

Fast-Charging Capability Limits of Electric Vehicle Battery Chemistries

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The fast cycling capability of Li-ion batteries is a key performance indicator for a range of applications including electric vehicles (EVs), in which systems must deliver/accept high currents while avoiding excessive degradation. We evaluate four leading EV battery chemistries in this context, encompassing cathodes focused on design-to-performance, $\text{LiNi}_{0.8}\text{Mn}_{0.1}\text{Co}_{0.1}\text{O}_2$ (NMC811), and design-to-cost, $\text{LiMn}_{0.6}\text{Fe}_{0.4}\text{PO}_4$ (LMFP), alongside current state-of-the-art (graphite and graphite@ SiO_x composite) and next-generation (high SiO_x) anodes. These chemistries are adopted as model systems to examine three common test protocols for evaluating fast cycling, specifically tuned to fast charge, fast discharge, or both. The capacity, degradation induced, and operational state-of-charge range were highly dependent on selected testing protocols. Significant loss of lithium inventory (LLI) was observed under fast-charging conditions, associated with lithium plating, observed via monitoring of anode potential in three-electrode cells, differential voltage analysis, and visual inspection of electrodes postmortem. LMFP cells induced a greater level of Li plating than NMC, reaching negative anode potentials in the early stages of fast charging. While the SiO_x anode performed relatively well at high rates, it suffered a greater degree of degradation, including both LLI and impedance increase associated with excess SEI growth. This study provides a basis for battery material design and charging protocols tuned for fast cycling.

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I. INTRODUCTION

Vehicle charging/refueling time is an area in which electric vehicles (EVs) continue to lag those based on internal combustion engines (ICE)—while an ICE vehicle can be refueled in < 5 min, EV Li-ion battery (LIB) systems typically require longer charge times (15–60 min) and often with a trade-off between charge time and capacity. This represents a significant challenge for consumer attraction and a significant added cost factor for commercial-use EVs with demanding duty cycles. It is little wonder that addressing this has attracted the attention of researchers in both academia and industry, and rapid charging in under 15 min has become an industry-wide goal [1–6]. In contrast, for certain other LIB applications, e.g., power tools, it is fast discharge capability that is critical, and a slow charge may be acceptable [7].

The factors limiting charge/discharge rate include those at the infrastructural level (capacity of charging station),

battery system level (current capacity of busbars), and battery cell level (determined by material choices and design) [2]. During charging (and indeed discharging), a cell becomes polarized, i.e., cathode and anode potentials deviate from their equilibrium values. If this deviation is sufficiently high, it can lead to electrode heterogeneity and drive potentials to unsafe values, e.g., outside the stability window of the electrolyte [8], into the oxygen loss regime of layered oxide cathodes [9], or inducing Li plating on the anode [10–12]. As the level of polarization is directly proportional to charge current, this effectively limits how quickly a cell can be charged. Often, design trade-offs are needed, whereby energy density is sacrificed for better fast-charge capability, utilizing, e.g., low electrode coat weight, low active material loading, highly porous electrodes, and extra/thicker tabs [13,14]. Similarly, active materials that provide high-energy density (e.g., Si) often do not provide good fast-charging capability and vice versa (e.g., lithium titanate) [15].

The design of optimized fast-charge strategies is an active area of research, with techniques such as preheating, pulsing, and combinations of constant current/power/voltage proposed [16–18]. As fast charge/discharge requirements vary with application, and capabilities vary with cell material/design, it is important to establish the key performance characteristics of any such combination. This is most often carried out in academia via the rate capability test, whereby

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cells are cycled at increasing rates sequentially [2,15,19]. Extracted capacity typically falls at higher rates, even dropping to near zero above a certain threshold rate. However, while such approaches can provide a general impression of fast cycling capability, they fail to link directly with use cases, nor do they typically examine degradation induced by such fast cycling [4,20]. A more meaningful, application-directed assessment would provide a greater level of predictive power, enabling more focused material/cell design rules and more successful fast-charge strategies. Alongside this, it is highly valuable to detect breaches of safe working limits during fast cycling via electrochemical means alone (i.e., without the need for time-consuming cell tear-down). This approach can thus be utilized in battery management systems to optimize fast charging in response to system state-of-health and operational conditions.

However, in standard Li-ion cells, only the full cell potential is detectable, while individual cathode/anode potentials are not, and thus it is difficult to determine electrochemically when these safety regimes have been breached. In practice, this leads to overly conservative fast-charge protocols and longer charge times [21]. The three-electrode (3E) cell configuration (using Li as a reference electrode) addresses this issue, as it enables the monitoring of individual cathode and anode potentials (alongside that of the cell) [22–24]. In this format, the identification of features associated with degradation events (such as anode potential < 0 V vs Li^+/Li) is straightforward. While the addition of reference electrodes to real cells is challenging and involves significant modification [25,26], 3E cells at lab scale enable identification of likely degradation events and their correlation with cycling protocols. This enables both the design of safe fast cycling protocols along with *in situ* monitoring via battery management systems to detect and prevent unsafe or degradation-inducing events during such cycling [27].

In contrast with most studies which examine a single cathode/anode pair, here we manufacture 3E cells using four popular combinations, encompassing two popular industry cathodes, LMFP and NMC811, and three anodes covering the current state-of-the-art (graphite and a graphite: SiO_x blend [22]) and next-generation (pure SiO_x). LMFP, in particular, is a relative newcomer to commercial batteries, and its fast cycling characteristics are largely unknown (though suspected to be poor due to low Li^+ diffusion) [2,28,29]. The fast cycling capability of these pairings is assessed and compared using multiple protocols designed for direct applicability to typical use cases, namely those placing importance on either fast charging or fast discharging. The measured performance and degradation induced during cycling show significant dependence on both cell chemistry and protocol, highlighting the need for appropriate test regimes and determining optimum chemistry-dependent operational conditions.

