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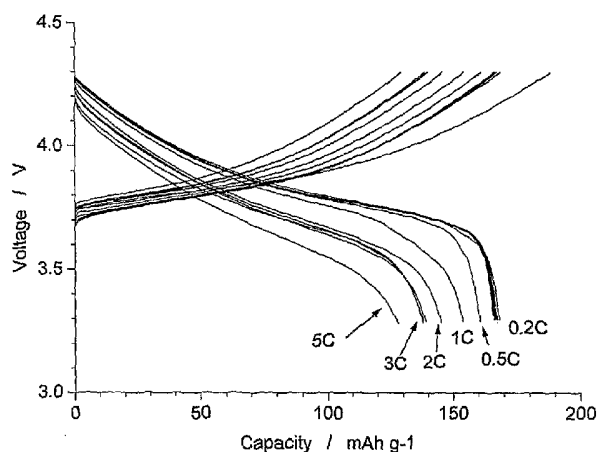
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(54) Title: POSITIVE ELECTRODE ACTIVE MATERIAL FOR LITHIUM ION SECONDARY CELL COATED HETERO METAL OXIDE ON THE SURFACE AND LITHIUM ION SECONDARY CELL COMPRISING IT



(57) Abstract: The present invention relates generally to a positive electrode active material for lithium-ion secondary cells and, more particularly, to a positive electrode active material made of a lithium-containing composite oxide whose surface is coated with a hetero metal oxide. The coated positive electrode active material according to the present invention may have a higher capacity and higher rate capability compared with an uncoated one, and be used for high-power lithium-ion secondary cells. In addition, the coated positive electrode active material may provide a simple method to significantly enhance capacity and rate capability of the lithium-containing composite oxides currently in use or to be developed in the future, hence, it is very usefully applicable to manufacture of lithium-ion secondary cells.



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【DESCRIPTION】**【Invention Title】****POSITIVE ELECTRODE ACTIVE MATERIAL FOR LITHIUM ION
SECONDARY CELL COATED HETERO METAL OXIDE ON THE SURFACE
AND LITHIUM ION SECONDARY CELL COMPRISING IT****【Technical Field】**

The present invention relates generally to a positive electrode active material for lithium-ion secondary cells and, more particularly, to a positive electrode active material coated with a hetero metal oxide.

【Background Art】

Corresponding to the recent widespread use of portable electronic appliances such as cellular phones and notebook computers, and the practical use of electrical vehicles in recent years, there have been continuous demands for small, lightweight, and high-capacity secondary cells.

) Currently, as high-capacity secondary cells according to these demands, non-aqueous electrolytic secondary cells are being commercialized. A typical example may be a lithium-ion secondary cell that includes Lithium Cobalt Oxide (LiCoO_2) and a carbon-containing material in its positive and negative electrodes respectively. Lithium-ion

secondary cells attract attention as a power source for the portable electronic appliances, because they may have favorable characteristics such as high energy density, compactness, and lightweight.

Specifically, the lithium-ion secondary cell may comprise a positive and negative electrode active materials, a current collector, and an electrolyte. The positive and negative electrode active materials generate electricity. The positive electrode active material may comprise a composite oxide containing lithium, preferably a lithium - containing transition metal oxide. The negative electrode active material may comprise a lithium, a lithium alloy, carbon (crystalline or amorphous), or a carbon compound. The current collector may provide a pathway which electrons generated from the active materials pass through, and be made of metals. The electrolyte may play a role as a medium for movement of ions, and comprise a non-aqueous solvent, a lithium salt, and other additives.

A representative positive electrode active material for the lithium-ion secondary cell is Lithium Cobalt Oxide (LiCoO_2). The theoretical discharge capacity of LiCoO_2 is 274mAh/g. However, because overcharge and overdischarge may cause phase changes of LiCoO_2 and shorten the cycle life of the lithium-ion secondary cell, the practical discharge capacity of LiCoO_2 ranges from 125 to 140mAh/g. LiCoO_2 as an

active material may have advantages in terms of easy manufacture and handling. On the other hand, because Cobalt (Co), a raw material of LiCoO_2 , is one of rare metals, Cobalt may cause a serious shortage of resources in manufacturing LiCoO_2 . In addition, because the price of Cobalt is high and varies widely, there exists strong need for alternative positive electrode materials having a low price and stable supply.

