



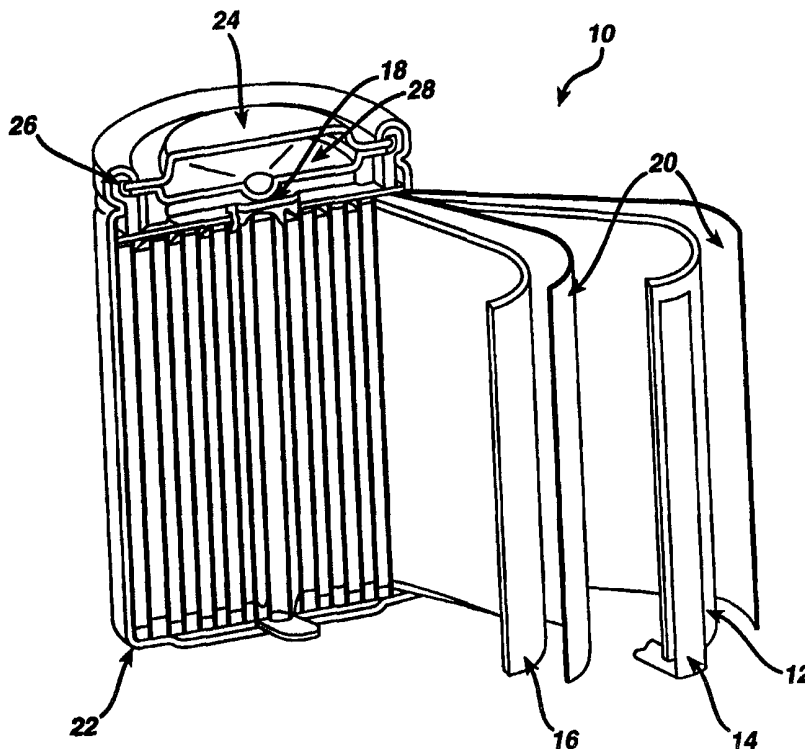
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(54) Title: BATTERY CATHODE MATERIALS

(57) Abstract

Materials having the empirical formula $\text{Li}_x\text{M}_y\text{M}'_{1-y}\text{O}_{2-z}\text{A}_z$ are disclosed. M and M' are different metals, and A is a halogen. x can have a value of from about 0.9 to about 1.2, y can have a value of from greater than zero to less than 1, and z can have a value of from greater than zero to less than 2. The distribution of fluorine atoms can be substantially uniform across the cross section of the materials. The materials can be used in battery cathodes (16) in lithium ion batteries (10).



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BATTERY CATHODE MATERIALS

The invention relates to battery cathode materials.

Batteries are commonly used as energy sources. Typically, a battery includes a negative electrode, called the anode, a positive electrode, called the cathode, and an electrolyte. The battery can further include one or more layers of material, called the separator, that electrically isolate the anode from the cathode when the battery is not in use.

When the battery is used, the anode and the cathode can be electrically connected through an external path so that electrons can flow from the anode to the cathode along the external path. This can cause the anode material to be oxidized while the cathode material can be reduced. During this process, ions can flow between the electrodes through the electrolyte.

One type of battery is called a lithium ion battery. In this type of battery, lithium ions can be transferred from the anode through the electrolyte to the cathode during battery use. During battery recharge, lithium ions can flow from the cathode through the electrolyte to the anode. During use or recharge, lithium ion batteries can heat up.

The invention relates to materials that can be used as cathodes in lithium ion batteries. The materials have good thermal stability, release limited amounts of oxygen upon heating and can exhibit good cyclability.

In one aspect, the invention features a battery cathode formed of a material having the empirical formula $\text{Li}_x\text{M}_y\text{M}'_{1-y}\text{O}_{2-z}\text{A}_z$. M and M' are different metals, and A is a halogen. x can have a value of from about 0.9 to about 1.2. y can have a value of from greater than zero to less than 1, and z can have a value of from greater than zero to less than 2. The battery cathode can be used in a battery that further contains an anode and a separator disposed between the cathode and the anode. Preferably, the material has a substantially uniform distribution of fluorine atoms across its cross-section. Preferably, the oxidation state of nickel is low relative to nonfluorinated lithium metal oxides.

In another aspect, the invention features a battery cathode having a peak power of less than about 100 Joules per gram-minute. Peak power is measured according to the peak power test described below.

