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(54) Title: LITHIUM-ION BATTERY FORMATION PROCESS

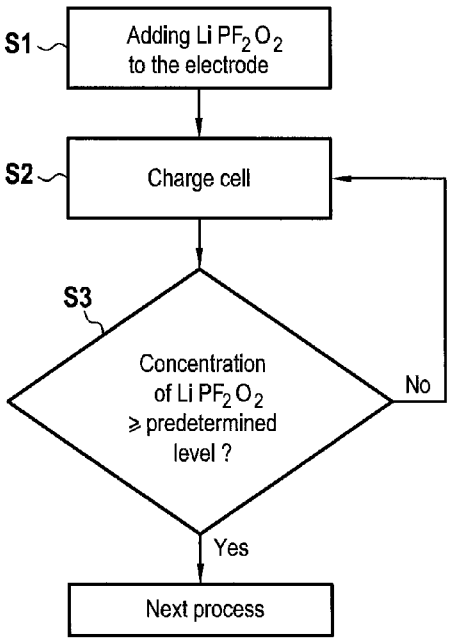

FIG.2

(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: (S1) adding lithium difluorophosphate $LiPF_2O_2$ as an additive to the electrolyte (22) for improving a solid electrolyte interface SEI (24) build-up on the anode (12); and (S2) charging the cell (10) up to a predetermined cell voltage (V_c) at a predetermined rate (C), so that (S3) the lithium difluorophosphate concentration in the solid electrolyte interface (24) reaches a predetermined minimum concentration level. The invention further relates to a lithium-ion cell (10) wherein the anode solid electrolyte interface SEI comprises at least 2 at % of lithium difluorophosphate $LiPF_2O_2$.

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LITHIUM-ION BATTERY FORMATION PROCESS

FIELD OF THE DISCLOSURE

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

BACKGROUND OF THE DISCLOSURE

[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.

[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.

[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.

[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.

[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 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] Additives have also been added to the electrolyte to improve the formation of the SEI and therefore enhancing the capacity stability (i.e. capacity retention).

[0010] JP2013098099 (A) describes a method of manufacturing a lithium ion secondary battery, wherein lithium difluorophosphate ($LiPF_2O_2$) is added as an additive to the electrolyte. The method is controlled based on measuring the amount $LiPF_2O_2$ in the electrolyte. However, the method may control the resulting capacity stability of the battery only in a limited manner.

SUMMARY OF THE DISCLOSURE

[0011] Currently, it remains desirable to control the amount of additive in such a manner that enhanced capacity stability can be realized reliably.

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

- adding lithium difluorophosphate ($LiPF_2O_2$) as an additive to the electrolyte for improving a solid electrolyte interface build-up on the anode; and
- charging the cell up to a predetermined cell voltage at a predetermined charge rate, so that the lithium difluorophosphate concentration in the solid electrolyte interface reaches a predetermined minimum concentration level.

[0013] By providing such a method, the amount of $LiPF_2O_2$ (i.e. its concentration in at%) on the anode surface can be determined. Hence, a direct measurement of the surface composition may be done. It has been found that by controlling the amount $LiPF_2O_2$ on the anode surface (i.e. the SEI) the capacity stability can be enhanced more reliably, than by controlling the amount of $LiPF_2O_2$ in the electrolyte. Moreover, the additive concentration in the SEI may be measured by accurate methods.

[0014] The determination of the predetermined cell voltage and/or the predetermined charge rate may be performed prior to applying a formation

process to a cell of a certain cell type. For example, test cells of the same cell type may be charged by applying different charge rates, respectively. The resulting concentration of $LiPF_2O_2$ of the SEI and the capacity stability (i.e. retention) may be measured subsequently. The concentration of $LiPF_2O_2$ in the SEI increases with a decreasing charge rate. Since the capacity stability increases with increasing concentration of $LiPF_2O_2$ of the SEI, a desired minimum capacity stability can be achieved by controlling that the $LiPF_2O_2$ of the SEI reaches a predetermined minimum concentration level.

[0015] Accordingly, once such a predetermined minimum concentration level is determined, also the corresponding maximum charge rate may be determined. Subsequently, when the formation process of regular cells of the same cell type as the test cells is carried out, charging may be controlled by controlling the charge rate (and eventually also the cell voltage) instead of measuring the concentration of $LiPF_2O_2$ in the SEI.

[0016] However, it is also possible that also the formation process of the regular cells is controlled by controlling the concentration level of $LiPF_2O_2$ of their SEIs.

