published by Prof. Dahn's group, which uses a titanium-based coating on SC NMC532. They could show more than 4500 cycles between 3.0 - 4.3 V, maintaining >90% of the original capacity.[11] Key for achieving such performance is complete prevention of transition metal dissolution by having a completely conformal coating[12].

At Powall, the process of coating metal oxide and metal organic coatings from the gas-phase has been developed from lab to industrial scale. Leveraging atomic layer deposition (ALD) chemistry established in the semiconductor industry and integrating it with state-of-the-art powder processing technologies, highly conformal coatings can be achieved even on rough or irregular particles (Figure 2).

Combining such coatings on SC NMC and an optimized electrolyte formulation enables battery cells which may maintain their charge capacity during thousands of cycles[11,13,14]. One main drawback of metal-oxide coatings is that it typically introduces additional internal resistance. This means that only very thin layers below a few nanometers can be used to avoid significant increase in internal resistance and to prevent degraded rate-performance.

Preventing electrolyte reactivity

In their delithiated (charged) state, most NMC crystals are unstable. An accelerated capacity fade occurs if NMCs are charged to high voltages beyond their H2-H3 phase transition threshold (which varies per Ni-content)[15]. In this state, oxygen loss causes a cascade of reactions like oxidation of solvents such as ethylene carbonate (EC).[16] The lattice stability of the layered structure can be increased by using dopants. Coating layers can change the surface redox activity as well by adding a surface layer[17] or doping the outer shell using a temperature treatment after coating.[18] Surface stabilizing groups, such as fluorides for nickel rich NMC's, can coordinate well with lithium atoms from the lattice.[19]

Another popular class of coatings is based on phosphates. The phosphate group has a high binding strength with oxygen, giving a high voltage stability at the surface of the crystal. Therefore, as protective coatings, these materials provide better protection to catalytic activity at high potentials, effectively shown on LMNO cycled between 3.8-5 V where $FePO_4$ prevents manganese dissolution[20] and on lithium-manganese-rich oxides (LMR) by stabilizing the surface oxygen electronic band structure[17]. Note the analogy with the well-known $LiFePO_4$ CAM.

Ionic and electronically conductive coating

Particularly of interest are coating that are ionically and/or electronically conductive. Such conductive coatings lower the charge transfer resistances in the battery.[21]

Ionically conducting layers become more important now that solid state batteries are emerging. With limited contact points between CAM and solid-electrolyte particles, enhanced surface conductivity ensures a higher portion of the active materials can effectively be used.

Examples of such coatings are lithiated metal oxides and mixed metal oxides. By lowering the charge transfer resistance, the homogeneity of lithiation is improved which relieves stresses and lowers the degree of cracking.[22]

$LiNbO_3$ offers an illustrative case of this approach, showing benefits in both liquid[23] and solid state batteries (SSB)[24]. In SSBs using thiophosphate electrolytes, the coating offers ionic conductivity and prevents severe side reactivity. However, oxygen loss from the coating at high voltages may still lead to unwanted CEI formation, as shown in a system with the thiophosphate argyrodite.[25]

A second promising candidate for conductive coatings is $Li_4Ti_5O_{12}$ (LTO), as it has shown cycling performance improvement when deposited on NMC811[26,27]. The LTO layer lowered the charge transfer resistance and reduced the capacity fade to 2% after 170 cycles, indicating the interface shows a stabilizing effected[28]. In solid state batteries LTO coatings may show a similar benefit[29].

Borates and silica[30] also show both protective and capacitive benefits[31] as they offer a protective amorphous surface which allows for an improved lithiation distribution.

The three coating functions named above show that several options exist to improve the NMC active material. An ideal coating should do all three: stabilize the interface between active material and electrolyte, allow ion and electronic conduction and prevent surface reformation. The combination of coating, active material and electrolyte is critical, as some coating layers have shown specific catalytic activity, are too rigid, have a lattice mismatch with the active material or might not fit the potential range intended for operation. Other coatings may need more costly precursors. For this reason, it is hard to pinpoint a 'one-coating-fits-all' strategy. Therefore, at Powall we use a 'fit-for-purpose' strategy where we consider the key specifications of the intended application before suggesting and applying a solution.

Powall's gas-phase powder coating technique

State-of-the-art coating methods

Comparing wet chemical methods to ALD for coating battery materials, ALD shows significant advantages.[32] ALD allows for the deposition of ultra-thin layers with Angstrom-level thickness control, minimizing the resistivity of the layer.

Powall has adapted the ALD process to create a scalable and cost-efficient gas-phase process (Figure 3). Powall's –beyond ALD– process uses a tubular

Figure 3 Powall's powder ALD equipment can continuously process CAM powder at a high throughput.

pneumatic transport reactor operating near atmospheric pressure. The need for purging and wasting of precursors is removed, which significantly reduces the cost of coating powders compared to conventional ALD and even other continuous techniques. The process is scalable by adjusting the tube diameter in line with normal powder transport techniques.

Additionally, post-processing is not required for the coated materials, an advantage compared to other coating methods which need post-drying. The elegant design and effective utility of chemicals makes powder coating processing using Powall equipment cost competitive.

In a laboratory environment, ALD is a technique which typically uses vacuum and generous dosing and purging times. Therefore, ALD is thought of as an expensive technique which is not scalable.

Powall has adapted the ALD process to create a scalable and cost-efficient gas-phase process.

By this technique, Powall is able to deposit conformal layers on various battery active materials (such as the ones listed in **Table *1***) under mild temperatures, leaving the inner structure of the active materials largely intact[33]. An ALD approach, therefore, results in minimal loss of capacity and rate, with the possibility to adjust surface properties without affecting other bulk characteristics. This platform technology enables the next generation battery chemistries with superior performance at relevant production scales.

Table 1 The ALD technique can deposit a wide variety of coatings on battery materials. Here are a few examples of effective CAM coatings

Class	**Type**	**Deposited on**	
Oxides	Al_2O_3	SC NMC622	7
	TiO_2	LNMO	34
	ZrO_2	SC NMC532	35
	Nb_2O_5	SC NMC532	36
Phosphates	$FePO_4$	LNMO	20
	$AlPO_4$	SC LMR	37
	Li_3PO_4	LMNO	38
	$LiZrPO_4$	NMC811	39
Borates	B_2O_3	SC NMC83	40
	$Li_2B_4O_7$	LMO	41
Lithium containing metal oxides	LTO	NMC811	27
	$LiNbO_3$	SC NMC83	42
Fluorides	$LiAlF_4$	PC NMC811	43
	LiF	LMNO	44

Contact

Have you become excited about our technology?

More information on our ALD technique, other applications for our coatings and services we provide can be found on our website https://powall.com/. Do you want be in contact? E-mail to info@powall.com to inquire about projects, our services and coating equipment.

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