

Article

Electrochemical Impedance Spectroscopy Investigation on the Charge–Discharge Cycle Life Performance of Lithium-Ion Batteries

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Abstract: In this work, we employed an electrochemical impedance spectroscopy analysis of commercial Li-ion Panasonic NCR18650B cells in order to monitor their cycle life performance and the influence of the C-rate on the charge/discharge processes. By applying a fast charge rate of 1.5 C, we investigated their speed degradation within three distinct discharge rates, namely, 0.5 C, 1 C, and 1.5 C. In our first approach, we assessed the dynamics of the lithium-ion transport processes, as well as their dependence on discharge rates, with the aim of understanding how their performance correlates with usage conditions. We observed that, as the discharge current increases while the number of cycles decreases, the ohmic resistance in the aged state reduces. Moreover, the charge transfer resistance is not affected by the discharge current, as the values are inversely proportional to the current rate, but mostly by the number of cycles. By performing a state of health analysis of Li-ion batteries with different C-rates until they were completely discharged, we offer a clear indication of how much of the battery's lifetime available energy was consumed and how much was left, anticipating further issues or when the battery needed replacing. Starting at 60% state of health, the battery degradation has a steeper increase at 0.5 C and 1 C, respectively, while for a deep 1.5 C discharge, it only increases when the battery charge rate can no longer be sustained. Finally, the resonance frequency results highlight a fast increase toward the end of life for 0.5 C and 1 C, which is directly correlated with the above results, as a potentiostatic electrochemical impedance spectroscopy sequence was applied every fourth charge/discharge cycle. When applied at 1.5 C, the linear trend is much more pronounced, similar to the state of health results.



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Keywords: electrochemical impedance spectroscopy; lithium-ion cell cycle life performance; state of health cycling evolution; resonance frequency; C-rate influence

1. Introduction

The high use of lithium-ion cells (LIBs) in various mobile equipment [1] and, in particular, in the development and commercial implementation of electrical cars [2,3] has made this technology progress rapidly in recent years, becoming the predominant choice in terms of rechargeable batteries. Besides their intense use for consumer electronics and electrical cars, LIBs have also been employed intensively in energy storage systems [4], grid services [5,6], UPS systems [7], electrified aviation [8,9], and even in tailored designed batteries for aerospace [10] or military applications [11]. In comparison with similar rechargeable technologies, such as nickel–cadmium, nickel–metal–hydride, or lead–acid types, lithium-ion cells (LIBs) have some particular advantages. They are capable of employing the highest energy density and can deliver up to 3.6 V, making them suitable

for high-power applications [12–15]. Corroborated with a low self-discharge rate [16] and low maintenance needs [17], LIBs are capable of a very long lifespan, which makes them currently the optimum choice for cleaning and/or decarbonizing our current economy's energy use.

Nowadays, on the market, a large range of distinct designs of LIBs are present, with different features related to lifetime performance, cost, and safety. They are mainly based on distinct anode or cathode materials, electrolytes, and separators. Starting from the first Li cells, which were mainly based on metallic lithium as the anode and lithium–metal insertion oxide as the cathode, new substitutes for the components of the conventional lithium-ion battery are continuously being developed as the issue of dendrite growth causes significant issues in terms of reliability or safety use [18]. New anode materials containing graphite intercalation electrodes have overcome this problem [19], and more recently, new anode materials have been added, for example, hard carbon [20], lithium–titanate [21], and silicon [22]. With regard to cathode cell developments, various metal oxides or phosphates, blends, and doped materials have been implemented [23,24]. Likewise, regarding anodes and cathodes, new liquid- [25] or polymer-type electrolytes [26], additives [27], binders, and separators have been innovated.

Their particular properties, which we refer to here as high voltage [28], high-power energy density [29], and/or low self-discharge rate [30], contribute to a much longer battery lifespan performance [31,32] when compared to other alternative technology (e.g., lead–acid batteries [33]). Beyond lead–acid batteries' intense use in the last 100 years, it is time to change the paradigm and provide a sustainable future for electric cars and large-scale renewable energies [34,35]. As such, the battery industry is required to address the present energy problem and should be capable of offering a viable solution for renewable energy sources with intermittency, which can stabilize the output of solar and wind power, increase their share in power generation, and ease their integration into the electric grid [36]. Adding the environmental constraints till 2035 in Europe, we are strongly pushed nowadays to solve the current major issues, including global warming, fossil-fuel depletion, and atmospheric pollution [37]. Technological engineering and the environmental entanglement of lithium batteries clearly have the potential to innovate and improve our future [38].

Despite the continuous development of LIBs, significant research disputes are present, seeking to gain a deep understanding of their degradation processes. These mechanisms are responsible for capacity and power fade in LIBs [39]; thus, a major focus of researchers is to develop more accurate examination techniques to be applied to LIBs. In this view, several modeling approaches have been developed in recent years, which are crucial to the continuous optimization of battery performance and/or lifespan. With regard to the state of charge (SoC) estimation techniques, we mention here Coulomb counting [40], open-circuit voltage (OCV) [41], Kalman filtering [42], and machine learning approaches [43], while for state of health (SoH) prediction, some of the current techniques are capacity fade tracking [44], data-driven models [45], artificial neural networks (ANNs) [46], electrochemical impedance spectroscopy (EIS) [47], and modeling through equivalent circuit models (ECMs) [48]. With the last two techniques, in particular, it is possible to highlight some important operating features inside lithium cells by describing the variable factors of the Randles equivalent model [49]. As such, EIS is capable of offering important knowledge about the power delivery capabilities in LIBs [50,51]. Through a single experiment, we can divide and quantify, in detail, the characteristic shapes of impedance plots in LIBs from very high-frequency domains, corresponding to inductor and/or cell resistance of the bulk, to high frequencies, describing the interface layer and/or the charge transfer reactions, or to low frequencies, describing the diffusion processes [52].

In these perspectives, we have applied an EIS analysis on three commercial Panasonic NCR18650B (Panasonic Energy Co., Ltd., Osaka, Japan) cells in order to investigate their degradation speed depending on distinct discharge rates. By keeping a fast charge rate of 1.5 C for all batteries, we have monitored their usage condition depending on three distinct discharge rates, namely 0.5 C, 1 C, and 1.5 C. The goal was to investigate their cycle life performance and the influence of C-rate on the charge/discharge processes, taking into account that, during usage or storage, batteries suffer from both energy and power fade. Moreover, by varying the cell qualities, we tried to analyze not only the performance degradation but also the safety of battery-powered products. Thus, the dynamics of the lithium transport process has been assessed, as well as its dependence on charge rate with the aim of understanding how their performance correlates with usage conditions. Qualitative information on the physical/chemical properties of the LIBs was provided, increasing our understanding on their lifetime prediction (or longevity) and/or aging. We also furnished some strong references for LIB management depending on their charge/discharge rates, taking into account that the monitoring of their charge and discharge is critical for ensuring their longevity and/or safety. Nevertheless, this investigation was not an easy task, due to the complex degradation mechanisms in LIBs, which are not completely elucidated. We refer here to the strong dependence between the performance parameters and the operating condition, that usually lead to a non-linear behavior.

2. Experimental Section

2.1. Li-Ion Cells

We have used three commercial Panasonic cells, type NCR18650B, acquired from the market. This particular choice is mainly due to the fact that this particular cell model was employed in previous TESLA Model S electrical vehicle, and thus has attracted tremendous attention within recent years [53,54]. Nowadays, they are used on a large scale in e-bike accumulators, notebook PCs, boosters, portable devices, etc. [55]. Being used in so many applications, an analysis of their behavior, when distinct fast charge/discharge rates are applied, is highly needed. In the same register, Panasonic is one of the world's largest battery manufacturers, and this particular rechargeable cell type is used mainly in packages and/or devices with built-in PCM/BMS electronic security. This means that the sold battery does not have electronic protection against overcharging and deep discharge, that allowed us to play easily with fast charge/discharge rates. Thus, we set fast charging at 1.5 C as a compromise between the irreversible side reactions and battery performance. Additionally, 0.5 C is the standard charging rate, for which the battery behavior is given in the manufacturer specification sheet.

The standard charging current for NCR18650B is typically 0.5 C, which is around 1.6 A (since $0.5\text{ C} \times 3.25\text{ mAh} = 1.625\text{ mA}$), which means that this is the optimal current for maintaining long-term health and cycle life of the cell. As mentioned before, NCR18650B can technically handle higher charge currents (up to 1.5 C in this study), thus the goal was to analyze how fast charge rates of 1.5 C, corroborated with different discharge rates, can lead to their degradation over time. We are fully aware that charging at 1.5 C will shorten the cycle life, and going beyond that, can risk overheating and lithium plating, especially if the battery is not equipped with adequate thermal management systems. Nevertheless, our goal was to find rapid and predictable approaches for determining their degradation speed over time and not to characterize the internal processes during the aging process.

The battery characteristics are summed up in Table 1.

Table 1. NCR18650B battery characteristics.

Rated Capacity at 25 °C	3200 mAh
Nominal Capacity at 25 °C	3250 mAh
Nominal Voltage	3.6 V
Internal Resistance	~50 mΩ
Charging Method	CC-CV
Charging Current	1625 mA
Charging Voltage	4.2 V
Charging Time	2 h
Cathode Material	Nickel Oxide

2.2. Experimental Set-Up

All the impedance measurements were performed by a VSP multichannel potentiostat from BioLogic [56] with an attached 100 A booster. The device booster uses the standard 1.5 m cables provided with the instrument. The controlled/measured potential and controlled and/or measured current were provided by a standard 1.5 m standard cable with Ref1/Ref2 and CA1/CA2, respectively, provided by the manufacturer and connected to the battery holder with crocodile clips. All measurements used the same set-up.

The results processing and interpretation were performed through the integrated EC-LAB software V11.46. Potentiostatic electrochemical impedance spectroscopy (PEIS) methodology was applied to LIBs cells in order to accurately monitor the frequency-dependent impedance. By this technique, the potentiostat will monitor and register the impedance data throughout an applied alternating current (AC) signal and then measure the resulting voltage response to gain insight into the system's impedance. As is known, impedance represents the resistance to the current flow but also includes the capacitive and inductive elements. This reveals important information about transport processes like charge transfer, diffusion, and double-layer capacitance.

During the initial capacity measurement, the CC-CV charge method was used with a 0.5 C charge rate up to a 4.2 V limit followed by a constant 4.2 V limited by 0.02 C. The discharge rate was set to 1 C and limited by a 2.5 lower voltage limit. During the fast-charging aging tests, the charging current was set to 4.875 A (1.5 C) with a voltage limit of 4.2 V followed by a constant voltage of 4.2 V with a cut-off current of 0.2 A to avoid overcharging during PEIS. We tested the discharge currents at 0.5 C, 1 C, and 1.5 C, respectively, with a 2.5 V lower voltage limit. Finally, it should be mentioned that all experiments were performed in a controlled room, maintained at 21 °C.

2.3. Electrochemical Impedance Spectroscopy (EIS)

EIS has been applied to carry out a detailed examination of the physical/chemical processes that occur inside the battery cell at both electrodes. In this method, a current (sinusoidal signal) of a certain amplitude and frequency was applied to the electrochemical cell, and then we measured the response, namely the phase shift and amplitude of the output current. The impedance has been calculated from the sinusoidal excitation at different frequencies as a response of the system to this perturbation. After a certain repetition of this procedure for a number of frequencies, the impedance spectrum was acquired, plotted as a Nyquist diagram, in which the horizontal axis reveals the real Ohm

impedance, while the vertical axis represents the imaginary impedance. The impedance at each frequency will highlight a distinct performance, encompassed in the shape of a so-called Nyquist diagram. Thus, our use was to generate frequency response and Nyquist plots, which were further fitted to a so-called equivalent Randles circuit.

Based on the various developments of battery cell models, we were able to associate the circuit elements with the processes that occur in direct dependence with the grid electrolyte interface changes, allowing us to obtain important qualitative information on the impedance behavior in each lithium-ion battery at different discharge rates. In Figure 1, the Randles equivalent circuits used to fit the PEIS are shown, together with an example of an explained Nyquist plot contour. As can be observed, we divided the scanned cell results supplied by the Nyquist plot and the equivalent Randles circuit into three distinct areas, namely: migration, charge transfer, and diffusion. The inductor (L2) and resistor (R2) elements are related to a very-high-frequency domain and are directly involved at the intersection $\text{Im}(Z) = 0$, between the impedance spectrum and real axis. In particular, L2 is limited to the part of the spectrum with positive $\text{Im}(Z)$, being mostly related to current collectors and cables, while R1 represents the resistance of the electrolyte [57,58].

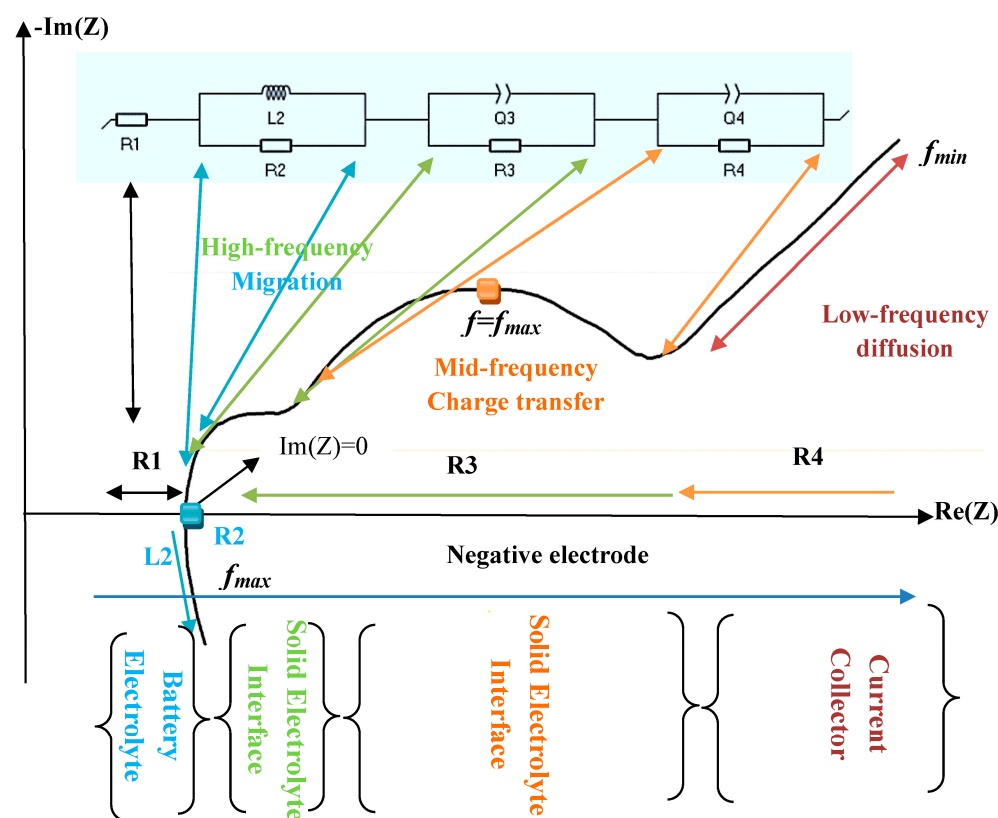


Figure 1. The equivalent Randles circuit (top); Nyquist plot for Li-ion cells (middle); Divided half-battery, describing the internal reactions allocated to each domain (bottom).

The migration mechanisms (on the left) which appear in high-frequency domains provide the resistive attributes of the cells in the presence of the solid electrolyte interface (SEI) and are mainly emphasized by R3-resistor and the constant phase element (CPE)-Q3 (expressed in Figure 2 as a small semicircle). This first semicircle is typically assigned to Li^+ transport through SEI [58]. By decreasing the frequency, the most important processes of charge transfer and battery kinetics of the double layer capacitance, shown as a large semicircle (in the middle), are represented by R4-resistor and CPE-Q4 (expressed in Figure 2 as the second, larger semicircle). The second broad semicircle, according to the literature, can actually encompass two mostly overlapped semicircles, and this is due to anodic and

cathodic charge transfer [58,59]. In this regard, in the ultimate frequencies domain, the low-frequencies (on the right) zone is attributed to the diffusion mechanisms that take place inside the Li-ion battery and is usually fitted by the Warburg element [59].

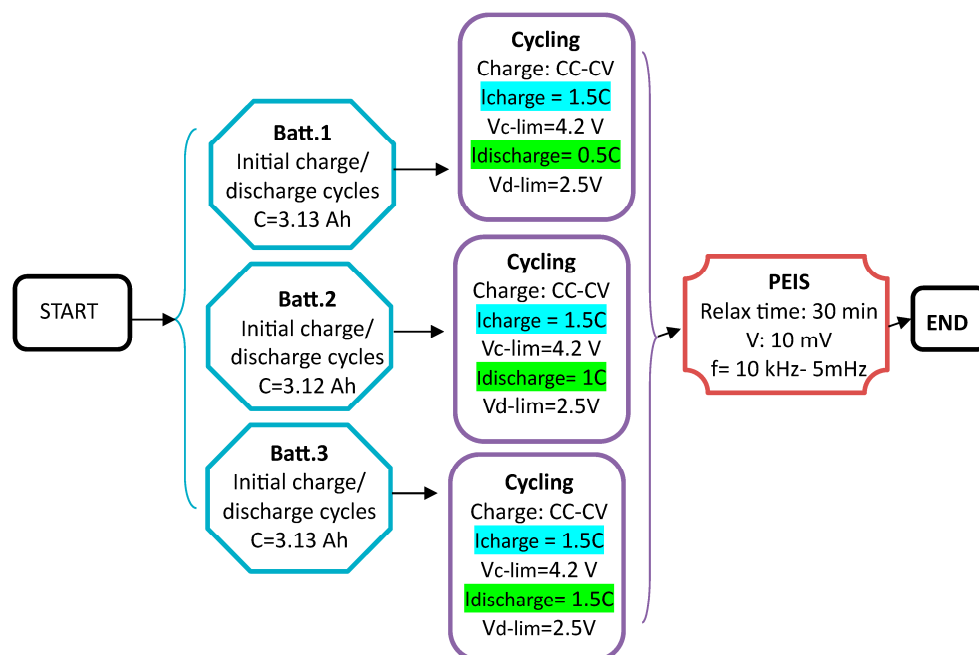


Figure 2. Aging protocol used for Li-ion batteries.

2.4. Aging Protocol

In a first approach, we have cycled each battery four times in order to measure their initial capacity, taking into account that, due to their complex chemical and/or mechanical structure, battery cells can never be fabricated with constant quality [60]. The initial capacity of all three batteries had close values, around 3.12 C.

We have performed EIS measurements by employing the PEIS technique, implemented throughout the Modulo Bat (MB) scheme. In this approach, the sinus signal was applied just around the DC potential, close to the equilibrium condition. The potential was set to 10 mV, within a frequency range of 5 mHz–10 kHz. Moreover, the sinus amplitude has been set to 20 mV, using two measurements per frequency for registering. At the beginning of each PEIS analysis, 30 min of tranquility was employed, as elsewhere [61]. It should be mentioned that we have performed two PEIS analyses on our lithium cells: one at SoC 100% fully charged and one in a completely discharged condition. In the diagram above, we have summarized the aging steps of the cells (see Figure 2).

The charge current for all the experiments has been fixed at 1.5 C, while the discharge rates were as follows: 0.5 C for Batt.1, 1 C for Batt.2, and 1.5 C for Batt.3. However, if the discharge current is too strong, it will rapidly compromise the battery performance, as we will see in the next sections. In the same register, we have limited the current during the charging to 200 mA, because of the possibility of overcharging, which will basically cause the device to stop due to safety conditions or to avoid any possible harm. Due to the large number of cycling steps, we have employed PEIS every fourth charge/discharge cycle. The aging procedure finished when the lithium cell was not able to sustain the charging, 1.5 C in this case. Figure 2 schematically shows the whole aging process.

3. Results

3.1. EIS Analysis

Taking into account the much more complex behavior of the circuit elements, electrochemical impedance spectroscopy measurements were applied to the Li-ion batteries by using a small excitation signal (in our case 10 mV), expressed as:

$$E_t = E_0 \sin(\omega t) \quad (1)$$

where E_t represents the potential at the time t , E_0 is the signal amplitude, and ω is the radial frequency. By encompassing the radial frequency and the frequency (Hz), we obtain the following equation:

$$\omega = 2\pi f \quad (2)$$

In a linear system, the current response (I_t) to a sinusoidal potential will highlight a sinusoid at the same frequency but, with a phase shift (ϕ), we have:

$$I_t = I_0 \sin(\omega t + \phi) \quad (3)$$

This allows us to calculate the impedance of the system in terms of magnitude Z_0 and the phase shift (ϕ) by using the following formula:

$$Z = \frac{E_t}{I_t} = \frac{E_0 \sin(\omega t)}{I_0 \sin(\omega t + \phi)} = Z_0 \frac{\sin(\omega t)}{\sin(\omega t + \phi)} \quad (4)$$

Going further, Nyquist plots were used for highlighting the impedance spectrum of the Li-ion batteries, in which the imaginary part of the impedance (presented with a negative sign) is plotted against the real part. In Figure 3, the results for our LIB cells, in both charge and discharge states, are shown. We used different colors in order to exhibit the impedance spectra evolution, which is directly connected with the battery's internal reactions. From left to right, their performance reduction (due to usage in time) is shown in a flowing manner, as already demonstrated in a recent work [62].

We have largely described these physical–chemical processes in different frequency domains, please see Section 2.3. In analogy with the Nyquist plot shown as an example in Figure 1, we can also split the diagrams into three major areas, from the high-frequency domain (migration) to the mid-frequency domain (charge transfer) and low-frequency domain (diffusion), within their corresponding parameters of fitted equivalent Randles circuits, thus allocating their influence in the aging process during the cycling. These elements of the battery model have a direct correlation with the LIB real-time performance, and after processing, potentially, with their remaining lifetime.

In Figure 3, all the measured Nyquist plots in both states of charge and discharge are shown, encompassing all impedance ranges at different frequencies, and there is a plot agglomeration in which it is quite difficult to visualize the difference in the reaction resistance between a new (pristine) and a degraded battery cell. Due to the many PEIS curves, there appears a semicircle in the Q3 region (see Figures 2 and 4), which is unexpected for new commercial cells. This phenomenon is known for deep charging and discharging (SoC between 0% and 100%) cycling, when degradation of charge transfer resistance is particularly noticeable in the Nyquist plot as reaction resistance. As such, in Figure 4 we show a comparison of the Nyquist plots between new and aged phases for Batt.1, Batt.2, and Batt.3, thus certifying that this semicircle appears later in the cycle aging when the battery deterioration has already started.

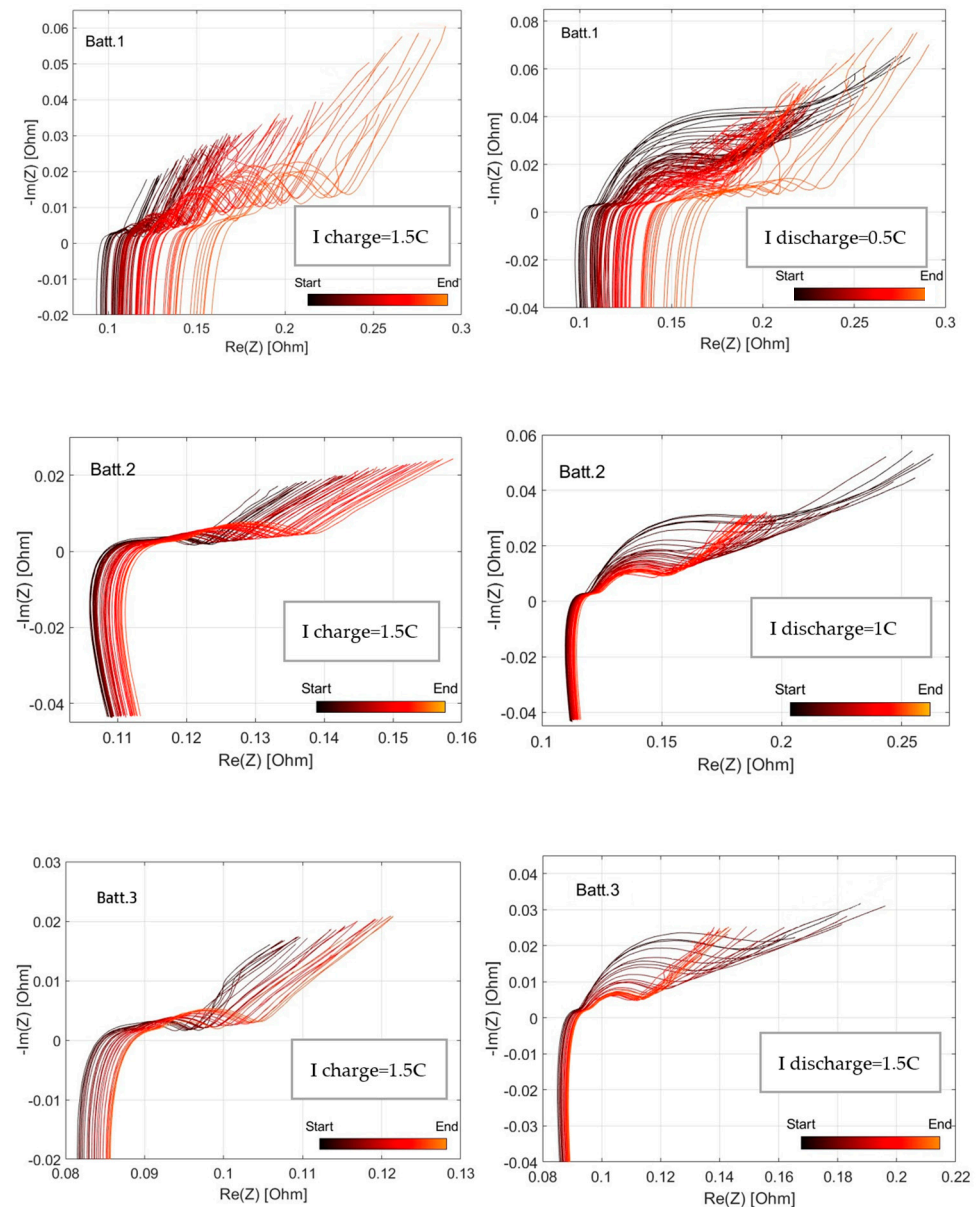
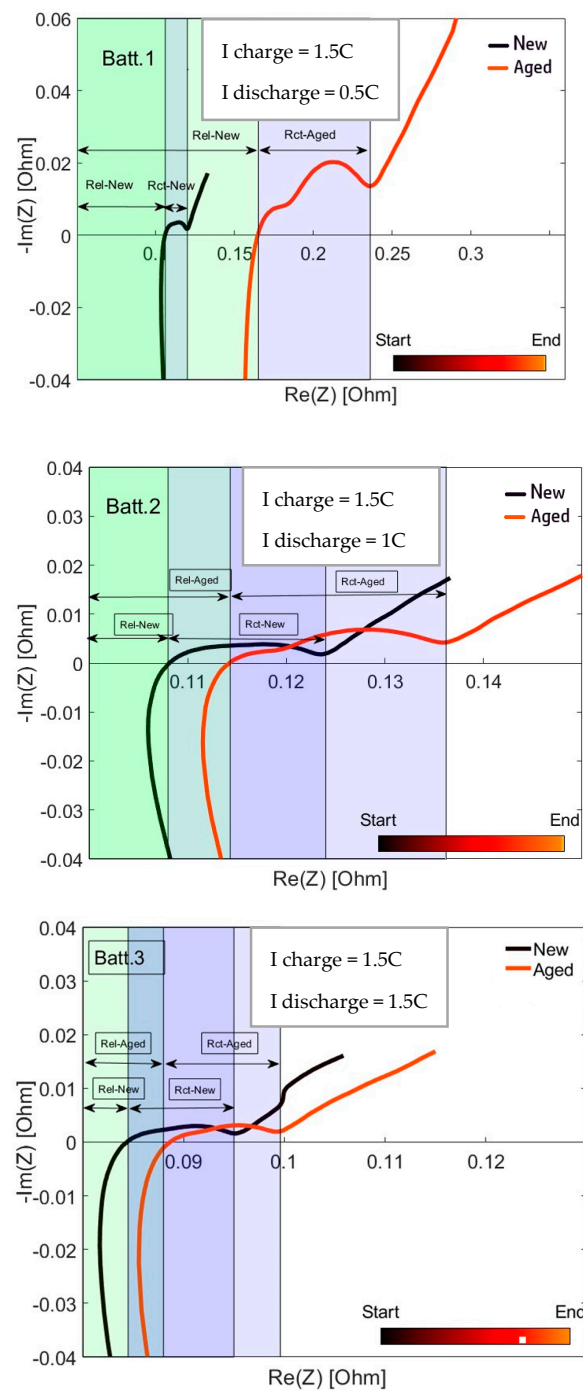


Figure 3. Left—Nyquist plots in charge state, Right—Nyquist plots in discharge state.

It is known that the main processes within LIBs, such as charging current, aging, or state of charge, can influence the expansion of the second semicircle and induce an increase in the charge transfer resistance, R_{ct} [63]. This will clearly influence the electrode production processes, or another reason may be that the electrode starts to react poorly on its surface. To determine how these factors affect this growth, two process frequencies were determined: the resonance frequency and the frequency at which the diffusion starts, which corresponds to the minimum frequency of the second semicircle. This semicircle is mostly defined by the degradation of charge transfer resistance, particularly noticeable in the Nyquist plot as reaction resistance. The electrolyte and charge transfer resistances in new and aged states are described below in Table 2.



Rel: Electrolyte resistance
 Rct: Charge transfer resistance

Figure 4. Comparison of the measured data for new and deteriorated lithium-ion batteries with a Nyquist plot.

Table 2. Battery resistance data.

Battery	Rel-New [Ω]	Rel-Aged [Ω]	ΔRel [Ω]	Rct-New [Ω]	Rct-Aged [Ω]
Batt.1	0.098	0.165	0.067	0.022	0.071
Batt.2	0.107	0.114	0.007	0.016	0.022
Batt.3	0.084	0.088	0.004	0.011	0.012

In the aging process, the new state of Rel is measured after four charge/discharge cycles and starts at 0.098 Ohm for a 0.5 C discharge rate, then increases to 0.107 at a rate of 1 C, and then drops to 0.084 Ohm at a 1.5 C discharge rate. The 0.009 Ohm increase for a 1 C-rate to a 0.5 C-rate is assumed to be a consequence of the increased discharge rate, while the 0.014 Ohm decrease for a 1.5 C discharge rate is merely caused by the increased temperature in the battery due to the high discharge rate. An increased temperature at a high SoH will decrease the cell ohmic resistance up to a point. Continuous aging enhances the cell impedance due to the electrolyte depletion of free electrons. According to Figure 4, as the discharge current increases while the number of cycles decreases, the ohmic resistance in the aged state increases for all three batteries. Thus, we can presume that a lithium plating phenomenon occurs due to the fact that the metal lithium cannot take part in the normal electrochemical processes. This will lead to a higher voltage drop during the discharge, translating to a lower efficiency and shorter operating time. As we have seen, degradation factors imply a reduced capacity and a cycle life reduction, leading to a significant drop in the number of usable charge–discharge cycles. Moreover, over time, lithium plating can lead to the growth of the solid electrolyte interphase (SEI) layer, which further contributes to increased resistance, both in the bulk material and at the interfaces.

ΔR follows a decreasing trend, as the number of cycles is significantly reduced, thus the electrolyte resistance is slightly affected by the discharge current but mostly by the number of cycles. Charge transfer resistance is inversely proportional to the discharge rate in both new and aged states.

3.2. SoH Dependency vs. Discharge Rate

SoH was employed here as a key metric used to assess the overall condition of lithium-ion batteries compared to their original (new) state. Two factors that affect SoH were taken into account: (a) capacity degradation over time and (b) cycle count and depth of discharge. Within the continual charge–discharge cycling, cell capacity continues to decline. In Figure 5, the SoH cycling evolution in terms of discharge rate for all three cells is described. The aim was to emphasize the general condition of the batteries and their ability to deliver energy over time, offering an indication of when the battery is experiencing problems or needs replacement. For determination of SoH behavior for each discharge rate, the following formula has been employed, as elsewhere [64]:

$$\text{SoH} = \frac{C_{\text{discharged}}}{C_{\text{initial}}} \times 100 [\%] \quad (5)$$

where $C_{\text{discharged}}$ represents the current available capacity and C_{initial} is the initial capacity measured by cycling each cell four times (which can differ a little bit from the initial capacity specified by the factory).

In terms of degradation level, we monitor the phenomena that occur when we apply to Li-ion batteries a deep I_{charge} of 1.5 C and then discharge with different C-rates, from standard to deep, namely 0.5 C for Batt.1, 1 C for Batt.2, and 1.5 C for Batt.3. For this type of battery (NCR18650B), the maximum recommended charging current is 0.5 C or 1.625 A [65]. A charging current above these values is considered fast charging and battery performance is reduced. The charging limitations of lithium-ion batteries are mainly influenced by lithium plating on the anode and electrolyte solution oxidation due to the high potentials at the cathode [66]. We set fast charging at 1.5 C as a compromise between these irreversible side reactions and battery performance. Additionally, 0.5 C is standard charging, and for that we know the battery behavior (it is given in the manufacturer specification sheet). This analysis is important in order to extend the battery's lifespan and, in particular, to optimize the battery's capacity and longevity.

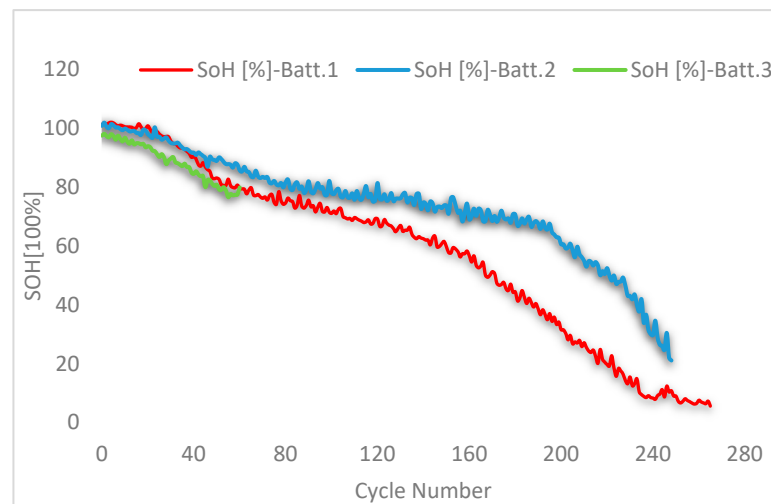


Figure 5. The dependence of SoH vs. cycle number for Batt.1, Batt.2, and Batt.3.

In Figure 5, it is clearly demonstrated that increasing the discharge rate will affect the process of lithium-ion extraction from one electrode and the intercalation to another. This process seems to be smoothly exponential for Batt.1 and Batt.2, but when the deep charge rate is equal to the deep discharge rate, the operation of inserting the lithium ions (embedded in the negative electrode during charging) into the positive electrode (during the discharge) becomes much more difficult.

As a direct consequence, battery damage occurs very fast when using a deep discharge rate of 1.5 C, in the case of Batt.3, when just ~60 cycling steps were required for the battery to become exhausted. In this case, cell reversal can cause an electrical short, thus demonstrating that the efficiency of the process is too strong, and the suffered stress is near to the maximum permissible tolerance, leading to a fast degradation of the electrode's elasticity. By contrary, for the lower discharge rates applied in the case of Batt.1 and Batt.2, a large number of cycles (above 240) were needed in order to fully discharge the cells. A slower discharge rate of 0.5 C or 1 C (Figure 5—Batt.1 and Batt.2) will extend the battery lifetime ~4 times more than the deep discharge rate of 1.5 C (Figure 5—Batt.3). For Batt.1 and Batt.2, the capacity loss is slow and, for Batt.3, the capacity loss accelerates quickly, thus decreasing the autonomy abruptly.

By performing an SoH analysis of Li-ion batteries till they are completely discharged (with different C-rates), a clear indication of how much of the battery's lifetime available energy has been consumed and how much is left, anticipating further issues or when the battery needs replacing, is offered. It is clearly noticeable that, starting at 60% SoH, the battery degradation has a significantly steeper increase for 0.5 C and 1 C. The last discharge rate of 1.5 C can only go up ~80% SoH, as the battery charge rate can no longer support the fast degradation.

Moreover, we have plotted the efficiency vs. cycle number in Figure 6 to check the cell state, taking into account the multiple challenges that can LIB cells can face over their long cycling period. The following chart highlights how the energy storage capability of Batt.1, Batt.2, and Batt.3, with a Coulombic efficiency of 99%, decays after cycling.

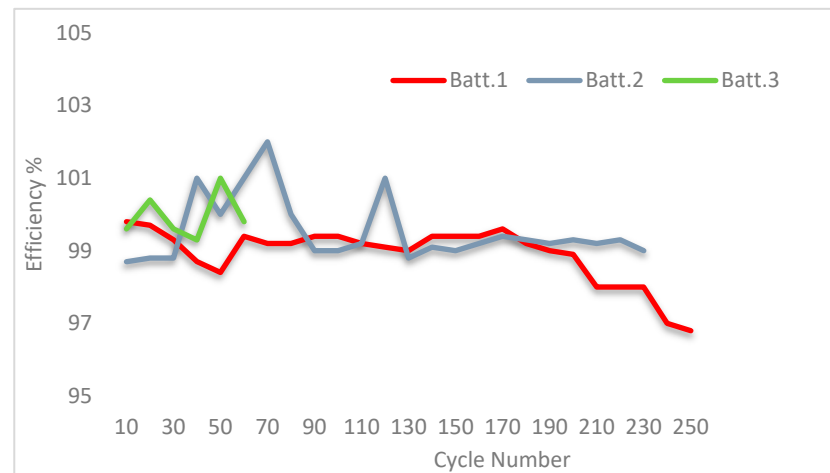


Figure 6. The efficiency of the LIB cells.

3.3. Resonance Frequency

The LIB performance and durability exploration has been carried out by gathering the results of resonance frequency, a single parameter fluctuation. As a common attribute of a circuit in alternative current, this element emphasizes the frequency where the resistance is minimal, and the scope here is to corroborate with other used methods for obtaining information on LIB SoH. Thus, the variations of the resonance frequency parameter are investigated during the charging–discharging cycles, highlighting the ability of electronic measurements to provide rapid predictability of cell health and lifespan.

It should be mentioned that single variations of resonance frequency were registered at each PEIS cadence, where the imaginary section of impedance is plotted as a frequency function. To be clearer, we show in Figure 7 an example of data obtained in a charging–discharging scheme for Batt.1.

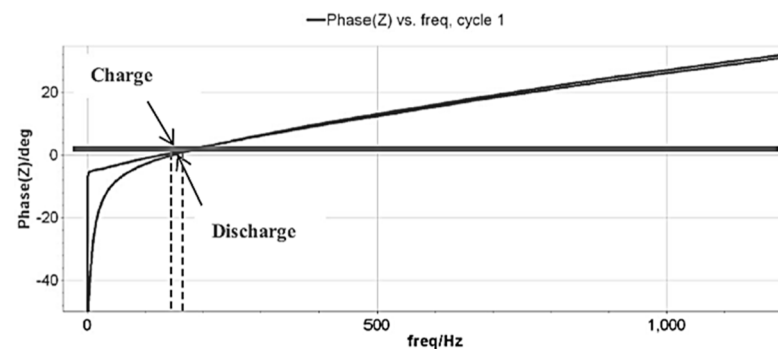


Figure 7. Data acquisition of Batt.1—resonance frequency in cycle 1.

In Figure 8 the results in all three cases are shown, and an ordering algorithm has been added in order to obtain a smoother version of the data and to better visualize the trends.

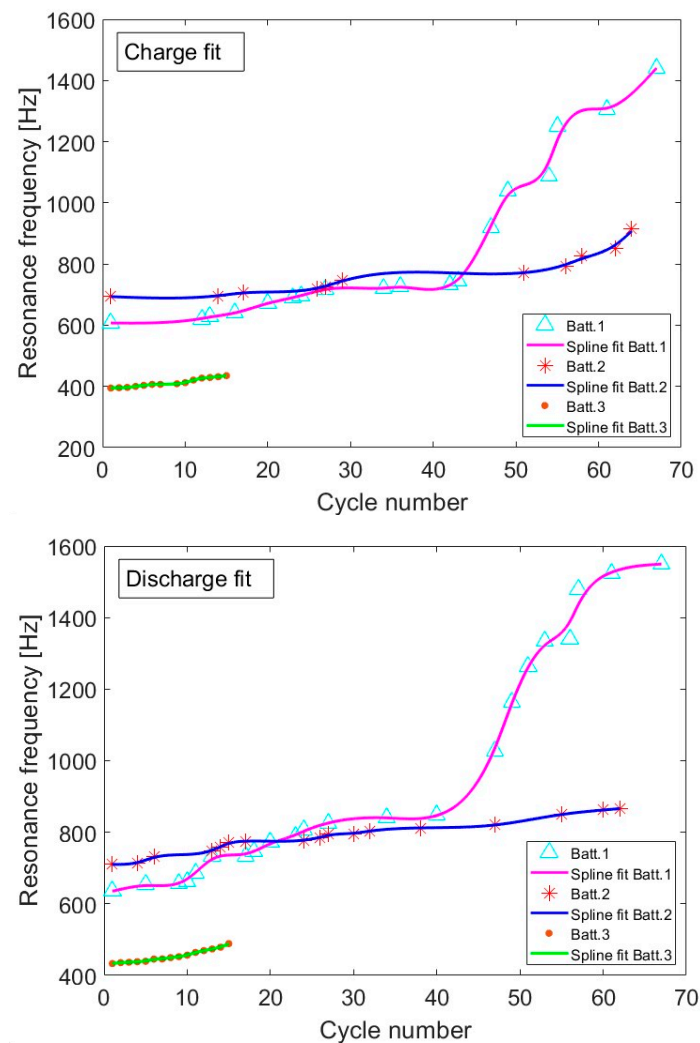


Figure 8. Resonance frequency with the spline fitting for charge–discharge rates of 0.5 C, 1 C, and 1.5 C.

A spline fitting was applied, namely a pink line for charge and a red line for discharge. For Batt.1, the resonance frequency starts at around 600 Hz and ends at around 1400 Hz. As the discharge rate increases to 1 C for Batt.2, a close value was obtained, around 680 Hz, but it ends much faster at around 900 Hz. As the discharge rate of Batt.3 increased at 1.5 C, the starting point of the resonance frequency gap falls to 395 Hz and ends very fast at around 435 Hz. It can be stated that, in the case of Batt.3, the resonance frequency moves completely into the middle-frequency range where all the current travels through Rct. A fast increase in resonance frequency can be seen toward the end of life for Batt.1 and Batt.2. This can be directly correlated with SoH, as a PEIS sequence was applied every fourth charge/discharge cycle. Therefore, at around 40 cycles/60% SoH (Batt.1) and 50 cycles/60% SoH (Batt.2), the resonance frequency develops a fast increase. For the 1.5 C discharge rate, the increase is almost linear, just as SoH decreases linearly in Figure 5.