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In another aspect, the invention features a method of making a lithium metal oxide material. The method includes combining spherical nickel hydroxide, a lithium precursor, a cobalt precursor and a fluorine precursor. As used herein, "spherical nickel hydroxide" refers to nickel hydroxide in the form of generally spherical particles as measured using scanning electron microscopy. Typically, spherical nickel hydroxide particles have a diameter of from about 5 microns to about 50 microns as measured by light scattering.

In another aspect, the invention features a method of making a material having a peak power of less than about 100 Joules per gram-minute. The method includes combining a nickel precursor, a lithium precursor, a cobalt precursor and a fluorine precursor.

Other features and advantages of the invention will be apparent from the description of the preferred embodiments thereof, and from the claims.

The figure is a sectional view of a lithium ion battery.

The figure shows a lithium ion battery 10 that includes an anode 12 in electrical contact with a negative lead 14, a cathode 16 in electrical contact with a positive lead 18, a separator 20 and an electrolyte. Anode 12, cathode 16 and separator 20 are contained within a case 22.

One end of case 22 is closed with a cap 24 and an annular insulating gasket 26 that can provide a gas-tight and fluid-tight seal. Positive lead 18 connects anode 16 to cap 24. A safety valve 28 is disposed in the inner side of cap 24 and is configured to decrease the pressure within battery 10 when the pressure exceeds some predetermined value.

Cathode 16 can include a lithium metal oxide material. This material can have the empirical formula $\text{Li}_x\text{M}_y\text{M}'_{1-y}\text{O}_2$ or the formula $\text{Li}_x\text{M}_y\text{M}'_{1-y}\text{O}_{2-z}\text{A}_z$. Preferably, the material has a substantially uniform distribution of fluorine atoms across its cross-section.

M and M' are different metals. Preferably, M is Sc, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Al or Mg, and more preferably M is Ni. Preferably, M' is Sc, Ti, V, Cr, Mn, Fe, Ni, Co, Cu, Zn, Al or Mg, and more preferably M' is Co.

A can be a halogen, including fluorine, chlorine, bromine or iodine. Preferably, A is fluorine.

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x is preferably from about 0.9 to about 1.2, more preferably from about 1 to about 1.1, and most preferably from about 1 to about 1.05.

y can have a value of greater than zero and less than 1. Preferably, y is from about 0.4 to about 0.95, more preferably y is from about 0.75 to about 0.85, and most preferably y is about 0.8.

z can have a value of greater than zero and less than 2. Preferably, z is from about 0.001 to about 0.4, more preferably from about 0.03 to about 0.2, and most preferably from about 0.05 to about 0.1.

The preferred lithium metal oxide has a low peak power and a low maximum rate of mass loss, corresponding to a material having good thermal stability.

The lithium metal oxide can have a peak power of less than about 100 Joules per gram-minute. Preferably, the lithium metal oxide from which cathode 16 is formed has a peak power of less than about 70 Joules per gram-minute, more preferably less than about 40 Joules per gram-minute, and most preferably less than about 30 Joules per gram-minute to about 40 Joules per gram-minute.

The peak power test is conducted as follows. 2.7 grams of lithium metal oxide is mixed with 0.15 grams of carbon black (Shawinigan Black, Chevron, located in California) and 0.15 grams of Teflon (DuPont, located in Wilmington, DE) to provide a cathode sample. 0.12 grams of the cathode sample is pressed into one half of an aluminum coin cell can (2430 size) and dried in a vacuum for about four hours at about 150°C. An excess of lithium metal (anode sample) is pressed onto the other half of the aluminum coin cell can. Once the cathode sample is dry, both the cathode sample and anode sample are immediately placed into an argon atmosphere drybox. One layer of separator material (HiPore H-4030V, Asahi Chemicals, Japan) is placed on the cathode sample, and one layer of polypropylene oxide (Pellon) is placed on the anode sample.

Both the cathode sample and the anode sample are soaked with a one molar solution of LiPF_6 in ethylene carbonate and dimethyl carbonate (50:50 by volume), assembled together with a plastic grommet to prevent shorting, and crimped.

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The coin cell is then charged at about 0.5 milliamps to about 4.8 volts until full delithiation occurred. The charged cell is pumped in the drybox and disassembled in the drybox without shorting the cell.

