

Article

Multi-Step Fast Charging of Commercial NMC/Graphite EV Pouch Cells: First-Life Extension, Degradation Mechanisms, and Second-Life Performance

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Abstract

Fast charging provides convenience for electric vehicle users but can accelerate Li-ion battery degradation and compromise lifetime energy utilization. Here, we investigate how fast-charging protocols govern first- and second-life performance of commercial 20 Ah NMC442/graphite pouch cells. A six-step 3C protocol (P6C2R1) completed 1009 first-life recorded cycles with >84.7% state of health (SOH) and limited resistance growth. Cells previously subjected to P6C2R1 subsequently demonstrated extended second-life cycling, including 1000 service cycles at 0.5C with ~80% SOH. Post-mortem scanning electron microscopy, X-ray diffraction, and X-ray photoelectron spectroscopy revealed reduced electrode deterioration, smaller structural changes, and a comparatively LiF-rich graphite interphase under multi-step fast charging (MS-FC). Increasing relaxation steps systematically delayed the onset of accelerated aging, while exploratory correlation analysis indicated that first-life aging trajectories contain information about subsequent second-life behavior beyond retirement SOH alone. Across measured first- and second-life operation, P6C2R1 delivered 64.6 kWh of cumulative discharge energy, 89% more than continuous fast charging. These results establish lifetime-aware MS-FC as a practical strategy for balancing charging speed, degradation, and second-life value in commercial lithium-ion batteries.

Keywords: multi-step fast charging; first-life performance; second-life batteries; Li-ion batteries; battery degradation; commercial EV batteries



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1. Introduction

The rapid electrification of transportation is creating an increasing demand for lithium-ion batteries (LIBs) that simultaneously deliver high energy density, rapid charging, long service life, and operational safety [1]. For electric vehicles (EVs), charging time remains a major barrier to broader adoption. The U.S. Advanced Battery Consortium defines extreme fast charging as restoring approximately 80% of a battery's rated capacity within 15 min [2]. Achieving this target, however, requires charging currents sufficiently high to drive the cell from electrochemical equilibrium, thereby accelerating degradation and potentially compromising safety.

During charging, Li^+ -ions are extracted from the cathode, transported through the electrolyte, and intercalated into the graphite anode [3]. At high charging rates, the incoming Li^+ -ion flux can exceed the combined rates of interfacial charge transfer and solid-state diffusion within graphite. The resulting concentration gradients and anode polarization lower the local graphite potential and promote metallic lithium plating, especially at high state of charge (SOC) and low temperature. Some plated lithium may subsequently become electrochemically inactive (as “dead Li”), while continued parasitic reactions consume cyclable lithium and increase cell resistance [4]. Fast charging can also accelerate electrolyte decomposition, nonuniform solid electrolyte interphase (SEI) growth, gas evolution, heat generation, and electrode structural degradation [5]. Recent studies of commercial pouch cells (1.8 Ah $\text{LiNi}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}\text{O}_2$ /graphite) charged at 4C–6C have associated rapid capacity loss with lithium plating, increasing polarization, and changes in SEI chemistry, including LiF accumulation [6]. SEI composition also plays a pivotal role in resistance growth and capacity loss. For example, organic SEI formed due to lithium plating and carbonate electrolyte decomposition during fast charging exhibits a low Young’s modulus, which cannot effectively suppress further lithium dendrite growth [5]. In contrast, inorganic SEI components, such as LiF and Li_2CO_3 , provide good chemical stability and mechanical strength. LiF is more desirable than Li_2CO_3 because of its superior electronic insulating properties, which can passivate graphite particles and suppress further lithium dendrite growth [4]. Fast-charge durability is therefore governed by coupled transport, interfacial, thermal, and structural processes.

Materials-based strategies for improving fast-charging performance include designing anodes with more rapid, uniform Li^+ -transport pathways [7] and modifying the electrolytes and constructing artificial SEI layers to stabilize the electrode–electrolyte interface [8,9]. Although effective, these approaches generally increase material costs and cell-manufacturing complexity. Charging-protocol optimization offers an attractive complementary route because it can potentially be implemented through the battery-management system without redesigning the cells [10]. Conventional LIBs are commonly charged using a constant-current/constant-voltage (CC-CV) protocol, in which the cell is charged at a CC stage until the upper-voltage limit is reached, followed by a CV stage with progressively decreasing current [11]. At high C-rates, however, the CC period can generate substantial polarization, while the CV period increases the overall charging time [12,13].

Pulse and multi-step charging strategies seek to overcome this tradeoff by periodically reducing or interrupting the charging current, thereby allowing Li-ion redistribution within the electrolyte and electrodes and partial relaxation of electrochemical polarization [14–18]. Properly designed relaxation periods reduce near-surface Li^+ -accumulation on graphite and suppress conditions favorable for lithium plating. Their effectiveness, however, depends strongly on current magnitude, pulse duration, relaxation time, SOC, temperature, and cell chemistry [19]. Inappropriately selected conditions can instead increase polarization, interfacial resistance, and parasitic reactions [20]. More importantly, much of the existing literature has focused on relatively small-format cells [21], limited cycling durations [17], or cell-level electrochemical response without direct correlation to post-mortem structural and chemical degradation [12,18,22,23]. Consequently, the mechanisms by which multi-step fast-charging (MS-FC) delays the transition from gradual aging to accelerated degradation remain incompletely understood.

An additional and largely unresolved question is whether the first-life fast-charging history determines the remaining second-life value of an EV battery. Retired EV cells often retain substantial capacity and may be repurposed for less demanding stationary applications or continued use in mobile applications with lower range and power requirements, such as e-bikes [24]. The end-of-life internal resistance of first-life batteries can affect

the performance of second-life batteries, thereby determining whether retired batteries can continue to be used in mobile applications. As illustrated in Figure 1, incorporating second-life use after vehicle retirement and before final recycling can increase lifetime energy utilization and delay material disposal. EV batteries are frequently considered for retirement near 70–80% state of health (SOH), although the appropriate threshold depends not only on capacity but also on resistance growth, power capability, degradation history, safety, and the requirements of the second-life application [25]. Mowri et al. reported that first-life degradation history can exert a stronger influence on second-life behavior than the nominal SOH at retirement [26], while Kim et al. demonstrated that changes in the slopes of SOH-related parameters can help identify transitions between degradation regimes [27]. However, electrochemical signatures such as capacity fade or knee-point analysis alone do not uniquely reveal the underlying physicochemical mechanism. Establishing a mechanistic connection between first-life fast-charging operation and second-life performance therefore requires electrochemical measurements to be correlated with structural, morphological, and interfacial characterization.

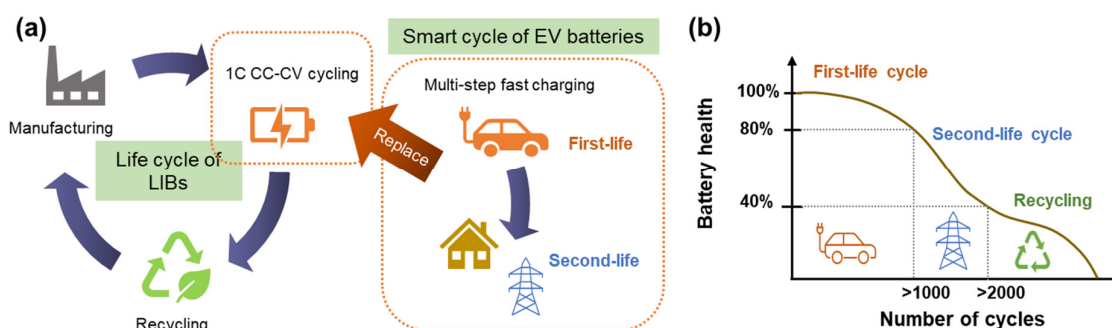


Figure 1. Integration of first-life optimization, second-life utilization, and recycling in the life cycle of EV batteries. (a) Conventional battery life cycle and the proposed integration of optimized fast charging and second-life deployment. (b) Schematic evolution of battery state of health during first- and second-life operation before recycling.

Here, we investigate how charging rate, SOC window, and multi-step fast charging (MS-FC) govern both first-life degradation and subsequent second-life performance in commercial 20 Ah EIG ePLB-C020 pouch cells. Continuous 3C and 4C fast charging is compared with MS-FC protocols containing controlled relaxation periods, with particular attention to a six-step 3C protocol operated within a restricted SOC window. Long-term cell aging is quantified through capacity retention, SOH evolution, internal-resistance growth, and the onset of accelerated degradation. Scanning electron microscopy (SEM), X-ray diffraction (XRD), and X-ray photoelectron spectroscopy (XPS) are then used to correlate cell-level aging with electrode morphology, crystal-structure evolution, and anode interphase chemistry. After first-life testing, the retired cells are further evaluated under 0.25C, 0.5C, and 1.0C cycling to determine how their prior fast-charging histories influence second-life durability. By integrating long-term cycling, internal-resistance evolution, degradation-mode analysis, post-mortem characterization, second-life testing, and cumulative energy accounting, this study establishes a direct link between fast-charging protocol and lifetime cell utilization. The results provide practical guidance for designing fast-charging strategies that balance charging speed against degradation and for determining when high-rate operation should be curtailed to preserve both first-life durability and subsequent second-life value.

