

Lithium-ion Battery High-Energy Cathode Innovation & Patent Review

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• Easpring / Dangsheng Technology - China	
• Ecopro / Ecopro BM - Korea	
• GEM / Gelinmei Wuxi / Jingmen Green Eco-Manufacture / Fuan Qingmei Energy Materials / EcoPro GEM - China	
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• Huayou Cobalt / Huahai / B&M Science and Technology / Bamo Technology - China	
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- [LG Energy Solution / LG Chemical - Korea](#)
- [Nano One - Canada](#)
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- [Reshine - China](#)
- [Ronbay / Rongbai / SKLD Technology / Tianjin Sikelande Technology - China](#)
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- [Sumitomo Chemical / Tanaka Chemical - Japan](#)
- [Sumitomo Metal Mining - Japan](#)
- [SVOLT Energy / Fengchao Energy Technology / Honeycomb Energy Technology - China](#)
- [Tesla - USA](#)
- [Toyota Group / Primearth EV Energy / Prime Planet Energy & Solutions - Japan](#)
- [UChicago Argonne LLC - USA](#)
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About the Author

Pirmin Ulmann obtained a diploma in chemistry from ETH Zurich (Switzerland) in 2004 and a PhD from Northwestern University (USA) in 2009. Thereafter, he was a JSPS Foreign Fellow in an ERATO academic-industrial project at the University of Tokyo (Japan). From 2010 to 2016, while working at a major battery materials manufacturer in Switzerland, he was a co-inventor of 7 patent families related to lithium-ion batteries. He was also in charge of a collaboration with the Paul Scherrer Institute, evaluated outside technologies for corporate strategy, and made customer visits to battery manufacturers in East Asia, North America & Europe. He holds the credential Stanford Certified Project Manager (SCPM) and has co-authored scientific articles with more than 1,800 citations.

Balancing Raw Materials Costs – Ni / Mn-containing vs. Fe / Mn-containing Active Materials

Ni / Mn-containing positive electrode (cathode) active materials (such as NMC, NMCA, Co-free NMx) typically account for >50% of overall Li-ion battery cell materials costs. For this reason, Li-ion battery cell manufacturers are continuously evaluating if a replacement with lower cost positive electrode active materials is feasible:

- by relying on lower-cost raw materials, such as Fe & Mn (price information [Ni](#) / [Li](#) / [Co](#) / [Mn](#) / [Fe](#)),
- or through reduced manufacturing process costs ([see below](#)).

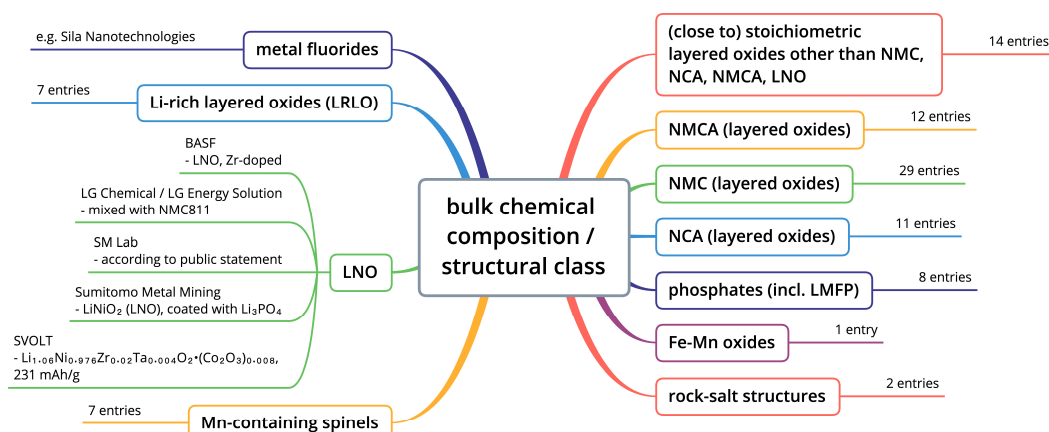
Recent developments (within the frame of liquid electrolyte Li-ion battery cells) suggest that two positive electrode material technology tracks will split up most electrified transport applications between them:

- cells with **Ni-dominant positive electrode active materials**, 90 to >99 mass% Ni (in relation to the total transition metal content), theoretical limit for LNO (LiNiO_2): 275 mAh/g reversible capacity, 3.8 V vs. Li^+/Li average potential.
- cells with **iron-based LFP (LiFePO_4) positive electrode active materials**, theoretical limit: 170 mAh/g, 3.43 V vs. Li^+/Li average potential. These materials will evolve towards **iron / manganese-based LMFP with increasing Mn content**, theoretical limit for LMP (LiMnPO_4): 170 mAh/g, 4.1 V vs. Li^+/Li .

