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Exponential-Offset Modeling and XRD Correlation of SOH Degradation in $LiFePO_4$ Batteries under Extreme Loading

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Abstract

Understanding the relationship between electrochemical degradation and structural changes is critical for improving the reliability of lithium-ion batteries. In this study, the state of health (SOH) of 18650-type lithium iron phosphate ($LiFePO_4$, LFP) cells was evaluated under extreme loading using discharge resistances of 2.5 Ω and 0.005 Ω. The SOH decreased sharply after the first cycle and then declined more gradually, and the degradation trend was well described by an exponential-offset model with RMSE = 2.87, MAE = 2.25, and R^2 = 0.90. Structural analysis was performed by X-ray diffraction (XRD) on electrode samples taken after one discharge at 2.5 Ω (L1), 100 discharges at 2.5 Ω (L100), and one discharge at 0.005 Ω (LD). The XRD results confirmed that the main phase was graphite, but with reduced peak intensities, peak broadening, and increased background noise, indicating crystallinity loss and partial amorphization. These findings demonstrate that SOH degradation is strongly correlated with the decline in crystallinity, and that extreme loading can trigger significant structural deterioration even within a single discharge.

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Abstrak

Pemahaman mengenai hubungan antara degradasi elektrokimia dan perubahan struktur sangat penting untuk meningkatkan keandalan baterai lithium-ion. Pada penelitian ini, state of health (SOH) dari sel lithium iron phosphate ($LiFePO_4$, LFP) tipe 18650 dievaluasi di bawah kondisi pembebanan ekstrem menggunakan hambatan pelepasan 2,5 Ω dan 0,005 Ω. Nilai SOH menurun tajam setelah siklus pertama dan kemudian mengalami penurunan yang lebih lambat, dengan tren degradasi yang dapat digambarkan dengan baik menggunakan model exponential-offset dengan RMSE = 2,87, MAE = 2,25, dan R^2 = 0,90. Analisis struktur dilakukan menggunakan X-ray diffraction (XRD) pada sampel elektroda yang diambil setelah satu kali pelepasan pada 2,5 Ω (L1), 100 kali pelepasan pada 2,5 Ω (L100), dan satu kali pelepasan pada 0,005 Ω (LD). Hasil XRD menunjukkan bahwa fase utama adalah grafit, tetapi dengan intensitas puncak yang menurun, pelebaran puncak, serta peningkatan derau latar (background noise), yang mengindikasikan penurunan kristalinitas dan kecenderungan menuju amorf. Temuan ini menunjukkan bahwa degradasi SOH sangat berkorelasi dengan penurunan kristalinitas, serta bahwa pembebanan ekstrem dapat memicu kerusakan struktural signifikan bahkan hanya dalam satu kali pelepasan.

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1. Introduction

The increasing demand for electric vehicles and renewable energy systems has driven the need for reliable energy storage materials (Latif et al., 2025). Lithium-ion batteries are widely used due to their high energy density, long cycle life, and stable performance (Ngoy et al., 2025). Among the available cathode chemistries, lithium iron phosphate ($LiFePO_4$, LFP) is regarded as one of the most promising materials because it is safe, thermally stable, relatively low cost, and capable of delivering adequate capacity compared with other candidates such as lithium cobalt oxide ($LiCoO_2$) and lithium nickel manganese cobalt oxide (NMC), which suffer from higher cost and safety concerns (Hasan et al., 2025).

The assessment of the state of health (SOH) of lithium-ion batteries in different working conditions is critical to security use and lifetime prediction (Lei et al., 2024). SOH provides information on the ratio of maximum capacity at rated cycles relative to the nominal value, and its degradation characteristics can be highly load dependent. In cases of extreme loading, failure can occur suddenly, and SOH characterisation is critical.

An appropriate mathematical model is needed to demonstrate SOH degradation and predict subsequent SOH. (Y. Wang et al., 2025). Traditional regression has been used in previous research (Huang et al., 2021; Yogianto et al., 2025), but the model does not capture the common pattern of an initial rapid decline followed by a slower one. In such cases the exponential offset model would be a better representation of this behavior and would yield more accurate degradation trend predictions.

