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► **To cite this version:**

Maxime Juston, Nicolas Damay, Christophe Forgez. Extracting the diffusion resistance and dynamic of a battery using pulse tests. *Journal of Energy Storage*, 2023, 57, pp.106199. 10.1016/j.est.2022.106199 . hal-03942882

HAL Id: hal-03942882

<https://hal.science/hal-03942882v1>

Submitted on 17 Jan 2023

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Extracting the diffusion resistance and dynamic of a battery using pulse tests

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Abstract

The modeling of a battery by an equivalent circuit model requires the determination of its parameters. This determination can be done by exploiting the transient response of a battery to a current pulse (often called GITT: galvanostatic intermittent titration technique). A classical approach is to first separate the open circuit voltage (OCV) and the overvoltage, and then to extract the model parameters from the latter. However, the estimation of the OCV can be difficult, which can lead to errors on the overvoltage, especially for slow dynamics such as diffusion. We propose here a method to estimate the OCV during GITT and a method to estimate the overvoltage which allow the extraction of parameters associated with slow dynamics. The results brought by the proposed method are compared with more classical approaches.

DOI : <https://doi.org/10.1016/j.est.2022.106199>

Keywords: Battery ; electrical characterization ; open-circuit voltage ; GITT ; diffusion

1. Introduction

Modelling a battery using an equivalent circuit model requires the determination of parameters that represent the physical phenomena taking place [1, 2]. The most classical method for determining the parameters is the use of current pulses, also called GITT (galvanostatic intermittent titration technique) [3, 4]. This method uses the transient response of the battery to characterize the model. However, this method faces two difficulties which are the estimation of an OCV during the test, and the exploitation of the resulting overvoltage which may not correspond to the theory. To estimate the OCV, the classical approaches are to use a linear interpolation along the current pulses or to use a so-called “pseudo-OCV” curve that is measured independently by applying a very low current. While the pseudo-OCV gives an estimate with an accurate shape, it requires more tests than a linear estimate. Several tests may be needed to consider the shape modification of the OCV due to: a different electrode balancing [5, 6, 7]; an heterogeneous operation inside the cell [8] and the hysteresis phenomenon between charge and discharge [9, 10, 11]. Determining pseudo-OCV curves for all these conditions can be long and may not be suited for certain applications.

In the case of a battery with a non-linear OCV, the use of simple methods such as linear interpolation does not allow satisfactory overvoltage estimates. Figure 1 illustrates

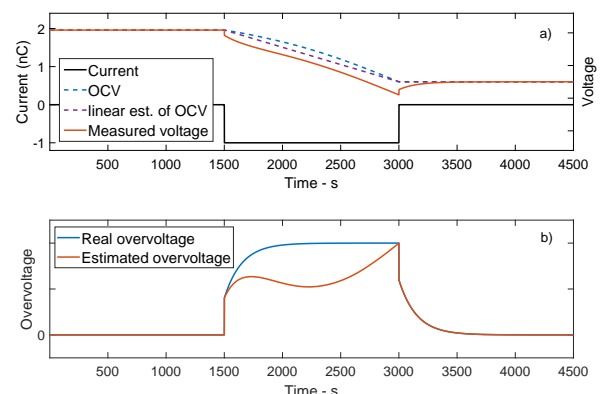


Figure 1: a) Simulation of the voltage response of a battery to a current pulse. b) Real and estimated overvoltage in the case of a linear estimation of the OCV.

this by presenting a voltage simulation and the overvoltage estimated by using a linear estimation of the OCV. The estimated OCV differs from the real overvoltage in shape, and cannot be used to determine electrical parameters.

The difficulty of the overvoltage estimation lies initially in the estimation of the open circuit voltage of the battery. A small error in proportion, a few mV, on the OCV estimate can lead to a large error on the overvoltage, which has an order of magnitude of the mV. Especially, the shape modification of the overvoltage can greatly impact the extracted parameters, as illustrated in figure 1. Several phe-

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nomena make it complex to estimate precisely the OCV. It has been proved by experiments that the following parameters vary the open circuit voltage of a battery [12]:

- the temperature;
- the immediately preceding operations of the battery, by hysteresis effect;
- age and state of health of the battery;
- history of the battery (all the operations of the battery during its life).

The estimation of the OCV during GITT is thus a difficult but important step toward a correct overvoltage and a correct model.

