MAT4BAT

Advanced materials for batteries

FP7-2013-GC-Materials

Theme GC.NMP.2013-1 - Improved materials for innovative ageing resistant batteries

Collaborative project

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Deliverable D5.6
Recommendations for the further development of regulations and standards

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Task	5.4	Regulations, Recommendations, Standards

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[1] Dissemination level: **PU** = Public, **PP** = Restricted to other programme participants (including the JU), **RE** = Restricted to a group specified by the consortium (including the JU), **CO** = Confidential, only for members of the consortium (including the JU)

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Deliverable abstract

Task 5.4 has as objective to review the current regulations and standards, the development of proposals for the improvement of regulations and standards with respect to the new state-of-the-art created in this project, if appropriate and necessary for the development and competitiveness of the European economy. The impact of current standards on the developed materials and cell architectures has to be investigated with a differentiation between test standards and regulations.

Deliverable 5.1 has provided an identification of relevant regulations and standards. It comprises an overview of test methods for Li-ion battery cells concerning characterisation tests, cycle life tests, abuse & reliability tests and labelling.

The regulations and standards as described in the previous deliverable of task 5.4 are used for a confrontation with the materials and methods as they are applied in the Mat4Bat project. The approach for this is a systematic survey and questionnaire to the project partners. From the resulting findings an analysis on shortcomings of the actual standards with respect to the needs in the Mat4Bat project is proposed in the current deliverable.

The information needed to mark batteries for recycling leads to different views. For the recycler it is good to know as much as possible on the composition of the batteries. However, for the national organisations involved with the collection it may be advantageous that as little information as possible is clearly visible to prevent upstream sorting of the battery waste stream.

The set of characterisation tests in the Mat4Bat project shows a number of deviations with the characterisation tests in standards:
- The slow discharge and charge test (<C20) is important in the Mat4Bat project for the physics-based simulations and the equivalent network approach as used in the battery management system. Nevertheless, it is not given in any standard today.
- The conclusion of dynamic stress test (DST) can be used to simplify the cycle life ageing test and to estimate the accuracy of voltage predicting battery models.
- Using a second order equivalent electric network model to describe the voltage response on pulses appears superior over a first order model. Combining a discharge block with pulses gives most information for a good determination of the parameters of an equivalent network scheme for the battery.
- The charge behaviour is not evaluated in any standard, although important e.g. for brake energy recuperation.

The material characterisation tests used in the Mat4Bat project are part of broadly used test methods. Most of them are not standardised and if so, officially only for nano-enabled electric energy storage materials. For ante and post mortem characterisation tests standardisation is judged not possible. However, for coin (half) cell tests standardisation may lead to better comparability of test results between laboratories for the development phase of new batteries. An emphasis has to be at the preparatory phase. More important than a test prescription appears the material preparation to be. The electrode configuration has to be studied at the beginning and trials must first be made to obtain a reliable result. The best is to study the half-cell design first on the pristine material. All involved laboratory personnel should afterwards use the same design for the ageing study.

The ageing test approach in the Mat4Bat project is totally different from the ones found in the standards. Ageing tests by IEC, ISO, etc. cover a lot of tests and are still not representative for electric drivelines and car usage. A more generic ageing approach would enable to calibrate aging models. These models can then be used for quantifying the aging for specific drivelines and car usage.

It appears that both the designation of a test being an abuse, reliability or safety and protection tests is arbitrary as well as the pass/fail criteria. The external and internal short circuit tests are recommended to be reviewed. The addition of adiabatic (accelerated rate) calorimetric (ARC) tests in ISO and IEC standards is encouraged since a lot of useful information for the application of batteries can be obtained. If the calorimetric tests are not judged as a safety test, they should be adopted in the characterisation (performance) test methods.

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1. Introduction: regulation and standardisation landscape

The Mat4Bat project comprises a task to investigate the impact of current standards on the developed materials and cell architectures and the compliance of the development with those standards. To do so, first an overview of applicable regulations and standardisation has been collected. This has resulted in the report:

"Mat4Bat Public deliverable D5.1: List of relevant regulations and standards, G. Mulder, K. Trad, S. Ried, D. Sotta, April 2016" or "MAT4BAT_D5.1_M31_v1.pdf", that can be downloaded here:
http://mat4bat.eu/wp-content/uploads/2017/02/MAT4BAT_D5.1_M31_v1.pdf

The standards are also listed as a searchable website (see Figure 1):
https://batterystandards.vito.be/

This inventory is used for a confrontation with the materials and methods as they are applied in the Mat4Bat project. The approach for this is a systematic survey and questionnaire to the project partners what will be described in chapter 2. From the resulting findings an analysis on shortcomings of the actual standards with respect to the needs in research projects is derived in chapter 3. The current chapter will give a short outline on regulation and standards related to rechargeable Li-ion batteries, based on the deliverable D5.1.

