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Review

Component Degradation in Lithium-Ion Batteries and Their Sustainability: A Concise Overview

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Abstract: Researchers are presently involved in the creation of materials for high-voltage lithium-ion batteries, with a particular emphasis on their practical uses. However, it is important to acknowledge that the components of lithium-ion batteries frequently undergo substantial loss of capacity during the cycling process, which hinders their potential for becoming commercially viable. Lithium-ion battery electrodes can degrade due to electrochemical surface reactions that occur with the electrolyte at the interface between the electrode and electrolyte, as well as from structural degradation within the electrode particles. The presence of structural degradation can be ascribed to the development of imperfections or unstable states, which are expedited by electrochemical processes arising from the electrolyte if unstable states emerge near the electrode/electrolyte contact. Underlying degradation mechanisms can enhance improvements in the electrochemical properties of the electrodes. This paper presents a comprehensive analysis of the various degradation mechanisms that impact the components of lithium-ion batteries to improve energy efficiency. It also discusses innovative methodologies used to analyze the degradation phenomena that occur at the surface of the electrode and within individual battery components.

Keywords: characterization; lithium-ion battery; sustainability; energy storage; electrode; degradation; electrolyte; electrochemical properties; energy efficiency

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1. Introduction

An effective approach to reducing the use of fossil fuels is to promote the use of electric vehicles, which obtain their energy from renewable energy sources. However, solar and wind energy generation is unreliable and intermittent, making it challenging to employ these important electric resources consistently and stably. To address this issue, the use of batteries as energy storage devices in conjunction with renewable energy may significantly increase the utilization rate and stability of renewable energy [1]. In this sense, electric vehicles are becoming increasingly popular as part of the worldwide decarbonization effort [1]. Despite notable developments since their creation, conventional combustion engines still have relatively low efficiency in modern cars. In the future, battery storage technology is expected to be the dominant way of storing electrical energy in automobiles [2]. Although battery technology offers various benefits, the method also has inherent drawbacks. For example, the length of time it takes to charge battery-powered vehicles might range from thirty minutes to several hours [3]. Hence, lithium-ion batteries (LIBs) are now widely used in many consumers' electronic products because of their favorable properties, including high energy density and the ability to be customized for different physical setups. An effective method to increase the energy density of a lithium-ion battery is to elevate and improve the properties of the cathode material [4]. As a result, there has been progress in the development of high-voltage lithium-ion cathode materials that undergo reduction–oxidation (redox) processes at elevated potentials [5]. However, it is important to mention that specific cathode materials, such as LiCoPO4, which have a high voltage, undergo substantial degradation when subjected to multiple cycles [6]. Under high potential, traditional electrolytes experience oxidation, resulting in the deterioration of the electrode–electrolyte interface [6]. Conversely, when the anode is at its operational potential, the electrolyte undergoes reduction and forms a solid electrolyte interphase (SEI). This SEI acts as a protective coating for the anode, preventing additional degradation caused by the electrolyte. However, the production of the cathode–electrolyte interphase (CEI) film can sometimes be unstable, which interferes with the electrode passivation process [7]. The structural degradation in cathode particles might be caused by the development of unstable de-lithiated phases and anti-site defects that are very susceptible to damage from the electrolyte [8]. Lithium-ion batteries have been the main method of storing energy for battery electric vehicles (BEVs). Nevertheless, the deterioration of the components of lithium-ion batteries and the subsequent decline in performance suggest that these batteries are encountering formidable obstacles in the market. To justify significant investments in storage batteries, the most important components of the battery electric vehicle (BEV) must have an exceptionally long lifespan. Researchers must fully understand and collect long-term data within a restricted timeframe about the degradation mechanisms found in lithium-ion battery components [9]. Nevertheless, understanding the degradation mechanisms may be impeded by the complex nature of the microenvironment found in lithium-ion battery electrodes. Moreover, the existence of various degradation environments stems from the composite nature of electrodes, which includes a binder, active material, a conductive additive, and an electrolyte. Additionally, the fluctuations in potential, stress, and temperature contribute to the complexity of understanding the degradation mechanism, necessitating the use of sophisticated characterization techniques. The utilization of diverse microscopy techniques can establish a direct relationship between microstructures and the chemical composition of the deteriorated material, hence providing valuable insights into the surrounding local environment [10]. This study aims to examine the deterioration of the components of lithium-ion batteries through the utilization of several characterization techniques. The objective is to comprehend the dominant degradation mechanism of the system components and improve their potential use in lithium-ion battery materials.

2. Using Batteries for Energy Storage

Batteries have become one of the most widely used and versatile technologies for energy storage. In the context of modern energy systems, they serve as essential components for both temporary and long-term storage of electrical energy. A battery stores energy chemically, which can be later converted back into electricity when required. This ability makes batteries invaluable in numerous applications, ranging from powering portable electronic devices to stabilizing power grids and enabling renewable energy integration [11,12]. Batteries play critical roles in renewable energy integration, where the battery allows for storing extra energy produced during periods of high output so that it can be utilized during periods of high demand or when production is low, ensuring a steady and reliable energy supply [13–15]. Batteries have also been shown to play a critical role in grid stability by providing services such as frequency regulation, voltage control, and load balancing [16]. By storing energy during periods of low demand and discharging it when demand spikes, batteries can help balance the supply and demand of electricity on the grid, reducing the risk of blackouts and improving overall grid efficiency. Another area of application is in electric vehicles, where batteries are allowed to store energy for propulsion. Batteries, in addition to their role in large-scale applications, are also used in small-scale, off-grid, or backup power systems. For example, uninterruptible power supplies (UPSs) for critical systems, e.g., in hospitals, data centers, and homes, rely on batteries to provide backup power in case of grid failures.

2.1. The Basics of Lithium-Ion Batteries

Batteries consist of interconnected electrochemical cells, functioning as devices that store chemical energy and then transform it into electrical energy [17]. Electrochemical cells depend on reduction–oxidation (redox) processes, where electron transfer happens between species, causing the reactants and products to undergo oxidation (electron loss) and reduction (electron gain) [18]. Physically separating the oxidation and reduction processes, which take place on distinct electrodes known as the anode and cathode, allows electrochemical cells to store energy [18]. After being discharged, the two electrodes are connected electrically, which allows the redox reaction to happen naturally. Electrons can be collected and utilized for practical applications, while ions are carried through a liquid electrolyte. Often, the anode is made up of a carbon-based active material that is adhered to a copper current collector [19]. The process of designing each electrode starts with the use of active material powder, consisting of spherical or ellipsoidal particles ranging from nanometer to micrometer sizes. In the design, the powder is blended with conductive additives and binders, resulting in a mixture called porous electrode that maintains liquid-filled pores. The structure contains the active material that forms the central component, selected to facilitate the battery's electrochemical reaction, generating electrical current while maintaining structural integrity during the intercalation and deintercalation of lithium ions.

Lithium ions (Li^+) are the primary charge-carrying entities in lithium-ion batteries, as depicted in Figure 1.