2. Experimental Methods

2.1. Cell Structure and Specifications

Commercial EIG ePLB C020 pouch cells (Energy Innovation Group Ltd., Cheonan, Republic of Korea), purchased in 2011, were used in this study. Each cell consisted of 20 graphite anodes with vapor-grown carbon fibers as conductive additive, 19 $\text{Li}(\text{Ni}_{0.4}\text{Mn}_{0.4}\text{Co}_{0.2})\text{O}_2$ (NMC442) cathodes, a Z-fold separator, and a liquid electrolyte, sealed in a pouch with dimensions of 217 mm $\times$ 130 mm $\times$ 7.1 mm, offering a nominal capacity of 20 Ah, a nominal voltage of 3.65 V, and a cell-level energy density of 174 Wh/kg. A total of 48 cells were evaluated in this work. All the electrochemical tests were performed using a 24-channel Chroma 17,010 Battery Cell Test System (Chroma ATE Inc., Irvine, CA, USA). Notably, prior to testing, all cells had been stored in their original packaging in a flammable storage cabinet at room temperature and at a nominal voltage of approximately 3.65 V (~30% SOC based on the OCV test, shown in Figure S1) since shipment from the manufacturer. Under these storage conditions, the impact of long-term calendar aging on cell performance is expected to be minimal and is unlikely to affect the comparative evaluation of the charging protocols.

2.2. Cell Testing Procedures for First-Life Cycling

Figure 2a illustrates the overall testing framework, including fast-charging cycling and periodic SOH assessments. Cell location and cycle-accounting methods are summarized in Tables 1–4. A total of 48 cells were divided into three batches. Batch 1 (B1, 4 cells) was cycled at 1C using a CC-CV protocol and served as the reference. Batch 2 (B2, 24 cells) was used to evaluate continuous and MS-FC protocols over the full SOC range, corresponding to voltage limits of 3.0–4.15 V. Batch 3 (B3, 20 cells) was used to systematically investigate different MS-FC protocols within a voltage limit of 3.385–4.114 V, corresponding to a restricted ~20–80% SOC window.

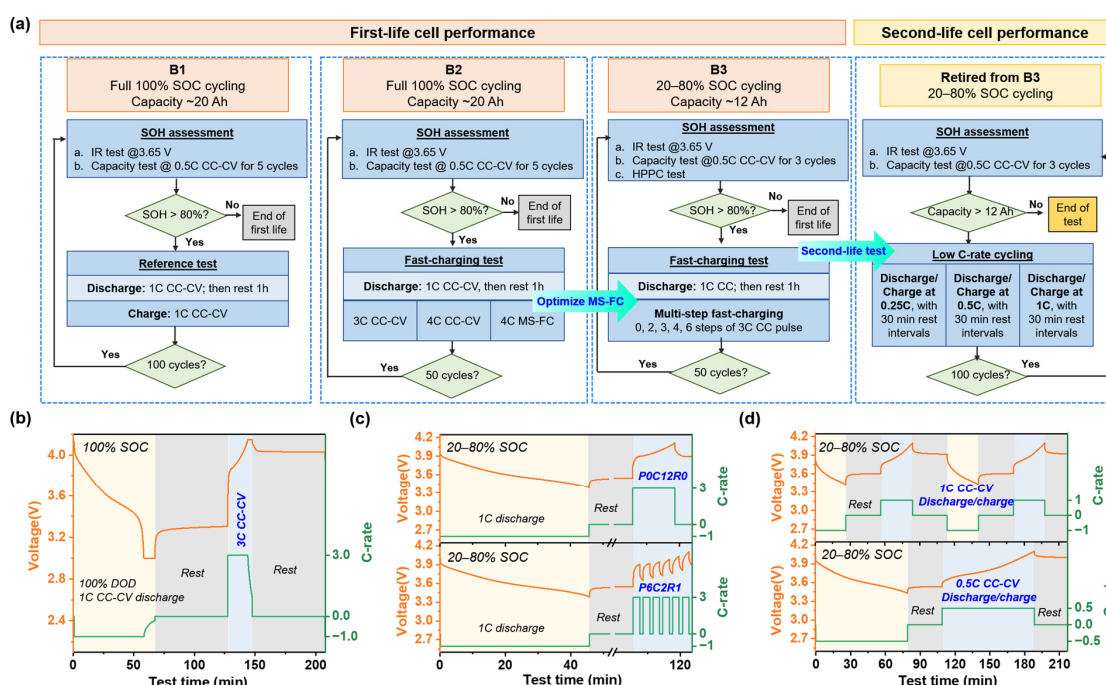


Figure 2. Experimental framework and representative charging/cycling profiles for first- and second-life testing. (a) Overall experimental framework comprising first-life testing of B1, B2, and B3 cells followed by second-life evaluation of selected B3 cells at low C-rates. Representative voltage and current profiles for (b) B2 cells under continuous 3C CC-CV fast charging; (c) B3 cells under P0C12R0 (top) and P6C2R1 (bottom); (d) second-life test at 1C (top) and 0.5C (bottom).

Table 1. B1 cell allocation and cycle-accounting method.

Protocol	No. of Cells	IR Test	Capacity-Test Cycles	HPPC Test	Service Cycles	Recorded Cycles per Round
1C CC-CV	4	At 3.75 V during the 1st cycle	3	During the last capacity cycle	100	103

Note: B1 cells served as the reference group and were cycled over the full SOC range.

Table 2. B2 cell allocation and cycle-accounting method.

Protocol	No. of Cells	Initial IR Test	Initial Capacity-Test Cycles	Service Cycles	Final IR Test	Final Capacity-Test Cycles	Total Recorded Cycles
3C CC-CV	8	At 3.75 V during the 1st cycle	5	50	At 3.75 V during the 56th cycle	5	60
4C CC-CV	8						
4C MS-FC	8						

Note: B2 cells were cycled over the full SOC range.

Table 3. B3 cell allocation and cycle-accounting method.

Protocol	No. of Cells	IR Test	Capacity-Test Cycles	HPPC Test	Service Cycles	Recorded Cycles per Round
P0C12R0	4	At 3.75 V during the 1st cycle	3 *	During the last cycle of capacity test	50	53 **
P2C6R2	4					
P3C4R1	4					
P4C3R1	4					
P6C2R1	4					

Note: B3 cells were cycled within an ~20–80% SOC window. * The first-round capacity test consisted of 5 cycles. ** Consequently, the first round contained 55 recorded cycles rather than 53.

Table 4. B3 cell allocation for second-life testing and cycle-accounting method.

First-Life Protocol	Second-Life Cycling Rate	No. of Cells	IR	Capacity-Test Cycles	Service Cycles	Total Recorded Cycles per Round
P0C12R0	0.25C	2	During the last cycle of capacity test	3	100	103
	0.5C	1				
P2C6R2	0.25C	1				
	0.5C	2				
P3C4R1	0.25C	1				
	0.5C	2				
P4C3R1	0.25C	1				
	0.5C	2				
P6C2R1	0.5C	1				
	1C	2				

Note: One cell from each B3 first-life protocol group was reserved for post-mortem analysis; therefore, three cells from each group were subjected to second-life test.

For B1, the cells were discharged to 3.0 V at 1C using a CC-CV protocol, rested for 1 h, and subsequently charged to 4.15 V at 1C using CC-CV charging with a cutoff current of 0.05C. Each aging round consisted of 100 cycles, following the manufacturer's recommended cycling protocol.

The B2 cells were divided into three groups of eight cells according to the charging protocols: (i) continuous 3C CC-CV charging, (ii) continuous 4C CC-CV charging, and (iii) six-step 4C MS-FC followed by a CV stage. For each cycle, the cells were discharged to 3.0 V at 1C using a CC-CV control with a cutoff current of 0.2C, followed by a 1 h rest. The cells were then charged either continuously at 3C for up to 20 min (3C CC-CV), continuously at 4C for up to 15 min (4C CC-CV) or using six 2.5 min charging steps at 4C separated by 1 min relaxation periods (4C MS-FC). Once the cell voltage reached the upper cutoff of 4.15 V, charging transitioned to the CV stage and was terminated when the current decreased to 0.2C. After every 50 fast-charging service cycles, cell health was assessed using a direct-current internal-resistance (DC-IR) measurement followed by five 0.5C CC-CV capacity-test cycles. SOH was calculated from the current remaining capacity ($Q_{\max}$) to the rated capacity (Q_0) [28], given as:

$$\text{SOH} = Q_{\max}/(Q_0) \times 100 [\%]$$

where $Q_{\max}$ is the averaged maximum capacity from the 0.5C CC-CV cycling test, whereas Q_0 stands for the rated capacity of 20 Ah from the manufacturer. Eight identical cells were used for each test group.

The B3 experiments were designed to isolate the effect of number and duration of relaxation periods during fast charging while restricting the cell operation to a cutoff voltage of 3.385–4.114 V, corresponding to ~20–80% SOC. The 20 cells were divided into five groups of four cells according to the charging protocol. After discharge to 3.385 V (~20% SOC) at 1C and a subsequent 1 h rest, the cells were charged at 3C to the cutoff voltage of 4.114 V (80% SOC) using 0, 2, 3, 4, or 6 charging steps separated by relaxation periods, as illustrated in Figure 2c. The protocols were designated P0C12R0, P2C6R2, P3C4R1, P4C3R1, and P6C2R1, respectively, where P, C, and R denote the number of relaxation interruptions, charging duration per step, and relaxation duration, respectively. These MS-FC protocols maintained the same cumulative 3C charging duration of 12 min, while systematically varying the number and duration of relaxation periods. For example, the P6C2R1 protocol consisted of six 2 min charging steps at 3C, separated by 1 min relaxation periods.

2.3. Second-Life Performance Tests

Cells were considered eligible for second-life testing if they retained >80% SOH and exhibited no severe swelling or other safety concerns after first-life cycling. Based on these criteria, B2 cells were excluded from second-life evaluation because of substantial gas generation and rapid SOH degradation. B3 cells retaining >80% SOH (~16 Ah) after first-life testing were selected for subsequent second-life evaluation within the same 20–80% SOC window.

Cells previously subjected to the P0C12R0, P2C6R2, P3C4R1, and P4C3R1 first-life protocols were evaluated at second-life cycling rates of 0.25C and 0.5C, whereas cells previously subjected to P6C2R1 were evaluated at 0.5C and 1C (Figure 2d). After every 100 second-life service cycles, cell health was evaluated by measuring internal resistance and performing three 0.5C CC-CV capacity-test cycles to determine SOH.

