



- (51) **International Patent Classification:**
H01M 10/0567 (2010.01) *H01M 10/44* (2006.01)
- (21) **International Application Number:**
PCT/EP2016/054216
- (22) **International Filing Date:**
29 February 2016 (29.02.2016)
- (25) **Filing Language:** English
- (26) **Publication Language:** English
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- (81) **Designated States** (unless otherwise indicated, for every kind of national protection available): AE, AG, AL, AM, AO, AT, AU, AZ, BA, BB, BG, BH, BN, BR, BW, BY, BZ, CA, CH, CL, CN, CO, CR, CU, CZ, DE, DK, DM, DO, DZ, EC, EE, EG, ES, FI, GB, GD, GE, GH, GM, GT, HN, HR, HU, ID, IL, IN, IR, IS, JP, KE, KG, KN, KP, KR, KZ, LA, LC, LK, LR, LS, LU, LY, MA, MD, ME, MG, MK, MN, MW, MX, MY, MZ, NA, NG, NI, NO, NZ, OM, PA, PE, PG, PH, PL, PT, QA, RO, RS, RU, RW, SA, SC, SD, SE, SG, SK, SL, SM, ST, SV, SY, TH, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, ZA, ZM, ZW.
- (84) **Designated States** (unless otherwise indicated, for every kind of regional protection available): ARIPO (BW, GH, GM, KE, LR, LS, MW, MZ, NA, RW, SD, SL, ST, SZ, TZ, UG, ZM, ZW), Eurasian (AM, AZ, BY, KG, KZ, RU, TJ, TM), European (AL, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HR, HU, IE, IS, IT, LT, LU, LV, MC, MK, MT, NL, NO, PL, PT, RO, RS, SE, SI, SK, SM, TR), OAPI (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, KM, ML, MR, NE, SN, TD, TG).

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(54) **Title:** LITHIUM-ION BATTERY FORMATION PROCESS

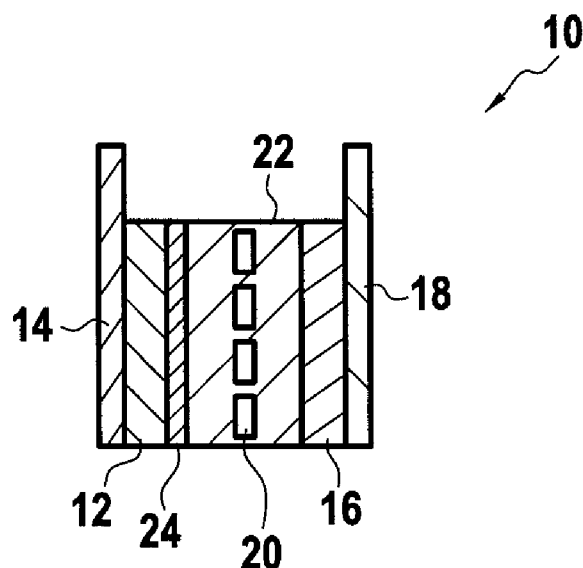


FIG.1

(57) **Abstract:** A method of performing a formation process for a lithium-ion cell (10) comprising an anode (12), a cathode (16), an electrolyte (22) and a separator (20), the formation process including adding an additive to the electrolyte (22) for improving a solid electrolyte interface (24) build-up on the anode (12), determining a SEI formation end voltage value at which the SEI build-up is completed by determining the additive concentration in the SEI (24), charging the cell (10) at the first predetermined rate up to the SEI formation end voltage, and then charging the cell (10) to a fully charged capacity at a second charge rate, the second charge rate being greater than the first charge rate.

WO 2017/148500 A1



Published:

— *with international search report (Art. 21(3))*

LITHIUM-ION BATTERY FORMATION PROCESS

FIELD OF THE DISCLOSURE

5 [0001] The present disclosure is related to lithium ion batteries or cells, and more particularly to an improved method for initially charging such batteries (formation process).

BACKGROUND OF THE DISCLOSURE

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[0002] Lithium-ion batteries are part of a family of rechargeable battery types in which lithium ions move from the negative electrode to the positive electrode during discharge and from the positive electrode to the negative electrode when charging.

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[0003] There are various types of lithium-ion battery. The anode comprises generally carbon and the cathode comprises a lithium compound. The anode and the cathode are separated by a separator made from a porous polymer, such as a micro-perforated plastic sheet, which allows ions to pass through. The anode, cathode and separator are immersed in an electrolyte.

