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Determination of Li-ion battery degradation mechanisms at high C-rate charging

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Abstract—A modification of the differential voltage analysis method is developed. It allows determination of the degradation mechanisms during charging of Li-ion batteries. A constant current (CC) with relatively high C-rate can be used in the analysis method. The modification of the differential voltage analysis method is based on measuring the heat generated during CC charging of Li-ion batteries. The advantages and the applicability of the presented modified differential voltage analysis (MDVA) method are verified by a comparison with currently existing and proven methods used in determining the Li-ion battery degradation mechanisms.

Keywords—Li-ion batteries, State-of-Health, MDVA method, degradation mechanisms determination, heat flux measurements.

I. INTRODUCTION

The popularity of battery powered electric vehicles (EVs) has increased significantly in recent years because of the aggravating problems related to the traditional economy based on fossil fuels, implied greenhouse gases emission and its effect on the environment. According to the International Energy Agency (IEA), the number of EVs may significantly increase up to 38.5 million by 2030 [1]. This definitely leads to a problem related to the utilization of the electric energy storage system, which is an essential element of EVs, at the end of its life.

Currently, Li-ion batteries are considered as the most suitable choice for EV energy storage, because of advantages, such as: acceptably high volumetric and gravimetric energy density, large cycle life, low self-discharge rate and no memory effect. Typically, Li-ion batteries can be used in EVs until the battery capacity has decreased by 20%. At this point the battery should be replaced as it may not be capable to provide the required performance for the EV. However, this battery still has 80% of its initial capacity, and can thus be used in other less-demanding applications, where the required power, volume and weight boundaries are not so critical [2]. Stationary grid-related applications that have lower energy and power demands are considered to be the most suitable systems for the second life application of an EV battery [3]. The use of EV batteries in such cases has been intensively analysed by many researchers [4]–[11]. In general, it was concluded that the second life usage of the EV battery systems may be economically attractive. However, the performance of the Li-ion battery in such an

application strongly depends on its previous history. Therefore, to achieve safe operation in second-life applications, the aging history of batteries in the first-life application has to be very well known [6].

The aging history should contain information about the degradation mechanisms, which were the reason of the ageing of the Li-ion battery. However, the non-observability of the electrochemical processes, which lead to the Li-ion battery degradation, complicate the determination of the degradation mechanisms. The evaluation of these mechanisms without Li-ion battery disassembly is only possible through indirect methods, e.g. by analysis of the open circuit voltage changes when a Li-ion battery is ageing. Such approach is used in relatively new techniques such as the incremental capacity analysis (ICA) and differential voltage analysis (DVA) methods, which are used in evaluating the degradation mechanisms of Li-ion batteries. The applicability of the ICA and DVA methods is demonstrated by many researchers in [12]–[15] and [16]–[21], correspondingly. Despite the applicability of these methods in evaluating the degradation mechanisms, the requirement that the Li-ion battery should be fully charged or discharged by a low non-stop continuous C-rate current to measure the incremental capacity ($\partial Q/\partial V$) or differential voltage ($\partial V/\partial Q$) curves, may limit practical on-board implementation of these methods in EVs. Therefore, methods, which allow fast measurements of the data required in ICA or DVA methods, are extremely desirable.

In the present work the applicability on the MDVA method in estimating the degradation mechanisms of four different Li-ion chemistries during cycling ageing is analysed. The MDVA method is based on the utilization of the measured heat flux and temperature data to obtain a modified differential curve, which can be analysed in a similar way as in the DVA method. The measurements are made at a relatively high C-rate charging of the Li-ion batteries. A comparison between the MDVA and DVA methods was done and conclusion about the applicability of the MDVA method is made.

II. MATERIAL AND METHODS

A. Li-ion cylindrical cells

Cylindrical Li-ion cells have been selected for the cycling tests. The chemistries and characteristics of the selected cells are given in Table I.