Decision Tree for High-Energy Cathodes

In Figures 2-7, R&D decisions by various companies are plotted, as reflected in the patent literature. The emphasis is on illustrating the commercially most relevant and promising options, without claiming comprehensiveness as to capturing all applicants active in a certain area. With few exceptions, only new patent families published since 2019 have been included (focus: new patent families published since 2021). It is therefore possible that earlier patents that constitute ‘foundational IP’ are not shown.

Figure 4: decision tree - bulk chemical composition / structural class (full review contains all entries)



- **LNO (stoichiometric, layered LiNiO_2):** LNO has very quickly expanded from the academic domain to being a very hot topic of commercially oriented product development. Doping (BASF - Zr, SVOLT - Zr / Ta / Co), coating (Sumitomo Metal Mining - Li_3PO_4), mixing with NMC (LG Chemical / LG Energy Solution) are strategies pursued to stabilize LNO towards sufficient cycling stability and 1st cycle efficiency. SM Lab's approach to LNO has not yet been published in the patent literature.

Because the theoretical energy density limits of LNO are very favorable (275 mAh/g reversible capacity, 3.8 V vs. Li^+/Li average potential) without the need for overlithiation (risk of gassing, accelerated aging at high voltages as described above for LRLO), LNO will continue to represent a highly attractive product development target for current manufacturers of stoichiometric layered oxides and other players that wish to develop positive electrode active materials that can be deployed in liquid, semi-solid and solid Li-ion battery cells (i.e., unlike overlithiated LRLO and Mn-rich spinels, the market success of LNO likely does not hinge on the successful establishment of solid catholyte / solid electrolyte supply chains).

AI-based Identification of Commercially Relevant Patents

b-science.net has developed a supervised AI methodology to assess the commercial relevance of patents, combined with an automatic translation framework that makes sure Non-English patents are also identified. The methodology was validated as shown in the Appendix. With this approach, we have comprehensively identified & classified patents by companies active in commercial R&D on high-energy Li-ion battery positive electrodes.

Table 1: Number of commercially relevant Li-ion battery high-energy positive electrode patent families between January 2019 and July 27th, 2022 (publication of earliest family member)

Company	Country	2019	2020	2021	2022 (until Jul 27)	Total (2020-2022)	Joint patent filings / JVs or technical partnerships / licenses / patent transfers
<u>LG Energy Solution / LG Chemical</u>	Korea	156	87	106	105	298	- <u>3M / Umicore</u> - <u>BASF Toda</u> - <u>B&M</u> - GS Energy - <u>UChicago Argonne</u>

305 additional companies are listed in the full review.

Focus of This Review

This review focuses on the global innovation & patenting activity by companies in Li-ion battery high-energy positive electrode active materials for large scale, high-energy applications (key application: EVs). Patent family publication dates (first / earliest family member) between January 2019 and November 2022 were considered and were classified into 5 categories: **A)** bulk properties; **B)** particle microstructure, composites, gradients; **C)** surfaces; **D)** manufacturing, reliability & safety; **E)** positive electrodes; **F)** active materials produced through recycling.

Each listed patent family has been labeled according to its active material category: Li-rich layered oxides - (LRLO, 10% or more overlithiation), Mn-containing spinels, LNO, (close to stoichiometric layered oxides other than NMC, NCA, NMCA, LNO (up to 10% overlithiation), NMCA, NCA, NMC, phosphates, Fe-Mn oxides, rock-salt structures. If applicable, listed patent families are flagged with 'possible use for commercial products' (if an elevated likelihood is seen by the author towards commercialization). The goal is that the reader understands which product development topics are of highest commercial interest to the industry, and how the high-energy positive electrode active materials supply chain will prospectively evolve in the coming years.

Assessment of Companies

Author comments are displayed in **maroon color**.

Contemporary Amperex Technology (CATL) / BRUNP Recycling / Dynanonic - China

Organization profile

Contemporary Amperex Technology Limited (CATL, <https://www.catl.com/en/>) is the largest Li-ion battery producer. CATL was founded in 2011 in Ningde, China. In 2017, CATL completed the split from its parent company ATL/TDK. With BRUNP Recycling (subsidiary), and Dynanonic (publicly traded, >60% floating stock), CATL jointly develops positive electrode active materials.

In addition to its own production capacity, CATL likely continues to source positive electrode active materials from multiple NMC suppliers in China.