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[0004] Lithium-ion batteries are classified according to the cathode material.

[0005] Once the lithium-ion battery is assembled, before the battery is suitable to be used, the lithium-ion battery may be put through at least one precisely controlled charge/discharge cycle to activate the working material. This step is called the formation process. This formation process provides the initial full charge of the battery.

25

[0006] During the formation process, a solid electrolyte interface (SEI) is formed on the anode. The SEI formation is important for the lifetime of the lithium-ion battery or cell.

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[0007] Methods for initial charging, i.e., for the formation process, of a lithium-ion battery have been proposed.

[0008] Typically, the battery is charged at a constant charge rate. The charge rate is also expressed as a C-rate, which represents a charge or a discharge rate equal to the capacity of a battery in one hour. It has been found that the SEI is best formed at small C-rate, which means that the

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initial charging is performed over an extended period of time. Indeed, fully charging a battery at a C-rate equal to C/5 would take approximately five hours. The battery is charged at a small C-rate up to the fully charged voltage of the battery in order for the SEI to form on the carbon anode during the first charge and then the battery is held constant at the fully charged voltage until the current drops below a threshold. The battery is then left to rest for two hours and is discharged at a small C-rate to a pre-set voltage, i.e., the discharge cut-off voltage. This formation process may be cycled at least once.

[0009] In order to reduce the manufacturing time of lithium-ion batteries, so-called dynamic forming processes have been proposed. In such processes, the battery is charged at a small C-rate up to the end of SEI layer formation on the anode, corresponding to a threshold voltage value, and then, a large C-rate is used to charge the battery up to the fully charged voltage. For example US 2015/060290 discloses such a formation protocol which still involves at least charging the battery up to the fully charged voltage at least twice, and resting the cell for two hours between each charge/discharge of the cell, the total duration of the dynamic formation process being greater than forty hours. However, in US 2015/060290, the end of SEI layer formation voltage value on the anode being determined by a method using differences of temperature, the determination is an approximate.

[0010] Additives have also been added to the electrolyte to improve the formation of the SEI and therefore enhancing the anode stability.

SUMMARY OF THE DISCLOSURE

[0011] Currently, it remains desirable to reduce the duration of formation process while having a battery that will exhibit good properties over a large number of charge/discharge cycles.

[0012] Therefore, according to embodiments of the present disclosure, a method of performing a formation process for a lithium-ion cell having an anode, a cathode, an electrolyte and a separator is provided. The method including:

- adding an additive to the electrolyte for improving a solid electrolyte interface build-up on the anode;

- determining a SEI formation end voltage value at which the SEI build-up is completed by determining the additive concentration in the SEI;
- charging the cell at a first predetermined rate up to the SEI formation end voltage; and
- then charging the cell to a fully charged capacity at a second charge rate, the second charge rate being greater than the first charge rate.

[0013] By providing such a method, the determination of the SEI formation end voltage value is accurate. Indeed, the additive concentration in the SEI may be measured by accurate methods. The determination of the SEI formation end voltage value is performed prior to applying a formation process to a cell.

[0014] Cells are charged at the first predetermined rate. Each cell is charged up to a given voltage and the additive concentration of the SEI partially formed on the anode is measured, allowing determining the SEI formation end voltage value. Thus, the SEI formation end voltage value is determined and may be used to initially charge cells having the same configuration and the same components as the cells used to determine the SEI formation end voltage value.

[0015] Charging the cell comprising an electrolyte to which additive has been added at the first predetermined rate allows for the formation of the SEI on the anode, the SEI having good electrochemical properties.

[0016] Once, the SEI formation is completed, the cell is charged up to a fully charged capacity at a second predetermined rate, the second charge rate being greater than the first charge rate. This allows reducing the duration of the formation process.

[0017] The SEI formation end voltage value may be determined as being the voltage at which the concentration of additive in the SEI becomes constant.

[0018] Indeed, when the additive concentration in the SEI does not increase with increasing charging time and increasing charging voltage, it may be concluded that the SEI formation on the anode is completed.