Figure 1: Screenshot of website on battery standards

1.1. *Regulation*

Regulation on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH)

The European Union Regulation No 1907/2006 on the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) is regulating the use of chemicals in Europe. It addresses the production and use of chemical substances and their potential impacts on human health and the environment. The substances must be registered with the European Chemicals Agency (ECHA). This database can be consulted here:

http://echa.europa.eu/information-on-chemicals

One of the obligations is to inform customers about the 'Substances of Very High Concern' (SVHC) that are listed on the 'Candidate List' and contained in products in concentrations higher than 0.1% weight by weight per article. These materials may be found in batteries, probably as an electrolyte solvent. Two substances used as electrolyte fluid are known as of Very High Concern. The Candidate List of substances of very high concern for Authorisation has the following internet address:

http://echa.europa.eu/candidate-list-table

European battery directive

Directive 2006/66/EC is the main European regulation on batteries[4]. The primary objective is to minimise the negative impact of batteries on the environment. It advocates a high collection and recycling rate for waste batteries and accumulators in the European member states, so as to achieve a high level of environmental protection and material recovery throughout the community. For the Mat4Bat project it is important to remember that it prescribes an additional label to the CE marking. All batteries, accumulators, and battery packs are required to be marked with the separate collection symbol (crossed-out wheeled bin) either on the battery or its packaging depending on size. If the battery contains more heavy metals than prescribed by the directive, their chemical symbols have to be added. Lithium ion batteries fall under the lowest category for recycling efficiencies: a recycling rate of at least 50% by average weight is required.

Search for Chemicals

Advanced search

| tetrahydrofurane | Search |

Search by name, EC number or CAS Registry number

Name	EC / List no.	CAS no.	
Tetrahydrofuran IUPAC name: **Tetrahydrofurane**	203-726-8	109-99-9	BP
(3S)-3-Hydroxytetrahydrofuran Synonym: **Tetrahydrofurane**, (S)-3-Hydroxy	617-795-0	86087-23-2	

Figure 2: Screenshot of website to search chemicals at ECHA.

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[1] http://ec.europa.eu/environment/waste/batteries/

Regulation on capacity labelling of portable secondary and automotive batteries

The European regulation 1103/2010 governs the capacity marking requirements of portable rechargeable batteries including specific requirements related to its minimum size and location. The capacity label shall include both the numeral and its units expressed in Ah or mAh. The capacity label is a marking which has to appear either on the battery label, the battery casing and/or the packaging.

UN 38.3 UN Manual of Tests and Criteria, 4th Revised Edition, Lithium Battery Testing Requirements

All lithium cell types have to be subjected to tests as described in UN 38.3. The tests have to be fulfilled to be able to transport cells and batteries. It contains 8 tests.

1.2. Standards

IEC 62660 series on Secondary lithium-ion cells for the propulsion of electric road vehicles

This series contains performance and life testing of secondary lithium-ion cells used for propulsion of electric vehicles including battery electric vehicles (BEV) and hybrid electric vehicles (HEV) (part 1), reliability and abuse testing for lithium- ion cells (part 2) and safety requirements (part 3).

ISO 12405 series on Electrically propelled road vehicles -- Test specification for lithium-ion traction battery packs and systems

This series contains three parts: high-power applications, high-energy applications, and safety performance requirements. The test procedures for lithium-ion battery packs and systems enable the determination of the essential characteristics of performance, reliability and abuse of lithium-ion battery packs and systems. They assist the user to compare the test results achieved for different battery packs or systems.

IEC 62620 Large format secondary lithium cells and batteries for use in industrial applications

This standard contains many aspects about secondary li-ion cells and batteries. It has clauses on battery labelling, including the main used active materials. It makes a division into high power, high energy, and intermediate cells. It also contains a cycle life test.

IEC 62619 Safety requirements for large format secondary lithium cells and batteries for stationary and motive applications

The title is clear on the application scope.

IEC TS 62607-4 series on key control characteristics in nanomanufacturing

It concerns:
– Part 4-1: Cathode nanomaterials for nano-enabled electrical energy storage - Electrochemical characterisation, 2-electrode cell method
– Part 4-2: Physical characterization of nanomaterials, density measurement
– Part 4-3: Nano-enabled electrical energy storage - Contact and coating resistivity measurements for nanomaterials
– Part 4-4 Thermal Characterization of Nanomaterials, Nail Penetration Method
– Part 4-5 Cathode nanomaterials - Electrochemical characterisation, 3-electrode cell method

Part 4-1 contains methods to characterise cathode materials via the 2 electrode cell method. The standard deals with the sample preparation, pre-treatment of the cathode material and the preparation of the screw or Swagelok cell. The standard comprises the following test methods:

– open circuit voltage
– potentiostatic impedance spectroscopy
– charge-discharge experiment (constant current + constant voltage) with a C-rate of 0,1C and 10 cycles.

The methods seem valid for 'micro-scale' as well.

IEC 62281 Safety of primary and secondary lithium cells and batteries during transport

Standard IEC 62281 is close to UN 38.3. It comprises three sections: transport tests, packaging test and safety information on packaging and transporting batteries.

Marking symbols for secondary batteries for the identification of their chemistry (under development by IEC)

This is a new IEC standard under development with the aim to provide information to battery recyclers. It contains battery labelling. The sign for lithium (ion) batteries is shown in.Figure 3.
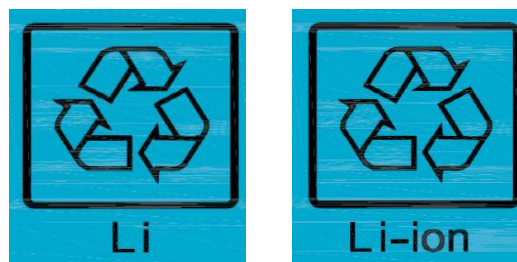

Figure 3: proposed marking for Li-ion batteries

(DOE) INL/EXT-07-12536 Battery test manual for plug-in hybrid electric vehicles

This manual defines a series of tests to characterise aspects of the performance and life behaviour of batteries for plug-in hybrid electric vehicle (PHEV) applications. The test procedures in this manual are directly applicable to complete battery systems. However, most can also be applied with appropriate scaling to the testing of modules, cells or sub-scale cells.