[0017] The predetermined charge rate may be chosen as a function of the concentration of the lithium difluorophosphate in the SEI.

[0018] The concentration of the lithium difluorophosphate of the SEI may be measured by XPS.

[0019] 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 light elements such 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.

[0020] The charge rate may be maximally 2C, desirably1 C, and more desirably smaller than or equal to 0.5 C.

[0021] The predetermined minimum concentration level of the lithium difluorophosphate in the SEI may be at least 2 at% (atomic %). Said concentration is desirably measured based on the peak area of the second lowest peak between 130 to 142 eV of the XPS spectrogram. Said second lowest peak indicates desirably a concentration level of more than 1 at% of the

lithium difluorophosphate, so that tiny peaks are excluded from the selection of the second lowest peak.

[0022] If the concentration doesn't reach to the desired concentration of 2 at%, several charging cycles may are applied to the cell in the method.

[0023] The predetermined cell voltage may be 3 V or more. However, carbonate type chemicals can react above 3V dominantly. To be accurate, a predetermined cell voltage below 3V is desirable.

[0024] The disclosure further relates to a rechargeable cell, in particular to a lithium-ion cell comprising:

an anode with a SEI,

a cathode,

an electrolyte, and

a separator, wherein

the SEI comprises lithium difluorophosphate ($LiPF_2O_2$) with a concentration of at least 2 at%.

[0025] In this concentration range of $LiPF_2O_2$ the cell can have an acceptable capacity stability.

[0026] The cell may be formed by the formation process as described above.

[0027] The anode may comprise graphite.

[0028] The cathode may comprise $LiNo_{1/3}Co_{1/3}Mn_{1/3}O_2$.

[0029] The separator may be made of a film comprising polyethylene.

[0030] The electrolyte may comprise a mixture of ethylene carbonate, dimethyl carbonate and ethyl methyl carbonate, in particular present in equal volume ratio.

[0031] The electrolyte may comprise $LiPF_6$, in particular at 1 mol/L.

[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.

[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.

BRIEF DESCRIPTION OF THE DRAWINGS

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

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

[0037] Fig. 3 shows a XPS spectrum indicating the $LiPF_2O_2$ concentration of the SEI.

DESCRIPTION OF THE EMBODIMENTS

[0038] 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.

[0039] 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.

[0040] Typically, the anode 12 is made of a carbonaceous material and/or graphite. The anode current collector 14 may be made of copper. The cathode 16 may be made of an intercalated lithium compound, e.g. $LiNi_{1/3}Co_{1/3}Mn_{1/3}O_2$. The cathode current collector 18 may be made of aluminum. The separator 20 may be made of a film comprising polyethylene.

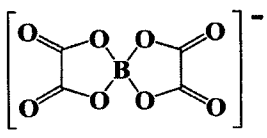
[0041] The electrolyte 22 may be a mixture of ethylene carbonate, dimethyl carbonate and ethyl methyl carbonate present in equal volume ratio. The electrolyte may also comprise $LiPF_6$ at 1 mol/L (mole/litre).

[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.

[0045] Examples of oxalate salts may include Lithium bis(oxalate)borate :

[0046] 

[0047] In particular, the additive may be lithium difluorophosphate ($LiPF_2O_2$), more in particular added at 5 wt% (weight percent) to the electrolyte 22.

[0048] 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.

[0049] Fig. 2 shows a flow chart illustrating an exemplary method according to embodiments of the present disclosure. This method is desirably carried out with test cells of the same cell type, in order evaluate for this cell type a suitable cell voltage and charge rate in the formation process according to the disclosure. This evaluation is based on the measured difluorophosphate ($LiPF_2O_2$) concentration. Once these parameters of a suitable cell voltage and charge rate are known for the test cells, subsequent formation processes of regular cells of the same cell type may be controlled based on these parameters.

[0050] In step S1, lithium difluorophosphate ($LiPF_2O_2$) is added as an additive to the electrolyte 22. This additive serves for improving the solid electrolyte interface 24 build-up on the anode during first charging.

[0051] In step S2 the first charging of the cell is started. This means that the SEI is also started to be built up. This further implies that the concentration of $LiPF_2O_2$ in the SEI is increased during charging.

[0052] The battery is charged up to a predetermined cell voltage, e.g. 3 V or more. Moreover charging is done at a predetermined charge rate C, e.g. a charge rate C of maximally 1 C, desirably smaller than or equal to 0.5 C.

[0053] In step S3 the concentration of $LiPF_2O_2$ is determined. This may be done by a XPS measurement. For this purpose the cell 10 is desirably disassembled.

