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Lithium-ion battery cell production processes

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Research Team

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CONTENTS

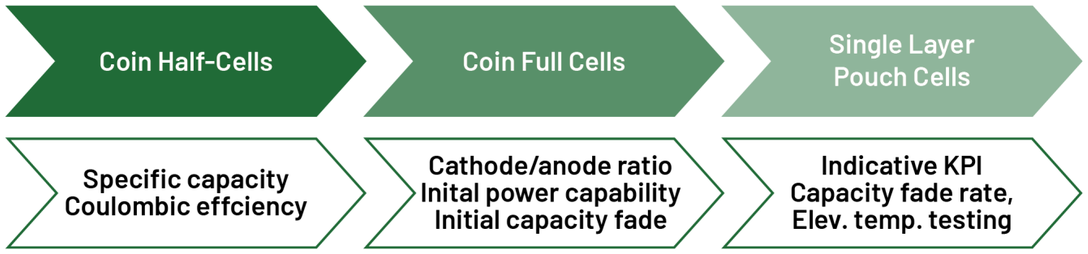
1. INTRODUCTION, PURPOSE AND SCOPE

This document provides an overview of standard cell format trends, cell fabrication and formation equipment as well as best practices established worldwide for Lithium-ion (Li-ion) cell/battery fabrication and formation equipment. The purpose of this report is to fill the knowledge and essential capability gap pertaining to testing and qualification workflow of Li-ion cell materials in reliable, reproducible cell formats that are also representative of industry standards. The target audiences for this report are domestic battery-grade materials suppliers and prospective suppliers as well as members of the emerging Australian battery industries.

Stringent benchmarking and qualification of Li-ion battery materials are critical. This is partially due to the heritage of Li-ion battery technology, which was established through high-end applications such as aerospace and defence. These processes are also critical due to the prominence of Li-ion batteries in the electric mobility market. All the above applications have developed sophisticated supply chains with highly traceable precursors, materials, and components, feeding into rigorous quality control systems for cell/battery makers and system integrators. The main reasons for Li-ion technology dominance in these markets are high specific energy and energy density, relative flexibility in electrode formulations and several viable cell formats. This report will lay foundations for the understanding of major trends in standard cell formats, with special emphasis on cell design and the workflows used in materials qualification.

Coin cells and single-layer pouch cells are key in initial and pilot stage qualifications of materials and components destined for commercial Li-ion batteries. The material qualification workflow usually starts from coin half-cell testing to determine basic properties of the material under validation, i.e., prospective cathode, anode, etc. This includes properties such as specific capacity vs. lithium metal, first cycle coulombic efficiency, etc. Coin half-cell testing is usually followed by coin full-cell evaluation to determine cathode/anode ratio, initial power capability, capacity fade and self-discharge properties. For the long-term life cycling component of material qualification, single layer pouch cells are commonly used to provide the indicative prediction of performance in commercial cells. Data derived from the above qualification workflow is used to select preferred material suppliers, set material performance KPIs and feed into commercial cell designs.

Figure 1: Typical initial/pilot stage qualification workflow for prospective Li-ion battery-grade materials, includes typical information derived from testing at each stage



2. OPERATING PRINCIPLE OF A LI-ION BATTERY CELL

Li-ion cell operation (Figure 2)[1] is based on shuttling lithium ions between cathode and anode electrodes. At each electrode, lithium ions are cyclically inserted (intercalated) into the structures of so called "active materials" via reversible, electrochemical reactions. The anode intercalates lithium ions during the charge step, while ion insertion into the cathode occurs during discharge step. A complete single set of charge and discharge steps are jointly described as a "cycle".

The main constituents of the dry mass of each electrode are referred to as the "active materials" (Table 1). In addition to active materials, each electrode also contains "inactive" constituents that support intercalation:

- Conductive diluents: nano/microscopic carbons, enhancing electronic conductivity within electrodes and facilitating conduction of electrons between electrode and the external electric circuit.
- Polymeric binders: binding electrode coatings to metallic current collectors and providing cohesion between electrode constituents. The exact chemistry and content of binders usually varies between cathodes and anodes.

A "live" Li-ion cell contains an electrolyte solution which acts as a medium for migrating Li-ions and as a wetting agent for electrodes. Electrolytes commonly consist of lithium salt (e.g., lithium hexafluorophosphate, $LiPF_6$) dissolved in a mixture of organic carbonates (e.g., ethylene carbonate and diethyl carbonate). Another important cell component is the polymer separator which provides electrical separation of electrodes while maintaining ionic movement between them.

Figure 2: Operating principle of Li-ion cell, modified after Heimes et al.[1]

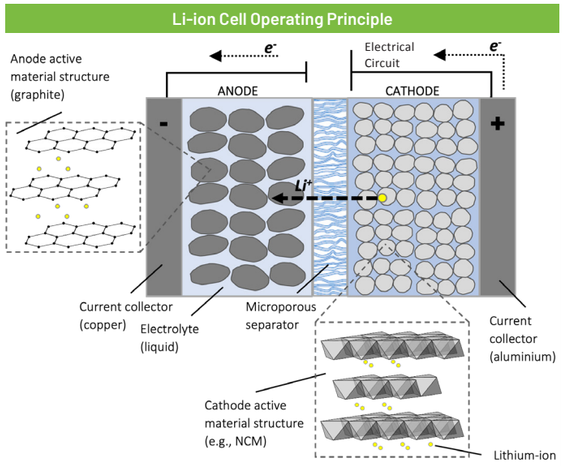


TABLE 1: COMMON SLURRY COMPOSITIONS FOR CATHODE AND ANODE, SOURCE: HEIMES ET AL.[1]

CONSTITUENT	CATHODE	ANODE
Active Material	Various, e.g., NCM or LFP (~90 wt.%)	Graphite (~90 wt.%)
Conductive Diluent	Nano-microscopic carbon, e.g., SuperP® (5 wt.%)	Nano-carbon (5 wt.%)
Binder	PVDF (5 wt.%)	CMC (3 wt.%)
Solvent	N-Methyl-2-Pyrrolidone (NMP)	Deionized water

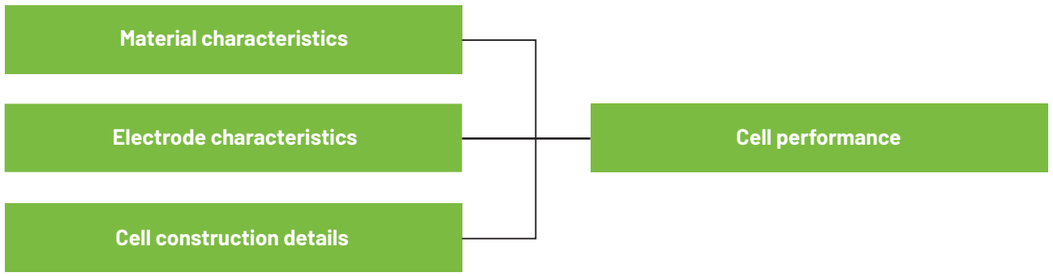
3. MATERIAL AND ELECTRODE CHARACTERISTICS VS CELL PERFORMANCE

Based on their purpose, two prominent types of qualification workflows can be identified:

1. Material qualification workflow; goal: indication of material performance or materials screening; often done in "idealised" cell conditions (e.g., cathode candidate tested vs. lithium metal anode, with thick or double separator, cell flooded with electrolyte).
2. Cell design qualification workflow; goal: indication of cell component or new design performance in "realistic" conditions ("real" N/P ratio, no oversupply of Li-ions or electrolyte).

Regardless of the qualification workflow type, the electrochemical performance of materials and components in Li-ion cell results from several factors acting together (Figure 3).[2]

Figure 3: Cell factors impacting the cell performance



Key factors which impact on cell performance are shown in Tables 2-4; also see Appendix I for relevant standards information. Electrode characteristics and cell construction details will be discussed in the report's sections on cell manufacturing process and its variations.

