Synthesis of Spherical Cathode Material $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$ by Melting-Salts Coating Process

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Abstract: A spherical cathode material $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$ was synthesized by melting-salts coating process in air using spherical $Ni(OH)_2$ as core material, $Al(NO_3)_3 \cdot 9H_2O$, $Co(NO_3)_2 \cdot 6H_2O$ and $LiNO_3$ as coating materials. The crystal structure, morphology and electrochemical performance of the synthesized powder were characterized by XRD, SEM and charge-discharge experiments. The experimental results show that the synthesized powder $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$ has the α-$NaFeO_2$ type ordered layered structure, spherical morphology and excellent electrochemical performance.

Key words: lithium ion battery; spherical cathode material; $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$; melting-salt coating process
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As a cathode material, $LiNiO_2$ is superior to $LiCoO_2$ in its lower cost and higher specific capacity; however, it suffers from poor cycling reversibility and thermal instability, which prevents its practical uses[1]. These drawbacks are mainly related to its crystal structure, which will undergo phase transformations during the charge/discharge processes especially under higher temperature[2]. In order to improve its stability, various metal cation-substituted $LiNiO_2$ cathode materials have been developed. Recently, $LiNi_{1-x-y}Co_xAl_yO_2$ is considered as a promising cathode material for its improved stability and electrochemical performance brought by Co and Al substitutions for Ni in $LiNiO_2$[3-7].

The cathode materials synthesized by traditional process or high temperature solid-state reactions have many disadvantages, such as inhomogeneity, irregular morphology, large particle size and broad particle size distribution. In this work, we synthesized the $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$ which has regular spherical morphology and narrow particle size distribution by melting-salts coating process.

1 Experimental

$Al(NO_3)_3 \cdot 9H_2O$, $Co(NO_3)_2 \cdot 6H_2O$ and $LiNO_3$ were mixed and heated into melt and coated on the surface of spherical $Ni(OH)_2$. The coated material was firstly prefired at 600℃ for 6 h and then calcined at 700~800 ℃ for 16 h in air.

The crystal phases of as-prepared powders were determined by X-ray diffraction (XRD, Bruker, D8-ADVANCE) using Cu K_α radiation in the 2θ ranges of $10^\circ \sim 70^\circ$. The particle morphology was examined by scanning electron microscopy (SEM, JEOL, JSM-5610LV) operated at 15 kV.

The electrochemical characteristics of the synthesized powders were carried out in a two-electrode test cell. The Charge-discharge measurements were performed at room temperature in a voltage range of 4.30~2.50 V at a constant current density of 0.50 mA using a computer controlled multi-channel battery test system.

2 Results and discussions

2.1 Crystal structure

Fig. 1 shows the XRD patterns of the powders synthesized at 700, 750 and 800 ℃. All of them can be indexed as α-$NaFeO_2$ type layered structure. No other diffraction peak being tested means that the synthesized powders are single phase solid solution $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$. The sharp and clear diffraction peaks means that the synthesized products are well crystallized. High intensity ratio of the (003) and (104) peaks as well as the clearer splitting of the

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(006) / (102) and (108) / (110) peaks indicate that the synthesized products possess high cation order. The intensity ratio of I_{003}/I_{104} of the sample synthesized at 750℃ being higher than those synthesized at other temperatures indicates that 750℃ is the optimal synthesis temperature.

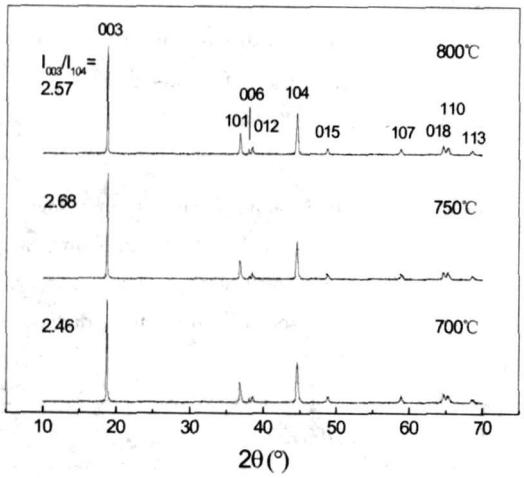

Fig. 1 XRD patterns of $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$ synthesized at different temperature

2.2 Morphology

Fig. 2 is the SEM Photos of $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$ synthesized at 750℃. As shown in fig. 2, the synthesized material possess spherical morphology and the average spherical particle sizes range between 10 and 15 μm. As shown in fig. 2, every spherical particle is composed of many small crystal particulates with a diameter about 1μm.

