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Research Papers



Experimental assessment of high-energy high nickel-content NMC lithium-ion battery cycle life at cold temperatures

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ABSTRACT

An essential element for the diffusion and environmental impact assessment of electrochemical storage systems is their lifetime. This lifetime also impacts the overall cost of the equipment in which the storage system is used. At the same time, a number of applications, e.g. electric vehicles, do not allow a temperature control of the batteries, although the performances of the latter are very strongly linked to them. The phenomena investigated, such as aging, behavior at low temperature and rapid charge for low temperature, are complex and difficult to model. Only the establishment of databases representative of the conditions of use can lead to elements of response. The exploitation of these databases by artificial intelligence tools is certainly relevant but requires a learning on data representative of the conditions of use and of the phenomena to be evaluated.

The work presented in this paper is in this context and is intended to model the influence of the rapid charge at low temperature (0 °C, -10 °C, -20 °C) on the lifetime (initial capacity loss) of lithium-ion NMC elements. The problem with data-based approaches is the large number of tests required and the development in relevant experimental designs. Faced to this challenge, a consortium of French laboratories and manufacturers has been working together since the 2000s to carry out this type of work. The paper proposes to present a test campaign and summarizes the main results to assess the service life (loss of capacity) for a fast-charging application at low temperature and position them against the manufacturer data established at 25 °C. The impact of two partial discharge processes (50 %) will also be assessed. In a last part, we show that for low temperatures, even for the nominal charge current the life time is reduced to few dozens of cycles and a mechanical destruction of the cell with no activation of the current interrupt device is achieved. This study demonstrates and quantifies the very important impact of low-temperature charging processes on the lifetime of high energy lithium-ion NMC batteries.

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1. Introduction

Reducing the environmental footprint of our lifestyles requires an optimization of energy use and a deployment of renewable energy sources. Electricity appears to be the most appropriate energy vector for successfully carrying out the ecological and energy transitions, which have been underway for several years and are inseparable. The issue of electricity storage is a central point in the optimization of energy use [1]. This explains the massive use of batteries, particularly lithium-ion batteries. These batteries, which were already widely used in embedded devices (telephony, computers, etc.), are now increasingly used in automotive applications [2]. Among the challenges that battery technologies must face, the control of the life span is essential. This has a direct influence on the economic model and the environmental impacts.

Any analysis of lithium-ion battery aging is based on experimental data that are necessary to validate numerical aging models. These models can be used for system sizing [3], optimal management [4,5] or life cycle prognosis [6]. They are also a precious source of inputs for defining thermal management constraints. Especially in automotive industry, where autonomy and charging speed are of primary interest. Maximizing both parameters with an optimized thermal management system while affecting the lifecycle into the lowest possible extent, appears as crucial.

The study of lithium-ion battery aging is confronted with the following specificities:

- Complexity and diversity of aging mechanisms, which lead to a high sensitivity of aging to usage profiles and conditions of use.
- The relative youth of the industrial production, which results in a multiplicity of available technologies and a permanent evolution of performance.
- The charge for low temperature conditions may induce a lithium plating phenomenon and so, may reduce dramatically the battery life span.

The first point means that an aging test campaign must use an experimental design containing a large number of conditions. This requires the mobilization of a large amount of equipment for times of the order of a year.

The second point means that these costly test campaigns must be repeated for each battery family and that the experimental data collected must be updated regularly.

As a result, there is an obvious interest in pooling experimental battery aging campaigns. This starting point led to the creation of the COMUTES² consortium. The results of its first test campaign are described below. Chapter 2 recalls the genesis of the COMUTES² project. Chapter 3 describes the experimental design carried out. Chapter 4 presents the results obtained.

2. The Comutes² project

The study of the aging of electrochemical batteries in the field of electrified vehicles was quickly identified as a major element for the diffusion of this type of vehicle.

The French government, through its national research agency (ANR), quickly identified this problem and launched calls for projects in the 2000s. It quickly became apparent that it was necessary to mobilize French manufacturers and academics around this issue. In addition, it was important to have a large number of test facilities in order to carry out significant experimental studies and propose relevant aging models.

In 2005, a consortium of automotive manufacturers (Renault, PSA, VALEO), battery and super-capacity manufacturers (SAFT and BATS-CAP), energy and engineering companies (EDF, Imagine), research organizations (CEA, IFP, INRETS) and university laboratories (Amiens, Bordeaux, Compiègne and La Rochelle) combined their skills to propose a “laboratory without walls”, thus pooling their scientific resources for

an ambitious and well-defined task.

This first project (SIMSTOCK) proposed over 3 years and for an amount of 4.1 M€ aimed at characterizing Li-Ion, Ni-MH and super-capacity batteries for “hybrid electric vehicle” applications. This SIMSTOCK [7,8] project has allowed to model the storage elements for significant cycling operation of the envisaged applications.

It quickly appeared that it was important to consider the phenomena occurring at rest (calendar aging) which represents about 90 % of the time in the life of the battery. A similar group proposed the SIMCAL [9] project (3.6 M€) in 2009.

