



Synthesis and electrochemical properties of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ cathode materials

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Abstract: A Co–Mg co-substituted $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ cathode material was prepared by a co-precipitation method. The prepared $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ exhibits excellent electrochemical properties, such as initial discharge capacities of 202.6 mA·h/g and 190.5 mA·h/g at 0.2C and 1C rate, respectively, in operating voltage range of 3.0–4.3 V (versus Li^+/Li). The capacity retentions are 96.1% and 93.4% at 0.2C and 1C, respectively, after 50 cycles. Moreover, the cycle performance of the sample was investigated in a 053048-type square Li ion battery. This type of battery can keep 81.7% of initial capacity after 500 charge–discharge cycles at 1C rate, which is close to that of commercial LiCoO_2 battery. Therefore, the as-prepared material is capable of such high energy applications as portable product power.

Key words: lithium-ion batteries; cathode material; co-precipitation method; cobalt–magnesium co-substitution; electrochemical properties

1 Introduction

LiNiO_2 is considered as a very promising cathode material for lithium-ion batteries due to its low cost and high specific capacity [1,2]. However, the material still exhibits several shortcomings, such as difficult synthetic technique, low thermal stability, and poor cycle life at the charged state [3,4]. It has been known that the cationic substitution of LiNiO_2 is one of the most important methods to improve the electrochemical reactivity. Among the possible element substitution or modification strategies, Co-substitution for Ni has been accepted as an effective path for the suppression of cation mixing (that is, some Ni ions in Ni layer move to Li layer) and the enhancement of its structural stability due to the formation of a perfect solid solution [5–7]. It has also been reported that a small amount of Al or Mg substitution for Ni in LiNiO_2 could also improve its

electrochemical properties, thermal stability, etc [8–10]. OHZUKU et al [11] reported that $\text{LiNi}_{1-x}\text{Al}_x\text{O}_2$, with partial Ni^{2+} ions substituted by Al^{3+} ions, could also show not only excellent structure stability, but also thermal stability ascribed to the suppressed phase transitions during charge–discharge cycling at high states of charge (SOC). Moreover, based on the similar ion size of Mg^{2+} and Li^+ , DELMAS et al [8] proposed that the substitution of Mg^{2+} ions in the Li layer of LiNiO_2 can also prevent local collapse of the structure effectively and enhance electrochemical performance of the $\text{LiNi}_{1-y}\text{Mg}_y\text{O}_2$ system during cycling. CHO [10] also investigated the effect of Mg^{2+} substitution in $\text{LiNi}_{0.74}\text{Co}_{0.26-x}\text{Mg}_x\text{O}_2$ and indicated that the improvement of electrochemical properties of $\text{LiNi}_{0.74}\text{Co}_{0.26-x}\text{Mg}_x\text{O}_2$ should be ascribed to the suppression of cation mixing to some extent. Thermal stability was also enhanced because of the enhanced structural stability with Mg^{2+} substitution. The most widely used Ni-rich material of $\text{LiNi}_{1-x}\text{M}_x\text{O}_2$ is Co and

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Al co-substituted $\text{LiNi}_{0.8}\text{Co}_{0.15}\text{Al}_{0.05}\text{O}_2$ due to its good thermal stability and cyclability [12,13]. High nickel cathode of $\text{LiNi}_{0.9}\text{Co}_{0.1}\text{O}_2$ synthesized by some researchers [14–16] showed high capacity. However, capacity fading was inevitable during cycling. From the above discussion, it is expected that a partial substitution of Co and/or Mg would have a positive effect to improve the battery performance. In this study, the synthesis and electrochemical properties of Co and Mg co-substituted for $\text{LiNi}_{0.87}\text{Co}_{0.1}\text{Mg}_{0.03}\text{O}_2$ cathode material were reported.