TABLE 2: CATHODE ACTIVE MATERIAL – PHYSICAL CHARACTERISTICS; OFTEN RELATED TO MECHANICAL PROCESSING

CHARACTERISTIC	DETAILS
Moisture	Content
Particle size	Particle size distribution: D_{min}, D_{10}, D_{50}, D_{90}, D_{99}, D_{max}
Surface area	BET specific surface area
Powder density	Cathode active material – powder tap density

TABLE 3: CATHODE ACTIVE MATERIAL – CHEMICAL CHARACTERISTICS; OFTEN RELATED TO CATHODE'S PRECURSOR AND LITHIATION STEP

CHARACTERISTIC	DETAILS
Formulation	Li/M ratio, transition metal contents
Additives	Contents
Impurities	Contents of magnetic impurities, contents of non-magnetic impurities
Crystal structure	Unit cell parameters, crystallite size, impurity phases, etc.

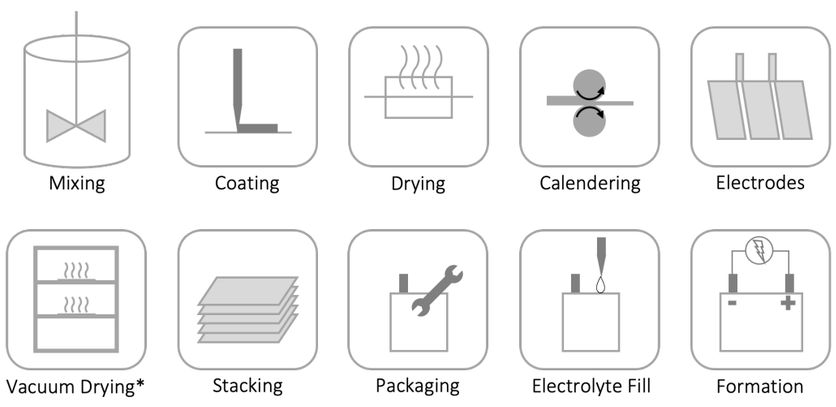
TABLE 4: CATHODE ACTIVE MATERIAL – ELECTROCHEMICAL PERFORMANCE; SUM OF MATERIALS CHARACTERISTICS AND CELL DESIGN FEATURES

CELL FORMAT	PERFORMANCE INDICATORS
Half cell	First charge capacity, first discharge capacity, coulombic efficiency
Full cell	Capacity fade (at 25 °C and/or at elevated temperature)

4. GENERIC LI-ION CELL MANUFACTURING PROCESS FLOW

This section gives a top-level overview of the generic Li-ion cell manufacturing process (Figure 4) and focuses on common parameters and methods, shared between different cell form factors (e.g., coin cells, pouch cells or cylindrical cells). The following sections will describe details of process variations, depending on the specific cell form factor.

Figure 4: Generic Li-ion cell assembly, modified after Liu et al.[3]



*Depending on the manufacturing process details, separation of electrodes is sometimes done after the vacuum drying step.

While Li-ion cells come in a variety of formats, all systems are produced following a standard general procedure of electrode fabrication, cell assembly and electrochemical activation. Each of these categories can be further extended into individual tasks, as depicted in Figure 4. The electrode fabrication steps of slurry mixing, coating, drying, calendering, electrode separation, and vacuum drying are often the most prone to variation between fabrication and testing facilities due to the numerous variables encountered. This is not only considering the physical and electrochemical characteristics of the active materials in question, but also the cost, energy requirements and throughput of the processes at the manufacturing facility.

4.1. Slurry mixing

The first fundamental step in the production of a Li-ion battery cell is the modification of the active material (AM) to a form that can be effectively implemented. To do this, the most common method is to create a slurry mixture consisting of the AM, a carbon additive, and a thermoplastic binder. These components are mixed in a controlled process that determines the distribution of the active material and the microstructure within the electrode. The ratios of these components are extremely important to the overall electrochemical performance of the resultant cell and typically follow the trend of AM = (100 – χ), carbon = (χ/2), and binder = (χ/2). Even for such a seemingly simple process, there are many components that can affect the overall performance of the completed cell. For example, the mixing order of the components alone can alter the cells' ionic conductivity and rate performance.

Typically, prior to being coated onto a metallic current collector, dry constituents of an electrode (cathode or anode) are dispersed within a solvent, forming the electrode slurry (Table 1). Electrode slurry mixing is an important step to achieve a homogeneous blend of active materials, conductive diluent(s) and binder dispersed in a solvent. Effective mixing is critical to produce continuous, smooth, even coatings and to achieve uniform electric current distribution within the electrode. Slurry preparation is a sequential and lengthy process, taking up to several hours.

First, binder is dissolved in a slurry solvent, typically followed by conductive carbon dispersion and finally active material addition. Mixing energy/shear delivered to the slurry at each of these steps is varied depending on powder surface area and tendency to form agglomerates as well as resilience of the mixed material. For example, the particles of cathode material can crack from excessive stirring force. The amount of solvent added also has a significant impact on the physical properties of the slurry, and a deficient amount of solvent can cause the slurry to crack upon drying, lessening the cycle life of the cells. Slurry solvent excess on the other hand can cause a lack of uniformity across the current collector. Furthermore, the ratio of carbon to overall mass of the mixture governs the electrode's surface area and therefore impacts the contact interface with the electrolyte. Consequently, excessive surface area of the electrode can encourage undesirable side reactions with electrolyte.

Depending on the sophistication of the manufacturing facilities, size of the slurry batch and the exact type of active material (e.g., cathode materials with >50 mol% nickel are more moisture-sensitive than cathodes with less nickel in their structure), slurry mixing can be done in clean rooms, under protective gas atmosphere or under vacuum.

Recent advances in slurry mixing include lowering solvent content (solvent recovery is expensive, especially for NMP), dry blending of selected electrode constituents prior to solvent addition and increased use of aqueous slurries (predominantly for anodes, although some research is also being done on aqueous cathode slurries development). Other improvements in slurry mixing are especially focused on mixing equipment to maximise slurry uniformity and solids content while minimising any potential particle damage and shortening the mixing time.

4.2. Electrode coating, continuous drying and calendering

During the coating step, the slurry is applied onto the metallic current collector (copper for the anode and bare or carbon-coated aluminium for the cathode). The exact type of application equipment varies depending on the slurry batch size, solvent content, slurry viscosity, allowable shear, target coating thickness range, desired coating uniformity, etc. The main types of coating technologies used in the Li-ion battery industry discussed here are doctor blade, comma bar and slot die coating.

These coating methods fall into two groups: pre-metered application and roll application. Slot-die coating belongs to the pre-metered application group in which slurry-pumping speed and line speed of a coated substrate determine the coating weight. Roll application involves slurry flow between two rotating rollers, where the gap between two rollers determines the weight (and thickness) of wet coating, for example, in comma bar coating or industrial-scale doctor blade coating. Doctor blade is also used in lab-scale bench-top coatings where slurry is spread directly onto the stationary substrate.

Pilot and industrial-scale coating machines include a continuous drying step directly following the coating stage. Drying removes solvent from the coating deposited onto the substrate using heat. Usually, there are several drying temperature zones and the temperature profile across the drying section of the coater is used to promote efficient and effective solvent removal and to target specific solvents. The solvents are drawn through an exhaust system and the flammable cathode solvent (usually NMP) is recovered or used for thermal recycling.

As-deposited coatings are highly porous. The process of calendering (coating compression between two rotating rollers) is done to decrease coating porosity, increase coating density and improve contact between active material, conductive carbons and current collector. Calendering process parameters such as pressure, roll temperature and number of passes can be tailored to control the electrode's physical attributes such as porosity, surface texture and density. Calendering also increases the materials' adhesion to the current collector, a characteristic that is of particular importance to cells with thickly coated electrodes. From an electrochemical standpoint, the process works to increase conductivity by reducing the space between the conductive carbon chains and reduces the polarisation of the electrodes, especially for those with low ratios of carbon and binder in the slurry.

Not all manufacturers use heated rollers in calendering. However, it has been shown that elevated calendering temperatures result in a lower coating porosity and better adhesion, likely due to the elastic deformity caused by the heating of the binder material (e.g., PVDF).