Besides the electrochemical performance of the electrode materials, a deep understanding of their structure evolution is also very important to understand the degradation processes and estimate whether electrodes are applicable for reuse or recycling. XRD offers crucial insights into the crystallinity of the electrode based on derivatives between peak intensity, breadth, and background level that indicate the extent of lattice disorder and the structural disorder (Kano et al., 2025). Establishing the relationship between SOH degradation and XRD results is therefore essential to explain how the electrochemical performance loss corresponds to the internal structural changes occurring in the electrode.

Before, we investigated the degradation of $LiFePO_4$ cathodes under various conditioning treatments, observed its morphology using Scanning Electron Microscopy (SEM), correlating it with SOH, and modeling it with linear regression. (Yogianto et al., 2025). From the study, we confirmed that particle fracture and agglomeration on the cathode surface are induced with extreme loads and increased cycles, and this fact can be attributed to the decrease in SOH. However, the study was limited to surface morphology and linear degradation models without understanding the crystallographic changes of $LiFePO_4$ which have not been studied.

In this paper, SOH degradation of 18650-type LFP cells was investigated under extreme loading at 2.5 Ω and 0.005 Ω. Unlike the earlier linear regression approach, an exponential-offset model was applied to represent the degradation more accurately, and structural changes were examined using XRD after 1 discharge, 100 discharges, and 1 discharge at 0.005 Ω. This combined modeling and structural approach provides new evidence that rapid SOH decline is directly associated with crystallinity loss under extreme loading conditions, thereby complementing and extending our previous SEM-based findings.

2. Method

Commercial cylindrical 18650-type $LiFePO_4$ cells with an original capacity of 1800 mAh and a nominal voltage of 3.2 V were used in this study. The cells were tested under two extreme discharge loads: 2.5 Ω and 0.005 Ω. These loading conditions were chosen to accelerate degradation and investigate the impact of different levels of degradation on battery performance and its crystal structure.

Charge–discharge experiments were carried out using an automated measurement system consisting of an Arduino Uno microcontroller, voltage sensors, relay modules, load circuit, battery and power supply. Charging was carried out up to 3.5 V, while discharging was terminated at 0.033 V. The system was designed to control charging and discharging automatically and to record voltage–current data throughout the tests. The schematic diagram of the experimental setup is presented in Fig. 1 (Yogianto, 2025).

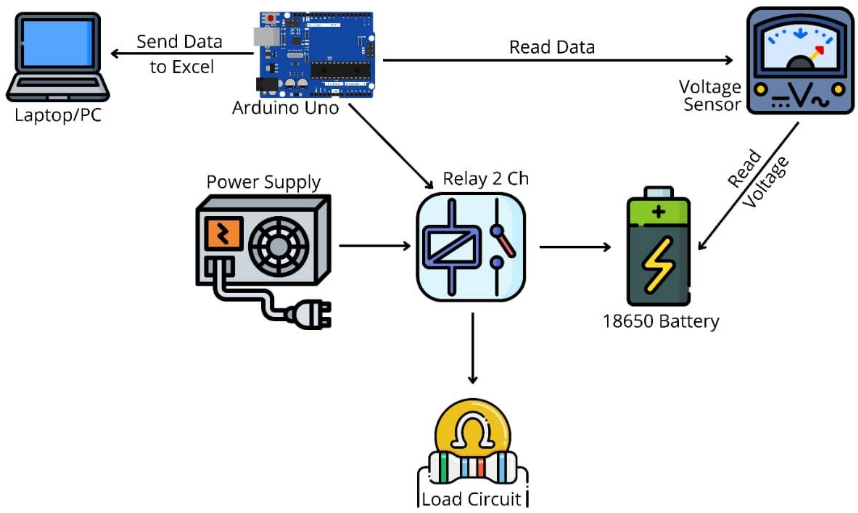

Figure 1. Schematic diagram of the experimental setup

Capacity was calculated from the charging process using the relation:

$$C = i \times t \quad (1)$$

where i is the charging current (3.6 A) and t is the charging time. SOH (%) was then calculated from the measured capacity relative to the original capacity according to:

$$\text{SOH (\%)} = \frac{C_{Current}}{C_{original}} \times 100 \quad (2)$$

where $C_{Current}$ is the measured capacity at a given cycle $C_{original}$ is the previous full charge capacity, and $C_{original}$ is the manufacturer-specified nominal capacity (1800 mAh).