In this paper we present a new method to precisely estimate the OCV during a GITT. We also present a new method to estimate the overvoltage that allows the determination of the parameters of an electrical circuit model (ECM). The main interest is the modelling of the diffusion phenomena, which have the largest time constant among all the phenomena taking place in an operating battery, and which are particularly sensitive to the estimated overvoltage shape.

In section 2 we will present the proposed method for separating the OCV and the overvoltage during GITT tests. In section 3 we will present the application of the proposed method to two lithium-ion batteries, compare it to the linear OCV estimation and to the pseudo-OCV measurement and then discuss the results. In section 4 we compare the parameters obtained for our model based on the three OCV estimation methods and discuss the validity and consistency of those results.

2. Proposed method to separate OCV and overvoltage

2.1. Definition of an equivalent OCV

By construction, the ECM are often made with several components that represent the cell overvoltage and with a voltage source that represents the OCV of the battery, measured at the thermal and electrochemical equilibrium [4, 13]. This definition of the OCV at equilibrium implies that any voltage variations from it must be included into the cell overvoltage.

We observed in our research team that this feature can lead to inconsistencies. For instance, the OCV shape is often less visible when current is getting higher, as if the OCV curve was smoothed. We found that this is potentially due to a heterogeneous operation of the cell [8]. We will demonstrate in this paper that this smoothing of the OCV shape should be modeled has a apparent change of the OCV curve itself, instead of a change of the overvoltage (thus a change of the impedance for an ECM). This apparent changes of the OCV curve lead to what we call an “equivalent OCV” OCV_{eq} .

2.2. Definitions of transient and quasi-steady states overvoltages

The exploitation of current pulses to determine parameters aims at better understanding and characterizing the transient operation of a cell from a relaxed state to an operating state. Our measurements have shown that, given enough time, the voltage curve from current pulses joins the trace of a voltage curve from a constant current discharge at the same regime, as presented in figure 2a. On these figures are displayed the traces from a constant current discharge and from a current pulse discharge. Each pulse is 480 sec long at 1C, which is approximately 13% DoD. It can be seen that after 480 seconds the traces are the same.

The overvoltage is a quantity that is used on ECM to represent various electrochemical effects such as the charge transfer, the diffusion or the electrolyte concentration gradient. Those phenomena depends on the current, the temperature, the state of health and the state of charge [14]. The physical phenomena such as the double-layer capacity or the solid and/or liquid diffusion are dynamic with a time-constant that can be characterized by EIS. Thus we believe that after a certain duration the diffusion gradients are the same in the active particles and in the electrolyte [15]. In this case, the overvoltages values of two discharges curves should be the same under the same experimental conditions (current, temperature, SoC, SoH): concentrations gradients are established.

Some time is required for all the phenomena to reach this state, but if we consider a battery in this quasi-steady state then we can remove time from the expression of the overvoltage $\eta_{GITT}(t, I, T, SoC)$, such as in Eq. 1:

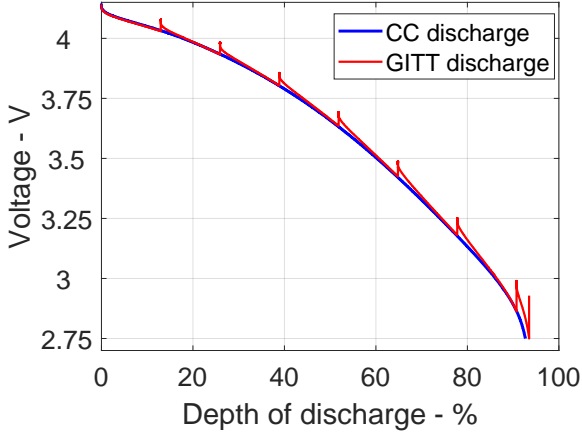
$$\eta_{GITT}(t, I, T, SoC) \xrightarrow{t \rightarrow +\infty} \eta_{GITT, qss}(I, T, SoC) \quad (1)$$

This assumption is supported by the fact that the traces of the constant current and pulse current discharges are overlapping after a certain duration: the steady-state is reached. Thus the overvoltage at the end of each pulse $\eta_{GITT, qss}$ is at the same level than the overvoltage from the constant current discharge (Eq. 2).