2 Experimental

$\text{LiNi}_{0.87}\text{Co}_{0.1}\text{Mn}_{0.03}\text{O}_2$ was prepared via the co-precipitation method. Metal sulphate aqueous solutions were mixed with molar ratio of Ni^{2+} to Co^{2+} to Mg^{2+} being 87:10:3 in a stirred tank reactor (CSTR, capacity 10 L). The total concentration of all the metal ions was 2 mol/L. At the same time, a NaOH solution (2 mol/L) and a desired amount of $\text{NH}_3\cdot\text{H}_2\text{O}$ solution (2 mol/L) as a chelating agent were also separately fed into the same reactor. The concentration of $\text{NH}_3\cdot\text{H}_2\text{O}$, feed rate, temperature and pH of the mixture in the reactor were controlled precisely. The green precipitate was filtered, washed, and dried in a vacuum oven. The morphology and chemical composition of the precipitate were revealed by scanning electron microscope/energy dispersive spectrometer (SEM/EDS, JSM–6010LV, JEOL Ltd.) to be spherical $\text{Ni}_{0.87}\text{Co}_{0.1}\text{Mg}_{0.03}(\text{OH})_2$. The precursor $\text{Ni}_{0.87}\text{Co}_{0.1}\text{Mg}_{0.03}(\text{OH})_2$ and $\text{LiOH}\cdot\text{H}_2\text{O}$ were mixed adequately with molar ratio of Li to Ni+Co+Mg being 1.05 and then fired in tube furnace at 750 °C for 12 h under oxygen atmosphere to form the final product $\text{LiNi}_{0.87}\text{Co}_{0.1}\text{Mg}_{0.03}\text{O}_2$.

The prepared material was characterized by X-ray diffraction (XRD) (Rigaku P/max 2200VPC using a Cu K_α radiation with $\lambda=1.5406$ Å). XRD data were obtained at diffraction angle ranges (2θ) from 10° to 110°, with a step size of 0.02° and a constant counting time of 10 s. The XRD patterns were also refined for their structural information by refinement software GSAS [17]. Chemical element analysis of the precursor and product was conducted by inductively coupled plasma-atomic emission spectroscopy (ICP-AES, ICPS–8100). The particle size and morphology of the samples were studied by SEM/EDS (JSM–6010LV, JEOL Ltd).

The composition of the composite electrode was $\text{LiNi}_{0.87}\text{Co}_{0.1}\text{Mg}_{0.03}\text{O}_2$, carbon black and polyvinylidene fluoride (PVDF) binder with a mass ratio of 80:10:10. The electrode was dried at 120 °C for 12 h in a vacuum oven before assembly of coin-type half cells (2025): $\text{LiNi}_{0.87}\text{Co}_{0.1}\text{Mg}_{0.03}\text{O}_2/\text{Celgard separator}/\text{Li}$, in an argon-filled glove box. The electrolyte was 1 mol/L LiPF_6 in a

mixture of ethylene carbonate (EC) and dimethyl carbonate (DMC) (1:1 in volume). All of these reagents were analytical reagents or better. The galvanostatic charge–discharge performance of the coin-type cells was conducted on an electrochemical test system CT2001A (Wuhan Land Electronic Co. Ltd., China) in operating voltage range of 3.0–4.3 V (versus Li^+/Li) at room temperature (25 °C). The charge–discharge rate of 1C was equal to 190 mA/g for coin-type half cells. The electrochemical performance of $\text{LiNi}_{0.87}\text{Co}_{0.1}\text{Mg}_{0.03}\text{O}_2$ was also investigated in a full cell (053048-type square Li-ion cell): $\text{LiNi}_{0.87}\text{Co}_{0.1}\text{Mg}_{0.03}\text{O}_2/\text{Celgard separator}/\text{standard graphite}$. The charge–discharge of the square cell was conducted at 600 mA (1C). Cyclic voltammogram (CV) measurements of the half cell were carried out at a Solartron 1287 electrochemical interface over a voltage range from 3.2 to 4.5 V at a scan rate of 0.2 mV/s.

3 Results and discussion

3.1 $\text{Li}^+/\text{Ni}^{2+}$ cations displacement

The chemical composition of the synthesized samples, obtained from ICP-AES, is listed in Table 1. Compared with the formula of the target samples, the results indicate that the composition of the synthesized samples is actually very close to that of the target products. The results prove that the desired products have been successfully synthesized by the co-precipitation method followed by high-temperature firing. Therefore, the compositions are used to describe the materials in the following sections.