For these reasons, lithium manganese oxides may be more promising than LiCoO_2 as the positive electrode active material. Among these, lithium manganese oxides having a spinel structure such as $\text{Li}_2\text{Mn}_4\text{O}_9$, $\text{Li}_4\text{Mn}_5\text{O}_{12}$, LiMn_2O_4 , $\text{Li}[(\text{Ni}_{0.5}\text{Mn}_{0.5})_{1-x}\text{Co}_x]\text{O}_2$ have attracted attention. Especially, $\text{Li}[(\text{Ni}_{0.5}\text{Mn}_{0.5})_{1-x}\text{Co}_x]\text{O}_2$ has good thermal stability and is regarded as one of the promising candidates for the positive electrode active material of next-generation high-power and high-capacity lithium-ion secondary cells. While $\text{Li}[(\text{Ni}_{0.5}\text{Mn}_{0.5})_{1-x}\text{Co}_x]\text{O}_2$ has relatively high capacity and good reversibility, its cobalt content which is a factor to increase electrical conductivity is lower than that of LiCoO_2 . Consequently, the rate capability of $\text{Li}[(\text{Ni}_{0.5}\text{Mn}_{0.5})_{1-x}\text{Co}_x]\text{O}_2$ falls short of our expectations, and its applicability to current high-power and high-capacity secondary cells is still unknown.

【Disclosure】**【Technical Problem】**

The object of the present invention is to solve some problems in conventional positive electrode active materials for lithium-ion secondary cells. The present invention provides through a simple means a new positive electrode active material for lithium-ion secondary cells which has a higher capacity and rate capability than the conventional one. In addition, the present invention provides lithium-ion secondary cells comprising the new positive electrode active material.

【Technical Solution】

In order to achieve these objects, the present invention provides both a new positive electrode active material for lithium-ion secondary cells that is a hetero metal oxide-coated lithium-containing composite oxide and a lithium-ion secondary cell comprising the same.

【Advantageous Effects】

The positive electrode active material for lithium-ion secondary cells according to the present invention is a lithium-containing composite oxide whose surface is coated with a hetero metal oxide. This coated positive electrode active material may have a higher capacity and higher rate capability compared with an uncoated one, and be used for high-power lithium-ion secondary cells. In addition,

through various applications of the hetero metal oxide to the lithium-containing composite oxide usable as a positive electrode active material, the hetero metal oxide may also be applied to the lithium-containing composite oxide to be developed in the future and be used for lithium-ion secondary cells having a significantly enhanced capacity and rate capability.

【Description of Drawings】

Fig. 1 is a graph showing rate capabilities of $\text{Li}_{1.05}\text{Ni}_{0.40}\text{Mn}_{0.40}\text{Co}_{0.15}\text{O}_2$ without a metal oxide coating.

Fig. 2 is a graph showing cycle characteristics of $\text{Li}_{1.05}\text{Ni}_{0.40}\text{Mn}_{0.40}\text{Co}_{0.15}\text{O}_2$ without a metal oxide coating, during 100 charge/discharge cycles with an applied current of 1C (140mA/g).

Fig. 3 is a photograph, taken by a scanning electron microscope (SEM), showing $\text{Li}_{1.05}\text{Ni}_{0.40}\text{Mn}_{0.40}\text{Co}_{0.15}\text{O}_2$ coated with aluminum oxide (Al_2O_3) according to the present invention.

Fig. 4 is an SEM-EDS (energy dispersive spectroscopy) pattern of $\text{Li}_{1.05}\text{Ni}_{0.40}\text{Mn}_{0.40}\text{Co}_{0.15}\text{O}_2$ coated with aluminum oxide (Al_2O_3) according to the present invention.

Fig. 5 is a photograph showing an SEM element mapping image of $\text{Li}_{1.05}\text{Ni}_{0.40}\text{Mn}_{0.40}\text{Co}_{0.15}\text{O}_2$ coated with aluminum oxide (Al_2O_3) according to the present invention.

Fig. 6 is a graph showing rate capabilities of $\text{Li}_{1.05}\text{Ni}_{0.40}\text{Mn}_{0.40}\text{Co}_{0.15}\text{O}_2$ coated with aluminum oxide (Al_2O_3)

according to the present invention, at an applied current of 1C (140mA/g).