2.4. Pearson Correlation Coefficient Analysis

Pearson Correlation Coefficient (r) analysis was performed to underline potential linear relationships between first- and second-life performance metrics. By definition, the r

value was calculated as the covariance of the two variables divided by the product of their standard deviations [29], given by

$$r = \frac{\text{cov}(x, y)}{s_x s_y} = \frac{\sum (x_i - \bar{x})(y_i - \bar{y})}{\sqrt{\sum (x_i - \bar{x})^2 \sum (y_i - \bar{y})^2}}$$

where $\bar{x}$ and $\bar{y}$ represent the mean of the x and y datasets; x_i and y_i represent the individual data points from the x and y datasets. Each dataset of the variables (descriptors) contains ten independent data points corresponding to the ten individual cells, respectively, undergoing both first/second-life testing from B3. In other words, i iterates from 1 to 10, corresponding to data points from the selective Cell 1 to Cell 10, used in the second-life 0.5C and 1C cycling test. First-life descriptors include the number of charging steps, SOH and internal resistance at EOL, and the number of degradation modes. Second-life descriptors are internal resistance at the 516th cycle and the SOH decay rate. For a reliable relationship, a strong linear correlation between two variables exists when the calculated PCC magnitude is greater than 0.8 [30].

2.5. Characterization

Selected cycled cells were disassembled in an Argon-filled glovebox (LC Technologies, Salisbury, MA, USA) with O_2 /Water levels below 1 ppm. After removal of the pouch packaging, the entire stack was extracted and photographed. The adhesive tapes and current collector tab connections were carefully removed, after which the anode and cathode sheets were separated from the Z-folded separator for post-mortem characterization.

Scanning electron microscopy (SEM) was used to image the morphology of active material particles on cycled electrodes with a FEI Quanta 450 FEG scanning electron microscope (FEI, Hillsboro, OR, USA), operated primarily at an accelerating voltage of 5 kV. Powder X-ray diffraction (XRD) characterization was performed on a Rigaku MiniFlex 6G-C diffractometer (Rigaku, Tokyo, Japan) using Cu $K\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$) at 40 kV and 15 mA. Data were collected over a 2θ range of $10\text{--}90^\circ$ with a step size of 0.01° and a scan rate of $10^\circ/\text{min}$. X-ray photoelectron spectroscopy was performed on a PHI 5600 XPS system (Physical Electronics, Chanhassen, MN, USA), operated at 250 W and 14 kV, with Al $K\alpha$ X-ray radiation with a photon energy of 1486.7 eV to characterize the SEI layer of the graphite anode before and after cycling. Ar^+ ion etching was performed at an ion energy of 2 keV and an ion current of 1 μA for depth profiling. All samples were cut from electrode sheets right after cell disassembly and fully rinsed with dimethyl carbonate (DMC) (anhydrous, >99%, Sigma-Aldrich, Burlington, MA, USA). All XPS samples were further rinsed with N,N -Dimethylformamide (DMF) (anhydrous, 99.8%, Sigma-Aldrich, Burlington, MA, USA) for 30 s to remove PVDF binder before being transferred out for characterization. Notably, the inorganic SEI components (LiF and Li_2CO_3) exhibit very low solubility in DMF. Therefore, DMF can selectively remove PVDF while largely preserving the inorganic SEI components. However, owing to its high polarity, DMF may also partially dissolve organic SEI components.

3. Results and Discussions

3.1. First-Life Performance Under Full SOC Fast Charging

B1 cells were cycled at 1C CC-CV over the full voltage range of 3.0–4.15 V, serving as the reference for evaluating the fast-charging behavior of B2 and B3 cells. In the following discussion, service capacity refers to the capacity delivered under the assigned fast-charging protocol. Recoverable capacity refers to the capacity measured during the 0.5C CC-CV capacity test. The knee point marks the transition from gradual capacity loss to accelerated degradation.

B2 cells were discharged to 3.0 V under 1C CC-CV and then charged to 4.15 V using 3C CC-CV, 4C CC-CV or 4C MS-FC protocols as described in Section 2.2. Figure 3a–d reveals a pronounced dependence of high-rate service capacity on charging protocol. By recorded cycle 50, the 3C CC-CV cells retained ~16 Ah of service capacity, whereas the 4C CC-CV and 4C MS-FC cells delivered only ~6 and 2 Ah, respectively. The corresponding service-capacity decays were 11.4% for 3C CC-CV and 65.5% for 4C CC-CV, demonstrating the strong rate dependence of capacity utilization during fast charging. The 4C CC-CV group also exhibited decreases in CE from 97% to 92% and in EE from 86% to 80.5% near the knee point at recorded cycle 21 (Figure 3d,e). In contrast, the 3C CC-CV group maintained CE above 99% and EE above 89% at the same recorded cycle. After the knee point, both 4C groups went through an accelerated degradation to a later self-limiting stage. Overall, continuous 3C charging provided substantially greater service-capacity retention than either 4C protocol.

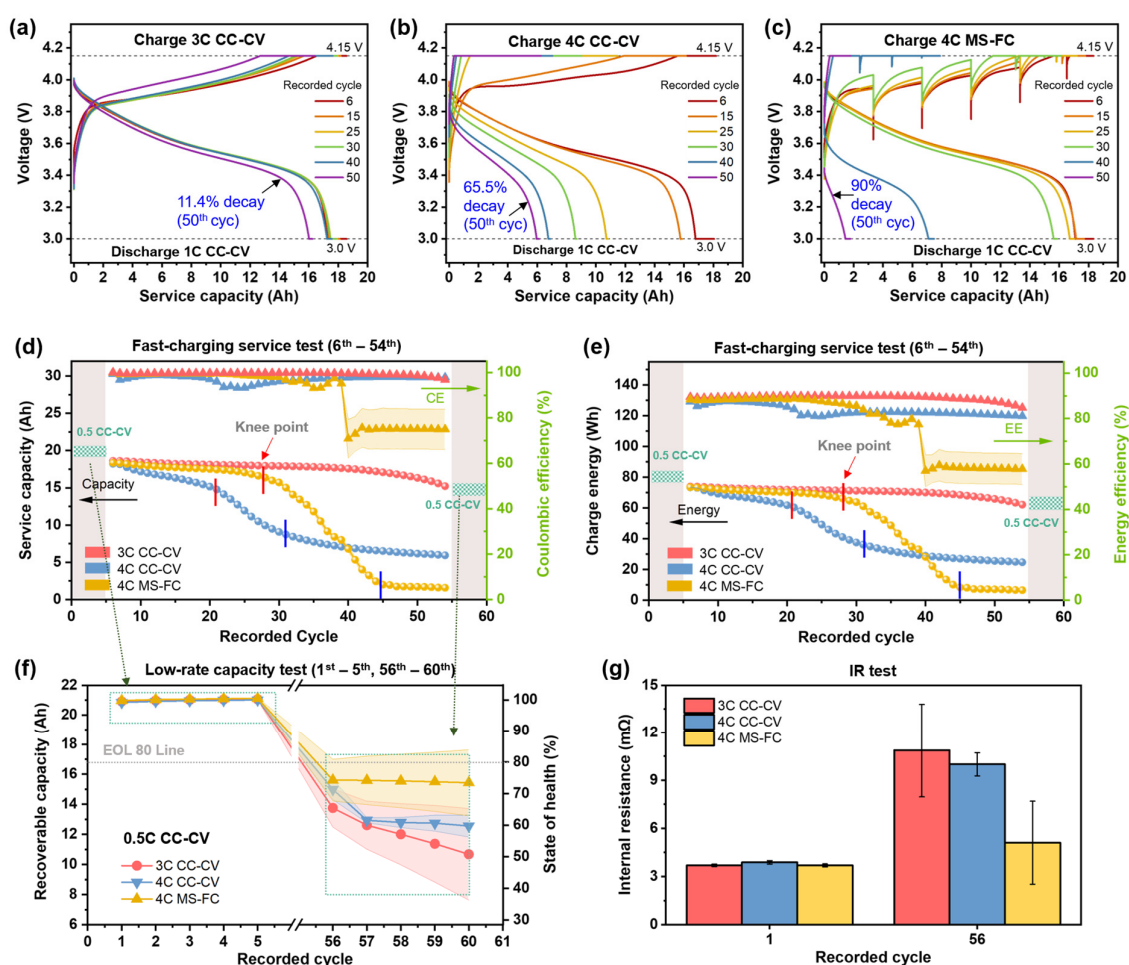


Figure 3. Effect of full SOC fast charging on the aging and internal-resistance evolution of B2 cells. Representative discharge–charge profiles under (a) 3C CC-CV, (b) 4C CC-CV, and (c) 4C MS-FC. (d) Service capacity and Coulombic efficiency; (e) charge energy and energy efficiency vs. recorded cycle number; (f) recoverable capacity measured at 0.5C CC-CV; and (g) internal resistance measured before and after fast-charging service test. In (d), the circle and triangle symbols represent the service capacity and Coulombic efficiency, respectively, and in (e), they represent the charge energy and energy efficiency, respectively. Data in (d–g) are averages of eight cells per group, with error bars representing standard deviations. The dashed line in (f) indicates the 80% SOH threshold.

Importantly, the low-rate recoverable-capacity and IR measurements revealed a different trend (Figure 3f,g). After 50 fast-charging cycles, the 4C MS-FC group exhibited the

highest recoverable capacity and the smallest increase in internal resistance among the three B2 groups, despite showing the lowest service capacity during later fast-charging cycles. This divergence between high-rate service capacity and low-rate recoverable capacity indicates that the apparent capacity loss under 4C MS-FC cannot be attributed entirely to irreversible loss of active materials or cyclable lithium. Instead, a substantial fraction of the unavailable capacity remained accessible at low rates, indicating an important contribution from kinetic limitations and polarization [6]. The relaxation periods therefore appeared to mitigate irreversible resistance growth while being insufficient to preserve high-rate capacity utilization near the upper-voltage limit.