The experimental SOH data were fitted to an exponential-offset model to capture the rate of degradation. Curve fitting and model evaluation were executed by built-in functions of Excel, including RMSE (root mean square error), MAE (mean absolute error), and R^2. The model was made to show how things break down quickly in the first cycle and more slowly in the next cycles, which is a truer way to show the breakdown process than a linear model.

Electrode samples for XRD characterisation were prepared by disassembling fully discharged cells carefully. Safety procedures were observed during disassembly to prevent a fire or explosion, and personal protective equipment (PPE) was used. The cell voltage was first measured, and it was verified that the potential was lower than 2.0 V so that the battery was safe to open (Rouhi et al., 2021). After opening the casing, the jellyroll electrodes were pried apart and allowed to dry in open air to evaporate the remaining electrolyte without water contact because of material reactivity. The electrodes were then cut to the appropriate size for the XRD measurement after drying.

The XRD measurements were collected with a Cu–Kα radiation (λ = 1.5406 Å) in the 2θ range of 10° to 80° by a step size of 0.02°. Peak intensity, peak width and background level changes were investigated as signs of loss crystallinity and partial amorphization. Three samples were investigated: one after the first discharge at 2.5 Ω (L1), another after 100 discharges at 2.5 Ω (L100) and for the third sample, a single discharge experiment was performed at 0.005 Ω (LD). These labels will be used universally in the discussion to describe the structural changes under distinct loading conditions, which enables a direct comparison between electrochemical performance and crystallography evolution.

3. Results and Discussions

To show how extreme loading affects capacity retention, the charge–discharge patterns from the first five cycles were compared to those from the last five cycles. This comparison makes it easier to see how the battery's performance changed during the course of the testing. It also shows how much performance drops when there are really high resistive loads, which helps us understand how quickly things break down when they are under such heavy loads.

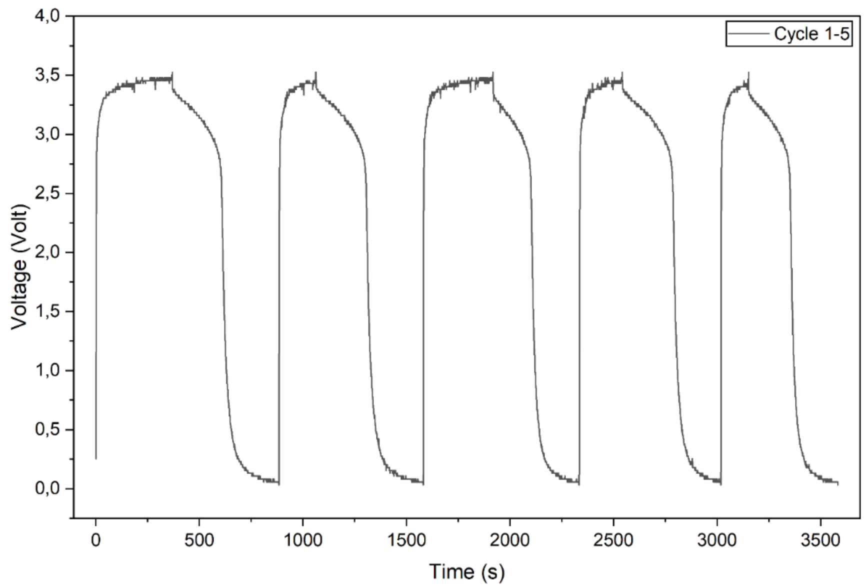

Figure 2. Charge–discharge profiles of the LFP battery during the first five cycles at a 2.5 Ω load

