

Date of publication xxxx 00, 0000, date of current version xxxx 00, 0000.

Digital Object Identifier 10.1109/ACCESS.2017.Doi Number

Suppressing Anodic Degradation of Lithium-ion Batteries by Sinusoidal Waveform Charging Method

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ABSTRACT The effects of Constant current- Constant voltage charging and combined sinusoidal waveform charging methods on the internal structure of LiFePO₄ battery have been discussed in detail in this study. Experimental results clearly indicated that after 600 cycles, the battery that utilized the charging method had retained about 94% of state of health. In the electrochemical impedance analysis and cyclic voltammetry tests, the portion of the SEI film impedance was increased about 0.08 mΩ. In the micro structured view, no significant SEI film formation was observed on the surface of the negative electrode that used the battery in the present charging method. Finally, in the analysis of the SEI film composition, it was found that the Li₂CO₃ and Li₂O content in the Li element was only about 15.97% and 4.42% in the battery employing this charging method, and the battery component contents in the CC-CV charging method were 27.2% and 6.69% respectively.

INDEX TERMS Lithium-ion battery; charging; sinusoidal waveform

I. INTRODUCTION

Recently, massive research emphasis is being laid on the discovery of alternative energy sources as the past few decades have witnessed a global upswing of environmental and energy predicaments [1]. However, as an electrochemical system, lithium-ion batteries will gradually degrade during its use or shelving, and the power capability and safety will also deteriorate. Energy security and environmental issues like urban air pollution due to internal combustion engine vehicles are increasing at a much faster rate. The field of automobile engineering is rapidly advancing in the production and utilization of energy efficient vehicles, in which electric vehicles (EVs) have become the most important technological development objective for governments and enterprises. Among EVs, the most well developed are the EVs that use Li-ion batteries as a power source owing to their superior performance and extended cycle life [2]. Transportation electrification has

also become a foremost ultimatum for energy conservation and for the advancement of sustainable, eco-friendly, and energy-intensive locomotors in almost all countries. Among all, Lithium-ion (Li-ion) batteries are much preferred due to various advantages like high specific energy and power along with a prolonged life [3,4]. The State of Health (SOH) of a battery has to be significantly monitored by battery management systems, where the variations between an aged and a fresh battery can be evaluated quantitatively [5]. A primary factor of battery degradation is the formation of a solid interface on the electrolyte-anode interface, which is called a solid electrolyte interphase (SEI). The SEI consists of solid Li complexes that are created by combining Li-ions and decomposed electrolyte with excess electrons during the first few charge cycles via reduction reaction. Furthermore, SEI and its crucial impacts are usually unkempt and its formation is tough to control [6].

Li-ion battery-based EVs have not been able to replace internal combustion vehicles on a large scale so far, largely due to issues with the batteries such as a limited longevity and long charging time. Battery aging is the primary limitation and the batteries decay every time they are used and age-induced degradation is irreversible. The aging strategy of these batteries is multifarious and complex that may be related to factors like operational conditions, types and reaction phases [7]. Improvements in the battery longevity can significantly improve battery performance throughout the life of EVs. Hence, the development of energy storage technologies has become critical in addressing the challenges of EVs. Although considerable efforts have been made in the development of materials for inner Li-ion batteries, less attention has been paid to solving aging problem via charging strategy [8, 9]. At the beginning of battery utilization, the primary role of the SEI is to protect the negative electrode from possible corrosion and the electrolyte from reduction. Due to repeated charging and discharging of the battery, the SEI is not stable enough and thus the battery operates in tension outside the electrochemical stability range of the electrolyte. The SEI progresses over time, inducing a loss of recyclable Li-ions and the decomposition of electrolyte. A compact and consistent SEI will prevent electrolyte penetration into the active material, thus preventing continual Li losses and thereby warranting an elevated battery coulombic efficiency [10].