[0019] The additive concentration of the SEI may be measured by XPS. [0020] X-ray photoelectron spectroscopy (XPS) is a qualitative and quantitative analysis technique that allows measuring the elemental

composition on the surface of a sample. XPS may detect elements as light as lithium and may measure the elemental composition at the parts per thousand range. XPS has also the advantage that the surface chemistry of the sample may be analysed without requesting additional treatments of surface preparation.

[0021] The first predetermined rate may be smaller than or equal to 2 C, preferably smaller than or equal to 1 C, more preferably smaller than or equal to 0.5 C.

[0022] The first predetermined rate allows for the formation of the SEI on the anode. This first predetermined rate allows for the formation of a SEI with good electrochemical properties while not extending too much the duration of the full formation process.

[0023] The second predetermined rate may be equal to or greater than 2 C, preferably equal to or greater than 3 C, more preferable equal to or greater than 4 C.

[0024] The second predetermined rate allows reducing the formation process duration, as the second predetermined rate is greater than the first predetermined rate. Indeed, as the second predetermined rate is greater than the first predetermined rate, the charge time to reach the fully charged voltage from the end of peak voltage value is smaller at the second predetermined rate than at the first predetermined rate.

[0025] Before charging the cell to the fully charged capacity, the cell may be discharged at the first predetermined rate down to a cut-off voltage value of the cell, and the cell is charged again at the first predetermined rate up to the SEI formation end voltage value.

[0026] Cycling between the cut-off voltage value and the SEI formation end voltage value may improve the capacity retention of the cell.

[0027] The additive provided in the electrolyte may be selected from an oxalate salt, an ethylene carbonate or a sulfone.

[0028] The additive may present a decomposition potential at a smaller voltage than the electrolyte.

[0029] These additives may improve the formation of the SEI on the anode and provide a SEI having better in-use characteristics than SEI formed from the electrolyte only. Thus, the life-time of the lithium ion cell may be improved and higher power may be reached.

[0030] The first predetermined rate may be chosen as a function of the additive.

[0031] By choosing the first predetermined rate in function of the additive, the cell performance may be enhanced. For example, the retention capacity of the cell may be improved. The first predetermined rate may be chosen by carrying out some simple tests on test cells prior to starting the initial charge of the cell. In particular, the first derivative of the charge capacity relative to the measured voltage (dQ/dV) may be calculated from the measurements of the charge capacity and the voltage.

10 The first derivative exhibits at least a peak which has a maximum peak value voltage which decreases on charging/discharging the cell at a predetermined rate. The first predetermined rate may be chosen in function of the decreasing trend of the dQ/dV curve.

[0032] It is intended that combinations of the above-described elements and those within the specification may be made, except where otherwise contradictory.

[0033] It is to be understood that both the foregoing general description and the following detailed description are exemplary and explanatory only and are not restrictive of the disclosure, as claimed.

20 [0034] The accompanying drawings, which are incorporated in and constitute a part of this specification, illustrate embodiments of the disclosure and together with the description, and serve to explain the principles thereof.

25 BRIEF DESCRIPTION OF THE DRAWINGS

[0035] Fig. 1 shows a lithium ion cell;

[0036] Fig. 2 shows a block diagram illustrating an exemplary method according to embodiments of the present disclosure;

30 [0037] Fig. 3 shows a XPS spectrum of a SEI with and without additive representing the intensity (in arbitrary units) as a function of the binding energy (in electronvolt); and

[0038] Fig. 4 shows a graph of the additive concentration (in atomic share percent) in the SEI as a function of the voltage (in volts).

DESCRIPTION OF THE EMBODIMENTS

[0039] Reference will now be made in detail to exemplary embodiments of the disclosure, examples of which are illustrated in the accompanying drawings. Wherever possible, the same reference numbers will be used throughout the drawings to refer to the same or like parts.

[0040] Fig. 1 shows a schematic representation of an exemplary lithium ion cell 10. The lithium ion cell 10 includes an anode 12 fixed on an anode current collector 14 and a cathode 16 fixed on a cathode current collector 18. The anode 12 and the cathode 16 are separated by a separator 20, the anode 12, the cathode 16 and the separator 20 being immersed in an electrolyte 22.