Table 1 Designated and analyzed chemical formulas of samples measured by ICP-AES

Designated chemical formula	Analyzed chemical formula
$\text{LiNi}_{0.87}\text{Co}_{0.1}\text{Mg}_{0.03}\text{O}_2$	$\text{Li}_{0.996}\text{Ni}_{0.867}\text{Co}_{0.105}\text{Mg}_{0.031}\text{O}_2$

In the XRD patterns, all the diffraction peaks can be indexed to space group $R\bar{3}m$. No other diffraction peaks are detected in the patterns, meaning that the synthesized sample is single phase with homogeneous element distribution. As shown in Fig. 1(a), the splitting of the hexagonal characteristic doublets 006/102 and 108/110 is evidence for layered structure, indicating a good ordering of the hexagonal lattice. The integrated intensity ratio of (003) to (104) peaks (I_{003}/I_{104}) has been considered as one of the indicators of the degree of cation displacement [18,19]. Typically, for the hexagonal layered oxide cathodes, the reversible capacity has been reported to decrease when the I_{003}/I_{104} value is less than 1.2 [20,21]. Note that the integrated intensity ratio

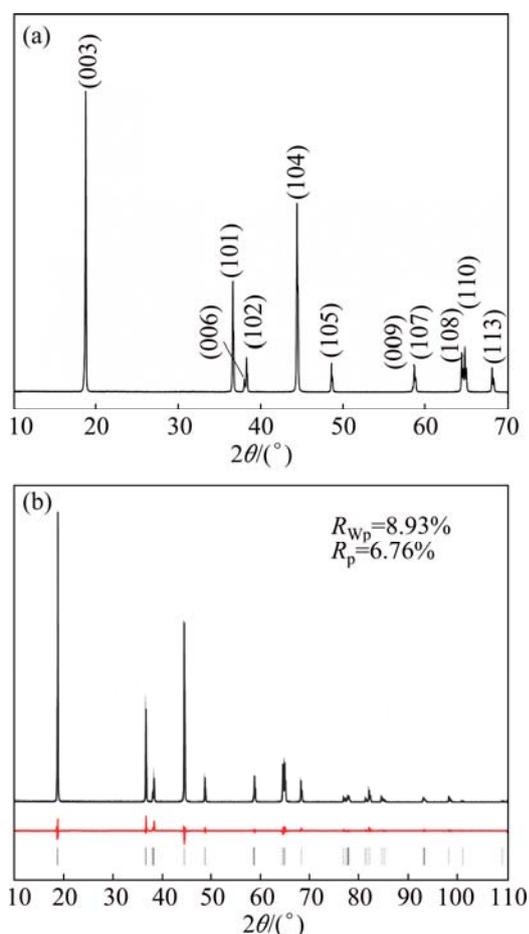


Fig. 1 XRD patterns of synthesized $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ (a) and its Rietveld refinement (b) (R_{wp} —Weighted R factor; R_{p} —Profile R factor)

I_{003}/I_{104} of the samples is around 1.6. The fact indicates that the crystal structure of the material keeps a good hexagonal ordering and good electrochemical performance will be expected. In order to determine the occupancy sites of Ni^{2+} and Mg^{2+} in the unit cell, the structural refinements (Rietveld refinements) have been performed with GSAS program. Ni^{2+} and Mg^{2+} , located at $3a$ site, could be exchanged with Li^+ , located at $3b$ site, due to the similar ionic radius (Li^+ , 0.76 Å; Ni^{2+} , 0.69 Å; Mg^{2+} , 0.72 Å). Meanwhile, the possibility of Co incorporation into Li layer is excluded from the refinement due to the fact that the ionic radius of Co^{2+} (0.545 Å) is much smaller than that of Li^+ . The occupancy sites of Ni^{2+} , Mg^{2+} and Li^+ ions in the unit cell of sample are listed in Table 2. As shown in Table 2, most of Ni^{2+} ions are determined to be located at transition metal sites, with about 4.3% of Ni^{2+} ions located at Li^+ site. Meanwhile, about 0.5% of Mg^{2+} ions are detected to be located at interlab space. Therefore, the formula of sample can be written as $(\text{Li}_{0.952}\text{Ni}_{0.043}\text{Mg}_{0.005})_{3b}(\text{Ni}_{0.827}\text{Co}_{0.10}\text{Mg}_{0.025})_{3a}\text{O}_2$, which is almost same as the formula of target one. According to the previous

reports [22,23], a small amount of Mg^{2+} ions in the interlab can play an important role in enhancing extra structural stability without blocking the reversible extraction/insertion of Li^+ ions. Thus, it is concluded that 10% Co and 3% Mg co-substitution into the unit cell can prevent the $\text{Li}^+/\text{Ni}^{2+}$ cations displacement effectively.