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TABLE I. CHARACTERISTICS OF THE SELECTED LI-ION CELLS

Chemistry	Characteristics				
	Voltage, V	Capacity, Ah	Mass, g	Diameter, mm	Length, mm
NMC/C	3.7	4.4	82	26.2	65.0
NCA/C	3.6	2.7	48	18.5	65.3
NCA/C+Si	3.6	3.2	48.5	18.65	65.15
LFP/C	3.3	2.5	70	25.85	65.15

During selection of the Li-ion chemistries for the tests, high-energy Li-ion cells were preferred. The commercially available INR26650EC cell (NMC/C) produced by O'cell and NCR18650PF cell (NCA/C) produced by Panasonic were selected, as these chemistries are the most popular in EV battery systems, nowadays. In addition, a non-commercial cell with improved energy density was tested. Lithium nickel cobalt aluminium oxide (NCA) was used for the positive electrode in the considered non-commercial cell. Moreover, the silicon (Si) was used as an additive in the graphite (C) negative electrode in this cell to improve the energy density. In addition to the selected energy type Li-ion cells, the power type A123 ANR26650M1B cell (LFP/C) was selected for the cycling test to expand the field of the MDVA method evaluation.

B. Cycling tests

The electrochemical experiments were done by using automatic battery testing equipment (Neware CT-4008-5V6A-S1). The ambient temperature was controlled by a climate chamber, which consists of a double-wall metallic box, where the temperature of the circulated liquid is controlled by a refrigerated-heating circulator (JULABO). All considered Li-ion cells were activated at 25 °C ambient temperature during 5 charging/discharging cycles before starting the cycling tests. Moreover, after activation a characterization of the cylindrical Li-ion cells was done. The characterization of the considered cells included the determination of the electromotive force (EMF), entropy change curve and differential voltage curves necessary for the current research. All measurements were done after a constant-current constant-voltage (CCCV) charging. The charging in the constant current (CC) mode was done by 1/2 C-rate continuous current up to cut-off charging voltage and further the constant voltage (CV) mode was applied during 2 h. The EMF curves for all cells were determined during charging and discharging of the considered Li-ion cells by different C-rate constant currents (1/10, 3/20, 1/5 and 5/20 C-rate) up to cut-off charging and discharging voltage, correspondingly. Before charging of the cell in the CC mode by the indicated C-rate charging currents all cells were discharging by 1/2 C-rate constant current down to discharging cut-of voltage. The EMF curves for charging and discharging were extracted by mathematical extrapolation from the measured cell terminal voltage curves obtained after charging or discharging of the considered Li-ion cells as it was done in [22]. The entropy change (ΔS) curves for all considered cells were measured by a potentiometric method similarly as it was done in [23]. The ΔS profile was measured by adjusting the ambient temperature with a selected temperature profile. The chosen ambient temperature profile is 35, 25, 15, 5 and 35 °C. The temperature holding time is 2 hours. The ΔS was measured for each 10% of SoC in range between 10 and 90 %.

After characterization, all considered Li-ion cells were cycled at 45 °C until the capacity has decreased by 15%. The rest time between CC charging and discharging during cycling was 30 min. After approximately each 5% capacity decrease, the cycling of the cells was stopped, and characterization was repeated. Additional information about cycling tests parameters is given in Table II.

TABLE II. CYCLING TESTS PARAMETERS

Chemistry	Cycling tests parameters			
	Cut-off discharging voltage, V	Cut-off charging voltage, V	C-rate at CC discharging	C-rate at CC charging
NMC/C	3.0	4.2	1	1
NCA/C	2.5	4.2	1	0.5
NCA/C+Si	2.5	4.2	1	0.5
LFP/C	1.6	3.6	2	2

C. Differential curves measurements

The differential curves required for the ICA and DVA methods were obtained during charging of the considered Li-ion cells in the CC mode. The different C-rate charging currents were used for the differential curve measurements with purpose to evaluate the effect of C-rate current on the important features of the differential curves. For the energy Li-ion cells, the 1/10, 1/4, 1/3 and 1/2 C-rate constant charging currents were used, and 1/10 1/2 1/1 and 3/2 C-rate constant currents were used at the power Li-ion cell charging. Before charging, the Li-ion cells were discharged in CC mode by 1/2 C-rate current up to discharging cut-off voltage. The $\partial Q/\partial V$ and $\partial V/\partial Q$ curves were obtained by a numerical differentiation of the capacity and cell terminal voltage of the considered Li-ion cells during charging. Before numerical differentiation the considered data were filtered to remove noise as it was suggested in [24]. In addition to $\partial Q/\partial V$ and $\partial V/\partial Q$ curves, the EMF curve derivative ($\partial U_{EMF}/\partial Q$) was used in the DVA method as a reference curve for the analysis of the C-rate current effect on the important features of the $\partial V/\partial Q$ curves.