The temporal evolution of service capacity further illustrates this tradeoff. Compared with continuous 4C CC-CV, 4C MS-FC delayed the knee point, the onset of accelerated service-capacity loss, from recorded cycle 21 to 28 (Figure 3d). However, this initial advantage diminished after 30 cycles as cell polarization increased. Following each relaxation period, reapplication of the 4C current caused the cell voltage to rapidly approach the 4.15 V cutoff (Figure 3c). In contrast, the 4C CC-CV cells remained in the CV stage while the current gradually decreased toward the 0.2C cutoff (Figure 3b). Consequently, the two protocols differed not only in the presence of relaxation periods but also in their exposure to high-current conditions near the upper-voltage limit. As aging progressed and polarization increased, repeated 4C pulses increasingly drove the MS-FC cells to the voltage cutoff before the targeted capacity could be accepted. Similar limitations of multi-step CC-CV charging under high polarization have been reported previously [31]. Thus, relaxation alone was insufficient to overcome the kinetic constraints imposed by 4C charging over the full SOC range.

Post-mortem observations supported the electrochemical behavior. As shown in Figure S2, a representative 4C CC-CV cell exhibited severe swelling after cycling, whereas a 3C CC-CV cell showed no visible expansion. The 4C MS-FC cell exhibited less pronounced swelling than continuous 4C CC-CV, suggesting that the relaxation periods mitigated, but did not eliminate, gas-generation side reactions. Gas evolution under high-rate and high-voltage operation is commonly associated with electrolyte decomposition and formation of gaseous products, including hydrocarbons such as CH_4 and C_2H_4 [32]. For the 4C CC-CV cell, prolonged operation near the upper voltage limit (4.15 V), shown in Figure S3, may accelerate electrolyte decomposition, SEI growth, and loss of cyclable lithium [33]. These observations are consistent with the larger efficiency losses observed for 4C groups; however, the individual contributions of gas formation, lithium plating, and electrolyte decomposition were not quantified directly in this study. SEM images of cathodes (Figure S4) collected in the charge state exhibited similar secondary-particle microcracking, with the 4C CC-CV cathode showing greater disruption of secondary-particle morphology than the 3C CC-CV cathode. The 4C CC-CV graphite anode (Figure S5) exhibited a denser surface morphology and reduced porosity, whereas the 4C MS-FC anode retained a more open morphology. These observations suggest that relaxation periods mitigated, although did not eliminate, electrode damage and interfacial degradation under 4C charging.

XRD patterns provide additional evidence of protocol-dependent structural changes (Figure S6). Compared with the 3C cathode, both 4C cathodes exhibited a larger shift in the relevant reflections toward higher 2-theta and greater separation of the (018)/(110) peaks, indicating differences in the lattice state after high-rate cycling [34]. The graphite anodes at 3C and 4C CC-CV showed broader (002) reflections and stronger features associated with lithiated graphite than the 4C MS-FC anode, suggesting greater heterogeneity in the lithiation state and structural disorder [35,36]. Collectively, the electrochemical and post-mortem results reveal a critical limitation of applying MS-FC over the full SOC range: relaxation periods reduced irreversible resistance growth and mitigated electrode

deterioration, but repeated 4C pulses near the upper-voltage cutoff progressively restricted high-rate capacity utilization as polarization increased. These results indicate that effective MS-FC requires not only appropriately designed relaxation periods but also control of charging rate and SOC window. This finding motivated the 3C pulse-controlled charging experiments within a restricted 20–80% SOC window described in Section 3.2.

3.2. First-Life Performance Under SOC-Restricted Multi-Step Fast Charging

The effect of the operating SOC (or cutoff voltage) window was first evaluated by comparing continuous 3C charging of B3 cells within 20–80% SOC (3.385–4.114 V) with continuous 3C charging of B2 cells over the full SOC (3.0–4.15 V). As shown in Figure 4a, the P0C12R0 cells operating within 20–80% SOC exhibited ~6.6% service-capacity loss by recorded cycle 50, corresponding to the 45th fast-charging service cycle. In comparison, the B2 cells subjected to 3C CC-CV exhibited ~11.4% service-capacity loss over the same period (Figure 3a). Restricting the SOC window therefore improved early-cycle stability, consistent with previous reports showing that avoiding deeply discharged and highly charged states can reduce degradation associated with deep cycling and prolonged high-voltage exposure [37,38]. During the second testing round, the P0C12R0 group exhibited ~7.4% service-capacity loss by recorded cycle 100. Service capacity subsequently decreased rapidly during the third round, falling by more than 70% and reaching ~4 Ah by recorded cycle 150. However, a subsequent recoverable capacity test at 0.5C CC-CV exhibited ~85% SOH (~17 Ah) (Figure 5b). The large difference between fast-charging service capacity and low-rate recoverable capacity indicates that the rapid decline in high-rate service capacity did not represent an equivalent loss of total cell capacity. Instead, increasing polarization and kinetic limitations progressively restricted capacity utilization at 3C [39]. Thus, restricting the SOC window delayed the onset of severe capacity limitation relative to full SOC operation, but continuous 3C charging remained insufficient to maintain long-term high-rate service performance.

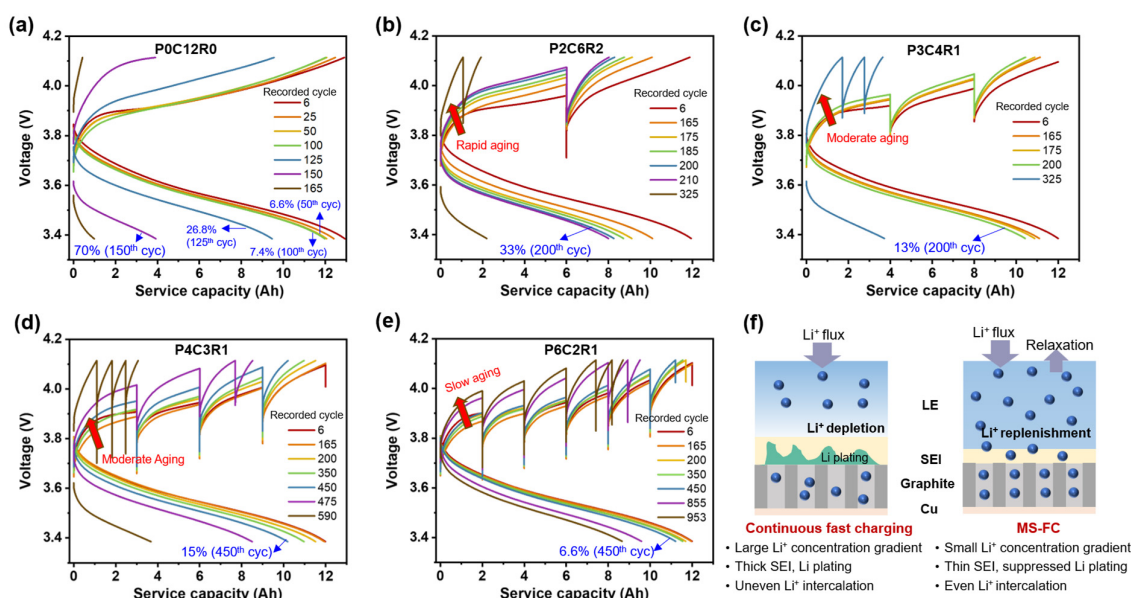


Figure 4. First-life cycling performance of B3 cells under 3C MS-FC within 20–80% SOC. Representative voltage-capacity profiles for (a) P0C12R0, (b) P2C6R2, (c) P3C4R1, (d) P4C3R1, and (e) P6C2R1 protocols. (f) Schematic illustration of Li⁺ intercalation under continuous and MS-FC conditions. Note that the cycling was controlled by the described voltage cutoffs (3.385–4.114 V), so the service capacity of P0C12R0 could exceed the nominal 12 Ah, corresponding to 60% of the rated 20 Ah capacity, but the variation does not affect the comparison of fast-charging performance among the MS-FC protocols.

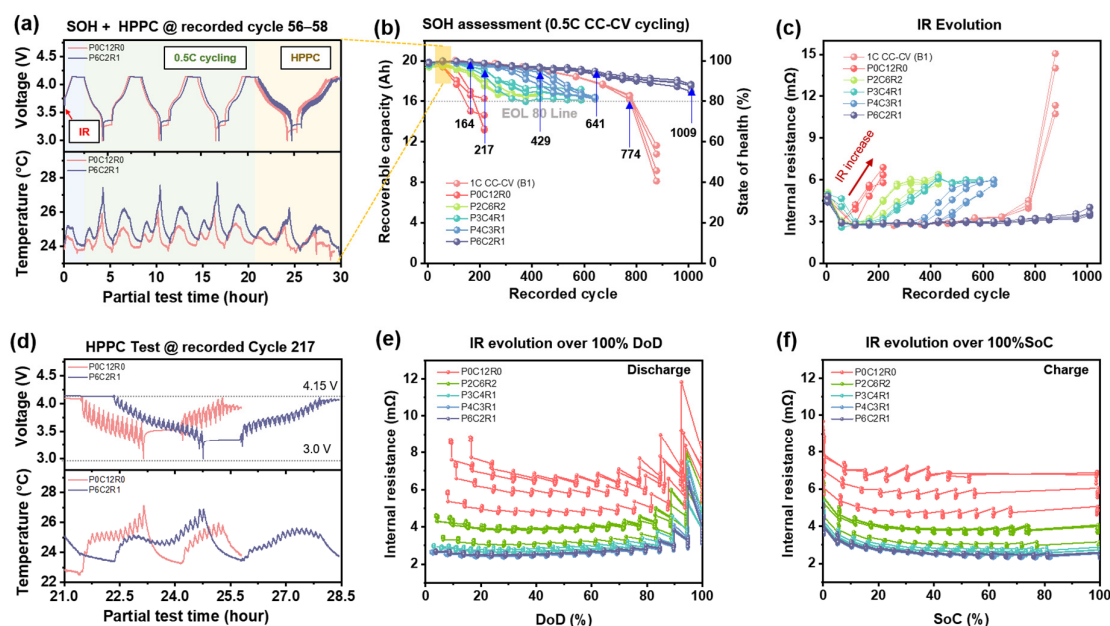


Figure 5. Evolution of SOH and internal resistance during MS-FC cycling of B3 cells. (a) Representative cell-voltage and temperature profiles during capacity characterization performed after each 50-cycle fast-charging interval. (b) Evolution of recoverable capacity and SOH, and (c) internal resistance as a function of recorded cycle number for P0C12R0, P2C6R2, P3C4R1, P4C3R1, P6C2R1, and 1C CC-CV reference group. (d) Representative cell-voltage and temperature profiles during an HPPC test performed at recorded cycle 217. IR evolution during (e) discharge and (f) charging. Each first-life test group contained four cells.