Unique capability: high-nickel positive electrode formulations based on single-crystal and polycrystalline NMC811, plus leading position in LMFP (presumably with 3 mol% Mg content).

Leap of faith: commercialization of Na-ion batteries (requires specialized positive electrode active materials, not covered in this review), while maintaining lead in Li-ion batteries.

Possible future commercial high-energy positive electrode materials

NMC

- NMC811 and beyond.

- doped with Al, Zr and/or W.
- slight oxidation of NMC hydroxide precursor with a controlled amount of oxygen gas, introduced as micro-bubbles (average particle size of 0.2-4.0 μm).
- coated with aluminum oxide (Al_2O_3) / boron oxide (presumably B_2O_3).
- combination of single-crystal ($D_{50} = 10 \mu\text{m}$) and polycrystalline NMC811 ($D_{50} = 3 \mu\text{m}$), approximately 3 : 7 by mass.

LMFP (Figure 19)

- $\text{LiMn}_{0.6}\text{Fe}_{0.37}\text{Mg}_{0.03}\text{PO}_4$.
- well-defined particle size (average diameter about 500 nm), obtained upon MPCVD treatment (5 min).
- amorphous carbon coating.

Electrochemically inactive components

- conductive additives: carbon black (IMERYS) and acetylene black.
- **filler: alumina (Al_2O_3).**
- binder: PVDF.

Figure 19: projected process for carbon-coated $\text{LiMn}_{0.6}\text{Fe}_{0.37}\text{Mg}_{0.03}\text{PO}_4$ (BRUNP / CATL)

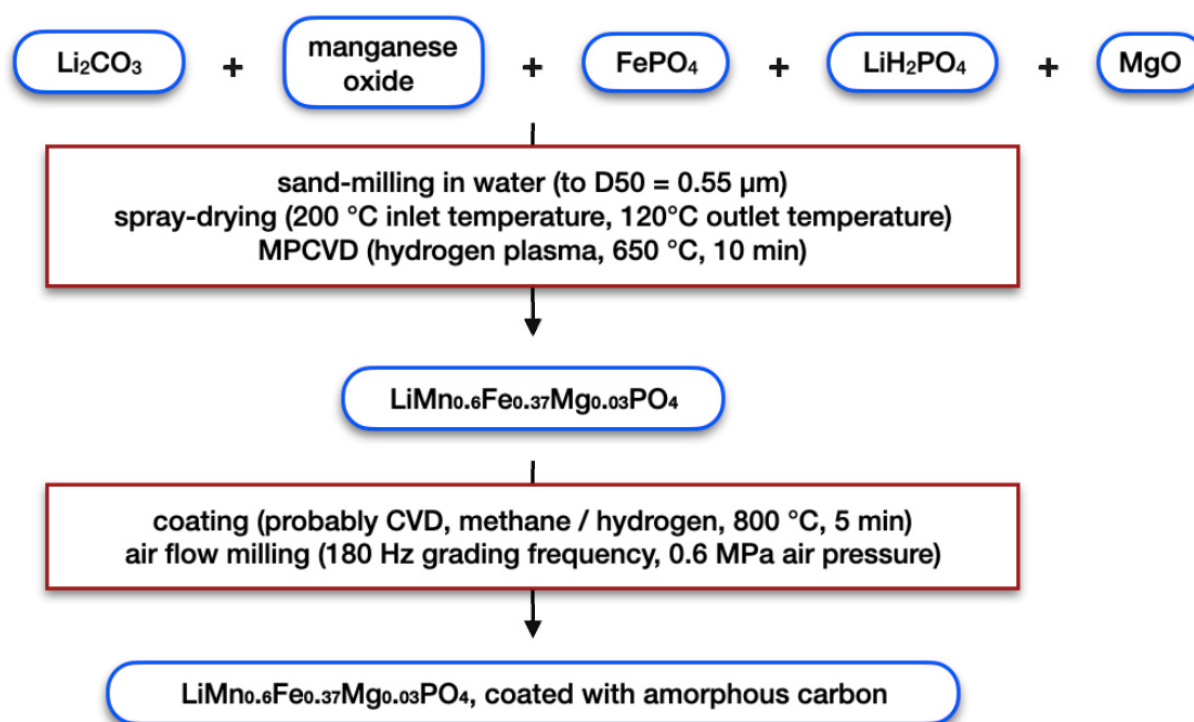
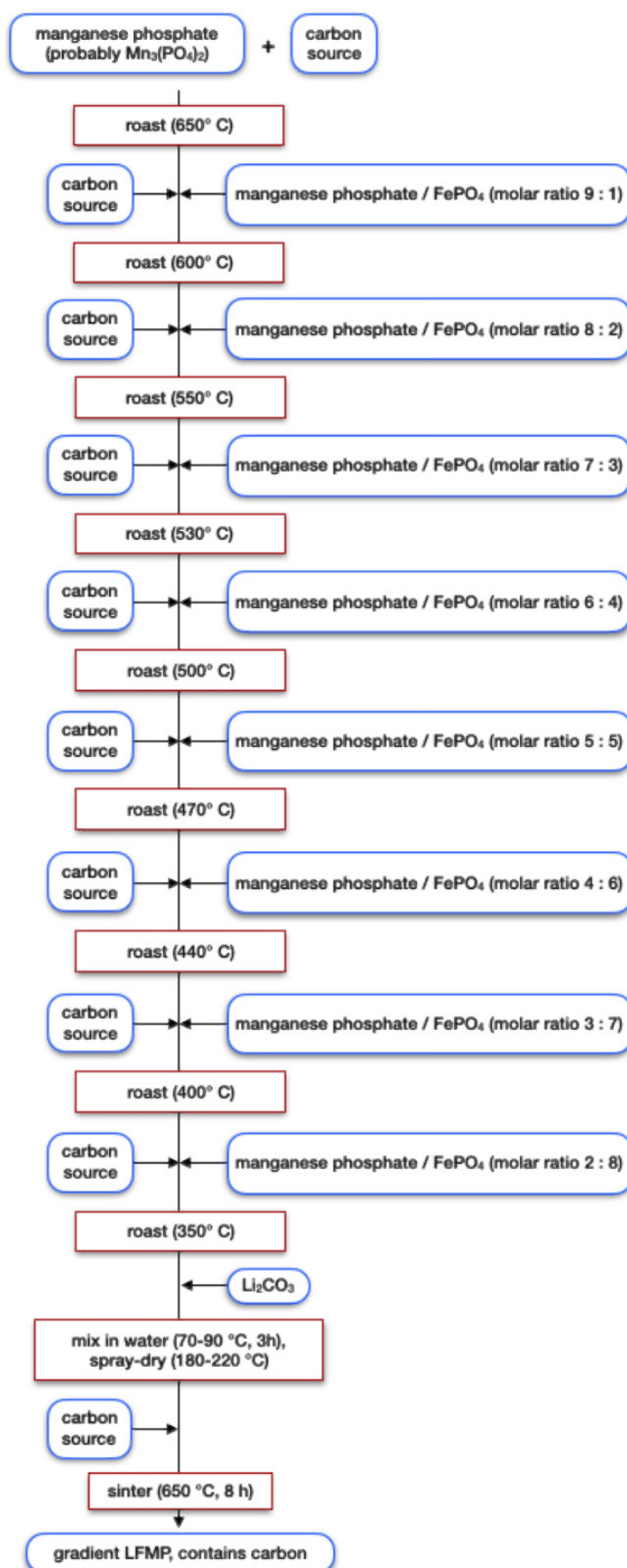