Loss of available Li for solid Li complex formation is the foremost reason for battery aging during storage periods. The aging mechanisms of Li batteries are multifarious and intricate which are strongly linked to many interactive factors, such as battery categories, electrochemical reaction phases, and operational situations. In that constant high-power charging has been identified as the main factor that accelerates aging [7]. High-power charging is capable of negatively affecting the resilience and security of Li batteries due to increased heat generation, which could induce strong risks like battery capacity degradation and thermal runaway. Nevertheless, substantial research and engineering efforts have been undertaken to stabilize the development of charging methods that could ensure lowered capacity loss and enable long-term Li-ion battery utilization [11]. Among various charging methods, CC-CV is the most extensively implemented technique. The CC-CV charge profile is extensively accepted for Li-ion battery charging due to its great efficiency and adequate protection of the battery SOH. However, SEI formation can occur during normal CC-CV charging, if the charging current rate reaches or exceeds a certain value. Thus, the charging performance of the CC-CV method cannot sufficiently satisfy the long-cycle-life requirement of the consumer. The development of new charging methods that could effectively balance both battery degradation as well as fast charging, remains a challenge. Reflex charging methods

that have been proposed can only apply to a very short discharging pulse during pulse charging and resting periods [12]. As passivation layers develop mainly via reduction reaction, the discharging pulse in reflex charging method could be used to reduce unwanted chemical reactions at the electrode surface, such as side chemical formation, crystal growth, and passivation in nickel-cadmium batteries. Charging techniques involving negative pulses have also been revealed to achieve high performance in Li-ion batteries. Monem et al., [13] have concluded that pulse charging could effectively enhance the battery charging capacity and reduce the resistance. This could obviously delay the aging process of the battery. Sinusoidal current charging improved the charging time, charging efficiency, maximum rising temperature, and the lifetime of the battery [14]. Although charging methods with negative pulse have been proposed for battery improvement, such charging strategies, involving discharging stages, have not been widely utilized due to the lack of a theoretical basis for the charging voltage. Most experiments have been conducted without considering the physicochemical aspects, which has made the integration of cross domain knowledge a huge obstacle to the development of this technology.

SEI formation in charging cycles has been considered as a major bottleneck for battery age which can be irreversible. The formation was caused by the reduction reaction of electrolyte molecule coupling of the Li-ion after receiving an excess electron. For instance, when using ethylene carbonate (EC) as an electrolyte, the reaction started with the formation of a $\text{LiOCOOC}_2\text{H}_4$ radical for the excess electron and breaking the CE-O_1 bond [15,16]. $\text{LiOCOOC}_2\text{H}_4$ can extensively react to form solid molecules, such as dilithium ethylene dicarbonate $[(\text{LiOCOOC}_2\text{H}_4)_2]$, lithium carbonate (Li_2CO_3), Li_2O , and stack on the graphite negative electrode. EC breakdown occurs in the succession of electron transfer to the Li-EC during charging. Although the electronic insulation property of the initial SEI film inhibits further reduction of the electrolyte, an improper charging method via constantly pumping electronic energy into the battery may enhance electron transfer and continue EC reduction. The ceaseless reduction reaction during the charge/discharge cycle is the key significant aging problem of Li-ion batteries [17]. Thus an endeavor has been attempted to introduce discharging in a charging strategy, which can be capable of enabling the inverse reaction of $\text{LiOCOOC}_2\text{H}_4$ radical formation and preventing SEI growth has been attempted in this research study.

Our previous research has evaluated the moderate energy for charging Li-ion batteries and all our preliminary experiments have been greatly successful. Compared with the CC-CV method, the proposed charging method had definitely enhanced the battery capacity of an aged 18650 Li-ion battery by 18.7%, and reduced the maximum temperature rise by 9 °C, and the charging time by 25 min. Nevertheless,

a important issue remains that the battery capacity has been used as a standard for assessing the effects. Direct spectral characterization will also be imperative. In order to fulfil the future broadening requirements of charging, development of a device for the generation of waveform is of utmost necessity. Thus, the application of these strategies in automobile engineering will assuredly be an inexorable success in the future.

II. MATERIALS and METHODS

Numerous studies have concluded that the most significant cause of Li-ion battery aging is due to the conjoining of molecules of the electrolyte with the Li atoms during charging to form a passivation layer of SEI, which will eventually lead to the depletion of recyclable Li ions in the electrolyte and a subsequent decline in battery capacity [18-19]. The CC-CV charging is the most commonly applied method for charging Li-ion batteries till date. However, in cases of high C-rate charging and discharging of electric vehicles, battery capacity is more likely to decline, leading to concerns like battery aging, decrease in battery life cycle and rated service life. This will result in expensive capital costs for maintenance and replacement of the battery pack, thereby hindering a wider use of electric vehicles due to cost ineffective nature [20]. Thus alternative charging methods for enhancing battery life span and uninterrupted service are the need of the hour. A sinusoidal waveform charging technique developed by our research team has an undeniable great potential for further development as it can assist in avoiding unnecessary energy expenditure and prevent battery aging. Hence, this charging strategy, can effectively solve the aging problem caused by Li-ion battery charging [4]. The charging section and discharge section are composed of two half cycle sine waves with different amplitude, as illustrated in Figure 1.