The effect of introducing controlled relaxation periods was therefore systematically investigated within the same 20–80% SOC window. Figure 4b–e compares B3 cells charged at 3C using P2C6R2, P3C4R1, P4C3R1, and P6C2R1 protocols. Importantly, all protocols maintained the same cumulative 3C charging duration of 12 min while varying the number and duration of relaxation periods. The corresponding voltage-current profiles are provided in Figure S7. Increasing the number of charging steps markedly extended the period over which high service capacity could be maintained. At recorded cycle 200, increasing the number of charging steps from two (P2C6R2) to three (P3C4R1) reduced service-capacity loss from ~33% to 13%. At recorded cycle 450, increasing the number of charging steps from four (P4C3R1) to six (P6C2R1) reduced the service-capacity loss from ~15% to 6.6%. These results demonstrate that more frequent relaxation during a fixed cumulative high-rate charging period substantially delayed the loss of usable service capacity. The systematic increase in cycling stability from P0C12R0 toward P6C2R1 strongly indicates that periodic interruption of the 3C charging current alleviated the progressive kinetic limitations observed during continuous charging. A mechanistic interpretation of this behavior is illustrated schematically in Figure 4f. During continuous fast charging, the high Li^+ flux toward the graphite anode can generate steep concentration gradients across the graphite/electrolyte interface and within graphite particles. As the rate of Li^+ arrival exceeds its redistribution and solid-state diffusion, local polarization increases, potentially promoting heterogeneous lithiation, lithium plating, SEI growth, and loss of cyclable lithium [36,40]. Introducing relaxation periods temporarily removes the applied charging current, allowing Li^+ redistribution [14–16]. This redistribution can reduce local polarization, promote more uniform intercalation, and decrease the likelihood of metallic lithium deposition. Previous studies have similarly associated interrupted or pulse charging with reduced interfacial polarization and diffusion-induced stress in graphite anodes [36].

Long-term SOH and internal-resistance evolution under the different MS-FC protocols are compared in Figure 5. Each protocol was evaluated using four cells. The P0C12R0 group fell below the 80% SOH threshold at recorded cycle 217, corresponding to 200 fast-charging service cycles, accompanied by an approximately twofold increase in internal resistance. The P2C6R2 group exhibited substantially slower degradation, retaining ~95% SOH at recorded cycle 164 before entering an accelerated degradation regime. Its SOH subsequently decreased to ~88% at cycle 217, accompanied by a decline in service capacity to below 8 Ah (Figure 4b).

Increasing the number of charging steps and associated relaxation periods produced progressively greater improvements in first-life durability (Figure 5b). At recorded cycle 217, the P3C4R1, P4C3R1, and P6C2R1 groups retained ~90.8%, 92.8%, and 96.8% SOH, respectively. Between recorded cycles 217 and 429, the P2C6R2 and P3C4R1 groups exhibited accelerated SOH decline toward the 80% threshold, whereas the P4C3R1 group showed a more gradual decay. By recorded cycle 641, the P4C3R1 group had decreased to 80% SOH, while the P6C2R1 group still retained 93.4% SOH. Notably, the P6C2R1 group completed 1009 recorded cycles while maintaining >84.7% SOH, demonstrating substantially greater first-life durability than the continuous and lower-step fast-charging groups. Over this extended cycling period, the internal resistance increased only modestly, from 2.96 m Ω to 3.4–4.0 m Ω . The 1C CC-CV reference cells also retained >80% SOH for over 700 cycles, accompanied by a gradual increase in internal resistance. During the subsequent cycles, however, the SOH declined rapidly to 40–50%, while the internal resistance increased by a factor of approximately 4–5 (Figure 5c).

In addition to SOH and single-point internal resistance measurements, HPPC was used to evaluate resistance evolution across the operating SOC range. Successive ~10 s current pulses were applied during stepwise discharge/charge, spanning 0–100% DOD and 0–100% SOC, respectively. The cell-temperature variation during the HPPC measurement remained within ~2 °C (Figure 5d), minimizing the influence of temperature variation on the measured resistance.

The resulting resistance profiles revealed a strong dependence of resistance evolution on the MS-FC protocol. At recorded cycle 58, the five B3 groups exhibited relatively similar resistance profiles (Figure S8), indicating limited differentiation among the protocols during the early stage of cycling. Internal resistance generally increased with increasing depth of discharge and subsequently decreased during recharge, approaching its initial value at high SOC, consistent with previous studies [41,42]. By recorded cycle 217, clear protocol-dependent differences had emerged (Figure 5e,f). The P0C12R0 group exhibited the most pronounced resistance growth, followed by P2C6R2, whereas P3C4R1, P4C3R1, and particularly P6C2R1 showed smaller changes. The progressive suppression of resistance growth with increasingly segmented MS-FC protocols paralleled the corresponding improvements in recoverable capacity, SOH, and high-rate service-capacity retention. These results indicate that the benefit of MS-FC extended beyond temporarily improving capacity utilization: frequent relaxation periods were also associated with slower accumulation of irreversible internal resistance during long-term fast charging.

3.3. Post-Mortem Characterization of Electrode Degradation Under MS-FC Protocols

The cathode and anode sheets, with dimensions of 12 × 19 cm² (Figure 6 and Figure S9), were visually examined immediately after cell disassembly. Localized dark regions were observed on the cathode recovered from the P0C12R0 cell (Figure 6c), whereas the extent of visible discoloration generally decreased with increasing numbers of MS-FC steps (Figure 6c–g). Because discoloration can arise from several sources, including electrolyte residues, surface-

film formation, and transferred electrode material, these photographs alone do not identify its chemical origin.

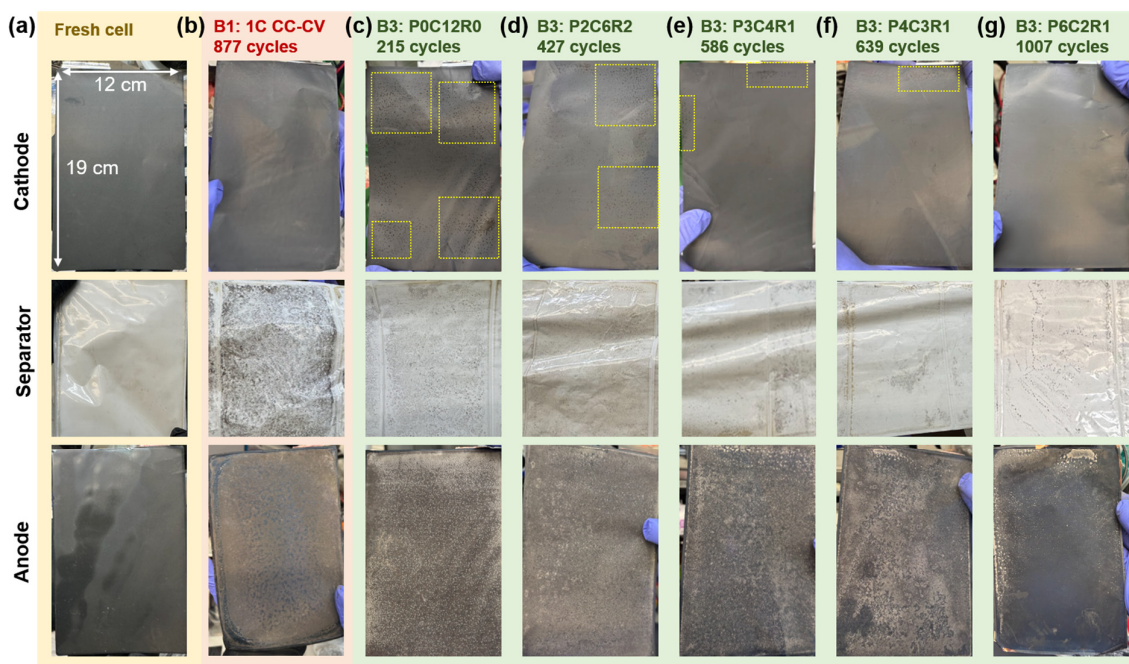


Figure 6. Post-mortem photographs of electrodes and separators recovered from (a) fresh, (b) B1, and (c–g) B3 cells after different cycles. Representative regions from the middle layers of the cathode, separator, and anode sheets are shown after cell disassembly. The corresponding charging protocol and recorded cycle number are indicated above each set of images.

More pronounced physical degradation was observed on the graphite anodes. The anode recovered from P0C12R0 exhibited more visible pulverization and delamination than the anodes subjected to the MS-FC protocols. Brown deposits were also observed on the separator recovered from the P0C12R0 cell. SEM images showed graphite-like particles within these regions (Figure S10), suggesting transfer of detached anode material onto the separator. Magnified photographs of $2.5 \times 2.38 \text{ cm}^2$ anode and separator regions are provided in Figure S11.