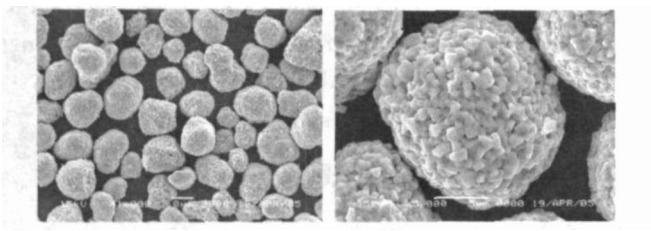

Fig. 2 SEM Photos of $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$

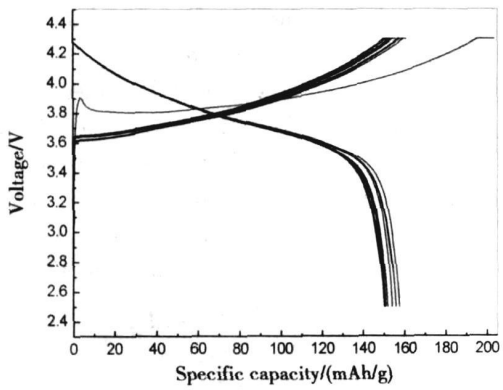

Fig. 3 Charge—discharge curves of $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$

2.3 Charge/discharge performance

Fig. 3 shows the first 10 cycles charge/discharge curves of $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$ synthesized at 750℃. As shown in fig. 3, the $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$ synthesized at 750℃ has excellent charge/discharge performance, whose discharge specific capacity is up to 157.7 mAh/g and remains above 150.4 mAh/g after 10 cycles.

3 Conclusions

A spherical cathode material $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$ was synthesized by melting—salts coating process in air using spherical $Ni(OH)_2$ as core material, $Al(NO_3)_3 \cdot 9H_2O$, $Co(NO_3)_2 \cdot 6H_2O$ and $LiNO_3$ as coating materials. The synthesized material $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$ by this process has the α-$NaFeO_2$ type ordered layered structure, spherical morphology and excellent electrochemical performance.

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曲杏壳制备炭膜支撑体

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摘　要：以杏壳为原料，通过碳化法制备了碳膜支撑体，利用泡压法表征了支撑体的孔径分布。对杏壳原料进行预碳化、粉碎、筛分得到碳粉，将碳粉和添加剂混匀挤出成型得到支撑体原膜，经干燥、碳化得到无缺陷碳膜支撑体。实验结果表明，粘结剂用量增加会导致支撑体平均孔径变小，纯水能量减小。随着原料颗粒的减小支撑体平均孔径减小，纯水能量下降。

关键词：碳膜支撑体；碳化；杏壳；孔径分布
中图分类号：TQ028.8

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融盐包裹法合成球形正极材料 $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$

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摘　要：以球形 $Ni(OH)_2$为核心原料，$Al(NO_3)_3\cdot 9H_2O$、$Co(NO_3)_2\cdot 6H_2O$和 $LiNO_3$为包裹原料，采用融盐包裹法在空气中煅烧合成了单相固溶体 $LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$。用 XRD研究了合成产物的物相和结构，用 SEM研究了合成产物的形貌，用电池性能测试仪研究了合成产物的电化学性能。实验结果表明，合成产物具有 $\alpha-NaFeO_2$型层状有序结构、球状形貌和良好的电化学性能。

关键词：锂离子电池；球形正极材料；$LiNi_{0.7}Co_{0.2}Al_{0.1}O_2$；融盐包裹法
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