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# 高容量正极材料 $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2-0.92\text{LiNiO}_2$ 的合成与电化学性能研究

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**摘要:** 在恒定 pH 值下将层状钴铝双羟基复合金属氧化物 ( $\text{CoAl-LDH}$ ) 均匀包覆在球状  $\text{Ni}(\text{OH})_2$  表面, 与  $\text{LiOH} \cdot \text{H}_2\text{O}$  混合均匀后, 经高温煅烧制得钴铝酸锂包覆镍酸锂  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2-0.92\text{LiNiO}_2$  正极材料. 电化学测试表明,  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2-0.92\text{LiNiO}_2$  正极比容量高, 具有良好的倍率性能和循环寿命, 其 0.1C 放电比容量为  $211 \text{ mAh} \cdot \text{g}^{-1}$ , 0.5C 放电比容量为  $195.6 \text{ mAh} \cdot \text{g}^{-1}$ , 3C 放电比容量为  $161 \text{ mAh} \cdot \text{g}^{-1}$ , 0.5C 30 周期循环后容量保持率为 93.2%, 明显优于  $\text{LiNiO}_2$  和钴酸锂包覆镍酸锂  $0.08\text{LiCoO}_2-0.92\text{LiNiO}_2$  正极.

**关键词:** 锂离子电池; 正极材料;  $\text{LiNiO}_2$ ;  $\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2$ ; 包覆

**中图分类号:** TM912.9

**文献标识码:** A

$\text{LiNiO}_2$  锂离子电池正极材料比容量高, 价格适宜, 有望替代  $\text{LiCoO}_2$  正极材料. 但镍离子易嵌入锂离子层发生锂、镍阳离子混排, 故  $\text{LiNiO}_2$  制备条件苛刻, 重现性差<sup>[1-3]</sup>, 且其易与电解液发生化学反应, 缩短  $\text{LiNiO}_2$  电极循环寿命<sup>[4]</sup>. 通过元素掺杂可提高  $\text{LiNiO}_2$  的结构稳定性, 钴、镍是处于同一周期的相邻元素,  $\text{LiNiO}_2$  和  $\text{LiCoO}_2$  有相同的  $\alpha\text{-NaFeO}_2$  结构, 因此钴常用作掺杂元素. 钴铝共掺杂能得到单相层状结构  $\text{LiNi}_{1-x-y}\text{Co}_x\text{Al}_y\text{O}_2$ <sup>[5-7]</sup>, 比  $\text{LiNi}_{1-y}\text{Co}_y\text{O}_2$ <sup>[8-10]</sup> 电极材料更稳定, 循环寿命更长, 因而备受关注. 然而元素掺杂易使  $\text{LiNiO}_2$  电极比容量较大幅度降低, 选用表面包覆 (如  $\text{ZrO}_2$ <sup>[11]</sup>、 $\text{MgO}$ <sup>[12]</sup> 等) 可减少  $\text{LiNiO}_2$  与电解液的直接接触, 提高电极循环寿命. 但  $\text{LiNiO}_2$  表面包覆通常不会改善  $\text{LiNiO}_2$  本体的比容量, 甚至还会导致比容量的降低. 因此包覆层的选择至关重要, 可考虑选用与  $\text{LiNiO}_2$  相同结构且更稳定的钴铝酸锂包覆材料.

本文将金属元素均匀分布的薄片状的钴铝双羟基复合金属氧化物 ( $\text{CoAl-LDH}$ )<sup>[13]</sup> 包覆于球状  $\text{Ni}(\text{OH})_2$  表面, 与锂源混匀经高温煅烧制得  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2-0.92\text{LiNiO}_2$  正极材料 (并合成了

$\text{LiNiO}_2$  和  $0.08\text{LiCoO}_2-0.92\text{LiNiO}_2$  正极材料作为对照), 以期在保持  $\text{LiNiO}_2$  高比容量特性的同时改善其倍率性能与循环寿命.

