



X-ray computed tomography comparison of individual and parallel assembled commercial lithium iron phosphate batteries at end of life after high rate cycling

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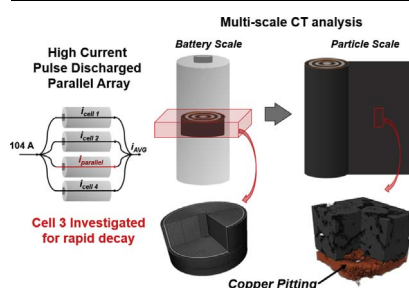
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HIGHLIGHTS

- X-ray CT is used to detect differing end of life mechanisms for commercial LFP cells.
- Rapidly decayed, parallel assembled cell shows evidence of copper dissolution.
- Carefully impedance matched cells assembled in parallel are at risk of expedited failures.
- Three different length scales of X-ray CT diagnostic analysis are demonstrated.

GRAPHICAL ABSTRACT



ARTICLE INFO

Keywords:

Computed tomography
Micro-CT
Battery degradation
Li-ion battery
Pulsed power
Copper dissolution
Overdischarge

ABSTRACT

X-ray computed tomography (X-ray CT) across multiple length scales is utilized for the first time to investigate the physical abuse of high C-rate pulsed discharge on cells wired individually and in parallel. Manufactured lithium iron phosphate cells boasting high rate capability were pulse power tested in both wiring conditions with high discharge currents of 10C for a high number of cycles (up to 1200) until end of life (< 80% of initial discharge capacity retained). The parallel assembly reached end of life more rapidly for reasons unknown prior to CT investigations. The investigation revealed evidence of overdischarge in the most degraded cell from the parallel assembly, compared to more traditional failure in the individual cell. The parallel-wired cell exhibited dissolution of copper from the anode current collector and subsequent deposition throughout the separator near the cathode of the cell. This overdischarge-induced copper deposition, notably impossible to confirm with other state of health (SOH) monitoring methods, is diagnosed using CT by rendering the interior current collector without harm or alteration to the active materials. Correlation of CT observations to the electrochemical pulse data from the parallel-wired cells reveals the risk of parallel wiring during high C-rate pulse discharge.

1. Introduction

Common battery failure mechanisms due to cell overcharge and

overdischarge are difficult to detect with battery management systems (BMS) and state of health (SOH) monitors [1–3], especially in the common Li-ion chemistry, lithium iron phosphate (LFP), which exhibits

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an extremely flat reaction plateau. The minimal change in voltage over a cycle makes determination of state of charge (SOC), an important parameter of cell monitoring, difficult. However, this chemistry is quite attractive for its cell safety and high rate capability, causing it to gain popularity among a range of applications, including grid storage and pulsed power [4].

For high power applications the LFP chemistry is most commonly selected, as a result of its high power performance and promising long cycle life. The chemistry has demonstrated extensive cycle-life, far surpassing 1000 cycles in normal operating conditions [4] (moderate current densities and ambient temperatures). However, if voltage slipping occurs, overcharge or overdischarge will be hard to detect. As the electrodes degrade the reaction potentials shift relative to each other allowing reactions in proximity to the conventional behavior to occur [5]. At the graphite anode, utilized in most Li-ion chemistries, the lithium plating reaction occurs close to (within 100 mV) graphite intercalation on charge, meaning that in overcharge, lithium deposition at the graphite anode can cause a catastrophic lithium short [6,7]. Additionally, the slightly lower operating voltage of the LFP system compared to systems with other cathodes, threatens copper short with overdischarge, as a result of, the proximity of the lithiation potential to the oxidation of the copper current collector (also within ~ 100 mV) [5,8,9]. The risk of these metallic shorts from voltage slip are heightened when operational limits are pressed. Pulsed power applications typically demand ultra-high currents (10C, 26 A) and complex parallel wiring assemblies, which force current sharing for voltage matching [10,11]. The safety risk of these conditions, therefore, requires extensive testing and demands highly effective BMS and SOH techniques capable of proper risk assessment and mitigation.

Non-destructive state of health monitoring techniques include derivative capacity [12,13], AC impedance [14,15], and acoustic response [16], which all require complex signal analysis specific to cell chemistry and configuration that can be time consuming and lack visual confirmation. Medical diagnostic techniques, like X-ray imaging, boast promising capabilities likely valuable to batteries, not only for SOH monitoring, but also for manufacturer quality control. This technique has been leveraged for the development of fuel cells [17–19] and other energy storage [20] and conversion [21,22] applications. Specifically for batteries, several groups have utilized this technique for the characterization of composites [23–27] and electrode fabrication [28,29], as well as the examination of lithiation/delithiation behavior [30–33]. Further, the technique has been used to observe catastrophic battery failure or thermal runaway initiated by cell heating [34] or puncture tests [35]. Herein, we utilize micro X-ray computed tomography (CT) for the first time to diagnose cell failure after extensive cycling, resulting in two distinct end of life scenarios.