(DOE) SAND2005-3123 FreedomCAR Electrical Energy Storage System Abuse Test Manual for Electric and Hybrid Electric Vehicle Applications

This manual defines abuse tests intended to simulate actual use and abuse conditions for electrical energy storage systems used in electric and hybrid electric vehicles. The manual incorporates improvements and refinements to test descriptions presented in the Society of Automotive Engineers Recommended Practice SAE J2464 "Electric Vehicle Battery Abuse Testing".

UL 1642 UL Standard for Safety of Lithium Batteries

It covers all types of lithium batteries for use as power sources in products. The requirements are intended to reduce the risk of fire or explosion when lithium batteries are used in a product. They are also intended to reduce the risk of injury to persons due to fire or explosion when user-replaceable lithium batteries are removed from a product and discarded.

2. Results from survey about the use and applicability of standards and regulation for the Mat4Bat materials and methodologies

2.1. REACH regulation

Based on the relevant standards and regulations a list of questions was prepared by VITO in order to identify

- if the MAT4BAT developments are compliant with those,
- if the MAT4BAT developments outperform some of them,
- if actions should be taken on European level aiming at extensions or modifications of existing standards and regulations.

Prior to the consortium meeting in May 2016, these questions were sent to the MAT4BAT partners and first feedback was collected. The consolidated feedback was then presented and discussed in May 2016. Further iterations until the end of the project were achieved. The present chapter presents the collected feedback.

Are new materials developed that are currently not registered at the European Chemicals Agency (ECHA)? & Are materials used, new for the use in batteries but already existing, that are of Very High Concern?

Concerning the 2nd GEN electrolyte, there is no new material that is not registered at ECHA. From those substances, there is no material that is listed as substance of very high concern. 3rd generation electrolyte materials and additives, like tetrahydrofurane are registered substances.

The materials that are added to the anode are registered materials and do not fall under Very High Concern. This seems for the cathode material also the case.

2.2. Labelling of batteries

Are the options to designate a battery cell according to IEC 62620 (Large format secondary lithium cells and batteries for use in industrial applications) complete enough for the Gen2&3 cells?

The main anode component is a special version of graphite, so covered by the current designation. The main cathode material is nickel, cobalt or manganese and therefore covered by the current possibilities.

Do recyclers need more information than just Li(-ion) as proposed in the IEC standard under development on marking symbols for secondary batteries to know how to recycle, based on the green value chain management approach?

Although MAT4BAT did not analyse technical options for the recycling of the developed cells, this question has received three different answers.

- No

The Li-ion battery designation as given in the two standards with a full battery designation will not be included in this proposed standard with the argument that additional marking of the different Li-ion chemistries is not necessary for the recycling.

- Yes

The recycling of (Li-ion) batteries definitely could benefit from more detailed information on the composition of the batteries (materials + quality). The focus of Li-ion battery recyclers is definitely not on Li because of its low economic value, but more on valuable metals such as Cu and Al. Note 'could' because this of course still depends of the capability of the recycler to separate the materials again.

In the next level towards a circular economy it would even beneficial to have information not only about the materials composition of the battery but on the components in the battery, to enable re-use of re-manufacturing of these components.

- Certainly not

Certainly not more information should be given. It should be avoided that (illicit) organisations collect batteries before they go to the collection points. This would lead to a situation that the national collection organisations would be left with worthless Li-ion batteries and that some other organisations can enrich themselves.

2.3. *Material characterisation*

A first observation is that the methods in the IEC TS 62607-4 series, especially, 4-1, are valid for a broader material range than nano-materials, covering micro-scale' as well.

Opposite feed-backs are given on the question, if the IEC TS 62607-4 series on nanomanufacturing should also cover secondary batteries:
– standardisation of material characterisation methods can be worthwhile to obtain repeatable results;
– the methods are such common sense that standardisation is unnecessary.

Are there characterisation methods that can/need to be standardised?

It appeared that the electrochemical coin cell measurements (half cells and full cells) as carried out amongst the research partners were difficult to compare. The test conditions were varying. Therefore, it has been suggested that normalised methods can be helpful. More important than a test prescription appeared the material preparation to be. The electrode configuration has to be studied at the beginning and trials must first be made to obtain a reliable result. Particularly if later on the material characterisation is performed at several laboratories this is a prerequisite. Additional problems to compare characterisation results are introduced if not the same device (not the same separator, number of spacer, etc…) is used. The best is to study the half-cell design first on the pristine material. All involved partners should afterwards use the same design for the ageing study. It would really improve the reliability and the comparison.

Standard IEC TS 62607-4 Part 4-1 (2-electrode cell methods for electrochemical characterisation of cathode materials) contains tests about:
– open circuit voltage
– potentiostatic impedance spectroscopy
– charge-discharge experiment (constant current + constant voltage) with a C-rate of 0,1C and 10 cycles.