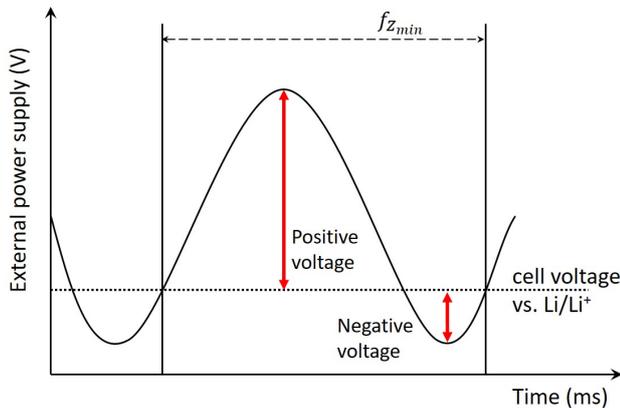


FIGURE 1. Representation of sinusoidal waveform.

The waveform of the charging section was designed according to the energy required for the Li ion migration in each stage during the charging process. The energy required in the process of Li ion from the positive electrode to the negative electrode during charging was estimated and then combined into the form of sinusoidal waveform as the upper

half amplitude of the charging. The lowest resistance frequency of AC impedance corresponded to the impedance frequency of SEI layer ($f_{R_{SEI}}$). It can be inferred that the minimum resistance frequency of AC impedance fulfilled the requirement of the time of Li ion migration from positive electrode to negative electrode during charging. Most importantly, considering the formation of SEI passivation layer at the coupling of electrolyte and negative electrode during charging, a discharge section is specially designed to inhibit the generation of SEI layer by Li ion to slow down the decline of capacitance.

The charging-discharging experiments were performed by a commercial instrument (N6705A, Keysight Technologies, Inc., Santa Rosa, CA, USA). Prior to spectral observation, the capacitance of 18650 batteries was measured using CC-CV charging method with the current of 0.5 C and those with similar capacitance were selected. Following this, the electrochemical impedance spectroscopy (EIS) analysis and cyclic voltammetry (CV) were employed to measure the internal impedance and electrochemical performance of the battery, and the battery with a similar initial state was selected as an experimental battery. Subsequently, the battery shall be charged and discharged at room temperature.

Both the sinusoidal waveform charging and CC-CV charging methods adopted the current of 5 A (about 3.4 C) for charging. In the CC-CV charging method, the cut-off voltage in the CC and CV sections were set as 3.65 V and 72.5 mA respectively, when the charging current was less than 0.05 C. For the sinusoidal waveform charging method, the cut-off condition was when the current value displayed by the charging instrument showcased positive and negative signs. Once the battery was fully charged, it was then allowed to rest for 15 min, till the internal ion concentration balance and the battery temperature reduced. The discharge part utilized 5 A current to discharge the battery at CC and the cut-off condition was customized to stop discharging when the battery voltage drops down to 2.5 V. There was another 15-minute cessation, for the internal ion concentration balance and battery temperature to abridge.

In order to compare the difference between the two charging methods in terms of life cycle, the standard practice of discontinuing the life cycle when one of the battery capacity declined to 80% was adopted. This cut-off condition was followed according to the standard of Li-ion battery for EV. The life cycle test for both charging methods was similar. Following every 100 charge and discharge cycles, the battery was allowed to repose for one day, and its internal impedance and electrochemical performance were measured by EIS and CV. Following the battery life cycle test, the battery was disassembled in the drying room and the negative electrode plate of the battery was extracted. It was then soaked in dimethyl carbonate (DMC) to remove impurities. Afterwards, the electrode was detached from the battery and placed in a vacuum oven following one day of static soaking, and the excess dimethyl carbonate on its surface was removed at a

temperature of 50 °C. The air in the oven chamber was completely extracted out for providing vacuum conditions. A field emission scanning electron microscope (FE-SEM) was used to observe the micro state of the plate and the structure of negative carbon was analyzed by Raman spectroscopy. The functional groups of the surface were then identified using FT-IR. The composition of SEI film on the negative electrode surface was determined by ESCA.