Table 2 Rietveld refinement analysis results of synthesized $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$

Li 3a	Li 3b	Ni 3a	Ni 3b	Co 3a	Co 3b	Mg 3a	Mg 3b
0	0.952	0.827	0.043	0.10	0	0.025	0.005

(3a—Transition metal site; 3b—Li site)

3.2 Electrochemical performance of battery

The SEM images in Fig. 2 indicate that the particle shape of both the hydroxide precursor and the lithiated product is spherical with the size ranging from 5 to 15 μm . Note that the spherical morphology and particle size are maintained even after the high-temperature sintering. The lithiated product has the large tap density of 2.3 g/cm^3 , which is very beneficial to the enhancement of battery energy density.

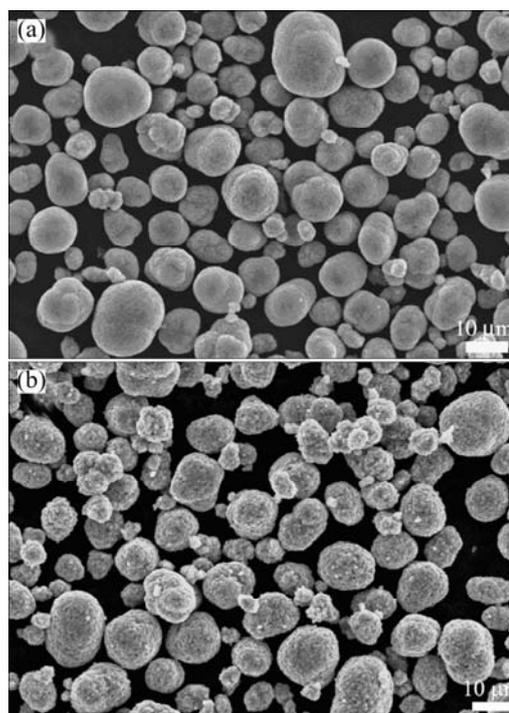


Fig. 2 SEM images of $\text{Ni}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}(\text{OH})_2$ (a) and $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ (b)

As shown in Fig. 3(a), $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ exhibits the initial discharge capacities of 202.6 $\text{mA}\cdot\text{h}/\text{g}$ and 190.5 $\text{mA}\cdot\text{h}/\text{g}$ at 0.2C and 1C, respectively, in a coin-type half cell. For the secondary battery, cycling stability is an important and desirable feature for practical applications. The specific discharge capacity