[0054] It is further determined in step S3, whether the determined $LiPF_2O_2$ concentration is equal to or greater than a predetermined concentration (a predetermined minimum concentration level), in particular 2 at% (atomic %). If this is not the case, charging is continued by returning to step S2.

[0055] However, in case the determined $LiPF_2O_2$ concentration is equal to or greater than the predetermined minimum concentration level, charging may be stopped. Anyhow, it may also be possible that charging is not stopped but charging is continued, e.g. in case the battery has not yet reached its fully

charged state. In such a case charging may be continued, but eventually with a different charge rate, in particular with a greater charge rate, e.g. 3 C.

[0056] The $LiPF_2O_2$ concentration in the SEI can be increased by continuing charging. The capacity stability (i.e. capacity retention) of a cell is a function of the $LiPF_2O_2$ concentration in the SEI. Therefore the $LiPF_2O_2$ concentration can be used as a threshold to determine, whether the cell has reached a satisfactory capacity stability. Accordingly test cells of a certain cell type may be used to determine the target concentration of the $LiPF_2O_2$.

[0057] It has been found that the capacity stability is in a desirable range, when the $LiPF_2O_2$ concentration is at least 2 at%. This has been determined in a test, as it is schematically described in the following.

[0058] A given number of test cells 10 (i.e. samples) of the same type having the same components were charged up to the same cell voltage, e.g. 3V, but at different charge rates, e.g. between 0.01 C and 5 C, respectively. All test cells have been provided before charging with the same additive $LiPF_2O_2$ to the electrolytes in the same concentration.

[0059] Table 1 summarizes the resulting characteristics of the cells after the formation process.

[0060]

	Charge rate	Terminal Voltage	Atomic % of Peak A	Capacity retention (%)
Sample 1	0.01 C	3V	3.73	98.4
Sample 2	0.1C		3.33	98.0
Sample 3	1C		3.08	97.5
Sample 4	3C		1.85	94.3
Sample 5	5C		1.21	93.6

Table1: Formation condition and result of XPS and capacity retention

[0061] As it can be seen in this test, samples 1 to 3 have a capacity retention in an acceptable range, i.e. at least 97.5% (cf. sample 3). These samples have been charged at a charge rate of maximally 1 C (cf. sample 3). Their minimum $LiPF_2O_2$ concentration is 3.08 at% (cf. sample 3). Accordingly, and in particular due to further corresponding tests, a suitable $LiPF_2O_2$ concentration at the end of the formation process has been found to be at least 2 at%.

[0062] The $LiPF_2O_2$ concentration has been determined by XPS measurement. The anodes of the samples were dipped in a solution of ethyl methyl carbonate for 10 minutes and dried. They were set inside a glove box and brought to measurement in a closed chamber. The anodes were then ready for XPS analysis.

[0063] The X-ray intensity used during the XPS analysis was 1500 eV (electronvolt) and the X-ray diameter was 200 µm (micrometre). The detected angle of the photoelectron was 45 degrees.

[0064] Fig. 3 shows a XPS spectrum indicating the $LiPF_2O_2$ concentration of the SEI.

[0065] The $LiPF_2O_2$ concentration relevant for the present disclosure can be measured by the lowest peak between 130 eV and 124 eV in the XPS spectrum of fig. 3. It is indicated in fig. 3 as "Peak A". The atomic % can be calculated by determining the peak area. Accordingly, this peak is representative for the $LiPF_2O_2$ concentration and may therefore be used to control the capacity retention (i.e. capacity stability) of the cell.

[0066] For determining the $LiPF_2O_2$ concentration the total share (concentration) of elements is calculated by between whole energy. From this calculation the phosphorus share (concentration) can be obtained. However phosphorus consists of two peaks. Therefore, separation by Gaussian distribution fitting is needed. And then, the share of the two peaks can be obtained (peak A and the other peak). The following equation may be used for determining the Peak A concentration:

(Phosphorus concentration) * (Peak A share) /100 = Peak A concentration

[0067] The fitting curve in fig. 3 may be used, because XPS peaks follow the normal distribution (Gaussian distribution). In case of multiple peaks overlapping, Gaussian fitting is necessary to calculate area separately. For instance, peak A is derived from $LiPF_2O_2$ but the other peak is derived from another phosphorus component.

[0068] The capacity retention was determined by applying a charge-discharge cycle test to the test cells. Said test included charge and discharge between 3 V and 4 V at current rate of 2C, wherein desirably 500 cycles were carried out at room temperature. The capacity retention may be calculated by the equation (Capacity retention) = ((Discharge capacity after cycle test) / (Discharge capacity after formation))*100 (%).