After the completion of the calendering process, some electrodes are prone to swelling. However, this effect can be reduced by undergoing a two-step calendering process rather than the standard one-step. The two-step calendering process also improves the capacity retention of the overall cell, as demonstrated by Liu et al.[3]

4.3. Electrode separation, vacuum drying and winding or stacking

Electrode separation means slitting, cutting, or die-punching of continuous coatings into desired shapes including: electrode strips (for wound electrodes), electrode sheets (for stacked electrodes) or electrode discs (for coin cells). Once processed, electrodes are typically re-dried under vacuum. Vacuum drying is one of the final stages of component preparation before cell assembly. Industrial vacuum ovens typically operate within the range of 60-150 °C for 12-30 hours at pressures graduating from 0.07 to 1000 mbar. Inert environments such as argon purging can also be applied in combination with vacuum drying.

Vacuum drying is a highly necessary procedure and if not completed correctly, any remnant moisture in the cell can react with the electrolyte salt (e.g., $LiPF_6$), causing a side reaction with the potential to form hydrogen fluoride (HF), a product that tends to damage the AM and cause a plethora of safety risks. HF traces also harm the overall electrochemical performance of the cell. This is of particular concern in case of negative electrodes, as the anode is often made using an aqueous CMC binder-containing slurry, greatly increasing the moisture content within the electrode, potentially driving forward the unwanted reactions.

Careful moisture removal is also key in processing of high-nickel (e.g., NCM-622 or NCM-811) cathode electrodes as electrochemical performance of these materials deteriorates when moisture traces are present. Once water is removed from processed electrodes, cell assembly usually occurs in a humidity-controlled environment (glove box for coin cells and dry rooms for larger cells; see Appendix III for cell fabrication environment specifications).

Separated and redried electrodes are then combined (stacked) into cathode-separator-anode assemblies in:

- wound cells, where the porous separator is wound between the dry anode and cathode electrode, forming a "jelly roll", ready to be inserted into the cell can
- mass-produced electrode assemblies, where electrodes are either stacked between separator sheets or inserted between the z-folded separator layers.

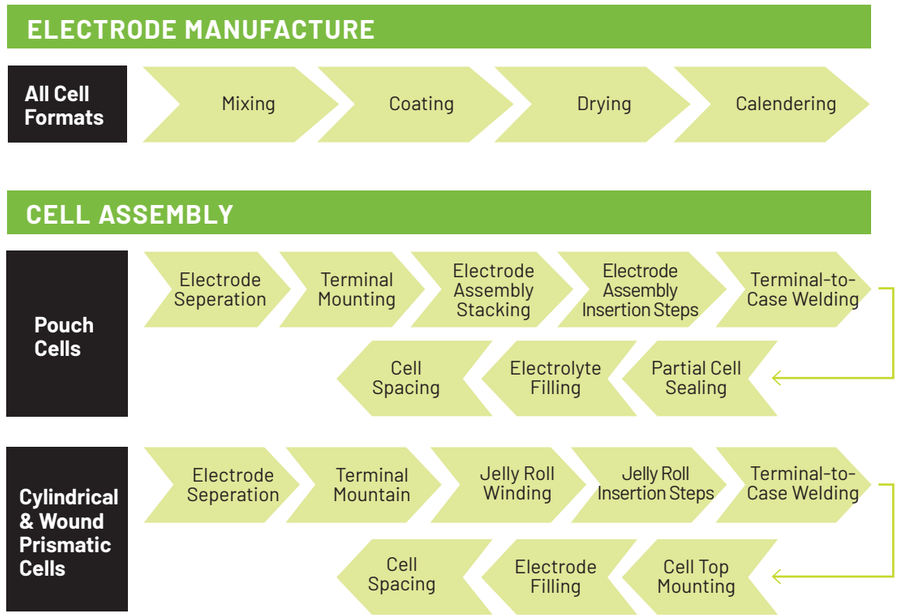
4.4. Cell assembly steps vs form factors

Cell assembly steps vary, depending on which cell form factor is employed.

TABLE 5: LEADING LI-ION CELL MAKERS WITH THEIR CELL FORM FACTORS [4]

COMPANY	BYD	CATL	LG CHEM	PANASONIC	SAMSUNG SDI	SKI
COUNTRY	China	China	Korea	Japan	Korea	Korea
FORM FACTOR	Prismatic	Prismatic	Pouch Cylindrical	Cylindrical	Prismatic Cylindrical	Pouch
MARKET SHARE, 2020	11%	24%	23%	16%	11%	3%

Figure 5: Simplified cell manufacturing process for various cell form factors.[5]



5. MANUFACTURING PROCESS AND EQUIPMENT VARIATIONS SPECIFIC TO CELL FORM FACTOR

5.1. Coin cells

Coin cells are used in the initial stages of materials qualification to screen the prospective materials and to provide the first approximation of expected electrochemical performance at reasonable cost. Coin cell fabrication and testing is often undertaken in an academic environment and as such can differ greatly in the production methods from their commercial counterparts. This is often due to a lack of suitable equipment, lack of appropriate training or because of the miniscule sample size. This can often result in testing data that does not reproducibly reflect the true electrochemical capabilities of the material. However, this discrepancy can be minimized by ensuring that format-appropriate processes are standardised and benchmarked to allow for comparable data between experimental cells.

Slurry composition and mixing: A study by Chen et al. shows that currently most "academic" coin cell slurries have an average AM content of around 80 wt.% with the balance split between the carbon and binder components.[6] However, this is a significantly lower AM amount than what is seen in industry-representative slurries for use in larger cells. Examples of "realistic" cathode and anode compositions, suitable for further scale-up into larger cells (e.g., as a short batch of cells for product demonstration purposes), are shown in Table 1. Slurry mixing for coin cell application is typically done on a lab scale, using stirring plates and stirrer bars or small propeller mixers.

Electrode coating, drying and calendering: Small coatings for coin cells are typically made either by hand or using small laboratory benchtop coaters (see Table 7 for an overview of equipment used in fabrication of various cell formats). This method is less precise than industrial, continuous coaters but is more suitable to small, varied material batches and has significantly less cost associated. For best results, careful calibration of instrumentation is recommended at this scale; see examples in Chapter 8 (best practices section) of this report.

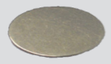
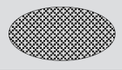
Due to the miniscule size of coin cell coatings, they are sometimes dried for only a few hours. Marks et al. proposed drying at 120 °C for 2 hours in a convection oven, mandatorily followed by drying the processed (compressed and punched) electrode discs overnight under vacuum to remove trace water.[7]

As stated previously, regardless of the cell format, electrodes typically undergo calendering (compression). By completing this process, coin cell positive electrodes can reach densities as high as 3.3 g/cm^3 whilst achieving 26% porosity, a value which is well in line with commercial cells.[7] Anode porosity is typically reduced to only ~30–35%, a value often dictated by electrode's wetting properties with respect to the employed electrolyte solution; this is discussed in more details in the formation section (Chapter 6) of this report.[8]

Electrode separation, vacuum drying and stacking: Coin cell electrodes are cut into their characteristic coin shape using a disk cutter. To improve repeatability of the following step in cell assembly, it is recommended that the anode be slightly larger than the cathode to reduce the risk of misalignment of the cell components. Long et al.[9] proposed a size enlargement of ~1.15 of the anode (N) vs. cathode (P), which increased data reproducibility and had no adverse effect on electrochemical performance of the cell. In addition to areal matching of electrodes, Long et al. also reported on testing of capacity-matched electrodes, with negative:positive (N:P) ratio ranging between 1.05 < N:P < 1.2. Capacity matching between the cathode and anode is part of an "art" of cell design calculations and often involves preliminary experiments in coin half-cells based on both cathode and anode active materials.

Cell assembly: The proposed coin cell assembly steps for various qualification workflows are shown in Table 6 and the rationale for parameter choices is discussed in the coin half-cell formation section (Chapter 6). The table illustrates the assembly of coin half-cells. Coin full-cells are built as per cell design qualification principles and instead of Li disks, mostly use graphite-based negative electrodes coated onto copper foil current collectors facing the separator.