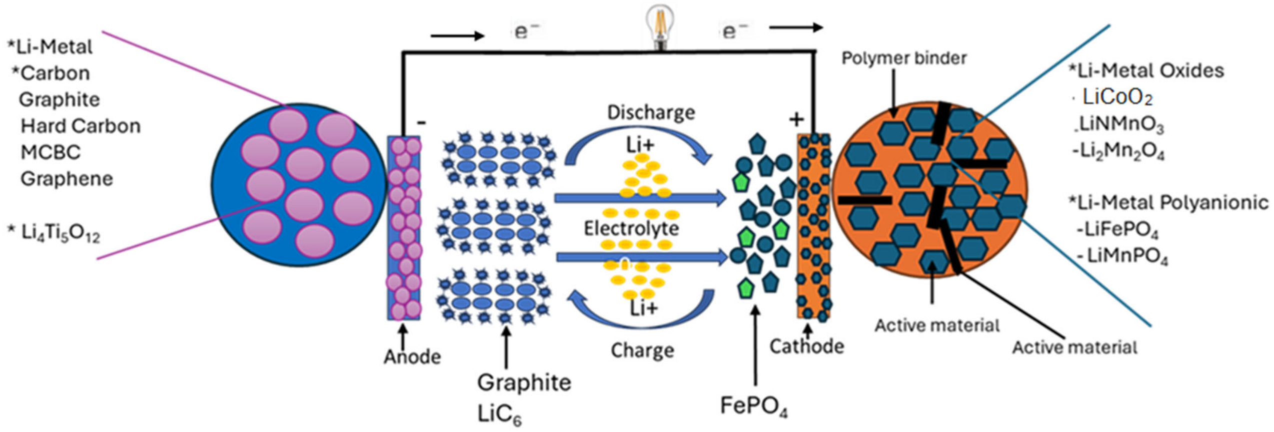


Figure 1. Schematic diagram of basic components of an operational lithium-ion battery.

2.2. Lithium-Ion Battery Components

Basically, there are four main parts of a lithium-ion battery. While "cathode" is used to designate the positive electrode, "anode" is designated as the negative electrode.

a. Anode (negative electrode): The anode's role is that of a host for lithium ions. It is mostly made from graphite, which it gives out during usage.

b. Cathode (positive electrode): The cathode is a positive electrode often made from lithium metal oxides. It plays a vital role of serving as the source of lithium ions during discharge.

c. Electrolyte: The liquid or gel electrolyte is lithium salt suspended in an organic solvent, which also helps in the conduction of ions between the electrodes.

d. Separator: Polypropylene and polyethylene are the materials used in the porous membrane, which separates the anode and the cathode from short-circuiting while allowing ions to pass through the electrolytic solution.

Although these components have been specifically designed to aid the usage and processes of the battery, they are also susceptible to damage over time due to several degradation mechanisms that exist across these components.

2.3. The Charging and Discharging Process of a Lithium-Ion Battery and Failure Mechanisms

The anode and cathode serve as the receptacles for the lithium ions that provide the battery's electricity. The porous separator serves to insulate the electrodes electrically, while the electrolyte aids in the transit of ions within the system. The lithium ions undergo charging and discharging, during which the lithium ions perform a simple movement from one electrode to another without undergoing any chemical reaction [20]. During operation, the lithium ions move to the spaces between atoms in the negative electrode (anode), which is usually made of graphite, when the battery is initially charged. The non-aqueous electrolyte that transports the ions is lithium hexafluorophosphate (LiPF6) dissolved in carbonates, which acts as a conductive salt [21]. During the discharge of a cell, almost all the lithium is inserted into the cathode's active material. During the charging process, lithium undergoes oxidation at the cathode due to the application of an external current. The polymer separator is positioned between the two electrodes to avoid direct contact between the anode and cathode, while enabling the passage of ions between the electrodes. At lower potentials, the potential range of a graphite anode exceeds the stability window of the electrolyte. However, during the lithiation process, the potential lowers to 100 millivolts (mV) compared to metallic lithium in typical operating circumstances [22]. The ions in the electrolyte begin to dissolve when potential is applied, and the movement of these ions generates the electric current flowing through the electrolyte. It is important to mention that most electrolyte components start to break down when exposed to a voltage of 1 volt (V) compared to metallic lithium [23]. According to reports, electrolyte reduction takes place on the surface of the anode, leading to the creation of a solid coating on the anode particles [23]. Throughout the film production process, the electrolyte components experience breakdown, leading to the depletion of lithium and producing an irreparable loss during the initial transformation. However, the main indication of aging is the gradual formation of the surface coating over the course of an individual's life in the LIB. During the initial formation phase, a significant amount of accessible lithium is used up in the first cycle. However, in the following cycles, the intake of lithium is significantly diminished. However, unlike many other battery technologies, LIBs require regular monitoring of their cell voltage and temperature [24].

3. Lithium-Ion Battery Degradation Mechanisms

The cathode and anode material of the battery affect the aging mechanism and the cycle life. There are many conflicting effects that produce aging effects on battery and energy storage systems, such as environmental conditions, product usage patterns, etc. [25–29]. Degradation can occur during rest periods, in which energy is not being extracted from the battery. Loss of active material (LAM) and loss of lithium inventory (LLI) are the two main degradation processes in LIBs. In the electrochemical process, lithium is consumed in such side reactions, reducing battery capacity. Electrode degradation leads to the loss of active material, which decreases the available sites for lithium intercalation. Zhu et al. [30] reported that the mechanism results in power and capacity fading at the anode and cathode. Degradation does affect all aspects of a battery and degrades with time and use, including the active materials and inactive components. Although there are numerous processes involved in the degradation of lithium-ion batteries, it is difficult to investigate these processes independently as they occur on comparable timescales and interact with one another.

3.1. Degradation of Lithium-Ion Battery Components

LIBs, or lithium-ion batteries, experience slow degradation of their components when they are used in various applications and for storage [31]. Degradation can occur in various components of LIBs through different methods. As LIBs are employed in different applications and storage, the components gradually degrade due to irreversible side reactions. Effects like capacity fade can result when environmental and operational conditions exacerbate such reactions [32]. Within LIBs, degradation can occur in a variety of different components and through a variety of different processes, which will be discussed subsequently.

3.2. Cathode Degradation

In lithium-ion battery degradation, cathode degradation is more unpredictable because cathode aging is strongly dependent on the material used, and there are many different cathode chemistry types in use [31]. The term cathode–electrolyte interphase (CEI) is commonly used to characterize the cathode-side surface layer formed of solid-phase electrolyte breakdown products, whereas solid–electrolyte interphase (SEI) refers to what forms on the anode side. The formation of the CEI and SEI depletes the available lithium in the battery and hinders lithium transport at the electrode–electrolyte interface. Cathode–electrolyte interphase (CEI) formation is a key process that impacts the performance of lithium-ion batteries. Cathode degradation mechanisms include losses of active material and CEI formation. Due to the cathode material composition and electrochemical process that occurs in the battery, transition metal dissolution (TMD) is often observed for active materials (Ni, Mn, Co, Fe), in which the cathode may dissolve in the electrolyte, leading to a loss of active material [33]. Zhang et al. [34] reported that the process is enhanced at elevated temperatures. Moreover, small amounts of water in the battery could react with LiPF6 salt via hydrolysis to produce hydrofluoric acid (HF), which also causes TMD to occur. On the other hand, batteries are susceptible to TMD when the electrode is completely discharged, and this behavior is predominantly associated with manganese-containing cathodes [35]. Li et al. [36] revealed that dissolved transition metals may be deposited on the anode, increasing conductivity that in turn promotes SEI growth, as well as the forming of dendrites [37].