[0069] 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 any use of the terms "substantially" and/or "approximately" and/or "generally" should be understood to mean falling within such accepted tolerances.

[0070] Although the present disclosure herein has been described with reference to particular embodiments, it is to be understood that these embodiments are merely illustrative of the principles and applications of the present disclosure.

[0071] 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.

[0072] The method is described in terms of a single cell. However, it may be easily adapted for batteries having multiple cells. Moreover it may also refer to other cell types than lithium-ion cells.

CLAIMS

1. A method of performing a formation process for a rechargeable cell (10) comprising an anode (12), a cathode (16), an electrolyte (22) and a separator (20), the formation process comprising:

- adding lithium difluorophosphate ($LiPF_2O_2$) as an additive to the electrolyte (22) for improving a solid electrolyte interface (24) build-up on the anode (12); and
- charging the cell (10) up to a predetermined cell voltage (V_C) at a predetermined charge rate (C), so that the lithium difluorophosphate concentration in the solid electrolyte interface (24) reaches a predetermined minimum concentration level.

2. The method according to claim 1, wherein the predetermined charge rate (C) is chosen as a function of the concentration of the lithium difluorophosphate in the SEI.

3. The method according to claim 1 or 2, wherein the concentration of the lithium difluorophosphate of the SEI (24) is measured by XPS.

4. The method according to any of claims 1 to 3, wherein the charge rate (C) is maximally 2C, desirably 1 C, and more desirably smaller than or equal to 0.5 C.

5. The method according to any of claims 1 to 4, wherein the predetermined minimum concentration level of the lithium difluorophosphate in the SEI is at least 2 at%, wherein said concentration is desirably measured based on the peak area of the second lowest peak between 130 to 142 eV of the XPS spectrogram.

6. The method according to any of claims 1 to 5, wherein the predetermined cell voltage (V_C) is maximally 3 V.

7. A rechargeable cell (10) comprising:

- an anode (12) with an SEI (24),

- a cathode (16),
- an electrolyte (22), and
- a separator (20), wherein

the SEI comprises lithium difluorophosphate ($LiPF_2O_2$) with a concentration of at least 2 at%,.

8. The cell (10) of claim 7, wherein

the cell (10) is formed according to any one of the preceding method claims 1 to 6.

9. The cell (10) of claim 7 or 8, wherein

the anode (12) comprises graphite and/or the cathode (16) comprises $LiNo_{1/3}Co_{1/3}Mn_{1/3}O_2$.

10. The cell (10) any one of claims 7 to 9, wherein

the separator (20) is made of a film comprising polyethylene.

11. The cell (10) any one of claims 7 to 10, wherein

the electrolyte (22) comprises a mixture of ethylene carbonate, dimethyl carbonate and ethyl methyl carbonate, in particular present in equal volume ratio, and/or the electrolyte (22) comprises $LiPF_6$, in particular at 1 mol/L.