## 1 实验

### 1.1 材料制备

按  $\text{Co}:\text{Al}:\text{Ni}$  为 0.06:0.02:0.92 (by mole) 的比例称取  $\text{Ni}(\text{OH})_2$ 、 $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  和  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  反应物. 然后将球状  $\text{Ni}(\text{OH})_2$  在剧烈搅拌下分散于去离子水作为反应底液,  $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$  和  $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$  溶于去离子水中配制成混合盐溶液 (金属离子总浓度为  $0.5 \text{ mol} \cdot \text{L}^{-1}$ ). 向反应底液滴入  $1 \text{ mol} \cdot \text{L}^{-1}$   $\text{NaOH}$  溶液, 调节 pH 值为 10.5, 同时滴加混合盐溶液和  $1 \text{ mol} \cdot \text{L}^{-1}$   $\text{NaOH}$  溶液 (控制  $\text{pH} = 10.5 \pm 0.2$ ) 直至混合盐溶液滴加完毕 (1 h). 反应溶液继续剧烈搅拌 2 h, 抽滤, 去离子水洗涤至滤液  $\text{pH} = 7$ , 得滤饼 (反应产物). 将产物  $100^\circ\text{C}$  真空干燥后即得  $\text{CoAl-LDH}$  包覆  $\text{Ni}(\text{OH})_2$  前驱体.

以  $\text{LiOH} \cdot \text{H}_2\text{O}$  为锂源, 按  $\text{Li}:[\text{Ni} + \text{Co} + \text{Al}] = 1.05$  (by mole) 与  $\text{CoAl-LDH}$  包覆  $\text{Ni}(\text{OH})_2$  前驱体混匀,  $600^\circ\text{C}$  煅烧 20 h, 粉碎,  $700^\circ\text{C}$  再煅烧 20 h, 可得  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2-0.92\text{LiNiO}_2$ .

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0.08LiCoO<sub>2</sub>-0.92LiNiO<sub>2</sub> 合成方法同上,但不加 Al 源. 纯 LiNiO<sub>2</sub> 由球状 Ni(OH)<sub>2</sub> 与 LiOH·H<sub>2</sub>O 混合煅烧而成,煅烧方法同上.

1.2 物理表征

使用 X 射线衍射仪 (XRD, Shimadzu XRD-6000 型) 测试材料, Cu K<sub>α</sub> 辐射, 步宽 0.02°, 扫描速率 5°·min<sup>-1</sup>, 扫描范围 2θ = 10° ~ 70°. 使用扫描电子显微镜 (SEM, Zeiss Super 55 型) 观察材料形貌.

1.3 电池组装与测试

将材料、乙炔黑导电剂和聚偏氟乙烯粘结剂按 80:10:10 (by mass) 混合, 涂于铝箔集流体上, 80 °C 烘干后冲压成正极极片 (φ = 1 cm), 与金属锂片负极、Celgard 2400 隔膜和 EC + DMC + EMC + 1 mol·L<sup>-1</sup> LiPF<sub>6</sub> 电解液, 在惰性气体手套箱 (德国 M. braun 公司, Unilab 型, O<sub>2</sub> 和 H<sub>2</sub>O 含量均小于 1 × 10<sup>-6</sup>) 组装成 CR2032 型扣式电池. 采用电池测试系统 (武汉蓝电, CT 2001A 型) 测试电极电化学性能, 电压范围为 2.8 ~ 4.3 V. 使用电化学工作站 (上海辰华, CHI 660C 型) 测试电极交流阻抗谱, 激励电压 5 mV, 频率范围 100 kHz ~ 10 mHz.

2 结果与讨论

2.1 前驱体表征

图 1 为不同前驱体和 CoAl-LDH 的 XRD 谱图. 由图可见, 与球状 Ni(OH)<sub>2</sub> (a) 相比, Co(OH)<sub>2</sub> 包覆

球状 Ni(OH)<sub>2</sub> (b) 的峰位相同, 峰形变宽, 而 CoAl-LDH 包覆球状 Ni(OH)<sub>2</sub> (c) 的峰位和峰宽基本不变, 且呈现 CoAl-LDH 特征峰 (d).