Fig. 7 is a graph showing cycle characteristics of $\text{Li}_{1.05}\text{Ni}_{0.40}\text{Mn}_{0.40}\text{Co}_{0.15}\text{O}_2$ coated with aluminum oxide (Al_2O_3) according to the present invention, during 100 charge/discharge cycles with an applied current of 3C (420mA/g).

Fig. 8 is a graph showing cycle characteristics of $\text{Li}_{1.05}\text{Ni}_{0.40}\text{Mn}_{0.40}\text{Co}_{0.15}\text{O}_2$ coated with aluminum oxide (Al_2O_3) according to the present invention, during 100 charge/discharge cycles with an applied current of 5C (700mA/g).

【Best Mode】

Hereinafter, the present invention will be described in detail.

The present invention may be best characterized in that it improves capacity and rate capability of lithium-ion secondary cells by coating a positive electrode active material for lithium-ion secondary cells in current use or to be developed with a hetero metal oxide.

In the present invention, the hetero metal oxide is a metal oxide that is different from the positive electrode active material for lithium-ion secondary cells and is an electrochemically inert material. This hetero metal oxide

coating of a lithium-containing composite oxide, a positive electrode active material, may suppress reactions between thermodynamically unstable Ni^{4+} and Co^{4+} created during charge and HF created in the electrolyte, thereby enhancing capacity and rate capability of lithium-ion secondary cells.

As the hetero metal oxide showing this effect, for example, metal oxides having high electro-negativity such as Al_2O_3 , TiO_2 , ZrO_2 are preferred and they may also be selected according to the kinds of positive electrode active materials.

As mentioned above, the hetero metal oxide is coated on the surface of the positive electrode active material. Any coating method commonly used in this field may be utilized for the hetero metal oxide coating, and coating methods are not limited in the present invention. For example, the coating may be conducted by melting the hetero metal oxide itself in a highly volatile solvent, wherein process conditions are adequately adjusted according to the hetero metal oxide and the positive electrode active material.

The thickness of the hetero metal oxide coating may be determined so as to enhance physical characteristics of the coated positive electrode active material. Specifically, it is preferable that the thickness of the hetero metal oxide coating ranges from 5 to 20nm. If the coating

thickness is less than 5nm, the coating may be too thin to enhance the physical characteristics. If the coating thickness is larger than 20nm, the coating may be too thick and make the positive electrode active material ineffective.

In the present invention, the lithium-containing composite oxide is a base material of the positive electrode active material for lithium-ion secondary cells, and may be any one of lithium-containing composite oxides currently in use or to be developed in the future. Specifically, the lithium-containing composite oxide may be a lithium-containing transition metal oxide, and preferably is LiCoO_2 , LiNiO_2 , LiMnO_2 , LiMn_2O_4 , $\text{Li}[(\text{Ni}_{0.5}\text{Mn}_{0.5})_{1-x}\text{Co}_x']\text{O}_2$ ($0 \leq x' \leq 0.2$), or $\text{Li}_{1+x}[(\text{Ni}_{0.5}\text{Mn}_{0.5})_{1-y}\text{Co}_y]\text{O}_2$ ($0 \leq x \leq 0.1$, $0 \leq y \leq 0.2$).

The positive electrode active material according to the present invention may be applicable to all kinds of lithium-ion secondary cells such as lithium ion and lithium polymer cells.

The lithium-ion secondary cell according to the present invention may be manufactured through publicly known methods using the positive electrode active material. For example, the positive electrode active material together with binders such as polyvinylidone and electrically conductive additives such as carbon black and acetylene black is added into an organic solvent such as N-methyl-2-pyrrolidone, resulting in a positive electrode

active material slurry. This slurry is applied onto a current collector such as an aluminum foil and dried, thereby forming a cathode. Next, the lithium-ion secondary cell is manufactured through a series of steps including placement of a separator between the cathode and an anode made of carbon or a lithium metal, winding under constant tension, insertion into a pouch which is a cell case, electrolyte injection, and sealing.

Hereinafter, embodiments of the present invention will be described in detail. It is to be understood that the following examples serve only as illustrative purposes and the present invention is not limited thereto.