Comparison of the different operating conditions further suggests contributions from both SOC-window restriction and charging relaxation. The P0C12R0 cell cycled within 20–80% SOC showed less anode delamination and separator discoloration than the cell subjected to continuous 3C charging over the full SOC range. Within the same 20–80% SOC window, cells subjected to more relaxation steps generally exhibited less visible electrode damage and less extensive separator discoloration. These observations are consistent with reduced mechanical and interfacial degradation under SOC-restricted and MS-FC conditions. However, because the cells were harvested after different accumulated cycle numbers, the individual contributions of charging protocol and total cycling history cannot be completely separated from the post-mortem photographs alone.

Figure 7a compares the XRD patterns of fresh and cycled NMC442 cathodes recovered in the charged state. NMC442 has an O3-type layered structure derived from $\alpha\text{-NaFeO}_2$, in which lithium and transition-metal layers alternate between oxygen planes [43]. During prolonged cycling, migration of Ni^{2+} into lithium sites can impede Li^+ transport and accompany surface reconstruction and cracking of NMC secondary particles [44–46]. The intensity ratio of (003) to (104) peaks, $I(003)/I(104)$, is commonly used as an empirical indicator of layered-structure ordering, with ratios above 1.2 generally associated with lower

$\text{Li}^+/\text{Ni}^{2+}$ disorder [47]. In the present samples, $I(003)/I(104)$ increased with increasing numbers of MS-FC steps (Figure 7a,b), consistent with better preservation of the layered structure. Additional protocol-dependent structural differences are evident in the enlarged diffraction patterns in Figure 7c. The continuously charged cathodes (both 1C CC-CV and P0C12R0 at 3C) exhibited larger shifts of the (101) and (012) reflections toward higher 2theta and greater separation of the (018)/(110) reflections. These changes are consistent with greater anisotropic lattice contraction and expansion during de-lithiation [48]. The weak or poorly resolved (006) reflection in the continuously charged samples may additionally be associated with increased structural disorder [49]. Taken together, the XRD results suggest that increasing the number of relaxation steps reduced the structural changes accumulated in the NMC442 cathode during prolonged fast charging.

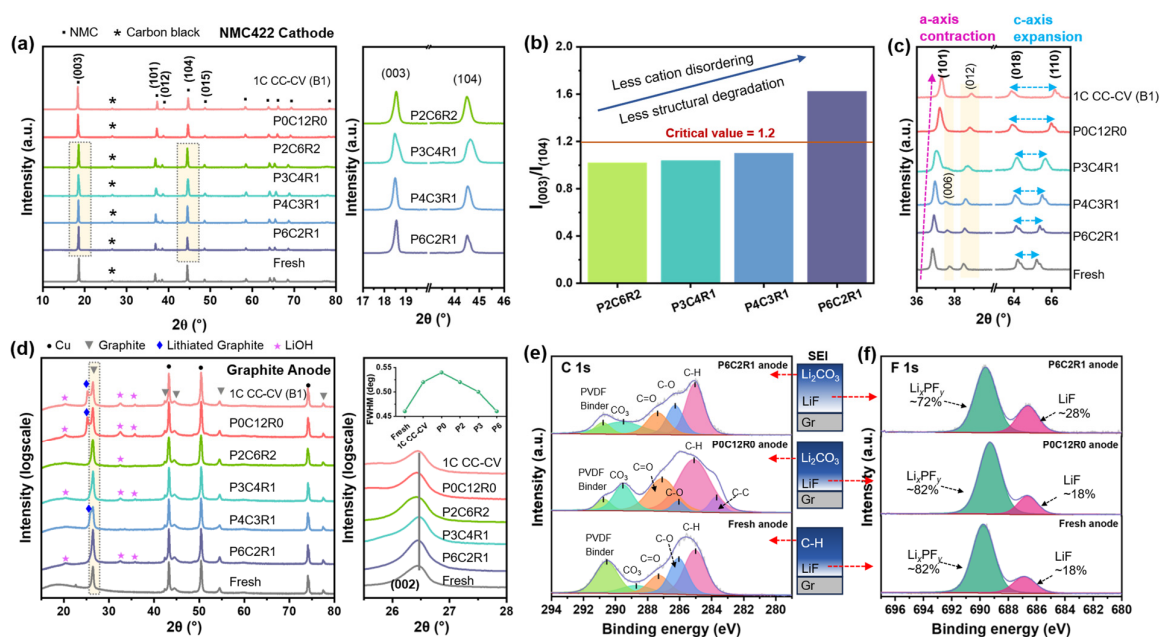


Figure 7. Structural and surface-chemical characterization of post-mortem electrodes. (a) XRD patterns of NMC442 cathodes; (b) intensity ratios of the (003) and (104) peaks; (c) 2-theta range of 36–40° and 63–67°; (d) XRD patterns of graphite anodes; and high-resolution (e) C 1s and (f) F 1s XPS spectra of graphite anodes recovered from fresh, P0C12R0, and P6C2R1 cells.

Figure 7d shows the corresponding XRD patterns of the graphite anodes. A weak additional reflection assigned to LiOH was detected after extended cycling. LiOH may form through reactions involving trace moisture or other lithium-containing surface products [50,51], but its origin cannot be determined from ex situ XRD alone. More importantly, the graphite (002) peak broadened from a full width at half maximum (FWHM) of $\sim 0.46^\circ$ for the fresh anode to $\sim 0.54^\circ$ for P0C12R0. In contrast, the MS-FC anodes exhibited FWHM values closer to that of the fresh anode. The greater (002) peak broadening under continuous 3C fast charging is consistent with increased structural heterogeneity, reduced coherence-domain size, and/or accumulated microstrain within the graphite structure [36]. These structural changes are consistent with the greater physical deterioration observed in P0C12R0.

XPS was subsequently used to compare the graphite surface and SEI chemistry of the fresh, P0C12R0, and P6C2R1 cells. Compared with the fresh anode, the P0C12R0 anode exhibited stronger carbonate-related contributions, including components assigned to Li_2CO_3 (C=O, CO_3), whereas the P6C2R1 anode showed a comparatively stronger LiF contribution in the F 1s spectrum (Figure 7e,f). Depth-sensitive XPS measurements of the P0C12R0 anode further revealed a compositional gradient across the interphase. As shown

in Figure S12a, Ar sputtering decreased the O 1s and C 1s signal intensities associated with surface species. Correspondingly, the carbonate-, C-O-, and C=O-related contributions in the C 1s spectrum decreased after sputtering, while the relatively stronger LiF contribution became more apparent (Figure S12b,c). These observations are consistent with a more carbonate-rich outer SEI and a comparatively LiF-rich inner region, in agreement with previously reported depth-dependent SEI compositions [52]. The different interphase compositions observed for P0C12R0 and P6C2R1 provide a possible chemical basis for their markedly different resistance evolution. Accumulation of carbonate-rich decomposition products during continuous fast charging may contribute to interphase thickening and increased Li^+ -transport resistance. In contrast, LiF-rich inorganic SEI components are generally associated with greater chemical and mechanical stability and can contribute to passivation of graphite against continued electrolyte decomposition [53]. The formation of a thin but robust self-limiting passivation layer on anodes can effectively stabilize interfaces and prevent transition metal crosstalk from NMC cathodes [54]. The P6C2R1 anode exhibited an approximately 10% greater LiF contribution than the fresh-cell anode, consistent with the formation of a more inorganic-rich interphase during cycling. No detectable transition-metal signals were observed in the XPS survey spectra of the analyzed graphite surface in this work [55]. Nevertheless, ion sputtering can induce the formation of LiF artifacts, potentially leading to an overestimation of the LiF content during quantification. XPS peak fractions alone do not establish SEI thickness, ionic conductivity, or causality between LiF content and improved cycling performance; the compositional differences are therefore interpreted in conjunction with the electrochemical and structural observations. Collectively, the post-mortem results establish a consistent relationship between charging protocol and accumulated electrode degradation.

3.4. Second-Life Performance at Reduced C-Rates

B3 cells satisfying the second-life eligibility criteria (>80% SOH without severe swelling or other apparent safety concerns) were subsequently evaluated at 0.25C, 0.5C, or 1C. The allocation of cells for second-life testing is summarized in Table 4, and their cycling behavior is compared in Figure 8 and Figures S13 and S14.

At 0.25C, cells previously subjected to the P0C12R0, P2C6R2, P3C4R1, and P4C3R1 protocols exhibited capacity decay of 6.6%, 2.6%, 2.7%, and 2.0%, respectively, as shown in Figure S13a–d. Their voltage-capacity profiles were also similar (Figure S13e,f). Thus, differences associated with first-life charging history were small under the low-rate 0.25C condition. Testing at 0.5C was therefore used to provide a more discriminating comparison of the effects of first-life charging history. Figure 8a–e compares the second-life cycling at 0.5C within a cutoff voltage of 3.426–4.098 V. At the beginning of second-life operation, cells previously subjected to more MS-FC steps generally delivered higher service capacities. For example, P6C2R1 cells delivered ~13.1 Ah, compared with 10.65 Ah for P3C4R1 and 9.96 Ah for P0C12R0 at recorded cycle 5 (Figure S14a). By recorded cycle 450, the P0C12R0 cell retained 97.2% (9.7 Ah) of its initial second-life capacity, whereas the P2C6R2, P3C4R1, P4C3R1, and P6C2R1 cells retained 56.8% (5.7 Ah), 56.0% (5.9 Ah), 79.4% (8.6 Ah), and 91.0% (11.9 Ah), respectively (Figure S14b). Thus, the P6C2R1 cell delivered the highest absolute capacity at recorded cycle 450, whereas the P0C12R0 cell showed the smallest percentage decline from its lower initial second-life capacity. Given the outstanding second-life performance observed at 0.5C, two additional P6C2R1 cells were further evaluated under the 1C condition (Figure 8f). By recorded cycle 450, the P6C2R1 cells retained 62.3% (5.6 Ah) of their initial second-life capacity.

