Nanosized octahedral LiNi0.5Mn1.5O4 with predominant (111) facet as high performance cathode for Lithium-ion batteries

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Abstract

Nanosized octahedral LiNi$_{0.5}$Mn$_{1.5}$O$_4$ with predominant (111) facet has been successfully fabricated using Mn$_3$O$_4$ nanoparticles precursors via a two-step synthesis, which involves a hydrothermal treatment and the subsequent calcination. The physical properties of the Mn$_3$O$_4$ precursor and the resultant LiNi$_{0.5}$Mn$_{1.5}$O$_4$ were characterized by XRD (X-ray diffraction), TEM (transmission electron microscopy) and SEM (scanning electron microscopy). The charge-discharge tests show that the resultant LiNi$_{0.5}$Mn$_{1.5}$O$_4$ exhibits excellent cyclability and rate capability, which delivers a discharge capacity of about 117 mAh g$^{-1}$ after 300 cycles, and maintains 94% of its initial discharge capacity (124.7 mAh g$^{-1}$) at 1C, even at a rate of 40C, a specific capacity of 99.2 mAh g$^{-1}$ could be still obtained for the O-LNMO. The superior electrochemical performance of the LNMO is mainly attributed to the synergistic effect of the nanosized octahedral structure and exposed (111) facets of the prepared LiNi$_{0.5}$Mn$_{1.5}$O$_4$. We found that the nanosized octahedral structure can not only accommodate the lattice stress caused by John-Teller distortion but also provide short paths for Li$^+$ ion transportation in the material. Additionally, the obtained predominant (111) facet is helpful to the formation of protective SEI film on the spinel LiNi$_{0.5}$Mn$_{1.5}$O$_4$ electrode.

1. Introduction

Li-ion batteries (LIBs) have achieved great success in portable electronics and are widely studied for electric vehicles (EVs) and hybrid electric vehicles (HEVs) due to its advantages such as high energy density, long lifespan, environment benignity and no memory effect [1–3]. In order to meet the applications in EVs and HEVs, further improvement of energy density for LIBs is required [4, 5]. An effective approach for improving the energy density of LIBs is to explore the cathode materials with higher operating potential and large specific capacity [6, 7].

Among the diverse materials that have been studied as potential cathode materials for LIBs, LiNi$_{0.5}$Mn$_{1.5}$O$_4$ (LNMO) has received attention as a promising candidate for high energy density LIBs because of its high operating voltage (4.75 vs. Li/Li$^+$), and high specific capacity (146.7 mAh g$^{-1}$) as well [8, 9]. Despite these outstanding features, the practical application of LNMO in LIBs is still restricted by two problems. First, presence of Mn$^{3+}$ formed during charge/discharge processes leads to the John-Teller distortion causing severe structural instability [10, 11]. Second, the dissolution of Mn resulting from the spinel lattice, especially at elevated temperature [12, 13]. Strategies including cations doping and/or surface coating have been proposed to address the problems as mentioned above [14–16], but these method always accompanied by compromising the theoretical capacity and complicated synthetic process [17, 18].

Recently, particle shapes and sizes of electrode materials are known to play a critical role in determining their electrochemical properties [19, 20]. LNMO with nano-structures shows enhanced electrochemical properties in terms of cyclic stability and rate capability [21, 22]. Nanostructure materials can increase the
contact between electrode and electrolyte and reduce the path length for the \( \text{Li}^+ \) diffusion, resulting in good rate capability. Additionally, nano-structure can buffer the lattice stress caused by John-Teller effect upon cycling and thus improve the cycling stability of LNMO material [23, 24].

It was reported that the crystallographic facets of the LNMO material can affect its electrochemical performance [25]. For example, the (111) facet in the spinel LNMO shows better cycling stability than other facets such as (100) and (110). Previous study reveals that the SEI layer formed on the (111) facet is thinner and smoother compared to the (100) and (110) facets [26]. What is more, the (111) facet can promote the \( \text{Li}^+ \) intercalation/deintercalation in the LNMO, resulting in the enhanced rate capability of the LNMO [27]. Therefore, the nano-structure of LNMO with exposed (111) facet could be considered as a promising method to improve the cycling stability and rate capability of the LNMO material.