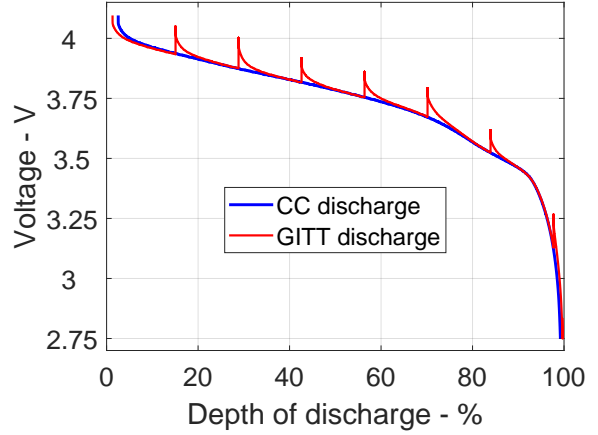
$$\eta_{GITT, qss}(I, T, SoC) = \eta_{CC}(I, T, SoC) \quad (2)$$

The value of this overvoltage is called here the quasi-steady-state overvoltage. Let us note η_{qss} this value. Recently, Chen *et al.* [16] also demonstrated that the concentration gradients in the electrolyte may change with the state of charge, leading to variations of the quasi-steady-state overvoltage with SOC. Given this, a constant current discharge is therefore a measure of the OCV and the quasi-steady-state overvoltage generated by the application of a current I as presented in the equation 3

$$U_{CC, I} = OCV + \eta_{qss, I} \quad (3)$$



(a) Voltages measured during the discharge of a cell "A"



(b) Voltages measured during the discharge of a cell "B"

Figure 2: Traces of voltages measurements for discharges at constant current (CC) and by GITT

Provided that the OCV is the same for a constant current discharge and a discharge from a GITT over the same SoC range, we can also write the equation 4 for each GITT. They allow us to observe a transient regime towards the overvoltage in quasi-steady-state. We note the overvoltage during the GITT $\eta_{transient,I}$. The transient overvoltage, which is usually called "overvoltage", allows us to characterize the behaviour of the cell from a relaxed state to a polarized state. The quasi-steady-state overvoltage, on the other hand, allows us to assess only the value of the overvoltage in the polarized state.

$$U_{GITT,I} = OCV + \eta_{transient,I} \quad (4)$$

2.3. Theoretical development

It is then possible to define the transient overvoltage as a quantity that no longer requires the determination of an OCV, but by a difference between the voltage U_{CC} and the voltage U_{GITT} thanks to the equation 5.

$$U_{GITT} - U_{CC} = \eta_{transitory,I} - \eta_{qss,I} \quad (5)$$

This we can rewrite to obtain the equation 6.

$$\eta_{transitory,I} = U_{GITT} - (U_{CC} - \eta_{qss,I}) \quad (6)$$

In order to exploit this relation, we need to determine the quasi-steady-state overvoltage. Its value, η_{qss} , is indirectly measured during the relaxation phase. Indeed, this phase represents the transition from the polarized state to a relaxed state. Independently of the dynamics of this relaxation, the voltage difference between these two phases gives us a value of the quasi-steady-state overvoltage for each of the GITT, noted $\eta_{qss,meas}$. The dynamic of the relaxation was not studied because it is different from the

polarization and requires a specific modeling [17]. By interpolating these overvoltages measurements with respect to the SOC during the GITT, we can obtain an estimate of the overvoltage for any intermediate state of charge $\eta_{qss,est}$. This interpolation is made with a polynomial of order 3 between each measured point to ensure a consistency between each measure. It is therefore possible to define the transient overvoltage thanks to these three quantities which are U_{CC} , U_{GITT} and $\eta_{qss,meas}$ the quasi-steady-state overvoltage measured during the relaxation phases.