III. RESULTS and DISCUSSION

Figure 2 demonstrates the capacitance decay curve of two charging methods at room temperature and at high temperature. After 600 charge and discharge cycles at room temperature, the capacity decline curve of the battery using the combined sine wave charging method was quite moderate, and when it reached the number of cut-off cycles, it still retained about 94% of the capacity. While the capacity of the battery using CC-CV charging method appeared to be lower than that of the battery using the combined sine wave charging method after about 100 cycles. Subsequently, after 400 cycles, the capacitance declined exponentially to 80% of the original value and the capacity of the battery using the sinusoidal waveform charging method appeared to be still higher than that using the CC-CV charging method. This proved that the charging method was highly effective in extending the service life of the battery even under high temperature environments.

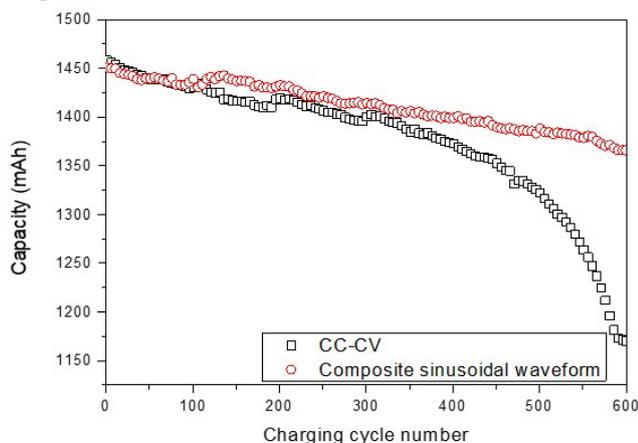
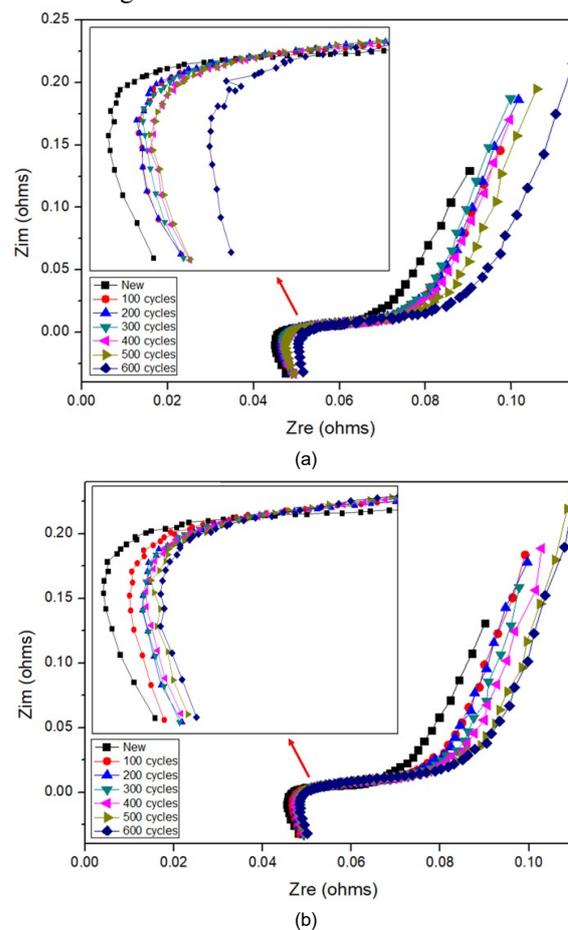


FIGURE 2. Capacity fade of Li-ion batteries charged by conventional constant current-constant voltage (CC-CV) and sinusoidal waveform method after 600 charging-discharging cycles.