Six milligrams of the cathode sample is placed into a gold sample pan. The cathode sample is soaked in about two microliters of a solution of one molar LiPF_6 in ethylene carbonate and dimethyl carbonate (50:50 by volume). The pan is then hermetically sealed with a gold lid.

The cathode sample is taken from the drybox and placed into a DSC instrument (TA Instruments model 2010, Wilmington, DE), and the temperature of the cathode sample is increased from room temperature to about 300°C at a rate of about 5°C per minute. During this temperature ramp, the cathode sample is kept under a flow of argon gas (about 50 cubic centimeters per minute).

The heat given off by the cathode sample is measured as the temperature of the cathode sample is increased, commonly referred to as an exotherm. The time over which this heat is given off is determined by the point where the exotherm begins to deviate from the baseline by more than the noise level to the point where the exotherm returns to within the noise level of the baseline. The total amount of heat given off by the cathode sample during this time is divided by the time and the mass of the cathode sample. To compensate for the fact that a portion of the mass of the sample was carbon and/or Teflon, the measured value is multiplied by 0.9.

The lithium metal oxide from which cathode 16 is made can have a maximum rate of mass loss of less than about 0.3% per $^\circ\text{C}$. Preferably, the lithium metal oxide from which cathode 16 is formed has a maximum rate of mass loss of less than about 0.25% per $^\circ\text{C}$, more preferably less than about 0.2% per $^\circ\text{C}$, and most preferably less than about 0.15% per $^\circ\text{C}$.

The maximum rate of mass loss can be measured as follows. 1.9 grams of lithium metal oxide is mixed with 0.1 grams of carbon black (Shawinigan Black, Chevron) to form a cathode sample. 50 to 100 milligrams of the cathode sample is pressed into an aluminum coin cell can (2430 size) having an aluminum mesh attached to it and dried under vacuum for about four hours at about 150°C . An excess of lithium metal (anode sample) is pressed onto the other half of the

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aluminum coin cell can. Once the cathode is dried, both the cathode sample and the anode sample are immediately placed into an argon atmosphere drybox. One layer of separator material (Asahi) is placed on the cathode sample, and one layer of polypropylene oxide (Pellon) is placed on the anode sample.

5 Both the cathode sample and the anode sample are soaked with a solution of one molar LiPF_6 in ethylene carbonate and dimethyl carbonate (50:50 by volume), assembled together with a plastic grommet to prevent shorting, and crimped.

The coin cell is then charged at about 0.2 milliamps to about 4.8
10 volts until full delithiation occurred. The charged cell is pumped in the drybox and disassembled in the drybox without shorting the cell. The aluminum mesh is pried away from the can and the cathode sample is washed into a centrifuge tube using dimethyl carbonate.

The cathode sample is then washed with excess dimethyl carbonate
15 and allowed to settle in the centrifuge tube. Most of the dimethyl carbonate is decanted, and the cathode sample is dried under vacuum at room temperature overnight. The dried sample is placed in a thermal gravimetry analysis (TGA) instrument (TA Instruments model 2950) under an argon atmosphere, and the rate of mass loss is measured as the sample is heated at a rate of about 10°C per minute to
20 a temperature of about 500°C .

The lithium metal oxide material can be made by combining and heating a nickel precursor, a cobalt precursor, a lithium precursor and a halogen precursor. For example, the precursors can be ground in a mortar and pestle to form a homogenized mixture and heated in a furnace. The peak temperature used
25 when heating the combined precursors can be, for example, from about 600°C to about 800°C . Preferably, the peak temperature is from about 650°C to about 720°C .

The nickel precursor can be any material which, upon being heated to at least about 600°C , readily decomposes to provide nickel atoms that can be
30 incorporated into the lithium metal oxide material. Nickel precursors include nickel hydroxide, spherical nickel hydroxide, nickel carbonate, nickel oxide and nickel acetate. Preferably, the nickel precursor is spherical nickel hydroxide.

The lithium precursor can be any material which, upon being heated to at least about 600°C, readily decomposes to provide lithium atoms that can be incorporated into the lithium metal oxide material. Lithium precursors include lithium carbonate, lithium hydroxide, lithium acetate and lithium oxalate.

5 Preferably, the lithium precursor is lithium hydroxide.

The cobalt precursor can be any material which, upon being heated to at least about 600°C, readily decomposes to provide cobalt atoms that can be incorporated into the lithium metal oxide material. Cobalt precursors include CoO_4 , Co_3O_4 , Co_2O_3 , CoO , cobalt carbonate and cobalt acetate. Preferably, the cobalt

10 precursor is Co_3O_4 .