In this study, we proposed the preparation of nanosized octahedral \( \text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4 \) (O-LNMO) with predominant (111) via a two-step synthesis, which is schematically shown in Fig. 1: we first synthesized the \( \text{Mn}_3\text{O}_4 \) nanoparticles precursor by a hydrothermal treatment, and then impregnated the obtained \( \text{Mn}_3\text{O}_4 \) precursor with an ethanol solution of Li and Ni precursors, followed by solvent evaporation and a high temperature solid state reaction. The crystal structure and morphology of the synthesized O-LNMO were characterized by XRD, SEM and TEM. When evaluated as cathode material for LIBs, the as prepared O-LNMO shows excellent electrochemical performance in terms of reversible capacity, cycling stability and rate capability. Based on these results, the as prepared product is a promising cathode candidate for high energy density LIBs, and we conclude that the preparation of O-LNMO from the \( \text{Mn}_3\text{O}_4 \) precursor has a beneficial role in the synthesis of LNMO materials. To the best of our knowledge, there is seldom report on the preparation of LNMO using \( \text{Mn}_3\text{O}_4 \) as precursor.

2. Experimental Section

2.1 Materials synthesis

\( \text{Mn}_3\text{O}_4 \) precursor was synthesized by a hydrothermal reaction. Briefly, 1.0 mmol \( \text{Mn(CH}_3\text{COO})_2\cdot4\text{H}_2\text{O} \) and 8 mL glycerol were dissolved into a solution mixture of 25 mL DI water and 25 mL isopropanol, and the above mixture was stirred for 0.5 h. Then, the formed mixture was transferred into a 100 mL Teflon-lined stainless steel autoclave, and heated at 180 °C for 6 h. After the autoclave was cooled naturally to room temperature, the \( \text{Mn}_3\text{O}_4 \) products were collected and washed by centrifugation several times with DI water and absolute ethanol before dried in a vacuum oven at 60 °C overnight.

For the preparation of O-LNMO, the obtained \( \text{Mn}_3\text{O}_4 \) precursor, stoichiometric amounts of the obtained \( \text{Mn}_3\text{O}_4 \) precursor, \( \text{Ni(CH}_3\text{COO})_2\cdot4\text{H}_2\text{O} \) and \( \text{LiCH}_3\text{COO}\cdot2\text{H}_2\text{O} \) (Li excessive 5%) were well dispersed in 10 mL ethanol. After ethanol evaporation, the resulting products were calcined at 800 °C in air for 10 h to obtain the final O-LNMO.
As a comparison, bulk LNMO (B-LNMO) was also prepared by a sucrose-assist combustion method to illustrate the effect of the prepared O-LNMO. Typically, 2 g sucrose was added to 30 mL DI water, Ni(CH\(_3\)COO\)_2\(\cdot\)4H\(_2\)O, Mn(CH\(_3\)COO\)_2\(\cdot\)4H\(_2\)O and LiCH\(_3\)COO\(\cdot\)2H\(_2\)O (Li excessive 5%) were added to the above mixture in a molar ratio. Then the resulting solution was heated to 90 °C under stirring condition to evaporate water. Finally, the obtained precursor was calcined at 800 °C for 10 h in air.

### 2.2 Materials characterization

Crystallographic information of the samples was investigated with X-ray diffraction analysis (XRD, Bruker D8 ADVANCE, Germany). The morphology and structures of the samples were examined using scanning electron microscopy (SEM, Merlin, Germany), and transmission electron microscopy (TEM, JEM-2100HR, Japan). After cycling, cells were carefully opened in a glovebox to retrieve the electrodes for SEM observation and ICP analysis. The cycled LNMO electrodes and Li foils extracted from the cells were rinsed with anhydrous DMC to remove residual Li salt and EC, and then followed by drying at room temperature overnight in the antechamber under vacuum. The contents of Mn and Ni deposited on Li foils were determined by ICP-MS (IRIS Intrepid II XSP, USA).