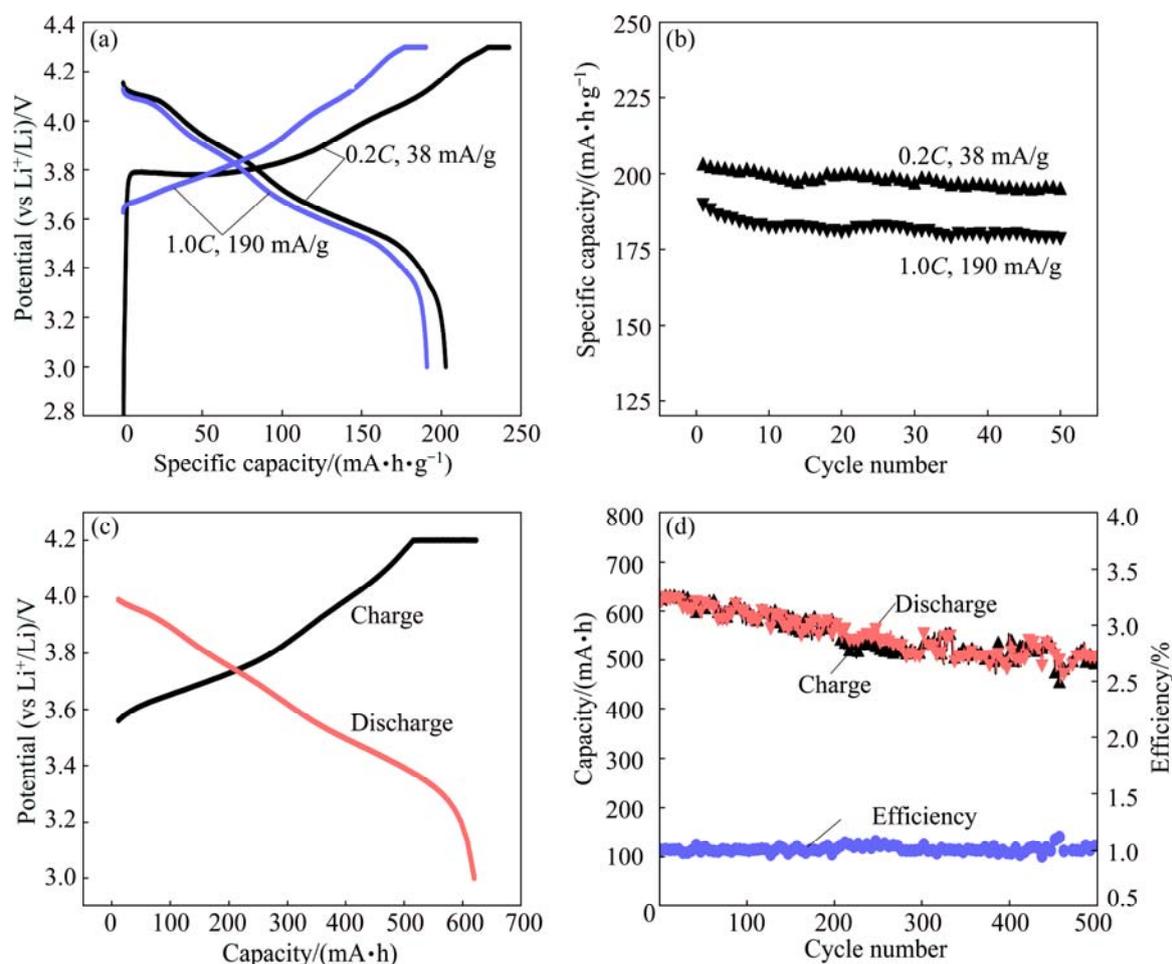


Fig. 3 Initial charge–discharge curves of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ sample (vs Li^+/Li) (a), cycling capability of as-prepared sample (b), the 2nd charge–discharge curves of sample (vs Li_6C) in 5S3048 square battery (c) and cycling performance of 5S3048 square batteries with $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ samples as cathode (d)

with cycle number of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ is presented in Fig. 3(b). Capacity retentions of 96.1% and 93.4% after 50 cycles at 0.2C and 1C, respectively, can be achieved in the coin-type cells, indicating good cycle performance. And it can be clearly seen from Table 3 that the $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ shows better electrochemical performance in contrast to some other reports. Furthermore, a 5S3048-type square Li-ion cell (see Fig. 4) was fabricated to study the electrochemical performance of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ in a full cell. The 2nd charge–discharge curves of the full cell are shown in Fig. 3(c). The cell exhibits a median discharge voltage of 3.5 V and a discharge capacity of around 600 mA·h. The cycling stability of the cell at 1C rate is given in Fig. 3(d). It can be seen that the 5S3048-type square cell shows a good cycle performance with 81.7% of its initial capacity retention after 500 cycles at 1C, which is really close to that of commercial LiCoO_2 cell.

CV plots have been recorded for $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ using coin-type half cell to reveal the phase transitions during the Li-deintercalation/

Table 3 Electrochemical properties of $\text{LiNi}_{0.9}\text{Co}_{0.1}\text{O}_2$ and $\text{LiNi}_{0.87}\text{Co}_{0.1}\text{Mg}_{0.03}\text{O}_2$ cathode material

Testing condition	Initial discharge capacity/(mA·h·g ⁻¹)	Capacity retention rate /%	Reference
50 mA/g, 3.0–4.3 V	195.7	91.1(20 cycles)	[14]
25 mA/g, 3.0–4.3 V	193.0	88.6(15 cycles)	[15]
25 mA/g, 3.0–4.35 V	199.0	94.5(5 cycles)	[16]
25 mA/g, 3.0–4.35 V	202.6	96.1(50 cycles)	–

intercalation process as seen in Fig. 5. The plots were collected at a scan rate of 0.2 mV/s for the 1st, 2nd and 10th cycles. Two oxidation peaks appear at 3.91 V (P1) and 4.14 V (P2) while their corresponding reduction peaks are located at 3.63 V (P3) and 3.91 V (P4) in the 1st cycle, which have some difference from the results reported in the similar Li–Ni–O compounds [24,25].



