



Thermophysical abuse couplings in batteries: From electrodes to cells

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Thermophysical couplings in batteries must be understood to ensure that batteries remain safe from potential immolation during operation. This article examines the ways in which thermophysical deformation of lithium-ion batteries can lead to explosions and other safety events and then provides a brief review of characterization methods to assess the behavior and results of such deformations. Finally, a recent example of an event “in the wild” is discussed and the mechanisms covered are applied to competing diagnoses of the failure.

Introduction

Between 2010 and 2020, secondary or rechargeable battery production increased by a factor of 50, and costs decreased by a factor of six, with average battery cell prices near \$USD100/kWh and battery pack prices below \$USD140/kWh.¹ A contributing factor to the decreasing capital cost of batteries has been the increase in energy density of batteries,² of which the usable capacity has increased by a factor of three in the same time period. This is due in large part to the safe implementation of anodes of graphite with increasing amounts of silicon compounds added (Gr-Si), and cathodes of layered lithium-metal oxides with increasing nickel and decreasing cobalt content with either manganese (NMC) or aluminum (NCA) stabilizing atoms, as well as cathodes of lithium-iron phosphate (LFP).

It may be surprising to the casual reader with a background in thermodynamics that lithium-ion batteries of increasing energy density have had relatively few publicly reported incidents of explosions, and more importantly, fewer deaths still.³ It may be more surprising to a lithium-ion expert that time travelled to the present from the year 2000 to see relatively low safety incidents.⁴⁻¹⁰

The US Occupational Safety and Health Administration (OSHA)¹¹ indicates that a safe battery should be a battery with (1) deterministic behavior of a cell in (2) a well-defined environment. If either of these two conditions are violated, then the

battery is not safe and should not be used. Standards setting and certifying organizations such as Underwriters Laboratories (UL) act as a nexus for application safety requirements and device physics and build recommendations to satisfy both.¹²

In this article, the multimodal physical and thermal measurement improvements of the last decade are explored, and discussed are how these methods enabled, *ex situ*, *in situ*, *operando*, and on-line, the improvement of nameplate energy density at decreasing costs without an increase in safety incidents. To do so, we explore a few of the known and understood critical failure mechanisms of lithium-ion batteries, and the methods that have been developed to check and ensure that these conditions do not exist in a cell after manufacture and in use.

What can go wrong in a battery

This article will focus on thermal and mechanical inconsistencies that lead to damage, which can then lead to the autocatalytic thermal runaway of batteries. Because batteries are composed of mutually reducing and oxidizing components, there are considerably biotoxic, carcinogenic, and mutagenic components in both the manufacture of batteries and the final product.¹³⁻¹⁷ This article will not discuss the toxic aspects of batteries beyond this: you should not eat batteries, and a ruptured battery should be physically isolated and delivered to a proper disposal facility as soon as possible.

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Mechanical damage to a battery that does not cause direct rupture of the cell packaging nonetheless has safety consequences. Per the OSHA definition of safety, this mechanical damage can alter the deterministic behavior of the battery and render it unsafe. Liu et al.¹⁸ provide a comprehensive review of mechanical damage loops in batteries; what follows is a brief overview to map cause–effect loops. It is by no means exhaustive, but intended to give the reader a framework to assess why mechanical and thermal damage are mutually reinforcing to batteries and can lead to thermal runaway events.

A closed-form electrochemical energy cell (“battery cell”) consists of a reducing agent (the “anode”), an oxidizing agent (the “cathode”), a medium to allow for the transport of ions to the surface while blocking electrons (the “electrolyte”), and a medium for transporting electrons to and from a surface, while keeping the mass constrained and contained within a cell (the “current collectors,” the “external wiring bus,” and the “load”). The ionic current is translated to the electric current at the electrode interfaces via electrochemical reaction, and complimentary reduction and oxidation reactions are required at the anode and cathode to balance the mass and charge transfer within the system. The only regions where, by design, there are simultaneous ionic and electronic current are the porous electrodes, which have physically overlapping regions of reductant and electrolyte, and oxidant and electrolyte (**Figure 1a**). This pattern may be repeated in reversing sequences many times per **Figure 1b**.

If the battery geometry is deformed in such a way that any of the described operations are hindered and/or altered, the application of current to the battery (via charge or discharge) can lead to an unexpected rate of heat generation in the cell which can then trigger thermal runaway. Next are a few examples of how deformations are instigated and triggered during both standard operation of batteries and within “abusive” conditions.