TABLE 6: COIN CELL SET-UP PRINCIPLES FOR DIFFERENT QUALIFICATION WORKFLOWS

ASSEMBLY SEQUENCE	PART IMAGE	PART DESCRIPTION	MATERIAL QUALIFICATION	DESIGN QUALIFICATION
9		Negative case with insulator ring. Standard coin cell part, for example CR2032 from Hohsen®		
8		Spring washer		
7		SS spacer	0.5 mm thick	Thicker SS spacer to offset different Li and separator thickness
6		Lithium disc	Very thick Li metal disc (≥250 µm)	Thin Li metal disc (≤100 µm thick)
5		Porous separator	Glass fibre separator, highly permeable and very thick (≥300 µm)[10]	PP/PE/PP tri-layer e.g., Celgard® 2325 25 µm thick
4		"Standard" electrolyte (e.g., LP30 or LP40)	Generous amount (e.g., >50-100 µL)	Limited amount (e.g., 30-50 µL)
3		Positive electrode disc, coated side facing the separator. Electrode design as per specific cell batch.		
2		SS spacer	0.5 mm thick	0.5 mm thick
1		Positive case		

5.2. Pouch cells and cylindrical cells

Slurry composition and mixing: Heimes et al.[1] reports cathode industrially-relevant slurry ratios of 90 wt.% AM, 5 wt.% carbon and 5 wt.% binder and anode slurry also containing 90 wt.% of AM (Table 1), which is significantly higher than coin cells reviewed by Chen et al.[6] In fact, ever-increasing AM content, progressing with every new product generation, is a well-known trend in mass-scale cell manufacturing, especially in high-energy cell designs. In terms of slurry mixing, the equipment differs depending on the cell batch size rather than the cell format. For short batches of single-layer pouch cells, a small propeller mixer can be used for slurry mixing, similarly to coin cell coatings. However, to produce coatings used in large, commercial batches of multi-layer pouch cells (or commercial series of cylindrical cells), planetary mixers with controlled atmosphere are commonly employed (Table 7). Main control parameters available for optimisation in industrial slurry mixing are:

- mixing sequence
- applied shear
- slurry viscosity
- slurry homogeneity.[10]

Electrode coating, drying and calendering: As with slurry mixing equipment, coating methods differ depending on the cell batch size and are often shared between various cell formats. For example, a benchtop coater can be employed in preparation of electrodes for a small batch of single-sided pouch cells, but a larger pouch cell batch may require a continuous coater/dryer (Table 7). Industrial coater/dryers further vary in size, ranging from R&D-style machines and pilot coaters to full scale industrial-scale continuous coaters capable of coating speeds in the range of 50 m min^{-1}. Slot-die coating options are the state-of-the-art choice [11].
Industrial coaters/dryers are equipped with multiple drying zones, set to gradually varied temperatures (e.g., 80 °C → 160 °C → 160 °C → 80 °C for NMP-based cathode slurry) to optimise the complex drying process conditions and avoid unwanted coating aberrations such as binder migration towards the coating surface or pinhole defects.[11]

The principle of operation for electrode calendering equipment does not significantly differ between different cell formats and batch sizes (Table 7). However, with the increasing fabrication line output (and larger cell sizes), the need for electrode parameter optimisation and monitoring is more significant. Therefore industrial calendering machines are commonly equipped with pressure gauges, often in addition to gap control devices.[12] Importantly, cathodes and anodes exhibit very different calendering behaviour, due to the dissimilar physical densities as well as variations in the compaction resistance of their active materials.[13] Therefore, at the industrial cell design and engineering stage, coating and calendering optimisation is necessary for both cathode and anode electrodes.[13]

Electrode separation, winding or stacking, cell finishing: The cell assembly consists of the preparation and stacking of electrodes as well as cell finishing steps. During the preparation step, the calendared coatings are cut into electrode plates or sheets for stacking. In the state-of-the-art process, electrodes are punched using dies or trimmed by a cutting blade. Repeated punching or cutting action on a brittle coating may limit the life of cutting tools and lead to burr formation or damaging the knife edge. This leaves room for improvement in this production unit operation, with laser cutting technology being one of the process alternatives (Table 12).

The following alternative stacking methods are used in fabrication of industrial cells:

- winding in cylindrical cells (18650 cell and 21700 cells, etc.)
- flat winding in prismatic cells (prevalent in Asia; heritage of consumer electronics cells)
- stacking of single sheets in pouch or prismatic cells (preferred in Europe)
- Z-folding in pouch or prismatic cells, which is one of the newer stacking methods

After the stacking process, cell finishing is the last part of the cell assembly and consists of the following steps:

- joining of the copper and aluminium conductors
- packaging the stack into a thermoformed aluminium barrier film or a rigid metal case
- filling in the electrolyte
- sealing the package under vacuum
- formation and further testing the cells.

A critical step in the cell finish is the connection of electrode assemblies to the electrical terminals, which is currently done by ultrasonic welding. Alternative processes like laser welding may offer improved electric resistance and mechanical durability of the welds but need further development prior to industry-wide implementation (Table 12). The seal quality of the filled and sealed cells is critical for overall cell quality, especially around the electrical terminals, which calls for advanced process control and sophisticated leak monitoring methods.[14]

Cell assembly: The build specifications of industry-representative, larger cells (e.g., cylindrical 18650 cells, multi-electrode pouch cells and prismatic cells) are highly dependent on the purpose for which they are designed. Consequently, the cells used in high-power applications (power tools and hybrid electric vehicles) will employ different grades of materials and follow different cell design principles than cells destined for high energy applications (portable electronics and electric vehicles). A common technique to induce higher power density in cells is to synthesise smaller AM particles for both the anode and the cathode, with the cathode material prioritising high porosity electrode architectures. To optimise Li-ion diffusion throughout the electrode, the path length must be minimised by reducing the resistance through lesser coating weights and areal capacities. An additional feature of high-power cylindrical cells is a multi-tab design. Tabs are welded onto the respective electrode current collectors (uncoated portion) and perform the function of conducting electrons to and from the external electrical circuit. High-energy cylindrical cells typically have a single tab per electrode (a pair of tabs per cell), located on the opposite sides of the cells, while some high-power cells have a 3-tab design (two tabs welded onto the anode and one per cathode) to minimise the ohmic resistance to electron conduction in and out of the cell.[15]

5.3. Examples of equipment for fabricating various cell form factors

TABLE 7: COMMONLY USED STATE-OF-THE-ART EQUIPMENT FOR VARIOUS CELL FORM FACTORS[1, 3, 12, 13]