We examine commercial LFP cells (LiFePO₄ cathode and graphite anode) electrochemically cycled under pulsed, high current discharge until end of life (EOL) where $\leq 80\%$ of initial discharge capacity). The pulse testing delivers a series of 10C discharge currents over short time periods separated by short rests and followed by conventional recharge. This type of cell abuse presents a unique avenue for failure diagnostics, where end of life is anticipated earlier than manufacture rating. Under this unique abuse, degradation can be probed in various wiring configurations (single, series, and parallel). Our investigation seeks to elucidate the expedited decay of the parallel assembly compared to the individual cell.

2. Experimental details

2.1. Electrochemical testing

An initial population of 180 cells, all of which were all newly obtained from the manufacturer, was cycled according to the Dunbarry et al. procedure [36]. This initial testing allowed 4 impedance (± 0.1 m Ω) and capacity ($\pm 0.2\%$) matched cells to be selected for parallel

testing in an attempt to eliminate cell-to-cell variation from the degradation mechanism. These cells were installed into the parallel test bed outlined in prior work [15] and the individual and parallel assembly were subsequently pulse power tested until EOL using a MACCOR Series 4000 computerized battery analyzer. Temperature, current, and voltage were logged for each cell.

The cells were initially pulsed 50 times with a 10C discharge current for 4 s followed by a rest of 2 s. While these discharges appear to be relatively shallow, they are intended to be representative of a real system under test for US Navy application. The cells were then discharged to 2.0 V at 1C and fully charged to 3.5 V at 1C. A new set of 50 pulses follows. After 25 pulse and recharge cycles are completed, state of health was assessed.

The cells were fully discharged and charged in the same manner as initial testing [36] and capacity retention was assessed. If cell capacity dropped below 80% of initial delivery, end of life was declared and the cycle was terminated. Otherwise, the cells were then charged to 50% SOC and each cell had an electrochemical impedance spectroscopy (EIS) sweep performed from 10 kHz to 0.1 Hz, with a 10 mV perturbation amplitude using a Solartron Analytical ModuLab XM system. The cell(s) were then fully charged to 100% SOC and pulse testing was initiated again. The entire cycle was automated by the ModuLab software.

2.2. Cell opening

An untested cell, the individual cell and each of the cells from the parallel assembly were fully charged and discharged according to manufacturer's recommendation, C/2. The parallel cell exhibiting the shortest discharge or lowest capacity was selected. The three cells were disassembled in a battery dry room with dew point $< 0^\circ\text{C}$ and temperature $\sim 20^\circ\text{C}$. A small Dremel tool with abrasive cutting wheel, additional photos provided in supporting information, was used to carefully score and weaken the case around the circumference at the edge of the cylinder close to the positive button. When the end began to detach from the case, the piece was carefully removed. The 4 positive tabs welded to the top were then carefully snipped with pliers, deactivating the cell. The cell was then carefully cut in the same fashion at the other end and the negative leads cut. The cell jelly roll was then carefully removed and unrolled. The cell was carefully observed and photographed, and small pieces ($\sim 2 \times 4$ mm) were cut from the middle of each electrode for tomographic investigation.

The acquired pieces were transferred to an Ar-filled glove box and rinsed in diethyl carbonate (DEC, anhydrous, $> 99.9\%$ sigma Aldrich) to eliminate electrolyte salt and solvent residue from tomographic analysis. The rinsed electrodes were vacuumed dried overnight.

2.3. X-ray computed tomography

Three-dimensional imaging of batteries was done with lab-scale Zeiss Xradia 520 Versa X-ray microscope (Carl Zeiss X-ray Microscopy, Pleasanton, CA, USA). The detection system consisted of a scintillator coupled to a 16-bit CCD. X-ray projections were collected over 360° rotation range and reconstructed with Zeiss XMReconstructor proprietary software using Feldkamp-Davis-Kress (FDK) algorithm. Battery and electrode-scale imaging were conducted with $0.4\times$ objective lens, resulting in voxel sizes of 9.77 and 2.37 μm , respectively. Particle-scale imaging was conducted with $4\times$ objective, resulting in voxel size of 218 nm. Image stack set was converted to 8-bits and imported into open-source Fiji/ImageJ software [37]. After image manipulation, such as cropping, rotation and translation, manual thresholding was performed to separate void and solid phases. Avizo 8.1 was used for volume rendering and visualization.

2.4. Tortuosity computation

Geometric tortuosity was calculated on direct volumetrically

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