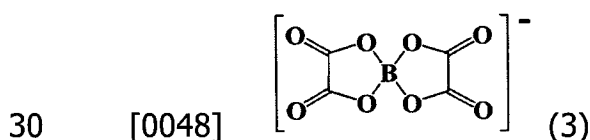
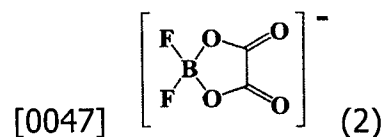
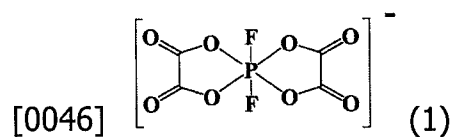
[0041] Typically, the anode 12 is made of a carbonaceous material, the anode current collector 14 is made of copper, the cathode 16 is made of an intercalated lithium compound and the cathode current collector 18 is made of aluminum. Lithium ions present in the electrolyte 22 move from the anode 12 to the cathode 16 during discharge of the cell 10 and from the cathode 16 to the anode 12 when charging the cell 10.

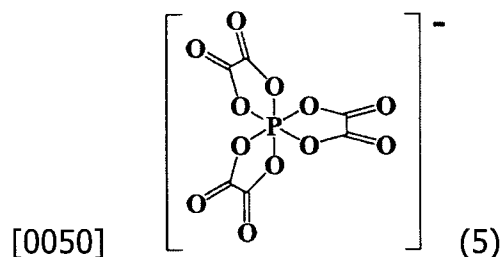
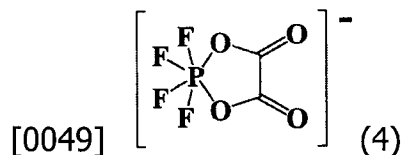
[0042] On the anode 12, a solid electrolyte interface (SEI) 24 is formed. The SEI 24 is formed during the formation process of the cell, i.e., during the initial charging of the cell.

[0043] Additive may be added to the electrolyte 22 to improve the formation of the SEI.

[0044] According to some embodiments, the additive provided in the electrolyte may be selected from an oxalate salt, an ethylene carbonate or a sulfone.

[0045] Examples of oxalate salts may include lithium salts of:





[0051] (1) is difluoro(oxalate)phosphate.

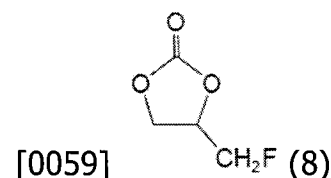
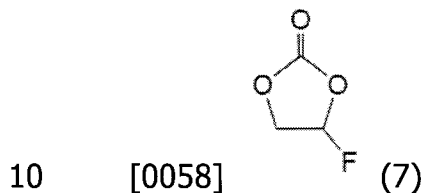
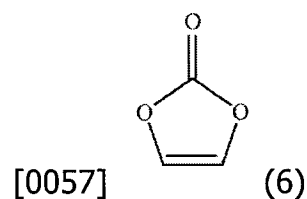
[0052] (2) is difluoro(oxalato)borate

5 [0053] (3) is bis(oxalato)borate.

[0054] (4) is tetrafluoro(oxalato)phosphate.

[0055] (5) is tris(oxalato)phosphate.

[0056] Examples of ethylene carbonate may include:

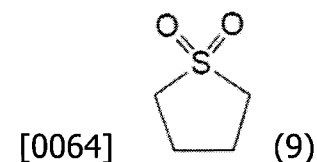


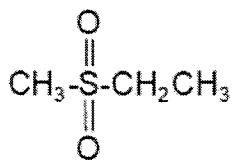
[0060] (6) is vinylene carbonate.

[0061] (7) is fluoroethylene carbonate.

[0062] (8) is (fluoromethyl)ethylene carbonate.

15 [0063] Examples of sulfone may include:





[0065] (10)

[0066] (9) is sulfolane.

[0067] (10) is ethyl methyl sulfone.

[0068] Fig. 2 shows a block diagram illustrating an exemplary method
5 according to embodiments of the present disclosure.

[0069] In step 26, the SEI formation end voltage value V_{SEI} is determined.

[0070] In step 28, the additive is added to the electrolyte 22 in the cell
10 for improving the SEI 24 build-up on the anode 12.

[0071] In step 30, the cell 10 is charged at a first predetermined rate
 C_1 up to the SEI formation end voltage value V_{SEI} determined in step 26.