PROCESS STEP	COIN CELLS	POUCH CELLS	CYLINDRICAL CELLS
Mixing	Small batches: (few hundred mL of slurry): overhead mixers are commonly used (electric or air propelled), various impeller shapes can be applied to control shear. Standard laboratory equipment can be used for this purpose. Mid-to-large batches: (few litres of slurry and more) are typically mixed using vacuum planetary mixers (e.g., Ross or Netzsch) for bubble-free slurry mixing in controlled atmosphere. Notes (applicable in all batch sizes): • Stainless steel propellers and shafts are commonly used in slurry mixing. • High-end facilities use separate mixing equipment for cathode and anode slurries. • Dispersion of nanoparticulate carbons often requires a homogeniser. • Mixing environment conditions are elaborated in Appendix III.		
Coating / Drying	Small batches: coating of individual sheets, ~A4 or smaller. Can be hand-drawn or semi-automated using doctor blade benchtop coaters; examples: MTI[16], RK Print[17], etc.	Mid-to-large batches: continuous coating process onto foil rolls, combined with drying in temperature-zoned chambers. Minimum quantity of coating is several metres (pilot coating size), with roll lengths reaching thousands of metres. Examples of continuous coater/dryer manufacturers: Durr-Megtec[18], Hirano[19], etc.	
Calendering	All batch sizes: regardless of batch and machine size, calendering equipment relies on the same operating principle of compression and compacting of the coated electrode sheet or roll between two rotating rollers. Example of calendering machine manufacturer: Breyer (Germany), Coatema (Germany), Saueressig (Germany), etc.		
Electrode Cutting / Punching / or Slitting	Coin cell discs: coin cell electrodes are manually punched using electrode disc cutter (e.g., from Hohsen).[20]	Pouch cell electrode sheets: short batches of pouch cell electrodes are often made using die cutters (e.g., MTI).	Cylindrical cell electrode strips or large batches of pouch cell electrodes: typically produced using electrode cutting/slitting machines (e.g., Nagano).[21]
Vacuum Drying	All batch sizes: standard laboratory equipment can be used for this purpose. Note: in laboratory conditions, when vacuum-drying NMP-based coating sheets for small cells, it is beneficial to initially drive off and collect (e.g., using scrubber or cold trap) excess NMP. This is done for operator safety (see notes on NMP in Appendix IV) and to counteract vacuum oven seal deterioration caused by NMP.		
Winding or Stacking	Coin cell discs: coin cell electrodes are manually assembled, usually under argon atmosphere, inside a glove box. Disc alignment is critical to achieve reproducible coin cell batch performance.	Pouch cell electrode sheets: short batches of pouch cell electrodes can be assembled by hand, using electrode stacking fixtures (e.g., Hohsen) Mid-to-large batches of pouch cell electrodes: stacking machines or integrated production systems are used (e.g., Manz)[22]	Cylindrical (or wound prismatic) cell electrode strips: semi-automatic or automatic winding machines are used to wind cathode, separator, and anode strips together. Winder manufacturers examples: Hohsen, Manz, etc.
Electrolyte Filling	Coin cells or short batches of pouch cells: electrolyte is injected into cells using standard lab micropipette, under argon atmosphere, inside a glove box.	Mid-to-large batches of pouch cells and cylindrical cells: electrolyte filling and degassing stations are used. Examples of electrolyte filling station manufacturer: Manz, Sovema, etc.	
Cell Packaging	Cell packaging equipment consists of multiple components that are cell format-specific and often can be purchased in sets. Examples of cell component makers and/or suppliers: Targray,[23] Hohsen, etc.		
Formation	Small-to-mid size batches: (few to tens of cells): short batches of cells are typically formed using battery cyclers per specific formation protocol. Examples of cycler makers are: Maccor, Arbin, BioLogic, Neware, etc.[24] Mid-to-large batches: (hundreds of cells and more) are typically formed using dedicated formation equipment. The modularisation of formation process is done to save cost on formation process, which (for large number of cells) is time and energy consuming operation. Example of formation charge/discharge platform manufacturer: Keysight.		

6. FORMATION PROCESSES IN VARIOUS CELL FORMATS

6.1. Formation process overview

One of the most significant steps in the cell fabrication process is the formation of the solid electrolyte interphase (SEI). Despite irreversibly consuming some amount of lithium from the fresh cathode material, SEI formation is a critical stage in Li-ion cell fabrication. SEI formation enables stable, reproducible cell cycling via controlled reactions at the electrode surface with the electrolyte and its relevant components (salt, additives, solvent). The product of this is a combined substance that passivates the surface layer of the electrode and hinders any further undesirable reaction with the electrolyte. This is particularly important for reactive and porous electrodes such as graphite anodes. According to Wood et al., the SEI layer should be thin, minimally porous, electrochemically inert, electronically resistive, and ionically conductive to Li-ions.[25] Typically, the process of SEI formation has three main stages:

- Wetting: During the formation of the SEI layer, electrolyte must be in constant contact with the electrode surface to ensure uniformity. This concept is referred to as wetting and can also be conducted at elevated temperatures of 40–60°C to encourage electrolyte access to the electrode mesopores as this is vital for the proper completion of the SEI layer formation. Due to this requirement, many companies often incorporate multiple wetting stages. However, this can impede the throughput of the formation process and/or increase its cost. Electrode wettability is key to successful SEI formation[26] and depends on many factors, such as electrode thickness, compression, composition, and cell format.

- Formation: Initial charge/discharge cycles are conducted at low and controlled currents to ensure the optimal characteristics are realised in terms of the SEI's composition and thickness, both of which are crucial in minimising the capacity fade of the resultant cell. According to several researchers,[25,27,28] the formation process occurs in two successive stages, the first stage being the creation of ionically resistive SEI layer at >0.25 V vs. Li/Li+ and the second stage involving SEI layer conversion to a highly ionically conductive film at <0.25 V vs. Li/Li+.

- Aging: After the completion of the wetting and formation stages, the aging process begins. This can take on average 1-2 weeks and involves ongoing leak current checks. Immediately after formation, leak currents of 20-50 μA cm^{-2} are expected to occur. However, this range will gradually decrease with the average reaching 2-5 μA cm^{-2} after only a few days and a couple of weeks realising a decrease to less than 1 μA cm^{-2}. This process requires large amounts of cycling equipment and floor space, potentially taking up to almost a quarter of a production area.

The above steps combined can take more than two weeks to accomplish and cannot be compromised as all three stages are prerequisite to the successful creation of the SEI layer. Many analytical studies have been published on the fundamental aspects of SEI, giving details on the formation process, reaction mechanisms, and material characterisation covering the investigation of morphological structure, material, and chemistry. However, practical details such as the exact formation protocols used in an industrial setting, including step duration and conditions, often remain a trade secret due to the impact on product (battery) quality and cost of manufacturing. This includes any use of additives used to control and optimise SEI formation. An example set of the industrial formation conditions (many possible variants exist) is given in Table 8.

TABLE 8: EXAMPLE OF AN INDUSTRIAL SEI FORMATION PROCESS FOR LARGE CELLS, MODIFIED AFTER WOOD ET AL.[29]

FORMATION STAGE	DURATION	CONDITIONS
First wetting	6-24 h	Rest step held at 25 °C
First formation	10-40 h	1-2 formation cycles at 0.1–0.2 C charge/discharge rate, 25 °C
Second wetting	12-24 h	Rest step held at 25 °C
Second formation	0.5-20 h	1-2 formation cycles at 0.2–0.5 C charge/discharge rate, 25 °C
Third wetting	12-24 h	Rest step held at 40-60 °C

6.2. Formation protocols for coin half-cells

Because coin half-cells use metallic lithium as the negative electrode instead of graphitic active AM, SEI formation proceeds differently to the full-cells. Specifically, instead of the formation of a stable, continuous interphase, the reactions between metallic lithium and electrolyte continue and an irregular, "mossy" SEI keeps re-forming on cycling of coin half-cells. Therefore, the term "formation" does not strictly apply to coin half-cells.[6]

Instead of relying on the formation of stable, passivating SEI layer during the initial charge/discharge cycles (as in full-cells), cycling stability of coin half-cells can be significantly improved by altering and controlling the following parameters:

- thickness of Li metal anode
- volume of electrolyte
- areal weight loading of cathode
- type, thickness and porosity of separator.

It is important to note that employing thick lithium metal electrodes, excessive volume of electrolyte, lighter cathode weight loadings and thick separators in coin half-cells is only rational in cases of analytical, specialised testing of materials but does not translate onto realistic scale-up to larger cell formats (e.g., full pouch cells, cylindrical or prismatic cells), which must be done using different cell design principles.

6.3. Formation protocols for coin full-cells

- Wetting: in general, there are no commonly shared wetting protocols used in coin full-cells across several key literature reports.[9, 29, 30] This is caused by the multitude of studied materials as well as variations in the employed cell design parameters (e.g., electrode architecture, thickness, compression, and porosity). Initial rest time allowed for the freshly constructed cells to soak up electrolyte varies between not reported (possibly, not controlled) to several hours (e.g., six).[9] For best results, allowing longer, controlled wetting times is recommended to achieve the most reproducible cell testing outcomes.

Coin cell assembly processes do not involve vacuum application, which somewhat facilitates wetting in larger cell formats. On the other hand, coin cells are often flooded with electrolyte and have favourable electrode-electrolyte contact area, therefore their wetting stage proceeds with less hindrance than in case of larger cells with stacked electrodes.