Fig. 4 Prototype of 053048 square Li ion battery

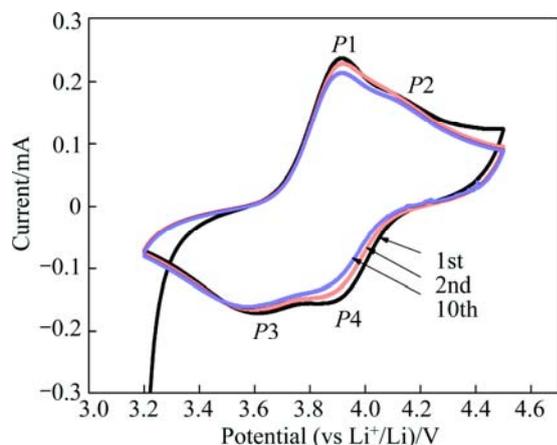


Fig. 5 Cyclic voltammograms of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ at scan rate of 0.2 mV/s

In Fig. 5, a broad oxidation peak appears in the potential range of 3.7–4.3 V and a broad reduction peak appears in the potential range of 3.2–4.0 V, indicating the phase transitions occurring during Li-deintercalation/intercalation, respectively [21]. These broad peaks remain almost unchanged during the 2nd–10th cycles, indicating good reversibility of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$.

As a result, Co^{2+} and Mg^{2+} co-substitution can greatly compress the irreversible phase transitions and improve the reversibility of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$. These results confirm that the high discharge capacity and good cycling stability of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ are directly related to the ordered layered structure. Apparently, Co^{2+} and Mg^{2+} co-substitution can improve performances of battery, such as high discharge capacity, high discharge-charge capacity and good cycling performance. Hence, Co^{2+} and Mg^{2+} co-substitution is a promising strategy to improve the electrochemical performance of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$.

4 Conclusions

1) Spherical Co^{2+} and Mg^{2+} co-substituted $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ material was successfully

synthesized via co-precipitation method and the subsequent high-temperature sintering. The results of XRD indicate that all the elements are incorporated into $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ structure. Rietveld refinement shows that 10% Co^{2+} and 3% Mg^{2+} substitution can suppress the $\text{Li}^+/\text{Ni}^{2+}$ cation displacement effectively.

2) The results of long-term cycling confirm that $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ exhibits excellent high cyclic performance in coin-type half cell, yielding 202.6 mA·h/g and 190.5 mA·h/g at 0.2C and 1C rate, respectively, and keep 81.7% of its initial discharge capacity after 500 cycles at 1C rate in 053048-type square full cell. Such performance of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ sample is similar to that of LiCoO_2 in commercial battery.

3) The results of CV prove that the Co^{2+} and Mg^{2+} co-substitution can greatly compress the degree of irreversible phase transitions and improve the reversibility of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$. It is concluded that the reduced cation mixing and improved structure stability by Co^{2+} and Mg^{2+} co-substitution contribute to the enhanced electrochemical performance of $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$.

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$\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ 正极材料的合成和电化学性能

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摘要: 采用共沉淀法制备钴镁共掺的 $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ 正极材料。结果表明, 所制备的 $\text{LiNi}_{0.87}\text{Co}_{0.10}\text{Mg}_{0.03}\text{O}_2$ 正极材料具备良好的电化学性能, 在 3.0~4.3 V (Li^+/Li) 的条件下, 该电极 0.2C 和 1C 的首次放电容量分别为 202.6 mA·h/g 和 190.5 mA·h/g; 经过 50 次循环后, 该电极 0.2C 和 1C 的容量保持率分别为 96.1% 和 93.4%。而且, 将该材料制作成型号为 053048 的全电池, 在 1C 充放 500 次后, 电池的容量保持率为 81.7%, 性能接近工业用钴酸锂电池的水平。因此, 采用该方法制备的正极材料能够用于高能量密度的移动数码产品。

关键词: 锂离子电池; 正极材料; 共沉淀法; 钴镁共掺; 电化学性能

(Edited by Xiang-qun LI)