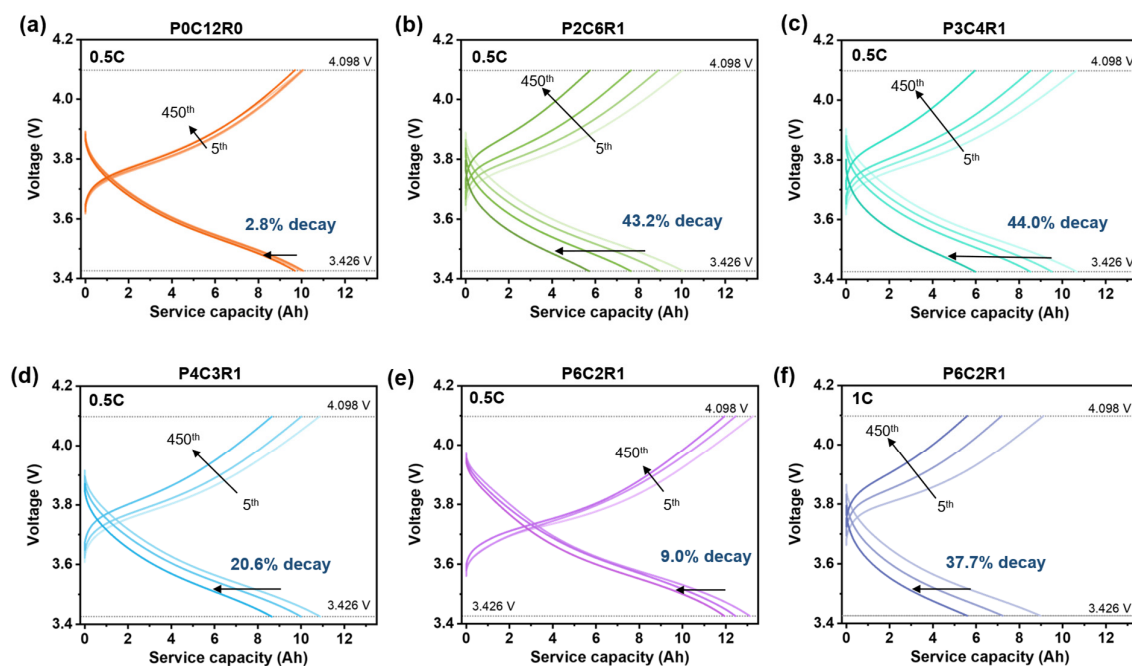


Figure 8. Second-life cycling performance of B3 cells with different first-life charging histories. Representative voltage-capacity profiles from the retired B3 cells previously subjected to (a) P0C12R0, (b) P2C6R2, (c) P3C4R1, (d) P4C3R1, and (e,f) P6C2R1 first-life protocols. Cells in (a–e) were cycled at 0.5C, and the P6C2R1 cell in (f) was cycled at 1C.

SOH and internal-resistance evolution during second-life operation are summarized in Figure 9. At 0.25C, the selected cells exhibited only limited SOH loss and a gradual internal resistance increase (Figure 9a,d). At 0.5C, the P6C2R1 cell completed 1033 recorded cycles while retaining ~80% SOH, corresponding to a recoverable capacity of 15.8 Ah, with an internal resistance remaining below 6 m Ω (Figure 9b,e). The P0C12R0 cell retained ~80% SOH through recorded cycle 621, after which its SOH progressively declined to ~60% SOH by recorded cycle 1033. In comparison, the cells previously subjected to the P2C6R2, P3C4R1, and P4C3R1 protocols exhibited shorter second-life cycling durations and higher internal resistance growth. Two independent P6C2R1 cells were additionally evaluated at 1C (Figure 9c,f). One cell has completed 1239 recorded cycles while retaining ~77.5% SOH and internal resistance reaching ~6 m Ω . Another one failed earlier. This result demonstrates that a cell that previously operated under the six-step MS-FC protocol can retain substantial capacity during extended second-life cycling, even under the 1C rate. Among the tested B3 cells, those previously subjected to P6C2R1 exhibited the highest measured absolute second-life capacities and strong long-term SOH retention. However, because the first-life protocols resulted in different cycle numbers and cumulative energy throughputs before retirement, and because the number of cells available for each second-life condition was limited, the influence of charging protocol cannot be completely separated from accumulated first-life history and cell-to-cell variability.

3.5. Relationship Between First-Life Aging and Second-Life Performance

Pearson correlation coefficient analysis was used to explore associations between first-life descriptors and second-life performance (Figure 10a). The first-life variables included pulse number, end-of-first-life SOH, end-of-first-life internal resistance, and the number of fitted degradation modes. Second-life variables included internal resistance at cycle 516 and SOH decay rate. Because the analysis contained only ten cells and several descriptors were derived from the same SOH trajectories, the coefficients are interpreted as exploratory associations rather than evidence of causation. The correlation matrix

suggests that second-life internal resistance growth and SOH decay were associated not only with end-of-first-life internal resistance and SOH, but also with the shape of first-life aging trajectory. This observation is consistent with previous reports that batteries retired at similar SOH can exhibit different second-life behavior because of differences in their first-life operating histories [26].

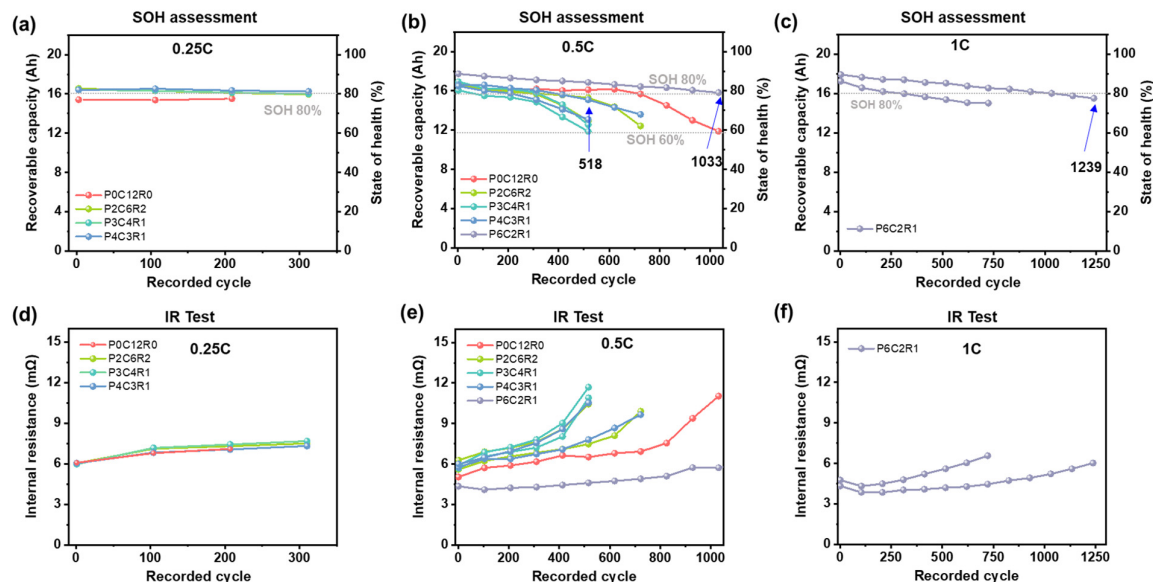


Figure 9. Evolution of SOH and internal resistance during second-life cycling. Recovery capacity and SOH evolution at (a) 0.25C, (b) 0.5C, and (c) 1C. Corresponding internal resistance evolution at (d) 0.25C, (e) 0.5C, and (f) 1C.

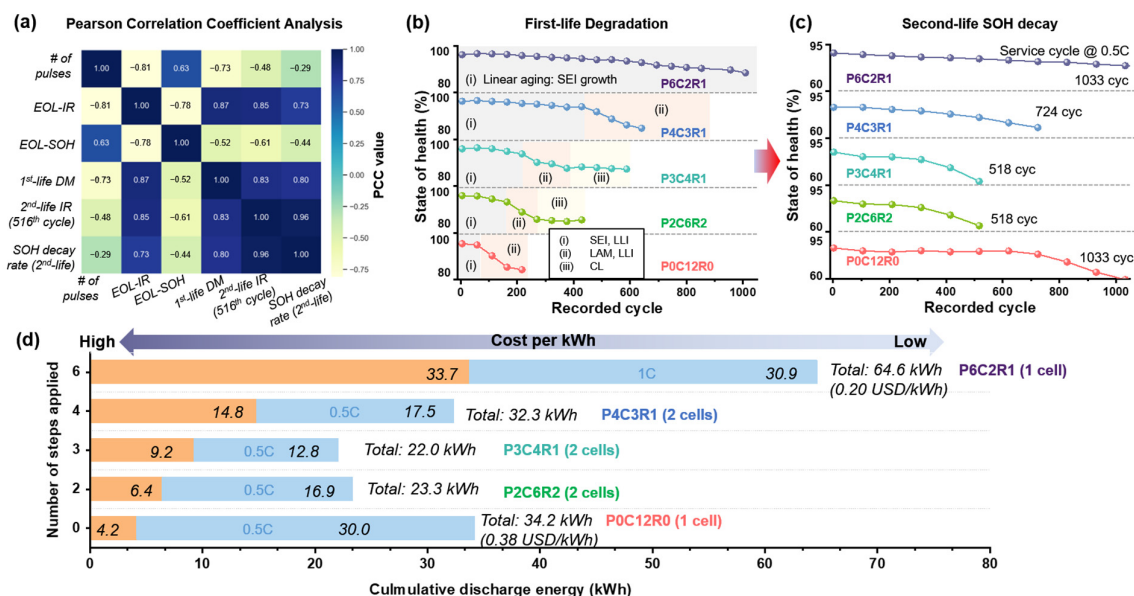


Figure 10. Correlation of first-life degradation with second-life performance and cumulative lifetime energy utilization. (a) Pearson correlation coefficient analysis of key first-life and second-life performance descriptors. (b) First-life aging regimes and (c) the corresponding second-life cycling performance for B3 cells. (d) Cumulative total discharge energy delivered during B3 first-life fast-charging operation and subsequent second-life cycling.