These projects have allowed to propose models of aging in cyclic and calendar modes.

From 2014, a new project [10], partially subsidized, and with a more industrial vocation, (MOBICUS) used the results and main actors of SIMSTOCK and SIMCAL to design and validate strategies to extend the life according to vehicle use and recharging modes, based on experimental measurements.

At the end of these subsidized programs, the main actors of the three above-mentioned projects wanted to maintain the network and the associated skills, without state subsidies.

Six laboratories (CEA, IFPEN, EIGSI, University of Bordeaux, University G. Eiffel, UTC) propose taking advantage of this experience and sharing it eventually through the COMUTES² consortium.

COMUTES² is an original consortium whose objective is to organize and execute battery aging campaigns. The 6 members of the consortium provide the testing facilities and their expertise to carry out each campaign, whose precise theme is chosen in agreement with industrial players. Each testing campaign is open to external partners who, after signing up to become members, may participate in the choice and definition of the tests and contribute to its funding. The databases generated from the results of test campaigns are owned equally between the experimental institutes and the members. Co-proprietors of this intellectual property may use these databases for their internal research purposes, to provide data for PhD theses or research projects.

After a legal construction phase, the COMUTES² consortium starts its first aging campaign. Five French industrial partners wished to join the adventure of this first campaign. (EDF, DGA, Hutchinson, Valeo, Siemens) around a common problem.

The first campaign started in mid-2020 on the theme “cold cycling aging of Li-ion batteries”. The present study is therefore derived from the environment described.

3. Aging campaign and experimental procedure

3.1. Lithium-ion cells

It was decided to study chemistry high Nickel-content NMC with a 21700 geometry. The manufacturer electrical characteristics of the cells are given in the Table 1.

In order to ensure a low dispersion, the cells were ordered from an identical manufacturing batch for all laboratories. They were supplied with welded tabs (width: 10 mm / thickness: 0.15 mm) for easy measuring.

3.2. Aging conditions investigated

The aim of this campaign is to study the aging impact of fast charges at temperatures below 0 °C. At these temperatures, calendar aging can

Table 1
Electrical characteristics of the studied cells.

Max charge voltage	4.2 V
Min discharge voltage	2.5 V
Max charge current (25 °C)	0.3C
Max discharge current (25 °C)	1.5C

be neglected and aging is mainly due to cycling [11,12].

Different mechanisms affect aging during cycling that depend on temperature, charge current and maximum depth of discharge (DoD) one of these mechanisms which is observed particularly at low temperature is the Lithium plating deposition [13]. This unwanted reaction occurs when the difference between the interface potential of the graphite electrode near the separator and the Li_+ / Li redox potential reaches 0. In a previous paper [14], we propose a method to determine the maximal current for low temperature to avoid this phenomenon. As a first approximation, it is assumed that most of the charge transfer takes place at the negative electrode. This allows us to estimate a maximum current by dividing the potential of the negative electrode by the charge transfer resistance. At $-20\text{ }^\circ\text{C}$ by measuring the charge transfer resistance R_{ct} at beginning of life using an Electrochemical impedance spectroscopy (EIS), considering a potential margin of 85 mV at the negative electrode in the high states of charge, which means a maximum current not to be exceeded C/7. The C/7 rate is very low. However, the method used as a first approximation neglects the diffusion present at the negative electrode.

Maintain a constant depth of discharge for the different points of the experimental plan remains difficult especially for high temperature variations. The presented results correspond to an expected DoD of 50 %. Two protocols are proposed:

- Protocol P1: The discharge is stopped when the cell voltage reaches 3,65 V. this voltage corresponds to a 50 % DoD under nominal conditions (0,2C@25 °C).
- Protocol P2: 50 % depth of discharge is obtained by a coulometric method and based on the initial cell capacity.

The six consortium’s experimenters divided up the 12 configurations selected for the experimental plan: a total of 36 cells were tested (3 cells by test). At the difference to Wang et Al. study on similar technology [15], cycling conditions were performed with partial discharge after full charge. To realize the experiment reported in Table 2, two successive waves with two different cycling protocols were carried out.

The impact of the protocols will be accurately described in the Section 4.1 for the conditions 0,3C @ 0 °C.

In all the experiments, the discharge was performed at 0.5C rate (see Section 3.3.2. for a more accurate description of the aging protocols P1 and P2).

Moreover, the maximum value for the state of charge (SoC) was set at 100 % and its minimum value around 50 % in the case of fresh cells (the minimum value is lower for aged cells).

3.3. Experimental procedure

A specific protocol for this test campaign was defined jointly between the 11 members.

Table 2
Experiment plan for 2 waves with all protocols.