In the Mat4Bat project also the galvanostatic intermittent titration test (GITT) method is used as an important characterisation method, especially to obtain the open circuit voltage as function of discharge capacity. It can also be used to derive the ion diffusivity although this is less known and

analysis intensive. Since it is a key characterisation method it is good to bring it into the standard. The opinion that it is so wide-spread and straightforward that it is not necessary to normalise it, exists also within the project.

Can the ante/post mortem cell characterisation be standardised? Are systematic approaches used?

The possibility to standardise ante/post mortem cell characterisation is denied since it depends on many factors. A procedure was presented in the review paper by the Mat4bat consortium (Waldmann et al. 2016), but the exact way how it is done cannot be standardized and has to be decided in each case.

The paper is:
'Review — 'Post-Mortem Analysis of Aged Lithium-Ion Batteries: Disassembly Methodology and Physico-Chemical Analysis Techniques ', T. Waldmann, A. Iturrondobeitia, M. Kasper, N. Ghanbari, F. Aguesse, E. Bekaert, L. Daniel, S. Genies, I. Jimenez Gordon, M.W. Löble, E. De Vito, M. Wohlfahrt-Mehrens, J. Electrochem. Soc. 163, A2149-A2164, 2016 open access, doi: 10.1149/2.1211609jes

From electrochemical modelling point of view it is interesting to know if the SEI built-up is limited by a kinetic rate growth or by a diffusion limited growth. Mathematical expressions exist for both cases and they can fit on the ageing data, but may lead to unrealistic parameters. Knowing which aspect is most important allows to trench before. A way to know the mechanism is determining the SEI quality like its porosity. In the Mat4Bat project no specific measurements on this were possible.

Some characterisation methods are used that are not in the standards. Why different approaches are used, what is their added value?

The pulse test is different from the standards. Almost all standards used 10 second pulses and in the Mat4Bat protocol 30 seconds. Only one pulse strength (1C) is used. A reason for increasing the pulse time might be that it allows to observe slower cell mechanisms as for example the diffusion.
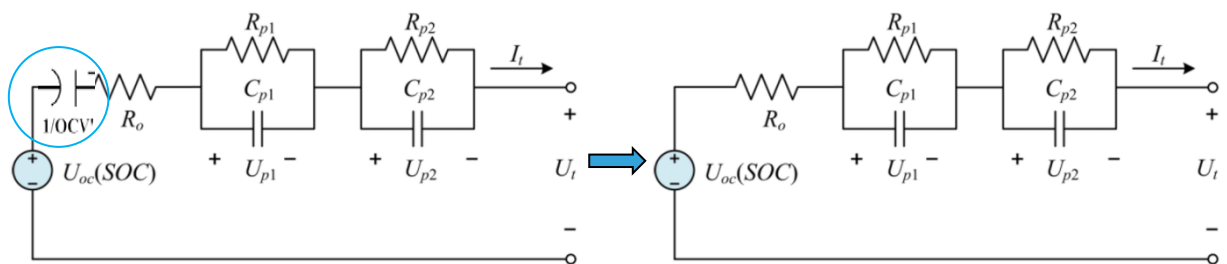

Figure 4: The FreedomCar model approach is used to obtain the Thévenin-model for each applied SOC level. Here a second order version is shown of both models.

VITO carried out a test to study the difference between 10 s and 30 s pulses. Also a series of pulse strengths was included, being C/10, C/2, 1C and 3C. The FreedomCar model is used from the 'Battery test manual for plug-in hybrid electric vehicles' standard. It has one RC-circuit, being a first order model. A second circuit was added in the mathematical expressions to obtain a second order model. To use the parameter results afterwards, the ultracapacitor (OCV') in the model must be neglected. This leads to the first and second order Thévenin model. This is explained in Figure 4. It

appears that fitting 10 s pulses up to 1C with a first order FreedomCar model does not lead to relaxation dynamics. To use a first order equivalent electric circuit model, the pulses must take 30 s. This is shown in Figure 5 A and B. However, if a second order model is used, then no difference is found in the parameters for all pulse strengths and for both durations.

10 s pulses can be used in combination with a first order model only for high strength pulses (3C or higher). This is the case for the pulses prescribed in the famous HPPC test of the 'Battery test manual for plug-in hybrid electric vehicles'.

A more complete picture on battery dynamics is obtained by including the discharge block to the next SOC level into the fitting. This makes a much slower relaxation visible, especially using a second order an equivalent electric circuit model like the second order Thévenin model. Adequate model parameters are only found by fitting the discharge block and the pulses together. Whether the pulses are of 10 s or 30 s, does hardly have an influence, see Figure 5 C and D.

The characterisation test in the Mat4Bat test set-up comprises a dynamic stress test (DST). This is an example of a load test and is used to verify that the battery can deliver its specified power when needed. The load is usually designed to be representative for the expected conditions in which the battery may be used. It can also be a particular driving cycle. In the Mat4Bat project the DST discharge profile is adapted with respect to the maximum power of the tested cells. Starting from the fully charged cells, the DST was repeated until the end-of-discharge voltage was reached. Two representative variables from the DST tests have been selected, cumulative capacity [Ah] and cumulative energy [Wh]. The cumulative capacities and energies are plotted against the respective values for CC discharging and a clear linear correlation appears to be present, see Figure 6. The added value that has been found is this linear correlation indicates that a static CC discharge is a good profile to estimate the capacity loss under dynamic conditions of the DST test.