Some research groups pre-soak electrodes or separators prior to coin cell assembly. However, this approach is normally not recommended due to potential reproducibility issues. Nevertheless, controlled soaking of the separator with electrolyte is fully justified in comparative separator studies,

where electrolyte uptake into separator pores is carefully measured and correlated to separator's material, porosity, tortuosity, etc.[31]

- Formation: coin full-cell formation protocols differ widely in literature reports with respect to the following parameters: formation voltage range, charge/discharge current rates, rest duration and formation temperature. Primarily, these differences are driven by the cathode active material type and the employed cell design. A representative set of standardised formation conditions for coin full-cells with NCM cathodes as proposed by Long et al.[9] is shown in Table 9. In contrast, coin cells containing LFP cathodes are typically tested within the 3.6-2.5 V voltage range and often with an extended wetting step/rest, e.g., 24 h.[32]

  Many full-cell protocols begin with a "tap charge" designed to bring the anode potential below the threshold of copper corrosion by reducing the surface oxides on graphite and slightly lithiating the graphitic anode AM. In this way, the copper current collector used in the vast majority of full cell formats is "protected" against corrosion during extended cell cycling.

TABLE 9: EXAMPLE OF STANDARDISED SEI FORMATION PROTOCOL FOR FULL COIN CELLS, AFTER LONG ET AL.[9]

FORMATION PARAMETERS	CONDITIONS
Tap charge	Charge to 1.5 V and hold for 15 minutes (or 0.33 C current cut-off)
Rest	6h duration
Voltage range	4.2-3.0 V (NCM cathodes)
Charge current rate	0.1 C constant current, 0.05 C current cut-off at the top of charge
Discharge current rate	0.1 C
Number of formation cycles	4
Temperature	25 °C

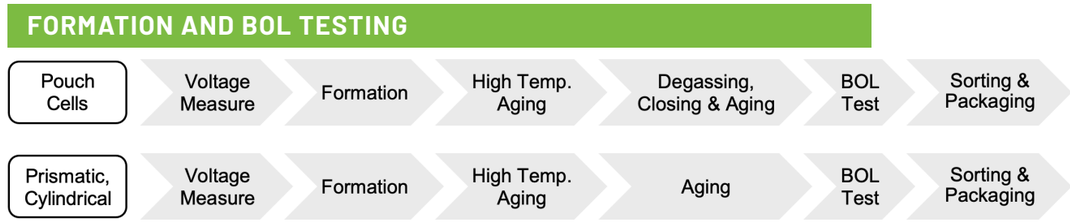
  A recent study by Pathan et al.[33] used an intentionally restricted voltage range during cell formation cycles (i.e., the upper voltage limit of 4.0 V), specifically to avoid higher voltage-induced cathode stress and to limit the possible formation of the CEI. In the same study, the lower formation voltage limit was decreased to 2.6 V for an in-depth study of the anodic SEI formation processes.

- Aging: in most cases the aging step is not done when testing coin cells unless their specific purpose is to investigate electrode aging processes. In which case aged electrodes are harvested upon cell disassembly and then a variety of surface analyses (e.g., XPS, TEM, XRD, Raman spectroscopy) are employed to identify the surface deposition products.

6.4. Formation process in pouch cells and cylindrical cells

Key stages of formation process in pouch cells and hard-case cells are shown in Figure 6

Figure 6: Key steps of cell formation and aging process for various industrial cell form factors[1, 3, 4, 5]



Protocol-wise, for equivalent AMs, there are many similarities in formation and testing protocols between coin full-cells, pouch cells and cylindrical cells, especially regarding the employed voltage ranges and current rates.

The differences in formation process stages for various cell formats predominantly originate from variations in cell packaging and/or cell geometry and are manifested during the following stages:

- electrolyte filling by injection
- electrolyte diffusion and electrode wetting
- cell degassing.

Electrolyte filling: Filling into pouch cells and cylindrical cells alike occurs by injection, using a high-precision dosing needle. In the case of pouch cells, the dosing needle is introduced into the partially sealed pouch just after electrode stack assembly and tab welding. In cylindrical cells, electrolyte is also injected through a dosing needle, just after the insertion of a jelly roll into the top-less cell case.

In both of the above cell formats, after electrolyte is injected into the cell, a supply of inert gas or vacuum application activates capillary action so that the wetting process may begin. Partial filling and evacuation steps may need to be repeated depending on the exact cell design details and the wettability of electrodes and separator.

Electrolyte diffusion and electrode wetting: State-of-the-art electrodes are often difficult for electrolyte solution to wet due to their heavy weight loadings, high compaction, and low porosity. Anode porosity is typically reduced to only ~30–35%, which affects the pore-size distribution (<100 nm for majority of pores) and hinders capillary action that enables electrode's wetting by electrolyte.[25,26] In larger Li-ion cells (i.e., larger than coin cells), there are additional barriers to electrode wetting, such as other cell components (e.g., separator pores, the electrode binder, and the conductive carbon black additives) which may wet preferentially. Hence, a period of ~12–24 h under vacuum may be required to wet the electrodes, and sometimes, the smallest pores remain unwetted.

Degassing of the cell: This refers to the disposal of gaseous by-products evolved during the formation process, as elaborated below:

- Under normal conditions (i.e., impurity- and moisture-free electrolyte) the main by-product of SEI formation is gaseous ethylene[35] and in pouch cells the volume of the evolved gas is significant enough to warrant using soft rubber-lined cell restraints (e.g., at 25 kPa gauge pressure)[30] during formation and cycling to improve cell testing reproducibility.

- Another common practice for handling formation gases, recommended for academic laboratories where short batches of pouch cells are fabricated, is re-opening of pouch cells after the formation under inert atmosphere (e.g., inside a glove box), followed by re-sealing of the cells.[30]

- In an industrial environment, degassing of pouch cells is accomplished by having excess volume of pouch (a "gas bag") available for gas collection and then, after formation, sealing off the gas-filled bag. Typically, gas bags are then pierced and re-sealed off inside of the dedicated electrolyte diffusion and degassing chambers.[1, 36]

7. AN OVERVIEW OF THE CELL TESTING PROTOCOLS

Standard cell testing is usually done for a specific purpose, follows highly detailed procedure(s) and generates structured datasheets as an outcome.[37] As already mentioned in this report, standard cell testing is usually employed for one of the two main purposes:

- materials qualification and/or in-depth examination of their electrochemical behaviour
- development and/or qualification of the new cell design.

Regardless of the testing purpose, the cell testing will basically follow the same top-level stages, as illustrated in Figure 7 and described below. The primary differences between various testing workflows manifest in the tested cell's formats and testing protocol details.

Figure 7: High level summary of Li-ion cell formation, Reference Performance Test (RPT) followed by further testing stages[38]

- Preparation: This stage is often overlooked in literature descriptions of standard cell testing protocols but can have significant effect on the experimental error and the results deviation. Therefore, during the preparation stage care should be taken to ensure adequate calibration of testing equipment, such as battery testers/cyclers, temperature and/or environmental chambers as well as ensure the proper care, maintenance and set-up of the lead cables and cell testing fixtures. Another important part of the cell test preparation is awareness of the risks and potential hazards associated with the given test, followed by pre-emptive risk mitigation, for example, including specific PPE descriptions into test protocols as well as requiring the ready availability of cooling aids (ice packs, sand buckets, etc.) where applicable.

- Formation: This stage was extensively covered in previous sections; therefore, this chapter will mainly focus on the post-formation testing of various Li-ion cells.

- Reference performance test (RPT): This is typically completed at regular intervals post-formation and throughout the cell testing regime. The purpose of RPT is to repeatedly assess the state of health (SOH) of the cell over the entire test duration. RPT should be performed at constant temperature and using reasonably "mild" testing conditions (e.g., non-aggressive current rates). Dubarry et al.[38] gave an example of a three-stage RPT, consisting of conditioning, low-rate cycling, and nominal rate cycling. The three-stage RPT is relatively complex and in some cases, only two slow-rate charge/discharge cycles (e.g., C/10 or similar) are sufficient. The main purpose of RPT is to gather information on the maximum capacity that a cell can deliver, its rate capability and resistance.