Figure 20: projected process for carbon-containing, gradient LMFP (Dyananonic)



News reports and press releases

In December 2022, it was reported that CALB lost a battery patent infringement lawsuit brought by CATL (apparently related to a current-collector patent).

In October 2022, it was announced that CATL targets mass production of sodium-ion batteries in 2023.

In September 2022, it was reported that Dynanonic's 110 kton/year LMFP manufacturing operation has been put into operation.

In September 2022, CATL introduced the swappable MTB (module to bracket) technology for heavy-duty electric trucks.

A news article from September 2022 describes CATL's battery swapping business in China and overseas.

A news article from August 2022 describes CATL's M3P batteries, which are based on LMFP. LMFP in turn is supplied by Shenzhen Dynanonic, which plans to put 110 ktons of (presumably annual) capacity into production in H2/2022. Two types of LMFP have been developed, without dopants and with aluminum, magnesium or other dopants. **Tesla has denied that it will use M3P / LMFP cells by CATL.** CATL stated that M3P batteries are based on a "ternary lithium battery of the phosphate chemistry system", and that 10-20% higher energy density can be reached as compared to LFP-based batteries.

In August 2022, it was reported that CATL again sued CALB (7th-largest EV Li-ion battery producer) for alleged patent infringement (the first lawsuit by CATL against CALB was filed in 2021).

In June 2022, CATL presented its third-generation cell-to-pack (CTP) battery technology (planned production start in 2023), labelled Quilin. The NMC version of these battery packs reaches 250 Wh/kg, the LFP version 160 Wh/kg (67% volumetric utilization efficiency of pack). The LFP pack exhibits 290 Wh/L and that the NMC pack exhibits 450 Wh/L volumetric energy density, respectively.