For the electrochemical impedance analysis, the battery was discharged to 2.5V at a current of 0.5C after every 100 charging-discharging cycles, and was measured after a resting period of one day. The amplitude set by the measuring instrument was 75 mA and scanning frequency range was 2000 ~ 0.01Hz. The measurement results are displayed in Figure 3(a) and 3(b). The curve was observed to be composed of semicircles in high frequency and intermediate frequency areas, along with an oblique line in low frequency area. Later, the measurement results were

simulated and analyzed by equivalent circuit. As illustrated in Figure 3(c), the impedance values of each resistance in the battery can be obtained. $R\Omega$ represents ohmic impedance, R_{sei} signifies SEI passivation impedance and R_{CT} denotes mass transfer impedance. It was perceived from the test results in Table 1, that in the 100th cycle at room temperature, the impedance of each part of the battery using CC-CV charging method increased, which might be due to the increase of the impedance in the process of adjusting the internal electrode configuration to make it more stable at the initial stage of charging and discharging. While in the 200th cycle, the electrode configuration of the battery was much more stable, which induced decrease in the impedance values. However, with the increase of charging times, the battery began to age, and its impedance value continued to rise. The results showed that the impedance value of SEI membrane of the battery using the combined chord wave charging method was decreased in the initial 400 cycles. This might be associated to the design of negative half wave in the charging method. The impedance value of SEI membrane increased by only about 0.08 m Ω at the culmination of 600 cycles. Compared with the battery using CC-CV charging method, the impedance of SEI membrane increased by about 0.94 M Ω , indicating that this charging method certainly has inhibited the growth of SEI film.



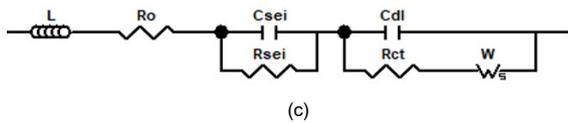


FIGURE 3. Electrochemical impedance spectroscopy of Li-ion batteries charged by (a) CC-CV and (b) sinusoidal waveform method. (c) Equivalent circuit model for the 18650 Li-ion battery.

TABLE I
Impedance value from equivalent circuit model.

	Sinusoidal waveform						
	New	100	200	300	400	500	600
R_{Ω} (m Ω)	45.66	46.89	47.59	47.59	47.72	48.05	48.38
R_{SEI} (m Ω)	3.63	3.51	3.48	3.55	3.62	3.64	3.71
R_{CT} (m Ω)	5.30	6.44	6.61	6.99	7.05	7.36	7.62
	CC-CV						
	New	100	200	300	400	500	600
R_{Ω} (m Ω)	45.11	46.71	46.61	46.99	47.46	47.50	50.50
R_{SEI} (m Ω)	3.31	3.91	3.49	3.56	3.67	3.81	4.25
R_{CT} (m Ω)	4.77	6.81	6.35	6.53	7.44	7.51	7.93

Cyclic voltammetry was used to substantiate the electrochemical activity of the battery according to the increase or decrease of the peak current value. When the lithium-ion battery was used for cyclic voltammetry analysis, reduction and oxidation peaks were observed in the process. The cyclic voltammetry of this experiment was carried out after the completion of electrochemical impedance analysis with the measurement range of 2.5-4.2 V and scanning frequency of 0.1 mV/s. The measurement results have been revealed in Figure 4, where IPA represents the current value of the reduction peak and IPC characterizes the voltage value of the oxidation peak. It can be inferred from the chart that the current values of the reduction and oxidation peaks of batteries using CC-CV charging method were much higher than that of batteries using combined chord wave charging method. According to the reaction equations of oxidation and reduction processes of lithium-ion batteries, it can be concluded that with the increase of charging and discharging cycles, the SEI film inside the battery gets continuously thickened, thus rendering the battery internal. The number of Li ions capable of participating in charge discharge cycle reaction was decreased thereby decreasing the overall current generation in the reaction process.

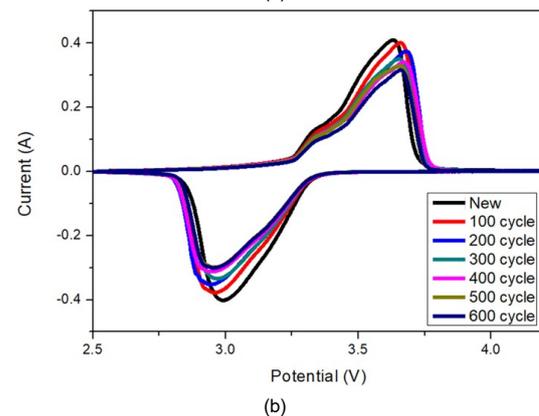
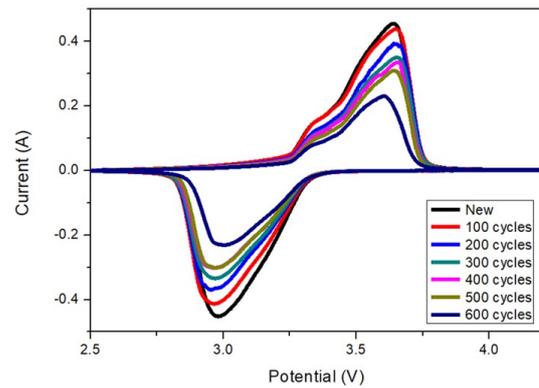