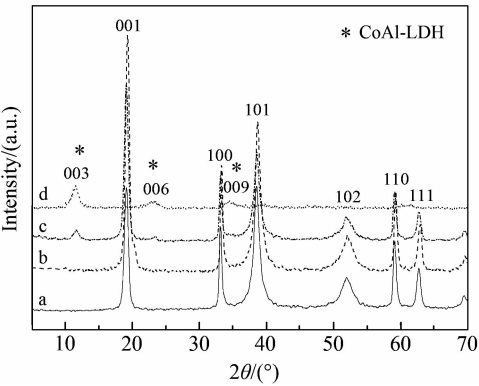


图 1 不同前驱体和 CoAl-LDH 的 XRD 谱图  
a. Ni(OH)<sub>2</sub>; b. 0.08Co(OH)<sub>2</sub>-0.92Ni(OH)<sub>2</sub>; c. 0.08CoAl-LDH-0.92Ni(OH)<sub>2</sub>; d. CoAl-LDH  
Fig. 1 XRD patterns of different precursors and CoAl-LDH  
a. Ni(OH)<sub>2</sub>; b. 0.08Co(OH)<sub>2</sub>-0.92Ni(OH)<sub>2</sub>; c. 0.08CoAl-LDH-0.92Ni(OH)<sub>2</sub>; d. CoAl-LDH

图 2 为不同前驱体的 SEM (低倍率: × 3000; 高倍率: × 100 000) 照片. 从图看出, 与球状 Ni(OH)<sub>2</sub> 照片 (A, A') 相比, Co(OH)<sub>2</sub> 包覆层 (B, B') 材料表面不甚光滑, 颗粒较大, 呈不规则片状, 片层较厚; CoAl-LDH 包覆层 (C, C') 很薄, 呈片状

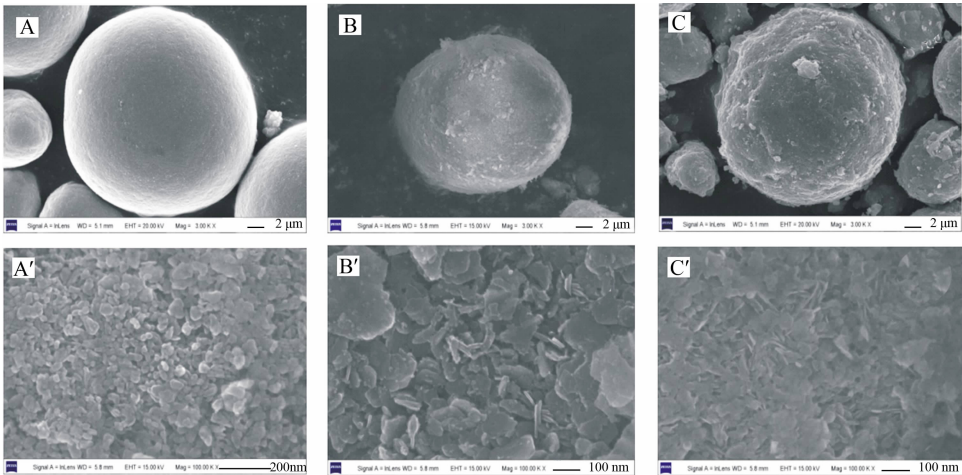


图 2 不同前驱体的 SEM (低倍率: × 3000; 高倍率: × 100000) 照片  
A, A'. Ni(OH)<sub>2</sub>; B, B'. 0.08Co(OH)<sub>2</sub>-0.92Ni(OH)<sub>2</sub>; C, C'. 0.08CoAl-LDH-0.92Ni(OH)<sub>2</sub>  
Fig. 2 SEM images of different precursors (magnifications: low × 3000 and high × 100000)  
A, A'. Ni(OH)<sub>2</sub>; B, B'. 0.08Co(OH)<sub>2</sub>-0.92Ni(OH)<sub>2</sub>; C, C'. 0.08CoAl-LDH-0.92Ni(OH)<sub>2</sub>

密堆积. 由此可见,  $\text{CoAl-LDH}$  能均匀地包覆于球状  $\text{Ni}(\text{OH})_2$  表面, 形成致密的包覆层.

## 2.2 正极材料表征

图 3 为不同正极材料的 XRD 图谱. 从图 3 看出, 由  $\text{Co}(\text{OH})_2$  表面包覆的球状  $\text{Ni}(\text{OH})_2$  前驱体制备的  $0.08\text{LiCoO}_2\text{-}0.92\text{LiNiO}_2$  和  $\text{CoAl-LDH}$  表面包覆的球状  $\text{Ni}(\text{OH})_2$  前驱体制备的  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$  均保持了  $\text{LiNiO}_2$  的层状结构.