The DST test is part of the (DOE) INL/EXT-07-12536 Battery test manual for plug-in hybrid electric vehicles. It is not part of IEC and ISO standards that contain performance tests for Li-ion batteries and cells. The above given reasoning shows that it is an interesting test and can simplify the ageing test.

The DST test can also be used to check the equivalent network battery model that is derived from the pulse test.
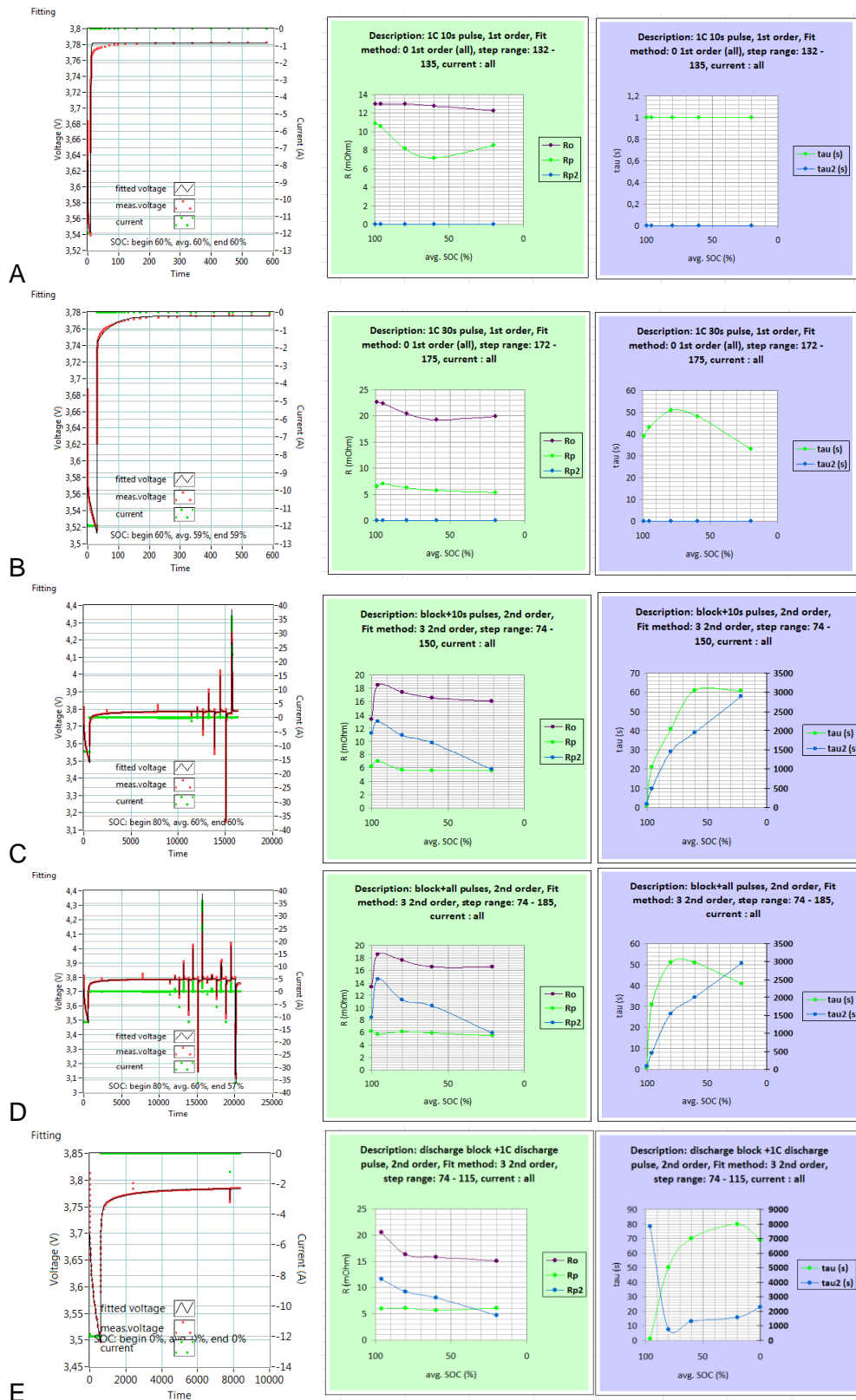
Figure 5: Fitting different pulses to obtain the parameters of a Thévenin model. In A 10 s discharge pulses are fitted. The characteristic time is 1 s, what was the minimum allowed in the fitting. The 30 s pulses (B) lead to a characteristic time of approximately 40 s. Fitting the discharge block and all 10 s pulses with help of a second order FreedomCar model (C) leads to a characteristic time of around 40 s and a slow one of around 3000 s. Adding 30 s pulses (D) does not influence the fit (Measurements by VITO). Using the discharge block followed by one 1C discharge pulse leads to the same parameters if a second order model is used (E).
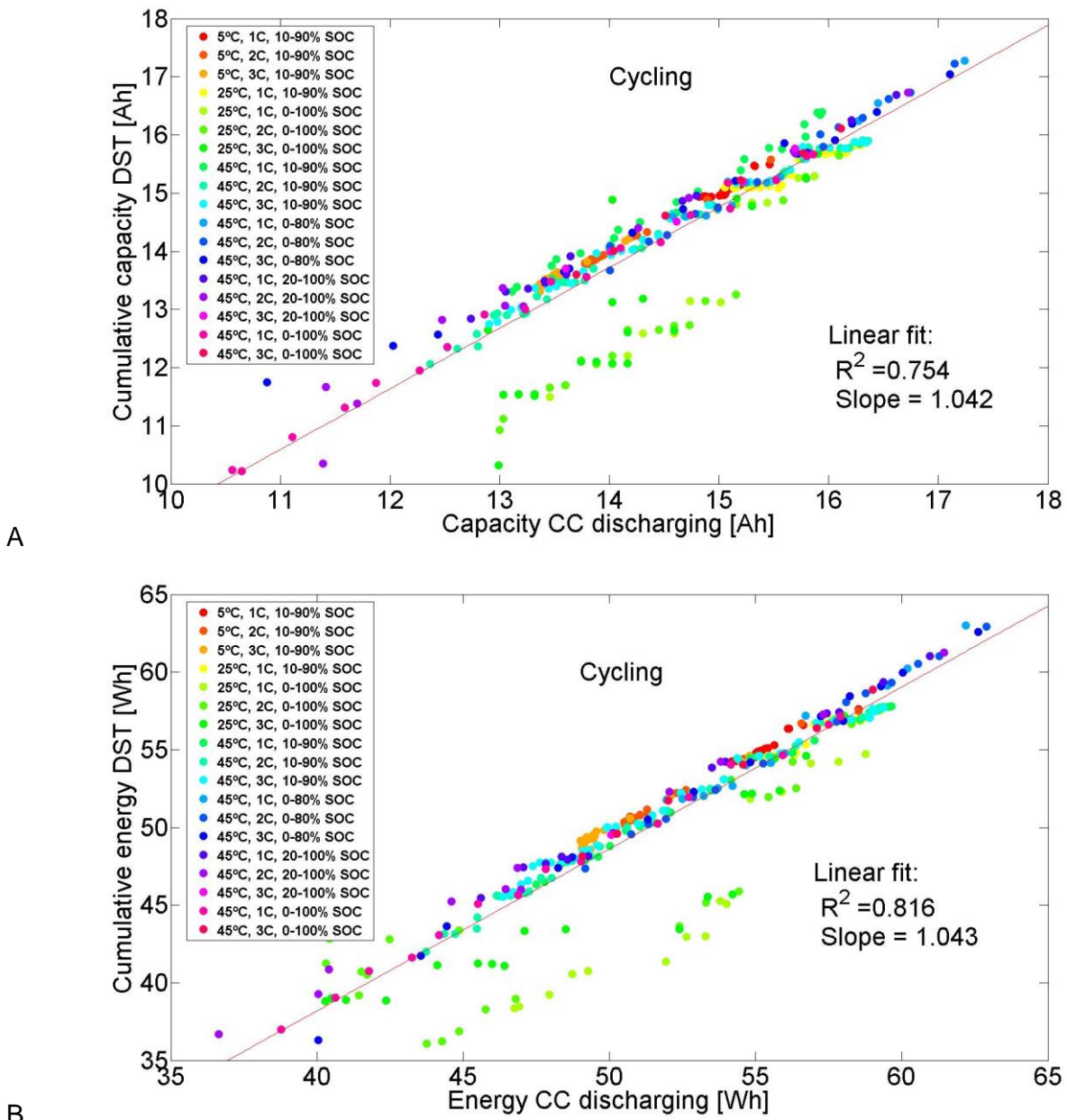
Figure 6: The cumulative capacity plotted against the constant current discharge capacity (A). A linear relation appears. B shows the equivalent relation expressed in energy.