<Example 1> Manufacture of the hetero metal oxide-coated positive electrode active material for lithium-ion secondary cells

(1) Synthesis of $\text{Li}_{1+x}[(\text{Ni}_{0.5}\text{Mn}_{0.5})_{1-y}\text{Co}_y]\text{O}_2$ ($0 \leq x \leq 0.1$, $0 \leq y \leq 0.2$)

Raw materials are nickel sulfate (NiSO_4), manganese sulfate (MnSO_4) and cobalt sulfate (CoSO_4). The stoichiometric ratio in the raw materials is $(\text{Ni}_{0.5}\text{Mn}_{0.5})_{1-y} : \text{Co}_y = 1 \sim 0.8 : 0 \sim 0.2$ ($0 \leq y \leq 0.2$). The value of y indicated in Figs. 1 to 8 is 0.15. These raw materials are dissolved in distilled water, and introduced into a reactor under an inert atmosphere. Ammonium hydroxide is continuously supplied to

the reactor. Obtained composite hydroxide is dried for 24 hours at about 110°C, and then physically mixed with a designated amount of lithium hydroxide, where the stoichiometric ratio between the lithium hydroxide and the composite hydroxide is 1.25. The resulting mixture is heat treated for about 10 hours at about 480°C, and 3 ~ 24 hours at 950 ~ 1200°C, giving the intended material having the formula of $\text{Li}_{1+x}[(\text{Ni}_{0.5}\text{Mn}_{0.5})_{1-y}\text{Co}_y]\text{O}_2$ ($0 \leq x \leq 0.1$, $0 \leq y \leq 0.2$).

(2) Al_2O_3 coating on the positive electrode active material

Aluminum triisopropoxide (1 wt %) is dissolved in a highly volatile solvent. After confirming its complete dissolution (transparent liquid), the synthesized positive electrode active material is put into this aluminum dissolved solution, stirred for reaction using an impeller until the solvent completely evaporates. After complete evaporation of the solvent, the obtained product is heat treated for 5 ~ 24 hours at 400 ~ 500°C.

<Example 2> Manufacture of a lithium-ion secondary cell comprising the hetero metal oxide-coated positive electrode active material

The positive electrode active material manufactured by the method of Example 1, polyvinylidene fluoride, and acetylene black are added into N-methyl-2-pyrrolidone, resulting in a positive electrode active material slurry. This slurry is

applied onto a current collector made of an aluminum foil and dried, thereby forming a cathode. Next, the lithium-ion secondary cell is manufactured through a series of steps including placement of a separator between the cathode and an anode made of a lithium metal, winding under constant tension, insertion into a pouch which is a cell case, electrolyte injection, and sealing.

<Experiment 1> Measurement of the rate capability of the positive electrode active material for lithium-ion secondary cells

In order to examine characteristics of a lithium-ion secondary cell utilizing the positive electrode active material, a charge/discharge experiment was performed using a charge-discharge cycler under the condition: a temperature of 25°C, an electric potential ranging from 3.3 to 4.3V, and a current density ranging from 0.2 to 5C (1C= 140mA/g).

To examine the rate capability of the positive electrode active material, the positive electrode active material having the hetero metal oxide (Al_2O_3) coating (refer to Figs. 3 to 5) manufactured by the method of Example 1 was compared with the positive electrode active material without the Al_2O_3 coating. The measurement results are shown in Figs. 1 and 6. Fig. 1 is related to the

positive electrode active material without the hetero metal oxide (Al_2O) coating, and Fig. 6 is related to the positive electrode active material having the hetero metal oxide (Al_2O_3) coating. It can be seen from Figs. 1 and 6 that the coated one has a higher capacity than the uncoated one. This result is a consequence of the fact that the coating of the hetero metal oxide, electrochemically inert material, suppresses reactions between thermodynamically unstable Ni^{4+} and Co^{4+} created during the charge and HF created in the electrolyte.

<Experiment 2> Measurement of the cycle characteristics of the positive electrode active material for lithium-ion secondary cells

To examine the cycle characteristics of the positive electrode active material for lithium-ion secondary cells, the charge/discharge curves of the positive electrode active material having the hetero metal oxide (Al_2O_3) coating manufactured by the method of Example 1 was compared with those of the positive electrode active material without the Al_2O_3 coating. The measurement results are shown in Figs. 2, 7, and 8. Fig. 2 shows the charge/discharge curves of the positive electrode active material without the hetero metal oxide (Al_2O_3) coating manufactured by the method of Example 1. Fig. 7 shows the

charge/discharge curves of the positive electrode active material having the Al_2O_3 coating during 100 charge/discharge cycles with applied currents of 3C (420mA/g). Fig 8 shows the charge/discharge curves of the positive electrode active material having the Al_2O_3 coating during 100 charge/discharge cycles with applied currents of 5C (700mA/g).