The halogen precursor can be any material which, upon being heated to at least about 600°C, readily decomposes to provide halogen atoms that can be incorporated into the lithium metal oxide material. Halogen precursors include lithium fluoride, nickel fluoride, cobalt fluoride, ammonium fluoride, fluorine gas, lithium chloride, nickel chloride, cobalt chloride, ammonium chloride, chlorine gas, lithium bromide, nickel bromide, cobalt bromide, ammonium bromide, lithium iodide, nickel iodide, cobalt iodide, and ammonium iodide. Preferably, the halogen precursor is nickel fluoride.

If the lithium metal oxide has the empirical formula $\text{Li}_x\text{M}_y\text{M}'_{1-y}\text{O}_2\text{A}_z$, the lithium metal oxide can be formed by flowing fluorine gas over material having the empirical formula $\text{Li}_x\text{M}_y\text{M}'_{1-y}\text{O}_2$ at temperature of at least about 600°C, preferably from about 600°C to about 800°C.

Anode 12 can include any materials suitable for use in the anode of a lithium ion battery. For example, anode 12 can be formed of a highly porous sintered, felt, or foam substrate having a coating of anode material thereon.

The anode material can be formed of an active material and a binder. The binder can be, for example, a polymeric binder. The active anode material can include lithium, carbon, graphite, acetylenic mesophase carbons, coke, polyacenic semiconductors, metal oxides, and lithiated metal oxides having an electrochemical potential that is greater than the electrochemical potential of the cathode.

30 The active anode material and binder can be mixed to form a paste which can be applied to the substrate of anode 12.

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Separator 20 can be formed of any of the standard separator materials used in lithium ion batteries. For example, separator 20 can be formed of polypropylene, polyethylene, a polyamide (e.g., a nylon), a polysulfone and/or a polyvinyl chloride.

5 Separator 20 preferably has a thickness of from about 0.1 millimeters to about 2 millimeters, and more preferably from about 0.2 millimeters to about 0.5 millimeters.

Separator 20 can be cut into pieces of a similar size as anode 12 and cathode 16 and placed therebetween as shown in the figure. Anode 12, cathode 16
10 and separator 20 can then be placed within case 22 which can be made of a metal such as nickel or nickel plated steel, or a plastic such as polyvinyl chloride, polypropylene, a polysulfone, ABS or a polyamide.

Other configurations of battery 10 can also be used, including the coin cell configuration or the classic (Leclanche) configuration.

15 Case 22 containing anode 12, cathode 16 and separator 20 can be filled with the electrolyte, which can be any electrolyte appropriate for use in lithium ion batteries.

Typically, the electrolyte includes one or more solvents and one or more lithium salts. Solvents include ethylene carbonate, propylene carbonate,
20 dimethyl carbonate, ethylmethyl carbonate diethyl ether, dimethyl ether, methyl propionate, ethyl propionate, methyl butyrate, gamma butyrolactone, dimethoxy ethane, diethoxy ethane, triethyl phosphate and trimethyl phosphate.

Lithium salts include LiPF_6 , LiAsF_6 , LiI , LiBr , LiBF_4 , LiAlCl_4 , LiClO_4 and LiCFSO_3 .

25 Preferably, the electrolyte is a 1 molar solution of LiPF_6 in a 50:50 mixture (by volume) of ethylene carbonate and dimethyl carbonate.

After disposing the electrolyte in can 22, it can be sealed with cap 24 and annular insulating gasket 26.

Example I

30 About 29.478 grams of $\text{LiOH}\cdot\text{H}_2\text{O}$ (available from Alfa Aesar, located in Ward Hill, MA), about 51.530 grams of spherical $\text{Ni}(\text{OH})_2$ (available from Tanaka Chemicals, located in Osaka, Japan), about 2.822 grams of $\text{NiF}_2\cdot 4\text{H}_2\text{O}$

(available from Alfa Aesar) and about 10.742 grams of Co_3O_4 (available from Alfa Aesar) were mixed and ground using a mortar and pestle.

The reactants were put into alumina crucibles and placed in a tube furnace. The reactants were heated to about 700°C over about three hours, kept at a temperature of about 700°C for about eight hours, and ambiently cooled to room temperature.