When the cathode is exposed to the electrolyte, both materials react with each other, consuming lithium inventory. As a result of continuous operation of the battery, lithiation and delithiation occur, causing volume changes that generate mechanical stresses and may lead to cracking, exposing additional reaction sites [38]. Inhomogeneous lithiation, such as Jahn–Teller distortion, gives rise to structural phase transitions occurring in the cathode material, unlike the anode, which further shrinks the amount of lithium ions that the cathode can absorb [39]. Although this effect is more pronounced at low state of charge (SOC), researchers [40] have reported that different dopants can be used to provide the stabilization of this structure. These structural alterations can reduce the active material available in the cathode and expose the cathode to electrolytic effects.

Other researchers [41] have shown that gas generation may also lead to cracking due to oxygen vaporization out of the metal oxide at elevated temperatures, or to the breakdown of electrolytes at elevated voltages. Point defects in the lattice is another phenomenon that can cause the degradation of the cathode. It has been revealed that, at high temperature

(150–310 °C, depending on the material), the cathode itself may decompose, resulting in deactivation of the active material and gases being released.

The SOC and temperature joint estimation based on ultrasonic reflection waves is a potential method for assessing the degree of degradation of lithium batteries by integrating SOC and temperature, both of which are key elements in battery health. SOC is a vital measure of a battery's charge level and gives useful information about its health. Monitoring SOC allows one to assess the amount of charge held in the battery and detect capacity loss over time, which is a primary cause of battery degradation. Temperature, on the other hand, has a significant impact on the depreciation of lithium batteries. High temperatures can accelerate chemical reactions within the battery, causing thermal runaway and further degradation. Elevated temperatures can also have an impact on the battery's internal structure, producing changes in material qualities, capacity loss, and potentially safety issues. This method estimates battery degradation more comprehensively by combining these two parameters, SOC and temperature, using ultrasonic reflection waves. Ultrasonic waves can be used to investigate the battery's interior properties, such as material degradation, and to reflect changes in impedance or stiffness caused by temperature and state of charge. This joint estimation enables the real-time monitoring of both the charge level and temperature, making it a more precise and dependable method for measuring the overall health and degradation of lithium batteries.

3.3. Anode Degradation

During the electrochemical process, lithium ions coming from cathode and the organic compounds coming from the electrolyte integrate with the graphite anode and form a thin film called the SEI [42]. Sarkar et al. [43] demonstrated that SEI formation is an irreversible process and that lithium is consumed during the process, which removes lithium from the cycling system, thereby diminishing battery capacity. The deposition of organic electrolyte solvents on conductive surfaces creates a stable SEI film, which coats the graphite electrode over tens to hundreds of angstrom thickness during the initial several cycles of electrochemical operation, and organic and inorganic salts entrap gas molecules to form the film [42]. Irreversibly, a significant amount of the initial capacity is lost due to the formation of the SEI, and after the formation of the SEI, the graphite electrode becomes fully covered and the reaction ceases. While the SEI passivates the anode against further reaction with the solvent, the process induces the insulation of the anode and allows the precise selection of permeable lithium ions. A stable SEI layer is one of the essential conditions for proper lithium-ion battery operation. However, because SEI formation is highly dependent on the kind of graphite, graphite microstructure, electrolyte concentration and composition, electrochemical circumstances, and cell temperature, it is difficult to control [44]. As the operation of the battery increases, the degradation of the SEI decreases and each SEI dissolution exposes the graphite to the electrolyte, which leads to further SEI growth and therefore further capacity loss [45]. Higher temperatures increase dissolution, and at elevated temperatures, rising voltage also becomes a relevant parameter in SEI formation [46]. Furthermore, there can be gas creation upon electrolyte depletion in the electrode, leading to increased pressure and stress [47]. Xu et al. [48] revealed that the volume of graphite expands and contracts by about 10% when cycling the battery, lithiating and de-lithiating its structure. Mechanical stresses produced by each mechanism can cause particle cracking. Agubra et al. [49] demonstrated that a battery's anode is significantly lithiated and has low potential at high SOC. The authors further revealed that the deposition of lithium on the anode becomes thermodynamically favorable below 0 volts vs. Li/Li^+ at the anode surface.

At these potentials, some of the lithium ions get deposited on the surface of the electrode as metallic lithium instead of diffusing into the anode during charging [50]. To mitigate this unwanted lithium plating, batteries are conventionally designed with a greater anode capacity compared to the cathode, resulting in an under-lithiated state for the anode.

Even with this precaution, researchers [51] have revealed that lithium plating due to overcharge is still possible due to a nitrogen/phosphate (N/P) ratio higher than expected. It was demonstrated that high charging rates, even with carefully planned ratios, can result in lithium plating if they exceed the rate at which lithium diffuses into the graphite. This gas generation is inhibited at low temperatures by competing processes, such as ion diffusion in the anode and/or electrolyte, which promote greater lithium plating and dendritic development [52]. During lithium-ion intercalation and de-intercalation, anode metallic lithium is stripped for discharge and the dead lithium acts as source of capacity loss if electrical contact with the anode is lost.

3.3.1. The Progress of Graphite in Anode Passivation Films

Graphite is commonly employed as the main active substance for the anodes of lithium-ion batteries (LIB) due to its advantageous performance characteristics. Nevertheless, issues with the creation of passivation coating, mechanical stress, and the deposition of lithium metal continue to exist. Introducing silicon oxides can enhance the performance of lithium-ion batteries (LIB), but this comes at the expense of increased side reactions [53]. The deposition of passivation films on the graphite active material surface, usually known as the solid SEI, is believed to be the most consistent and continuous kind of deterioration that happens at the anode [54]. The irreversible electrochemical reaction that depletes lithium is caused by the thermodynamic unpredictability of the electrolyte at the operating potential [55]. The by-products of the process create a layer that functions as an electrical insulator. This barrier prevents electrolyte molecules from entering but permits the passage of lithium ions, resulting in the formation of a passivating layer. Lui et al. [56] demonstrated that ionic conductivity decreases and internal resistance rises as a result of electrolyte breakdown. The scientists stated that the process is worsened by the continuous exposure of the active material to the electrolyte because of volume variations and mechanical stresses. Oxidation–reduction processes can occur in the copper foil current collector due to high electrode potentials during over-discharge [57]. Moreover, inadequate adhesion between the active material and the current collector might result in the loss of active material, leading to a substantial decrease in capacity [57]. During this process, the capacity is further diminished, as dissolved copper species can be deposited on the surfaces of the electrodes, obstructing intercalation sites and diminishing the capacity and longevity of the anode.

3.3.2. Silicon Oxide Volume Fluctuation

Silicon (Si) and silicon oxides have shown promise as anode materials in lithium-ion batteries (LIBs), and they are commonly used in combination with graphite [58]. Graphite degradation and the processes of lithiation and delithiation in silicon have been shown to result in substantial changes in volume [59]. The rapid increase in the solid electrolyte interphase (SEI) and the loss of lithium are a result of variations in volume, and induce the pulverization of silicon particles and the electrode.

A degradation mechanism spectrum for the cathode may encompass numerous essential processes, such as capacity fading, structural degradation, electrolyte dissolution, side reactions, active material loss, high-voltage instabilities, and cycling-induced stress. The degradation mechanism spectrum varies based on the kind of cathode material, battery architecture, cycle conditions, and environmental factors, including temperature. Understanding these mechanisms is critical to increasing the longevity and performance of electrochemical cells.