Figure 10b compares the phenomenological aging regimes identified from piecewise-linear fits to the first-life SOH curves. All cells initially exhibited gradual SOH loss (i). The P0C12R0 cell entered an accelerated degradation regime (ii) between approximately

recorded cycles 50 and 100. Gradual early-life capacity loss may be consistent with SEI growth and loss of lithium inventory (LLI), while accelerated degradation may involve increasing polarization, lithium plating, loss of active material (LAM), and disruption of ionic and electronic pathways [27]. The post-mortem observations in Section 3.3 provide complementary evidence for structural and interfacial degradation. In comparison, P2C6R2, P3C4R1, and P4C3R1 cells exhibit delayed transitions near recorded cycles 162, 215, and 427, respectively. P2C6R2 and P3C4R1 cells also exhibited a second transition to a slower apparent degradation rate (iii) after approximately 268 and 374 cycles, respectively. This late-stage flattening should not be interpreted as recovery or stabilization of battery health. As accessible capacity declines, transport limitations and loss of conductive pathways may reduce the fraction of active material participating in each cycle, producing an apparently lower capacity-decay rate. Accordingly, the fitted regimes are described as (i) gradual, (ii) accelerated, and (iii) late-stage transport-limited aging. Most notably, the P6C2R1 cell maintained a comparatively gradual SOH decline for more than 1000 recorded cycles, accompanied by limited resistance growth (Figure S15). Thus, increasing the number of charging steps and associated relaxation periods was associated with a delayed onset of accelerated first-life aging.

Figure 10c relates these first-life aging trajectories to subsequent second-life performance. Cells exhibiting more complex first-life aging generally showed poorer second-life retention. P6C2R1 cells, which maintained a gradual degradation regime, exhibited both the strongest first-life durability and favorable long-term second-life SOH retention. In contrast, P2C6R2 and P3C4R1 cells, which exhibited three first-life aging regimes, showed rapid second-life degradation. P0C12R0 and P4C3R1 each exhibited two first-life aging regimes but differed substantially in the duration of accelerated aging and in their subsequent second-life behavior. These observations suggest that the shape and transition points of the first-life SOH trajectory may provide information about subsequent cell performance that is not captured by the retirement SOH alone. However, because this analysis is based on a limited number of cells with unequal first-life histories, this relationship should be considered hypothesis-generating and requires validation using larger, independently replicated datasets.

Cumulative discharge energy was used to account for differences in first-life duration and second-life performance (Figure 10d). Across the measured first- and second-life tests, the P6C2R1 cell delivered a cumulative discharge energy of ~64.6 kWh, compared with ~34.2 kWh for P0C12R0. This result demonstrates that extending first-life operation through an appropriately designed MS-FC protocol can provide a substantial lifetime energy benefit, even when cells enter second life with different cycling histories. As an illustrative measure of cell utilization, dividing an assumed cell market price of USD 12.91 by the cumulative discharge energy yield ~USD 0.38 kWh⁻¹ for P0C12R0 and USD 0.20 kWh⁻¹ for P6C2R1 corresponds to an ~47% reduction. This simplified calculation is not a levelized cost of storage or full techno-economic analysis. Nevertheless, the calculation illustrates the practical importance of evaluating fast-charging strategies in terms of total lifetime energy delivered rather than first-life cycle count or retirement SOH alone.

4. Conclusions

This study evaluated how charging rate, SOC window (or cutoff voltage), and multi-step fast charging (MS-FC) affect the first- and second-life performance of commercial 20 Ah NMC442/graphite pouch cells. Full SOC 4C charging caused rapid loss of service capacity, reduced Coulombic and energy efficiencies, and cell swelling. Restricting operation to 20–80% SOC improved cycling stability, while introducing relaxation periods further delayed the transition from gradual to accelerated degradation. Among the evaluated MS-FC

protocols, P6C2R1 achieved the strongest first-life performance, completing 1009 recorded cycles with >84.7% SOH and only modest internal-resistance growth. Post-mortem photographs, SEM, XRD, and XPS showed that MS-FC was associated with less electrode deterioration, smaller electrode structural changes, and a LiF-rich interphase, consistent with suppressed structural and interfacial degradation. The benefits extended into second-life operation, where P6C2R1 cells maintained substantial recoverable capacity during prolonged cycling. Piecewise and exploratory correlation analyses further suggest that the first-life aging trajectory may provide information about second-life potential beyond retirement SOH alone. Across the measured first- and second-life tests, P6C2R1 delivered 64.6 kWh of cumulative discharge energy, compared with 34.2 kWh for P0C12R0, representing an ~89% increase in lifetime energy delivery and ~47% reduction in cost per kWh. These results highlight MS-FC as a practical battery-management strategy for balancing fast charging, degradation mitigation, and second-life value. Further optimization of charging protocols using machine learning algorithms, such as artificial neural networks, could be explored in future studies to dynamically adapt charging strategies to the complex operating conditions of EV batteries.

Supplementary Materials: The following supporting information can be downloaded at <https://www.mdpi.com/article/10.3390/batteries12100403/s1>. Figure S1. Open-circuit voltage measured at different controlled state-of-charge in the first charge cycle of capacity test from B3 experiment. The cell voltage data points were the average of 24 cells from B3 group, with standard deviations presented as the orange shade. The nominal 3.65 V is equivalent to 30% SOC. Figure S2. Photographs of dismantled B2 pouch cells, cycled over the full SOC range. Side views of the electrode stacks from (a) a fresh, uncycled cell; (b) a cell cycled using 3C CC-CV; (c) a cell cycled using 4C CC-CV; and (d) a cell cycled using 4C MS-FC. The orange and green arrows indicate the anode Cu current collector and cathode Al current collector, respectively. Figure S3. The partial test time profiles of (B2) 3C CC-CV (orange) and 4C CC-CV (blue) representative cells. The voltage cut-offs were 3.0 and 4.15 V, with 100% SOC. An example cycle includes steps of 1C CC-CV discharge, 1h rest interval, and fast charging at 3C CC-CV and 4C CC-CV, respectively. Figure S4. SEM images of cathode active materials recovered from B2 pouch cells after cycling and stopped in the charged state. (a1, a2) 3C CC-CV, (b1, b2) 4C CC-CV, (c1, c2) 4C MS-FC, and (d1, d2) fresh cathode without cycling. Figure S5. SEM images of graphite anodes recovered from B2 pouch cells after cycling and stopped in the charged state. (a1, a2) 3C CC-CV, (b1, b2) 4C CC-CV, (c1, c2) 4C MS-FC, and (d1, d2) fresh anode without cycling. Figure S6. XRD patterns of cathode and anode active materials. (a, b) XRD patterns of NMC cathodes recovered from fresh, 3C CC-CV, 4C CC-CV, and 4C MS-FC cells and (c, d) corresponding XRD patterns of graphite anodes. Figure S7. Representative voltage and current profiles as a function of test time for (a) P2C6R2, (b) P3C4R1, (c) P4C3R1, and (d) P6C2R1. Figure S8. HPPC measurements of B3 cells at recorded cycle 58. Internal resistance measured during (a) pulse-discharge steps from 4.15 to 3.0 V, corresponding to 0–100% depth of discharge (DOD), and (b) pulse-charge steps from 3.0 to 4.15 V, corresponding to 0–100% state of charge (SOC). Figure S9. Photographs of a graphite-anode sheet recovered from an uncycled pouch cell, showing its dimensions: (a) width, approximately 12 cm; and (b) length, approximately 19 cm. Figure S10. SEM images of the separator surface facing the graphite anode in a B3 cell (P0C12R0) after 215 recorded cycles. (a) Localized region corresponding to a brown deposit observed on the separator; (b) higher-magnification image showing graphite-like particulate residue. Figure S11. Magnified photographs of graphite-anode and separator surfaces from uncycled and cycled pouch cells, acquired immediately after cell disassembly. From top to bottom, the images show the anode (left) and separator (right) from the uncycled cell, 3C CC-CV cell cycled over the full SOC range, P0C12R0 and P6C2R1 cells cycled within 20–80% SOC. Figure S12. XPS spectra of P0C12R0 graphite anode before and after Ar sputtering. (a) Survey spectra before and after 1 min of sputtering, showing decreased O 1s and C 1s signal intensities after surface removal; high-resolution (b) C 1s and (c) F 1s spectra after sputtering. Figure S13. Second-life cycling performance at 0.25C. Voltage-capacity profiles of retired B3 cells previously subjected to (a) P0C12R0,

(b) P2C6R2, (c) P3C4R1, and (d) P4C3R1 first-life protocols. Overlaid voltage-capacity profiles of the four groups at (e) recorded cycle 15 and (f) recorded cycles 200/262. Figure S14. Second-life cycling performance at 0.5C. Overlaid voltage-capacity profiles of retired B3 cells previously subjected to P0C12R0, P2C6R2, P3C4R1, P4C3R1, and P6C2R1 first-life protocols at (a) recorded cycle 5 and (b) recorded cycle 450. Figure S15. Internal-resistance evolution measured before and after each round of B3 first-life fast-charging tests. Representative B3 cells previously subjected to P0C12R0, P2C6R2, P3C4R1, P4C3R1, and P6C2R1 were subsequently evaluated at 0.5C during second-life cycling.

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