- Custom tests: Often, this part of testing profile is also called "duty cycle". This testing section strongly depends on the intended application and battery use conditions. Details of the specific duty cycles are extensively described elsewhere.[37, 38, 39] An example of the duty cycle testing (in this case, cycle life testing at two different temperature conditions) is included in Table 10, along with the rest of the sample testing protocol.

- Final RPT: This is usually, done according to the same test conditions as mid-testing RPT stages. It serves as the final point of reference for tracking the SOH of the cell.

7.1. An example of the cell testing protocol

TABLE 10: EXAMPLE CELL TESTING PROTOCOL

TESTING STEP	C-RATE	TEMPERATURE
Formation	C/10 (repeated 2x)	25 °C
Reference Performance Test (RPT)	C/5, C/2, 1 C (all repeated 3x)	25 °C
Cycle Life Testing	50 cycles 1 C charge/discharge	25 °C
	Re-RPT	25 °C
	50 cycles 1 C charge/discharge	45 °C
	Re-RPT	25 °C

7.2. Specification datasheets for commercial cells

Specification datasheets provided with commercial cells provide an insight into the key metrics reported by the commercial cell makers and provide a basis for cell validation services to be executed as needed by the third-party testing and validation (or certification) providers. Currently, there is no general consensus on which information must be included in a commercial cell specification datasheet. Depending on the cell manufacturer, the intended application, or even on the target customers, the datasheet may contain varying content. There are ongoing efforts at standardisation of specification datasheets, with one proposed layout [37] shown in table 11. Other typical parameters given in cell specification datasheets are internal cell resistance, peak power for charge and discharge, maximum continuous or peak current and voltage limits.

TABLE 11: AN EXAMPLE OF THE COMMERCIAL CELL SPECIFICATION DATASHEET [37]

SPECIFICATION	DESCRIPTION	
Rated Capacity Cn	Minimum achievable capacity measured at ambient temperature of 20 °C, using constant current discharge, during specified time base (n).	
	Typical time basis values: • 5 hours (C5) for portable applications • 3 hours (C3) for battery electric vehicles, BEV • 1 hour (C1) for hybrid electric vehicles, HEV	Applicable standards: IEC 61960, IEC 62620 IEC 62660-1, ISO 12405-2 IEC 62660-1, ISO 12405-1
Nominal Voltage	Mean voltage achieved during the discharge that leads to the rated capacity (= the standard discharge)	
Capacity	Minimum achievable capacity measured at ambient temperature of 25 °C, using specified discharge current rate.	
Charging	Standard charging method, as specified by the cell manufacturer. Includes information on the current rate, upper cut-off voltage limit for constant current charging (CC) and total charging time limit or a cut-off current for constant voltage (CV) charging.	
Weight	Maximum cell weight is given. Median weight is used for a batch of cells reported on a single datasheet.	
Temperature	Recommended values for charge, discharge and storage are given. These are the maximum (or minimum) ratings at the surface of the cell.	
Specific Energy	Specific energy ($Wh \cdot kg^{-1}$) is calculated using minimum capacity at 25 °C, nominal voltage, and maximum cell mass.	
Energy Density	Energy density ($Wh \cdot L^{-1}$) is calculated using minimum capacity at 25 °C, nominal voltage, and maximum cell dimensions.	
Dimensions	Technical drawing of the cell with dimensions of cell and tabs.	

8. RECENT PROCESS DEVELOPMENTS REPORTED IN LITERATURE

TABLE 12: MANUFACTURING PROCESS METHODS UNDER DEVELOPMENT, MODIFIED AFTER LIU ET AL.[3]

MANUFACTURING PROCESS STEP	CONSIDERED METHOD	SIGNIFICANCE	CHALLENGE
Mixing	3D hydrodynamic shear mixing	Low cost, easy to scale up	Limited improvement
	Ball milling	High efficiency, good uniformity	The risk of damaging the structure of active materials particles
	Ultrasonic mixing	High efficiency, especially for high concentration slurry	Instrumental cost and hard to scale-up
	Different mixing sequence	Improve the uniformity without marginal modifications	Need more study for different materials
Coating / Drying	Low solvent content extrusion	Lower the drying time and energy	Cannot avoid the toxic organic solvent and potential instrumental investment
	Dry calendering	Save the drying time and energy	Potential instrumental investment, scale-up ability
	Dry printing		Potential instrumental investment, scale-up ability, and spray uniformity
	Electrostatic coating		Potential instrumental investment, scale-up ability, and coating uniformity
	Three-stage drying	Lower the drying time without extra instrument cost	Limited improvement
	Laser annealing	Lower the drying time and energy consumption	
	Infrared heating		
Slitting	Laser cutting	Improve the cutting quality and flexibility for different shape design	Potential instrumental investment and relatively low throughput
Vacuum Drying	Argon purging	High throughput and room-temperature operation	The efficiency after scale-up needs to be verified
Welding	Laser welding	Low contact resistance and high tensile force	Difficulty to join dissimilar and high-reflective materials
	Wire bonding	Low energy consumption	Only feasible for cylindrical cell
Formation	Narrow the voltage window	Save the formation time without extra cost	Mechanism still unclear
	Pulse current charging	Save the formation time, low cost	Specific frequency needs to be discovered for different types of cells
	Artificial SEI layer	Save the formation time	Potential instrumental investment and scale-up ability

APPENDICES

Appendix I. Selected standards: material specifications and analyses [41]

Generic NCM Cathode		File Name	Example Product Specification		
Department		**File Type**	Technical Standard	**Version**	
Confidentiality		**File Number**		**Page Number**	
Created by		**Audited by**		**Approved by**	
Date		**Date**		**Date**	
1 Definition This specification example summarises technical indicators, test methods, packaging, product shelf life, handling and storage of the generic high energy lithium nickel cobalt manganese oxide (NCM) lithium-ion battery cathode material. **2 Scope** Example: For use in prismatic, cylindrical and polymer pouch cells **3 Properties**					
Main Physical Characteristics					
Category	Unit	Specification (Example)	Comments	Test Method or Instrument	
Appearance	/	Black powder	/	Visual inspection	
Tap density	g/cm^3	≥2.30	High TD is critical	Tap density meter	
Specific surface area	m^2/g	≤0.50	Low SA is beneficial	Surface area meter	
H_2O	ppm	≤300	The lower, the better	KF moisture meter	
D10	μm	≥5.00	Particle Size Distribution depends on the specific material design	Laser particle size analyzer (e.g.: Malvern)	
D50	μm	12.00±2.00			
D90	μm	≤25.00			
Main Chemical Characteristics					
Li	wt%	7.40±0.20		AAS or ICP	
Ni	wt%	Elemental composition depends on the specific material design		Dimethylglyoxime gravimetric method or ICP	
Co	wt%			Potentiometric titration test or ICP	
Mn	wt%			Potentiometric titration or ICP	
Fe	ppm	≤50	Trace elements will differ depending on the material's origin and processing details	ICP test	
Cu	ppm	≤50			
Na	ppm	≤500			
Ca	ppm	≤200			
Mg	ppm	≤200			
Free Li^+	ppm	≤2500	Critical for High-Ni cathodes	Titration	
pH	/	11.60±0.20		PH test	
ROHS	/	EU standard		Third party testing	
Magnetic impurities（Fe+Cr+Zn）	ppb	≤50	Extremely important metric, lowest possible	ICP test	
Crystal Structure					
Particle morphology	/	Spherical	/	Scanning Electron Microscope (SEM)	
Crystal structure	/	No impurity peaks	/	X-ray diffractometer (XRD)	
Electrochemical Performance					
Specific capacity	mAh/g	Depends on cathode composition, typical values available in open publications		Coin half-cell，4.30-3.0V，25 ，0.1C charge and discharge	
First cycle efficiency	%	爱85.0	Depends on e.g.: cycling voltage limits	Coin half-cell，4.30-3.0V，25 ，0.1C charge and discharge	
4 Packaging					
5 Storage and Shelf Life Stored in a cool and dry place under vacuum-sealed bag. The shelf life is one year. **6 Handling and Storage** 6.1 Avoid disturbing and turning over during transport. 6.2 Store in clean, cool and dry environment. 6.3 Avoid sunlight, fire and rain during transport. 6.4 Use immediately after opening the bag. If not used immediately, store in re-sealed bag to prevent moisture absorption.					
7 Particle Size Distribution Diagram					
8 SEM Micrographs					
9 XRD Pattern					

Appendix II. Selected standards: cell testing

The overall landscape of standards related to Li-ion cells and batteries is complex. Leading standards relate to cells/batteries safety, especially transportation safety. Performance standards do exist, however, are mostly application (end use) driven. Many established cell makers and system integrators rely on agreed upon, customary suites of testing methods, verified by a nationally and/or internationally recognised testing laboratories. Furthermore, high-end applications (including aerospace and defence) have their own set of standards.