An external short circuit (ESC) drives the potential difference of the current collectors to near zero. Most lithium-ion batteries operate between 2.7 and 4.2 V, and as a result, at any state of charge, the cell feels a driving force to try to further equilibrate such that the reductant and the oxidant reach the same chemical potential. The consequences of this

are instant heat generation within the cell, and given enough time (enough being seconds to minutes), gas generation can occur as a result of both heating of the liquid electrolyte as well as electrochemical oxidation and reduction of the electrolyte.^{19,20} Additionally, since the positive electrode is asking for current from a negative electrode that is depleted or near depleted of lithium, depending on the electrode/current collector design, copper can electro-dissolve to copper ion from the negative current collector.²¹ Under significant enough ESC, the external heat generated can push the battery into an oxidant-driven thermal runaway (i.e., thermal decomposition of the metal oxide positive electrode) if the heat generated raises the temperature sufficiently before enough lithium ion has returned to the positive electrode upon discharge. A lithiated (i.e., discharged) metal-oxide cathode is far less prone to thermal runaway, as the lithiated metal oxide has a thermal reduction temperature $>600^{\circ}\text{C}$.²²

It is critical to note that heat is also generated in the conductor external to the cell creating the ESC. Regardless of the volatility and stability of the cell components, if the ESC is physically allowed to continue, heat is generated as:

$$\dot{Q} = I^2 R \quad (1)$$

If that heat cannot be sufficiently dissipated, the external cell temperature will rise according to the sensible and latent heats of the environment. Many, if not most, battery fires are triggered by a failure to manage this heat generation.

If the cell survives the ESC, and the ESC was not detected nor heeded, once the battery goes into charge mode, copper will likely deposit on the negative electrode as a mossy or dendritic film instead of redepositing uniformly on the negative current collector. Given the approximately $15\ \mu\text{m}$ separating the negative and the positive electrode, this copper metal filament can create a non-penetrating internal short circuit (NPISC) (often referred to as an internal short circuit or ISC, the difference in this article will be elaborated shortly).^{23–26} Unlike an ESC, a NPISC cannot be (readily) eliminated by removing the short. Also unlike an ESC, which is assumed to have almost zero resistance, the non-penetrating internal short circuit can have significant resistance (e.g., it will not always drive the cell potential to zero).

Depending on the nature of the short, if the impeding metal is thin enough and/or of a small enough cross section when touching both electrodes, it may disconnect itself through chemical oxidation or mechanical shifts. These events are referred to as “soft shorts” and may appear in a voltage signal as momentary dips or noise.^{27,28} If the short-circuit metal is sufficiently large and chemically robust, it will permanently bridge the positive and

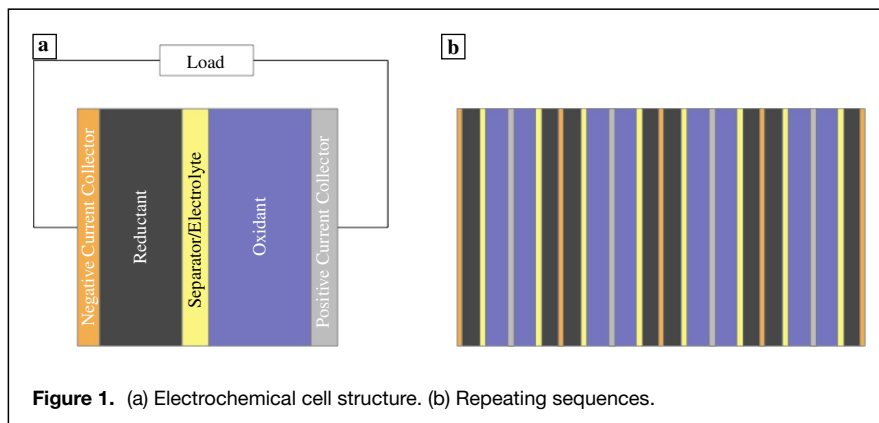


Figure 1. (a) Electrochemical cell structure. (b) Repeating sequences.

negative electrode and will continually discharge the battery internally. The combination of volatile organic electrolytes, thermally unstable metal oxides, and sub-20- μm separator differences exacerbate potential safety triggers.

The chemical nature of the metal filament in a NPISC has a significant impact on the potential danger it creates. Beyond the copper case previously discussed, a well-monitored trigger is iron filament from manufacturing, as well as ferric or ferrous ions left unwashed on cathodes before initial charge.^{23,29} Ions of copper and iron will be drawn to the negative electrode upon charge, but once plated, they are galvanically protected by the active lithium ion in the system until a zero volt event, which is to be avoided for reasons detailed above.