2.4.   *Ageing tests (cycle-life and calendar life)*

Why different approaches are used, what is the added value? To be standardised?

Several test methods and especially many test conditions are found (SOC windows, temperatures) in the available standards. In the Mat4Bat project a totally different ageing test approach is used.

Ageing tests by IEC, ISO, etc. cover a lot of tests (if you want to fulfil them all) and are still not representative for electric drivelines and car usage. A more generic ageing approach that enables a model for the cell ageing behaviour would be better if the test results are planned to be used for calibrating aging simulation models. Based on such models the ageing can be derived for specific drivelines and car usage.

Similar approaches to the Mat4Bat ageing test methodology are also used in other European research projects like Batteries2020 and eCAIMAN.

2.5.   *Reliability and abuse tests*

Are the used tests methods and conditions according to standards?

Yes, except for ARC. An ARC test is only prescribed in (DOE) SAND2005-3123 FreedomCAR Electrical Energy Storage System Abuse Test Manual for Electric and Hybrid Electric Vehicle Applications. The given test was not studied in detail.

We did ARC tests like in literature. It's a quasi-standard, since all publications do it like this.
e.g. Roth and Doughty J. Power Sources 128 (2004) 308.

3. Identified shortcomings of the actual standards with respect to the needs in the Mat4Bat project

3.1. *Battery labelling*

The Mat4Bat project does not need to include new chemistries in the battery designation. It can be remarked that a new anode materials as silicon and lithium polysulfide are currently not covered.

3.2. *Characterisation tests*

The characterisation tests in standards have the emphasis on system level for specific EV applications. Cell behaviour cannot be compared since the test conditions need not to be identical for cells. In fact, the test conditions deferred from the final application and are downsized to module and cell level by a dimensioning parameter that is chosen by the manufacturer.

The charge behaviour is not evaluated in current standards, although important e.g. for fast charging and brake energy recuperation. Only the prescribed charge current of the battery manufacturer is used. It is advised to include this.