In December 2021, it was reported that CATL and BRUNP builds a positive electrode active material plant with with 220 ktons/year LFP capacity and 180 ktons/year NMC precursor capacity.

In September 2021, CATL signed an agreement with BASF including on positive electrode active materials in the context of establishing a sustainable battery value chain.

Insights from recently published patent filings

It makes sense that the deployment of a short (5 min) MPCVD (650 °C) procedure after spray-drying results in a unique nanoscale powder to which a uniform carbon coating can be applied. The nano-scale nature of the powder might also help with keeping the favorable crystal structure (in terms of Li-ion diffusion) and nature of the carbon coating intact upon extensive cycling.

With regards to NMC, multiple doping and coating approaches have been evaluated. It appears that the combination of aluminum and boron in coatings might offer benefits as compared to aluminum oxide coatings.

Earlier technical information that remains relevant

The combination of single-crystal (large, slightly anisotropic particles) & polycrystalline (small, spherical particles) NMC in positive electrodes leads to an optimal balance between energy density & cycling stability.

To improve safety, alumina is used as a filler in positive electrode formulations.

General patent portfolio characteristics

246 new patent families by CATL / BRUNP / Dynanonic related to Li-ion battery high-energy positive electrodes were published between 2020 & July 27th, 2022. BRUNP Recycling holds a license from BASF for Argonne NMC patents.

Examples from the patent portfolio

A) Bulk properties

- **Phosphates (LMFP) - possible use for commercial products - process in Figure 19:** Preparation method and application of a kind of cathode material (Google, BRUNP): manganese oxide, ferric phosphate, lithium dihydrogen phosphate, lithium carbonate and magnesium oxide were mixed in water and sand-milled (to a particle size D50 of 0.55 µm), followed by centrifugal spray-drying (200 °C inlet temperature, 120 °C outlet temperature), followed by an MPCVD (microwave plasma assisted chemical vapor deposition) treatment (hydrogen plasma, 650 °C, 10 min), which resulted in $\text{LiMn}_{0.6}\text{Fe}_{0.37}\text{Mg}_{0.03}\text{PO}_4$ powder. This powder was carbon-coated (probably CVD) with methane / hydrogen gas (800 °C, 5 min), followed by air flow milling (180 Hz grading frequency, 0.6 MPa air pressure) to obtain **$\text{LiMn}_{0.6}\text{Fe}_{0.37}\text{Mg}_{0.03}\text{PO}_4$ coated with amorphous carbon.**

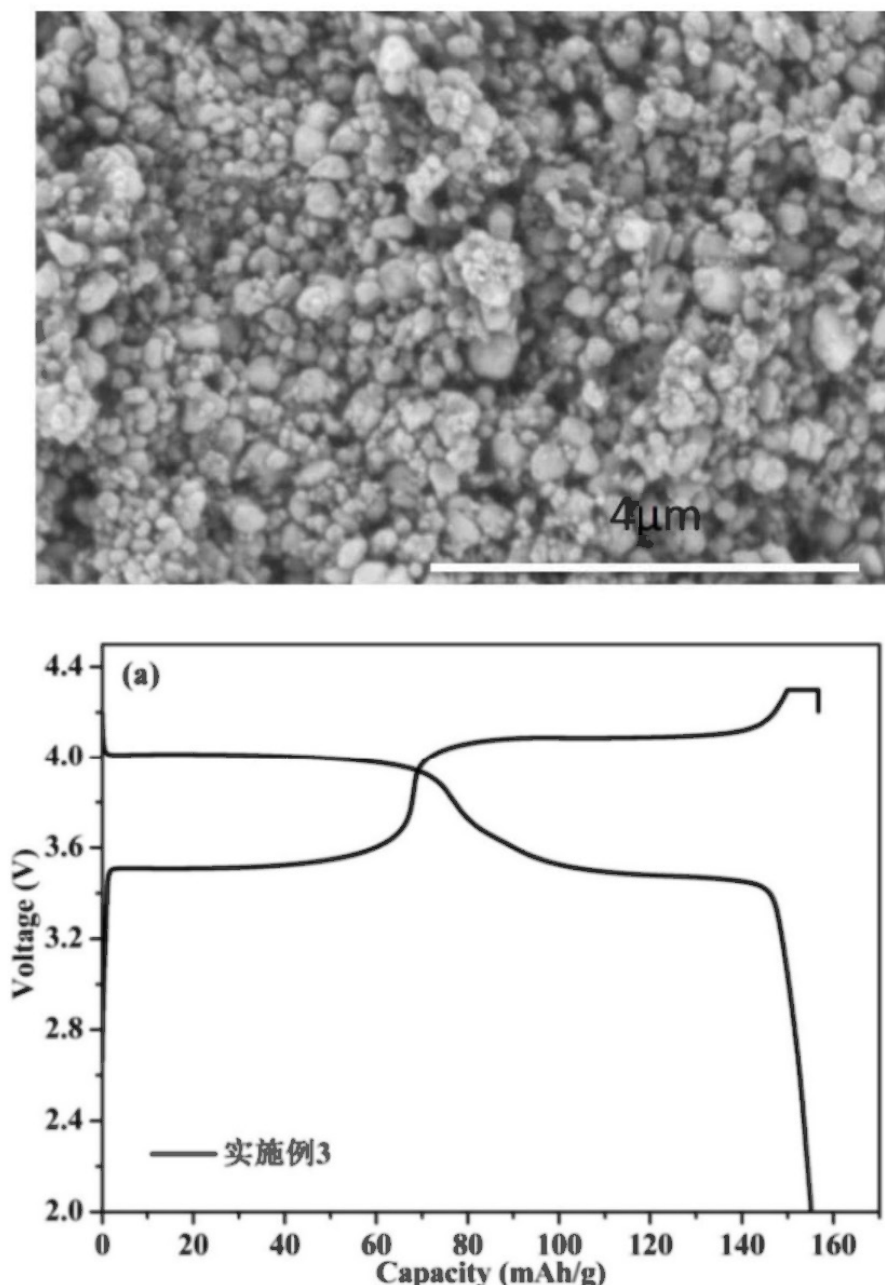
It is emphasized how MPCVD allows for uniform nano-scale particle formation (Figure 21, top), which is beneficial for shortening the Li-ion migration path during the de-intercalation process. It is explained that introduction of the Mg dopant lengthens the Li-O bond lengths in the olivine crystallites, allowing for easier Li-ion diffusion.