As can be seen from Figs. 7 and 8, the positive electrode active material having the Al_2O_3 coating has maintained very good characteristics during repeated cycles - a capacity of about 140 mAh/g in the case of 3C (charge for 20 minutes, discharge for 20 minutes), and a capacity of about 130 mAh/g in the case of 5C (charge for 12 minutes, discharge for 12 minutes). However, the positive electrode active material without the Al_2O_3 coating did not show these results.

【Industrial Applicability】

Hetero metal oxides usable for coating a positive electrode active material for lithium-ion secondary cells may be variously applicable to lithium-containing composite oxides available for positive electrode active materials. The hetero metal oxides may also be variously applicable to the lithium-containing composite oxides to be developed in

the future, thereby contributing to enhancement of capacity and rate capability of lithium-ion secondary cells.

【CLAIMS】**【Claim 1】**

A positive electrode active material for lithium-ion secondary cells comprising a lithium-containing composite oxide whose surface is coated with a hetero metal oxide.

【Claim 2】

The positive electrode active material for lithium-ion secondary cells of claim 1, wherein the hetero metal oxide is Al_2O_3 , TiO_2 , or ZrO_2 .

【Claim 3】

The positive electrode active material for lithium-ion secondary cells of claim 1, wherein the hetero metal oxide coating has a thickness ranging from 5 to 20nm.

【Claim 4】

The positive electrode active material for lithium-ion secondary cells of claim 1, wherein the lithium-containing composite oxide is LiCoO_2 , LiNiO_2 , LiMnO_2 , LiMn_2O_4 , $\text{Li}[(\text{Ni}_{0.5}\text{Mn}_{0.5})_{1-x}\text{Co}_x]\text{O}_2$ ($0 \leq x \leq 0.2$), $\text{Li}_{1+x}[(\text{Ni}_{0.5}\text{Mn}_{0.5})_{1-y}\text{Co}_y]\text{O}_2$ ($0 \leq x \leq 0.1$, $0 \leq y \leq 0.2$) or $\text{LiNi}_{1-x-y}\text{Co}_x\text{M}_y\text{O}_2$ ($0 \leq x \leq 1$, $0 \leq y \leq 0.2$, $0 \leq x+y \leq 1$, wherein M is Li, Al, Sr, Mg, or La).

【Claim 5】

A lithium-ion secondary cell comprising the positive electrode active material claimed in any of claims 1 to 4.

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FIGURES

FIG. 1

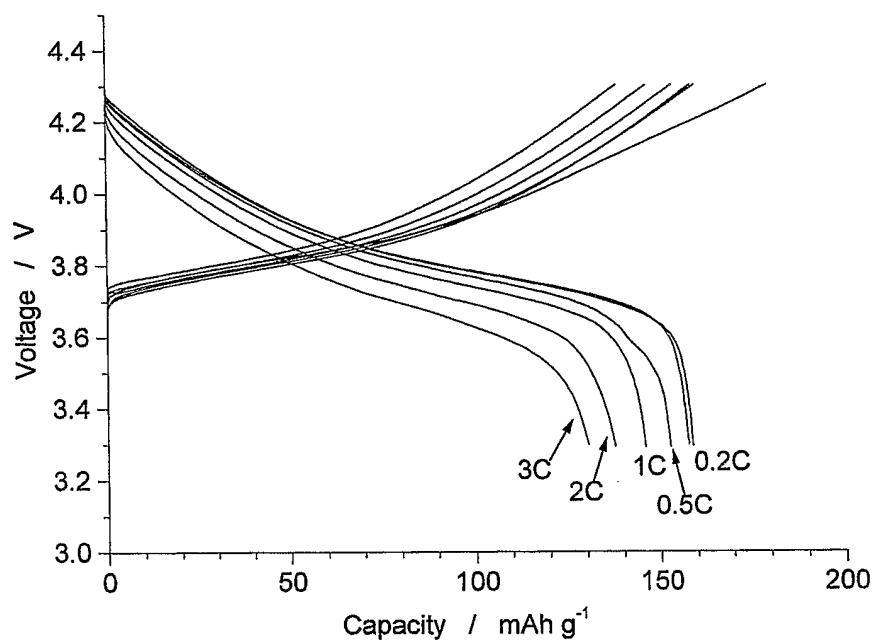
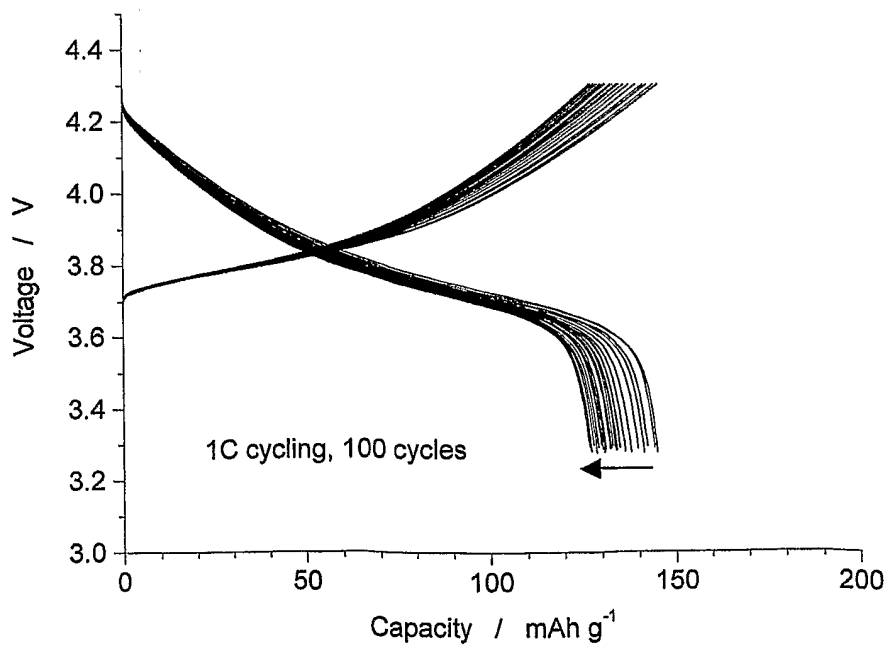