II. METHODS

A. Electrode manufacture

All electrodes utilized in this study were manufactured as per the parameters shown in Table I. The electrode manufacturing process was as described in our previous publications [2,23,29]. Briefly, cathode active powders (LMFP, $\text{LiMn}_{0.6}\text{Fe}_{0.4}\text{PO}_4$, Shandong Gelon LIB or NMC811, Ronbay), were mixed with carbon black (CB, Alfa Aesar) and polyvinylidene fluoride (PVDF, Solvay, predissolved in N-methylpyrrolidinone, NMP, Sigma Aldrich), before further dilution with NMP to a coatable viscosity. In the case of LMFP, a 500 g solid mixture of 93 : 3.5 : 3.5 LMFP : PVDF : CB was incorporated into a slurry at a solid content of 46%. For NMC811, a 700 g solid mixture of 95.5 : 2 : 2.5 NMC : PVDF : CB was produced at a solid content of 65%. The slurries were cast onto

TABLE I. Electrode manufacturing parameters. Cathode binder was PVDF while anode binder was SBR + CMC. Areal capacities were based on practical capacities of 160 mAh g^{-1} (LMFP), 190 mAh g^{-1} (NMC), (340 mAh g^{-1}) (graphite) and 1500 mAh g^{-1} (SiO_x). Cathode coat weights (and thus areal capacities) were chosen to align with commercial cells, i.e., higher for NMC and lower for phosphate materials, ensuring industrial relevance [30].

	Cell type	Active material	Active content %	Binder content %	Carbon %	Coat weight (gsm)	Thickness (μm)	Density (g cm^{-3})	Porosity (%)	Areal capacity (mAh cm^{-2})	NP ratio
Cathode	LMFP \ \ Gr	LMFP	93	3.5	3.5	135	65	2.15	36	2.1	
	NMC \ \ Gr	NMC811	95.5	2.5	2	165	47	3.5	23	3	
	NMC \ \ Gr@SiO _x	NMC811	95.5	2.5	2	165	47	3.5	23	3	
	NMC \ \ SiO _x	NMC811	95.5	2.5	2	165	47	3.5	23	3	
Anode	LMFP \ \ Gr	Graphite	95.25	2.25 + 1.5	1	74	53	1.4	37	2.4	1.14
	NMC \ \ Gr	Graphite	95.25	2.25 + 1.5	1	100	71	1.4	37	3.25	1.08
	NMC \ \ Gr@SiO _x	Graphite@SiO _x	85.75 + 9.5	2.25 + 1.5	1	73	52	1.4	37	3.25	1.08
	NMC \ \ SiO _x	SiO _x	70	7 + 7	16	29	21	1.35	33	3.25	1.08

15 μm Al foil (Avocet) to coat weights of 135 gsm (grams per square meter, LMFP) and 165 gsm (NMC811) on a roll-to-roll system within WMG's battery pilot line facility. Anodes were similarly fabricated via coating of aqueous mixtures of active material (graphite powder and SiO_x , BTR), styrene butadiene rubber (SBR), sodium carboxymethyl cellulose (CMC) and CB onto Cu foil (10 μm). The formulation adopted was 95.25 : 1 : 2.25 : 1.5 graphite:CB:SBR:CMC at a solid content of 50%. Electrodes were dried within convection ovens in the roll-to-roll system at temperatures of 95–130 $^{\circ}\text{C}$ (cathode) and 35–40 $^{\circ}\text{C}$ (anode). After drying, electrodes were calendered to the desired densities.

B. Cell assembly and testing

Three-electrode (3E) “PAT” cells were purchased from EL-cell. Discs of 18 mm diameter were punched from the cathode and anode, dried at 120 $^{\circ}\text{C}$ under vacuum for 16 h, and assembled into the 3E cell along with glass fiber separator, a Li reference ring, and 200 μl of electrolyte (1 M LiPF_6 in 3:7 EC:DMC+ 2% VC). Assembled cells were held in an EL-cell temperature chamber at 25 $^{\circ}\text{C}$ during

testing. Cycling was carried out using a Biologic VMP-3 cyclor coupled with EC-lab control software. All cells underwent an initial formation process involving a 10 h wetting stage followed by two cycles at a slow rate of C/20. C-rates were determined based on the practical capacities of each cathode type, namely 160 and 190 mAh g^{-1} for LMFP and NMC, respectively. LMFP // Gr and all NMC cells were cycled in voltage ranges of 2.5–4.3 V and 2.5–4.2 V, respectively. Cycling was carried out using a constant-current-constant-voltage (CCCV) charging strategy, in which the constant-voltage step terminated when current fell below 10% of initial. Full coin cells (2032) were assembled for teardown and *ex situ* inspection, using the electrodes as above (14.8 and 15 mm diameter discs for cathode and anode, respectively), along with a Celgard 2325 separator and 80 μl electrolyte.

III. RESULTS AND DISCUSSION

Figure 1 shows the evolution of cell, cathode, and anode potentials for the four EV-focused cathode/anode combinations studied in this work during a charge-discharge cycle at a rate of C/20. Electrode composition clearly