Moreover, we can observe a smoother rate of change for the resonance frequency element when SoH is high, in particular for Batt.1 and Batt.2, and then it becomes more pronounced as the battery ages. In the case of the applied 1.5 C deep charge/discharge rate for Batt.3, an important reduction in the smoother part is observed due to the fast degradation. In fact, we can observe a linear growth, which seems to become much faster as we equalize the charging/discharging rate. When the imaginary term of the Nyquist coefficient crosses the real axis, the frequency of battery processes is canceled. That is, the resonance frequency for all three batteries increases towards the end of life.

4. Conclusions

We have investigated, by EIS methodology, the cycle life performance and the influence of C-rate on the charge/discharge processes of Li-ion Panasonic NCR18650B commercial batteries. A similar I_{charge} of 1.5 C has been applied in all cases, while the $I_{\text{discharge}}$ was modified from 0.5 C to 1 C and 1.5 C. The current was limited during the charging to 200 mA because of the possibility of overcharging during the PEIS sequence, and PEIS was employed every fourth cycling due to the large number of cycling steps. The aging process ended when the cell was not able to sustain the charging, at 1.5 C in our case. In order to support this affirmation, we have added below in Figure 9 the voltage characteristic for the first and last cycle of Batt.3 (1.5 C discharging) where it can be seen that charging can no longer be sustained.

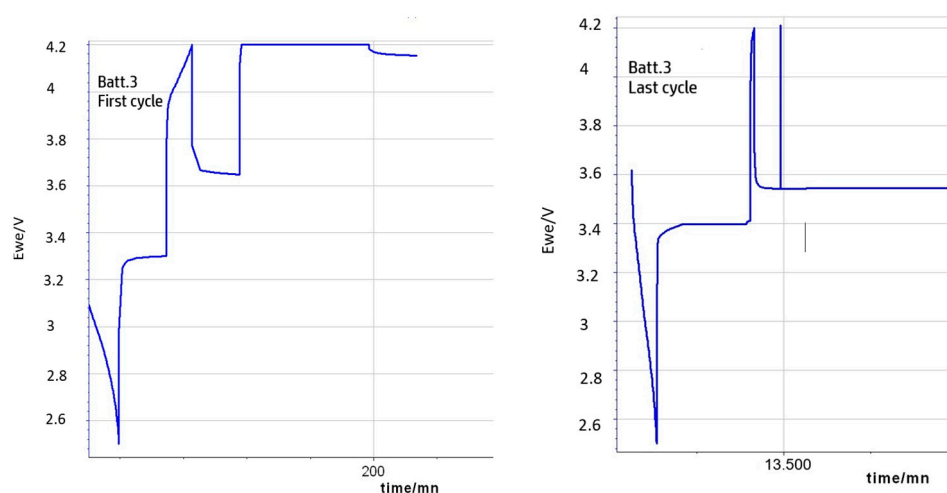


Figure 9. Voltage characteristic for the first and last cycle of Batt.3 (1.5 C discharging).

Starting from these conditions, EIS measurements were firstly performed on the LIBs in order to gain some insights into the lithium-ion transport processes, as well as the dependence on discharge rate, with the aim of understanding how their speed of degradation correlates with usage conditions. As such, their lifetime degradation depending on charge/discharge rate has been investigated by analyzing impedance fluctuations. Besides that, we have seen a particular behavior of LIBs: when discharge current increases and the number of cycles decreases, the ohmic resistance in the aged state increases for all LIBs. Thus, we can assume that the electrolyte resistance is slightly affected by the discharge current, but mostly by the number of cycles, and the charge transfer resistance is inversely proportional to the discharge rate in both new and aged states. By monitoring the correlations between the impedance data of the battery cells at the beginning of life with the cycLIB aging data, the cycle life performance of the battery cells can be predicted based on the impedance data, even at the early phase of the battery life.

In a second approach, we analyzed the SoH cycling evolution of Li-ion batteries till they were completely discharged with different C-rates with the aim to emphasize the general condition of the batteries and their ability to deliver energy over time, offering an indication of when the battery is experiencing problems or needs replacement. A further goal was to perform extensive laboratory testing of commercial Li-ion cells to develop and validate novel test methods based on the EIS approach for cycle life tests. As such, we have demonstrated that, by increasing the discharge rate, the process of lithium-ion extraction from one electrode and the intercalation to the other is significantly affected. This result shows that battery damage occurs very fast when using deep discharge rate of 1.5 C, in the case of Batt.3, when just ~60 cycling steps were required for the battery to become

exhausted. In contrast, for lower discharge rates applied in the case of Batt.1 and Batt.2, a large number of cycles (above 240) were needed in order to fully discharge the cells. In a real data comparison, a slower discharge rate of 0.5 C or 1 C will extend the battery lifetime ~4 times more than the deep discharge rate of 1.5 C. Another particular feature found here concerns the capacity loss, which is slow for Batt.1 and Batt.2, while for Batt.3, the capacity loss accelerates quickly, thus decreasing the autonomy abruptly. By measuring the SoH of Li-ion batteries till they are completely discharged, a clear indication of how much of the battery's lifetime available energy has been consumed and how much is left, anticipating further issues or when the battery needs replacing, has been offered. It is clearly noticeable that, starting at 60% SoH, the battery degradation has a steeper increase for 0.5 C and 1 C. From this perspective, if the battery end of life is set at 80%, more than 50% of its energy is lost, as the longest cycling part takes place between 80% and 60% SoH. When considering a fast-charging rate of 1.5 C, 60% of the initial capacity can be safely regarded as the end of life of Li-ion batteries for discharges up to 1 C. As for the last discharge rate (1.5 C), the degradation trend is nearly linear and merely lasts until 80% SoH, when the fast-charging rate can no longer be supported.

Another investigation of LIB performance and durability has been carried out through the analysis of the resonance frequency element. The results obtained are nicely aligned with the SoH data, and the rate of change is smoother at a high SoH for Batt.1 and Batt.2 and becomes more pronounced as the battery ages, while for Batt.3, a significant abatement in the smoother part is emphasized due to faster degradation. A linear growth can be seen that accelerates drastically when equalizing the charging/discharging rate. Thus, we validated our methods and, more importantly, we demonstrated that the SoH of a working LIB can be estimated during its service by monitoring the resonant frequency.

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