Figure 2 illustrates the impact of the expansion and contraction of the Si during lithiation and delithiation on the fractures in the SEI layer. These cracks enable the previously inaccessible electrode surfaces to be exposed to the electrolyte.

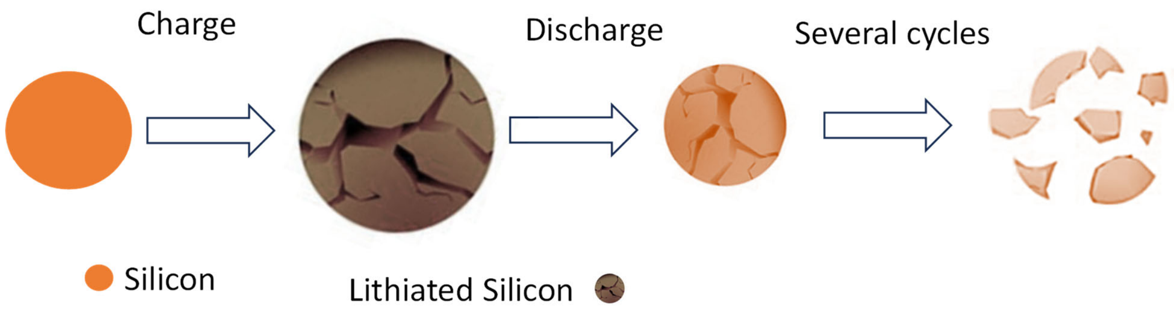


Figure 2. A schematic figure illustrating the process of silicon particle cracking that occurs during the charging and discharging of a battery.

When comparing silicon oxide species (SiOx) to graphite, it has been demonstrated that the changes in volume in SiOx are less significant than in elemental silicon [60]. However, the specific mechanism behind this phenomenon remains a significant area of concern. Elemental silicon exhibits a higher initial coulombic efficiency compared to silicon oxides due to the irreversible production of lithium silicates during the first lithiation process [61]. Throughout the next delithiation process, the lithium silicates remain inert. Nevertheless, the Si that is still present can form a reversible alloy with lithium, as documented by Kim et al. [62].

3.3.3. Degradation of the Anode Material Caused by Mechanical Processes

The deterioration of graphitic active materials is further exacerbated by the mechanical stress produced during intercalation. Lithium intercalation in the gaps between graphite planes can lead to an expansion of the graphite layers [63]. The cyclical expansion and contraction that occurs during electrochemical cycling has the potential to eventually cause microcracks to disperse over the polycrystalline graphite's grain boundaries [64]. The level of cracking in the active material is directly proportional to the magnitude of the electric current flowing through it [65]. Research has demonstrated that particle cracking is exacerbated by the process of solvent co-intercalation, gas development, and electrolyte reduction with graphite [66]. These factors might collectively contribute to a rapid degradation of the electrode. Furthermore, the occurrence of significant cracking might cause the graphite particles to undergo irreversible expansion while cycling, resulting in an overall increase in the volume of the active material [67]. This phenomenon arises when graphite is exposed to repetitive mechanical strain. Consequently, the anode graphite surfaces become exposed to the electrolyte.

The state of health usually deteriorates after degeneration. The same is true of micro-health metrics, which represent the performance of the battery's electrolytes and active material. Variations in these values might reveal the internal health status of the battery. Xu et al. [68] developed a method based on a simplified pseudo two-dimensional model, employing the Padé approximation for micro-health parameters that characterize the electrolyte and negative electrode material in $LiFePO_4$ batteries. The findings showed that the quick identification method for micro-health parameters describing negative electrode material and electrolyte performance can enhance the performance consistency of regrouped retired batteries and offer a multi-dimensional sorting basis for the second-use application of retired batteries from electric vehicles.

3.4. Phase Transitions During Lithium Intercalation/Deintercalation

Lithium-ion batteries (LIBs) operate through the insertion (intercalation) and extraction (deintercalation) of lithium ions (Li^+) into and from the host material of the electrode, typically graphite for the anode and various transition metal oxides (such as $LiCoO_2$) for the cathode. These processes are accompanied by phase transitions in the electrode materials, which significantly affect the electrochemical performance, efficiency, and stability of the battery [69]. The phase transitions during lithium intercalation/deintercalation have several critical effects on the performance of lithium-ion batteries, including capacity fade, hysteresis and voltage plateau, kinetic limitations, and thermodynamic and structural stability. Understanding the phase transitions during lithium intercalation/deintercalation is crucial for enhancing the capacity, cycling stability, and rate performance of lithium-based batteries.

3.4.1. Intercalation and Deintercalation Procedure

Lithium ions migrate through the electrolyte and intercalate into or deintercalate from the crystal structure of the electrode materials throughout a lithium-ion battery's cycle of charging and discharging. This process typically involves releasing lithium ions from the cathode materials that travel through the electrolyte and intercalate into the anode (mostly graphite during the discharge process). On the other hand, lithium ions are deintercalated from the anode material, travel back through the electrolyte, and intercalate into the cathode material during the charging process, which is called lithium deintercalation. These processes are often accompanied by significant structural changes within the electrode materials, leading to phase transitions that can influence the electrochemical properties.

3.4.2. Phase Transitions in Graphite (Anode Material)

Graphite, a frequently used anode material in lithium-ion batteries, experiences intricate phase changes during the intercalation and deintercalation of lithium ions. At low levels of lithium intercalation solid solution phase, lithium ions are inserted into the graphite's layered structure, forming a solid solution. In this phase, lithium ions occupy intercalation sites between the graphite layers, the structure remains relatively stable, and the material retains its layered structure [70]. However, during the stage transitions, different arrangements of lithium ions are represented in the interlayer space of the graphite. In this process, as lithium is inserted, the spacing between the graphene layers increases, which can lead to a corresponding change in the overall structure. At higher levels of lithium insertion, a transition occurs from the stage model, resulting in the formation of a lithium–graphite alloy, marking a distinct phase transition [71]. Sheng et al. [72] revealed that the lithium graphite alloy formation phase is characterized by the formation of a solid solution rather than distinct stages, and the graphite undergoes substantial structural changes. Further studies [73] have reported that the phase transitions in graphite are highly reversible, but the excessive insertion or extraction of lithium can lead to irreversible changes, such as the formation of cracks or the collapse of the graphite structure, affecting the long-term stability of the anode.

3.4.3. Phase Transitions in Cathode Material

In cathode materials, particularly in lithium metal oxides such as $LiCoO_2$, $LiFePO_4$, and $LiNiMnCoO_2$, lithium intercalation/deintercalation is accompanied by significant phase transitions that influence the material's electrochemical behavior. As $LiCoO_2$ is among the cathode materials most frequently utilized in LIBs, the phase transition in $LiCoO_2$ occurs as lithium ions are deintercalated from the material during charge, leading to the oxidation of Co^{2+} to Co^{3+}. When lithium is re-intercalated during discharge, Co^{3+} is reduced back to Co^{2+}, and the primary phase transition in $LiCoO_2$ involves the transition between the α-phase and β-phase [74]. Zhu et al. [75] demonstrated that at the high-voltage end of the discharge, the material undergoes a first-order phase transition characterized by a significant change in the lattice structure, which is accompanied by a volume change and increased stress in the material. Researchers [76] have proposed several strategies to mitigate the impact of phase transitions on the performance of lithium-ion batteries by designing materials with more stable phase transitions, or those that exhibit smoother transitions. Other researchers [77] have suggested that nanostructuring the electrode materials or using composite materials can help reduce the strain during phase transitions and improve the overall performance by providing more pathways for lithium-ion diffusion. Further studies [78] have also recommended that modifying the electrolyte to reduce the adverse effects of phase transitions, such as by using additives that stabilize the SEI layer or prevent side reactions, can improve the overall efficiency and lifetime of batteries.