This material exhibits a discharge capacity of 155 mAh/g (0.1 C discharge) / 141 mAh/g (1 C discharge), voltage window 2-4.3 V vs. Li^+/Li (Figure 21, bottom).

This work is consistent with CATL's statement that the positive electrode active material in its

new M3P battery can be labeled “ternary lithium battery of the phosphate chemistry system”.

Figure 21: SEM image (top) and voltage capacity diagram (bottom) of carbon-coated $\text{LiMn}_{0.6}\text{Fe}_{0.37}\text{Mg}_{0.03}\text{PO}_4$ (BRUNP / CATL)



- **Phosphates (LMFP):** [Preparation method of ammonium iron manganese phosphate, lithium iron manganese phosphate and application thereof \(Google, BRUNP\)](#): a mixed metal sulfate solution (solution A, 0.5 mol/L, **molar ratio Fe / Mn = 1 : 1**) and an ammonium dihydrogen phosphate solution (solution B, 0.5 mol/L) were separately mixed with an organic solution (2 mass% SDBS surfactant in cyclohexane / n-butanol = 8 : 1 by volume, all steps under nitrogen). For both solutions, 1 volume% of the organic solution was combined with the aqueous solution. Ammonia water (solution C, 8.0 mol/L) was added to solution B until a pH

of 8.5 was reached. Solutions A, B, C were concurrently combined with each other (pH = 8, 30 °C, stop upon reaching a D50 particle size of 5 µm). A solid-liquid separation step was carried out, followed by washing with deionized water and ethanol to obtain ferric manganese ammonium phosphate. After crushing, the material was spray-dried (20 mass% solid content in water) in combination with cane sugar (0.3 equivalents with respect to Fe + Mn) and lithium carbonate (1 equivalent with respect to Fe + Mn), followed by carbonization (600 °C, 20 h) to obtain LMFP active material.

It is claimed that this manufacturing approach leads to a homogeneous distribution of iron and manganese and therefore to favorable performance.

This material exhibits a reversible capacity of **143 mAh/g** / 381 mAh/cm³ (0.2 C discharge, 2.2–4.3 V vs. Li⁺/Li) due to a comparably high density of 2.66 g/cm³.

This work could allow for improved volumetric energy density with LMFP.

- **NMC - possible use for commercial products:** [Single-crystal ternary positive electrode material and preparation method thereof \(Google, BRUNP\)](#): procedure for manufacturing of **single-crystal NMC (Ni / Mn / Co = 0.65 : 0.2 : 0.15), doped with Zr & W** (precursors: ZrO₂ and WO₂). The NMC material is obtained through co-precipitation/sintering with lithium carbonate (up to 900 °C) and is then treated with Zr(OC₃H₇)₄ in propanol to obtain a gel, which is treated with a secondary sintering process (2 °C per min temperature increase to 500 °C, decrease to 300 °C at 1 °C per min, followed by natural cooling to room temperature).