[0072] For example, in the cell 10, the anode 12 is made of graphite,
the cathode 16 is made of $\text{LiNo}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2$ and the separator 20 is
made of a film comprising polyethylene. The electrolyte 22 is a mixture of
15 ethylene carbonate, dimethyl carbonate and ethyl methyl carbonate
present in equal volume ratio. The electrolyte also comprises LiPF_6 at
1 mol/L (mole/litre). The additive is a lithium difluoro(oxalate)phosphate
salt added to the electrolyte 22 at 5 wt% (weight percent).

[0073] For example, the first predetermined rate C_1 may be equal to
20 0.3 C. This first predetermined rate C_1 allows for the formation of a SEI 24
with good electrochemical properties while not extending too much the
duration of the full formation process.

[0074] Then, the cell 10 is charged at a second predetermined rate C_2
from the SEI formation end voltage value V_{SEI} to a fully charged capacity.

[0075] As shown at Fig. 2, after charging the cell 10 up to the SEI
25 formation end voltage value V_{SEI} , the cell 10 may be discharged at the first
predetermined rate C_1 from the SEI formation end voltage value V_{SEI}
down to a cut-off voltage value V_{cut} .

[0076] For example, the cut-off voltage value V_{cut} may be equal to 3 V.

[0077] The cell 10 is then charged at the first predetermined rate C_1
30 up to the SEI formation end voltage value V_{SEI} (step 30).

[0078] This charge/discharge cycle, i.e., steps 30 and 32, may be
repeated, allowing for better SEI formation.

[0079] The cell 10 is then charged at the second predetermined rate C_2 from the SEI formation end voltage value V_{SEI} to a fully charged capacity.

[0080] Although repeating steps 30 and 32 may increase the capacity retention of the cell 10, it will increase the duration of the formation process.

[0081] The SEI formation end voltage value V_{SEI} may be determined as follows.

[0082] A given number of cells 10 having the same components were charged at the first predetermined rate C_1 up to different voltages. Table 1 summarizes the voltages up to which the cells 10 are charged at the first predetermined rate C_1 .

[0083] Table 1: end voltage

	A	B	C	D	E	F	G	H
End voltage (in V)	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0

[0084] The anodes of Samples A-H were then dipped in a solution of ethyl methyl carbonate for 10 minutes and dried. The anodes of Samples A-H were ready for XPS analysis.

[0085] The X-ray intensity used during the XPS analysis was 1500 eV (electronvolt) and the X-ray diameter was 200 μm (micrometre).

[0086] As shown at Fig. 3, XPS spectrum of the SEI 24 formed on the anode 12 without additive added to the electrolyte 22 (dashed line) does not exhibit a peak while the XPS spectrum of the SEI 24 formed on the anode 12 with lithium difluoro(oxalate)phosphate additive added to the electrolyte 22 (dashed line) exhibits a peak at 134.85 eV.

[0087] This peak, representative of the additive is therefore used to measure the lithium difluoro(oxalate)phosphate concentration in the SEI 24.

[0088] As shown at Fig. 4, the additive concentration in the SEI (in %) as a function of the voltage (in volts) exhibits a plateau, i.e., the concentration of additive in the SEI does not increase above a given voltage value. This voltage value is the SEI formation end voltage value V_{SEI} for a given cell, at a given first predetermined rate C_1 . In this example, the SEI formation end voltage value V_{SEI} is equal to 2.5 V (volts).

[0089] Various formation processes have been applied to similar cells, i.e., having the same components, to validate performance of a cell 10 in which the SEI 24 is formed on the anode 12 according to the exemplary disclosed method.

- 5 [0090] The various formation processes are summarized in Table 2 together with the duration of the formation process and the capacity retention of the cells obtained with the listed formation processes.

[0091] Table 2: formation process

	C ₁	C ₂	Duration (min)	Capacity retention (%)
Sample 1	3 C	3 C	21	82
Sample 2	0.3 C	3 C	30	88
Sample 3	0.3 C	0.3 C	200	89

- 10 [0092] Sample 1 was charged at 3 C up to the fully charged capacity.

[0093] Sample 3 was been charged at 0.3 C up to the fully charged capacity.

- 15 [0094] Sample 2 was charged at the first predetermined rate C₁, which is equal to 0.3 C in this example, up to the SEI formation end voltage value V_{SEI}, and then from the SEI formation end voltage value V_{SEI} up to the fully charged capacity at the second predetermined rate C₂, which is equal to 3 C in this example.