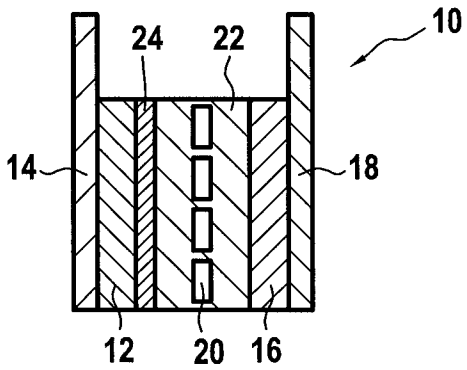

10
24
22
14
18
12
20
16

FIG.1

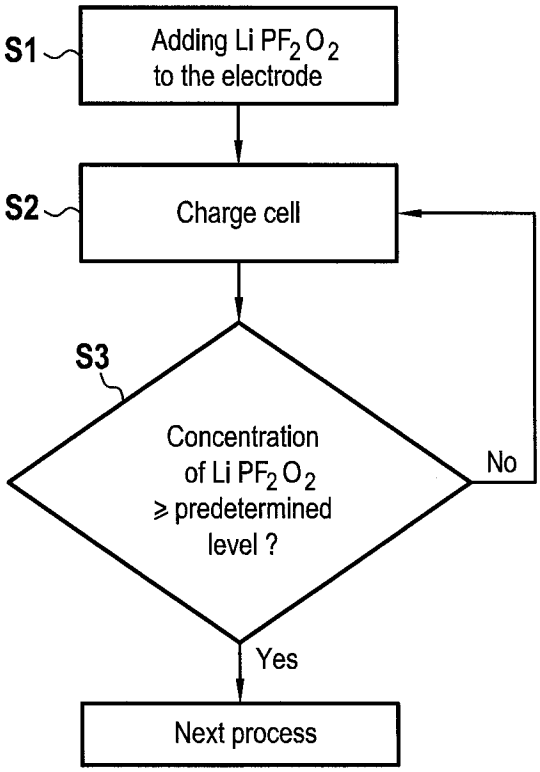

S1
Adding Li PF_2O_2 to the electrode
S2
Charge cell
S3
Concentration of Li PF_2O_2 ≥ predetermined level ?
No
Yes
Next process

FIG.2

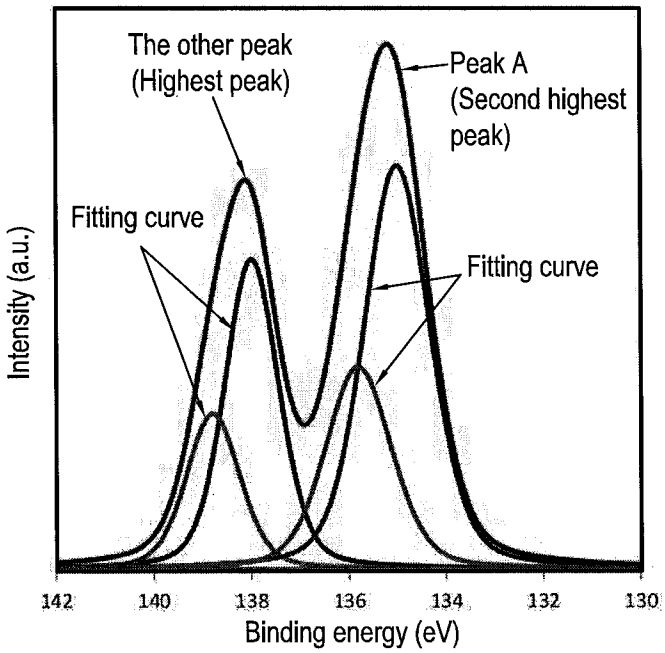

The other peak
(Highest peak)
Peak A
(Second highest
peak)
Fitting curve
Fitting curve
Intensity (a.u.)
142
140
138
136
134
132
130
Binding energy (eV)

FIG.3

INTERNATIONAL SEARCH REPORT

International application No
PCT/EP2016/054117

A. CLASSIFICATION OF SUBJECT MATTER
INV. H01M10/0567 H01M10/44
ADD. H01M10/0525 H01M10/42

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 A	US 2014/099555 A1 (ONIZUKA HIROSHI [JP] ET AL) 10 April 2014 (2014-04-10) paragraphs [0003], [0010], [0021] - [0023], [0027], [0045], [0051] - [0062], [0073], [0087] - [0089]; claims 1, 2, 3, 5, 6, 7, 8; tables 1, 2 ----- -/--	1-6 7-11

[X] Further documents are listed in the continuation of Box C.

[X] See patent family annex.

* Special categories of cited documents :

"A" document defining the general state of the art which is not considered to be of particular relevance

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"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
28 April 2016	19/05/2016

Name and mailing address of the ISA/	Authorized officer
European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016	Fauché, Yann

1

Form PCT/ISA/210 (second sheet) (April 2005)

C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	KO-EUN KIM ET AL: "A combination of lithium difluorophosphate and vinylene carbonate as reducible additives to improve cycling performance of graphite electrodes at high rates", ELECTROCHEMISTRY COMMUNICATIONS, vol. 61, 31 October 2015 (2015-10-31), pages 121-124, XP055268760, NL ISSN: 1388-2481, DOI: 10.1016/j.elecom.2015.10.013	7-11
A	abstract; parts "2. Experimental", "3. Results and discussion"; figure 3	1-6

A	US 2014/045042 A1 (TANAKA HIROFUSA [JP] ET AL) 13 February 2014 (2014-02-13) paragraphs [0001], [0006], [0026], [0036] - [0038]; claims 1, 2, 3, 4, 13	1-11

1

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No
PCT/EP2016/054117

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2014099555 A1	10-04-2014	CN 103715451 A	09-04-2014
		JP 5729613 B2	03-06-2015
		JP 2014078360 A	01-05-2014
		US 2014099555 A1	10-04-2014
US 2014045042 A1	13-02-2014	JP 2014035954 A	24-02-2014
		US 2014045042 A1	13-02-2014