3.5. Electrolyte Degradation

The utilization of additives during manufacturing has a notable influence on the degradation of electrolytes and the occurrence of breakdown reactions at the surface of the anode and cathode, as well as inside the overall electrolyte [79]. Evidence suggests that the presence of di-fluorophosphate (DFP) and phosphorus fluoride oxide (POF3) in tested samples indicates the decomposition of the conductive salt, lithium hexafluorophosphate (LiPF6), due to water impurities. This decomposition leads to a decrease in the ionic conductivity of the electrolyte and an increase in the ohmic resistance of the cell [80]. The development of the SEI on the anode surface has been seen to primarily involve the polymerization of ethylene carbonate (EC), resulting in solvent degradation [80]. The advancement of this reaction with electrolyte additives is of utmost significance to the subsequent functionality of the LIB, particularly during the initial few cycles of battery operation. High temperatures, particularly when lithium carbonate ($LiCO_3$) is present, intensify the release of carbon dioxide (CO_2) gas. Carbon dioxide (CO_2) can lead to the dehydration of specific areas on the electrode, and it plays a role in the self-discharge processes that bring lithium back to the cathode.

3.5.1. Electrolyte Degradation in Lithium-Ion Batteries: Salts and Solvents

Electrolyte degradation in LIBs critically affects their performance, lifetime, and safety. During charge and discharge cycles, the electrolyte conducts ions between the anode and cathode. Its degradation has reported to be mainly caused by the instability of the salts and solvents included that play an integral role in total electrolyte performance [81].

3.5.2. Electrolyte Composition

A common lithium-ion battery electrolyte is lithium salt (typically lithium hexafluorophosphate, LiPF6) dissolved in an organic solvent or solvent mixture. The solvents generally include carbonates, such as ethylene carbonate (EC), diethyl carbonate (DEC), dimethyl carbonate (DMC), etc., and in the solvent, the lithium salt splits into lithium (Li^+) and anions (e.g., $PF6^-$), enabling ionic conduction. However, it has been reported that the absence of a stable electrolyte can be a major barrier to battery technology because the electrochemical stability, ionic conductivity, and the overall life of LIB can be negatively affected due to electrolyte degradation [82].

3.5.3. Degradation of Lithium Salts

The decomposition of lithium salt is an important contributor to the degradation of the electrolyte. LiPF6, the most widely employed salt in commercial LIBs, is prone to hydrolysis and thermal decomposition, both of which impair the performance of the battery. During the hydrolysis process, the LiPF6 can react with water in the presence of moisture to produce hydrofluoric acid (HF) and lithium fluoride (LiF). The produced HF is extremely corrosive and detrimental to the electrodes and current collectors within the battery. Additionally, the HF promotes the electrolyte's breakdown and the development of an SEI on the anode that is detrimental to the cycling stability of the battery. On the other hand, during the thermal decomposition, LiPF6 decomposes thermally at high temperatures to form toxic reactive gases [83]. These products destabilize the electrolyte even more and can break down the internal components of the battery. Decomposition of the LiPF6 at elevated temperatures has also been shown to present a serious threat to batteries running under high-power conditions, and alternative lithium salts are being sought that have higher thermal and moisture stability [84].

3.5.4. Degradation of Solvents

Electrolyte solvents, mainly carbonates, are also susceptible to degradation during battery operation, particularly at high voltage, temperature, and cycling conditions. The formation of side products due to this degradation may negatively affect the performance of the electrolyte. These decompositions produce different organic and inorganic products, some of which are detrimental to the performance of the battery. At elevated voltages, solvent molecules can undergo oxidation reactions, generating gaseous species and contributing to the pressure build-up in sealed battery cells. This decomposition results in the inability of the solvent to transport lithium ions efficiently, reducing the overall capacity of a battery. At the anode, solvents can also be reduced, resulting in the generation of solid deposits on the surface of the anode, which can affect the structure factor of the SEI layer [85]. These materials block the transport of lithium ions and lead to capacity loss. This reaction also raises the resistance of the battery, decreasing the efficiency with which it works. Attempts to prevent electrolyte degradation have centered around both the salt and solvent components. Although LiPF6 is the most studied lithium salt, new lithium salts are proposed to exhibit better thermal stability and hydrolysis resistance than LiPF6, decreasing the generation of HF and other reactive by-products [86]. Moreover, a number of solvent additives have been designed in order to enhance electrolyte stability. These additives potentially form stable passivation layers on electrodes to inhibit solvent decomposition or serve as free-radical scavengers [87].

3.6. Preprocessing of Materials with Organic Solvents or Heating for Volatilization in Lithium-Ion Battery Degradation Analysis

3.6.1. Importance of Preprocessing in Degradation Analysis

Several components of lithium-ion batteries experience chemical changes that lead to degradation when in operation. The release of volatile organic compounds (VOCs) or gases can occur because of the degradation of components due to electrochemical reactions within the cells. Often the formation of solid by-products is also experienced as part of the degradation phenomenon, which contributes to the overall degradation and failure of the battery. Thus, knowledge of these phenomena is necessary to improve volatilization and battery safety. This knowledge will also help in identifying degradation pathways through tracking the volatile components released during degradation, thereby providing valuable insights into the chemical reactions occurring inside the battery. Moreover, some volatile products, like gaseous components, can lead to the swelling, leakage, or even thermal cracking of batteries [88]. In the realm of lithium-ion battery degradation research, it is critical to emphasize the effective preprocessing of materials to comprehend failure, spills of volatile products, and performance decay over time. Maintaining the active battery components (electrodes, electrolytes, and separators) at room temperature may be undertaken by heating them up or treating them in organic solvents to evaporate certain components. This will aid the battery degradation analysis, and the use of solvents or heating will enable the evaporation of the components. Apart from the chemical reactions, other interactions alter the behavior of the battery over time; when li-ion batteries are actively used, degradation is considered as the source of side effects.

3.6.2. Preprocessing with Organic Solvents

Organic solvents are often used to extract, clean, or dissolve components of a lithium-ion battery that can volatilize during degradation. This technique is especially useful for solubilizing polymeric components where organic solvents can dissolve polymer binders, which are used in electrodes. Fink et al. [89] reported that preprocessing with organic solvents can help in removing surface contaminants and studying electrolyte degradation. In the preprocessing with organic solvents, some of the common organic solvents used include acetone, which is employed for cleaning electrode materials or dissolving polymer binders without interfering significantly with the material's properties [90]. Other organic solvents include N-Methyl-2-pyrrolidone (NMP), which is frequently used to dissolve binders in electrode slurry formulations and can be analyzed for its role in the degradation of binder materials.