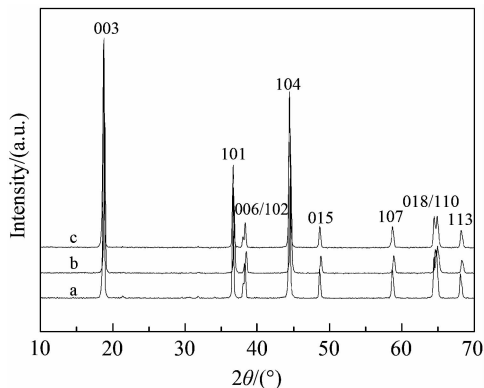


图 3 不同正极材料的 XRD 谱图

a.  $\text{LiNiO}_2$ ; b.  $0.08\text{LiCoO}_2\text{-}0.92\text{LiNiO}_2$ ;  
c.  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$

Fig. 3 XRD patterns of different cathode materials

a.  $\text{LiNiO}_2$ ; b.  $0.08\text{LiCoO}_2\text{-}0.92\text{LiNiO}_2$ ;  
c.  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$

图 4 为不同正极材料的 SEM 照片. 由照片可见,  $0.08\text{LiCoO}_2\text{-}0.92\text{LiNiO}_2$  (图 4B) 颗粒与  $\text{LiNiO}_2$  (图 4A) 类似, 呈较大不规整形, 包覆层较疏松, 而  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$  (图 4C) 的表面包

覆层呈花状薄片, 均匀致密.

## 2.3 电化学性能测试

图 5 给出  $\text{LiNiO}_2$ 、 $0.08\text{LiCoO}_2\text{-}0.92\text{LiNiO}_2$  和  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$  三种正极  $0.5\text{C}$  倍率的循环寿命曲线. 由图可见, 30 周期循环  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$  正极放电比容量为  $183\text{mAh}\cdot\text{g}^{-1}(\text{c})$ , 而  $0.08\text{LiCoO}_2\text{-}0.92\text{LiNiO}_2$  和  $\text{LiNiO}_2$  正极放电比容量只有  $144\text{mAh}\cdot\text{g}^{-1}(\text{b})$  和  $133\text{mAh}\cdot\text{g}^{-1}(\text{a})$ , 表明  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$  正极有最好的循环寿命. 这归因于包覆前驱体 ( $\text{CoAl-LDH}$ ) 片层薄、粒径小, 煅烧形成的  $\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2$  包覆层更致密, 可更好地隔绝  $\text{LiNiO}_2$  与电解液接触, 减少二者之间的副反应.

$\text{LiNiO}_2$ 、 $0.08\text{LiCoO}_2\text{-}0.92\text{LiNiO}_2$  和  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$  正极在不同倍率的放电循环寿命如图 6 所示, 电压范围  $2.8 \sim 4.3\text{V}$ .  $0.1\text{C}$  倍率三种正极的放电比容量相差不大,  $\text{LiNiO}_2$  和  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$  正极均可达到  $211\text{mAh}\cdot\text{g}^{-1}$ ,  $0.08\text{LiCoO}_2\text{-}0.92\text{LiNiO}_2$  正极为  $204\text{mAh}\cdot\text{g}^{-1}$ . 倍率增大,  $\text{LiNiO}_2$  电极放电比容量大幅度降低, 而  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$  电极放电比容量下降最少.  $3\text{C}$  倍率  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$  电极放电比容量为  $161\text{mAh}\cdot\text{g}^{-1}$ ,  $0.08\text{LiCoO}_2\text{-}0.92\text{LiNiO}_2$  电极放电比容量为  $122\text{mAh}\cdot\text{g}^{-1}$ , 而  $\text{LiNiO}_2$  电极放电比容量仅有  $50\text{mAh}\cdot\text{g}^{-1}$ , 这说明  $\text{LiCoO}_2$  包覆  $\text{LiNiO}_2$  电极可较大改善倍率性能, 而  $\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2$  包覆  $\text{LiNiO}_2$  电极倍率性能更优异.