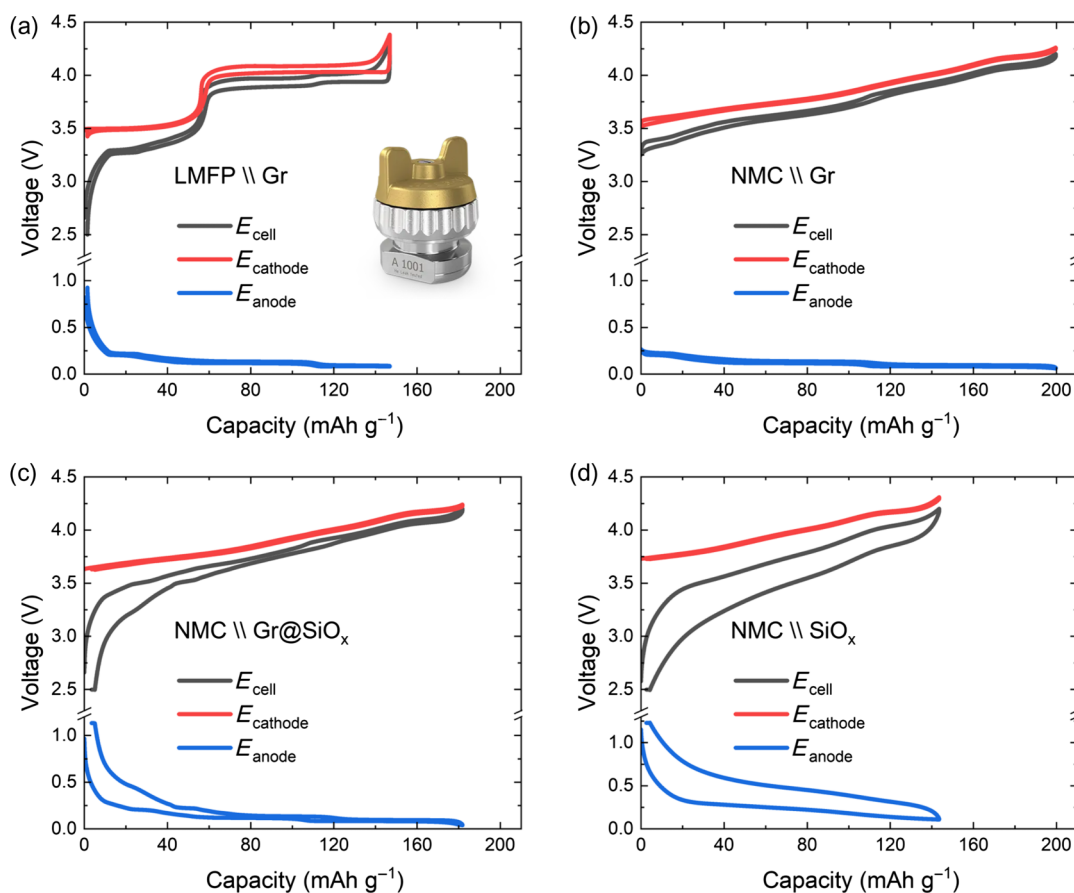


FIG. 1. Voltage profiles of 3E cells of the four battery chemistries examined in this study, (a) LMFP // Gr, (b) NMC // Gr, (c) NMC // Gr@SiO_x, and (d) NMC // SiO_x. Profiles were taken from the second formation cycle during cycling at C/20. Inset in (a) is a picture of the 3E cell. Capacity (mAh g^{-1}) is based on the cathode active mass.

impacted the resultant potential evolution, e.g., LMFP showing a dual-plateau vs NMC's gradual slope and graphite exhibiting a staged profile compared with a higher potential and more sloped profile for SiO_x . Performance was also impacted, as the SiO_x -containing cells suffered from a higher first-cycle loss (FCL) and thus lower subsequent capacity. The increased polarization of the SiO_x electrode (likely including a contribution from open circuit voltage hysteresis [31]) compared with graphite is apparent here [Figs. 1(b) and 1(d), Fig. S1 in Supplemental Material [32]]. The 3E configuration also enables determination of the limiting electrode, e.g., at the top of the charge of the LMFP \(\backslash\) Gr cell [Fig. 1(a)], it can be observed that the graphite electrode is not fully lithiated, and thus the cell is cathode limited (expected given the designed NP ratio of 1.14). Impedance spectra obtained after formation (Fig. S2 in Supplemental Material [32]) revealed superior kinetics for NMC compared with LMFP, consistent with its higher ionic and electronic conductivities [29], while the impact of anode composition on impedance was minimal.

High-rate cycling is expected to be a particular challenge for LMFP chemistries, as this material exhibits low ionic and electronic conductivity compared with other cathode materials (LFP and NMC) [29]. LMFP \(\backslash\) Graphite 3E cells (representing a low-cost cell design) were assembled using industry-style LMFP electrodes (high coat weight of 135 gsm and high active loading of 93%) to ensure commercial relevance. The cells were subjected to rate capability tests using three distinct protocols: (i) "Symmetric," whereby the same constant current was applied during both charge and discharge; (ii) "Fast charge," whereby only the charge current was varied and discharge current was held constant at C/3 throughout the test; and (iii) "Fast discharge," whereby discharge current was varied and charge current held constant. In all cases, a CCCV charging strategy was employed (Fig. S3

in Supplemental Material [32]), in which the CV step terminated when current reached a threshold of 10% of initial. The symmetric protocol represents the most common approach to rate capability testing in the literature [2,9,15,19,33,34], while the other protocols represent more realistic representations of common battery use cases, e.g., fast charge for EVs and fast discharge for power tools. The nature of the protocol with which battery materials are tested would be expected to impact both capacity extraction and degradation induced, and therefore evaluation in appropriate conditions is important. Ultimately, EV battery systems are tested using advanced cycling regimes such as the Worldwide Harmonised Light Vehicles Test Procedure (WLTP, recreating common driving patterns) [35], and state-of-the-art fast-charging strategies are often complex multistage protocols [20,36]. The fast-charge protocol utilized in this study represents somewhat of a middle ground, easy to replicate in typical battery labs while possessing significantly more relevance to the application.

Figure 2(a) shows the capacity extracted using the three protocols. At low C-rate, the capacities were similar for all protocols, i.e., approximately 141 mAh g^{-1} . Notably, a small increase in capacity was observed during the first five cycles at C/3 (e.g., 143.7 to 144.1 mAh g^{-1} for the symmetric protocol), attributed to continued electrolyte wetting unlocking more capacity. At faster rates, however, performance diverged significantly. The common symmetric cycling protocol exhibited the lowest capacity at medium and high rates, while fast-charge and fast-discharge mirrored one another at rates until 10 C, at which point the fast-charge protocol provided the highest capacity (103 mAh g^{-1}). This evolution reflects the limitations on capacity imposed by the different protocols and confirms the importance of evaluation using appropriate application-focused methods. During fast cycling conditions, extractable capacity is typically limited by slow Li^+ diffusion in both the pores of the active layer and within