In order to return to a more classical definition of the overvoltage, we propose to define an equivalent OCV as in eq. 7.

$$OCV_{eq,I} = U_{CC} - \eta_{qss,I} \quad (7)$$

which allows to rewrite the equation 6 into the equation 8. This term of equivalent OCV makes it possible to specify that what we estimate is not what we could define as a OCV, i.e. a measurement of the voltage in a relaxed and homogeneous state. This OCV is measured for a current regime, of value I, and corresponds to the voltage which allows us to find the values of U_{CC} when we add the measured overvoltage $\eta_{qss,meas}$. This equivalent OCV has a form that depends on the current, and the current regime is also indicated as a subscript to show the conditions for determining this OCV.

$$\eta_{transient,I} = U_{GITT} - OCV_{eq,I} \quad (8)$$

In contrast to a linear estimation of the OCV, the use of the voltage U_{CC} to estimate an equivalent OCV allows to keep an acceptable form of OCV. This method is also faster than a measurement of a pseudo OCV since the equivalent OCV estimation does not require to tend to a

relaxed state at each instant. Thus, this method seems to be a good compromise between the use of a linear interpolation, which is fast but not very accurate, and the use of a pseudo-OCV which is long and is bound to be outdated when the OCV changes.

3. Application to a Li-ion cell

In order to test and compare the results of parameter extraction according to the OCV estimation method, we performed CC and GITT discharges on 2 types of batteries. Battery A is a power battery and battery B is more of an energy battery. These batteries have different chemistries, and therefore different OCV shapes. Battery A has a smooth OCV with little variation in slope, while battery B has an OCV with highly non-linear areas.

3.1. Experiments performed and experimental protocol

The experimental protocol consists of a charge at constant current and then at constant voltage (CC-CV) up to the maximum voltage given by the manufacturer, followed by a 2h relaxation period. A discharge at constant current until the minimum voltage given by the manufacturer is then carried out. After 2h of relaxation, the battery is recharged according to the same protocol as before. 2h of relaxation are again observed before carrying out partial discharges at constant current (GITT) for a time of 8 minutes, i.e., 480 seconds. This time was chosen to be able to observe overvoltage whose time constant is of the hundreds of seconds. It also makes it possible to highlight the phenomenon of variations of the steady-state overvoltage value. When the battery reaches its minimum voltage during a GITT, a period of relaxation of 2h is observed and the test is stopped. Figure 2 show the measurement from this protocol.

The pseudo OCV used in this article was measured at C/100 under constant current in a climatic chamber at 25 °C.

All the other tests are performed at 1C and 20 °C. They take place in a climatic chamber regulated in temperature, and the cell are tested one by one, to keep them at the regulated temperature as much as possible. Under the hypothesis of a low heating and a good thermal dissipation, which should be reasonable due to the experimental protocol presented in the this section, the temperature can be considered constant. The state of health will be considered identical because the tests (CC discharge and GITT discharge) are done consecutively. Based on this we considered an overvoltage that depends on current and SoC only in section 1.

3.2. Results

The estimate of the three different OCV estimations for cell B are shown in the figure 3. The three methods

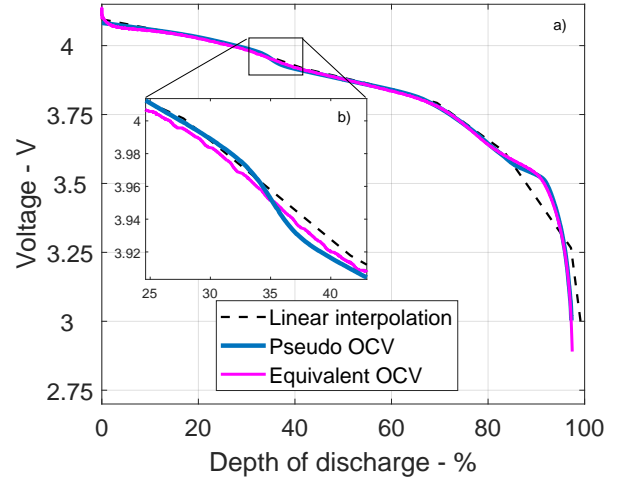


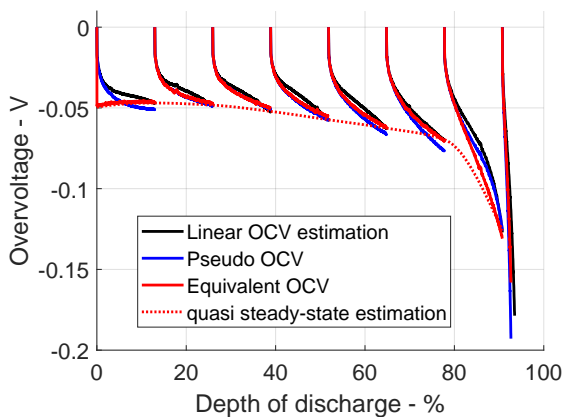
Figure 3: a) Estimated OCV from a linear interpolation, a pseudo-OCV and the equivalent OCV obtained using proposed method. b) Zoom around 35% DoD.