FIGURE 4. In-situ cyclic voltammetry spectroscopy of Li-ion batteries charged by (a) CC-CV and (b) sinusoidal waveform method.

After 600 charge and discharge cycles at 25°C, the battery was disassembled and the negative electrode was removed. The surface microstructure of the battery was observed under a field emission scanning electron microscope. The analysis results are publicized in Figure 5(a-c). SEI film formations were not detected on the negative electrode surface of the battery using the combined sine wave charging method, and the surface state of the battery was very similar to that of the new battery. However, few SEI film white spots were noticed on the surface of the battery using the CC-CV charging method. Through EDS element content analysis [Figure 5(d)], the content of the battery charged by sinusoidal waveform charging was identified to be very analogous to that of the new battery. The use of this charging method to charge the battery at room temperature can effectively reduce the generation of SEI film and oxide on the negative electrode surface, thus effectually extending its service life.

In order to understand how charging method affects the graphite electrode, Raman analysis was performed. The Raman spectra were excited by the laser of 2.41 eV and a 60X objective lens was used to focus on the specimens with spot size of 1 μ m. The spectra ranged from 1000 to 3000 cm^{-1} including D-, G-, and 2D-band correspondingly, as shown in Figure 6(a).

Since the G-band originated from the E_{2g} mode of the graphitic structure, the intensity ratios of D- (I_D/I_G) and 2D-band to G-band (I_{2D}/I_G) were calculated to evaluate the defectiveness and stacking order of the graphite electrodes under different charging conditions. The intensities of the Raman signals were obtained from Lorentz fitting and the intensity ratios were plotted in Figure 6(b).

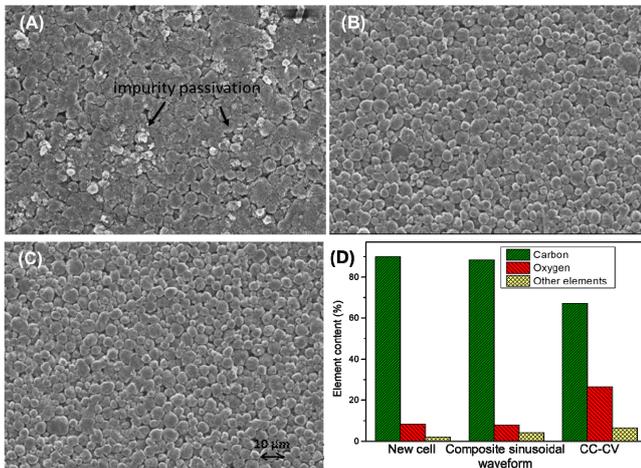


FIGURE 5. Scanning electron microscope (SEM) images of anodic surface of aged batteries respectively charged by (a) CC-CV, (b) sinusoidal waveform method, and (c) a new battery. (d) Energy-dispersive X-ray spectroscopy of the three batteries.

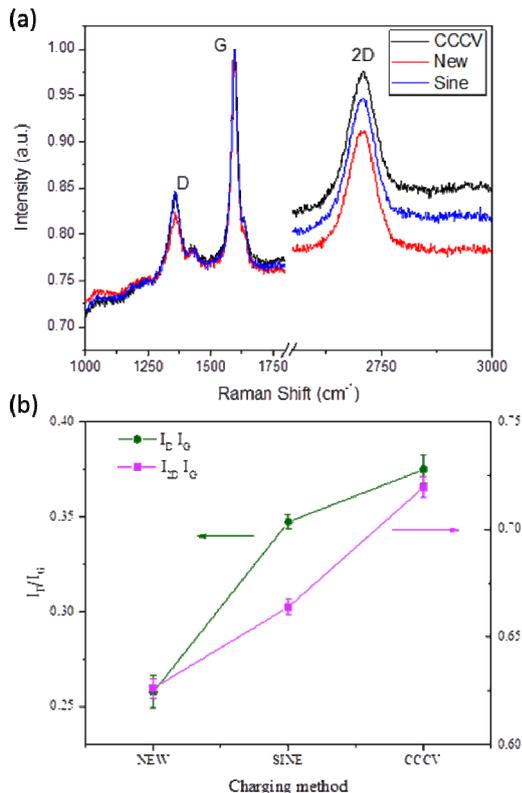


FIGURE 6. (a) Raman spectroscopy and (b) intensity ratio of I_D/I_G and I_{2D}/I_G of anodic graphite charged by CC-CV and sinusoidal waveform method.

Estimation of I_D/I_G , revealed that the quality of the graphite electrode appeared to have significantly reduced by the CC-CV and the Sine exhibited moderate degradation of electrode. Data from several sources have been used to identify the 2D-band associated with stacking order of graphitic layers and that was closely linked to the intercalation of Li ion in the graphitic layers. Astonishing results were observed from the I_{2D}/I_G and they indicated that the Sine charging method was capable of suppressing the intercalation than compared with that of CC-CV.

Chemical electronic energy spectrometer was used to measure the batteries under sinusoidal waveform charging and CC-CV charging methods along with the brand-new battery. According to the in-depth analysis of the main components of SEI membrane, such as lithium (Li 1s), oxygen (O 1s), carbon (C 1s), the proportion of the components of SEI membrane in the battery was indicated alongside the fact that the sinusoidal waveform charging method was capable of inhibiting the growth of SEI membrane [1,2,4,6,7,8,10,11].

The analysis results have been presented in Figure 8(a,b,c) and the characteristic peaks of Li 1s were positioned between 52 and 58 eV, and they included LiOH at 54.9 eV, LiF at 56.7 eV, Li_2CO_3 at 55.2 eV and Li_2O at 55.6 eV. The batteries with the sinusoidal waveform charging method exhibited lower contents of Li_2CO_3 , Li_2O and O, equaled to batteries charged with CC-CV method. These two compounds are the final products in the growth process of SEI film. Their bond energy with Li is large, which might have been a great hindrance for its participation in the charge-discharge cycle. Consequently, reduction of the generation of these compounds might have effectively prolonged the service life of the batteries.

After the completion of 600 charge-discharge cycles at room temperature, the results of C 1s analysis are shown in Figure 7(d,e,f). Two characteristic peaks of C were noticed between 281 and 293 eV and in this range, there were Li_2CO_3 at 290 eV, along with C-C, C-H, ROCOOLi and C-C-O bonds at 284.4 eV, 285 eV, 288 eV and 286.5 eV respectively. ROCOOLi was the main component at the initial stage of SEI film formation and its the content of ROCOOLi complex in batteries using sinusoidal waveform charging method was found to be much elevated than that of batteries using CC-CV charging method. This significantly specified that this particular charging method was effective not only in reducing the formation of compounds with strong bonding forces in SEI membrane, but also maintaining them in the form of ROCOOLi complex in batteries. This phenomenon eventually lead to prolongation of the Li^+ participation in charging and discharging Number of electrical cycles.