This work illustrates how a secondary treatment of NMC with Zr(OC₃H₇)₄ in propanol followed by sintering leads to single-crystal NMC with improved capacity and cycling stability.

B) Particle microstructure, composites, gradients

- **Phosphates (LMFP) - process in Figure 20:** [Polyphosphate positive electrode material, preparation method thereof, and secondary battery \(Google, Dynanonic, covered in patent update\)](#): **gradient LMFP** particles were prepared in a multi-step procedure:
 1. mix manganese phosphate with a carbon source, roast at 650 °C
 2. mix the prior material with manganese phosphate / iron phosphate (molar ratio 9 : 1) and a carbon source, roast at 600 °C
 3. mix the prior material with manganese phosphate / iron phosphate (molar ratio 8 : 2) and a carbon source, roast at 550 °C
 4. mix the prior material with manganese phosphate / iron phosphate (molar ratio 7 : 3) and a carbon source, roast at 530 °C
 5. mix the prior material with manganese phosphate / iron phosphate (molar ratio 6 : 4) and a carbon source, roast at 500 °C
 6. mix the prior material with manganese phosphate / iron phosphate (molar ratio 5 : 5) and a carbon source, roast at 470 °C
 7. mix the prior material with manganese phosphate / iron phosphate (molar ratio 4 : 6) and a carbon source, roast at 440 °C
 8. mix the prior material with manganese phosphate / iron phosphate (molar ratio 3 : 7) and a carbon source, roast at 400 °C
 9. mix the prior material with manganese phosphate / iron phosphate (molar ratio 2 : 8) and a carbon source, roast at 350 °C

10. mix with lithium carbonate solution in water (70-90 °C, 3 h)
11. spray-dry (180-220 °C)
12. mix with carbon source
13. sinter (650 °C, 8 h)

This material exhibits a reversible capacity of **160.2 mAh/g**.

This work illustrates an approach towards gradient LMFP particles supported by a carbon matrix. No voltage was reported (lithium manganese phosphate exhibits a voltage of 4.1 V vs. Li/Li⁺).

- **NMC: POSITIVE ACTIVE MATERIAL PRECURSOR, PREPARATION METHOD THEREFOR, POSITIVE ACTIVE MATERIAL, LITHIUM ION SECONDARY BATTERY AND APPARATUS** (Google, CATL): **NMC811** in which the density increases from the inside to the outside of the particle. This density gradient is achieved through a linear decrease of the pH by 0.02 h⁻¹ (starting at a pH of 11.7-11.75) and by varying the ammonia concentration during the co-precipitation procedure, until a D50 of 11 µm is reached for the NMC811 precursor.
This work could allow for improved cycling stability/improved resistance against crack formation during cycling.

C) Surfaces

- **NMC - possible use for commercial products: Positive pole piece for secondary battery, secondary battery, battery module, battery pack and device** (Google, CATL): **polycrystalline and single-crystal NMC811** were each mixed with **Al₂O₃** (0.2 mass%) and **boric acid** (0.2 mass%), followed by sintering (5 h, 500 °C).
The coated polycrystalline and single-crystal NMC811 were mixed (7 : 3 by mass) prior to the formation of positive electrodes. The resulting mixture exhibits a favorable tap density of 3.58 g/cm³.
Full cell (graphite-based negative electrodes) cycling tests exhibit 94.1% capacity retention after 400 cycles (1 C) at 45 °C.
This work illustrates how an active material mixture that consists of polycrystalline and single-crystal NMC with an Al/B-based coating leads to favorable cycling stability and volumetric energy density.
- **NMC: Composite coated Ni65 type nickel-cobalt-manganese ternary positive electrode material as well as preparation method and application thereof** (Google, BRUNP): coating of **NMC (Ni / Mn / Co = 0.65 : 0.3 : 0.05) with AlPO₄-11 molecular sieves** and **LiAl_{0.3}Ti_{1.7}(PO₄)₃** solid electrolyte to facilitate Li-ion diffusion while preventing transition metal leaching.
This work could allow for improved cycling stability with Mn-rich cathode materials.