TABLE 13: INTERNATIONAL BATTERY TESTING STANDARDS, AFTER RECHARGE ASSOCIATION[42]

ORGANISATION	STANDARD NO.	TITLE
United Nations (UN)	UN 38.3	Recommendations on the transport of Dangerous Goods, Manual of Tests and Criteria, Part II, Section 38.3
International Electrotechnical Commission (IEC)	IEC 62133	Secondary Cells and Batteries Containing Alkaline or Other Non-acid Electrolytes – Safety Requirements for Portable Sealed Secondary Cells, and for Batteries made from Them, for Use in Portable Applications
	IEC 62281	Safety of Primary and Secondary Lithium Cells and Batteries During Transportation
	IEC 62660-1, Ed 1 Dec 2010	Secondary Li-ion cells for the propulsion of electric road vehicles – Part 1: performance testing
	IEC 62660-2, Ed 1 Dec 2010	Secondary Li-ion cells for the propulsion of electric road vehicles – Part 2: reliability and abuse testing
International Standard Organisation (ISO)	IEC 62133	Secondary Cells and Batteries Containing Alkaline or Other Non-acid Electrolytes – Safety Requirements for Portable Sealed Secondary Cells, and for Batteries made from Them, for Use in Portable Applications
	IEC 62281	Safety of Primary and Secondary Lithium Cells and Batteries During Transportation
	IEC 62660-1, Ed 1 Dec 2010	Secondary Li-ion cells for the propulsion of electric road vehicles – Part 1: performance testing
	IEC 62660-2, Ed 1 Dec 2010	Secondary Li-ion cells for the propulsion of electric road vehicles – Part 2: reliability and abuse testing

Appendix III.
Electrode and cell fabrication environmental conditions

TABLE 14: FABRICATION ENVIRONMENT CONDITIONS, SOURCE: HEIMES ET AL.[1]

MANUFACTURING PROCESS STEP	CLEAN ROOM CLASS	DRY ROOM (DEW POINT)	TEMPERATURE	ANNOTATIONS
Mixing	ISO 8	N/A	22 ± 2 °C	The electrode manufacturing takes place under clean room conditions, since foreign particles in the coating cannot be removed in the later process by cleaning methods (e.g., suction).
Coating	ISO 7	Semi-dry (5 °C to -5 °C)		
Drying				
Calendering	ISO 7 – ISO 8			
Slitting				
Vacuum Drying		Dry (0 °C to -30 °C)		
Separation	Min. ISO 7	Dry (-25 °C to -35 °C)	22 ± 2 °C	The cell assembly must be carried out under dry conditions, as water inside the cell leads to strong quality losses (service life) and to safety risk (formation of hydrofluoric acid).
Stacking / Winding		Dry (-40 °C to -50 °C)		
Packaging				
Electrolyte Filling		Extra Dry (-50 °C to -70 °C)		
Formation	N/A	N/A	22 ± 3 °C	Cell finishing takes place in a normal environment. Since the cell is already sealed and degassing takes place in a vacuum chamber, there are fewer requirements for the particle environment and humidity.
Degassing				
HT Aging			30 °C to 50 °C	
NT Aging			22 ± 3 °C	
BoL Testing				

Appendix III. Glossary

AM – Active material: Either the cathode active material or anode active material.

CAM – Cathode active material: High purity structured materials that intercalate lithium ions.

Cathode electrolyte interphase (CEI): The CEI is usually formed spontaneously at the high voltages (e.g., ≥4.4 V) when cathode active material undergoes the initial charge and discharge cycles in the presence of electrolyte. CEI contains inorganic components and organic species, deposited via complicated chemical surface reactions between electrolyte and electrode.

Cell design: A set of multiple parameters, optimised for a specific cell application/end use (e.g., high power or high energy cell use). A non-exhaustive list of cell design parameters is summarised below.

- Electrode parameters: electrode coating weight loading, porosity, active material particle size distribution, carbon content/type, binder content/type, etc.
- Current collector parameters: thickness, adhesion-promoting coating(s), etc.
- Separators: thickness/type, porosity, tortuosity, etc.
- Electrolyte: conductivity, Li salt concentration, additive amounts/types, etc.
- Current conducting tabs: thickness, material, single/multiple tabs, etc.

Cell format: Also known as cell form factor, cell format describes cell casing, packaging material/type and, in most cases, provides cell dimensions. Li-ion cells come in three basic form factors: cylindrical, prismatic, and the flat rectangular shape of lithium-polymer cells. The standard formats for metal-encased cylindrical cells are 18650 (18 mm in diameter × 65 mm tall), 21700 (21 × 70 mm), or 26650 (26 × 65 mm). Li-ion cylindrical and prismatic cells are packaged in metal cans, whereas lithium-polymer cells are encased in thin (0.1 mm) inflexible aluminium-foil laminate pouches with custom footprints.

CMC - Carboxymethyl cellulose: a polymer used as an electrode binder.

C-rate: The C-rate is a measure of the electric current rate, at which a cell or battery is being charged or discharged. C-rate is defined as the actual current experienced by the cell or battery, divided by the theoretical current draw under which the battery would deliver its nominal rated capacity in one hour. C-rate has the unit of h^{-1}.

LFP – lithium ferro phosphate: a cathode active material.

Lithium dendrites: Lithium dendrites are undesired metallic microstructures that can form on the negative electrode during the charging process of a Li-ion cell, especially if the cell is subjected to overcharging or low temperature charging. Metallic lithium dendrites are formed when excess lithium ions accumulate on the negative electrode's surface and cannot be absorbed into the anode in time. Dendrites can cause short circuits and lead to cell or battery failure or even fire event.

Li/M ratio: Lithium to Metal ratio in cathode active materials.

LP30, LP40: Commercially available electrolytes, lithium hexafluorophosphate dissolved in a mixture of diethyl carbonate and ethylene carbonate

NCM – nickel, cobalt, manganese: a cathode active material

NMP: N-methyl pyrrolidone (NMP) is an organic solvent, commonly used to fabricate the electrodes for Li-ion cells, especially cathodes (there is an aqueous process alternative for anode coatings). The use of NMP in cell manufacturing adds capital and operating costs due to the necessary solvent evaporation needs and presents environmental control and occupational safety problems as NMP is an irritant to multiple organs, teratogenic and toxic (e.g., to the reproductive system). Appropriate PPE and precautions should be employed when working with NMP.

N/P ratio: the ratio between the capacity of the anode and the capacity of the cathode in a cell.

PE: polyethylene polymer used in separators.

PP: polypropylene polymer used in separators

PPE: personal protective equipment

PVDF: polyvinylidene difluoride, a polymer used as a binder for electrodes.

Solid electrolyte interphase (SEI): The SEI is a passivating layer formed on the graphitic anode's surface during the initial Li-ion cell charge/discharge cycles as a result of the electrode surface's side reactions with the electrolyte solvent and Li-containing salt.

SS: stainless steel

TEM: transmission electron microscopy

XPS: x-ray photoelectron spectroscopy

XRD: x-ray diffraction

18650 cell: a cylindrical battery cell 18 mm in diameter and 65 mm tall.

21700 cell: a cylindrical battery cell 21 mm in diameter and 70 mm tall.

Appendix V. Selected patents

DEGASSING & FORMATION

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2. Split Charge Forming Process for Battery, S.-Y. Yoon et al., A123 Systems, Inc., US20100192362A1
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