The modification of the DVA method was done with the help of the measured heat generation data during charging of the Li-ion batteries. The heat generated during the cell charging was estimated by using a cylindrical cell thermal model and the measured information about temperature at the cell surface and heat flux from this surface. The estimation of the heat generation during cylindrical Li-ion cell operation was previously presented in [25]. The heat generation curve obtained after cylindrical cell charging was modified by using a simplified form of the battery heat equation presented in [26]. Its differential is presented as:

$$\frac{\partial(\dot{Q}/I - U)}{\partial Q} = -\frac{\partial U_{EMF}}{\partial Q} + \frac{\partial(T \cdot \frac{\Delta S}{nF})}{\partial Q} \quad (1)$$

where $\dot{Q}$ is the heat generated in the Li-ion cell (W), I is the charging current (A), U is the terminal voltage (V), Q is the cell capacity (Ah), U_{EMF} is the EMF voltage (V), T is the cell temperature (K), ΔS is the entropy change (J/mol/K), F is the faraday constant (C/mol) and n is the stoichiometric number of electrons participating in the cell reaction (for a Li-ion cell, $n = 1$). Further, the term $(\dot{Q}/I - U)$ is denoted as H while $\partial H/\partial Q$ is called the differential heat curve.

III. RESULT AND DISCUSSION

A. Differential curves analysis

The measured $\partial Q/\partial V$ curves are presented in Figure 1 as a function of the terminal voltage for all considered cells. In addition, $\partial V/\partial Q$ curves and $\partial U_{EMF}/\partial Q$ curves are presented in Figure 2 as a function of the state of charge (SoC) of the Li-ion cells. The SoC was calculated by using the maximum cell capacity obtained for each of the considered Li-ion cells during EMF determination. The EMF curves used for the determining the maximum capacity were obtained after discharging the cells at different C-rates.

Fig. 1. Effect of the C-rate charging current on $\partial Q/\partial V$ curves of LFP/C cell (a), NMC/C cell (b), NCA/C cell (c) and NCA/C+Si cell (d).

Fig. 2. Effect of the C-rate charging current on $\partial V/\partial Q$ curves of LFP/C cell (a), NMC/C cell (b), NCA/C cell (c) and NCA/C+Si cell (d).

As expected, the analysis of Figs. 1 and 2 demonstrates the high influence of the C-rate value on the important features of the $\partial Q/\partial V$ and $\partial V/\partial Q$ curves. The $\partial Q/\partial V$ curve of the LFP/C is the most sensitive to the C-rate. The smallest changes of the peak position and intensity are noticeable on the $\partial Q/\partial V$ curve of the NCA/C+Si cell. Also, the change in the $\partial V/\partial Q$ curves for

this cell with increasing C-rate is small, which may be an effect of the silicon additive in the negative electrode. The energy density of silicon is much higher than the energy density of graphite, which allows to decrease the C/Si electrode thickness. Decreasing the electrode thickness increases the electrode maximum current density, which may explain the decreasing effect on the C-rate in the Li-ion cell overvoltage. In addition, the analysis of the differential curves shows that the identification of the peak, which is corresponding to the positive electrode and is close to the charging cut-off voltage, is not possible at high C-rate charging. This is well visible in Fig. 1 (c) and (d) where the $\partial Q/\partial V$ curve peaks at about 4.15 V noticeable at 1/10 C-rate current cannot be found at higher than 1/4 C-rate charging. This fact may complicate the determination of the degradation mechanisms associated with positive electrodes such as a loss of the active material of the lithiated positive electrode (LAM_{LiPE}). As the $\partial V/\partial Q$ curve peaks represent the phase transition locations instead of the phase plateau locations as in the $\partial Q/\partial V$ curves, the $\partial V/\partial Q$ curves have better visibility of the peaks at high SoC in case of the high C-rate charging. However, increasing the charging current leads to smoothing of the $\partial V/\partial Q$ curve important features, which complicates utilization of the $\partial V/\partial Q$ curves measured at high C-rate charging for determination of the degradation mechanisms. In general, we can conclude that the measurements of the $\partial Q/\partial V$ or $\partial V/\partial Q$ curves for all considered cells in the CC-charging mode should be done with lower than 1/10 C-rate current in order to use these curves for the determination of degradation mechanisms.