Given the small separator gap, a simple “pinch short” can be the NPISC. Pinch shorts can be the result of poor cell manufacturing processes, damage during cell to battery packing, or unexpected impact (e.g., car crash, dropping a cell phone).^{25,30}

Finally, a penetrating internal short circuit (PISC) represents a foreign body creating an electrical short circuit within a cell. The canonical example of this is the nail penetration test, in which a nail is driven through a battery to emulate its behavior during an NPISC. While the nail penetration test is a facile way to test cell response, it is sufficiently different from NPISCs such that it should not be used as a sole estimate of a cell’s NPISC response.^{25,27,31} For example, the cross section of a nail is far larger than that of an internal filament. While the electrical conductivity of this nail is high, the thermal conductivity is high as well, and the nail is connected to the outside world.

Charging is also a heat-generating event, and the localized heat of a poorly monitored or designed charging system can lead to overheating at the positive electrode during charge. Since the positive electrode is lithium-depleted during charge, it undergoes thermal reduction at a lower temperature. Thus, the coupling between heat generation during charge and thermal runaway is positive, since the lithium is leaving rather than entering the electrode. Upon overcharge, electrolyte gassing due to electrochemically driven redox^{19,32,33} must be considered as well. Fast charge of batteries has been shown to deposit lithium metal upon the graphite electrodes (instead of intercalated into the graphite electrodes).^{34–36} The safety consequences of this lithium metal within the battery are still being studied, it is understood that this lithium metal is not designed to be within the battery, however, and therefore treated as “unsafe.”

Finally, the last damage mechanism discussed here will be, broadly, disconnection, where components within the battery are physically isolated rather than connected. Isolation of particles electrically can be caused by physical damage, and isolated surfaces can be created because of gassing due to the previously mentioned overdischarge and overcharge reactions. The danger of disconnection is complementary to the danger of the “over-connection” of the various short circuits. In batteries, charge rates are normalized internally

to current densities, but disconnection events are inherently heterogeneous, as a result, the current density becomes non-uniform. In turn, a 1 C charge rate globally may mean a 10 C charge rate locally, and the safety concerns for overcharge apply.³⁷

A brief, incomplete survey of thermophysical analyses relevant to battery safety

Since thermophysical couplings lead to battery safety events, and the couplings are difficult to fully normalize across different form factors of cells, the field has developed a variety of methods to measure thermal and physical behavior of commercially relevant cells.

Thermal methods

Calorimetric methods may be the oldest form of physical cell analysis, but they are still critical for measuring battery behaviors. Differential scanning calorimetry (DSC) is a common method for identifying the point of thermal runaway for many lithium-ion cathodes as well as understanding phase behavior of individual components.^{6,38–40} DSC measures the difference in the amount of heat added or removed to change the temperature of an experimental sample in comparison to a reference of well-defined heat capacity. DSC is particularly interesting for measuring the impact of surface and structural enhancements to prevent unwanted phase changes and exothermic events, since the components in the DSC coupon are purely chemical driven. DSC chambers, however, are typically small and not intended for *operando* studies of electrochemical systems.

Accelerating rate calorimetry (ARC) methods^{38,41–45} are excellent “big siblings” to DSC in that they are similar to the heat addition to DSC, but does so in a fully adiabatic setting in which the sample is “allowed” to self-heat while the extent of that self-heating is measured as a function of time and temperature. Whereas DSC is useful for understanding predetermined reaction in simulated cell environments, an ARC experiment is useful for emulating the “full cocktail” of physical–thermal–chemical couplings that may occur in a battery as it drives itself to thermal runaway or other degradation modes.

Thermogravimetric analysis (TGA)^{46–48} is typically used to measure material or simulated cell environment (e.g., cathode-in-electrolyte), measuring the mass loss of a sample on a microbalance as it is heated. Since positive electrodes typically lose oxygen (exothermically) as they heat, and electrolytes vaporize or react, to form gases, TGA is an excellent method to quantify the extent of reaction (which can then be correlated to the expected pressure increases within a cell). When combined with spectroscopic methods, the gaseous compounds can be further classified and/or quantified depending on the methods (discussed in more detail next).

While typically not used to measure critical safety events directly, isothermal micro- or nanocalorimetry (ITC) can be

used on full cells to measure the onset of reaction over longer time scales (minutes to hours) as a function of the fixed temperature and electrochemical operation on commercially relevant cell:^{49–58} specifically, electrochemical and temperature conditions are set by the user, and the resulting heat flow in or out of the cell is measured over time. This thermodynamic data can then be analyzed to assess the nature of reactions occurring. This method is good at ascertaining events that form prior to critical damage in cells.