Temperature (°C)	Charge rate current				
	C/7	0.3C	1C	2C	3C
25		P1		P1	P2
0		P1	P1	P1	
		P2			
-10		P1	P1		
			P2		
-20	P2	P2			

3.3.1. Preliminary test

Before starting the aging tests, we checked that the whole available cells had similar characteristics. To do this, each experimenter carried out an electrical impedance spectroscopy measurement on 10 cells. Between the 60 cells, the different spectra revealed that there was a difference of at most 4 % on the ohmic resistance characterized by a shift of the spectra. Among these 60 cells, 36 were selected to perform the campaign.

3.3.2. Aging protocols

For each of the two waves planned for an experiment, the aging protocol is divided into 5 different steps described in Fig. 1.

During the running-in, 3 charge and discharge cycles are first carried out at constant current. The charges were performed at 0.3C until the voltage reaches 4.2 V and the discharge at 0.2C until the voltage reaches 2.5 V. After the running-in, a first checkup is performed to control the state of health (SoH) of the cell (see Section 3.3.3. for checkups description). Then cyclic aging is launched, which consists of a succession of charges and discharges hereinafter referred to as micro-cycles.

This campaign consisted of 2 waves of tests, each of it with a different protocol (P1 and P2). These two protocols result from two different strategies: for P1, cycling is carried out with a target in voltage; for P2, cycling is carried out with a target in discharged capacity.

Each protocol contains a succession of micro-cycles with 3 different steps which are presented in Fig. 2.

Every 10 cycles for P1 and every 30 cycles for P2, a full discharge at 0.5C rate until voltage 2.5 V is reached is carried out in order to measure the capacity at that moment of the experiment. Then, a full charge is performed and the cycling continued.

When the capacity loss is greater than 8 % compared to the previous measurement, a short checkup is performed at 25 °C.

The experiment stops when the total capacity loss reaches 50 % or when it is no longer possible to perform the required protocol. At the end, a final checkup is performed.

3.3.3. Checkup protocols

The objectives of the checkup protocol are to determine the evolution of the capacity and resistance of cells at the reference temperature (25 °C). This characterization phase has been done regularly for all the cells.

From this checkup phase, only the capacity is measured during the discharged phase at C/10.

3.3.4. Experimental setup

36 cells have been tested to realize the 12 experiments planed in the campaign. To have a nice reproducibility, the cells were distributed to obtain 3 independent cells by test condition. Cells were stored in different controlled climatic chambers with a tolerance about 2 °C. Digatron and Biologic power supplies controlled the cell current during charges and discharges.

For each cell, the voltage, current, temperature and capacity were recorded after the checks were completed. Cell voltage was measured in situ while current and capacity were directly measured by the power supplies. In order to limit the impact of the tab’s resistance on the voltage measurement, the power cables were connected to the opposite side of the tabs as shown on the Fig. 3 (4-wire sensing method). In addition, cell temperature was measured with a thermocouple stuck on the skin of the cell and the ambient temperature was also measured close to the cell.

Before starting the aging campaign, an evaluation of the dispersion of impedance measurements between laboratories was performed to ensure that there was no bias in the comparisons due to the experimental set up. The cell-to-cell impedance variability observed in one laboratory is close to the variability between cells in several laboratories, which allows the validation of the experimental set-up used by all laboratories.

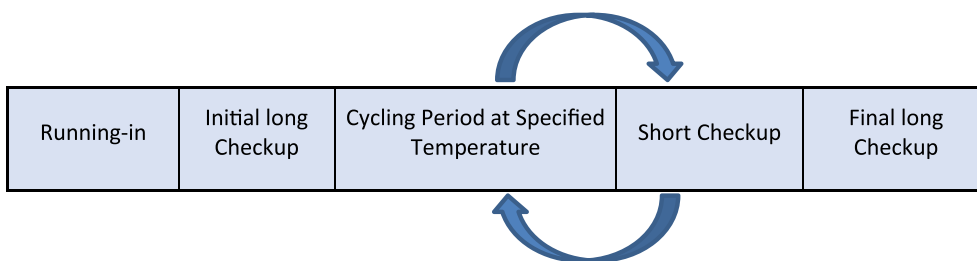


Fig. 1. Different steps of the aging protocol.

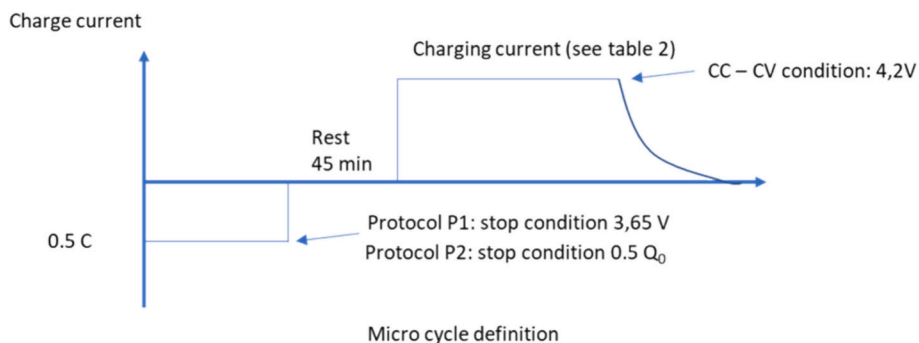


Fig. 2. Micro-cycle used in the aging protocols cycling tests.