FIG. 2



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FIG. 3

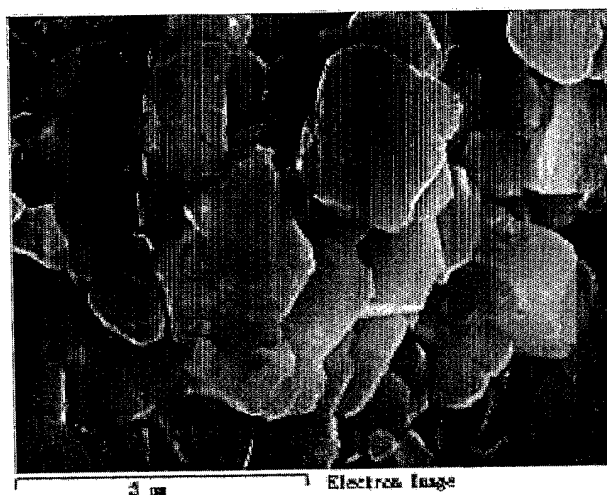


FIG. 4

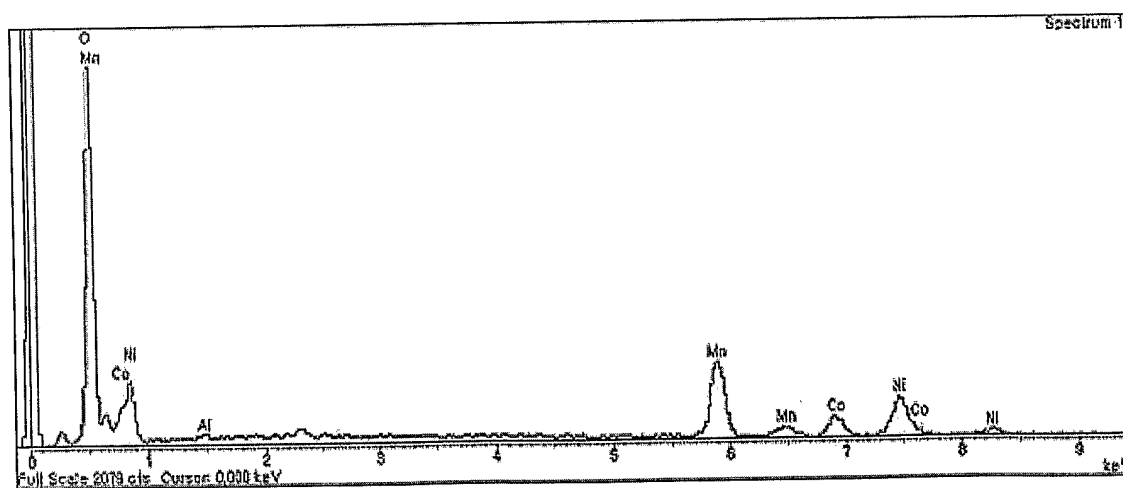
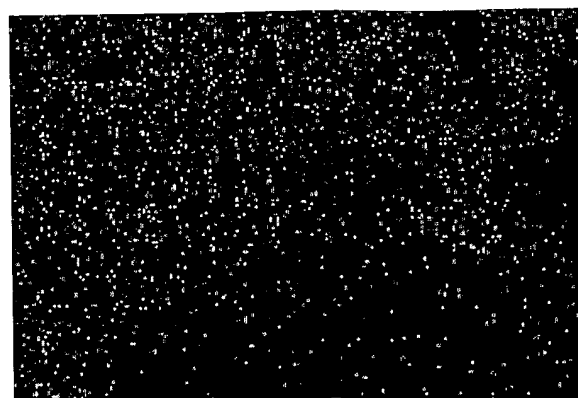
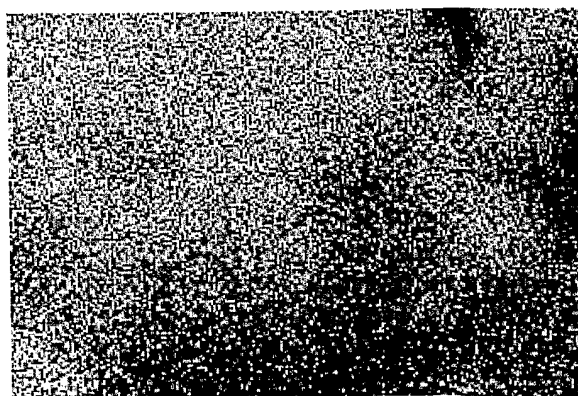