3.6.3. Employing Heating for Volatilization Preprocessing and Analytical Techniques

Another method to induce volatilization is heating the materials to specific temperatures. The process supports simulating the conditions that batteries may experience during accelerated aging or overheating, thereby facilitating the release of volatile compounds. Zhang et al. [91] revealed that subjecting materials to elevated temperatures accelerates the volatilization process and mimics conditions found in faulty batteries. In another study, Chombo et al. [92] demonstrated that heating materials like electrodes, separators, and electrolytes in an inert or controlled atmosphere (such as nitrogen or argon) helps identify gases released during degradation, such as CO_2, CO, HF, or other VOCs. This implies that heating the lithium-ion battery component can help researchers to study the thermal stability of materials and to understand the temperature ranges at which certain degradation processes become pronounced.

To improve the study of processes of degradation and volatilization types, several analytical techniques are used. While thermogravimetric analysis (TGA) is a technique used to measure changes in the mass of materials as the materials are heated and/or cooled and as a function of time, the method is also used to assess mass loss due to the volatilization of volatile components or degradation products. Another technique is Gas Chromatography–Mass Spectrometry (GC-MS), in which the released volatile organic compounds from battery materials can be identified and quantified [93]. The test provides a precise chemical fingerprint of the gases released when heated. Fourier-Transform Infrared Spectroscopy can be utilized for the characterization of vapors and of the surface of heated/degraded materials also, and to detect some specific functional groups which might have formed or volatilized during the degradation process. Moreover, electrochemical impedance spectroscopy (EIS) coupled with heating can track how the degradation of the electrolyte or electrode material affects cell performance. While the above methods of preprocessing materials with organic solvents or heating have some benefits, Zhao et al. [94] showed that some materials change permanently upon heating, making it difficult to interpret the output. Furthermore, the choice of solvent can have an impact on the stability of the battery components themselves, and solvents can also degrade, making it difficult to untangle degradation pathways.

4. Methods for Determining LIB Degradation

It is imperative to characterize degradation in lithium-ion batteries to understand performance loss and increase lifespan. Different aspects of degradation are studied by various techniques. EIS (electrochemical impedance spectroscopy) gives insight into internal resistance and aging effects, and CV (Cyclic Voltammetry) shows alterations in electrochemical behavior. Capacity fade is monitored over charge/discharge cycles, as shown in Figure 3.

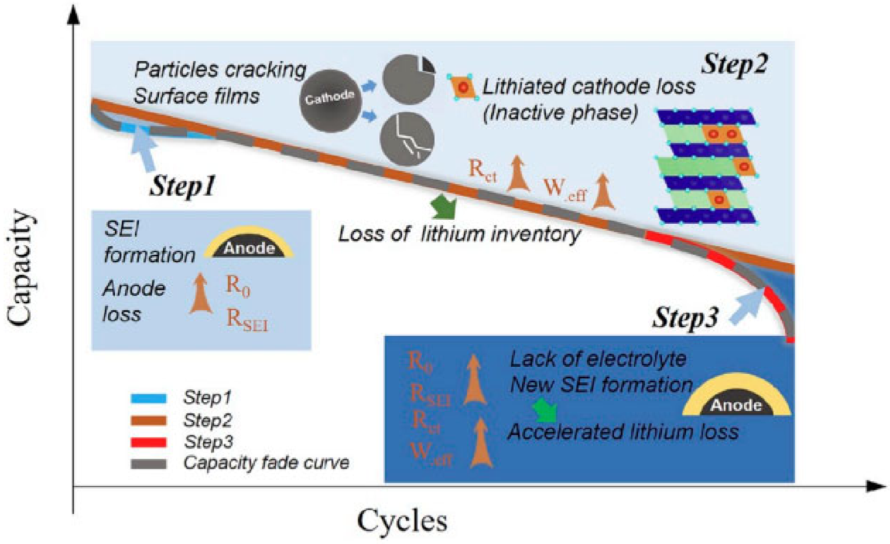
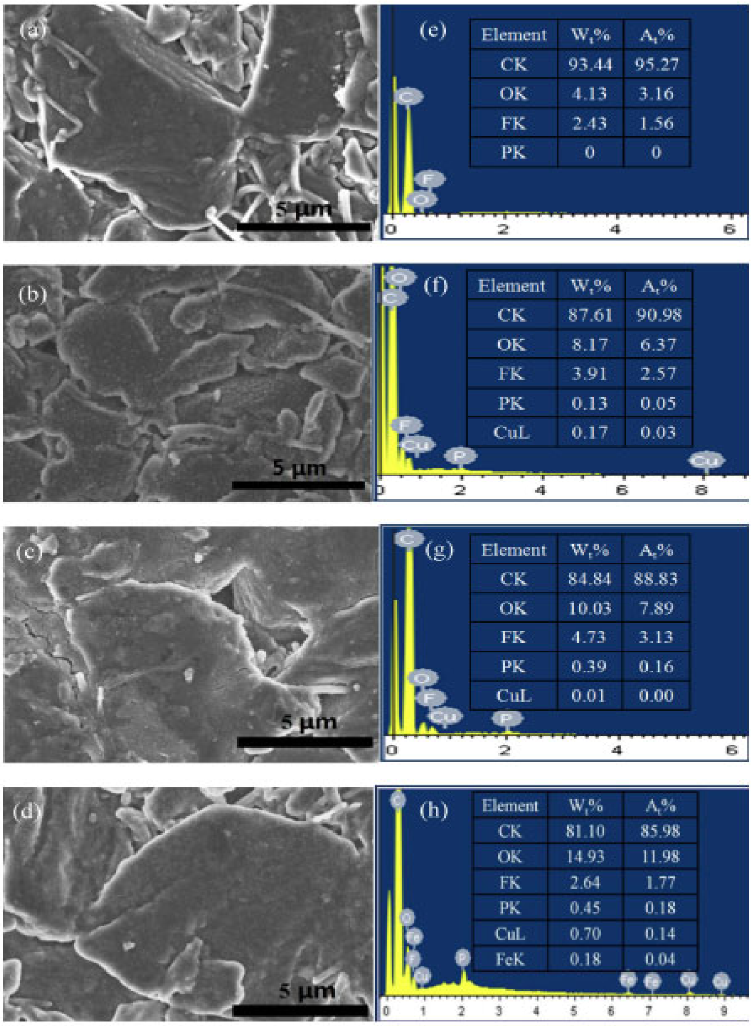


Figure 3. Diagram showing how the battery deteriorates as its capacity decreases [80].

Figure 3 graphically illustrates how the three steps divide the capacitance fading curve. Early in the cycling, there is a noticeable rapid capacity loss for CY0-CC, CY25-CC, and CY25-CC-CV. This is primarily caused by some initial SEI development and anode loss using up the mobile lithium inventory. However, not only are the phase and roughness changes relevant data for certain material breakdown and electrode surface changes, which were used in this article to identify surface electrochemical aging, but the phenomenon of irradiation with X-rays causing powder atomic order can occur due to crystallographic coupling, which is a result of the preferential orientation of crystallite grains. We use X-ray Photoelectron Spectroscopy (XPS) to study the chemical changes occurring at the battery's surface, including solid–electrolyte interphase (SEI) formation. Combined, all these methods result in a detailed understanding of the degradation mechanisms present in Li-ion batteries.

The main objective of LIB degradation research has been to study lithiation mechanisms using different analytical techniques [95]. Some of these techniques encompass X-ray diffraction (XRD) for phase analysis, X-ray absorption spectroscopy (XAS), nuclear magnetic resonance (NMR) for electronic structure analysis, scanning/transmission electron microscopy (S/TEM), and comprehensive XRD refinements for crystal structure defect analysis [96]. Except for S/TEM, the prevailing methods used have been those of "bulk" analysis [97]. Hwang et al. [98] stated that bulk analysis lacks the ability to offer spatial resolution of the phases or electronic structural characteristics observed in LIB materials. Conducting a spatially resolved investigation would enhance the understanding of the components of LIBs and contribute to the progress of knowledge in the field. The examination of deterioration and the usage of spatially resolved techniques have been highly important in improving the understanding of the mechanisms behind lithium battery degradation. The study conducted by Lu et al. [99] utilizing spatially resolved analysis reveals how the structure of the sorbent, particularly the properties of its active site, affects the distribution of components in the aqueous environs of the LIB. However, the application of transmission electron microscopy (TEM) for phase mapping demonstrated that the particles being examined were composed solely of either $LiFePO_4$ or $FePO_4$ [100]. Only a few in-operando experiments have been undertaken to examine the lithiation mechanisms of LiCoPO4 [100]. These studies utilize techniques such as X-ray diffraction (XRD) or operating cells to analyze the degradation behavior of a component in a system. Recently, new approaches have been developed that can assess and describe different mechanisms of LIB [101]. The combination of X-ray computed tomography (XCT) and X-ray diffraction (XRD) allows for a thorough analysis of the distribution of different phases inside the three-dimensional structure of an electrode. Energy-dispersive X-ray spectroscopy (EDS) has been used in combination with cross-sectional scanning electron microscopy (SEM) and XCT to examine chemical changes happening within the electrode [101]. However, XCT has a lower ability to accurately distinguish sub-micron details compared to scanning/transmission electron microscopy (S/TEM) techniques.

Li detection and characterization methods for batteries can be distinguished in many ways. The observational length scale in relation to the cell determines whether Li detection approaches are local or global. Local techniques provide information on specific cell portions, while global methods involve the whole mechanism process. Local methods reveal degraded spatial heterogeneity and cell processes. Global assessments allow cell deterioration to be compared to battery performance. Since ex situ and in situ approaches do not detect Li over time, the cell must reach equilibrium/steadiness. Lastly, dynamic cell information with non-equilibrium components is provided via real-time operando techniques during cell cycling. Because Li is so reactive, ex situ techniques necessitate careful sample handling and preparation to minimize damage and preserve cell state. Cold temperatures may be necessary for ex situ methods like scanning or transmission electron microscopy. In contrast, in situ and operando methods involve modified cell designs but little sample preparation. Finally, Li detection methods can be distinguished by whether they provide qualitative or quantitative cell deterioration data. Quantitative approaches can determine the relative impact of degradation pathways to battery performance locally or globally. Qualitative methods often have a greater spatial resolution and provide a deeper understanding of loss processes and their causes, but they cannot quantify relative magnitudes. Li recognition and classification methods include methods available in most research labs and at user facilities, like X-ray synchrotrons and neutron sources. Laboratory-based methods are accessible and include global electrochemical, chemical, and local imaging methods. However, synchrotron and neutron sources enable multiscale, sophisticated characterizations and research that would take much longer using a laboratory source. Nevertheless, compared to laboratory-based characterizations, such technology is less accessible due to its high cost and limited availability at certain shared user facilities across the globe. Nonetheless, the preparation of TEM samples requires them to be less than 1 µm thick to allow electron passage, which can limit the accuracy of determining the exact location of analyzed particles within the electrode. In contrast, XCT methods do not have this constraint. SEM was utilized to detect any potential alterations in the electrode's morphology following cycling by Zheng et al. [102]. Figure 4a–d display the SEM images of graphite electrodes of LIB prior to and following 600 cycles. Prior to cycling, the graphite particles exhibit clear visibility and are easily distinguishable from one another, as can be seen in Figure 4. During a discharge at a temperature of 25 °C, the aging process causes a slight roughening of the particle faces. After cycling at a high discharge rate of 10 °C, the graphite particles exhibit minimal cohesion and surface cracks are visible. The crack may be attributed to the significant volumetric fluctuations in graphite particles during 10 °C cycling. EDS analysis was conducted on the fresh and cycled graphite anode to determine the deposition of Fe and its elemental composition on the anode surface, as depicted in Figure 4e–h.



Element	W_t%	A_t%
CK	93.44	95.27
OK	4.13	3.16
FK	2.43	1.56
PK	0	0

Element	W_t%	A_t%
CK	87.61	90.98
OK	8.17	6.37
FK	3.91	2.57
PK	0.13	0.05
CuL	0.17	0.03

Element	W_t%	A_t%
CK	84.84	88.83
OK	10.03	7.89
FK	4.73	3.13
PK	0.39	0.16
CuL	0.01	0.00

Element	W_t%	A_t%
CK	81.10	85.98
OK	14.93	11.98
FK	2.64	1.77
PK	0.45	0.18
CuL	0.70	0.14
FeK	0.18	0.04

Figure 4. Graphite electrodes' SEM pictures and EDS analysis prior to and following cycling: fresh graphite in (**a**,**e**) and aged at (**b**,**f**) 1 °C and 25 °C; (**c**,**g**) 10 °C and 25 °C; and (**d**,**h**) 10 °C and 55 °C [102].

A little amount of iron (Fe) deposition was seen on the graphite anode following cycling at a discharge rate of 10 °C and a temperature of 55 °C. Interfacial resistance may have increased as a result of the Fe2+ ions in the solution being reduced at the graphite anode surface and acting as a catalyst during the cycling process [102]. When a cyclic test was performed, it was found that the graphite anode's carbon content decreased while the cycled graphite electrode's levels of phosphorus, fluorine, and oxygen increased noticeably. When the batteries were cycled at a temperature of 55 °C, the behavior became noticeably worse. The main components of the SEI film are acknowledged to be phosphorus, fluorine, and oxygen [102]. The authors revealed that the presence of LiPF6 salt in the electrolyte resulted in the formation of several compounds, including LiF, LixPFy, and LixPOFy, which include fluorine and phosphorus. This occurrence confirms that during cycling, the lithium compounds created by the interface reactions were deposited onto the anode's surface. As a result, the graphite anode degrades, and the composition of the solid SEI film varies. The variations in the structure and composition of the element at the anode likely affect the impedance profiles observed during cycling. Previous investigations [103] have shown that when electrodes are exposed to ambient circumstances, such as air or other atmospheres, it can lead to changes in surface characteristics, valence states, and interfacial interactions.

Batteries are naturally dynamic systems; therefore, studying them outside of their natural environment may lead to the omission of important details about complex structural changes and the intermediate stages of charging and discharging. Zhang et al. [104] completed a study on in situ and operando characterization techniques for solid-state lithium batteries. The study found that metastable phases were present throughout the de-lithiation process of $LiFePO_4$. This was detected using operando XRD [104]. Transitioning towards the utilization of in situ/in operando techniques for characterization is essential to build a relationship between theoretical models and the observations gained using electrochemical techniques. This methodology aims to thoroughly examine dynamic processes with a greater degree of accuracy in comparison to current state-of-the-art methodologies. Many studies investigating the de-lithiation mechanism have used in operando X-ray diffraction as a characterization technique. Other researchers have applied in situ XAS to investigate lithiation mechanisms [105]. Until now, the current in operando studies on the de-lithiation mechanisms of $LiCoPO_4$ have not been able to attain spatial resolution in their findings since they have used bulk techniques, such as XRD. A lack of spatial resolution may result in an insufficient understanding of the underlying mechanism. Nevertheless, there are multiple in operando strategies accessible that facilitate the spatial resolution of phases while undergoing the charging and discharging process. Table 1 displays some of the studies performed on LIB using different characterization techniques.