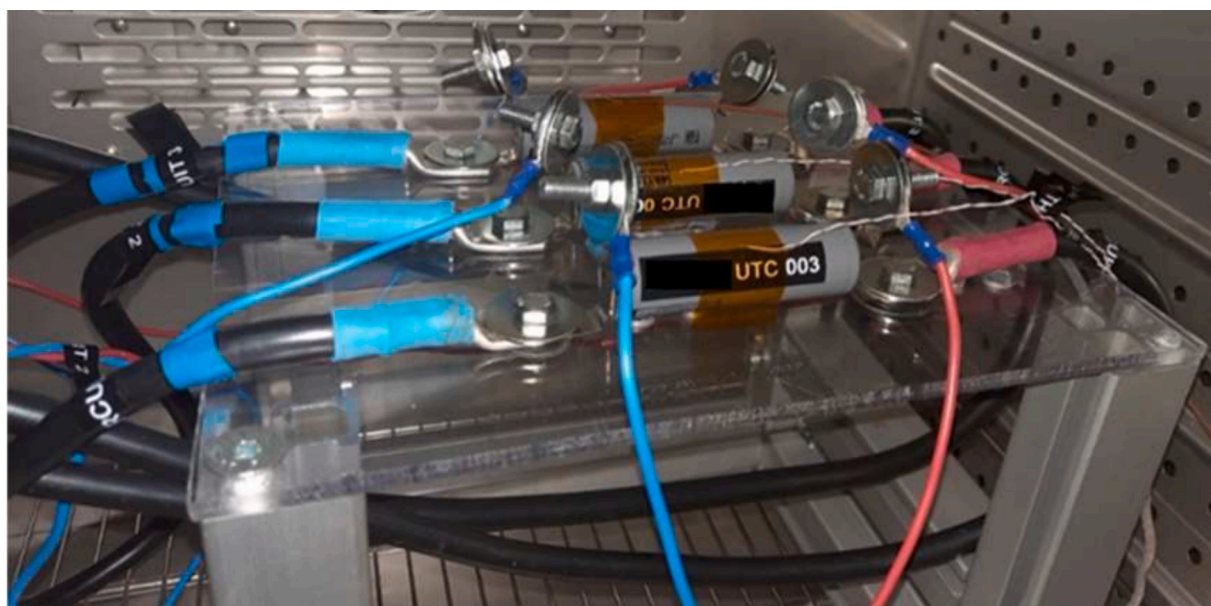


Fig. 3. Example of cells instrumentation.

4. Results and discussions

4.1. Comparison between aging conditions

Results of the first year of the COMUTES² test campaign are summarized in Fig. 4 representing the evolution of SoH as a function of total discharged capacity. For operating point described in Table 1, the evolution of the average SoH obtained on the three test cells is represented in solid line. The envelope represents the standard deviation of the measurements. The operating point and the protocol used are specified on the graph.

The operating point (−20 °C/ 0.3C) is not represented in this figure

because important swelling is observed after few cycles. This behavior makes it impossible to characterize the cell. Effect of this operating point is discussed in more detail in the next paragraph dedicated to complementary analysis.

The operating point (25 °C/0.3C) is considered as the reference point because the cells were cycled according to the conditions recommended by the manufacturer. The nominal capacity defined by the manufacturer is Q_0 . On this reference point, the cells have cycled according to the P1 protocol (Ref Table 2) more than 1030 Q_0 in discharge leading to a capacity loss of 15 %. The evolution of the capacity of these cells on this reference point shows a very low dispersion of the aging observed on the three cells.