The characterisation of a battery (cell) produces an incomplete description to enable a battery management system (BMS) to identify the cell. A slow discharge and charge, typically C20 or slower, lacks in all standards. Nevertheless, this lead is often used to derive the open circuit voltage (OCV) as function of state of charge (SOC) for batteries as used in the BMS.

Regarding the pulse tests it appeared from the tests performed in the previous chapter on this topic, that it is important to use a second order model. This leads to consistent model results hardly dependent on the pulse strength and pulse length that is used. Combining the discharge block to the next SOC level with one or more pulses makes a quick and slow relaxation visible, describing well the measurement data. This leads to an improved characterisation by equivalent network modelling as often used in the BMS. The performed test and the positive results can be shared with standardisation committees.

A dynamic stress (DST) test is neglected in almost all standards with battery characterisation methods or performance tests. In the Mat4Bat project the DST discharge profile is adapted with respect to the maximum power of the tested cells. Starting from the fully charged cells, the DST was repeated until the end-of-discharge voltage was reached. Two representative variables from the DST tests have been selected, cumulative capacity [Ah] and cumulative energy [Wh]. The cumulative capacities and energies are plotted against the respective values for CC discharging and a clear linear correlation appears to be present.

– The added value that has been found is that this linear correlation indicates that a static CC discharge is a good profile to estimate the capacity loss under dynamic conditions of the DST test.
– If this linear relationship can be proven then the ageing test can be simplified.
– The voltage response can also be used to test the voltage prediction possibilities of algorithms in the BMS.

3.3. *Ageing tests*

The ageing test approach in the Mat4Bat project, like in many scientific projects is totally different from the ones prescribed in the standards. The tests based on the classic approach with specific drive cycles and a specifically dimensioned battery, are not entirely fit-for-purpose since they are not always representative for the electric drivelines and car usage.

A more generic approach that enables the development of a model for ageing, either empirical law or physics-chemical phenomena based, would be beneficial. For this a minimum viable test scheme with a combination of calendar life and cycle life tests needs to be standardised.

3.4. *Material characterisation tests*

Material characterisation tests are found in standards for nano-enabled electrical energy storage. This seems not well fit-for-purpose as the test protocols are equally valid for 'above nano/ micro scale' materials. The given characterisation methods are some examples of methods that are broadly used. Some comments to 62607-4-1 can be given in more detail:

– Title: the restriction to nanomaterials depends on the definition of nanomaterials. Most cathode materials are larger than 100 nm in all directions and have at least one direction lower than 1 micron. It would make sense to directly make no reference to the particle size (also because there are primary particles and secondary particles –agglomerates-, etc.) or not to restrict the standard to nanomaterials.
– 3.2: NCM is defined as LiNi1/3Mn1/3Co1/3O2 but Ni-rich compositions will probably be in the market soon (if not already) therefore it is better to define NCM (or NMC) materials as $LiNi_{1-x-y}Mn_zCo_yO_2$. $LiMn_2O_4$ is missing in the list (it is also a commercial cathode material). Non-commercial chemistries are not considered.
– 4.5: Disassembly: it is also important to avoid the exothermic reaction of metallic lithium with moisture or air.
– The standard mentions that other cell designs are possible although does not mention any. Coin cells are commonly used alternatively to the Swagelok cell design shown.
– SEI layer is not relevant here since mainly forms on the negative electrode but the same standard could be valid for anode materials (obviously changing U upper and lower limits etc.) including a SEI formation cycle.

It is recommendable to include more common test methods galvanostatic intermittent titration test (GITT) method. Less known learnings from tests can be added like how to derive the ion diffusivity from a GITT. It can also make it possible to obtain a better level of comparison between laboratories since it appeared that the electrochemical coin cell measurements (half cells and full cells) were difficult to compare. It has to be noted that GITT are, although commonly used, often misused: very long relaxation times should be used for reliable results as well as 3-electrode cells if possible.

An important class of material tests for batteries are the ante/post mortem cell characterisations. However, the possibility to standardise these tests is doubted since it depends on many factors. An exact way how to perform these tests cannot be standardized and has to be decided from one case to the other. More important than a test prescription appeared the material preparation to be. The electrode configuration has to be studied at the beginning and trials must first be made to obtain a reliable result. The best is to study the half-cell design first on the pristine material. All involved

partners should afterwards use the same design for the ageing study. It would really improve the reliability and the comparison.

3.5.   *Safety tests*

A comparison of safety test methods, see "Mat4Bat Public deliverable D5.1: List of relevant regulations and standards", has shown that an identical test can be categorised as an abuse, reliability or safety and protection tests. So, the categories are ambiguous.

Looking into comparable test methods, pass/fail criteria seem arbitrary and changing from standard to standard.

A special mention has to be given to the short circuit test. Most short circuit tests create hardly a high current for large cells according to the test conditions. Cells of 2Ah have typically a resistance of 10 mOhm whereas cells of 10 Ah have a typical resistance of 2 mOhm (see G. Mulder e.a. , 'Comparison of commercial battery cells in relation to material properties', Electrochimica Acta, 2013, figure 8D). If an external resistance of 100 mOhm is used, this results approximately in 40A or 20 C for a small cell but only 4 C for a 10 Ah cell. The test condition of IEC 62660-2 is with 5 mOhm much harder, but is confined to 10 min. only.