show globally the same shape, there are differences in areas where the OCV is non-linear. The linear estimation of the OCV can be very different in these areas. A zoom on the OCV shapes around the depth of discharge (DoD) 35 % is shown on figure 3b. It can be seen that the equivalent OCV - estimated thanks to the proposed method - is smoothed compared to the pseudo-OCV (see section 2). This smoothing is also visible at for higher DoD. We will see now that overvoltages estimated thanks to these three OCV can greatly differ.

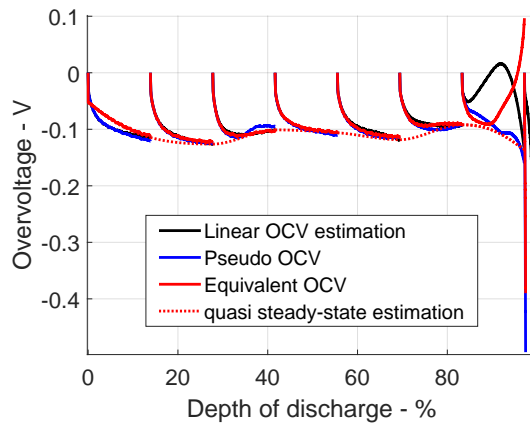
Our estimates of overvoltages are presented in figures 4a and 4b for type A and B cells respectively. However, the results are not satisfactory, especially in terms of the shape of the overvoltages, which are different from what is expected in theory, despite our observations on the voltages measured. The overvoltages do not seem to converge towards a limit value but increase without any significant sign of a limit value. As the electrical model, presented later, used in this work has been used in several publications [18, 19, 20], it is considered more robust than the overvoltage estimate. Therefore, these results do not question the model itself but our way of estimating the overvoltages.

3.3. Discussion

In figures 4a and 4b, the overvoltages were simply defined as in equation 8. However, as we saw when we constructed the equivalent OCVs, the quasi-steady-state overvoltage $\eta_{qss,I}$ is a quantity that has variations with the SoC. $\eta_{qss,I}$ has been reproduced on figures 4a and 4b to demonstrate that the transient overvoltage $\eta_{transient,I}$ tends toward $\eta_{qss,I}$ for cells A and B. This effect is particularly strong for cell B, because its OCV is very non-linear.



(a) Overvoltages estimated for cell A



(b) Overvoltages estimated for cell B

Figure 4: Overvoltages estimated with the different OCV estimation methods & estimated quasi-steady-state overvoltage for both cells

From figure 4b, we can see some benefits of the proposed method for estimating the equivalent OCV. The overvoltage appears to tend smoothly towards the quasi-steady-state, needing almost the same duration to reach it for each of the GITT pulses. To the contrary, the overvoltages estimated by the other methods (linear estimation and pseudo-OCV), appear to be less robust: they are correct for linear parts of the OCV, but they are skewed for cell B around DoD 35 %, 60 % and 80 % (see this cell OCV on figure 3). Finally, the overvoltage estimations after DoD 80 % are all bad. This can be explained by the complexity of the cell behavior in this operation range, mixing several effects due to heterogeneity and diffusive phenomena [8, 21].

To better observe the diffusion dynamic, we therefore propose to divide the transient overvoltage obtained by the estimated value of the quasi-steady-state overvoltage. This gives a dimensionless signal, whose value is between 0 and 1, and which is defined by the equation 9. In this equation the OCV term represents the chosen estimate of the OCV (linear, measured pseudo-OCV, equivalent OCV).

$$\eta_{adim} = \frac{U_{GITT} - OCV}{\eta_{RP,est}} \quad (9)$$

This method allows us to obtain the signals presented in figure 5a for a cell of type A and in figure 5b for a cell of type B. We observe a change in shape in both cases. The overvoltages have more regular responses and are closer to the expected overvoltage shape (see figure 1). The comparison of the overvoltages shows that the proposed method allows us to have overvoltages that can be modelled with the chosen electrical circuit, presented in the next section. In the case of a linear estimation of the open circuit voltage, the selected electrical circuit cannot model it, which leads to important errors. Although the final values of the

overvoltages are comparable for the first overvoltages, the shape of makes it difficult to automatically optimize the circuit parameters. The direct consequence is either an error on the value of the overvoltage or an error on the dynamics of the battery response. For the type A cell, the contribution of this method is lighter but still leads to parameters without outliers, which is the case for the estimation based on a linearly estimated OCV.