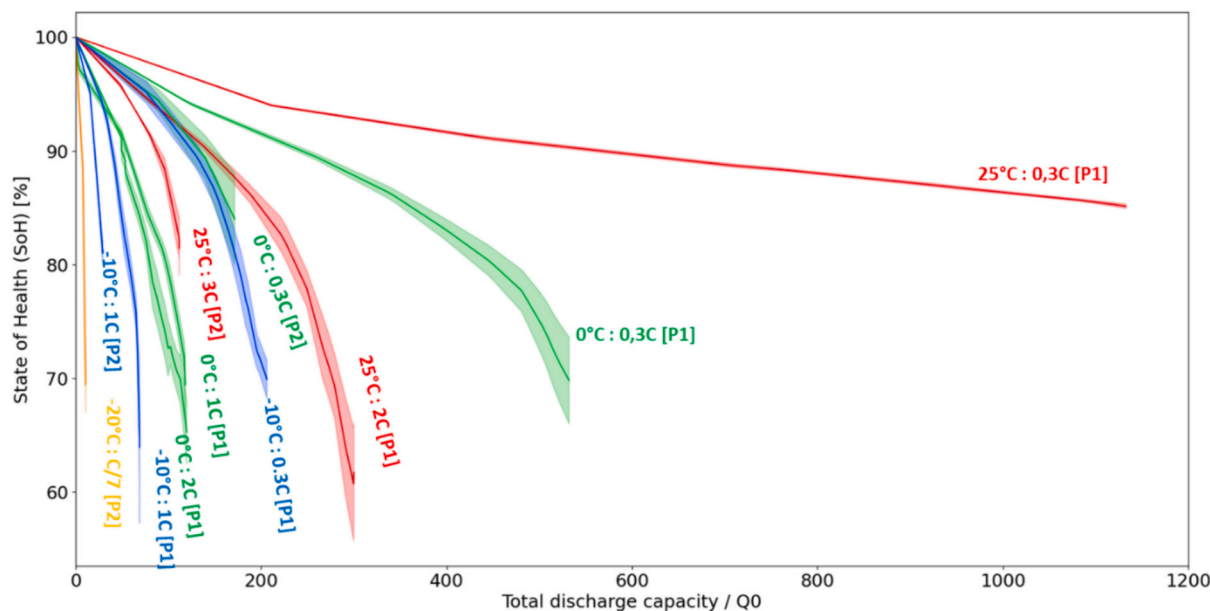


Fig. 4. SOH evolution during the first COMUTES² campaign.

As expected [16,17], cycling at low temperatures and/or high current induces accelerated cell aging. The Crates chosen for each temperature have been calculated to allow plating to a greater or lesser extent, except for 25 °C/0.3C, which is a reference for “normal” aging. These results also show that the method presented in Section 3 and used in this work to estimate the maximum C/7 current at –20 °C can be improved, since the current is slightly overestimated, leading to lithium plating from the very first cycles.

In addition to contributing to the database on rapid charge/low-temperature aging, two major points stand out from these results (Fig. 4). The first is the low dispersion of the results for the reference points but the dispersion increases for other operating points. This is an important point for the calibration or development of aging models under these critical operating conditions. The second point is the effect of the protocol. These results highlight that two protocols initially close for fresh cells can have significant effects on the results of an accelerated aging campaign. Indeed, the voltage or capacity cycling target, although similar on new cells at 25 °C, brings a clear change in operating conditions as the cell ages.

The Fig. 5 illustrates this point. Indeed, for two temperature-load current operating conditions (0.3C/0 °C and 1C/–10 °C), P2 protocol degrades the cells faster than P1 protocol at iso-condition of current and temperature.

To clarify the effect of the protocol observed on Fig. 5, the charge-discharge micro-cycles that constitute the major part of the aging phases are analyzed. This analysis is based on the observation of the chronograms of voltages and quantity of electricity for the new state, and an aged state corresponding to a 20 % loss of capacity. The comparison is made between protocol P1 (discharge to a voltage limit) and protocol P2 (discharge of a constant amount of Ah) for the same charge conditions (i. e. 0.3C). These curves are presented in Fig. 6.

In the fresh state, the difference in the amount of electricity exchanged can be clearly seen in the case of protocol P1 (cyan curves) where $Q_{cha} = Q_{dch} = 33\%$ of Q_0 and in the case of P2 (pink curves) where $Q_{cha} = Q_{dch} = 50\%$ of Q_0 (Figure 6right). In the aged state, this situation is accentuated and Q barely reaches 20 % of Q_0 in the case of the P1 protocol. Indeed, as the impedance of the cell rises with aging, the discharged capacity decreases because the reaching of the discharge limit voltage of 3.65 V occurs more and more earlier, leading to a reduction of the charged capacity with aging. This result illustrates the effect of DoD on aging. The P1 protocol, with its lower DoD, causes less

aging for the same energy discharged. The time spent recharging with P2 protocol is therefore longer than with P1 protocol, leading to an increase in the quantity of lithium plating at each recharge in cases where the C rate enables this phenomenon.

4.2. Particular/noteworthy cases / complementary analysis

Important swelling has been observed on several cells especially those tested in conditions: 0 °C/2C (P1 protocol) and –20 °C/0.3C (P2 protocol). For each of these conditions, we observe longitudinal casing breakage on one of the three cells tested (Fig. 7.a-b). These events happened during cell testing after ~800 cycles and at SoH ~ 54 % for 0 °C/2C condition and few dozens of cycles for –20 °C/0.3C condition (check-up had not been possible after for this cell). No any additional damage (exhaust, fire) and no mass loss has been noticed after cell breaking.

The damaged cell is opened to evaluate safety devices state. The metal casing of the cell is cut just under the positive terminal by taking care to not damage the terminal (Fig. 7.c). The terminal is dipped into a bath of epoxy resin to achieve the maximum of rigidity. Then, the sample is cut and polished in 2 parts to see the internal part namely the position of the Current Interrupt Device (CID) and the venting valve. It can be observed that the current collector remains connected to the terminal (Fig. 7.d) indicating no activation of CID occurred during or after the event. Also, the venting valve is not opened. So, we can assume that the damage comes from the expansion of internal materials of the cell during the cycling.

