REVIEW

Surveys on Physical Based Methods for State of Charge, State of Health and Fault Detection on a Li-Ion Battery

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Abstract

The quest for efficient and reliable energy storage systems has driven significant advancements in the field of physical-based methods for State-of-Charge and State-of-Health assessment of batteries. This paper presents a comprehensive overview of these methods, leveraging fundamental principles of physics and materials science to unveil the intricate dynamics within battery systems. Covering techniques like Coulomb counting, Open Circuit Voltage analysis, Peukert's equation, Electrochemical Impedance Spectroscopy, Gas Chromatography, and cutting-edge imaging approaches such as X-ray Diffraction and Magnetic Resonance Imaging, this review elucidates the principles, instrumentation, and applications of each method. Moreover, it delves into recent breakthroughs that enhance their accuracy and applicability. These physical-based methods not only empower battery management systems but also hold the key to advancing electric vehicles, renewable energy solutions, and a sustainable energy future.

Keywords State of charge · State of health · Battery · Physical-based method · White box method

Introduction

Battery energy storage systems play a vital role in various applications, including electric vehicles, renewable energy integration, and portable electronic devices. Accurate assessment of the State-of-Charge (SoC) and State-of-Health (SoH) of batteries is crucial for optimizing their performance, ensuring reliability, and extending their lifespan. Over the years, numerous assessment methods have been developed, ranging from empirical techniques to more advanced physics-based or white-box methods.

This literature review focuses on the SoC and SoH assessment of batteries using physics-based or white-box methods. These methods rely on fundamental electrochemical principles, mathematical models, and system dynamics to estimate the SoC and SoH of batteries. Unlike empirical methods,

physics-based approaches provide a deeper understanding of battery behavior, enabling more accurate and reliable assessment.

The review begins by discussing the importance of SoC and SoH assessment in Battery Management Systems (BMSs) and the challenges associated with traditional empirical techniques. It then delves into the principles and concepts of physics-based methods, emphasizing their ability to capture the underlying electrochemical processes and battery characteristics.

The review highlights the importance of integrating physics-based methods with other diagnostic techniques to achieve comprehensive battery health assessment. This integration allows for a holistic understanding of battery behavior, combining electrical, thermal, and electrochemical measurements to provide a more accurate assessment of SoC and SoH.

Overall, this literature review aims to provide a comprehensive overview of the current state of the art in SoC and SoH assessment using physics-based or white-box methods. Critically evaluating the principles, advantages, and limitations of these techniques will contribute to the existing knowledge and assist researchers, engineers, and practitioners in selecting appropriate methods for BMS and diagnostics. Ultimately, the adoption of physics-based approaches

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can enhance the reliability, safety, and performance of battery systems in various applications.

The paper is organized as follows: in Section"Overview of Physics-Based Methods", an overview of all the methods discussed in this paper is provided; electrical methods are presented in Section"Direct Electrical Methods"; analytical chemistry methods are described in Section"Chemical Analysis Methods"; spectroscopic methods are covered in Section"Spectroscopic Methods"; imaging and microscopy methods are examined in Section"Imaging and Microscopy Methods"; mechanical methods are outlined in Section "Mechanical Methods". The paper concludes with a summary in Section"Conclusion".

Overview of Physics-Based Methods

Accurate estimation of the SoC and SoH of batteries is essential for optimizing their performance, ensuring safe operation, and prolonging their lifespan. The SoC represents the amount of available energy in a battery, while the SoH reflects its remaining capacity or health over time.

A wide range of physics-based methods have been developed to estimate SoC and SoH in batteries. These methods leverage the physical characteristics and properties of the battery components to derive meaningful information about their state. They offer advantages such as direct measurement, non-destructiveness, and the ability to capture internal phenomena that affect the battery's performance.

This survey paper aims to provide a comprehensive overview of the state-of-the-art physics-based methods for SoC and SoH computation in batteries. It explores their common features, challenges, and potential applications, shedding light on their capabilities and limitations.

Inputs

The metrics used for SoC and SoH estimation in batteries vary depending on the specific technique employed. However, several common metrics are frequently utilized across different estimation methods. Here are some of the key metrics used for SoC and SoH estimation:

- **Voltage**: Battery voltage is one of the most fundamental metrics used for SoC estimation. It involves measuring the battery's terminal voltage and relating it to voltage characteristics at different SoC levels. Changes in voltage during charging or discharging can provide valuable information about the battery's state.
- **Current**: Battery current, measured during charge and discharge cycles, is often used in conjunction with voltage to estimate SoC. Integrating current over time (ampere-hour integration) provides an estimation of the total charge delivered or extracted from the battery.
- **Impedance**: Electrochemical Impedance Spectroscopy (EIS) and impedance-based techniques utilize the frequency-dependent behavior of the battery's impedance to extract information about its electrochemical processes. Impedance-based metrics, such as resistance, capacitance, and impedance magnitude or phase, are often used in SoC and SoH estimation methods.
- **Capacity**: Battery capacity is a key metric for SoH estimation. It represents the amount of charge a battery can store and deliver. Capacity fade analysis, which involves monitoring the degradation of battery capacity over time, is a common approach for estimating SoH.
- **Temperature**: Temperature is an important metric that affects battery performance and degradation. Temperature-based methods utilize temperature measurements during battery operation to estimate SoC and SoH. Fluctuations in temperature can indicate the internal state of the battery and its thermal behavior.
- **Electrochemical Parameters**: Some estimation methods rely on electrochemical parameters, such as diffusion coefficients, exchange current density, or concentration profiles, to infer SoC and SoH. These parameters are typically obtained through modeling or experimental calibration.
- **Aging Indicators**: SoH estimation often involves tracking specific indicators of battery aging, such as capacity fade rate, impedance growth, or changes in voltage behavior. These indicators provide insights into the degradation mechanisms and remaining battery lifespan.
- Other factors may also be considered.

It's important to note that different estimation methods may rely on a combination of these metrics, and the specific algorithms and models used for estimation can vary. Additionally, advanced techniques may incorporate machine learning or data-driven approaches to enhance the accuracy and reliability of SoC and SoH estimation.

Validation

Validating SoC and SoH estimation methods for batteries is crucial to ensure their accuracy and reliability. The validation process involves comparing the estimated values obtained from the estimation method with reference values or ground truth data. Here are some common approaches and techniques used to validate SoC and SoH estimation methods:

- **Reference Techniques**: Comparing the results of the estimation method with those obtained from reference techniques is a common validation approach. Reference

techniques are well-established, highly accurate methods for measuring SoC and SoH, such as coulomb counting, laboratory-grade testing, or destructive testing. By comparing the estimated values with the reference values, the accuracy of the estimation method is assessed.

- **Experimental Validation**: Conducting controlled experiments is another approach to validate SoC and SoH estimation methods. This process involves subjecting batteries to controlled charging and discharging cycles while simultaneously monitoring and recording the actual SoC and SoH using reference techniques. The estimated values are then compared against the measured reference values to evaluate their accuracy.
- **Field Testing**: Deploying the estimation method in real-world applications and comparing the estimated values with field measurements is another form of validation. Field testing involves using the estimation method in actual BMSs, such as electric vehicles or energy storage systems, and collecting data over an extended period. The estimated SoC and SoH values are validated against field measurements obtained from monitoring systems or periodic testing.
- **Long-Term Evaluation**: Battery aging is a long-term process, and validating SoH estimation methods over extended periods is important. Long-term evaluation involves monitoring batteries over their entire lifespan and periodically assessing the estimated SoH values against actual degradation measurements. This helps assess the method's ability to accurately track battery aging and predict its remaining useful life.

Statistical analysis techniques are often used to evaluate the performance of SoC and SoH estimation methods. This involves analyzing the differences between estimated values and reference values using metrics such as mean absolute error, root mean square error, or correlation coefficients. Statistical analysis helps quantify the accuracy and precision of the estimation method and provides insights into its performance.

Battery Management System

The BMS plays a crucial role in ensuring the safe operation and effective power management of batteries. In addition to its primary functions, advanced BMSs offer valuable insights into the SoC and SoH of the Lithium-Ion battery (LIB) (see Fig. 1). However, accurately estimating SoC and SoH from limited input and output measurements (voltage, current, and surface temperature) poses a challenge due to the intricate electrochemical side reactions within the battery.

Internal factors, including charge/discharge rates, operating temperature, internal aging, abnormal charging-dischar-
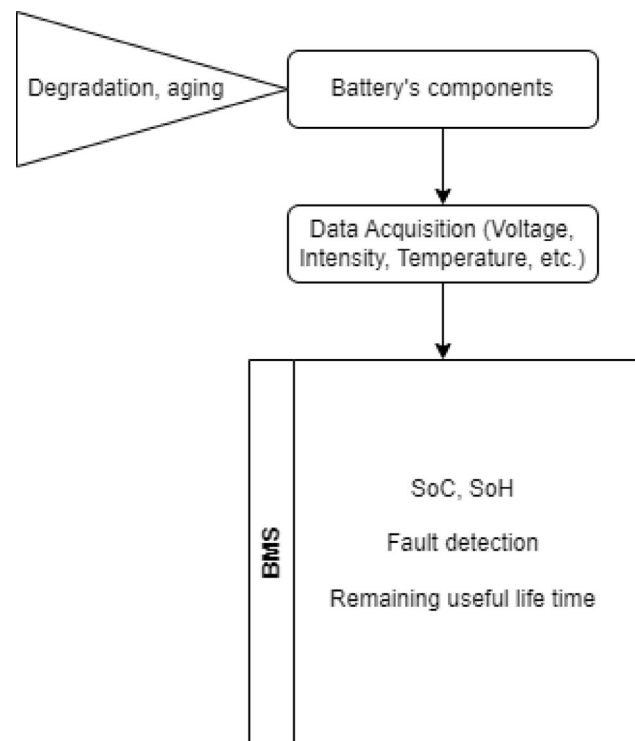

Fig. 1 Battery Management System

ging cycles, and internal faults, can significantly impact the health of the LIB. Developing intelligent and resilient BMSs capable of health-conscious decision-making requires a comprehensive understanding of both internal and external degradation mechanisms. This paper provides a thorough review of these degradation mechanisms, focusing on both the anode and cathode of LIBs. Mathematical models and correlations with SoH metrics such as capacity and power fade are discussed, emphasizing the integration of various electrochemical models with internal degradation mechanisms.

Considering these degradation mechanisms, various methods have been developed to detect specific types of aging. Each of these aging mechanisms is influenced by factors such as time, temperature, voltage, current load, cell design, pressure, and mechanical stress. Among the degradation mechanisms, the presented methods are useful for detecting the following:

- **Anode Aging**: solid-electrolyte interphase (SEI) growth or decomposition, plating/dendrite formation, electrode particle cracking, graphite exfoliation, and structural disordering.
- **Cathode Aging**: Cathode electrolyte interface formation, material phase transition, deposition on the anode, migration of dissolved products, and electrolyte decomposition.

- **Other Factors**: Binder decomposition, corrosion of the current collector, loss of electrical contact, and decrease in porosity.

White-Box Models

Researchers are exploring more sophisticated and detailed white-box models that consider additional factors such as temperature, aging mechanisms, and operating conditions. These models aim to improve accuracy and capture the complex electrochemical behavior of batteries more comprehensively.

Future research may focus on developing multiscale white-box models that integrate information from different length and time scales [1, 2]. These models could bridge the gap between microscopic battery electrode processes and macroscopic system behavior, providing a more accurate representation of the battery's SoC and SoH.

White-box models can be extended to incorporate multiphysics phenomena, such as mechanical stresses, thermal effects, and electrochemical–mechanical coupling [3, 4]. This holistic approach enables a more comprehensive analysis of battery performance and degradation, leading to improved SoC and SoH assessment.