Figure 2 shows the charge–discharge curves of the first five cycles at a 2.5 Ω load. Despite the cell exhibiting prolonged charging and discharging times with voltage plateaus at 3.3–3.5 V, the initial cycle yielded a capacity of roughly 368 mAh, far lower than the rated capacity of 1800 mAh. This steep decay demonstrates that imposing extreme resistive loads has resulted in significant degradation from the onset of operation. As another example, Wei and Wu (2024) found that $LiFePO_4$ cells cycled at moderate current rates revealed almost no capacity decline in the first 5 cycles, preserving over 98% SOH (Wei & Wu, 2024). Similarly, Sun et al. (2018) reported that they only found a minor lengthening of the voltage plateau after initial few cycles and the capacity was maintained higher than 95% (Sun et al., 2018). These comparisons clearly show that the extreme loading used in the present study caused far more rapid degradation even during the early cycling stage.

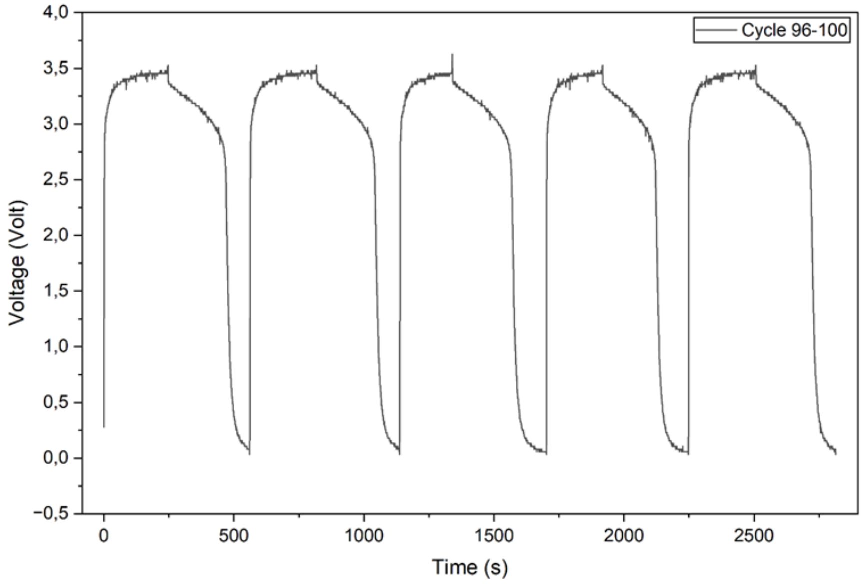

Figure 3. Charge–discharge profiles of the LFP battery during the last five cycles (96–100) under extreme loading.

Figure 3 shows the charge–discharge curves of the last five cycles (cycles 96–100). Compared with the early cycles, both the charging and discharging durations became much shorter, and the voltage plateaus nearly disappeared, indicating a severe loss of active lithium and increased polarization within the electrode. The shortening of the plateau in this stage suggests higher polarization and slower lithium-ion transport, consistent with earlier studies on LFP cells cycled at high rates, where the plateau voltage region becomes less visible as cycling proceeds (L. Wang et al., 2022). The charging capacity in cycle 100 was approximately 259 mAh, which corresponds to about 14.4% of the rated nominal capacity (SOH ≈ 14%). In contrast, Wei & Wu (2024) reported that $LiFePO_4$ cells subjected to moderate cycling conditions still retained more than 90% SOH after 100 cycles (Wei & Wu, 2024), and Sun et al. (2018) observed a gradual capacity decrease to about 85% after 200 cycles under 1C rate (Sun et al., 2018). These comparisons clearly demonstrate that the extreme loading used in this study resulted in much faster degradation, with nearly complete loss of usable capacity by the 100th cycle.

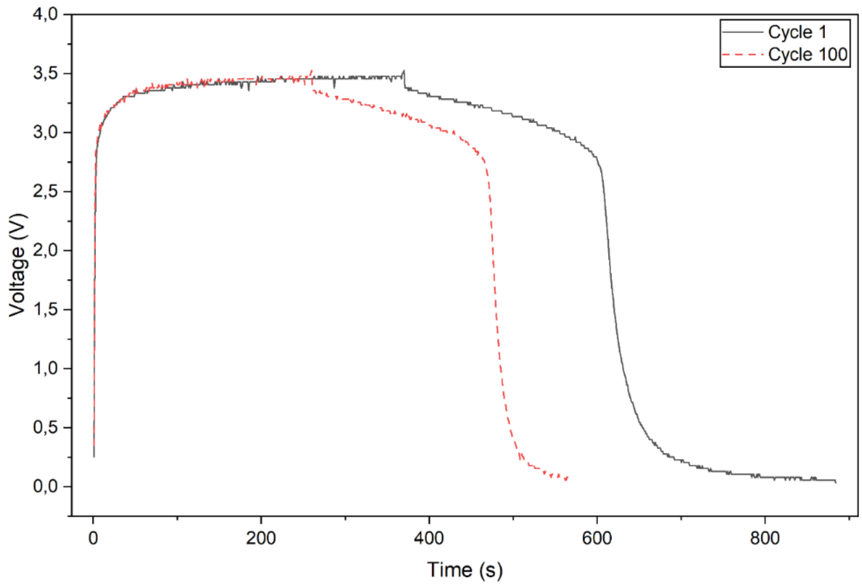

Figure 4. Charge–discharge profiles of the LFP battery comparing cycle 1 and cycle 100

The overall trend is highlighted more clearly in **Figure 4**, which directly compares cycle 1 and cycle 100. The cell initially delivered about 368 mAh, but after 100 cycles, this value had decreased to approximately 259 mAh. The discharge profile also showed a shorter plateau and a reduced delivered capacity. These results confirm that the SOH declined drastically as cycling progressed. This observation aligns with reports in the literature showing that under high-rate or extreme cycling, electrochemical and structural degradation can accelerate substantially in LFP electrodes(Pender et al., 2020; L. Wang et al., 2022).

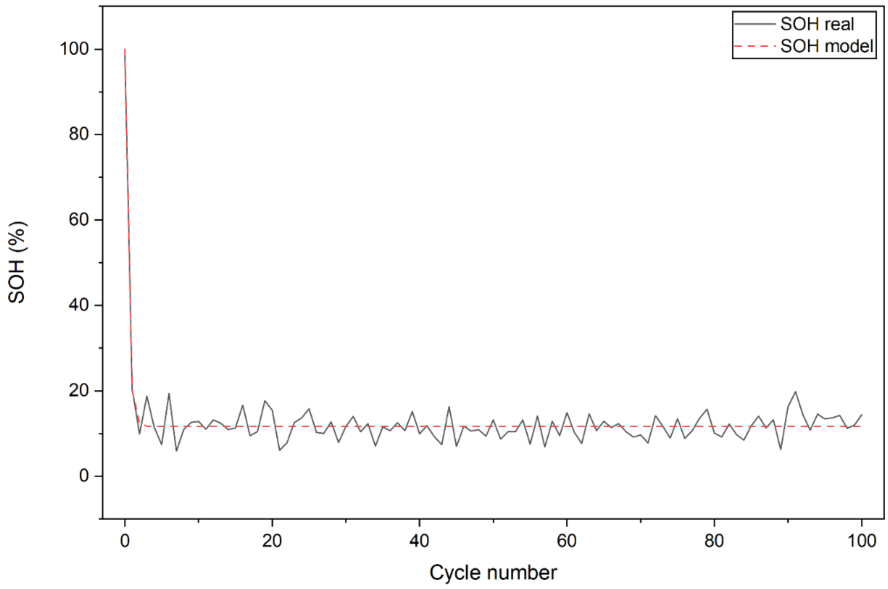

Figure 5. SOH degradation of the $LiFePO_4$ cell at a 2.5 Ω load fitted with the exponential-offset model

Figure 5 shows the SOH degradation of the $LiFePO_4$ cell under extreme loading, together with the exponential offset fitting. The experimental data exhibited a very sharp drop in SOH after the first cycle, where the value decreased from 100% to less than 20% by the second cycle. After this abrupt decline, the SOH fluctuated around much lower values, showing only gradual changes during the remaining cycles. This pattern of degradation is significantly different from the usual SOH trends found in the literature, which typically exhibit a nearly linear decline when cells are used under moderate or normal loading conditions. For example, Wei & Wu (2024) observed a more gradual, quasi-linear reduction in SOH during standard charge–discharge cycles.(Wei & Wu, 2024).

The exponential-offset model successfully described this behaviour, as it is able to represent both the steep initial drop and the slower trend at later stages. In this work, the SOH trend was modelled using the exponential-offset function expressed as:

$$SOH(n) = a \cdot e^{-b \cdot n} + c_0 \quad (3)$$

where n is the cycle number, a represents the initial amplitude of degradation, b is the decay constant, and c_0 is the offset corresponding to the residual SOH after prolonged cycling. The fitting parameters were obtained as a = 88.39, b = 2.34, and c_0 = 11.62. The quality of the fit was confirmed by error evaluation, yielding RMSE = 2.87, MAE = 2.25, and R^2 = 0.90. Compared with the linear regression model applied in our previous work, which gave an RMSE of 9.11, the exponential-offset approach clearly provided a better representation of the degradation behaviour (Yogianto, 2025). This improvement demonstrates that non-linear modelling is more appropriate for describing SOH loss under harsh conditions.

Most previous studies of LFP degradation under moderate cycling conditions have relied on linear or near linear models, as the capacity fade in those regimes typically follows a gradual trend (Sivalertporn et al., 2025; Zhang et al., 2023). Although these approaches can describe long-term behaviour under mild conditions, they fail to capture the sharp early drop observed in our case. Contrastingly, the exponential-offset model offers both accuracy and physical relevance, as it captures the rapid initial fading and subsequently more gradual decline from optimal performance levels in later cycles.

The catastrophic nature of the LD sample also indicates the sensitivity of an electrode under the extreme loading. After a single discharge at 0.005 Ω, the cell could no longer be recharged, resulting in zero measurable capacity and SOH. Although the SOH degradation model fails to capture this behavior, the apparent decrease in electrical performance warrants further investigation, especially when compared with the structural properties revealed by the XRD results in Fig. 6.