Furthermore, after rest time of several days, cell voltage drastically reduced near to 0 V. On cell tested at 0 °C/2C condition, voltage drop is observed during C/3 charge at 25 °C (Fig. 8). The voltage rapidly reduces to 2.6 V and continue to decrease slowly after. A slight increase of cell temperature happens before the event indicating an exothermic parasitic reaction. However, any warming is revealed during and after the event. Hence, we can conclude that a high resistance internal short circuit occurred conducting to complete discharge of cell.

We can notice that these events occurred only on 2 cells even if other cells were cycled in similar conditions. More generally, other cells tested in condition 1C/–10 °C and 2C/25 °C has been aged to SoH 46 % without observing this phenomenon. This observation confirms a very high dispersion of performances and failures between cells for low temperatures.

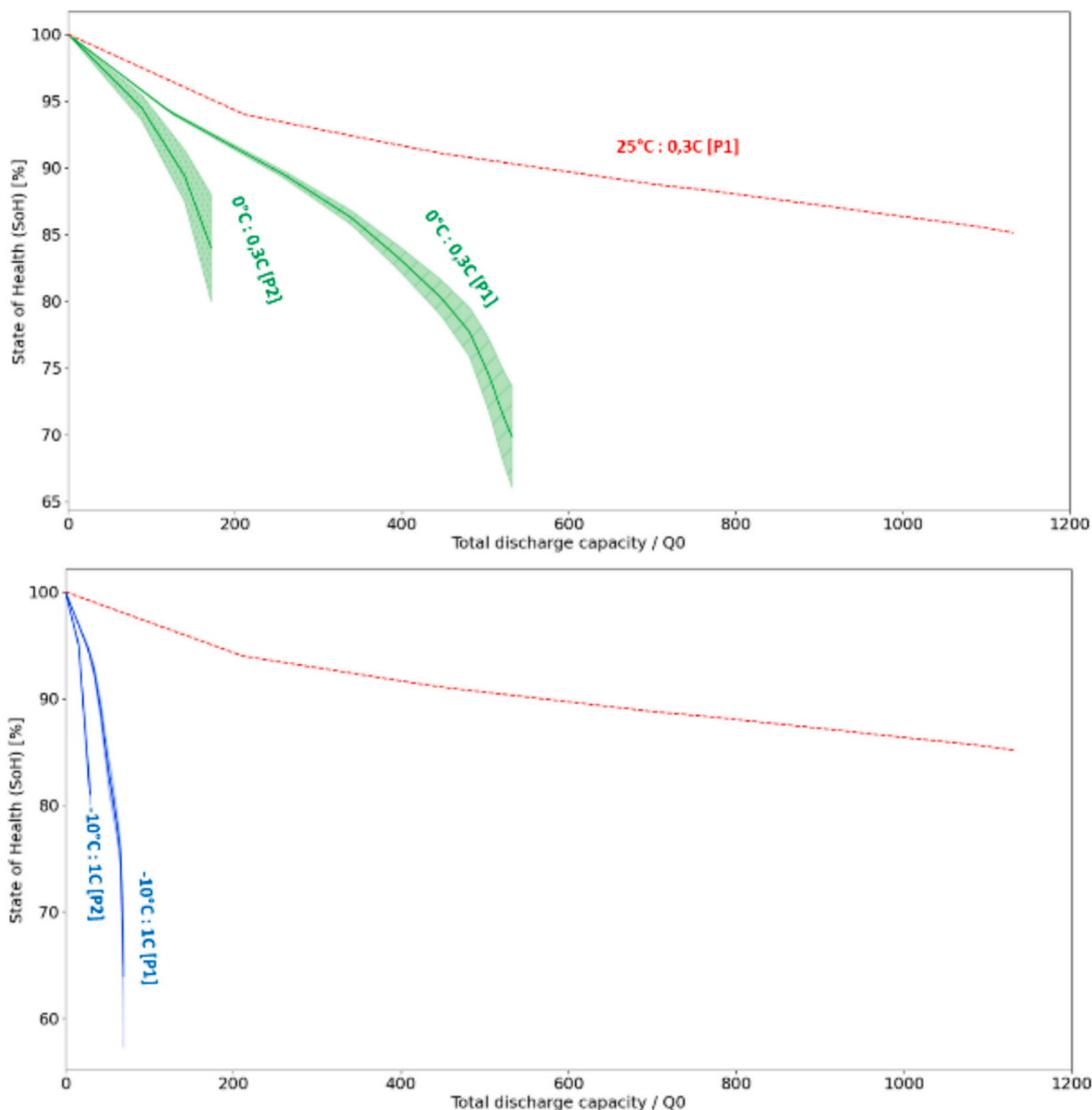


Fig. 5. Evolution of the SoH as a function of the total discharge capacity or different protocol at 0 °C and 0.3C (up) and at -10 °C and 1C (down).