Regarding the first GITT pulse, the proposed method cannot be applied because the GITT and CC discharge curves have approximately the same trace, thus our theoretical development gives for this peculiar case:

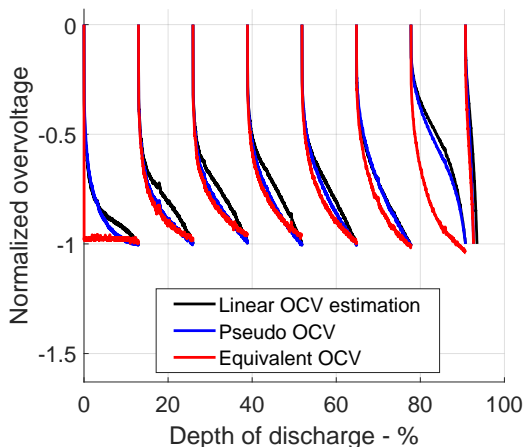
$$\eta_{adim} = \frac{U_{GITT} - U_{CC} + \eta_{qss,I}}{\eta_{RP,est}} \approx \frac{0 + \eta_{qss,I}}{\eta_{RP,est}} = 1 \quad (10)$$

4. Extraction of parameters

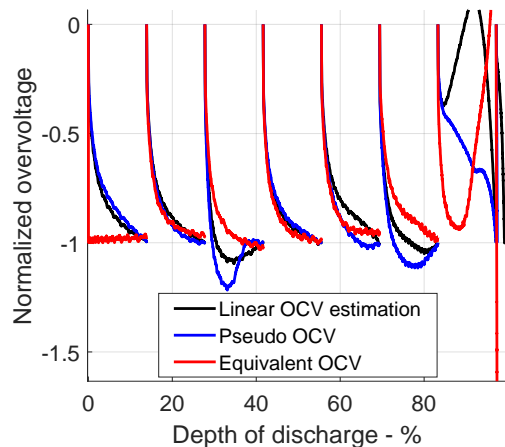
4.1. Electrical model of a battery

A battery is an imperfect source of voltage that varies according to the current delivered. The simplest model of a battery is therefore a voltage source coupled to an impedance that models the effects of the current on the voltage. This impedance must model the overvoltage that will be seen at the terminals of the battery by the external system. Physical considerations [22, 23, 24, 18, 19, 20] have led to equivalent circuit models of the U-R-RC-nRC type as shown in figure 6.

A voltage source U_{OCV} represents the open-circuit voltage of the cell, as defined in section 2. The overvoltage of the cell is modeled by several components. A first resistor R_s represents an instantaneous overvoltage that comes to model the contributions of the current collectors, the tabs, the electronic flow in the active material as well as



(a) Normalized overvoltages for cell A.



(b) Normalized overvoltages for cell B.

Figure 5: Normalized overvoltages for the three methods.

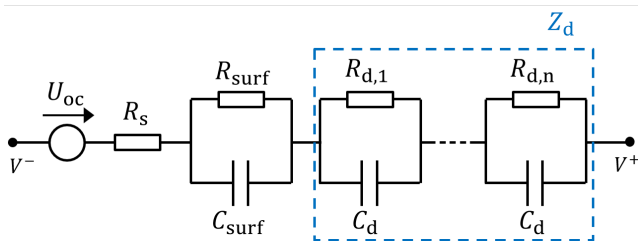


Figure 6: Equivalent circuit model of a battery that allows to model the main phenomenon.

the various connections from the battery to the system in which it operates. Then a surface resistance R_{surf} associated to a surface capacitance C_{surf} are linked to a fast dynamic. The surface resistance models the reactions at the electrode/electrolyte interfaces, as well as the possible contribution of a SEI-type layer on the electrode surface. Finally, on a slower dynamic, of the order of a hundred seconds, the solid and liquid diffusion phenomena are modelled by a number n of $R_d C_d$ circuits, in theory infinite. These RC circuits approximate a specific impedance Z_d related to these phenomena, which is in general a Warburg impedance [24]. In practice, to avoid large computation times, this number is limited. In this paper, which focuses on a new method of parameter extraction more than on the accuracy of the modelling, we have chosen to use only one RC circuit to model the effects of diffusion.