图 7 为三种正极首次充电至  $4.3\text{V}$  的电极交流阻抗谱图. 可见  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$

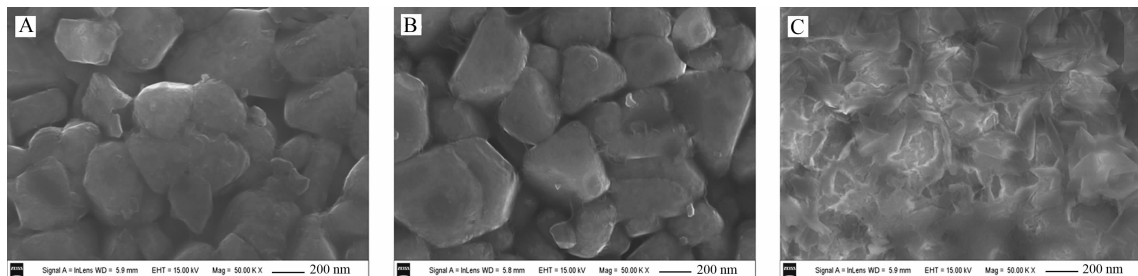


图 4 不同正极材料的 SEM 照片

A.  $\text{LiNiO}_2$ ; B.  $0.08\text{LiCoO}_2\text{-}0.92\text{LiNiO}_2$ ; C.  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$

Fig. 4 SEM images of different cathode materials

A.  $\text{LiNiO}_2$ ; B.  $0.08\text{LiCoO}_2\text{-}0.92\text{LiNiO}_2$ ; C.  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2\text{-}0.92\text{LiNiO}_2$

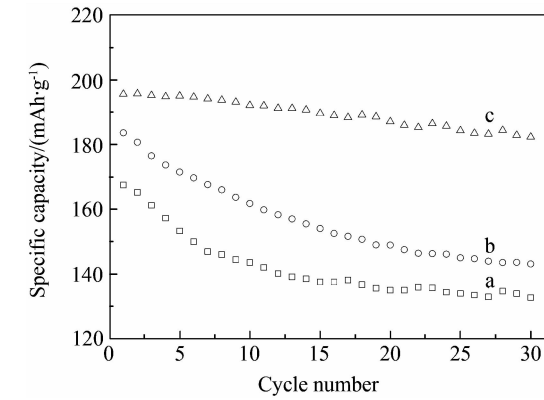


图 5 三种正极的循环寿命曲线  
a. LiNiO<sub>2</sub>; b. 0.08LiCoO<sub>2</sub>-0.92LiNiO<sub>2</sub>;  
c. 0.08LiCo<sub>0.75</sub>Al<sub>0.25</sub>O<sub>2</sub>-0.92LiNiO<sub>2</sub>

Fig. 5 Cycling performance of different cathodes  
a. LiNiO<sub>2</sub>; b. 0.08LiCoO<sub>2</sub>-0.92LiNiO<sub>2</sub>;  
c. 0.08LiCo<sub>0.75</sub>Al<sub>0.25</sub>O<sub>2</sub>-0.92LiNiO<sub>2</sub>

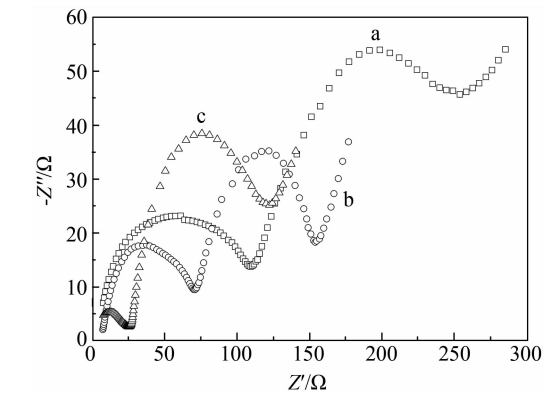


图 7 三种正极首次充电态(4.3 V)电极交流阻抗谱图  
a. LiNiO<sub>2</sub>; b. 0.08LiCoO<sub>2</sub>-0.92LiNiO<sub>2</sub>;  
c. 0.08LiCo<sub>0.75</sub>Al<sub>0.25</sub>O<sub>2</sub>-0.92LiNiO<sub>2</sub>