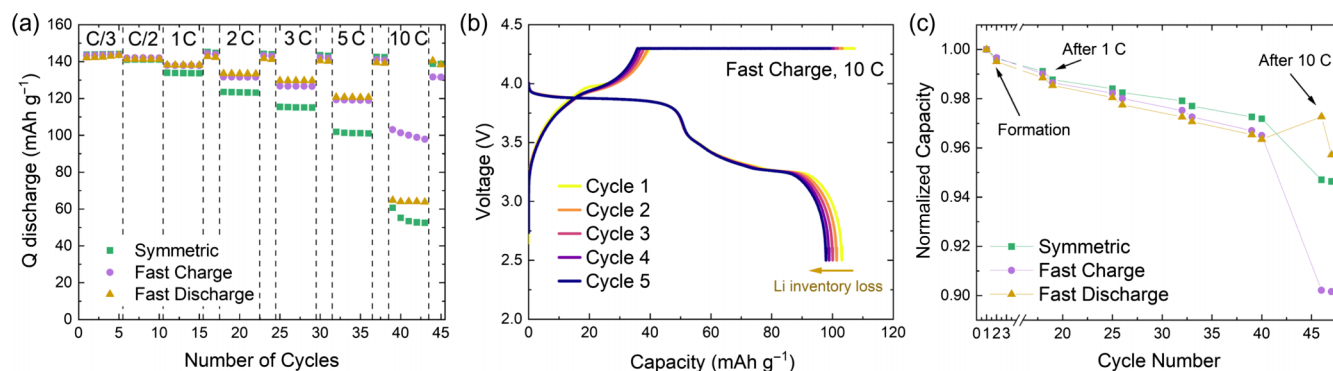


FIG. 2. Behavior of three-electrode LMFP \(\backslash\) Graphite cells during rate capability test using three protocols. (a) Discharge capacity as a function of rate. Here the stated rates are those applied during the constant-current phase of charge or discharge, as applicable. (b) Cell voltage profiles during the five cycles at 10 C using the fast-charge protocol. (c) Capacity extracted during C/20 diagnostics performed after each fast cycling rate. While the complexity of the 3E system limits potential for evaluation of many replicate cells, performance as measured here aligns well with similar coin and pouch cells tested in our previous work [2].

the active materials themselves (with the former typically dominating) [37–39]. A consequential buildup of electrode-level heterogeneity, e.g., more rapid delithiation of cathode particles near the separator during fast charge [40], implies a high level of polarization within the cell, leading to premature triggering of cell voltage limits, thus limiting extracted capacity.

Notably, a decay in the capacity extracted during the application of higher rates was observed, most visible in the fast-charge protocol at 10 C where it decayed from 103 mAh g⁻¹ to 98 mAh g⁻¹. This effect is often observed during rate capability testing in the literature, though not typically highlighted [2,15,19]. The source of this capacity fall was investigated via examination of voltage profiles during 10 C cycling [Fig. 2(b)]. Here it can be seen that the discharge curves fully overlap except at low voltage, clearly indicating the loss of Li inventory (LLI) and electrode slippage [41]. This mechanism was also apparent for the symmetric protocol, but not for the fast discharge (Fig. S4 in Supplemental Material [32]), and points to an induced degradation mechanism at a high rate.

The incorporation of slow C/20 cycles performed after each rate is a useful tool here. These cycles serve to both reset the cells to 0% SoC, ensuring a consistent starting point before each rate is applied (a voltage hold at the bottom of charge may achieve a similar effect), while also providing diagnostic information. Figure 2(c) shows the capacity as measured by these diagnostic cycles for each of the three protocols examined. The extent of capacity loss was quite low (< 4%) and similar for all protocols after rates ≤ 5 C, with the symmetric protocol showing slightly better retention. Capacities, however, diverged significantly after cycling at 10 C, whereby capacity loss was greatest for the fast-charge protocol (~10%) and lowest for fast discharge (~4%). A small capacity decay was observed for the fast discharge C/20 cycles (after 10 C), which may have been due to the beneficial effect of residual high temperature from the previous 10 C cycle. Differential voltage analysis (DVA) of the cell undergoing the fast-charge protocol, comparing pristine and after the five cycles at 10 C, confirms the dominant source of capacity loss as LLI (Fig. S5 in Supplemental Material [32]). While LLI at lower rates can be ascribed largely to SEI formation (which would be expected to be similar for all protocols), the divergence after 10 C points to an onset of more significant LLI degradation modes.

A comparison of the rate test with and without diagnostic cycles reveals slightly higher capacity extraction with them included (Fig. S6 in Supplemental Material [32]). This was attributed to the “resetting” effect, whereby the diagnostic cycles fully reset SoC to 0% before the next rate was applied, along with providing time for a capacity recovery via electrode homogenization and recovery of any plated Li. It is recommended that researchers include these cycles in the standard rate capability test protocol to

enable better comparison between materials and track cell degradation induced during fast cycling.

The evolution of capacity with cycling rate was investigated further via examination of the voltage curves (cell, cathode, and anode) for each protocol. Figures 3(a)–3(c) show the evolution of anode potential during charging at increasing rates for the three protocols. Charging at fast rates lowered the anode potential significantly, reaching < 0 V vs Li⁺/Li at rates ≥ 5 C for the symmetric and ≥ 3 C for the fast-charge protocol (reaching -0.18 V vs Li⁺/Li at 10 C), suggesting that the anode was subjected to conditions under which Li plating may have occurred. Notably, this effect was most prominent at relatively early stages of the charge cycle at low SoC (< 20%). Clearly, the anode potential was lowest, and thus the likelihood of Li plating was the highest, during the initial constant-current phase of charging. This is in contrast with traditional understanding, whereby Li plating is considered likely only under extreme circumstances of high rate and high SoC/overcharge, often combined with low temperature [42–45]. Here it is important to consider the geometry of the 3E cell, whereby the Li reference electrode is located between the anode and cathode, exhibiting a ring shape running around the perimeter of the cell stack [46]. It should be noted that while the measured anode potential would be expected to be representative of the true value across the cell area at low applied current, fast cycling may induce significant spatial heterogeneity across the anode. This cannot directly be observed via 3E measurements, which provide only a single value, and so it is possible that, e.g., certain areas of the anode would reach < 0 V vs Li⁺/Li, while other areas remain positive or vice versa. This may lead to plating even when measured anode potential is positive. Nevertheless, this approach represents the state-of-the-art in reference electrode design and has been used to track electrode potential under fast charging [47].

Voltage evolution during fast cycling at 10 C was examined in more detail, with curves shown in Figs. 3(d)–3(f). Both cathode and anode made significant contributions to total cell polarization, with the anode dominating at low SoC [57% at 20 mAh g⁻¹ in Fig. 3(f)] and the cathode dominating at high SoC [63% at 100 mAh g⁻¹ in Fig. 3(h)]. Here the limited electrode can also be elucidated, whereby for symmetric and fast-discharge protocols, extracted capacity is limited by the combination of cathode and anode polarization at the end of discharge, while, for fast charge, the anode is fully delithiated at the end of discharge, and thus capacity is limited by the polarization at the top of charge (cathode-dominated).