The internal short circuit test needs inclusion of other initiation methods. The nail penetration test suffers from poor reproducibility and is not fit-for-purpose. A meeting between standardisation committees on internal short circuit tests and researchers of the Mat4Bat project and other projects that studied the internal short circuit test like the Stallion project is recommendable.

Testing in adiabatic (accelerated rate) calorimeters (ARC) is not standardized by ISO and IEC, although a lot of useful information for the application of batteries can be obtained. If the calorimetric tests are not judged as a safety test, they should be adopted in the characterisation (performance) test methods.

3.6.   *Working groups to address recommendations from 3.1 to 3.5*

The following target was defined within the project description of work. "Only when our developments significantly outperform current standards, we will indicate this new state-of-the-art to the respective working group."

Since our test approaches differ significantly from the standards, a feed-back to the respective working groups seems interesting. A first attempt was given at the workshop "Putting Science into Standards (PSIS) 2016, Driving Towards Decarbonisation of Transport: Safety, Performance, Second life and Recycling of Automotive Batteries for e-Vehicles", organised by the Joint Research Centre (JRC) together with the European Committee for Standardization (CEN), the European Committee for Electrotechnical Standardization (CENELEC), and the European Commission Directorate-General for Internal Market, Industry, Entrepreneurship and SMEs. Grietus Mulder was invited as member of the Belgian standardisation committee and in personal name to give a view on lacks in current battery standards. The conclusions are handed over to the mentioned organisations. The contents can be found here:
https://ec.europa.eu/jrc/en/event/workshop/putting-science-standards-driving-towards-decarbonization-transport

The international committees that should be considered to give feed-back to are:
- IEC SC21A Batteries with alkaline and other non-acid electrolytes
  This committee makes standards on NiCd, NiMH, Li-ion batteries.
- IEC TC113 Nanotechnology standardization for electrical and electronic products and systems
  The committee treats nano-enabled electrical energy storage. They make standards for key control characteristics in nanomanufacturing and on material specifications in nanomanufacturing.
- ISO TC22 Road vehicles
  This committee takes the system level of electrically propelled road vehicles into account.

The project partners can also express the findings to the national mirror committees of the international standardisation committees. This is the straightforward and normal approach to comment on standards under development and under revision.

To start a new standard for battery tests it is also possible to address to the CENELEC committee CLC/TC21X that covers secondary battery standards.

Figure 7: Presentation about the performance assessment of automotive battery tests.

4. Conclusion

4.1.   *Legislation*

No issues have been found in relation with the legislation.

The European regulation on registration, evaluation, authorisation and restriction of chemicals (REACH) is followed and all materials used for the MAT4BAT cells are registered with the European Chemicals Agency (ECHA). None of them is listed as 'Substances of Very High Concern' (SVHC), being carcinogenic, very toxic or very persistent and very bio-accumulative (vPvB).

The battery directive prescribes a recycling rate of over 50% in average weight for Li-ion batteries. It does not give additional hurdles for the project.

No legislation on nanomaterials has been found.

4.2.   *Standardisation*

The Mat4Bat project does not need to include new chemistries in the battery designation.

The information needed to mark batteries for recycling leads to different views. For the recycler it is good to know as much as possible on the composition of the batteries (materials + quality) and to go further by including the used components. However, for the national organisations involved with the collection it may be advantageous that as little information as possible is clearly visible to prevent upstream sorting of the battery waste stream.

The set of characterisation tests in the Mat4Bat project shows a number of deviations with the characterisation tests in standards:
   – The slow discharge and charge test (<C20) is important in the Mat4Bat project for the physics-based simulations and the equivalent network approach as used in the battery management system. Nevertheless, it is not given in any standard today.
   – The conclusion of dynamic stress test (DST) can be used to simplify the cycle life ageing test and to estimate the accuracy of voltage predicting battery models.
   – Using a second order equivalent electric network model to describe the voltage response on pulses appears superior over a first order model. Combining a discharge block with pulses gives most information for a good determination of the parameters of an equivalent network scheme for the battery.
   – The charge behaviour is not evaluated in any standard, although important e.g. for brake energy recuperation.

The material characterisation tests used in the Mat4Bat project are part of broadly used test methods. Most of them are not standardised and if so, officially only for nano-enabled electric energy storage materials. For ante and post mortem characterisation tests standardisation is judged not possible. However, for coin (half) cell tests standardisation may lead to better comparability of test results between laboratories for the development phase of new batteries. More important than a test prescription appears the material preparation to be. The electrode configuration has to be studied at the beginning and trials must first be made to obtain a reliable result. The best is to study the half-

cell design first on the pristine material. All involved laboratory personnel should afterwards use the same design for the ageing study.

The ageing test approach in the Mat4Bat project is totally different from the ones found in the standards. Ageing tests by IEC, ISO, etc. cover a lot of tests and are still not representative for electric drivelines and car usage. A more generic ageing approach would enable to calibrate aging models. These models can then be used for quantifying the aging for specific drivelines and car usage.

It appears that both the designation of a test being an abuse, reliability or safety and protection tests is arbitrary as well as the pass/fail criteria. The external and internal short circuit tests are recommended to be reviewed. The addition of adiabatic (accelerated rate) calorimetric (ARC) tests in ISO and IEC standards is encouraged since a lot of useful information for the application of batteries can be obtained. If the calorimetric tests are not judged as a safety test, they should be adopted in the characterisation (performance) test methods.