Chemical methods

Chemical analysis methods have evolved over the last two decades from *ex situ* tools to near real-time *operando* tools capable of measuring complex couplings in full cells. TGA methods combined with Fourier transform infrared (FTIR) spectroscopy have been used to directly correlate thermal events to evolved gas quantity and composition in a variety of systems.^{46,59,60}

Differential electrochemical mass spectroscopy (DEMS) is a cousin to TGA-FTIR, but rather than drive the system thermally, the system is driven electrochemically.^{61–68} As a result, it can directly quantify and classify redox driven off

gassing in addition to thermally driven off-gassing. Rowden and Garcia-Areaz⁶⁹ provide a thorough review of gas evolution analysis methods.

Finally, x-ray fluorescence (XRF),^{70,71} atomic absorption spectroscopy (AAS),^{72–74} inductively coupled plasma (ICP),^{75,76} and scanning electron microscopy with energy-dispersive spectroscopic methods (SEM-EDS) are industrial and academic work horses that classify impurities in samples, both electrode powders samples as well as finished electrodes. Water content in lithium-ion batteries must be kept to an absolute minimum for both operational and safety reasons; Karl Fischer titration is the standard tool for assuring water content is sufficiently low.

Structural methods

Just as crystal structure analysis and accompanying diffraction methods are bedrock methods for understanding equilibrium and desired performance aspects of battery materials, these methods are important for safety consideration, particularly when combined with thermal characterization in *operando* experiments. For example, time-resolved x-ray diffraction (TR-XRD)¹⁰ in conditions relevant to thermal runaway of

nickel-rich cathodes was done to understand the coupling of structure change and oxygen gas generation (Figure 2). Recently, localized TR-XRD methods have been employed to spatially map structural changes in cathodes⁷⁷ as well as unwanted lithium deposition within lithium-ion batteries.

The small x-ray cross section of lithium metal makes x-ray analysis difficult. These recent methods are testaments to the ability of scientists to maximize signal from noise. The tools have lower availability; neutron diffraction and absorption methods have been used frequently to study the impact of lithium metal on lithium-ion batteries, as well as structural changes across full-format lithium-ion batteries.^{19,78–81} The interpretation of neutron data is more difficult than x-ray data as there are less prior data available, but the transmissivity of neutrons while being lithium-sensitive makes it a powerful tool for full cell analysis.

Optical imaging methods

While visible optical imaging of batteries yields little direct data, clever *in situ* mock cells allow for