During the constant-voltage phase, the current dropped and the anode potential increased, climbing back to positive values by the end of charging [Figs. 3(d) and 3(f)]. To verify the presence of plating, equivalent full coin cells undergoing each protocol were disassembled in the

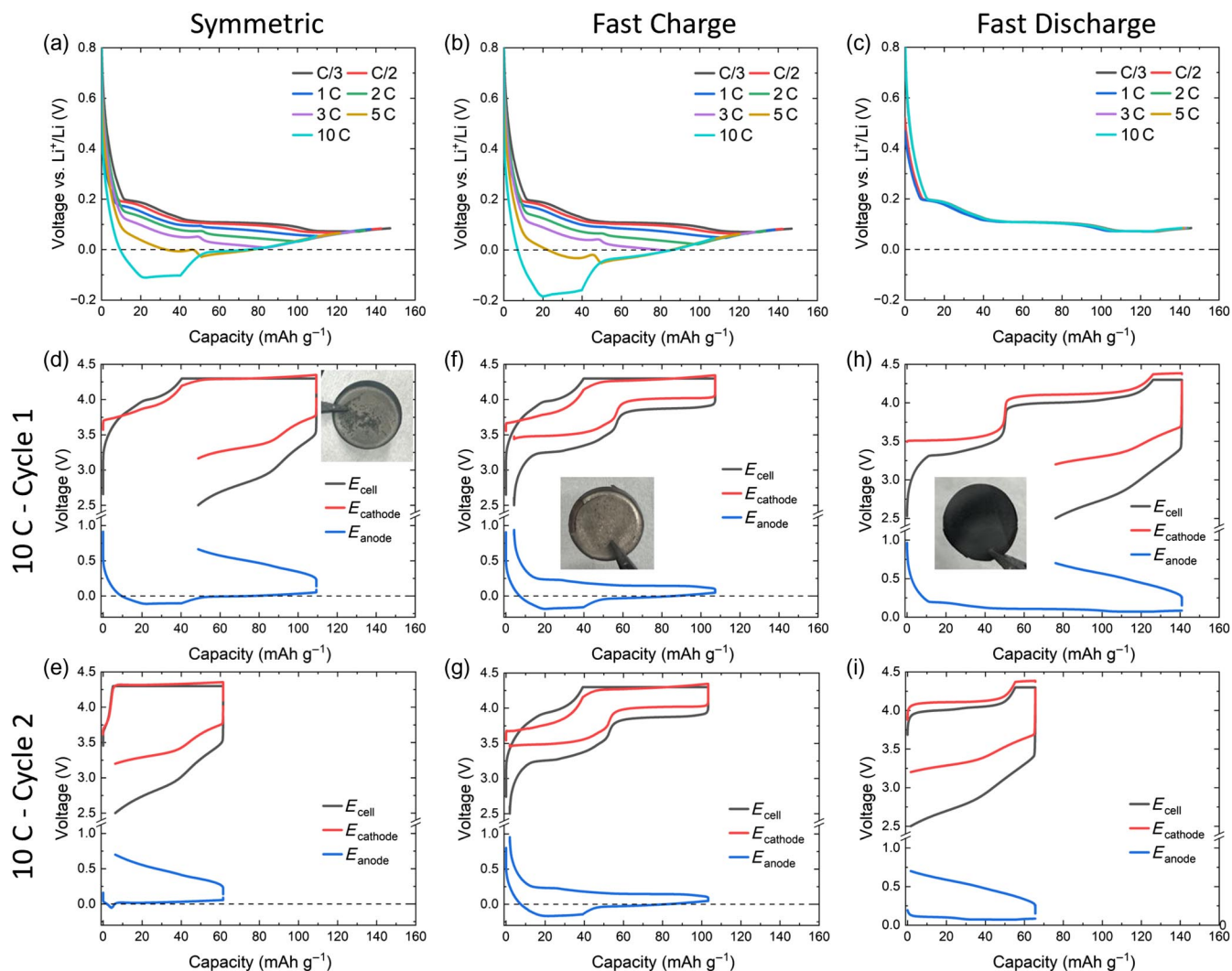


FIG. 3. Voltage profiles of LMFP || Gr3E cells. Evolution of anode potential during charging at various rates using (a) symmetric, (b) fast charge, and (c) fast discharge protocols. Data is taken from the first cycle applied at each rate. Cell, cathode, and anode potential curves during 10 C cycling using three protocols: symmetric (d),(e), fast charge (f),(g), and fast discharge (h),(i). For each protocol, the first and second cycles at 10 C are shown. Inset in (a),(c),(e) are images of anodes harvested (in the charged state) from equivalent coin cells cycled using each protocol. Cell potential curves for the fast-charge protocol at each rate are shown in Fig. S7 in Supplemental Material [32].

charged state after cycling at 10 C, with photographs of harvested anodes shown in insets in Figs. 3(d), 3(f) and 3(h). The anodes from cells that underwent symmetric and fast-charge protocols showed a clear grayish color of metallic Li on the surface of the electrode, confirming that plating had occurred. The anode from the fast-discharge protocol did not show any such signs, consistent with the maintenance of anode potential > 0 V Li^+/Li at all times. It should be noted that while this visual inspection confirmed Li plating, it does not indicate precisely when during the charge cycle it occurred (for which we rely on monitoring of anode potential from 3E measurements). Further study involving direct observation of the formation of metallic Li at low SOC, e.g., via *in situ* nuclear magnetic resonance, is warranted.

This further confirms the LLI origin for the capacity loss observed within the 5-cycles at 10 C [Fig. 2(a)], whereby for symmetric and fast-charging protocols, Li plating had been induced. Longer-term cycling at 10 C using the fast-charge protocol (Fig. S8 in Supplemental Material [32]) confirmed the high rate of performance loss under such circumstances, with capacity falling to < 20 mAh g^{-1} after 100 cycles. Here, the capacity was only marginally recovered during diagnostic cycles, confirming the irreversibility of this LLI. This neatly highlights a limitation of the common rate capability test, whereby examination of the performance of the fast charge cell in Fig. 2(a) may lead one to believe the cell is eminently capable of cycling at 10 C due to the reasonable capacity measured (> 100 mAh g^{-1}). However, it is clear that irreversible

Li loss and associated Li plating [determined via diagnostic cycling at C/20, Fig. 2(b), and observation of harvested anodes] had occurred.