5. Conclusions and future works

5.1. Conclusions

The behavior of batteries at low temperatures is complex and many phenomena drastically reduce their life span. Current models are not able to predict the functioning and aging of batteries. Thus, a measurement campaign to build a database was carried out on 21700 Li-Ion high Nickel-content NMC cells. A precise checkup protocol was drawn up and the article presents, among the results, the evolution of the SOH (capacity loss) as a function of the total number of A.h cycled. The charging modes were the subject of an experimental design and 2 discharge modes were considered. P1 Protocol: stopping the discharge at a voltage of 3.65 V and P2 protocol: systematic discharge of 50 % of nominal capacity Q_0 .

The main results are presented in relation to the nominal conditions of the cell considered (25 °C at 0.3C).

- Under nominal conditions, the total number of A.h cycled is greater

than 1030 Q_0 for a SoH greater than 85 % and the dispersion between cells is very low.

- For the P1 protocol at 0 °C, the dispersion between elements is much greater and the SoH reaches 80 % for less than 412 Q_0 cycled.

- For the P2 protocol at 0 °C, the dispersion is also important and the SoH of 80 % is reached for a value of 247 Q_0 cycled.

These last two points show that the reduction in temperature leads to a drastic reduction in service life and that the choice of end-of-discharge criteria (available energy) has a major impact on service life.

- For the battery under consideration, even at 25 °C, a charge at 2C has a significant impact on service life. For a SoH of 80 %, the number of A.h cycled drops to 247 Q_0 and the same charge conditions at 0 °C lead to less than 103 Q_0 cycled.

- In low temperature cases (0 °C - 2C), cases of destruction have been detected without triggering the safety systems.

This database, in addition to contribution to the development and calibration of aging models of Li-ion cells of high Nickel-content NMC chemistry, will allow by complementary analyses (post mortem,

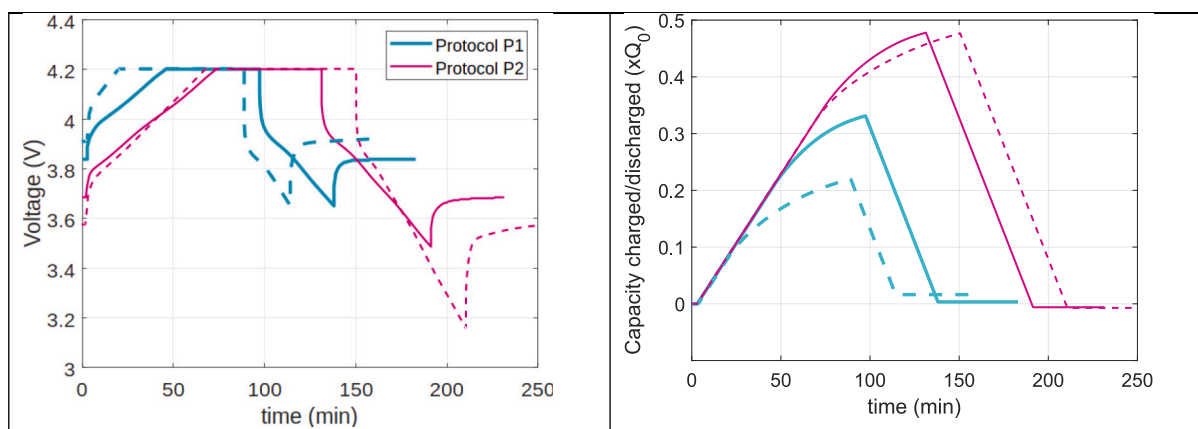


Fig. 6. Voltage (left), and algebraic capacity (right) during one micro-cycle at beginning of life (solid line) and after 20 % capacity loss (dashed line) for both P1 (cyan curves) and P2 (pink curves) protocols at the same conditions, i.e. charge at 0.3C and temperature 0 °C. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)

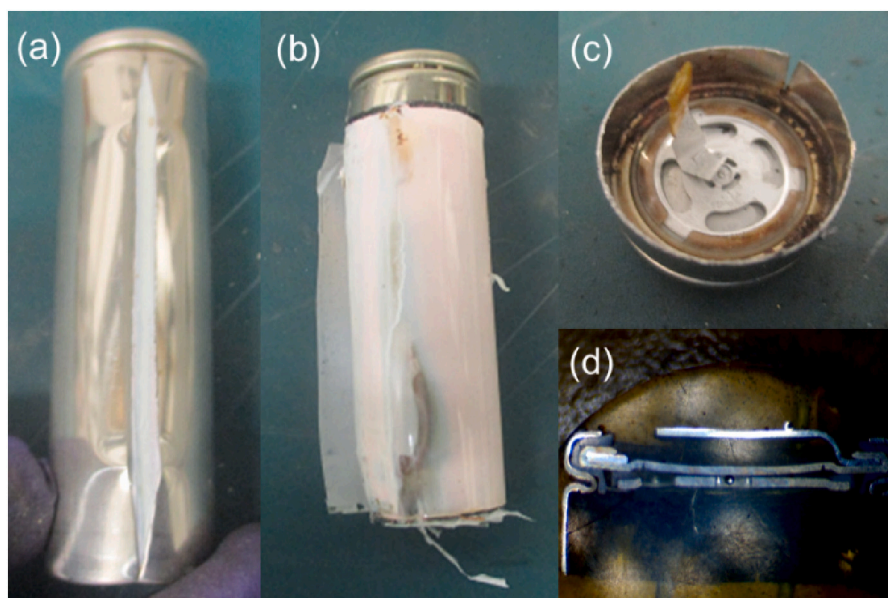


Fig. 7. Observation of longitudinal casing breakage (a) before opening, (b) after opening, (c) positive terminal, (d) cross-sectional view of positive terminal.