To mitigate external factors, studies could focus on developing application-specific white-box models tailored to particular industries or sectors. These models can account for specific operating conditions, load profiles, and environmental factors relevant to applications like electric vehicles, renewable energy storage, or portable electronics.

As new battery chemistries and technologies emerge, there is a need to develop white-box models specifically tailored for these systems. Future research may focus on expanding the applicability of white-box models to include emerging battery chemistries, such as solid-state batteries, lithium-sulfur batteries, and beyond. Additionally, research may emphasize the development of standardized benchmarks and validation protocols for white-box models in SoC and SoH assessment. Standardization ensures comparability and facilitates the adoption of these models in various applications and industries.

White-box models can benefit from the integration of sensor technologies, such as advanced monitoring systems and embedded sensors, to enhance model accuracy and enable real-time estimation of SoC and SoH. Combining data from sensors with white-box models can provide a comprehensive and reliable assessment of battery performance. Furthermore, advanced sensor technologies have been discussed in previous sections in the context of specific methodologies.

Efforts are being made to enhance parameter estimation techniques for white-box models. This includes the development of advanced optimization algorithms, data-driven approaches, and machine learning techniques to determine model parameters accurately, even in the presence of uncertainty or limited data [5, 6]. There is growing interest in leveraging artificial intelligence techniques, such as machine learning and deep learning, to enhance white-box models. AI-driven approaches can assist in data-driven modeling, pattern recognition, and predictive analytics, enabling more accurate and adaptive estimation of SoC and SoH.

A grey-box model refers to a modeling approach that combines elements of both white-box (physics-based) and black-box (empirical or data-driven) modeling techniques. Grey-box models aim to strike a balance between the accuracy of white-box models, which are based on a deep understanding of the underlying physical processes, and the flexibility of black-box models, which are built solely from observed data.

A grey-box model typically incorporates some knowledge of the battery's internal physics and chemistry, such as its electrochemical behavior, thermal characteristics, or aging processes [7, 8]. However, it may also use empirical data or machine learning techniques to account for complex or poorly understood phenomena. Future research will focus on further exploring and improving these grey-box models.

Methods Overview

This paper presented the following methods with all their references:

- Direct Electrical Methods (Section "Direct Electrical Methods")
  1. Coulomb Counting, Ampere-Hour Integration (Subsection "Coulomb Counting"): [9–15]
  2. Open Circuit Voltage (Subsection "Open Circuit Voltage"): [16–24]
  3. Peukert's Equation (Subsection "Peukert's Equation"): [25–28]
  4. Electrochemical Impedance Spectroscopy (Subsection "Electrochemical Impedance Spectroscopy"): [29–40]

- Chemical Analysis Methods (Section "Chemical Analysis Methods")
  1. Gas Evolution Analysis (Subsection "Gas Evolution Analysis"): [41–44]
  2. Electrochemical Noise Analysis (Subsection "Electrochemical Noise Analysis"): [45, 46]
  3. Gas Chromatography (Subsection "Gas Chromatography"): [42, 47–49]
  4. Secondary Ion Mass Spectrometry (Subsection "Secondary Ion Mass Spectrometry"): [50–54]
  5. Electrochemical Mass Spectrometry (Subsection "Electrochimical Mass Spectrometry"): [42, 55–57]

- Spectroscopic Methods (Section "Spectroscopic Methods")

  1. Raman Spectroscopy (Subsection "Raman Spectroscopy"): [58–64]
  2. Laser-Induced Breakdown Spectroscopy (Subsection "Laser-Induced Breakdown Spectroscopy"):[65–68]
  3. Positron Annihilation Spectroscopy (Subsection "Positron Annihilation Spectroscopy"): [69–75]
  4. X-ray Diffraction Spectroscopy (Subsection "X-ray Diffraction Spectroscopy"): [76–82]
  5. X-ray Absorption Spectroscopy (Subsection "5.5"): [80–87]
  6. X-ray Photoelectron Spectroscopy (Subsection "X-ray Photoelectron Spectroscopy"): [51, 81, 82, 88–90]

- Imaging and Microscopy Methods (Section "Imaging and Microscopy Methods")

  1. Neutron Imaging (Subsection "Neutron Imaging"): [91, 91–95]
  2. X-ray Microscopy Imaging (Subsection "X-ray Microscopy Imaging"): [81, 82, 96–98]
  3. X-ray Computed Tomography (Subsection "X-ray Computed Tomography"): [81, 82, 99–108]
  4. Magnetic Resonance Imaging (Subsection "Magnetic Resonance Imaging"): [99, 109–123]
  5. Atomic Force Microscopy (Subsection "Atomic Force Microscopy"): [124–128]
  6. Scanning Electron Microscopy (Subsection "Scanning Electron Microscopy Imaging"): [99, 129–147]

- Mechanical Methods (Section "Mechanical Methods")

  1. Mechanical Stress (Subsection "Mechanical stress"): [148–150]
  2. Ultrasonic Testing (Subsection "Ultrasonic Testing"): [99, 151–158]

Direct Electrical Methods

Electrical methods play a fundamental role in the assessment of battery health and performance. These methods, including Coulomb Counting, Open Circuit Voltage (OCV), Peukert's Equation, and EIS, offer valuable insights into the SoC and SoC of batteries. By analyzing electrical characteristics and parameters, these techniques provide crucial information about a battery's capacity, efficiency, and overall condition.

Together, these electrical methods form the foundation of battery assessment, aiding in the development of accurate models and management strategies for batteries used in various applications, from consumer electronics to electric vehicles and renewable energy systems. Understanding these methods is essential for optimizing battery performance, prolonging battery lifespan, and ensuring safe and efficient operation.

Coulomb Counting

Method

Coulomb counting is a widely used method for estimating battery charge and health. It calculates the SoC or SoH by integrating the current flowing in or out of the battery over time, considering the fundamental principle that one Coulomb of charge is equivalent to one Ampere-second of current. While this method is simple and effective, its accuracy can be affected by factors such as battery aging and temperature variations. Movassagh et al. [9] provide an enhanced Coulomb counting method that addresses measurement error, integration error, capacity uncertainty, and oscillator error.

To improve the Coulomb counting method, it is useful to examine the Ampere-Hour Integration method [10]. First, it is necessary to distinguish this method from Coulomb Counting. In simple terms, the Coulomb Counting method is actually a special case of the Ampere-Hour Integration method. The Coulomb method calculates charge by assuming a current intensity of one ampere for a second.

Since the Coulomb unit is too small, leading to large numerical values for battery capacity, the Ampere-hour (Ah) unit is more suitable. The process used for measuring SoC and SoH is the same in both methods, with the only difference being the units of measurement.

In conclusion, Coulomb counting is a widely used method for estimating the SoC of LIBs. It is simple, cost-effective, and does not require model parameters. However, this method has limitations, particularly for SoH estimation, and its accuracy can degrade over time. Therefore, it is important to consider the use of complementary methods, such as data-driven approaches [11], alongside Coulomb counting to achieve more accurate and reliable SoC and SoH estimates.

Experimental Protocol

The materials used in a typical Coulomb counting experiment include the battery under investigation, a precision ammeter, a voltmeter, and a data acquisition system. The battery is connected to the ammeter, which measures the electric current flowing in or out of the battery during charge and discharge cycles. Simultaneously, the voltmeter monitors the voltage across the battery terminals, providing crucial information about the electrochemical processes occurring within.

To initiate the experiment, the battery is charged with a known current while recording the voltage and time data.

This charging phase is closely monitored until a predefined endpoint, such as reaching a specific voltage level or a designated elapsed time. Subsequently, the battery undergoes a controlled discharge, and the corresponding current, voltage, and time data are recorded again.

The acquired data is then analyzed using the Coulomb counting algorithm, which integrates the measured current over time. This integration yields the total electric charge that has flowed in or out of the battery during the experiment.

SoC Estimation

It relies on the principle that the SoC is directly proportional to the accumulated charge. The method involves measuring the current flowing into or out of the battery using a current sensor. The measured current is integrated over time to calculate the accumulated charge. The accumulated charge is then divided by the battery's total capacity to determine the SoC, as shown in Eq. 1.

$$SoC(t) = SoC_{ini} + \frac{nf}{Q} \int_{t_{ini}}^{t} I(t)\, dt \qquad (1)$$

This equation is widely used to estimate the SoC of a battery over time using the Coulomb counting method (also known as electric charge integration). Below is an explanation of the variables in the equation:

- SoC(t) represents the SoC at a specific moment t, denoting the battery's charge level at that point. It is expressed as a percentage, where 0% signifies complete discharge, and 100% signifies full charge.
- SoC_{ini} is the initial SoC of the battery at the start of the estimation process.
- nf (Farad conversion factor) adjusts the calculation based on the battery's charging or discharging behavior, improving accuracy by considering battery-specific characteristics.
- Q is the nominal battery capacity, expressed in Ah.
- $I(t)$ is the electric current flowing through the battery at a given moment t.
- t_{ini} is the initial moment when SoC estimation begins, serving as the starting point for current integration.

When the battery is charging ($I > 0$), its actual capacity may deviate from the nominal capacity. The nf factor accounts for this variation, often being greater than 1, indicating an effective capacity higher than the nominal capacity. When the battery is discharging ($I < 0$), the nf factor may be equal to 1 (if capacity remains unchanged) or differ from 1 to reflect a different effective capacity.

The Coulomb counting method remains unaffected by power fluctuations that cause battery voltage drops, ensuring that accuracy remains consistent regardless of battery usage. For a more detailed discussion on this method and recent enhancements, refer to [12].

Coulomb counting is typically combined with other SoC estimation methods to improve accuracy and mitigate limitations. By integrating Coulomb counting with voltage-based methods, EIS, or advanced modeling techniques, more precise SoC estimation can be achieved, leading to better BMS performance across various applications.

Temperature fluctuations affect SoC computation. To mitigate these errors, incorporating temperature compensation techniques is recommended. Regular calibration of the current sensor and adjustment of calibration factors can also enhance accuracy. Additionally, the use of advanced algorithms, such as state estimation filters (e.g., Kalman filters [13]), helps reduce measurement errors and improve SoC estimation accuracy.

SoH Estimation

Coulomb counting can also be used to estimate the SoH of a LIB [14]. This is done by comparing the actual battery capacity over time to the capacity predicted by Coulomb counting. The difference between these two values indicates capacity fade, which can serve as an estimate for the battery's SoH.

However, Coulomb counting has limitations in accurately estimating SoH since it does not account for other factors affecting battery health, such as impedance growth and voltage deviation. Additionally, the accuracy of Coulomb counting-based SoH estimation declines over time due to measurement errors and the need for periodic calibration [15].

Results and Deepening

The survey by Balasingam et al. [9] provides numerous references related to Coulomb counting equations and algorithms. Their work considers factors such as current measurement error, integration approximation error, battery capacity uncertainty, and timing oscillator error/drift. Recent findings from their research are also presented.

Open Circuit Voltage

Method

The battery's terminal voltage is measured when it is in an open-circuit state, i.e., no current is flowing into or out of it. A calibration curve or lookup table is established by relating the measured terminal voltage to the corresponding SoC val-

ues. The measured terminal voltage is then compared to the calibration curve or lookup table to estimate the SoC. The SoH can be inferred from SoC trends over multiple charge–discharge cycles.

Voltage-based methods are widely used for estimating the SoC and SoH of LIBS [16, 17]. These methods benefit from being based on a measurable physical parameter-battery voltage-and can be combined with other methods, such as Coulomb counting and capacity fading, to enhance accuracy. However, it is important to note that the accuracy of voltage-based methods is influenced by factors such as temperature and measurement errors [18].

By integrating the OCV method with Coulomb counting, Kalman filters, or advanced modeling techniques [19], a more precise SoC estimation can be achieved, enabling better BMS control for various applications.

Experimental Protocol

The materials for an OCV experiment typically include a test battery, a voltmeter with high input impedance (to minimize current flow and prevent measurement interference), and appropriate connecting wires. The experiment begins by disconnecting the battery from any external load or circuit, allowing it to stabilize in an idle state. This ensures that the measured voltage represents the inherent electrochemical potential of the battery without external influences.

The voltmeter is then connected across the battery terminals, and the voltage reading is recorded over a defined period. It is essential to allow sufficient time for the OCV to stabilize, as some batteries exhibit transient behavior before reaching a steady state. Extended monitoring is often performed to observe long-term voltage trends. In some cases, tests are conducted within a temperature-controlled chamber to assess the influence of temperature variations on battery SoH. Battery aging can be quantified by conducting these tests under simulated driving and monitoring conditions.

SoC Estimation

To estimate a battery's SoC using the OCV method, one must first measure the battery's voltage at rest, meaning when no charge or discharge is occurring. Subsequently, it is necessary to construct a calibration curve using empirical data or manufacturer-provided specifications. SoC estimation is then performed by comparing the battery's OCV with this empirical curve. A higher OCV corresponds to a higher SoC, and vice versa.

In summary, the OCV method relies on associating measured OCV values with an established SoC correlation curve.

While this method is straightforward to implement, calibration is often required to ensure accuracy.

To obtain an OCV curve with sufficient resolution, it is necessary to refine the measurement interval to 5% SoC or lower at the beginning and end of a charge or discharge cycle. Reducing the SoC interval below 5% increases experimental duration, making the process more time-consuming In the mid-SoC range, larger intervals are acceptable because OCV variations with SoC are less pronounced.

It is important to note that OCV decreases as temperature drops for the same SoC level, as demonstrated by Xing et al. [20]. To address this issue, they propose an improved battery model to minimize temperature-related errors. Their findings indicate that even minor voltage deviations in OCV estimation can lead to significant SoC errors under the same temperature conditions.

To mitigate these errors and improve SoC accuracy, model-based techniques such as the unscented Kalman filtering approach [21] can be implemented.

SoH Estimation

OCV can also be used to estimate battery SoH. One common approach is the voltage deviation method, which evaluates deviations of battery voltage from its nominal value as an indicator of SoH. This method is based on the assumption that battery voltage decreases as SoH deteriorates, with the degree of voltage deviation correlating to the extent of degradation.

Another voltage-based SoH estimation technique is the voltage-based capacity fading method [22], which combines OCV measurements with capacity fade analysis. This method leverages voltage deviations to correct accumulated errors in capacity fade estimation, yielding higher accuracy than capacity fade analysis alone.

Since temperature also impacts SoH, Schmalstieg et al. [23] propose a holistic model incorporating multiple variables. Their research demonstrates that cycling a battery around mid-range voltages minimizes aging effects. Their proposed model consists of three components: Impedance-based electrical model, Thermal model, Aging model. In this approach, voltage, temperature, SoH, and current data from the combined electro-thermal model serve as inputs for the aging model, enabling accurate aging predictions under different battery cycling conditions and BMS strategies.

Results and Deepening

Pillai et al. [24] provide an in-depth review of OCV models, parameter optimization techniques, and their integration with data-driven and error-approximation algorithms.

Peukert's Equation

Method

Peukert's Equation is a mathematical model used to estimate battery capacity and discharge characteristics. It describes the relationship between discharge current, time, and available capacity. The Peukert exponent (k) (see Eq. 2, where C is the available capacity, I is the discharge current, and t is the discharge time) depends on battery chemistry and discharge characteristics. It quantifies how battery capacity varies with different discharge rates.

Battery capacity, expressed in Ah, represents how much current a battery can supply over time. For example, if a 100Ah battery is discharged at a constant current of 5A, it will be fully depleted in 20 h. The discharge rate is typically expressed using the C-rate, which indicates the time remaining before depletion. Higher discharge rates result in lower available capacity. The relationship between slow and fast discharge rates is characterized by Peukert's law, with k indicating how battery capacity changes at different discharge currents.

Peukert's Equation is a simplified model that provides a rough estimation of battery capacity based on discharge characteristics. It is often used alongside other battery modeling techniques and system simulations to enhance capacity prediction accuracy [25]. However, the accuracy of Peukert's Equation varies depending on battery chemistry, age, temperature, and other factors affecting battery performance [26].

$$C = I^k * t$$
$$k = \frac{\log(t_2) - \log(t_1)}{\log(I_1) - \log(I_2)} \qquad (2)$$

Experimental Protocol

The primary materials required include the test battery, an ammeter to measure discharge current, a timer or data acquisition system, and a method for recording battery capacity. The experiment begins with a fully charged battery at a known SoC.

The battery is then discharged at a controlled constant current while measuring discharge time. Simultaneously, discharge current and voltage are monitored and recorded. Maintaining a constant discharge current is essential to accurately apply Peukert's equation. The collected data is analyzed using regression analysis or other curve-fitting techniques to determine the Peukert exponent (k) and constant values. Conducting tests in a temperature-controlled chamber significantly improves measurement reliability.

The use of specific resistive loads ensures uniform discharge conditions across all tested cells. This protocol simulates real-world discharge scenarios, including temperature variations and current fluctuations, to enhance relevance for electric vehicle applications.

SoC Estimation

To calculate the Peukert exponent, two nominal battery capacity values are required. Battery ratings can be found in the manufacturer's datasheet. However, Peukert's exponent provides only an approximate estimation of battery SoC. At very high discharge currents, the battery delivers even less capacity than predicted by a fixed Peukert exponent.

SoH Estimation

By comparing initial battery capacity and internal resistance at the beginning of life, Peukert's equation can be used to estimate SoH. To perform this estimation, the battery must first be tested to determine its actual capacity and internal resistance. Peukert's equation is then applied to this data to estimate the current battery capacity and SoH.

However, research [27] has demonstrated that a battery can still deliver additional capacity even after reaching full discharge if allowed to rest before further discharge. This means that significant capacity can still be extracted at lower discharge currents. Peukert's equation is strictly valid for batteries discharged under constant temperature and at a fixed current. When applied to batteries with variable discharge rates and fluctuating temperatures, Peukert's equation typically underestimates the remaining capacity, unless adjustments are made using an effective or average current.

Results and Deepening

Galushkin et al. [28] analyze variations in Peukert's equation and its applicability to LIB. Their research optimizes Peukert's model for multiple battery types, enhancing its predictive accuracy.

Electrochemical Impedance Spectroscopy

Method

EIS involves measuring the impedance response of a battery at different frequencies to extract valuable information about its electrochemical behavior [29]. The method applies a small-amplitude sinusoidal voltage signal across the battery terminals and measures the resulting current response. This process is repeated across a range of frequencies, typically spanning from low to high.

The response is measured in terms of complex impedance, consisting of both magnitude (amplitude) and phase information. The obtained impedance data is typically visualized using Nyquist or Bode plots [30, 31], which reveal the elec-

trical properties of the battery across different frequencies. Analyzing this impedance data provides insights into the battery's electrochemical processes and internal behavior.

Experimental Protocol

The materials required for an EIS experiment include: An electrochemical cell or battery under investigation (non-destructive method); A potentiostat/galvanostat to control potential or current; A reference electrode, counter electrode, and working electrode; A frequency response analyzer to apply small-amplitude perturbations across a frequency range.

The experiment begins with the assembly of the electrochemical cell, ensuring proper connections and electrode placement. The cell is then subjected to a small-amplitude sinusoidal perturbation, and the resulting current response is recorded. This process is repeated across multiple frequencies, often spanning several orders of magnitude.

The collected impedance data is represented as a Nyquist or Bode plot, where impedance maps are generated and analyzed using SIM software. The data is validated against the Kramers-Kronig law, ensuring consistency and accuracy.

To interpret the EIS results, an equivalent circuit model is established based on the impedance curve and electrochemical knowledge [32]. Parameter values are determined using nonlinear least squares fitting theory, allowing extraction of key electrochemical properties such as charge transfer resistance, double-layer capacitance, and other battery characteristics.

Experimental conditions, including electrolyte composition, temperature, and applied potential or current, can be systematically varied to investigate their impact on impedance behavior.

SoC Estimation

Ran et al. [32] propose a method for estimating SoC using EIS. They analyze the EIS response of LIB, which consists of an inductive arc at high frequencies and two capacitive arcs at lower frequencies.
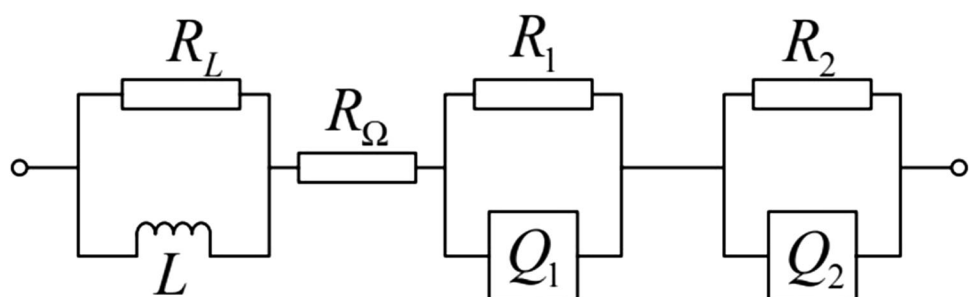
By combining equivalent circuit modeling and nonlinear least squares fitting, they extract circuit parameters that vary with SoC, establishing a correlation curve (see Fig. 2).

SoH Estimation

Li et al. [33] explain two main methods for estimating SoH by EIS: one is based on equivalent circuit model method [34, 35], the second one is based on deep learning method. However, both approaches fail to consider the effects of temperature and SoC variations during testing. To address this limitation, Wang et al. [36] propose a new model that incorporates these factors.

Building on this research, Zhang et al. [37] develop a probabilistic model for SoH estimation, incorporating charge transfer resistance, temperature, and SoC as key input variables. Their model achieves an estimation error of approximately 4% when accounting for both temperature and SoC. Under specific conditions (80% SoC at 30°C), the estimation error is further reduced to 1.29%.

Implementing this method requires expertise in experimental techniques, data analysis, and battery electrochemistry. Combining EIS with complementary techniques, such as voltage-based methods or impedance spectroscopy across different temperatures, can improve battery health assessments and performance predictions.

Results and Deepening

Vadhva et al. [38] provide a detailed explanation of the EIS experimental protocol and its variations. Liu et al. [39] offer an in-depth review of SoH estimation techniques, while Wang et al. [40] explore SoC estimation using EIS.

Chemical Analysis Methods

Electrochemical methods are at the forefront of battery analysis and diagnostics, providing crucial insights into the SoC and SoH of energy storage systems. This group of techniques, which includes EIS, Gas Evolution Analysis, and Electrochemical Noise Analysis (ENA), employs the principles of

Fig. 2 Equivalent circuit to carry through EIS simulation

electrochemistry to scrutinize the inner workings of batteries and cells.

Analytical chemistry methods have revolutionized our ability to comprehensively assess the SoC and SoH of batteries and energy storage systems. Among these methods, Gas Chromatography (GC), Secondary Ion Mass Spectrometry (SIMS) and Electrochimical Mass Spectrometry (MS) stand out as powerful tools in the field of battery diagnostics and analysis.

Collectively, these electrochemical methods offer a holistic view of a battery's condition, enabling researchers and engineers to optimize battery design, predict end-of-life scenarios, and ensure the safe and efficient operation of energy storage systems across various industries, from electric vehicles to renewable energy installations.

Gas Evolution Analysis

Method

Gas evolution analysis involves monitoring and analyzing gaseous emissions, primarily hydrogen (H_2) and oxygen (O_2), to assess battery condition and detect potential degradation [41]. During charging, discharging, or resting periods, gases emitted from the battery are collected and analyzed. This is typically performed using gas sensors or GC techniques, which detect and quantify the concentrations of different gases. For an overview of this method, refer to [42].

Calibrating gas sensors and establishing correlations between gas emissions and battery conditions are critical for accurate analysis. Calibration experiments using well-characterized battery samples under controlled operating conditions help establish accurate relationships between gas evolution and degradation mechanisms.

To ensure precise gas measurements and minimize interference, environmental factors such as temperature, humidity, and ventilation must be controlled. Standardizing test conditions and procedures further enhances the reliability and comparability of gas evolution analysis results.

Experimental Protocol

Materials used include the electrochemical cell or system under investigation, gas collection apparatus (e.g., gas burette or gas chromatograph), electrodes, a potentiostat/galvanostat to control the electrochemical processes, and a suitable electrolyte. The setup should allow for the safe and efficient collection of evolved gases.

The experimental setup must allow for the safe and efficient collection of evolved gases. Careful monitoring of gas volume and composition over time is essential. Tests typically vary experimental parameters such as applied potential, current density, and electrolyte composition to analyze their impact on gas evolution.

Collected gases are analyzed using GC to identify and quantify individual components. Calibration with known gas concentrations is necessary to ensure accurate mass spectrometry signal interpretation.

The study also considers aging-related gas emissions, accounting for pressure variations during experiments. Gas evolution is examined under different cycling conditions, including variations in C-rates and voltage limits. Open-circuit potential measurements follow each charge–discharge cycle.

SoH Estimation

By analyzing the type and quantity of emitted gases, battery SoH can be evaluated. Mattinen et al. [43] demonstrate that the most significant capacity loss occurs during high-rate cycling, specifically in 3C and 4C charge–discharge sequences.

Results and Deepening

Rowden et al. [42] and Narayan et al. [44] provide an in-depth examination of gas evolution processes and their relationship to battery faults.

Electrochemical Noise Analysis

Method

ENA is a technique used to study the electrochemical behavior and performance of batteries by analyzing small electrical fluctuations or noise signals that occur during battery operation. The recorded noise signals are analyzed using various techniques, including statistical analysis, power spectral density analysis, autocorrelation analysis, and wavelet analysis [45]. These analyses help extract information about underlying electrochemical processes, such as corrosion, charge transfer reactions, and diffusion mechanisms.

Experimental Protocol

Materials for an ENA experiment typically include the electrochemical cell or system under investigation, electrodes, a potentiostat/galvanostat for controlling the electrochemical conditions, and a suitable electrolyte. The setup should be designed to minimize external interference and noise. The experimental setup must be designed to minimize external interference and noise, ensuring accurate signal acquisition.

Electrochemical noise data is typically analyzed using statistical methods, power spectral density analysis, and time-domain or frequency-domain techniques. These analyses

provide insights into electrochemical processes, revealing information such as corrosion rates, localized corrosion events, and the effectiveness of corrosion protection mechanisms.

Experiments are often conducted under various conditions, including temperature variations, electrolyte composition changes, and the presence of inhibitors, to assess their impact on electrochemical behavior. To validate and quantify intrinsic noise, control voltage measurements are performed without the battery.

SoC Estimation

Martemianov et al. [45] present promising results regarding the application of ENA for in situ diagnostics of commercial LIB. The primary focus of this study is the methodology of ENA for monitoring commercial LIB. The researchers measure the standard deviation of noise signals across different SoC levels, demonstrating good reproducibility of results.

SoH Estimation

In the same study [45], preliminary results were obtained for battery SoH monitoring using ENA.

However, the accuracy of SoH estimation using this method remains limited. Specifically, the noise signal curve for an aged battery falls below that of a fresh battery between 95% and 60% SoC, but rises above it between 60% and 15% SoC.

The observed differences in aging effects at high and low SoC may be related to the dominance of different underlying mechanisms (electrochemical vs. transport phenomena). However, further research is required to confirm these findings and refine the method for improved SoH estimation.

Results and Deepening

Martemianov et al. [45] provide an in-depth analysis of this method and its application to LIBs. Xia et al. [46] detail the experimental setup and methodology.

Gas Chromatography

Method

GC is a technique used to separate and analyze gas components based on their differential interactions with a stationary phase (typically a coated column) and a mobile phase (an inert gas carrier).

The process begins by injecting the sample gas mixture into the GC system. As the sample passes through the chromatographic column, individual gas components separate based on their partitioning between the stationary and mobile phases. The separated components are then detected and quantified using various detectors, such as a flame ionization detector or a thermal conductivity detector.

The analysis focuses on identifying gases produced during battery operation, such as electrolyte decomposition products or side-reaction by-products. Liu et al. [47] provide a comprehensive review of the process and advanced techniques used in battery research.

Experimental Protocol

Materials for a GC experiment in battery analysis include the battery under investigation, a gas-tight sample collection system, a gas chromatograph instrument, a chromatographic column suitable for gas separation, a carrier gas, and a detector. The sample collection system must be designed to safely capture gases released during battery operation.

The chromatographic column facilitates gas separation, and the detector records signals corresponding to each gas component as it elutes from the column. Common detectors used in battery analysis include thermal conductivity detectors and flame ionization detectors. Additionally, mass spectrometry can be coupled with GC for a more detailed chemical analysis of individual gas components.

The resulting chromatogram provides a visual representation of the gas composition over time, enabling researchers to identify and quantify the gases released during different stages of battery operation. This information is crucial for understanding the mechanisms of gas evolution, detecting potential safety issues, and optimizing battery design for enhanced performance and safety.

SoH Estimation

GC can be coupled with other analytical techniques, such as mass spectrometry, to enhance the identification and quantification of gas components [48]. This combination provides additional insights into the chemical structure and molecular weight of separated gas species.

As batteries degrade or experience faults, they may release gases such as hydrogen (H_2) or carbon dioxide (CO_2) due to electrochemical reactions or physical damage. GC can be used to analyze the composition of these gases, offering valuable insights into battery health.

In some cases, GC is also used to analyze electrolyte composition, providing information about the presence of contaminants or electrolyte degradation over time.

Results and Deepening

Rowden et al. [42] present a detailed methodology for GC applications in battery research. Horsthemke et al. [49]

describe an in-situ experimental protocol specifically developed for lithium-ion cells.

Secondary Ion Mass Spectrometry

Method

SIMS is a technique used to analyze the elemental and isotopic composition of battery materials by bombarding the sample surface with a primary ion beam, typically from a liquid metal ion source [50]. The impact of the primary ions causes the ejection of secondary ions, which are then analyzed based on their mass-to-charge (m/z) ratio.

SIMS can be performed in imaging mode, where the ion beam scans the sample surface to create spatial maps of element distributions. Additionally, it can be used for depth profiling, where sequential sputtering reveals elemental composition as a function of depth.

This technique is particularly useful for studying electrode surfaces and solid materials within batteries, providing detailed information about the distribution of elements and their isotopic ratios. Depth profiling further enables researchers to track changes in material composition over the battery's lifecycle.

Experimental Protocol

A SIMS experiment requires battery components of interest, such as electrodes or electrolytes, a SIMS instrument, and a sample preparation system. The components are typically prepared as thin sections or surface samples to facilitate detailed elemental analysis.

The experiment begins with sample preparation, which may involve sectioning and polishing to ensure a flat and uniform surface. The sample is then loaded into the SIMS instrument, where it is bombarded with a primary ion beam, causing the release of secondary ions from the sample surface.

The mass spectrometer analyzes the ejected ions, sorting them by mass-to-charge (m/z) ratio to determine elemental composition. Because SIMS is highly sensitive, it provides high-resolution elemental and isotopic data.

In battery research, SIMS is applied to various studies, including the composition of electrode materials, ion migration within the electrolyte, and SEI formation on electrode surfaces.

SoH Estimation

Lee et al. [51] compare various techniques, including X-ray Photoelectron Spectroscopy (XPS), electron microscopy, and SIMS, for studying the SEI layer on graphite in full Li-ion battery cells. The study first employs static Time of Flight (TOF)-SIMS to assess the chemical homogeneity* of anode surfaces. Two batteries with different levels of degradation are analyzed:

1. A nearly unused cell
2. A heavily cycled cell that underwent 495 cycles (0%-50% SoC) and an additional 1317 cycles (50%-100% SoC) at 45°C

Results demonstrate the appearance of Mn+ and Na+ during battery cycling, likely due to cathode dissolution. Additionally, the presence of Na insertion may be indicative of cycle-induced contamination [52]. Surface scanning further confirms uniform Na+ contamination across cells.

Dynamic SIMS is then used to analyze SEI composition at different depths, allowing researchers to estimate SEI thickness and correlate it with capacity fade. The analysis reveals that $(C_2H_2)_nLi+$ species play a key role in SEI formation, likely originating from vinylene carbonate decomposition into lithiated polyacetylene, as suggested by Ota et al. [53]. To further validate SEI thickness variations, complementary XPS studies are recommended to provide additional chemical insights.

Results and Deepening

The review of Lombardo et al. [54] describes the SIMS best practices and limitations for various batteries while Zhou et al. [50] provide an in-depth explanation for LIBs.

Electrochimical Mass Spectrometry

Method

MS is an analytical technique used to identify and quantify the chemical composition of samples by measuring the m/z ratios of ions. In battery research, MS is employed to study gas emissions, electrolyte degradation, and material composition [55].

The sample is ionized using various techniques depending on its nature, including electron impact, electrospray ionization, or matrix-assisted laser desorption/ionization [56]. Once ionized, the generated ions are separated based on their m/z ratios using a mass analyzer, such as a quadrupole, TOF, or magnetic sector analyzer. The analyzer filters ions, allowing only those with specific m/z ratios to pass while eliminating others.

The separated ions are then detected by a Faraday cup or an electron multiplier, generating electrical signals proportional to ion abundance. This process provides quantitative insights into the chemical composition of battery components and helps track degradation mechanisms over time.

Experimental Protocol

Materials for a MS experiment in battery analysis include the battery components of interest, such as electrodes, electrolytes, and other relevant materials. Sample preparation typically involves techniques like liquid chromatography or GC to separate different species before mass analysis.

The experiment begins with introducing the prepared sample into the mass spectrometer. Depending on the specific instrument, different ionization techniques may be used, such as electrospray ionization or matrix-assisted laser desorption/ionization, to convert the sample into ionized species. Once ionized, the ions are accelerated and separated based on their m/z ratios within the mass analyzer.

The detector records ion abundance, generating a mass spectrum that provides information on the individual chemical components present in the sample. By interpreting the mass spectrum, researchers can identify the composition of battery materials and analyze chemical changes occurring during electrochemical processes.

Mass spectrometry is applied in battery research to investigate electrolyte degradation products, additive performance, and gas evolution mechanisms during battery operation. Isotope labeling techniques further enhance insights into reaction pathways and degradation mechanisms.

SoH Estimation

MS and SIMS are both essential analytical tools for assessing SoH in LIBs. These techniques provide critical insights into the chemical composition of battery components, aiding in the evaluation of battery performance and durability.

MS employs various ionization techniques, to generate ions from battery components. These techniques are suited for analyzing the electrolytes and solid battery materials. SIMS involves bombarding the surface of battery components with a primary ion beam, typically sourced from a liquid metal ion source. This process causes desorption and sputtering of secondary ions from the solid surface of the components.

Results and Deepening

The review of Rowden et al. [42] provides details about MS, the uses, limitations and advantages. Herl et al. [57] describes the protocol and experimental design of MS.

Spectroscopic Methods

Spectroscopic methods are instrumental in unraveling the intricate details of battery materials and understanding their SoC and SoH. Within this group, Raman Spectroscopy,

Laser-Induced Breakdown Spectroscopy (LIBS), Positron Annihilation Spectroscopy (PAS), X-ray Diffraction (XRD), X-ray Absorption Spectroscopy (XAS), and XPS emerge as indispensable tools for the comprehensive analysis of energy storage systems.

These spectroscopic methods explore the intricacies of battery materials at multiple levels, from the atomic and crystallographic scale to the molecular and chemical composition level. This wealth of information aids in battery design, optimization, and diagnostics for applications ranging from consumer electronics to renewable energy systems.

Raman Spectroscopy

Method

Raman Spectroscopy operates by directing a monochromatic laser beam onto the sample. Upon interaction with the material, most of the scattered light undergoes elastic (Rayleigh) scattering, maintaining the same energy as the incident laser. However, a small fraction of the light experiences inelastic (Raman) scattering, where its energy shifts due to molecular vibrations and rotations.

The Raman scattered light encodes valuable information about the molecular bonds and vibrational modes present in the sample. The resulting Raman spectrum is then analyzed and compared to reference databases to identify the molecular components and their structural characteristics [58].

Experimental Protocol

A Raman Spectroscopy experiment in battery analysis requires the battery components of interest, such as electrodes, electrolytes, and separator materials. Samples may take various forms, including powders, thin films, or even intact battery cells.

One of the advantages of Raman Spectroscopy is its non-destructive nature, requiring minimal sample preparation. The experiment begins by illuminating the sample with a laser beam, which interacts with the material, generating inelastic scattering and producing a Raman spectrum.

The collected Raman spectrum is analyzed to identify molecular species and gain insights into structural characteristics. This technique is particularly effective in studying carbonaceous materials, such as graphite electrodes, and detecting the formation of SEI on electrode surfaces.

SoH Estimation

According to the works of Lin et al. [59], Raman spectroscopy has given rise to the following methods: Spatially Offset Raman Spectroscopy [60], Tip-Enhanced Raman Spectroscopy [61], Surface-Enhanced Raman Spectroscopy

[62], XRD and Atomic Force Microscopy (AFM). Many of these methods have been discussed in other sections of this paper. Each technique provides crucial insights into battery component evolution, such as electrode and electrolyte behavior throughout charge–discharge cycles. Together, they significantly contribute to SoH monitoring and degradation analysis.

Results and Deepening

Flores et al. [63] propose an advanced in situ and operando Raman spectroscopy method, enhancing real-time monitoring of electrochemical reactions. Fredi et al. [64] investigate the graphitic microstructure of LIBs, demonstrating how Raman Spectroscopy enables detailed structural analysis.

Laser-Induced Breakdown Spectroscopy

Method

LIBS is a powerful technique used to analyze the elemental composition of battery materials. In this method, a high-energy laser pulse is focused onto the sample surface, causing rapid heating and vaporization of the material [65, 66]. This process generates a plasma plume composed of ionized atoms and ions.

As the excited atoms and ions return to their ground state, the plasma emits characteristic light (emission spectra). This emitted light is collected and analyzed using a spectrometer, which separates different wavelengths of light. By comparing the obtained spectral data with known elemental spectral signatures, researchers can identify and quantify specific elements within the battery sample.

Experimental Protocol

A LIBS experiment requires the battery components of interest, such as electrodes and electrolytes. Samples may be in various forms, including powders, thin films, or solid materials. Sample preparation ensures that the surface is representative and well-exposed for laser-induced plasma generation.

The experiment begins with applying a focused laser pulse onto the sample surface. This intense laser pulse ablates the material, creating a plasma plume that emits characteristic light. The emitted spectral lines correspond to the elemental composition of the sample and are collected and analyzed using a spectrometer.

The recorded LIBS spectrum provides real-time information about the presence and concentration of elements within the battery materials. LIBS is particularly advantageous for simultaneously detecting multiple elements with high preci-

sion. The technique consists of three primary components: laser excitation, light detection, and analytical processing.

SoC Estimation

Smyrek et al. [66] demonstrate that LIBS enables rapid elemental characterization of LIBs. A single laser pulse in ambient air is sufficient to assign a local SoC, offering a non-intrusive, qualitative, and quantitative approach to elemental analysis.

LIBS was used as a characterization method to determine the elemental composition of $Li(NiMnCo)O_2$ cathodes at various SoC levels, confirming its effectiveness for real-time state-of-charge estimation.

SoH Estimation

Researchers found that laser-calendered or laser-structured $Li(NiMnCo)O_2$ cathodes exhibited lower discharge capacities than purely calendered or structured cathodes.

The findings suggest that the LIBS method could be used to improve SoH assessment by analyzing cathode modifications and their impact on battery performance.

Results and Deepening

Pamu et al. [67] expose a calibration-free LIBS method, protocol and experimental design for LIBs. Smyrek et al. [68] describe the LIBS method for various electrodes.

Positron Annihilation Spectroscopy

Method

PAS is a technique that analyzes defects and electronic environments in materials by measuring the interaction between positrons (the antiparticles of electrons) and electrons. A positron source emits positrons into the material under investigation, where they eventually encounter electrons and undergo annihilation, producing gamma-ray emissions.

The characteristics of the annihilation process depend on the electronic environment and the presence of defects in the material. Doppler broadening analysis, a commonly used PAS technique, provides information about the momentum distribution of electrons, allowing researchers to identify vacancies and lattice defects [69].

Experimental Protocol

A PAS experiment requires battery components of interest, such as electrodes and electrolytes. Samples can be in various forms, including powders, thin films, or structured solid

materials. Sample preparation ensures a geometry compatible with positron annihilation measurements.

The experiment begins with positron injection into the sample. These positively charged antimatter particles penetrate the material and interact with electrons. Upon annihilation, gamma-ray photons are emitted, and their detection provides insights into defect structures and electron density [69].

PAS is particularly sensitive to vacancy-type defects, dislocations, and lattice irregularities. In battery research, it is used to study electrode material degradation, defect concentration changes during cycling, and the long-term structural stability of cathodes and anodes. While PAS is less common than other spectroscopic methods in battery studies, its unique sensitivity to defects makes it a valuable tool for analyzing the structural and electronic properties of battery materials.

SoC Estimation

According to Pagot et al. [69], PAS provides valuable insights into the lithiation state of $LiCoO_2$ thin films used in cathodes of LIBs.

Positron lifetime analysis offers a two-fold insight: Initial positron lifetimes reflect the cathode's charge state; Later positron interactions provide information about surface conditions at grain-graphite junctions.

These findings demonstrate the potential of PAS as a non-invasive technique for monitoring cathode lithiation in LIBs.

SoH Estimation

The study also found that discharge capacities for laser-calendered and structured $Li(NiMnCo)O_2$ cathodes were lower than those of purely calendered or structured cathodes.

A key aspect in understanding cathode microstructure evolution during operation is the influence of grain boundaries and internal grain properties. The presence of graphite in proximity to these structures appears to impact grain potential energy, which in turn affects the cathodic oxide's characteristics, including positron lifetime measurements.

Barbiallini et al. [70] use PAS to determine electron-positron correlations, predicting that this method can track lithium intercalation effects in oxide cathodes.

However, interpreting PAS lifetime data for materials like $LiFePO_4$ and $LiCoO_2$ remains challenging due to their complex microstructures [71, 72]. To address this, researchers suggest combining PAS with complementary techniques, such as soft and hard X-ray analysis, to gain a more comprehensive understanding of battery aging and degradation.

Another study [75] proposes using PAS to monitor lithiation and delithiation processes in $LiFePO_4$ cathodes, employing gradient correction approaches to improve measurement accuracy.

Results and Deepening

Pagot et al. [69] describes the PAS method adapted to LIBs. Many studies use PAS to various electrolyte systems but cannot be exposed individually in this section.

X-ray Diffraction Spectroscopy

Method

XRD is a widely used technique for analyzing the crystal structure and composition of materials, including battery electrodes and active materials. It provides valuable insights into the structural state and transformations occurring in battery components [76].

XRD works by exposing a sample to an X-ray beam and measuring the diffraction pattern produced by X-ray interactions with the crystal lattice. The resulting pattern reveals information about atomic arrangements, lattice spacing, crystallographic phases, and crystal size. By analyzing changes in diffraction patterns, researchers can track structural modifications in battery materials over time.

Experimental Protocol

Battery components such as electrodes and electrolytes are commonly analyzed using XRD. Samples are typically prepared as finely powdered materials, thin films, or other configurations suitable for X-ray analysis. The preparation process ensures uniformity and random crystallite orientation, which is critical for obtaining reliable diffraction data.

During the experiment, the prepared sample is exposed to an X-ray beam. The incident X-rays interact with the crystal lattice, producing constructive and destructive interference that forms a diffraction pattern. A detector captures this pattern, and the resulting data is analyzed to extract crystal structure, lattice parameters, and phase composition information.

XRD is commonly employed to identify the crystalline phases of electrode materials and monitor structural changes during charge–discharge cycles. It is particularly useful for studying the formation and degradation of SEI on electrode surfaces. Analytical methods such as Rietveld refinement [77] and pattern matching [78] are often used to identify crystalline phases and quantify their relative abundances.

SoC Estimation

According to Bartsch et al. [79], both ex situ and operando XRD techniques can be used to determine the SoC of semi-

conductor batteries. Using XRD data collected from a liquid electrolyte lithium-ion cell, researchers established a correlation between cathode lithiation/delithiation levels and SoC.

The study employed synchrotron-based operando transmission XRD and ex situ XRD to analyze cathode material transformations during charging and discharging cycles. As lithium content fluctuates within the cathode, its crystalline structure changes, affecting the battery's SoC.

XRD provides several advantages for SoC monitoring, as it is non-destructive and enables real-time observation of crystalline structural changes. In addition to SoC estimation, the same study concluded that XRD could also identify inactive electrochemical electrodes, making it a valuable tool for assessing the SoH of batteries. This application is particularly relevant for developing advanced cathode composites.

SoH Estimation

According to Balasubramanian et al. [80], XRD is a powerful method for evaluating the SoH of LIBs. The technique provides high-quality diffraction patterns, allowing for in-depth analysis of cathode materials and graphite reflections in the anode. The presence of relaxation processes in the cathode-which persist for several minutes after charging-was confirmed using XRD.

To obtain a comprehensive understanding of battery degradation, XRD should be complemented with other characterization techniques. Scanning Electron Microscopy Imaging (SEM), Transmission electron microscopy (TEM), and spectroscopic methods such as XPS can provide additional structural and chemical insights.

Results and Deepening

Li et al. [81] and Bak et al. [82] provide in-depth details on X-ray methods on LIBs.

X-ray Absorption Spectroscopy

Method

XAS is a powerful characterization technique used to investigate the chemical and electronic structure of battery materials. It provides critical insights into the local coordination environment and oxidation states of elements within battery components [83, 84].

XAS operates by exposing a sample to X-rays and measuring the absorption spectrum as a function of energy. It is typically conducted at synchrotron radiation facilities, which provide intense, tunable X-ray beams. The two primary types of XAS include X-ray Absorption Near-Edge Structure (XANES) [85] and Extended X-ray Absorption Fine Structure (EXAFS) [86], both of which offer detailed information about the electronic and structural environment of elements.

Successful XAS experiments require careful sample preparation, such as thin-film deposition, powder synthesis, or electrodeposition, to ensure compatibility with the experimental setup. Advanced data analysis techniques, including theoretical modeling, Fourier transform analysis, and simulation-based fitting, are crucial for extracting meaningful information from XAS spectra.

Experimental Protocol

Battery components such as electrodes, electrolytes, and separator materials are commonly analyzed using XAS. Samples can be prepared in various forms, including powders, thin films, or other geometries compatible with X-ray absorption measurements. Proper sample preparation ensures accuracy and reproducibility in spectral analysis.

The experiment begins by exposing the sample to monochromatic X-rays, which are tuned to the absorption edge of a specific element of interest. When the X-rays interact with the material, they excite inner-shell electrons, leading to element-specific absorption features in the recorded spectrum. The intensity of transmitted X-rays is measured as a function of energy, producing a spectrum that reveals details about the local atomic environment of the probed element.

XAS is widely used to study oxidation states in transition metal electrode materials, monitor electronic structure changes during charge–discharge cycles, and investigate elemental behavior in electrolytes and SEI. Its ability to analyze local coordination environments in complex materials makes it particularly valuable for understanding battery degradation mechanisms.

Data analysis in XAS involves comparing experimental spectra with reference spectra and applying theoretical models to extract quantitative structural information. EXAFS analysis [87] is particularly useful for determining bond distances, coordination numbers, and structural disorder within battery materials.

SoH Estimation

The XAS method plays a significant role in assessing the SoH of LIBs by tracking changes in the chemical structure of electrodes and battery materials over time. It is particularly effective in detecting variations in the oxidation states of elements in electrode materials.

As batteries undergo repeated charge and discharge cycles, chemical transformations occur in the active materials. XAS enables researchers to identify and quantify these changes, providing crucial insights into battery degradation and aging mechanisms [80]. However, it is important to note that XAS is primarily used for ex-situ analyses, meaning that

it is often conducted on battery samples removed from the operational system rather than in real-time.

Despite this limitation, XAS remains a highly effective tool for diagnosing long-term material stability, helping to optimize battery performance and extend cycle life.

Results and Deepening

Li et al. [81] and Bak et al. [82] provide in-depth details on X-ray methods on LIBs.

X-ray Photoelectron Spectroscopy

Method

XPS, also known as Electron Spectroscopy for Chemical Analysis (ESCA), is a surface-sensitive technique used to investigate the elemental composition, chemical states, and electronic properties of materials, including battery components [88].

The XPS process begins with X-ray irradiation of the sample, which causes the ejection of photoelectrons from its surface. These emitted photoelectrons are energy-analyzed using a spectrometer, allowing the determination of the binding energy of each element present in the sample. Additionally, chemical states are identified based on shifts in peak positions and spectral features, providing crucial insights into oxidation states, chemical bonding, and surface composition.

Experimental Protocol

Battery components such as electrodes, electrolytes, and separator materials are commonly analyzed using XPS. The sample is typically prepared as a thin film or other configurations suitable for surface analysis. Proper sample preparation is essential, often requiring sputtering or ion milling to remove surface contaminants and obtain a well-defined surface for analysis.

The experiment begins with irradiating the sample with X-rays, leading to photoelectron ejection from the surface material. The kinetic energy and intensity of the emitted electrons are measured, yielding detailed information about the elemental composition and chemical states of the material.

XPS is particularly effective for studying the oxidation states of elements in electrode materials, identifying surface contaminants, and tracking chemical changes at the electrode-electrolyte interface. It provides valuable insights into the formation and evolution of the SEI on electrode surfaces.

Data analysis in XPS involves deconvoluting spectral peaks corresponding to different elements and chemical states. The binding energies of photoelectrons offer precise information about chemical environments and oxidation state variations, making XPS a powerful tool for monitoring redox reactions in battery materials.

SoH Estimation

Previous studies on the SEI layer in complete lithium-ion battery cells employed XPS, electron microscopy, and SIMS to analyze interfacial composition changes [51].

Comprehensive XPS studies are necessary to assess variations in the relative thickness of the SEI layer over time. It is important to note that XPS requires a longer spectral accumulation time compared to SIMS, meaning that analyses can only be performed once a certain electrode thickness is achieved.

In one study, XPS revealed concentration profiles of fluorine atoms within battery sample [51]. A major F1s peak at 686 eV confirmed that LiF is a primary component of the SEI, a finding consistent with earlier work by Zheng et al. [89]. While Mn was absent at the surface, XPS depth profiling revealed strong correlations between Mn distribution and the SEI structure prior to graphite peak emergence.

Another study by Andersson et al. [90] demonstrated that the chemical composition of the SEI layer correlates with the high-temperature performance of Li/graphite half-cells. This finding highlights the influence of temperature on the SEI layer, emphasizing its role in battery degradation and SoH assessment.

While XPS effectively tracks surface chemical changes over time, it does not provide direct information about a battery's overall SoH, such as capacity retention, residual lifespan, or energy delivery reliability. To fully evaluate battery degradation and longevity, electrochemical testing and performance-based analyses should be conducted alongside XPS studies.

Results and Deepening

Li et al. [81] and Bak et al. [82] provide in-depth details on X-ray methods on LIBs.

Imaging and Microscopy Methods

The field of battery SoC and SoH estimation has witnessed a remarkable evolution with the integration of advanced imaging and microscopy techniques. These cutting-edge methods provide unique insights into the internal structures and processes of batteries, enabling a deeper understanding of their performance and degradation mechanisms. Imaging and microscopy methods, including Neutron Imaging, X-ray Microscopy Imaging (XRM), Magnetic Resonance Imaging (MRI) and AFM are detailed. These techniques offer a window into the intricate world of battery materials and offer the

potential for enhanced accuracy in assessing and managing the SoC and SoH of energy storage systems.

Neutron Imaging

Method

Neutron imaging is a non-destructive technique that utilizes neutrons to probe the internal structure and composition of materials, including battery components [91, 92]. This method provides valuable insights into element distribution, moisture content, and structural changes within batteries.

The process involves interactions between neutrons and the sample, followed by detection and visualization of transmitted or scattered neutrons. Two primary techniques are employed:

- **Neutron Radiography**: This method captures the attenuation of neutrons as they pass through the battery sample. The intensity of the transmitted neutrons is recorded, forming a 2D image that reveals internal structures, density variations, and element distribution.
- **Neutron Tomography**: This technique combines multiple radiographic projections from different angles to reconstruct a 3D image of the battery sample. By acquiring a series of 2D images at different rotation angles, researchers can visualize the internal structure and spatial distribution of materials in three dimensions.

For successful neutron imaging, sample containers and holders must be carefully designed to minimize neutron attenuation and ensure proper positioning. Additionally, neutron imaging should be combined with other characterization techniques, such as XRM, electron microscopy, or spectroscopy, to obtain a more comprehensive understanding of battery materials and their behavior. Integrating multiple techniques enhances the accuracy of battery assessments.

Experimental Protocol

A neutron imaging experiment in battery analysis requires a fully assembled battery cell, including electrodes, electrolytes, separators, and other relevant components. No extensive sample preparation is needed, as neutron imaging is a non-destructive technique that allows intact battery cells to be examined directly.

The experiment involves exposing the battery cell to a beam of neutrons. Since neutrons interact differently with various elements, the resulting contrast in the image provides insights into internal battery structures. This method is particularly sensitive to light elements like hydrogen, making it suitable for studying materials that are difficult to image with traditional techniques.

Neutron imaging is highly effective in analyzing electrolyte distribution within electrodes, tracking gas pocket evolution, and observing electrode morphology changes during charge–discharge cycles. It is particularly valuable for non-destructively investigating electrolyte filling, electrode swelling, and structural modifications under different operational conditions.

The acquired neutron images are analyzed to extract data on spatial distribution, density, and material composition within the battery. Advanced data processing techniques allow for 3D reconstructions, providing a detailed visualization of internal battery structures.

SoC Estimation

According to Senyshyn et al. [93], neutron imaging has been used to analyze the crystalline structure evolution of LIBs based on their SoC and SoH. Using Rietveld's refinement technique, researchers examined neutron radiography data and tomographic reconstructions, revealing the local neutron absorption density-a parameter that primarily reflects lithium distribution and battery design details.

Through this study, neutron imaging has been successfully applied to evaluate neutron absorption contrast as a function of SoC and SoH, providing a non-invasive approach to monitoring battery charge levels.

SoH Estimation

In another study, Song et al. [94] used neutron radiography to monitor lithium dendrite growth in real-time. The research demonstrated that short circuits in LIBs are primarily caused by lithium dendrite growth.

By employing neutron imaging, researchers visualized dendrite formation and tracked its progression over time, allowing for a detailed analysis of this aging mechanism. This study confirmed that neutron imaging is a powerful tool for identifying lithium accumulation and dendritic growth, which are major contributors to battery degradation and failure.

Results and Deepening

Kardjilov et al. [91] discuss the latest advancements in neutron imaging, highlighting its capabilities in battery diagnostics and beyond. Gao et al. [95] explore various applications of neutron imaging for lithium-ion battery analysis, demonstrating its potential in assessing internal battery structures and degradation mechanisms.

X-ray Microscopy Imaging

Method

XRM is an imaging technique that utilizes X-rays to produce high-resolution 2D or 3D images of the internal structure of materials, including batteries. It is particularly useful for observing the distribution of phases, pores, cracks, and other structural features inside a battery, providing critical insights into morphology and spatial distribution of battery components.

Two commonly used techniques in XRM are Transmission X-ray microscopy (TXM) [96] and Scanning transmission X-ray microscopy (STXM) [97], both of which enable nanoscale analysis of battery structures and compositions.

In TXM, a broad X-ray beam is transmitted through a thin sample, and differences in X-ray absorption based on electron density and chemical composition are recorded. A detector captures the transmitted X-rays, allowing for the reconstruction of 2D or 3D images that reveal internal structures at the nanoscale.

In STXM, a highly focused X-ray beam scans a small region of the sample with sub-micrometer spatial resolution. The intensity of transmitted X-rays at each scanned point is measured, generating detailed images with extremely high spatial resolution. This technique also enables chemical mapping by analyzing X-ray absorption at different locations within the sample.

The primary difference between TXM and STXM lies in how X-rays interact with the sample. TXM uses a broad beam to capture the entire sample, whereas STXM scans small regions with a focused beam. Both techniques are essential for analyzing battery materials, as they provide detailed insights into lithium distribution (SoC) and electrode degradation (SoH).

Experimental Protocol

A XRM experiment for battery analysis requires a fully assembled battery cell, including electrodes, electrolytes, separators, and other internal components. Since XRM is a non-destructive imaging technique, no extensive sample preparation is required.

The experiment involves exposing the battery cell to X-rays with short wavelengths. These high-energy X-rays penetrate the sample, and interactions such as absorption and phase contrast are detected to generate highly detailed images of the internal structure. The technique provides micrometer-to-nanometer spatial resolution, making it particularly useful for examining fine features within battery components.

XRM can be applied to observe electrode microstructures, track active material distribution, and analyze electrode-electrolyte interfaces. It is especially useful for monitoring morphological changes during charge–discharge cycles, as well as studying dendrite growth and electrolyte penetration.

The recorded X-ray microscopy images undergo advanced processing and analysis to extract quantitative data on internal structural features. Tomography techniques allow for full 3D reconstructions, offering a comprehensive visualization of battery architecture.

SoC Estimation

Using TXM, researchers can examine cathodes and anodes in LIBs to observe how lithium species distribution evolves during charging and discharging [98]. Cathode materials, for instance, typically contain lithium when charged and release it upon discharge. By analyzing electron density changes in cathode materials throughout charge–discharge cycles, SoC can be inferred.

STXM provides even higher spatial resolution, allowing for precise tracking of lithium at the nanoscale within electrodes. This capability is crucial for analyzing localized lithium concentration variations, making it a powerful tool for high-precision SoC estimation.

SoH Estimation

TXM is widely used for monitoring electrode integrity and internal degradation phenomena [98]. Researchers can assess the formation of deposits (such as lithium dendrites), material degradation, and microstructural changes that impact battery performance.

With its exceptional spatial resolution, STXM enables the detection of subtle chemical and structural changes in battery materials, such as variations in crystalline structure or chemical composition at the nanoscale. By tracking these early degradation indicators, STXM provides a highly accurate assessment of a battery's SoH before significant capacity loss occurs.

Results and Deepening

Li et al. [81] and Bak et al. [82] provide in-depth details on X-ray methods on LIBs.

X-ray Computed Tomography

Method

X-ray Computed Tomography (X-ray CT) is a 3D imaging technique widely used for industrial inspection and battery analysis. It allows researchers to reconstruct the internal structure of a sample non-destructively by scanning X-ray signals at various rotation angles [99].

Using the inverse Radon transform, 2D slice images of the scanned material are reconstructed, which can then be stacked to generate a complete 3D representation of the sample [100].

The limitations of conventional X-ray CT can be reduced by using synchrotron X-rays, which are highly intense and produce stronger transmitted signals. This enhancement increases imaging speed and allows for advanced modeling approaches, such as 4D (3D + time) and 5D (3D + time + energy) imaging. For example, research on lithium-sulfur batteries has employed 4D imaging to track cathode microstructure evolution [101].

As Deng et al. [99] highlight, X-ray CT is not limited to characterizing battery microstructure and morphology. The volumetric data obtained from imaging can also be used for real 3D computational modeling of LIBs. For example, Bronte et al. [102] developed a 5D computational model by integrating energy and time components into a 3D X-ray CT dataset. Another 5D imaging method (3D + time + diffraction) has been applied in X-ray diffraction tomography, enabling non-destructive crystallographic mapping of battery electrode materials [103].

Experimental Protocol

A typical X-ray CT experiment for battery analysis requires an assembled battery cell, which includes electrodes, electrolytes, separators, and additional relevant components. Since X-ray CT is non-destructive, no special sample preparation is required for the imaging process.

The experiment begins by placing the battery cell inside the X-ray CT system. X-rays are transmitted through the sample from multiple angles, and detectors measure the intensity of the transmitted X-rays at each angle. The collected data is then processed using computational algorithms to reconstruct a detailed 3D image of the internal battery structure.

X-ray CT is particularly valuable for examining the spatial distribution of electrode materials, identifying porosity and voids, and analyzing electrode-electrolyte interfaces. It also provides insights into how cycling affects the internal structure of a battery over time, making it an essential tool for understanding structural degradation mechanisms.

The reconstructed 3D images allow for advanced quantitative analysis, including electrode porosity measurements, active material distribution, and long-term structural evolution of battery components.

SoC Estimation

Yu et al. [104] combined spectro-tomography and soft X-ray ptychographic imaging to determine the 3D morphology and oxidation states of metal cations in lithium-iron phosphate (LiFePO4) cathode nanoparticles. By analyzing a set of LiFePO4 nanoplates extracted from a battery electrode at 50 percent SoC, the researchers investigated *the relationship between particle size and battery SoC.

This method for determining the SoC needs further research. The first reason is that the precise conditions that dictate how electrochemical transformations occur within individual particles of battery electrodes are still widely debated within the scientific community. The second reason is that the actual resolution is imprecise because Fourier Shell Correlation [105] reduces the signal-to-noise ratio of the data by a factor of two at all spatial frequencies. Lastly, the third reason could be that a portion of the electrode exhibits either a carbon deficiency or inadequate electrolyte wetting, which could lead to transport deficiencies that delay their reaction.

SoH Estimation

In the context of LIBs, 3D imaging enables researchers to investigate degradation mechanisms by mapping material morphology and composition across different size scales-from micrometers to nanometers.

One crucial factor in battery degradation is critical current density, which influences short-circuit behavior in solid electrolytes. Shen et al. [106] examined the effect of critical current density on structural transformations in solid electrolytes. Their findings revealed that the likelihood of short-circuit formation is inversely proportional to the evolution of interconnected pores, suggesting that pore structure plays a key role in electrolyte failure.

Additionally, Dixit et al. [107] demonstrated that electrolytes with highly anisotropic tortuosity tend to have lower critical current densities, making them more prone to degradation.

Another major aspect of battery SoH degradation is electrode deterioration. Singer et al. [108] focused on understanding the nucleation of mobile dislocation networks in lithium-rich layered oxide nanoparticles. Their study suggested that using LLZO (lithium lanthanum zirconium oxide) as a cathode material in next-generation LIBs could enhance voltage management, making it a promising candidate for improving battery SoH and lifespan.

Results and Deepening

Li et al. [81] and Bak et al. [82] provide in-depth details on X-ray methods on LIBs.

Magnetic Resonance Imaging

Methods

MRI is a non-invasive imaging technique that utilizes strong magnetic fields and radio waves to generate detailed images

of internal structures and material composition. In battery research, MRI is used to analyze internal characteristics, monitor SoH, and investigate electrochemical phenomena within battery cells [109, 110].

The MRI process involves subjecting the battery to a strong static magnetic field while applying radiofrequency pulses, which stimulate atomic nuclei (such as hydrogen or lithium) within the battery materials. The nuclei then emit electromagnetic signals, which are detected and processed to construct high-resolution images. The choice of targeted nuclei depends on specific research objectives, enabling researchers to study different aspects of battery composition and behavior.

Future advancements in MRI aim to improve imaging resolution and speed, particularly through stronger magnetic fields. MRI is already a powerful tool for studying lithium-ion systems and electrochemical processes, and further development of UV/visible/IR imaging techniques-such as in-situ cell monitoring, laser scanning methods, and thermal imaging-continues to enhance battery diagnostics [111].

Additionally, advanced image reconstruction and post-processing algorithms are improving data accuracy by reducing artifacts and enhancing quantitative analysis. Combining MRI with complementary imaging techniques, such as X-ray imaging and electrochemical analysis, provides a more comprehensive assessment of battery health and performance.

Experimental Protocol

A typical MRI experiment in battery analysis requires a fully assembled battery cell, including electrodes, electrolytes, separators, and other key components. While MRI is non-destructive, specific sample preparation may be necessary to ensure compatibility with the MRI setup. For instance, the battery may need to be placed in a non-magnetic holder to prevent interference with the strong magnetic fields.

The experiment begins by exposing the battery to a strong external magnetic field, aligning the nuclear magnetic moments of specific atomic nuclei (e.g., lithium, hydrogen, or sodium). Radiofrequency pulses are then applied, causing the nuclei to absorb and reemit energy. These emitted signals are detected and processed using computer algorithms to construct detailed images of the battery's internal structure, revealing information about material composition, electrolyte distribution, and electrode behavior.

MRI is particularly useful for visualizing liquid-phase components, mapping electrolyte concentration gradients, and tracking material changes during charge–discharge cycles. The resulting images provide valuable spatial information, allowing researchers to study how different components interact within the battery system over time.

SoC Estimation

MRI produces a map of spatial nuclear spin density, obtaining a spatial image by reconstructing the acquired signals [112, 113]. Ilott et al. [114] use a 2D MRI model to determine SoC. They demonstrate a relevant correlation between a magnetic field and the SoC of a battery.

SoH Estimation

MRI is widely used to monitor internal battery processes, offering key advantages over other imaging techniques. Unlike traditional imaging methods, MRI resolution depends on the shift in additional magnetic field gradients, which allows it to detect real-time changes occurring within battery materials [115, 116].

The foundations of nuclear magnetic resonance imaging were laid by Lauterbur et al. [117], while Suits et al. [118] later demonstrated its applicability to solid materials. Subsequent studies expanded the use of MRI for battery diagnostics, enabling detailed investigations into electrode behavior and electrolyte dynamics.

Several studies have identified specific nuclear isotopes (e.g., ^{23}Na, ^{7}Li, ^{1}H, ^{19}F) that can be detected via MRI, offering insight into chemical composition changes and degradation mechanisms [119, 120].

A notable application of MRI in SoH monitoring is its ability to track dendrite and lithium foam formation on battery electrodes. Bhattacharyya et al. [121] conducted in-situ lithium nuclear magnetic resonance spectroscopy experiments, successfully monitoring dendrite and foam growth on negative electrodes during battery cycling.

Dendrites form at high current densities and develop into sharp metallic protrusions, which can puncture the separator, leading to short circuits and thermal runaway. Lithium foam, on the other hand, accumulates at lower current densities, forming a dense layer of deposited lithium that gradually increases over multiple charge–discharge cycles. Understanding these degradation processes is critical for improving battery safety and longevity [122].

In another study, Ilott et al. [123] used 3D MRI models to demonstrate that dendrite growth is not unidirectional but follows a twisted path until it reaches a short circuit. These findings highlight the importance of 3D imaging for real-time SoH assessment, allowing for more accurate predictions of battery lifespan and failure mechanisms.

Results and Deepening

Mohammadi et al. [111] expose MRI for various electrochemical cells. For more details on LIBs, refer to Deng et al. [99].

Atomic Force Microscopy

Methods

AFM is a high-resolution imaging technique that uses a sharp probe tip attached to a cantilever to analyze the surface properties of materials at the nanoscale. The probe interacts with the sample surface, experiencing attractive and repulsive forces that depend on surface morphology and composition. As the tip moves across the surface, these interactions are recorded, producing a detailed topographic map that provides insights into surface roughness, particle size, defects, and film morphology.

Improving AFM for enhanced estimation of SoH in LIBs remains an active area of research [124, 125]. Researchers are developing:

- Specialized AFM sensors and probes tailored for battery analysis, enabling higher spatial resolution.
- Integration with spectroscopic techniques like Raman or infrared spectroscopy to provide chemical composition insights.
- Nanoscale mechanical property measurements to track changes in electrode surfaces during cycling.
- Real-time AFM analysis for in situ and operando monitoring of electrode degradation.
- Machine learning and artificial intelligence algorithms for automated detection of surface changes and degradation patterns.
- Electrochemical stability improvements to ensure accurate readings during AFM measurements.

By advancing these techniques, AFM is establishing itself as a key tool for characterizing LIB health, helping develop more reliable and long-lasting energy storage technologies.

Experimental Protocol

A typical AFM experiment for battery analysis requires electrode materials, electrolyte films, or separator coatings. The sample can be prepared in various forms, including thin films, coatings, or polished surfaces, to ensure optimal imaging conditions. Surface preparation is critical and often involves techniques such as polishing, cleaving, or sputtering to create a clean and flat sample.

The experiment begins with placing the sample onto the AFM stage, followed by positioning the sharp probe tip near the surface without making direct contact. As the tip moves across the sample, interactions with the surface cause the cantilever to deflect, and these deflections are recorded to generate a topographic image.

AFM is particularly useful for studying electrode surfaces, monitoring the formation of the SEI, and tracking changes in material morphology at the nanoscale. The technique allows researchers to examine the impact of charge–discharge cycling on surface roughness, particle agglomeration, and the growth of unwanted surface films.

Additionally, AFM can be combined with other analytical techniques, such as Scanning Electrochemical Microscopy (SECM), to gain complementary insights into electrochemical processes [126].

SoH Estimation

In situ AFM imaging has been successfully combined with ex situ SEM to monitor battery degradation mechanisms [127]. Becker et al. [127] demonstrated that mechanical damage accumulates during cycling in batteries containing organic electrolytes, leading to irreversible surface modifications.

The effectiveness of AFM for studying SoH in lithium-ion batteries has been widely validated, with numerous studies confirming its reliability for analyzing electrode aging, mechanical wear, and surface evolution [124, 128].

Results and Deepening

Wang et al. [124] and Zhao et al. [125] provide in-depth AFM details for LIBs.

Scanning Electron Microscopy Imaging

Methods

SEM utilizes a focused electron beam, typically accelerated at high voltage, to interact with the sample surface. When the beam strikes the sample, multiple interactions occur, including elastic and inelastic scattering, secondary electron emission, and backscattering. Secondary electrons provide high-resolution surface topography, while backscattered electrons offer compositional contrast. This technique is widely used to analyze material morphology, composition, and interfacial structures, making it an essential tool for optimizing advanced battery systems.

In a review of imaging methods, Deng et al. [99] divide electron microscopy techniques into two categories: TEM and SEM. Both methods are crucial for studying the evolution of electrode materials and elucidating the energy storage mechanisms of LIB materials at the atomic level.

However, conventional LIB electrolytes, which are typically volatile liquids, complicate lithium-ion imaging using electron beams. A promising advancement in electron microscopy is Cryogenic Electron Microscopy (cryo-EM), a technique that operates at low temperatures and with low electron doses to prevent beam-induced damage.

Cryo-EM was developed by Jacques Dubochet, Joachim Frank, and Richard Henderson, who were awarded the Nobel

Prize in Chemistry in 2017. Since then, highly efficient cryo-EM systems have been developed [129, 130], enabling researchers [131] to study lithium dendrites and the formation of the SEI.

Moreover, cryo-EM can be integrated with complementary techniques such as Energy Dispersive X-ray Spectroscopy, Focused Ion Beam, and Electron Energy Loss Spectroscopy, providing a comprehensive understanding of lithium loss and SEI deterioration mechanisms [132–136].

Experimental Protocol

A typical SEM experiment for battery analysis requires electrode materials, electrolyte films, separators, and other relevant components. Samples can be prepared in different forms, including thin sections, powders, or intact battery cells, depending on the research objectives.

Before imaging, non-conductive samples are often coated with a thin conductive layer (e.g., gold or carbon) to enhance image quality and reduce charging effects. The experiment begins with placing the prepared sample in the vacuum chamber of the SEM.

A focused electron beam is then scanned across the sample surface, generating various signals, such as secondary electrons, backscattered electrons, and characteristic X-rays. These signals are detected and used to produce high-resolution images that reveal surface morphology, particle size distribution, and structural integrity.

SEM is particularly valuable for investigating electrode microstructures, analyzing the formation of SEI, and assessing material degradation over multiple charge–discharge cycles. Its high spatial resolution allows researchers to observe features at the nanoscale, making it a powerful tool for studying the aging mechanisms of battery components.

SoH Estimation

Assessing changes in battery structure to determine SoH requires careful handling of the vacuum electron beam to avoid damaging volatile electrolytes. A key approach is using solid-state electrolytes [137, 138] or non-volatile ionic liquid-based electrolytes [139] to enable detailed electrochemical imaging without evaporation artifacts.

Several studies have successfully employed TEM and SEM to analyze battery degradation:

- Huang et al. [140] demonstrated that the deterioration of oxide anode materials is primarily caused by irreversible volume expansion.
- A combined Electron Energy Loss Spectroscopy and Scanning Transmission Electron Microscopy study [141] identified insulating side-reaction products at the LiCo-

O2/LiPON interface as the primary source of interfacial resistance.
- Chen et al. [142] used in situ TEM to study lithium metal deposition, providing insights into lithium plating behavior.
- Electron holography was employed to analyze interface charge distribution during lithiation in various electrode materials, including $Li_4Ti_5O_{12}$ [143], $LiCoO_2$ [144], and Ge nanowires [145].

Results and Deepening

Ul-Hamid's book [146] is a beginner's guide for SEM and its applications. Wu and Liu [147] provide details about SEM on batteries.

Mechanical Methods

This section delves into another crucial aspect of battery investigation: mechanical methods. These methods encompass two vital aspects, namely mechanical stress and ultrasonic testing. In a world where the reliability and durability of LIBs are of utmost importance, understanding these two methods and their relevance in studying SoC and SoH is essential. This section dives into the exploration of these methods and their impact on the optimization and maintenance of LIBs.

Mechanical stress

Method

To assess the SoC and SoH of a LIB, mechanical parameters can be used as supplementary constraints alongside conventional BMS methods. This approach specifically examines uniaxial mechanical stress resulting from electrode expansion during charge and discharge cycles.

Cannarella et al. [148] demonstrated that stack stress is a dynamic parameter that fluctuates with SoC, driven by electrode deformation, and accumulates over the battery's lifespan. These fluctuations offer valuable insights into battery performance and degradation mechanisms.

Experimental Protocol

The materials required for mechanical stress analysis in batteries include electrodes, separators, and structural components, which can be tested individually or as part of an assembled cell. Samples must be carefully mounted to simulate real-world conditions or specific stress scenarios.

The experiment begins by applying mechanical stress to the battery through methods such as mechanical testing

equipment, pressure cells, or controlled external forces. The material's response to stress is monitored using strain gauges, accelerometers, or other mechanical sensors.

Mechanical stress analysis helps:

- Assess the mechanical integrity of electrode materials and current collectors.
- Evaluate the impact of mechanical forces on battery safety and performance.
- Identify structural failures that could lead to battery degradation or failure.

This type of testing is particularly relevant for electric vehicles and portable electronics, where batteries are subject to frequent mechanical loading during operation and transport.

The collected data provides insights into mechanical behavior, including elasticity, stiffness, and failure points. These findings are crucial for optimizing battery designs and ensuring their long-term structural stability.

SoC Estimation

By measuring stack stress changes [149], which correspond to changes in electrode thickness, it is possible to determine the Capacity/Charge over stress (COS) if a stress-COS relationship for a given cell is known. The use of stack stress measurements to determine the SoC is advantageous because in many systems the stress is more sensitive to SoC than conventional parameters such as voltage. The use of a mechanical constraint to determine the SoC also has the advantage of being able to measure the self-load, as the stress/expansion is directly related to the lithium content of the electrodes. Conventional coulomb counting methods that measure the load passed through an external circuit cannot resolve the internal discharge.

The authors find a stress-SoC relationship dependent on the cell's SoH. This dependence on SoH is the result of the irreversible increase in thickness that occurs during cellular aging, This causes all stress measurements to shift to higher stresses when the electrodes expand irreversibly with the decrease in SoH.

SoH Estimation

Mechanical stress can serve as an effective metric for estimating SoC. By measuring stack stress changes, which correspond to electrode thickness variations, it is possible to establish a Capacity Over Stress (COS) correlation-provided

that a stress-COS relationship is defined for a given cell type [149].

This method offers several advantages over conventional voltage-based or Coulomb counting techniques: Stack stress is more sensitive to SoC fluctuations than voltage in many systems; Unlike external Coulomb counting methods, stack stress measurements can detect internal discharge, providing a more comprehensive assessment of the battery's charge state.

Moreover, Cannarella et al. [149] found that the stress-SoC relationship is dependent on the cell's SoH. As the battery ages and electrodes irreversibly expand, stress measurements shift to higher values, reflecting capacity degradation over time.

Results and Deepening

Grazioli et al. [150] describe a modeling of electrolytes to test mechanical stresses impact on LIBs performance.

Ultrasonic Testing

Method

Ultrasonic testing is a non-destructive evaluation technique that employs high-frequency sound waves to inspect materials, measure thickness, and characterize internal structures [99]. In battery research, it is used to assess internal integrity, detect defects, and monitor battery performance.

This technique relies on ultrasonic transducers that emit and detect sound waves as they travel through battery components such as electrodes, separators, and electrolytes. The interaction of these waves with internal structures produces echoes, which are analyzed to extract valuable information about the SoC and SoH of the battery.

For optimal performance, transducer selection (in terms of frequency and size) should be tailored to the specific battery components under inspection. Advanced signal processing techniques, such as TOF analysis, waveform processing, and imaging algorithms, enhance data interpretation. Additionally, combining ultrasound with complementary methods like X-ray imaging, electrochemical testing, or visual inspection provides a more comprehensive assessment of battery health.

Experimental Protocol

The materials required for ultrasonic testing in battery analysis include electrodes, separators, electrolytes, and assembled battery cells. The samples can be tested individually or as

part of a complete battery system. Proper sample preparation ensures optimal ultrasonic wave transmission and reception.

The experiment begins with ultrasonic wave generation using a transducer. The transducer applies high-frequency sound waves to the sample surface, which propagate through the material. These waves interact with interfaces, boundaries, and defects, causing reflections and scattering. The transducer detects these reflected waves, and the data is analyzed to visualize the battery's internal structure.

Ultrasonic testing can help:

- Detect defects such as delamination, cracks, voids, and electrode degradation.
- Assess electrolyte distribution by monitoring wetting processes.
- Identify gas accumulation resulting from unwanted chemical reactions.
- Measure acoustic impedance to track material degradation over time.

The collected ultrasonic signals are processed to generate images and plots representing the battery's internal composition. Variations in amplitude and TOF indicate structural defects or material property changes.

SoC Estimation

Research has demonstrated a linear relationship between ultrasonic wave transmission and SoC. Specifically, two key parameters-wave amplitude and TOF-have been shown to correlate with battery charge state [151].

Further studies [152–154] confirm these relationships using similar ultrasonic scanning devices. Deng et al. [99] conclude that SoC significantly influences ultrasonic properties, though the exact underlying mechanisms remain an area of ongoing research.

SoH Estimation

Ultrasonic testing provides insights into two critical aspects of battery SoH: the electrolyte wetting process and gas formation.

In Electrolyte Wetting Process, the ultrasound signal is highly sensitive to the electrolyte's interaction with electrode materials [155]. By monitoring this process, researchers can optimize electrolyte selection, improve battery design, and assess long-term material stability.

In Gas Generation Detection, gas evolution inside the battery is a key indicator of degradation, often linked to lithium deposition and side reactions. Ultrasound imaging enables real-time gas detection, allowing researchers to pin-

point degradation sources and assess their impact on battery longevity.

Results and Deepening

Wu et al. [156] provide the protocol for health monitoring, Robinson et al. [157] for cycling behavior, and Shen et al. [158] for in situ errors detection.

Conclusion

This paper has explored the critical role of SoC and SoH estimation in BMSs through the lens of white-box modeling. Grounded in fundamental physics and chemistry principles, white-box models provide a rigorous framework for predicting battery behavior and assessing its long-term health. These models bridge the gap between theoretical understanding and practical applications, enabling real-time monitoring, performance optimization, and extended battery lifespan.

With the increasing demand for electrification, renewable energy storage, and electric vehicles, the accurate estimation of SoC and SoH has become more important than ever. White-box models serve as a cornerstone in this endeavor, offering deeper insights into battery processes and enabling more efficient and reliable energy storage solutions. However, their deployment is not without challenges. The complexity of battery systems, the need for precise parameterization, and real-world uncertainties require ongoing research and refinement to improve the accuracy and adaptability of these models.

While white-box models offer high interpretability and accuracy, they often struggle with real-world uncertainties such as manufacturing variations and environmental influences. To address these challenges, grey-box models have emerged as a hybrid approach, integrating physics-based modeling with empirical data.

Grey-box models balance accuracy and flexibility, making them well-suited for real-world battery applications. By combining the physical understanding of white-box models with the adaptability of data-driven black-box models, grey-box approaches capture the dynamic and nonlinear behavior of batteries under diverse operating conditions. However, their effectiveness relies on access to high-quality experimental data for calibration and validation.

The development of more intelligent and adaptive battery management systems will likely involve a combination of white-box, grey-box, and black-box models. Advancements in machine learning, sensor technology, and electrochemical modeling will further enhance our ability to predict battery degradation, optimize charging strategies, and

improve energy efficiency. Continued research into hybrid modeling approaches will be essential to overcoming the limitations of individual models and ensuring sustainable, high-performance energy storage solutions for the future.

Data Availability No datasets were generated or analysed during the current study.

Declarations

Conflict of interest The authors declare no Conflict of interest.

Ethics approval Not applicable.

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