During fast cycling, another phenomenon becomes apparent. Given that capacities at 10 C ($60\text{--}100\text{ mAh g}^{-1}$) were significantly lower than that exhibited at low C-rate (140 mAh g^{-1}) or indeed theoretical for LMFP (170 mAh g^{-1}), the question arises as to over what SoC range the cell was operating. This is important for two reasons. First, certain battery degradation mechanisms are SoC dependent, e.g., Mn dissolution and Jahn-Teller structural distortions in LMFP, and oxygen loss from NMC, both occur under high levels of delithiation [9,48–50]. Second, cell kinetics (particularly the ionic diffusivity of cathodes) are known to be affected by lithiation level [51,52]. This kinetic aspect was confirmed for the LMFP \ Gr cells in this study via analysis of polarization as a function of SoC (Fig. S9 in Supplemental Material [32]), whereby it was approximately 70–100 mV and 170–220 mV in the Fe and Mn redox regions, respectively. Taking the cell cycled symmetrically at 10 C as an example [Fig. 3(d)], and starting from an initially fully discharged state, we observed a relatively high charge capacity of 110 mAh g^{-1} , and a smaller discharge capacity of 60 mAh g^{-1} . Clearly capacity was discharge-limited, and the cell remains at a relatively high SoC at the supposed end of discharge. In the subsequent 10 C cycle [Fig. 3(e)], the cell achieves charge and discharge capacities of $50\text{--}60\text{ mAh g}^{-1}$. Given a total capacity of $\sim 145\text{ mAh g}^{-1}$, this indicates that the cell is operating in the range of 35%–75% SoC. While such high cycling rates undoubtedly introduce particle and electrode-level heterogeneity, this range likely consists largely of Mn redox activity (given the Mn/Fe ratio of 60:40 for this material). In the case of fast charge (Figs. 3(f) and 3(g)), the cell discharged at C/3 every cycle (effectively to 0% SoC), and so the capacity of 105 mAh g^{-1} indicates operation over an SoC range of 0%–72%, encompassing all of the Fe redox activity and approximately half of the Mn. Finally, the cell undergoing the fast-discharge protocol [Figs. 3(h) and 3(i)] was charged slowly and thus reached high SoC (and high cathode voltage) every cycle. Achieved capacities of $\sim 65\text{ mAh g}^{-1}$ indicated operation in the range of 55%–100% SoC, i.e., Mn redox activity dominating, and thus this protocol may be expected to induce greater levels of cell degradation. This typically overlooked aspect of protocol-dependent operational SoC range would be expected to impact directly on cell capacity loss during extended cycling and should be considered (alongside other potential mechanisms such as Li plating) when selecting aging test protocols and operational conditions. The effect on degradation rate was verified via comparing the capacity loss experienced during 200 cycles by cells cycled with fast-charge and fast-discharge protocols (Fig. S10 in Supplemental Material [32]). The

fast-discharge protocol (operating in a high SoC range) induced a greater capacity loss than that of the fast-charge protocol (low SoC range).

Given the greater level of degradation experienced during the “fast-charge” protocol and its clear relevance to common applications such as EVs, the question arises as to how different anode chemistries are affected by such conditions. Silicon, offering a high capacity and low potential, is a compelling alternative to graphite and is widely adopted in state-of-the-art Li-ion batteries [22]. This is most often in the form of a SiO_x @graphite composite, with 5%–20% SiO_x , balancing the high capacity of Si with the stability of graphite. Here we directly compared the fast-charge capability of three anode chemistries: graphite, SiO_x , and a 90:10 composite of graphite: SiO_x . For these cells, an NMC811 cathode was utilized in place of LMFP, as Si-containing anodes are typically paired with NMC in design-to-performance, high-energy-density chemistry typical in high-end EVs (while phosphate cathodes are often paired with graphite-only electrodes in a design-to-cost approach [53]). Figure S11 in Supplemental Material [32] shows the voltage curves of these cells during the first formation cycle. All cells exhibited a high and similar charge capacities of $230\text{--}235\text{ mAh g}^{-1}$, and both the NMC \ Gr and NMC \ Gr@ SiO_x exhibited high discharge capacities of 215 and 206 mAh g^{-1} , respectively. The NMC \ SiO_x cell suffered from high first cycle loss (FCL) and a low Coulombic efficiency of just 70% attributed to excess SEI formation and material pulverization associated with large Si volume expansion/contraction, typical of this chemistry [54]. Figure 4(a) shows capacity extracted from the three 3E cells cycled from C/3 to 10 C using the fast-charge protocol, again with C/20 diagnostic cycles performed after each rate. The Gr-only cell maintained a capacity advantage over the other chemistries at all rates, exhibiting only minor capacity loss. Conversely, the Si-containing cells underwent significant capacity loss and did not recover their full capacity when the cycling rate was returned to C/3. Given that the capacity extracted during the diagnostic cycles also fell, this degradation was likely not kinetic in origin. Notably, the relative advantage of the Gr-only cell was reduced at higher rates, with all three chemistries converging somewhat at 10 C. The better high-rate performance of Si-containing anodes is consistent with their reduced anode coat weight, thickness, and tortuosity for a given areal capacity [55].

The composition of the anode may be expected to alter its lithiation potential and overall cell kinetics, impacting on the likelihood of Li plating and other degradation mechanisms at high rate (including those associated with the cathode). Here Figs. 4(b), 4(c), and 4(d) show electrode potential curves for each of the three anode chemistries, obtained from 3E cells cycled at 10 C using the fast-charge protocol. Monitoring of the cathode potential revealed