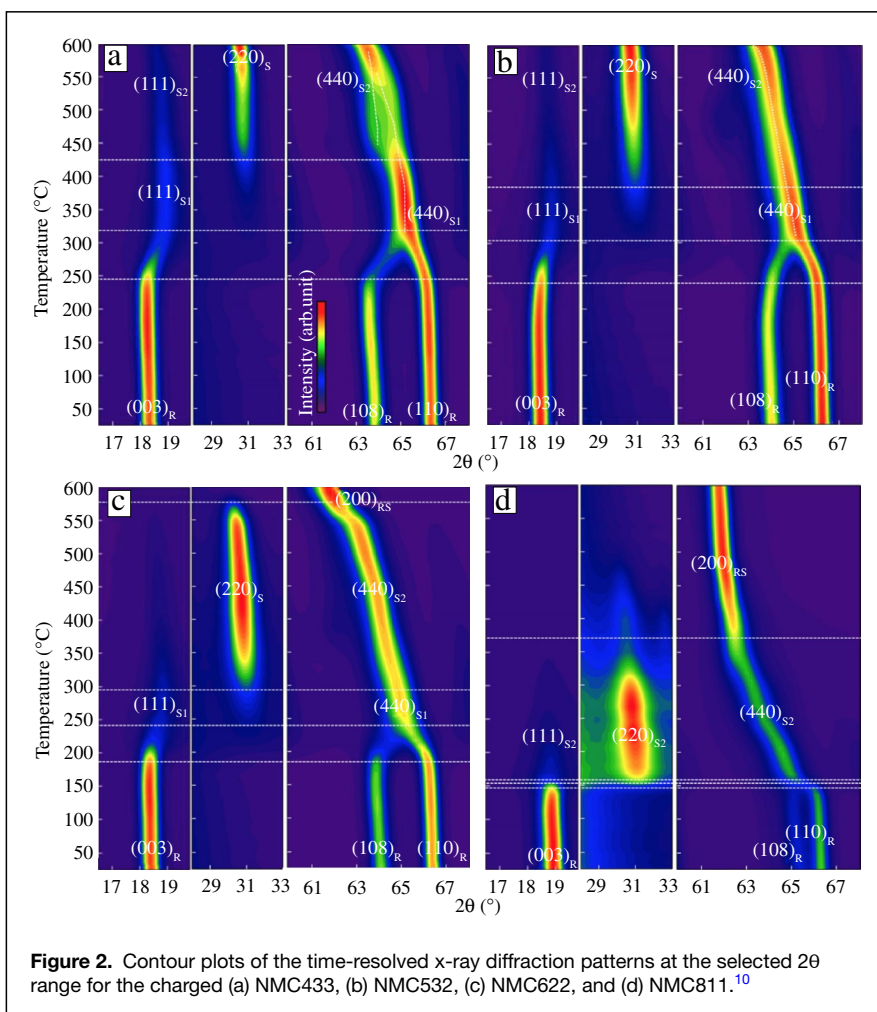
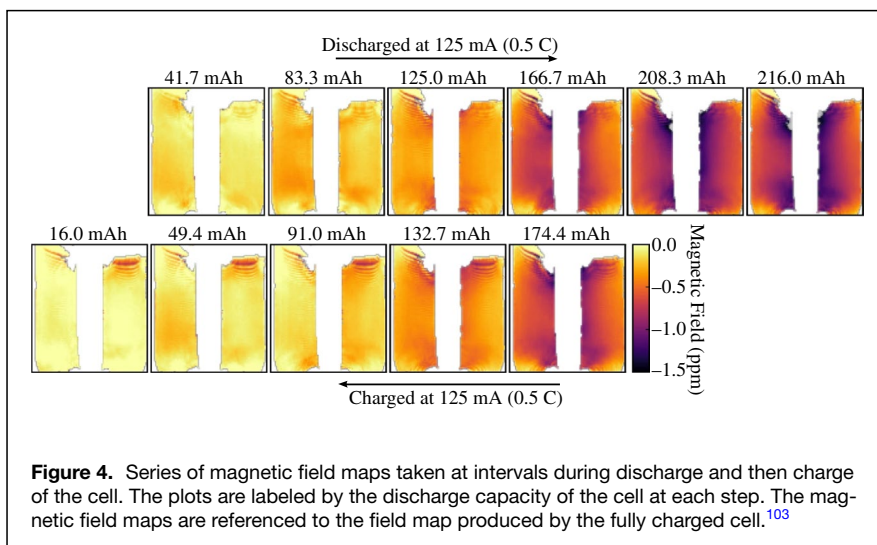
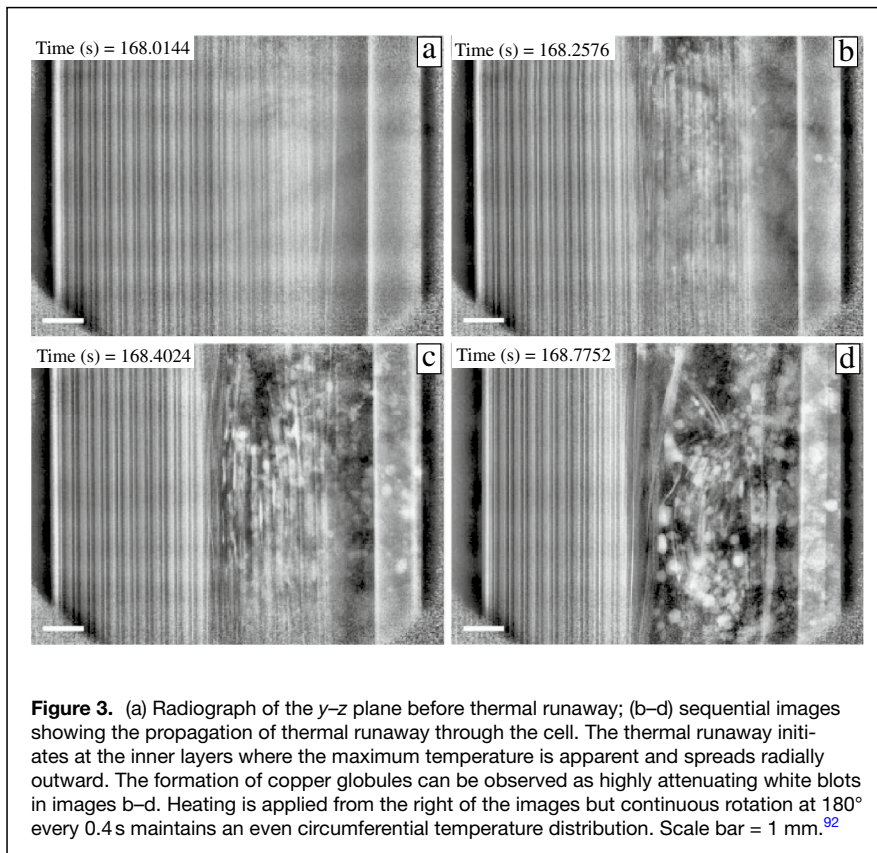


Figure 2. Contour plots of the time-resolved x-ray diffraction patterns at the selected 2θ range for the charged (a) NMC433, (b) NMC532, (c) NMC622, and (d) NMC811.¹⁰



undercomponent-level understanding. For example, as graphite lithiates, it changes in color from black to gold, with specific hues indicating stages,^{82,83} and this can be exploited to examine strain and heterogeneous behavior in cells.^{84,85} Again, heterogeneous behaviors in batteries are often the root cause of safety events, so methods such as this allow researchers to foresee and develop preventive measures to avoid such needle-in-haystack problems. Video rate methods have also keyed into key challenges in current distribution and metal

detachment for metal (Li, Zn, etc.) anode systems problems that can presage disconnection-related safety issues.⁸⁶⁻⁸⁸

In the last decade, x-ray and neutron tomography in near real time has been made available, and with x-rays almost to the laboratory scale. Rates of greater than 10 Hz are available, and this allows researchers to connect lower-resolution maps of the previously mentioned phenomena to full cells, creating a “zoomable map” in four dimensions.⁸⁹⁻⁹¹ A particularly dramatic example by Finegan and co-authors is the examination of a battery undergoing thermal runaway in real time.⁹² This example illustrated (Figure 3) not only where the runaway starts, but how the heat spreads and how cell design can accelerate or mitigate runaway events.⁹³

Magnetic methods

Since metal objects of various sizes can wreak havoc on a lithium-ion battery, and the lithium-ion positive electrode is a collection of valence-changing materials, magnetic interrogation of cells is natural, and similar the other methods described, there are complementary component-level analyses as well as full cell methods available. Lithium-metal behaviors in full cells have been examined via nuclear magnetic resonance (NMR) methods in imaging mode.⁹⁴⁻⁹⁷ Electrolyte behaviors, particularly decomposition modes, can be teased out of NMR data as well.⁹⁸⁻¹⁰²

Recently NMR imaging methods (nmMRI) have been applied in clever ways to large-format batteries by Jerschow,^{103,104} and maps

can be completed quickly enough to image extent of reaction as a function of space, and thereby extract current density (Figure 4).

Mechanical methods

The mechanical correlations of reversible electrochemical reactions have been studied for as long as batteries have been in field use, but the last decade has seen a