Such a model has a response to a current pulse that is constituted of several, 2 here, RC charges until a steady-state is reached. Studies [25, 14] have shown that all the resistive phenomena of a cell depend on the state of charge. The steady-state value that is normally reached is then here a quasi-steady-state which vary with the SoC, as observed in our measurement presented in section 2.

In this paper we used an algorithm that seeks, for each overvoltage curve, the best set of parameters to minimize an error function between the estimated (*via* measurement) and simulated overvoltage. The simulated overvoltage comes from the chosen equivalent circuit model. Regarding the value of the series resistance, their values are low, and the optimization was struggling to determine them, or sometimes returned negatives values to have a better fit. To avoid this, we used an EIS to determine the values at different state of charges and fixed the value in the optimization algorithm. It appeared that the serial resistance value is almost invariant to a SoC change, which leads to the same value being used for all optimizations.

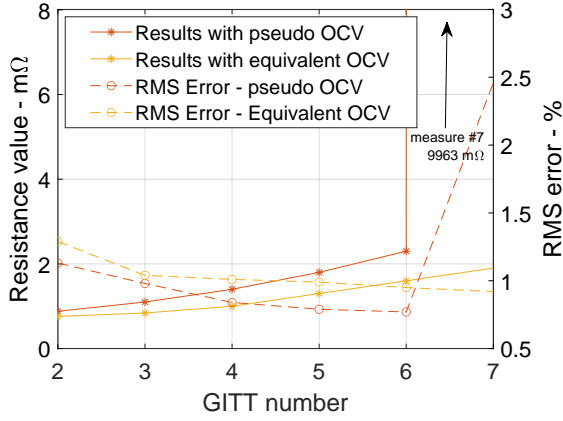
4.2. Results of ECM fitting

The diffusion results are the most influenced by shape variation, so we only present here the results for the resistance diffusion on the figure 7 and the time-constant diffusion on figure 8.

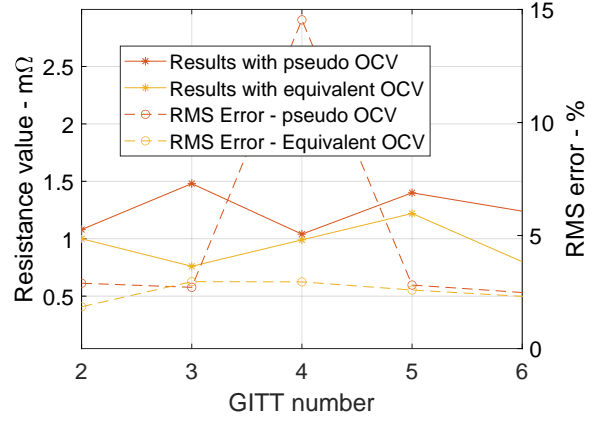
5. Analysis

5.1. Signal shape

For the two batteries studied, and for all overvoltages, the proposed method provides a signal shape can be modelled by the selected electrical circuit. Compared to a simple piecewise linear estimation of the OCV, we observe an improvement, especially in the case of non-linear OCV, such as that of battery A, or that of battery B during plateau transition. This phenomenon is particularly marked on the figure 5b for overvoltages 3 and 5, 6 & 7. Before a plateau transition, we see an increase in the equivalent resistance of the battery, and then a decrease after the plateau transition. Where an overvoltage will be poorly defined because of these variations in estimated

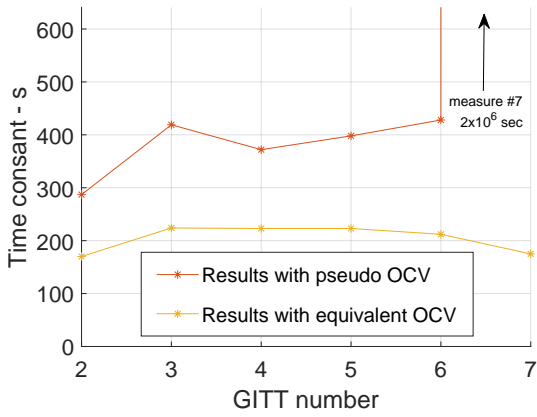


(a) Diffusion resistances fitted for cell A.