The reaction mixture was removed from the furnace, reground in a mortar and pestle, and placed back into the furnace. The mixture was heated to about 700°C over about three hours, kept at a temperature of about 700°C for about eight hours, cooled to about 300°C over about eight hours, and then ambiently cooled to room temperature. This yielded about 31.93 grams of material having the empirical formula $\text{Li}_{1.05}\text{Ni}_{0.8}\text{Co}_{0.2}\text{O}_{1.95}\text{F}_{0.05}$.

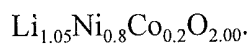
All steps performed in the furnace were done under flowing oxygen.

X-ray diffraction showed that the phase purity of the $\text{Li}_{1.05}\text{Ni}_{0.8}\text{Co}_{0.2}\text{O}_{1.95}\text{F}_{0.05}$ was about 100% (within detection limits) as measured with copper K-alpha radiation using a Rigaku Miniflex X-ray apparatus.

Example II

About 14.739 grams of $\text{LiOH}\cdot\text{H}_2\text{O}$ (Alfa Aesar), about 24.825 grams of spherical $\text{Ni}(\text{OH})_2$ (Tanaka), and about 5.371 grams of Co_3O_4 (Alfa Aesar) were weighed mixed and ground using a mortar and pestle.

The mixture was treated in a furnace as described in Example I. The reaction yielded about 32.03 grams of material having the empirical formula



X-ray diffraction showed that the phase purity of the $\text{Li}_{1.05}\text{Ni}_{0.8}\text{Co}_{0.2}\text{O}_{2.00}$ was about 100% (within detection limits) as measured by the method disclosed in Example I.

Other embodiments are in the claims.

C L A I M S

1. A battery cathode, comprising a lithium metal oxide having an empirical formula $\text{Li}_x\text{M}_y\text{M}'_{1-y}\text{O}_{2-z}\text{A}_z$ wherein M is a first metal, M' is a second metal different from the first metal, A is a halogen, x is from about 0.9 to about 1.2, y is greater than zero and less than 1, z is greater than zero and less than 2, and the lithium metal oxide has a substantially uniform distribution of fluorine atoms across a cross-section of the lithium metal oxide.
2. The battery cathode according to claim 1, wherein z is less than about 0.1.
3. The battery cathode according to claim 1, wherein z is greater than about 0.05.
4. The battery cathode according to claim 1, wherein z is from about 0.05 to about 0.1.
5. The battery cathode according to claim 1, wherein M is selected from the group consisting of Sc, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Al and Mg.
6. The battery cathode according to claim 1, wherein M' is selected from the group consisting of Sc, Ti, V, Cr, Mn, Fe, Ni, Co, Cu, Zn, Al and Mg.
7. The battery cathode according to claim 1, wherein M is Ni and M' is Co.
8. The battery cathode according to claim 7, wherein y is about 0.8.
9. The battery cathode according to claim 8, wherein z is from about 0.05 to about 0.1.
10. The battery cathode according to claim 9, wherein A is fluorine.
11. The battery cathode according to claim 1, wherein A is fluorine.
12. A battery, comprising:
 - a cathode, including a lithium metal oxide having an empirical formula $\text{Li}_x\text{M}_y\text{M}'_{1-y}\text{O}_{2-z}\text{A}_z$, wherein M is a first metal, M' is a second metal different from the first metal, A is a halogen, x is from about 0.9 to about 1.2, y is greater than zero and less than 1, z is greater than zero and less than 2, and the lithium metal oxide has a substantially uniform distribution of fluorine atoms across a cross-section of the lithium metal oxide;
 - an anode; and