D) Manufacturing, reliability & safety

- **NMC - possible use for commercial products: Method for preparing ternary precursor through microbubble pre-oxidation and application of ternary precursor** (Google, BRUNP): slight oxidation of NMC hydroxide precursor (to slightly increase trivalent metal content, decrease cation mixing) was carried out with a small amount of oxygen gas instead of potassium persulfate, leading to improved product purity. A key aspect is that a micro-bubble (average particle size of 0.2-4.0 µm) generator was used to introduce oxygen into the co-precipitation reaction vessel.
This work could allow for improved product purity and selectivity for partial NMC hydroxide precursor oxidation.

E) Positive electrodes

- NMC - possible use for commercial products:** [A BATTERY](#) (Google, CATL, covered in patent update): an electrode formulation that includes **alumina filler** was developed to improve performance in the puncture test while maintaining cycle life close to cells with lower alumina content that partially failed the puncture test (puncture with 5-10 mm steel nails, check for indication of burning or explosion, see Table 2).

This work could allow for improved safety.

Table 2: top: positive electrode compositions; bottom: cell puncture test & electrochemical results (CATL)

	Current collector	The second positive active material	Composition of the underlying positive active material layer							Adhesion force between the positive electrode film layer and current collector (N/m)
			The first polymer material		The first conductive material		Other inorganic filler		Thickness of the underlying layer H (μm)	
			material	wt%	material	wt%	material	wt%		
Comp. Plate 2-1	Al foil	NCM811	PVDF	75	SP	20	Alumina	5	8	160
Plate 2-2	Al foil	NCM811	PVDF	75	SP	15	Alumina	10	8	170
Plate 2-3	Al foil	NCM811	PVDF	75	SP	10	Alumina	15	8	180
Plate 2-4	Al foil	NCM811	PVDF	60	SP	10	Alumina	30	8	135
Plate 2-5	Al foil	NCM811	PVDF	60	SP	8	Alumina	32	8	140
Plate 2-6	Al foil	NCM811	PVDF	55	SP	15	Alumina	30	8	120
Plate 2-7	Al foil	NCM811	PVDF	50	SP	25	Alumina	25	8	110
Plate 2-8	Al foil	NCM811	PVDF	40	SP	15	Alumina	45	8	95
Plate 2-9	Al foil	NCM811	PVDF	35	SP	5	Alumina	60	8	75
Comp. Plate 2-10	Al foil	NCM811	PVDF	25	SP	5	Alumina	70	8	50

Battery	Positive electrode	Negative electrode	Puncture Test	Cycle Life (cycle)
Battery 6	Comp. Plate 2-1	CPlate N	5 fail, 5 pass	2502
Battery 7	Plate 2-2	CPlate N	10 pass	2351
Battery 8	Plate 2-3	CPlate N	10 pass	2205
Battery 9	Plate 2-4	CPlate N	10 pass	2251
Battery 10	Plate 2-5	CPlate N	10 pass	2000
Battery 11	Plate 2-6	CPlate N	10 pass	2408
Battery 12	Plate 2-7	CPlate N	10 pass	2707
Battery 13	Plate 2-8	CPlate N	10 pass	2355
Battery 14	Plate 2-9	CPlate N	10 pass	1800
Battery 15	Comp. Plate 2-10	CPlate N	4 fail, 6 pass	1715

Patent Analysis AI Methodology & Validation

The patent information source for this review is the European Patent Office (EPO), which covers patent filings from more than 100 patent offices around the world. >2.4M patent documents are included in the b-science.net database that were published since 1980, which either contain the words 'battery' or 'batteries' in the title or abstract, or were assigned to one of the energy storage-related CPC (cooperative patent classification) or IPC (international patent classification) codes: H01M (batteries & fuel cells) or H01G (capacitors). An AI model was defined for commercially relevant high-energy positive electrodes of Li-ion batteries. Patent documents were grouped into patent families and scored with the AI model. An AI relevancy score cutoff

value of 40 was applied (100: very relevant, 0: not relevant). In this review, patent families published between January 1st, 2019 and July 27th, 2022 were included in statistical [Table 1](#) on patent family publications (publication date of earliest family member). A range of key patent families published between August and November 2022 are also listed and discussed in company chapters of this review, without being part of statistical [Table 1](#).

The **methodology was validated** with patent families by Sumitomo Metal Mining (patent families first published in 2020-2022). 176 patent families by Sumitomo Metal Mining were manually classified as relevant, and 48 patent families were manually classified as not relevant. Of these 176 relevant patent families, 173 exhibit an AI score of >40, while 3 relevant patent families with AI scores of 37, 35, 31 were below the cutoff value of 40 (false-negatives). For 2 patent families, AI scores of 46 & 42 were above the cutoff value even though they were manually classified as not relevant (false-positives).

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