- 20 [0095] As shown in Table 2, although the duration of the formation process is relatively small, the capacity retention of Sample 1 is relatively poor.

[0096] Better capacity retentions are obtained for Samples 2 and 3. Capacity retentions for Samples 2 and 3 are nearly the same while the duration of the formation process at least six-fold less for Sample 2 than for Sample 3.

- 25 [0097] Thus, it is demonstrated that accurate determination of the SEI formation end voltage value V_{SEI} together with different predetermined rate of charging may produce cells with good capacity retention and relatively short duration of formation process.

- 30 [0098] Throughout the description, including the claims, the term "comprising a" should be understood as being synonymous with "comprising at least one" unless otherwise stated. In addition, any range

set forth in the description, including the claims should be understood as including its end value(s) unless otherwise stated. Specific values for described elements should be understood to be within accepted manufacturing or industry tolerances known to one of skill in the art, and
5 any use of the terms "substantially" and/or "approximately" and/or "generally" should be understood to mean falling within such accepted tolerances.

[0099] Although the present disclosure herein has been described with reference to particular embodiments, it is to be understood that these
10 embodiments are merely illustrative of the principles and applications of the present disclosure.

[0100] It is intended that the specification and examples be considered as exemplary only, with a true scope of the disclosure being indicated by the following claims.

15 [0101] The method is described in terms of a single cell. However, it may be easily adapted for batteries having multiple cells.

CLAIMS

1. A method of performing a formation process for a lithium-ion cell (10) comprising an anode (12), a cathode (16), an electrolyte (22) and a separator (20), the formation process comprising:
- adding an additive to the electrolyte (22) for improving a solid electrolyte interface (24) build-up on the anode (12);
 - determining a SEI formation end voltage value (V_{SEI}) at which the SEI build-up is completed by determining the additive concentration in the SEI (24);
 - charging the cell (10) at the first predetermined rate (C_1) up to the SEI formation end voltage (V_{SEI}); and
 - then charging the cell (10) to a fully charged capacity at a second charge rate (C_2), the second charge rate (C_2) being greater than the first charge rate (C_1).
2. The method according to claim 1, wherein the SEI formation end voltage value (V_{SEI}) is determined as being the voltage at which the concentration of additive in the SEI (24) becomes constant.
3. The method according to claim 1 or 2, wherein the additive concentration of the SEI (24) is measured by XPS.
4. The method according to any of claims 1 to 3, wherein the first charge rate (C_1) is smaller than or equal to 2 C, preferably smaller than or equal to 1 C, more preferably smaller than or equal to 0.5 C.
5. The method according to any of claims 1 to 4, wherein the second charge rate (C_2) is equal to or greater than 2 C, preferably equal to or greater than 3 C, more preferable equal to or greater than 4 C.
6. The method according to any of claims 1 to 5, wherein before charging the cell (10) to the fully charged capacity, the cell (10) is discharged at the first predetermined rate (C_1) down to a cut-off voltage value (V_{cut}) of the cell (10), and the cell (10) is charged again at the first predetermined rate (C_1) up to the SEI formation end voltage value (V_{SEI}).
7. The method according to any of claims 1 to 6, wherein the additive provided in the electrolyte (22) is selected from an oxalate salt, an ethylene carbonate or a sulfone.
8. The method according to any of claims 1 to 7, wherein the first predetermined rate (C_1) is chosen as a function of the additive.

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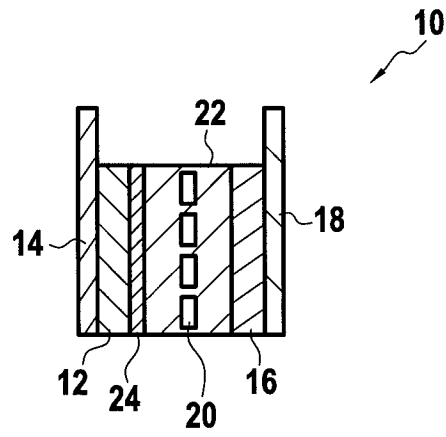


FIG.1

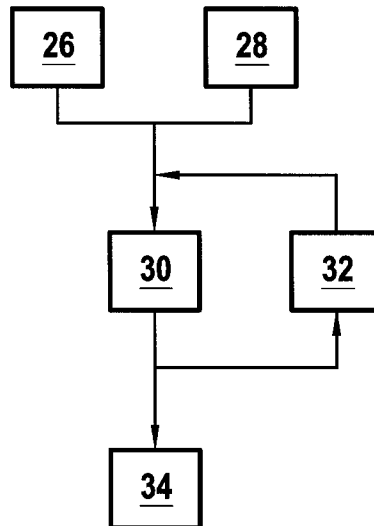


FIG.2

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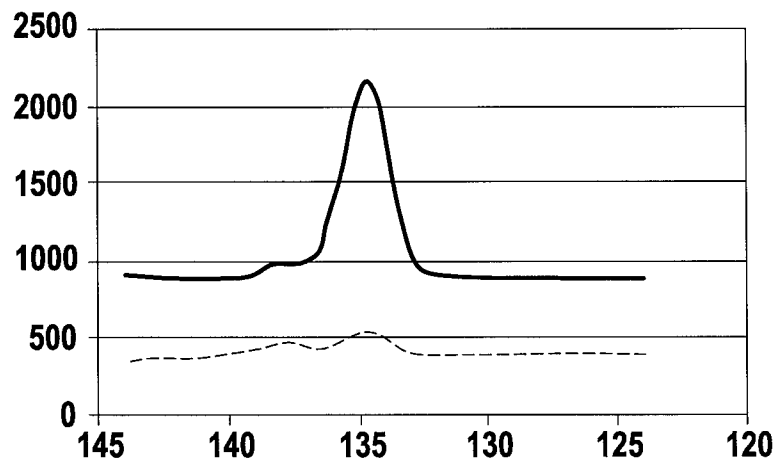


FIG. 3

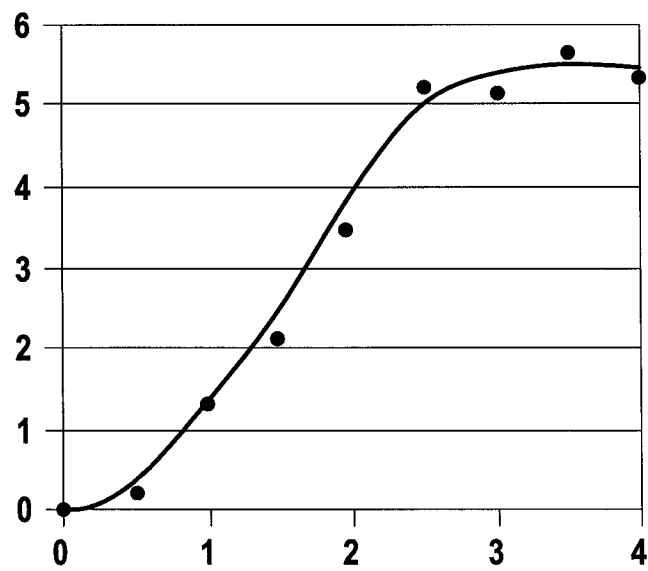


FIG. 4

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2016/054216

A. CLASSIFICATION OF SUBJECT MATTER
INV. H01M10/0567 H01M10/44
ADD.

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
H01M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

EPO-Internal, WPI Data

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	KANG XU ET AL: "Graphite/Electrolyte Interface Formed in LiBOB-Based Electrolytes", JOURNAL OF THE ELECTROCHEMICAL SOCIETY, vol. 151, no. 12, 1 January 2004 (2004-01-01), page A2106, XP55167733, ISSN: 0013-4651, DOI: 10.1149/1.1812732 page A2107, left-hand column, line 27, page A2109, left-hand column, line 9 page A2110, right-hand column, line 11, page A2111, right-hand column, line 18 ----- -/-	1-9



Further documents are listed in the continuation of Box C.



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INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2016/054216

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	HITOSHI OTA ET AL: "TPD GC/MS analysis of the solid electrolyte interface (SEI) on a graphite anode in the propylene carbonate/ethylene sulfite electrolyte system for lithium batteries", JOURNAL OF POWER SOURCES, ELSEVIER SA, CH, vol. 97, 4 February 2001 (2001-02-04), pages 107-113, XP028142748, ISSN: 0378-7753, DOI: 10.1016/S0378-7753(01)00738-8 [retrieved on 2011-02-15] Section 3.2	1-9
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Information on patent family members

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2015060290	A1	05-03-2015	NONE