FIG. 5



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FIG. 6

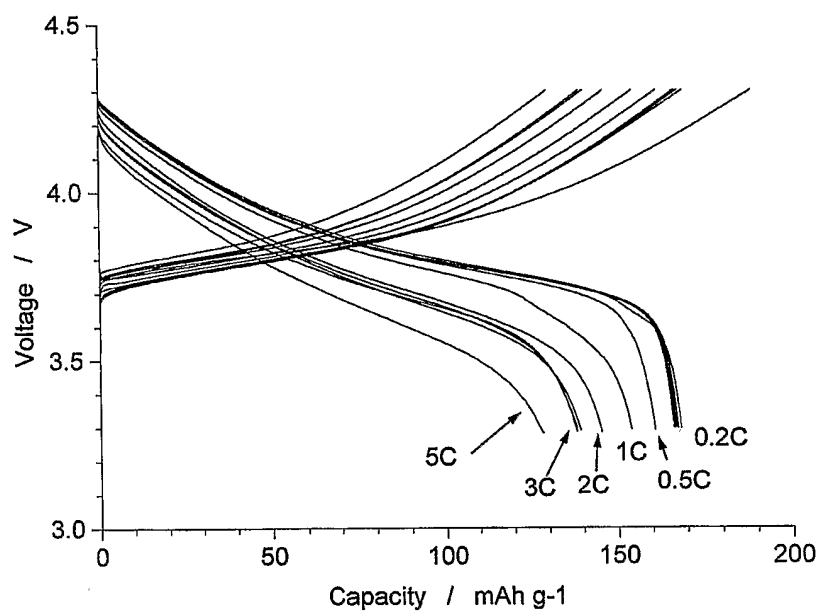
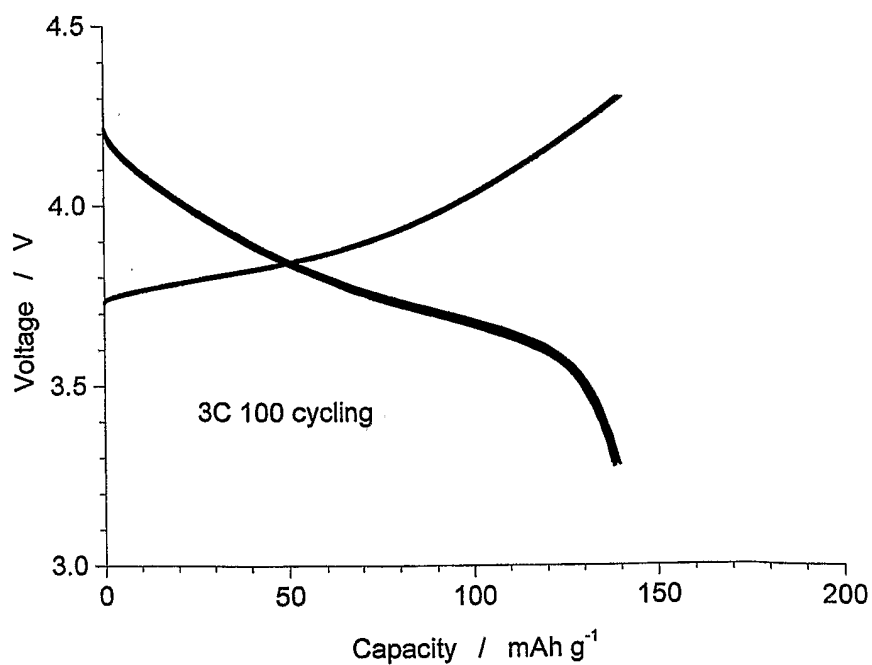
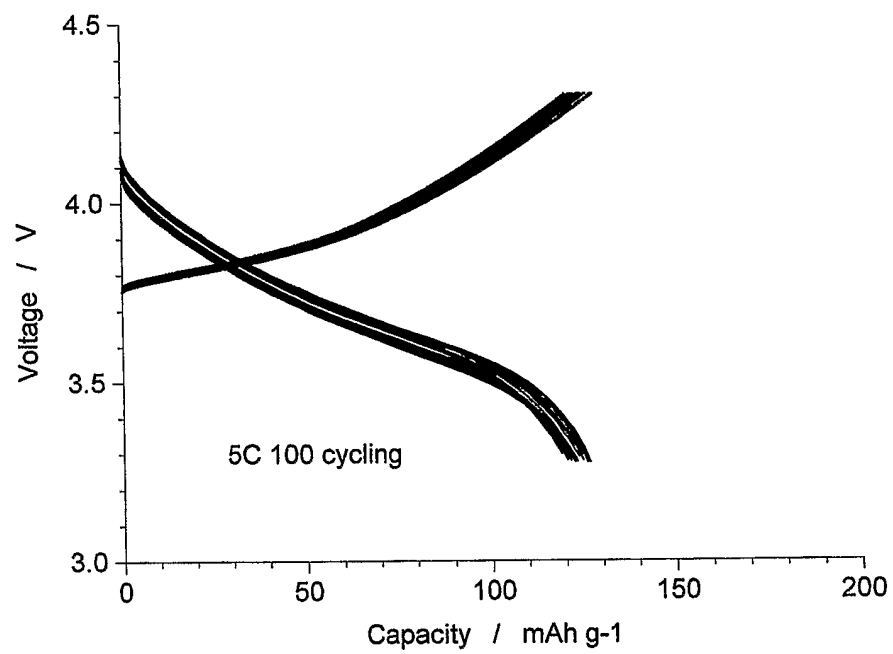


FIG. 7



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FIG. 8



INTERNATIONAL SEARCH REPORT

International application No.
PCT/KR2005/001552**A. CLASSIFICATION OF SUBJECT MATTER****IPC7 H01M 4/58**

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)

IPC7 H01M

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Korean patents and applications for inventions since 1975: IPC as aboveElectronic data base consulted during the international search (name of data base and, where practicable, search terms used)
Delphion and keywords: positive, electrode, active, material, metal, oxide, coating, lithium, battery and similar terms**C. DOCUMENTS CONSIDERED TO BE RELEVANT**

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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X	US 6551571 B2 (TODA KOGYO CORPORATION) April 22, 2003; claim 1-2	1, 4, 5
A	US 2002/0034583 A1 (WON-IL JUNG) March 21, 2002; abstract	1-5
X	EP 1035600 A1 (SAMSUNG DISPLAY DEVICES CO. LTD) September 13, 2000; claim 1-3; column 3, line 19-22	1-5
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X	US 6183911 B1 (SAMSUNG DISPLAY DEVICES CO. LTD) February 6, 2001; claim 1-2	1, 3, 4, 5

☐ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

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