Table 1. Lithium-ion battery degradation and characterization.

Degradation Mode	Battery Component	Test Method	Results	Ref.
Volume shrinkage	Anode	Cryo-atom probe tomography	Showed evidence of Si anode corrosion from the decomposition of the Li salt.	[106]
Solid–electrolyte interphase	Anode	SEM technique	Observed the formation of species such as ROCO2Li and CO2OLi on the anode surface.	[49]
Crack formation	Anode	X-ray tomography	Discovered irreversible electrode deformation, fracture initiation and development along preexisting defects.	[107]
Solid–electrolyte interphase (SEI)	Anode	Scanning electron microscopy (SEM)	Revealed the buildup of SEI on the surface of the graphite anode.	[108]
Solid–electrolyte interphase (SEI)	Anode	X-ray computed tomography with nanoscale resolution (nano-CT)	Demonstrated the SEI buildup inside the pore structure of the anode.	[108]
Formation of the rock salt phase	Cathode	Combined XRD and TEM	The cutoff voltage conditions determine the type of irreversible transformation observed in the surface lattice structures of $LiNi_{0.5}Co_{0.2}Mn_{0.3}O_2$	[109]
Formation and dissolution of lithium mossy formations	Electrolyte	Optical imaging	The findings indicate the presence of two distinct growth modes with a typical non-tip growth resulting from lithium metal insertion into the metallic moss backbone.	[110]
Crack and void formation	Cathode	Analytical transmission electron microscopy	Cracks and voids are produced by repeated volumetric change, which is linked to a decline in electrochemical performance.	[111]

Table 1. *Cont.*

Degradation Mode	Battery Component	Test Method	Results	Ref.
Temperature	Electrolyte	Quantitative NMR spectroscopy	Degradation is caused by the electrolyte components' chemical instability against water, and rising temperatures only hasten the effects of water on degradation.	[112]
Solid–electrolyte interphase (SEI)	Anode	Non-destructive in situ nuclear magnetic resonance (NMR)	Understood the corrosion behavior of the Li metal.	[113]
	Cathode	Non-destructive electron paramagnetic resonance imaging (EPRI)	Discovered that the evolution of the inactive metallic Li in $LiCoO_2$/anode-free pouch cells was influenced by the regularity of Li plating.	[114]
Temperature	Battery cell	Infrared thermography	Showed the quantitative analysis of heat generated in battery cells.	[115]
Corrosion of material	Anode	Non-destructive in situ nuclear magnetic resonance (NMR)	Understood the corrosion behavior of the Li metal.	[113]
Li plating	Cell	Non-destructive electron paramagnetic resonance imaging (EPRI)	Discovered that the evolution of the inactive metallic Li in $LiCoO_2$/anode-free pouch cells was influenced by the regularity of Li plating.	[114]

Although extensive research has been conducted to understand the degradation mechanisms of the components of lithium-ion batteries (LIBs), it is widely acknowledged that the surface coating of battery materials, particularly the solid–electrolyte interphase (S/CEI) layer, is vulnerable to changes in the atmosphere that can occur when the battery is removed from the cell and exposed to air. Hence, it would be beneficial to perform an in situ or in operando analysis of the S/CEI layer to comprehend the process of degradation. The expansion of the SEI has previously been documented by the application of a sealed liquid cell in in situ TEM, as well as a liquid cell in in situ SEM. However, there are various specific difficulties related to each method in terms of gathering data, setting up an in situ or in operando cell analysis, and establishing a connection between the produced outcomes. Having a system that can monitor changes in both the material and the electrolytes at high voltages would be beneficial. This is because high-voltage battery materials, such as $LiCoPO_4$, undergo severe deterioration.

5. Sustainability of Lithium-Ion Batteries

Although fossil fuels are limited, renewable sources such as sunlight, wind, and hydropower offer more sustainable alternatives in the long term. The need for efficient energy systems underscores the complex relationship between energy use and sustainability. Inefficient practices and operations that harm the environment pose a threat to long-term planetary sustainability, highlighting the importance of efficiency improvements to mitigate environmental impact and increase the lifespan of energy storage and source components [116]. As electric vehicles become more prevalent, improving energy storage systems, like LIBs, is essential for reducing fossil fuel dependence and curbing greenhouse gas emissions. However, the sustainability challenges of LIBs span their entire life cycle, from raw material extraction and battery manufacturing to use in vehicles and end-of-life management [117]. Recent research has emphasized concerns regarding the sustainability of graphite, a key material in LIBs, due to geopolitical and supply chain risks [118]. The global reliance on graphite from politically unstable regions has raised concerns about resource availability and price volatility, and ensuring long-term stability in cobalt supply requires diversification of the supply chain and increased recycling efforts [119]. Innovations in LIB materials and manufacturing aim to reduce the environmental and social impacts of raw material extraction while enhancing battery performance in terms of energy density and lifespan [120]. These advancements could significantly decrease the electricity consumption and environmental impact during the battery's operational life. However, even with improved lifespans, LIBs will eventually require management after their initial use in different applications. The recycling and reuse of end-of-life LIBs offer promising solutions to retain material and energy value. Circular economy principles can be explored to manage LIB waste effectively through direct reuse, cascading reuse, material recovery, and minimized disposal [121]. Cascading reuse in applications like electric vehicle charging stations or utility-scale storage has shown significant potential for reducing waste and environmental impacts [122]. Despite the potential for circular economy solutions, there are still challenges, such as variability in battery life after vehicle use and the need for rapid testing and remanufacturing techniques [123]. While battery recycling is mandated in some regions, reuse is not always clearly addressed in policy. To ensure the success of reuse systems, both regulators and manufacturers must address issues like liability and safety, and evaluate the entire life cycle to avoid unintended consequences [124]. Further advancements in the battery life cycle, particularly in the development of high-performance electrode materials, electrolyte, sustainable battery designs, and system-level assessments of energy storage technologies, are necessary for the sustainability of lithium-ion battery components and the overall performance of batteries. These ongoing contributions will be essential for realizing the full sustainability potential of energy storage technologies and advancing global efforts toward cleaner, more efficient energy systems.

6. Conclusions

This study has provided an analysis of the various components of the lithium-ion battery and the current degradation mechanisms that exist. It is important to understand that repeated charging and discharging of LiBs frequently results in a significant decline in the batteries' capacity to store energy, which restricts their potential for commercial success. Both internal structural deterioration inside the electrode particles and undesirable electrochemical integration between the electrolyte and the electrode at their contact point can cause the electrodes of lithium-ion batteries to degrade. The deterioration process is expedited by undesired reactions originating from the electrolyte, especially if unstable phases are formed in close proximity to the interface between the electrode and electrolyte. A thorough grasp of the underlying degradation mechanisms is necessary to enhance the electrochemical characteristics of electrodes and create efficient mitigation strategies. This paper presents a comprehensive examination of electrode degradation and the approaches employed to comprehend the processes of degradation. This study provides a thorough evaluation of the innovative techniques in electron and ion microscopy that have been developed to examine the deterioration processes taking place on the electrode surface and within individual primary particles.

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