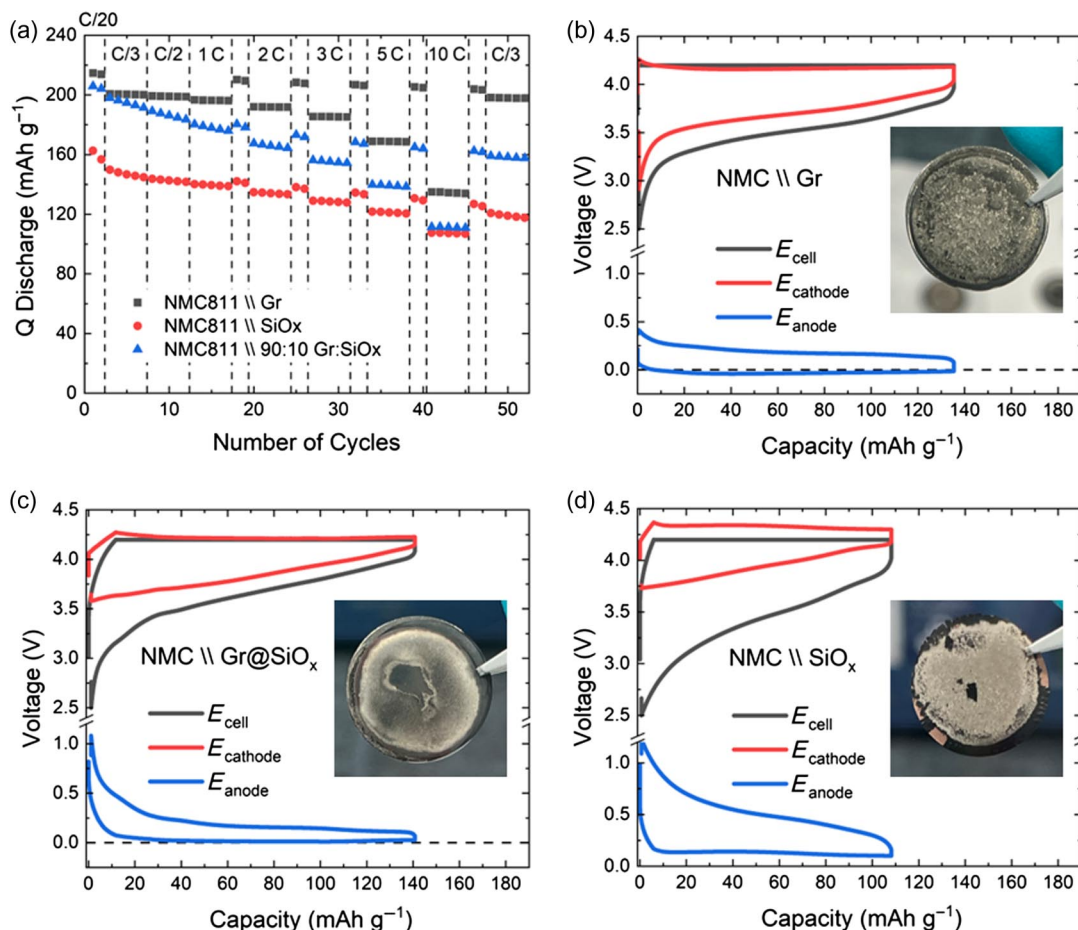


FIG. 4. Rate capability testing of NMC811 cells with three different anode chemistries. (a) Capacity as a function of cycling rate. Three-electrode voltage profiles measured at 5 C for the three cell chemistries: (b) NMC811 || Gr, (c) NMC811 || Gr@SiO_x, and (d) NMC811 || SiO_x.

significant differences based on anode composition. In the case of NMC || Gr, cathode potential did not exceed 4.21 V, while for NMC || Gr@SiO_x, it reached slightly higher at 4.25 V. For the NMC || SiO_x cell chemistry, however, NMC potential reached 4.34 V vs Li⁺/Li, potentially inducing electrolyte decomposition and cathode oxygen loss [9]. This has implications for optimum cell operational windows, whereby, if fast charging is desired, cell upper voltage (UCV) cutoff must be adjusted to account for anode composition. Cell polarization exhibited notable dependence on anode chemistry, with values of 0.7, 0.71, and 0.59 V for Gr, Gr@SiO_x and SiO_x, respectively, measured at 60 mAh g⁻¹.

On the anode side, the potential reached minima of -40 mV for NMC || Gr, +12 mV for NMC || Gr@SiO_x, and +101 mV for NMC || SiO_x. Because the potential of the graphite-only anode dipped below 0 V vs Li⁺/Li, one may expect Li plating to be more likely with NMC || Gr than with the other chemistries. To examine this, cells were disassembled in the charged state (charged at 10 C) with harvested anodes shown in the inset in Fig. 4. Indeed,

Li plating was apparent for the NMC || Gr cell [inset in Fig. 4(b)], to a lesser apparent extent than that of the similar LMFP || Gr cell [inset in Fig. 3(d)], which is consistent with the observed variance in anode potential. The SiO_x-containing electrodes [inset in Figs. 4(c) and 4(d)] also showed signs of significant degradation, whereby plated Li was present for Gr@SiO_x to a lesser extent than Gr (consistent with previous studies [56]) and located predominantly at the electrode edges. The SiO_x electrode appeared extremely degraded, though this is expected to exhibit significant material pulverization in addition to any plating. Degradation was quantified via DVA (curves shown in Fig. S14 in Supplemental Material [32]), with the losses associated with Li inventory loss (LLI) and the loss of active material (LAM, for each cell component) shown in Table II. LLI was higher for the Gr-only anode; however, LAM was far higher in the case of SiO_x-containing electrodes. This is consistent with the anode potential evolution (suggesting more Li plating and thus more LLI for Gr-only) and with an expected higher level of material pulverization in the case of Gr@SiO_x.

TABLE II. Capacity losses suffered by each of the three NMC cell types during the rate capability test. Values were extracted via DVA with curves shown in Fig. S13 in Supplemental Material [32], comparing the second formation cycle and the second diagnostic cycle after 10 C cycling. Capacity loss values are expressed as a % of initial, i.e., the LAM-SiO_x of -24% corresponds to approximately 8% of total anode capacity.

	NMC \ Gr	NMC \ Gr@SiO _x	NMC \ SiO _x
Capacity loss	-5%	-21%	-20%
LLI	-10%	-6%	Indeterminate
LAM-Gr	-2%	-6%	
LAM-SiO _x		-24%	Indeterminate
LAM-cathode	-2%	-3%	-3%

These factors were not quantifiable for the SiO_x anode via DVA due to the flat, featureless curve (Fig. S14 in Supplemental Material [32]), exhibiting only a cathode-related peak. LAM for the cathode was consistently low for all three cell types. EIS measurements of this cell obtained before and after the rate test (Fig. S15 in Supplemental Material [32]) confirmed worsening kinetic properties of the SiO_x-containing cell. In particular, the growth of the high-frequency impedance element indicated excess SEI formation, acting to both consume active Li and increase impedance.