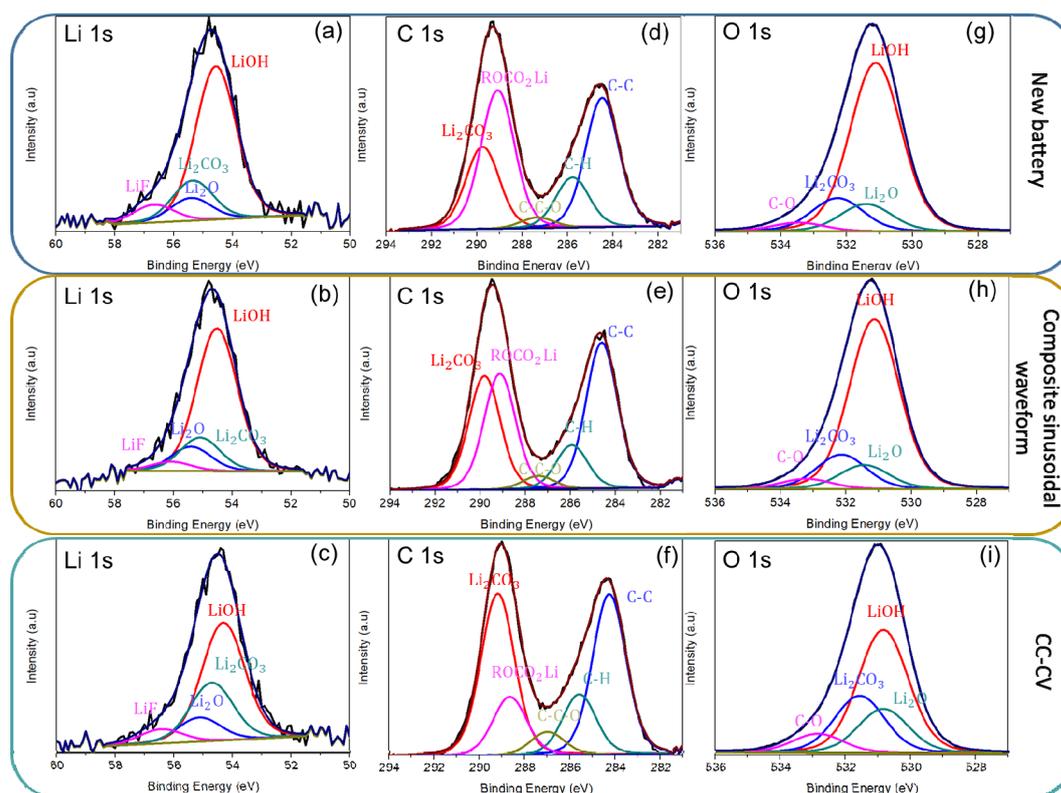


FIGURE 7. X-ray photoelectron spectroscopy.

The O 1s analysis is displayed in Figure 7(g,h,i) and it can be witnessed that the characteristic peak of O 1s was between 527 and 537 eV. In this range, LiOH at 531.5eV, LiOH at 532 eV, LiO at 531.3 eV, and CO bond at 533.5 eV were included. Among them, Li₂CO₃ and Li₂O were the final products of SEI film formation, and CO bond was the oxide bond formed by negative substrates. The proportion of the contents of Li₂CO₃, Li₂O and CO bond in the batteries with the sinusoidal waveform charging method were also found to be lower than that of the battery with CC-CV charging method, which was the important evidence that the type of charging can effectively influence and inhibit the growth of SEI film and also reduce the formation of oxides during battery use.

V. CONCLUSIONS

The significant deductions of this present experimental study has been presented in a point-by point manner as follows

- An innovative LiFePO₄ battery for electric vehicles was designated as the experimental battery.
- A designated 600 charge-discharge cycles were conducted at room temperature, to compare the differences in electrochemical performance, internal structure of the battery and the shape of SEI film between the battery using two different charging methods namely combined chord wave charging method and the traditional CC-CV charging method.

- The experimental results after the charging-discharging cycle demonstrated that the capacity of the battery using this charging method was about 94%, while the impedance of SEI film in the electrochemical impedance test had merely increased by about 0.08 mΩ.
- In the cyclic voltammetry analysis, the peak current values of reduction and oxidation peaks were abridged only by about 0.09361 A and 0.15126 A correspondingly, which was considered superior than that of the battery using CC-CV charging method.
- In the internal structure analysis of the battery, it was interestingly revealed that the I_D/I_G value of this charging method was 0.30599, which was very much reduced than that of CC-CV charging method.
- Finally, in the composition analysis of the SEI membrane, the contents of Li₂CO₃ and Li₂O in Li 1s and O 1s were fewer than those of CC-CV charging method, while the content of ROCOOLi in C 1s was less than that of CC-CV charging method.

ACKNOWLEDGMENT

We thank the financial support from Ministry of Science and Technology, Taiwan, under grant numbers MOST 108-2113-M-002 -021-MY2. Financial supports from the Center of Atomic Initiative for New Materials (AI-Mat) at National Taiwan University (108L9008), and the Featured Areas Research Center Program within the framework of the Higher Education Sprout Project by the Ministry of Education (MOE) in Taiwan are also acknowledged.

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