- a separator disposed between the cathode and the anode.
13. The battery according to claim 12, wherein z is less than about 0.1.
14. The battery according to claim 12, wherein z is greater than about 0.05.
- 5 15. The battery according to claim 12, wherein z is from about 0.05 to about 0.1.
16. The battery according to claim 12, wherein M is selected from the group consisting of Sc, Ti, V, Cr, Mn, Fe, Ni, Cu, Zn, Al and Mg.
17. The battery according to claim 12, wherein M' is selected from the
10 group consisting of Sc, Ti, V, Cr, Mn, Fe, Ni, Co, Cu, Zn, Al and Mg.
18. The battery according to claim 12, wherein M is Ni and M' is Co.
19. The battery according to claim 18, wherein y is about 0.8.
20. The battery according to claim 19, wherein z is from about 0.05 to about 0.1.
- 15 21. The battery according to claim 20, wherein A is fluorine.
22. The battery according to claim 12, wherein A is fluorine.
23. The battery according to claim 12, wherein the battery is a lithium ion battery.
24. A battery cathode, comprising a material having a peak power of less
20 than about 100 Joules per gram-minute.
25. The battery cathode according to claim 24, wherein the material has a peak power of less than about 70 Joules per gram-minute.
26. The battery cathode according to claim 24, wherein the material has a peak power of less than about 40 Joules per gram-minute.
- 25 27. The battery cathode according to claim 24, wherein the material comprises a lithium metal oxide having an empirical formula $\text{Li}_x\text{M}_y\text{M}'_{1-y}\text{O}_{2-z}\text{A}_z$, wherein M is a first metal, M' is a second metal different from the first metal, A is a halogen, x is from about 0.9 to about 1.2 y is greater than zero and less than 1, z is greater than zero and less than 2, and the lithium metal oxide
30 has a substantially uniform distribution of fluorine atoms across a cross-section of the lithium metal oxide.

28. A battery, comprising:
a cathode, comprising:
a material having a peak power of less than about 100 Joules per
gram-minute;
5 an anode; and
a separator disposed between the cathode and the anode.
29. The battery according to claim 28, wherein the material has a peak
power of less than about 70 Joules per gram-minute.
30. The battery according to claim 28, wherein the material has a peak
10 power of less than about 40 Joules per gram-minute.
31. The battery according to claim 28, wherein the material comprises a
lithium metal oxide having an empirical formula $\text{Li}_x\text{M}_y\text{M}'_{1-y}\text{O}_{2-z}\text{A}_z$, wherein M is a
first metal, M' is a second metal different from the first metal, A is a halogen, x is
from about 0.9 to about 1.2, y is greater than zero and less than 1, z is greater than
15 zero and less than 2, and wherein the lithium metal oxide has a substantially
uniform distribution of fluorine atoms across a cross-section of the lithium metal
oxide.
32. A method of making a lithium metal oxide, the method comprising:
combining spherical nickel hydroxide, a lithium precursor, a cobalt
20 precursor and a fluorine precursor to form the lithium metal oxide.
33. The method according to claim 32, wherein the lithium precursor is
selected from the group consisting of lithium carbonate, lithium hydroxide, lithium
acetate and lithium oxalate.
34. The method according to claim 32, wherein the cobalt precursor is
25 selected from the group consisting of Co_3O_4 , Co_2O_3 , CoO, cobalt carbonate and
cobalt acetate.
35. The method according to claim 32, wherein the fluorine precursor is
selected from the group consisting of lithium fluoride, nickel fluoride, cobalt
fluoride and ammonium fluoride.
- 30 36. The method according to claim 32, wherein the cobalt precursor
comprises Co_3O_4 , the lithium precursor comprises lithium hydroxide and the fluorine
precursor comprises nickel fluoride.

37. A method, comprising:
combining a nickel precursor, a lithium precursor, a cobalt precursor and a fluorine precursor to form a material having a peak power of less than about 100 Joules per gram-minute.
- 5 38. The method according to claim 37, wherein the nickel precursor is selected from the group consisting of nickel hydroxide, nickel carbonate, nickel oxide and nickel acetate.
39. The method according to claim 37, wherein the lithium precursor is selected from the group consisting of lithium carbonate, lithium hydroxide, lithium
10 acetate and lithium oxalate.
40. The method according to claim 37, wherein the cobalt precursor is selected from the group consisting of Co_3O_4 , CoO , Co_2O_3 , cobalt carbonate and cobalt acetate.
41. The method according to claim 37, wherein the fluorine precursor is
15 selected from the group consisting of lithium fluoride, nickel fluoride, cobalt fluoride and ammonium fluoride.
42. The method according to claim 37, wherein the nickel precursor comprises spherical nickel hydroxide.
43. The method according to claim 42, wherein the cobalt precursor
20 comprises Co_3O_4 , the lithium precursor comprises lithium hydroxide and the fluorine precursor comprises nickel fluoride.
44. The method according to claim 37, wherein the cobalt precursor comprises Co_3O_4 , the lithium precursor comprises lithium hydroxide and the fluorine precursor comprises nickel fluoride.

FIG. 1