Figure 5(a) shows the minimum potentials reached by the anode during fast charging of the four chemistries examined in this study, as a function of charging rate. While the minimum potential at low C-rate may be largely determined by small variations in NP ratio, the value at higher rates could be expected to be more heavily influenced by cell kinetics and charging strategy. The values for SiO_x-containing anodes were relatively constant across all charging rates, compared with graphite, which tended to decrease at higher rates. Notably, the anode of

the LMFP \ Gr cell reached a far lower potential at 10 C (-0.18 V) than the NMC \ Gr cell (-0.04 V). While the two cell designs utilized different cathode and anode areal capacities (and thus applied areal current density), it is important to note that these manufacturing parameters were chosen to reflect optimized, real-world LMFP and NMC cell types, in an endeavor to maximize the relevance of results. To illustrate the mechanism here, Fig. 5(b) shows the amount of capacity delivered during the constant-current (CC) charging phase (as opposed to the subsequent constant-voltage phase). At low C-rate, both cells delivered most of their charge capacity during the CC phase (88% and 95%, respectively). At higher C-rates, the greater polarization caused cell voltage to reach the UCV prematurely, and thus only a small capacity was delivered in the CC phase. LMFP maintained a higher CC capacity at high C-rates due to the presence of the low-voltage Fe redox plateau, which kept cell voltage below the UCV for longer. While this is a positive for enabling fast-charging rates for LMFP, the side effect was to deliver more capacity at high current, thereby decreasing anode potential and inducing Li plating.

IV. CONCLUSION

Fast-cycling capability is a key performance indicator for the evaluation of Li-ion battery designs, encompassing the ability of a cell to process high currents while avoiding excessive degradation. In this work, we evaluate four leading battery chemistries in this context, covering design-to-cost (LMFP \ Graphite), design-to-performance (NMC811 \ Gr and NMC811 \ Gr@SiO_x) along with next generation (NMC811 \ SiO_x). The common method of fast cycling evaluation in the literature, the rate capability test, lacks much in terms of standardization with many varying protocols utilized. We demonstrate that the measured performance at higher cycling rates was highly

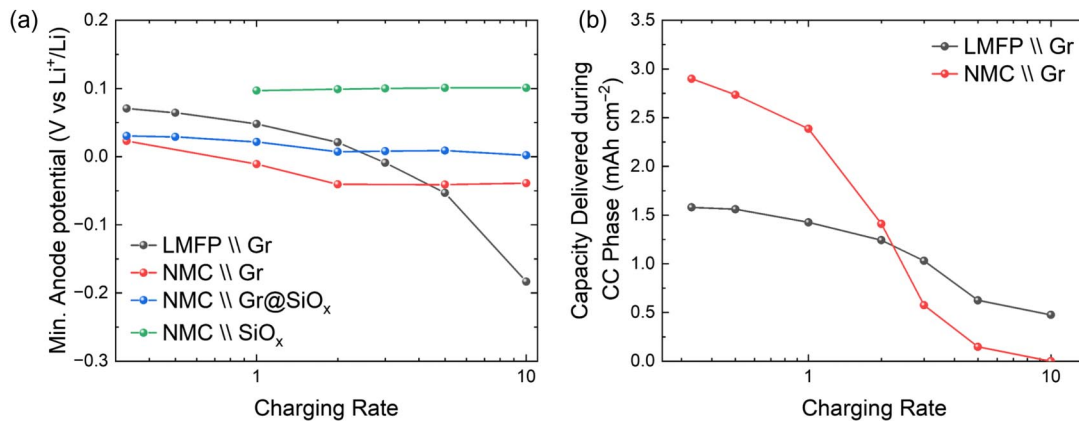


FIG. 5. (a) Minimum recorded anode potential during charging of 3E cells of four cell chemistries, and variation with charging rate. Some data from the NMC \ SiO_x cell are omitted due to failure of the reference electrode, attributed to the high levels of electrode expansion. (b) Amount of charge delivered during the constant-current phase of the charging protocol.

dependent on the rate protocol chosen, with a fast-charge/slow-discharge protocol providing the highest capacity and a symmetric protocol providing the lowest. Degradation mechanisms experienced during fast cycling were similarly protocol-dependent, whereby the fast charge and symmetric protocols induced Li plating (and thus significant LLI of 5%–10%) at 10 C as determined via inspection of harvested anodes and diagnostic cycling, while fast discharge did not. However, the fast-charge protocol also induced cell operation in a safer, lower SoC range, providing superior capacity retention during extended cycling at 5 C. Standardization of protocol within the field would provide more application-focused evaluation and enable more effective comparison between studies, while the simple insertion of slow, diagnostic cycles into the protocol provides useful information on degradation induced.

3E cell measurements enabled monitoring of both cathode and anode potentials. For NMC cells, the cathode reached higher (potentially degradation-inducing) potentials in the case of an SiO_x anode (4.34 V vs Li/Li⁺) compared with a graphite anode (4.21 V vs Li/Li⁺), highlighting the need for precise cell voltage control (adapted for anode chemistry) when fast charging is desired. The minimum potential reached by the anode during fast charging (a key indicator of Li plating) was similarly dependent on composition, with Si-containing anodes maintaining a positive potential even at the very fast rate of 10 C. The degree of Li plating was greater for LMFP \\\ Gr chemistry than for similar NMC \\\ Gr (consistent with reaching a lower minimum anode potential), induced as a side effect of greater capacity delivered during the constant-current phase of charging. The use of the 3E cell enabled the rapid, *in situ* identification of cycling conditions and charge rates, which pose significant degradation and safety risks. This data can be utilized to inform models with which BMS can detect and prevent such events.

Follow-on work should focus on the evaluation of chemistry-specific fast cycling capability in commercial format cells, potentially utilizing “instrumented” versions with inserted reference electrodes for *in situ* detection of Li plating. Additionally, the use of *in operando* techniques (such as XRD) to track graphite phase evolution may provide insights into Li plating in a nondestructive manner [9].

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DATA AVAILABILITY

There are no publicly available research data or software supporting this manuscript. Requests for further information or data should be sent to the authors.

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