The analysis of the proposed modification of the DVA method is shown in Figure 3. The analysis was done by comparison of the $\partial V/\partial Q$ and $\partial H/\partial Q$ curves measured during charging of the Li-ion cells at the same C-rate. The 3/2 C-rate current was used for the LFP/C cell charging and 1/3 C-rate current was applied for the charging of other considered Li-ion cells. Moreover, the $\partial U_{EMF}/\partial Q$ curves, which are corresponding to $\partial V/\partial Q$ curves measured at Li-ion cell charging by a very small C-rate current, are also shown in Fig. 3.

As it can be seen in Fig. 3, the suggested modification of the DVA method allows the detection of the important features of the differential curve, required for the determination of the degradation mechanisms, even if a relatively high charging C-rate current is used for the differential curve measurements. The deviations of the $\partial H/\partial Q$ curves from $\partial U_{EMF}/\partial Q$ curves are caused by the ΔS change during Li-ion charging as it can be seen in equation (1) and in Fig.3. The analysis of the ΔS variation with increasing SoC shows that the positive change of the ΔS leads to a bigger value of the $\partial H/\partial Q$ curve than the $\partial U_{EMF}/\partial Q$ curve value. Similarly, the negative change of ΔS with increasing of SoC leads to a smaller value of the $\partial H/\partial Q$ curve than the $\partial U_{EMF}/\partial Q$ value. If ΔS does not change significantly with increasing SoC, the $\partial H/\partial Q$ curve is almost corresponding to the $\partial U_{EMF}/\partial Q$ curve. Such dependence of the $\partial H/\partial Q$ curve on ΔS change may be considered as an additional advantage of the proposed modified DVA method as the ΔS change with Li-ion battery aging may be estimated from the $\partial H/\partial Q$ curves.

Fig. 3. $\partial U_{EMF}/\partial Q$, $\partial V/\partial Q$, $\partial H/\partial Q$ and entropy change curves of LFP/C cell (a), NMC/C cell (b), NCA/C cell (c) and NCA/C+Si cell (d).

B. Determination of Degradation mechanisms

The determination of degradation mechanisms of the considered Li-ion cells was done by analysing the $\partial U_{EMF}/\partial Q$ curve variations as a function of aging of the Li-ion cells. The $\partial U_{EMF}/\partial Q$ curves were created by a numerical differentiation of the EMF curves obtained after charging the Li-ion cells with different C-rate currents. The $\partial U_{EMF}/\partial Q$ curves at different ageing stages are shifted up for the purpose to improve the visibility of the curve important features changes upon Li-ion battery ageing. The considered $\partial U_{EMF}/\partial Q$ curves are shown in Figure 4. Moreover, all peaks in the $\partial U_{EMF}/\partial Q$ curves for all considered Li-ion cells, except of NCA/C+Si cell, are aligned with respect to the peak β , which are denoted by a green colour. The peaks of the differential curves for NCA/C+Si cell are aligned with respect to the peak α , which is denoted by a red colour.

Fig. 4. $\partial U_{EMF}/\partial Q$ curves for different ageing stages of LFP/C cell (a), NMC/C cell (b), NCA/C cell (c) and NCA/C+Si cell (d).

The analysis of the degradation mechanisms by using $\partial U_{EMF}/\partial Q$ curves showed that a loss of the active material of the lithiated (LAM_{LINE}) and delithiated (LAM_{deNE}) negative electrode are probably the dominant degradation mechanisms for the considered LFP/C cell. The indication of these degradation mechanisms is well seen in Fig. 4 (a) as the α peak is moving closer to the β peak upon ageing [14], [27]. The moving of the right end of the differential curve to the β peak is