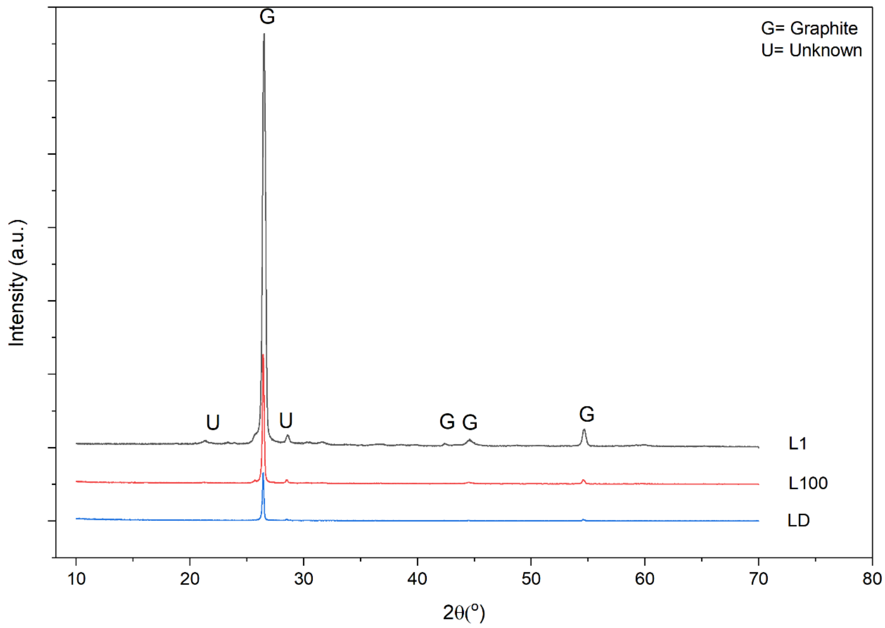

Figure 6. XRD patterns (Cu Kα radiation) of samples L1, L100, and LD

The XRD patterns of the electrode samples L1, L100, and LD are shown in **Figure 6**. In all cases the primary phase was determined to be graphite, with the strongest peaks around 2θ = 26.5° corresponding to the (002) plane and weaker diffraction near 44° and 54° that can be indexed to reference data for graphite (ICDD PDF33-1487)) (Zheng et al., 2025). The crystalline peaks of the L1 cathode samples are sharp and strong, with the (002) peak intensities ≈ 33224 counts after a single discharge at 2.5 Ω, suggesting that cathodes remained high crystallinity in initial cycling stage.

Significant structural changes were already induced by 100 discharges at the same resistance (L100). The intensity of the (002) peak reduced to~11374 counts, which is nearly 66% decrease from that of L1. The peak also broadened and the background level increased, indicating a partial amorphization and a reduction in long range order. These structural damage results are consistent with the electrochemistry results which shows the cell with shorter charge–discharge circle and lower capacity retention.

The most severe damage was observed in the LD that experienced only one discharge at 0.005 Ω Here, the intensity of the (002) peak decreased to only 3,318 counts that is almost one order of magnitude compared with L1. The diffraction pattern appears diffuse, with weak peaks and a high background suggesting that the graphite crystal structure was destroyed. The catastrophic failure with a SOH immediately down to zero comes from structural decay, which makes the crystallinity be disordered and thus will prevent reversible lithium intercalation of the electrode.

The intensities discussed here are measured values, offset-corrected for the offsets used in the data that was plotted (+5000 for L1 and +2500 for L100). The above results highlight the close correlation between electrochemical degradation and structural instability. With SOH model combined with XRD measurement, better insight into the performance degradation behaviour of $LiFePO_4$ cells under harsh resistive operation could be obtained.

4. Conclusions

In this study, we explored the electrochemical performance and structural alterations of 18650-type $LiFePO_4$ cells under extreme resistive conditions, employing charge–discharge evaluations, SOH modelling, and XRD analysis. The charge-discharge tests at a 2.5 Ω load revealed a swift decline in capacity, dropping from about 368 mAh in the first cycle to 259 mAh after 100 cycles, which is considerably below the nominal capacity of 1800 mAh. A single discharge at 0.005 Ω caused immediate damage to the battery cell, highlighting the detrimental effects of extreme loading on the electrode's electrical properties. The SOH degradation pattern was successfully modelled using an exponential-offset method, achieving an RMSE of 2.87, MAE of 2.25, and R^2 = 0.90, surpassing the linear regression used in earlier studies. XRD analysis indicates that graphite remains the dominant phase but experiences a gradual reduction in crystallinity, as evidenced by a 66% decrease in peak intensity after 100 cycles at 2.5 ohms (L100) and nearly 90% after a single discharge at 0.005 Ω (LD). These structural modifications are directly linked to the electrochemical degradation observed during cycling. Overall, the findings illustrate that extreme resistive loading hastens both capacity reduction and structural deterioration, leading to catastrophic failure in a short timeframe. The integration of SOH modelling and XRD characterisation offers a comprehensive framework for understanding degradation mechanisms under severe conditions. Furthermore, the exponential-offset model shows significant promise for incorporation into battery management systems for real-time SOH prediction, while XRD validation provides a structural foundation for enhancing reliability and informing recycling-oriented strategies.

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6. Declaration of Assistive Technologies in The Writing Process

The authors declare that generative artificial intelligence (AI) tools were used to assist with language refinement and enhance readability. All scientific concepts, experimental designs, data analyses, and interpretations were fully developed and validated by the authors, who take full responsibility for the accuracy and integrity of the research.

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