Incremental capacity analysis (ICA) among others) to identify the degradation mechanisms involved in the capacity losses observed according to the parameters of the study (temperature, cycling current and protocol).

5.2. Future works

The database obtained as part of the COMUTES² consortium could subsequently be supplemented by other operating points (cycling and calendar) and by post-mortem analysis of the cells studied. These post-mortem analyses will enable us to confirm or refute the targeted degradation mechanisms as a function of aging conditions. This additional information will enable the development of aging models for 21700 Li-Ion high Nickel-content NMC cells, with a specific focus on cold cycling and the damage mechanisms encountered.

In view of the diversity of formats and chemistries available for today's Li-ion batteries, the need for experimental data for the development and validation of aging models is more pressing than ever.

The COMUTES² consortium is continuing its collaborative work on aging databases with the launch of a second aging campaign [18].

CRediT authorship contribution statement

Mathieu Lecompte: Writing – original draft, Validation, Methodology, Investigation, Data curation. **Julien Bernard:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Elisa Calas:** Investigation, Data curation. **Lucas Richardet:** Validation, Investigation, Data curation. **Aurelien Guignard:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **François Duclaud:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Damien Voyer:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Maxime Montaru:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Bruno Crouzevalle:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Loic Lonardon:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Catherine Arnal:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Olivier Briat:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Armande Capitaine:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Jean-Michel Vinassa:** Validation, Methodology,

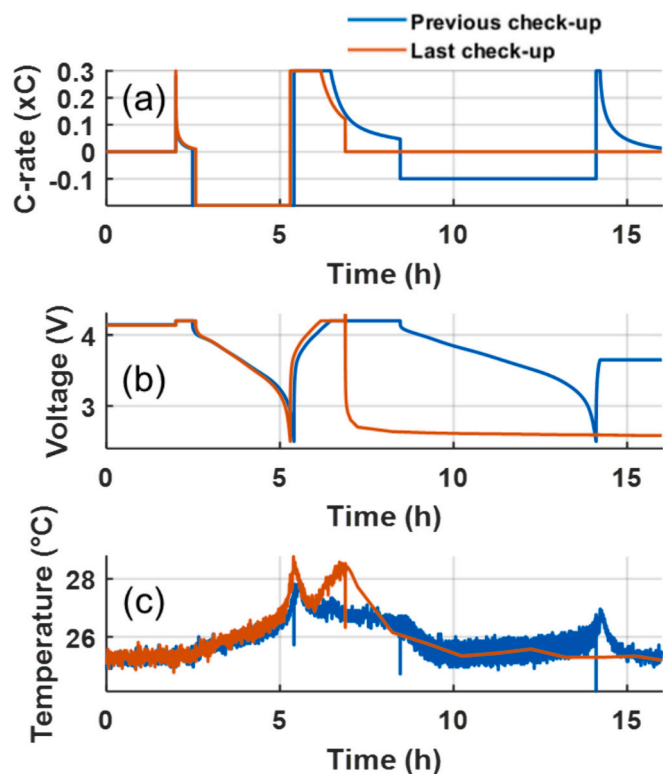


Fig. 8. Measurements performed on cell tested in 0 °C/2C condition during the two last check-up tests: (a) Current, (b) Voltage, (c) Battery temperature.

Investigation, Data curation. **Eduardo Redondo-Iglesias:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Serge Pelissier:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Christophe Forgez:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Guy Friedrich:** Writing – original draft, Validation, Methodology, Investigation, Data curation. **Laurent Torcheux:** Validation, Methodology. **Guillaume Bretin:** Validation, Methodology. **Abdelhadi Asseban:** Validation, Methodology. **Franck Sellier:** Validation, Methodology. **An Li:** Validation, Methodology. **Cedric De Vault:** Validation, Methodology. **Kamel Azzouz:** Validation, Methodology. **Jeremy Guazzagioloppa:** Validation, Methodology. **Loic De Francqueville:** Validation, Methodology.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

The authors do not have permission to share data.

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Glossary

- DoD depth of discharge:
 EIS Electrochemical impedance spectroscopy:
 SOC State of Charge:
 SoH State of Health:
 Q₀ Nominal cell capacity:
 CID Current Interrupt Device:
 ICA Incremental capacity analysis: