Electrical Properties of Lithium-Ion Batteries

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Lithium-ion batteries have good performance and environmentally friendly characteristics, so they have great potential. However, lithium-ion batteries will age to varying degrees during use, and the process is irreversible. There are many aging mechanisms of lithium batteries. In order to better verify the internal changes of lithium batteries when they are aging, post-mortem analysis has been greatly developed. The state of health (SOH) of Li-ion batteries is usually assessed using two main aspects: the loss of capacity and the increase in impedance. In addition, different kinds of lithium-ion cells can be divided into full cells and half cells. Coin cells are a kind of represented half cells, which can be investigated by a coin cell tester, which are carried out in a climate chamber. Coin cells can also build from full cells to investigate the electrical performance of independent electrodes. The electrodes from full cells could be thoroughly rinsed three times with diethyl carbonate (DEC) to remove the deposition of the electrolyte and Li salt and then be pouched into a specific area to use as the positive electrode as the coin cell.

lithium-ion batteries state-of-health Electrical Properties

1. Introduction

With the rapid development of green energy solutions, such as electric vehicles (EVs) and renewable power generation, lithium-ion batteries (LIBs) attract a lot of interest these days. Lithium chemistry offers high electrode potential (−3.04 vs. standard hydrogen electrode), low molecular weight and small ionic and atomic radii, ensuring fast diffusion and possibility of intercalation or integration into crystalline lattice of various materials. This helped Li-based batteries to outperform other secondary batteries both in terms of highest power and energy density as well as substantial lifetime [1]. However, uncertainties related to cost, degradation and weak performance in extreme weather conditions slow down large-scale adoption of Li-batteries. In particular, to meet the requirements in electrical vehicle (EV) applications, Li-ion batteries need to exhibit large capacity, stable thermal properties and a long lifetime [2][3][4][5][6][7]. Thus, detailed understanding of Li-ion batteries degradation behavior and mechanisms becomes a focal point of current battery research.

The aging process of a battery can be monitored by performing charging and discharging cycles while measuring the electrochemical characteristics, e.g., impedance and capacity [8][9][10][11][12][13].

For years, the internal physical and chemical reactions of Li-ion batteries were investigated [14]. As accurate and advanced electrochemical analysis devices were invented, people gained a better understanding of the aging processes of different materials and structures the batteries were composed of. To quantify the battery health, the most common indicator or concept, used in the literature, is State-of-Health (SOH). The SOH indicates the specific performance and health status of a battery, at a certain point, compared to the pristine state of the same battery [15]. Although there is no clear definition of the SOH, different parameters of a battery can be used to describe the SOH, such as capacity and impedance, corresponding to the battery's energy and power, respectively [16]. Then, the diagnosis and estimation of Battery SOH can be promoted to determine battery characteristic parameters. The monitoring gives a measure of SOH which decreases as the battery ages. The decrease in SOH cannot commonly be attributed to a particular physical or chemical mechanism occurring inside the battery, but rather a complicated interplay of various mechanisms [7][17][18][19][20].

With the development of the interdisciplinary approach to battery ageing, in addition to the traditional electrical properties curve-based method, post-mortem analysis has attracted a wide interest for battery SOH estimation and/or diagnostics. With these methods, the battery lifetime and SOH can be deduced from the morphology and composition changes observed in a disassembled cell. This approach can provide understanding of the ageing processes based on the observed chemical and physical changes and link them to changes in electrical characteristics. While several recent research have addressed the electrical diagnostics methods, a detailed research of post-mortem analysis methods is missing [21][22].

2. Open-Circuit-Voltage (OCV) Curve-Based Analysis

As the voltage of Li-ion batteries is mainly influenced by internal electrochemical reactions and also changes in a variety of aging conditions, many people are trying to employ health indicator (HI) to describe SOH. Thus, much research on SOH estimation is focused on appropriate HIs. In general, two models of the terminal voltage can be established by OCV and polarization voltage, reflecting thermodynamic and kinetic aspects. For OCV, this means measuring the terminal voltage while the battery is in the condition of an open circuit until it reaches the thermodynamic equilibrium state. In addition, the polarization refers to the deviation of the terminal voltage from the OCV. The OCV curve-based methods included two main parts, geometric features and electrical characteristics. It is essential to employ OCV to build a Li-ion batteries voltage model [21][23][24][25][26]. The geometric features-based method is a common way of the diagnostic methods, which can be used to detect aging mechanisms by focusing on OCV variation to realize the estimation of SOH. There are two main aging mechanism identification methods, incremental capacity analysis (ICA) and differential voltage analysis (DVA).

According to many studies, ICA has been employed to diagnose the battery aging mechanisms [10][27][28][29][30][31][32]. The incremental capacity (IC) curve is calculated by derivation of the voltage curve, which can reflect the electrochemical process in the charging procedure [33][34][35][36][37]. The analysis of the mechanism of battery aging is carried out by the position and shape of the peaks. For example, Yang et al. used IC curves to present the aging mechanisms.

The DVA method is also commonly applied in the aging mechanism identification [6][38][39]. Ira et al. found that lithium-capacity-consuming side reactions were mainly occurring at anodes [23].

For coin cells, OCV test are similar to the full cell [34]. Alexander et al. extracted coin cells from commercial Li-ion cells [39]. As the influence of the reassembly of the coin cells was not investigated clearly, they developed a method to compare the different parameters such as washing the electrode or changing the ratio of the electrolyte, which influenced the performance [40][41][42][43][44]. They used a BaSyTec Cell System to realize the formation and the cycling of coin cells. The cycling method of the cells are constant-current-constant-voltage (CCCV) in discharge and constant-current (CC) in discharge process.

Another study by the same group attempted to relate the characteristic features of interest (FOI) to the evolution of the IC curve and the mechanism of aging [45]. The result also proved that the principle of aging is the same between full cell and coin cell, which attributed to the original cells and the corresponding coin cells exhibited LLI at room temperature while an increasing trend of active materials loss at high temperatures. The three FOIs are studied for the IC analysis in coin cells. The general trend of the FOIs by the separated aging mechanisms was also discussed by Berecibar et al. [46]. They studied all the curves separately and considered independent parameters and combinations of different parameters. According to their results, LLI and LAM in negative and positive electrodes are exhibited, respectively.

3. Electrochemical Impedance Spectrum (EIS) Analysis

Electrochemical Impendence Spectrum (EIS) is a useful method to measure the degradation level and the state-of-health without opening the batteries. The EIS is applied to the open circuit as well as the stable DC polarization electrochemical system.

The EIS result is usually described by Nyquist plot, which involves the imaginary part (y axis) and the real part (x axis) of the impedance of the cells. The internal impedance of a cell is usually defined as a turning point at the low frequency area of EIS in a battery management system [13][47][48]. Fitting the Nyquist plot to the LIB equivalent circuit model allows for the modelling of battery dynamics [49][50][51][52]. R_{ohm} stands for the voltage drops because of the conductivity dropping during aging, such as current collector, binder, electrode and electrolyte electronic particles. R_f stands for R_{flim}, which refers to passivating film resistance. The process of charge transfer and conduction through the passivation layer should be considered as two sub-circuits consisting of pure resistors and pure capacitor elements in parallel.

Daniel et al. investigated the amplitude alternating current impedance which was measured at the voltage of 3.72 V. They found that both cathode and anode exhibit capacitive tail frequencies which are higher than 10 kHz, middle frequencies (from 1 kHz to 1 Hz) show a semi-circular arc and a diffusion tail at low frequencies lower than 1 Hz [33]. According to their study, the tail at high frequencies area is the result from the interaction between the impedance analyzer and the electrochemical cell. The depression of the semicircle in the mid frequency range may be due to simultaneous electrochemical reactions on active particles inside the cells.

For coin cell, EIS has proven to be a strong technique for analyzing phenomena in Li-ion battery. EIS spectroscopy in principle allows the separation and quantification of several achievements to the whole cell impedance, such as charge transfer, diffusion, and electrolyte resistance at the solid/electrolyte interface. To separate the influence from anode and cathode, respectively, the reference electrode (RE) is introduced into the cell system, and the impedance is measured between the working electrode (WE) and RE, as well as between the counter electrode (CE) and RE. Locating the RE is not easy because the EIS spectrum is distorted for a particular three-electrode battery configuration and useful analysis of the data is difficult [53][54]. Two models were developed to simulate EIS spectra by Delacourt et al., the 2D mathematical model and the equivalent circuit model of button cell based on the finite-element method (FEM) [53][55]. Most of the simulations were performed using the FEM model, which is easier to be realized. The purpose of the equivalent circuit is to simply elucidate the origin of the impedance distortion effect, while preserving the basic characteristics of the 2D model.

4. Electrochemical Parameter-Based Methods

The electrochemical methods to investigate lithium-ion batteries are mainly divided into the following parameters: the intercalation rate of lithium electrode, the diffusion coefficient in the solid or liquid phase, and the change of resistance in the formation process of solid electrolyte interphase layers (SEI) and so on. [21][22][27][28][56].

Rujian et al. observed that the side reactions and deposits appeared not only on the SEI surface but also between the anode and separator [28]. On the basis of their investigations, they proposed a model to represent Li-ion cells degradation by using the aspects as the following: the volume fraction of the active anode accessible; the resistance of the SEI layers and the deposition layer; the diffusion coefficient of the electrolyte. These characteristic parameters are influenced by real experiments. They concluded that the degradation was mainly caused by the deposition of anode side reaction via different methods including SEM, XRD and XPS.

5. In Situ Operando Characterization

The degradation of battery performance is caused by both external factors (such as temperature, stress, and charging-discharging method) and internal factors (such as loss of lithium-ion, loss of active materials, and decomposition of electrolyte). Most of the current SOH estimation methods treat batteries as a black box for empirical fitting, overlooking the actual electrochemical meaning of the parameters. Therefore, an accurate aging evaluation algorithm, i.e., in situ operando characterization, must be based on comprehensive understanding of the internal aging mechanisms of Lithium-ion batteries.

Laisuo et al. have employed in operando energy dispersive X-ray diffraction (ED-XRD) measurements to monitor the evolution of $LiCoO_2$ crystal structure during cycling [57]. The measurement process is illustrated. The first charge-discharge cycle of the pristine and Poly (3,4-ethylenedioxythiophene) (PEDOT)-coated $LiCoO_2$ electrode, and the lower intensity is represented by blue, and the higher intensity is red. It is shown that the structural evolutions are different between the two electrodes during cycling, which in turn confirms that ED-XRD is a powerful technique to investigate the mechanical behavior and the structure change of battery electrodes during cycling, and it can better explain the capacity fading caused by changes in the internal structure during cycling without destructive methods.

Operando transmission X-ray microscopy (TXM) has also been employed to visualize the morphological evolution of the electrodes. Laisuo et al. monitored $LiCoO_2$ electrodes during the over-lithiation test [58]. The images corresponding to five representative points on the curve are displayed as P1–P5. It is shown that during the over-lithiation, $LiCoO_2$ particles and agglomerates are broken and eventually pulverized. Overall, larger particles showed stronger resistance to cracking, remained intact during most of the charging process and only started to crack at the end of the over-lithiation process. On the other hand, small particles fractured early. This example shows how in situ techniques help to improve understanding of the electro-chemo-mechanical behavior of electrodes during cycling and provides an easy way to estimate the SOH of lithium-ion batteries without destroying the batteries.

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