Fig. 7 Nyquist plots of different cathodes charged to 4.3 V at 1st cycle  
a. LiNiO<sub>2</sub>; b. 0.08LiCoO<sub>2</sub>-0.92LiNiO<sub>2</sub>;  
c. 0.08LiCo<sub>0.75</sub>Al<sub>0.25</sub>O<sub>2</sub>-0.92LiNiO<sub>2</sub>

电极界面阻抗最小,其电荷传递阻抗与 0.08LiCoO<sub>2</sub>-0.92LiNiO<sub>2</sub>的相近,而 LiNiO<sub>2</sub> 电极的界面阻抗和电荷传递阻抗均很大,即前者倍率性能最优。

3 结 论

由 CoAl-LDH 包覆球状 Ni(OH)<sub>2</sub> 前驱体制备钴铝酸锂包覆镍酸锂 0.08LiCo<sub>0.75</sub>Al<sub>0.25</sub>O<sub>2</sub>-0.92LiNiO<sub>2</sub> 正极材料,并组装成 CR2032 扣式电池。

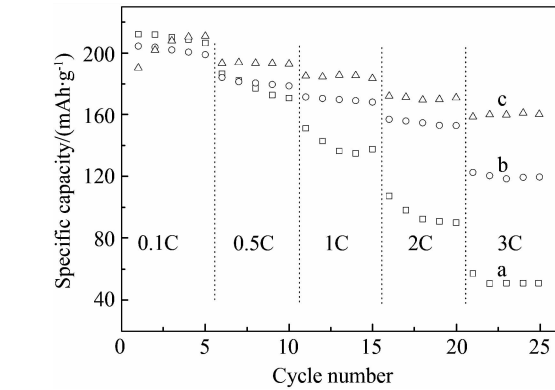


图 6 三种正极倍率寿命曲线  
a. LiNiO<sub>2</sub>; b. 0.08LiCoO<sub>2</sub>-0.92LiNiO<sub>2</sub>;  
c. 0.08LiCo<sub>0.75</sub>Al<sub>0.25</sub>O<sub>2</sub>-0.92LiNiO<sub>2</sub>

Fig. 6 Rate performances of different cathodes  
a. LiNiO<sub>2</sub>; b. 0.08LiCoO<sub>2</sub>-0.92LiNiO<sub>2</sub>;  
c. 0.08LiCo<sub>0.75</sub>Al<sub>0.25</sub>O<sub>2</sub>-0.92LiNiO<sub>2</sub>

0.1C、0.5C 和 3C 倍率该电极放电比容量分别为 211 mAh·g<sup>-1</sup>、195.6 mAh·g<sup>-1</sup> 和 161 mAh·g<sup>-1</sup>, 0.5C倍率 30 周期循环其容量保持率为 93.2%,电极倍率性能和循环寿命最优。

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## Synthesis and Electrochemical Properties of High-Capacity Cathode Material $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2$ - $0.92\text{LiNiO}_2$

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**Abstract:**  $\text{CoAl-LDH}$  or  $\text{Co}(\text{OH})_2$  coated spherical  $\text{Ni}(\text{OH})_2$  precursors were obtained via a coprecipitation method at a constant pH. The mixtures of the precursors and  $\text{LiOH}\cdot\text{H}_2\text{O}$  were annealed at high temperature in  $\text{O}_2$  atmosphere, and then the cathode materials of  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2$ - $0.92\text{LiNiO}_2$ ,  $0.08\text{LiCoO}_2$ - $0.92\text{LiNiO}_2$  and  $\text{LiNiO}_2$  were synthesized. Effects of the coating layer were also studied. The results showed that the  $0.08\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2$ - $0.92\text{LiNiO}_2$  material possessed the best rate and cycle life performance. The discharging capacities at 0.1C, 0.5C and 3C were  $211\text{ mAh}\cdot\text{g}^{-1}$ ,  $195.6\text{ mAh}\cdot\text{g}^{-1}$  and  $161\text{ mAh}\cdot\text{g}^{-1}$  respectively, and the capacity retention ratio after 30 cycles at 0.5C was 93.2%. These performances were much better than those of both pure  $\text{LiNiO}_2$  and  $0.08\text{LiCoO}_2$ - $0.92\text{LiNiO}_2$ .

**Key words:** Li-ion battery; cathode material;  $\text{LiNiO}_2$ ;  $\text{LiCo}_{0.75}\text{Al}_{0.25}\text{O}_2$ ; coating