probably caused by LAM_{LINE} and loss of the lithium inventory (LLI). The detection of the LLI by analysing the $\partial U_{EMF}/\partial Q$ curves changes is difficult in the considered case as the effect of the LLI on the considered differential curve is probably decreased by the effect of the LAM_{deNE}. Similar degradation mechanisms are observed for the NMC/C cell. However, smoothing the α and β peaks is also noticeable during NMC/C cell ageing. The smoothing of the α peak is probably caused by LAM_{LINE} [14]. The smoothing of the β peak may be caused by the structural degradation of the negative electrode. The analysis of changes of the $\partial U_{EMF}/\partial Q$ curve important features during NCA/C cell ageing showed that both electrodes are degraded during cell operation at the investigated conditions. The moving of the α to the β peak is caused by loss of the active material of the negative electrode. An addition the γ peak, which belongs to the positive electrode [28], moves closer to β peak, which indicates the LLI during the NCA/C cell ageing. The degradation of the positive electrode is indicated by decreasing α and γ peaks, which may lead to the disappearance of these peaks with further ageing [14]. The degradation of the NCA/C+Si cell is slightly different than the degradation of the NCA/C as it can be seen in Fig. 4 (d). The $\partial U_{EMF}/\partial Q$ curve of the NCA/C+Si cell has two additional peaks (δ and ϵ), which are probably attributed to the silicon. The distance between α and β peaks does not change with cell aging. This allows concluding that the degradation of the graphite in the NCA/C+Si cell is not significant. However, the moving of the ϵ peak closer to the α peak is well seen in Fig 4. (d). Such a change of the differential curves may be explained by the potential changes of silicon during its degradation. This was demonstrated in [29]. Moreover, significant moving of the γ to the β peak indicated that the LLI may be considered as the dominant degradation process in the NCA/C+Si cell. Continuous consumption of lithium because of SEI formation is caused by continuous side reactions at the silicon/electrolyte interface because of the repeated silicon volume expansion and contraction [29].

Fig. 5. $\partial H/\partial Q$ curves at for different ageing stages for LFP/C cell (a), NMC/C cell (b), NCA/C cell (c) and NCA/C+Si cell (d).

The changes of the $\partial H/\partial Q$ curves upon aging are shown in Figure 5. The $\partial H/\partial Q$ curves were measured during charging of the cells in the CC mode. The LFP/C cell was charged by 3/2 C-rate current and the 1/3 C-rate current was used during charging of other considered Li-ion cells. Similarly, as it was done at $\partial U_{EMF}/\partial Q$ curves analysis, the $\partial H/\partial Q$ curves were shifted up and aligned with respect to the α and β peaks for the NCA/C+Si and other considered Li-ion cells, correspondingly.

Comparison of the changes in $\partial U_{\text{EMF}}/\partial Q$ and $\partial H/\partial Q$ curves during ageing of the considered Li-ion cells showed that the detected degradation mechanisms during $\partial U_{\text{EMF}}/\partial Q$ curve analysis in general can be obtained after $\partial H/\partial Q$ curves analysis. The behaviour of all peaks of the $\partial H/\partial Q$ curves was corresponding to the behaviour of the $\partial U_{\text{EMF}}/\partial Q$ curve peaks despite the relatively high C-rate charging current was used during the measurements of the $\partial H/\partial Q$ curves. Such advantage of the DVA method modification is considered as significant and can simplify the applicability of this method in real systems. Therefore, the use of the presented $\partial H/\partial Q$ curves in determining the degradation mechanisms is recommended for the application in real systems such as, for example, in EVs.

IV. CONCLUSIONS

The modification of the DVA method, which permits to use a relatively high C-rate charging current for the differential curves measurements, is presented in this article. The modification of the DVA method was done by accounting the heat generated in the Li-ion batteries during operation. The information about the generated heat was obtained from the heat flux and temperature measurements and with help of the cylindrical cell thermal model. The applicability of the modified DVA method in determining the degradation mechanisms was verified for four different Li-ion chemistries. The verification was done by comparison of the results obtained from the $\partial U_{\text{EMF}}/\partial Q$ curves analysis method and results obtained from the presented modification of the DVA method. A good correlation of the results obtained from the $\partial U_{\text{EMF}}/\partial Q$ curves analysis method and presented modification of the DVA method was observed. The demonstrated advantages of the MDVA method make it recommendable to application in real battery powered systems.

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