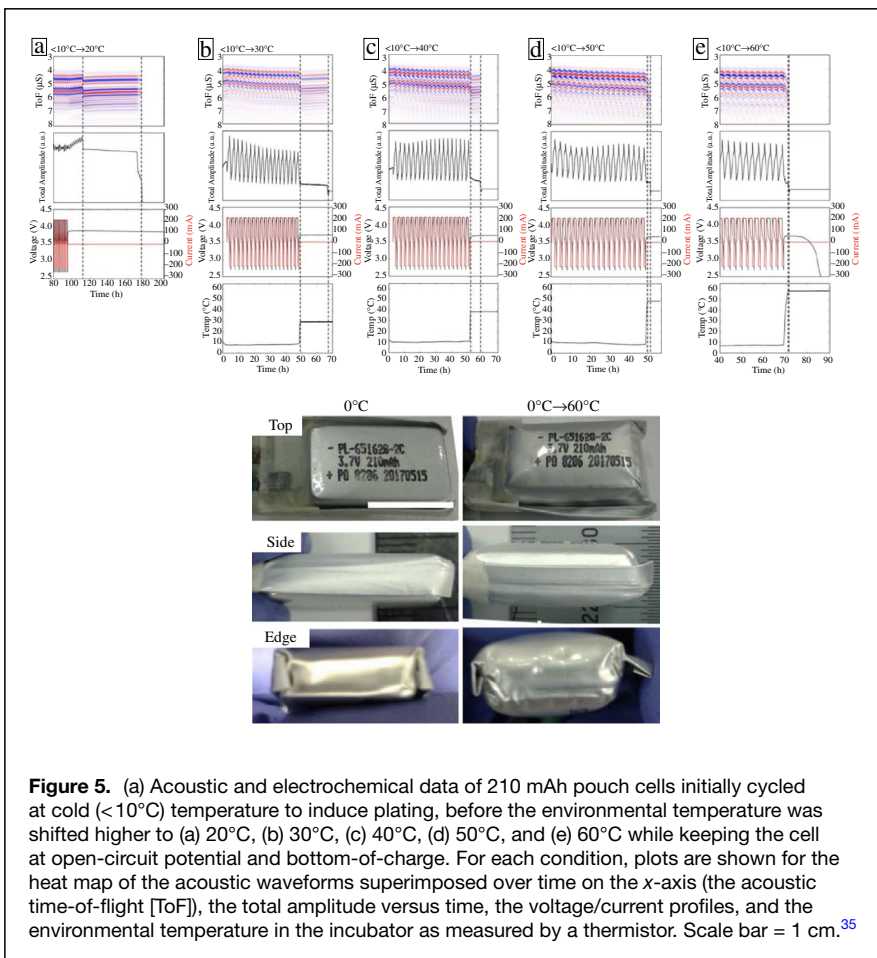


Figure 5. (a) Acoustic and electrochemical data of 210 mAh pouch cells initially cycled at cold ($<10^{\circ}\text{C}$) temperature to induce plating, before the environmental temperature was shifted higher to (a) 20°C , (b) 30°C , (c) 40°C , (d) 50°C , and (e) 60°C while keeping the cell at open-circuit potential and bottom-of-charge. For each condition, plots are shown for the heat map of the acoustic waveforms superimposed over time on the x-axis (the acoustic time-of-flight [ToF]), the total amplitude versus time, the voltage/current profiles, and the environmental temperature in the incubator as measured by a thermistor. Scale bar = 1 cm.³⁵

For example, Chang et al. showed the progression of lithium-metal deposition to dead lithium to gas formation in a multilayer stack.³⁵ In this case, the lithium metal showed itself not to be a danger because of rapid heating during a short circuit, but rather, because the reactive lithium metal is not stable in the context of the lithium-metal electrolyte, which causes excess chemical gassing and leads to physical disconnection (Figure 5).

Conclusion and case study in these methods

The preceding descriptions of thermophysical safety challenges for lithium-ion batteries and methods to assess these dangers are in some part responsible for the relatively low number of battery safety incidents that have occurred despite the significant increase in high energy density lithium-ion batteries over the last decade. But as mentioned, safety is a measure of what does not happen, and engineering for safety requires statistics and event analysis in equal proportion to hypotheses and fundamental understanding.

The former qualities can be difficult to access for academic battery researchers, as liability and confidentiality often accompany analyses of real-world safety events. A fire on April 19, 2019, at the APS McMicken Battery in Surprise, Arizona, is unusually openly documented for a large-scale battery-safety event. An entire battery module was destroyed, and several firefighters were injured when combating the fire, and two teams of battery-safety experts analyzed immolated cells with methods such as those previously mentioned to

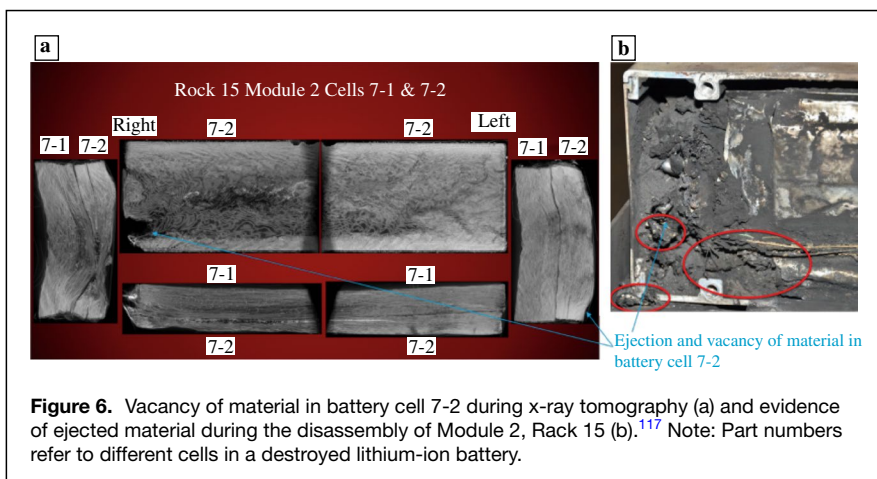


Figure 6. Vacancy of material in battery cell 7-2 during x-ray tomography (a) and evidence of ejected material during the disassembly of Module 2, Rack 15 (b).¹¹⁷ Note: Part numbers refer to different cells in a destroyed lithium-ion battery.

distillation of practical know-how to scientific understanding. Stress-strain relationships for lithium and lithium-ion systems have been extensively studied with classical tools of physical metallurgy,¹⁰⁵⁻¹¹¹ and recently, acoustic analysis of such systems has revealed similar information in addition to structural mappings.^{35,36,112-116}

piece together what may have happened.