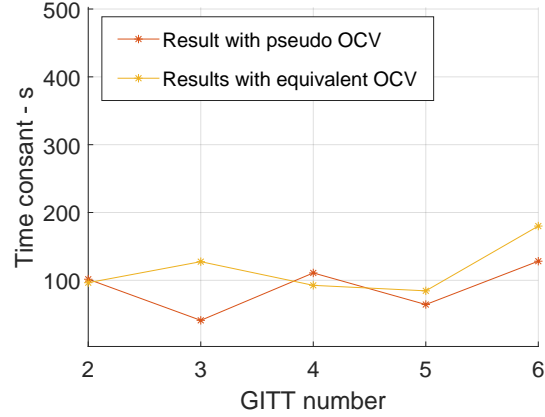


(b) Diffusion resistances fitted for cell B.

Figure 7: Diffusion resistances fitted for normalized overvoltages with a pseudo OCV and the proposed method.



(a) Diffusion time constants fitted for cell A.



(b) Diffusion time constants fitted for cell B.

Figure 8: Diffusion time constants fitted for normalized overvoltages with a pseudo OCV and the proposed method.

OCV, the signal we propose to study has a form that can be used for parameter determination.

The applied shape correction requires to estimate the steady-state overvoltage for the range of SoC covered during the GITT. However, the variations obtained allow notable improvements, such as for example the slot n°7 of battery A (figure 4a) which becomes exploitable on figure 5a.

5.2. Value of the model parameters

We also observe that an electrical model applied to classical surges converges to inconsistent solutions, especially on the diffusion parameters with values several orders of magnitude above the values found with the pro-

posed method or above a coherent value. The richness of the chosen electrical circuit allows us to propose a model even though it is physically incorrect. Although the RMS errors are comparable for the three methods, and of the order of percent, the value of the parameters allows us to discard the linear estimation method of the OCV.

The extremely large time constant values found for the battery A for a linear estimation of the OCV imply that the curves of the voltages U_{CC} and U_{GITT} should not have the same trace after a few hundreds of seconds, which we observe. For battery A, even the time constant values obtained by using a pseudo-OCV are about 400 sec, which would imply that the traces displayed on figure 2a should not be the same after only 480 seconds. The proposed method allows us to obtain lower values, of the order of

two hundred seconds for the diffusion time constant, which allow us to validate the concept of steady-state overvoltage and is coherent with the graphic observations.

The values of the parameters obtained for an estimation of the OCV by a pseudo-OCV, and the proposed method give results which are globally similar. However, the use of a pseudo-OCV requires an accurate estimation of the OCV and faces problems of sustainability in time as said in the introduction. The open circuit voltage of a cell is a quantity which evolves with the ageing of the cell, and modifications of form can appear. This operation of determination of the OCV is long [8] and has to be repeated in a regular way. The proposed method makes it possible to avoid this operation by proposing an estimation at an identical state of health thanks to a discharge with constant current, while giving similar results.

Besides, we found that the diffusion time constant determined thanks to our approach does not depend on the SoC for the studied battery. It may be explained by the fact that this time constant represents the dynamic of the lithium concentration gradients creation inside the active materials and the electrolyte during operation [15]. The creation of this gradients would thus have the same speed regardless of the SoC, but the effects of these gradients would be depending on SoC (namely the diffusion resistance whose shape varies in relation with the OCV shape).

6. Conclusions

We proposed a characterization method that allows the extraction of the diffusion resistance and time constant of a LIB battery. This method requires GITT and constant current tests.

The proposed method is based on a more realistic separation of the battery OCV and overvoltage. It has been tested on two different chemistries and gives reliable results, both on areas where the OCV is relatively linear and on areas where it is strongly non-linear. It brings results that seem more consistent with the electrochemical phenomena occurring inside a battery.

The benefits of the proposed approach are the characterization of the diffusion resistance as a nearly continuous function of the SoC and a more accurate determination of the diffusion time constant compared to the classical approach (that is linear interpolation of the OCV between GITT pulses).

Acknowledgement

The authors would like to thank the ANRT for its financial support (No. 2017/0855), SNCF and Bogdan Vulurescu for their participation to the study.

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