An analysis by DNV-GL¹¹⁷ suggested that a possible trigger for thermal runaway was a significant amount of plated lithium that formed a hard short in the lithium-ion cell. Figure 6 shows an x-ray tomograph of a failed cell, where missing material is considered evidence of ejecta

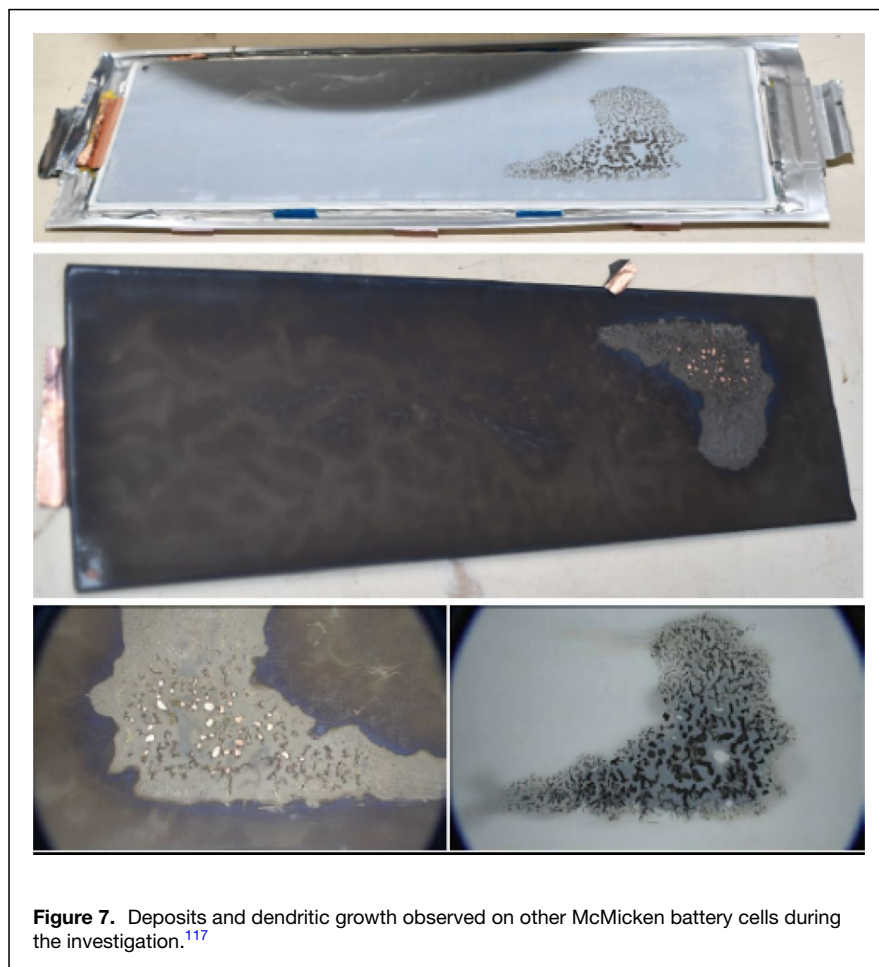


Figure 7. Deposits and dendritic growth observed on other McMicken battery cells during the investigation.¹¹⁷

from a fire triggered by internal thermal runaway. **Figure 7** shows a cell that was not destroyed, displaying significant evidence of lithium-metal deposition in the lithium-ion battery.

However, a second report on the incident by Exponent questioned the root cause analysis by DNV-GL,¹¹⁸ questioning the ability of lithium metal to persist long enough in a short-circuit configuration before oxidizing, thus self-limiting instead of allowing enough heat to be generated to cause thermal runaway. The Exponent report hypothesized that an ESC from a misconfigured wiring bus started the heating event which then triggered the cell rupture which then led to a fire. In short, the second theory is that the heat came from outside the cell.

As of the writing of this piece, the root cause has not been publicly agreed upon, but the reader is strongly encouraged to read both reports to see how the methods outlined here are applied in practice, and to develop an appreciation of how well lithium-ion batteries have been “hardened” such that events such as this are few and far between. While method development, specifically for online analysis, needs to be improved so events such as McMicken can be studied in real time, the tools for assessing thermophysical

correlations within battery are sufficiently developed such that forensic engineers can begin to piece together and learn from battery safety events to ensure future systems do not suffer the same fate.

Conflict of interest

On behalf of all authors, the corresponding author states that there is no conflict of interest.

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