### 2.3 Electrochemical measurements

Electrochemical properties were measured using two-electrode coin cells (CR2025) in an argon-filled glovebox, in which the moisture and oxygen concentration were less than 1.0 ppm. The working electrode was fabricated by blending the prepared LNMO samples, acetylene black and polyvinylidene fluoride at a ratio of 8:1:1 in N-methyl-2-2pyrrolidone. The slurry was mixed and coated onto a piece of Al foil followed by drying at 120 °C for 2 h. Lithium metal was employed as the counter electrode and reference electrode, Celgard 2400 as separator, and the electrolyte was 1.0 M LiPF\(_6\) dissolved in ethylene carbonate/dimethyl carbonate/ethyl methyl carbonate (1/1/1 in volume). A battery test system (LAND, CT2001A, China) was used for charging/discharging tests of the cells. Cyclic voltammetry is conducted on an Arbin testing system (BT2000, USA).

### 3. Results And Discussion

Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) provide insights into the morphology and detail structure of the samples. As shown in Fig. 2a, the as-prepared Mn\(_3\)O\(_4\) precursor exhibits nanoparticle shape, demonstrating a random particle distribution with a range of diameters of 30 to 60 nm. The structure of the as-prepared Mn\(_3\)O\(_4\) precursor could further be demonstrated by their TEM image shown in Fig. 2b, where the Mn\(_3\)O\(_4\) nanoparticles with sizes of 40 nm could clearly visualized. The relatively darker locations in Fig. 2b appear to indicate the as-prepared Mn\(_3\)O\(_4\) nanoparticles with slight agglomeration. As is well known, nanoparticles tend to aggregate into large secondary forms owing to their high surface energies and small dimensions. To synthesis the LNMO, the as-prepared Mn\(_3\)O\(_4\) precursor are immersed in ethanol containing Ni(CH\(_3\)COO\)_2\(\cdot\)4H\(_2\)O and LiCH\(_3\)COO\(\cdot\)2H\(_2\)O, followed by evaporation. After ethanol evaporation, the resulting products were calcined at 800 °C in air to obtain O-LNMO, and the formation of O-LNMO is illustrated by Fig. 1. It can be seen
from Fig. 2c that the obtained O-LNMO powders show octahedral structure with clean and smooth surface facets, suggesting the O-LNMO are well crystallized, and the particle size ranging from 600 to 800 nm, which is typically different from the Mn$_3$O$_4$ nanoparticles precursor as discussed above, indicating the octahedral structure is well formed after calcined at high temperature. TEM observation (Fig. 2d) reveals that the LNMO particles display octahedral shapes with well-defined edges and corners, which is in good agreement with SEM examination.

XRD was conducted to identify the crystallographic structure of the prepared samples. As can be seen from Fig. 3a, all the XRD patterns can be indexed to the well-known tetragonal hausmannite crystal structure model of Mn$_3$O$_4$ (JCPDS no. 24–0734) [28], which is in accordance with the previous reports. No characteristic peaks related to impurities are observed, which suggests the pure Mn$_3$O$_4$ can be prepared by the hydrothermal process. It can be seen from Fig. 3b that the LNMO displays typical profile of spinel phase (JCPDS no. 80-2162) with Fd3m cubic space group [29]. The as-prepared LNMO shows sharp and strong diffraction peaks, indicating its good crystallinity, which is beneficial to cycling performance and high discharge capacity. As shown, minor residues peaks (as marked asterisks) at 2θ = 37.52, 43.62 and 63.42 that can be attributed to Li$_x$Ni$_{1-x}$O$_2$ [21]. This is a common phenomenon that such impurities can be detected in the preparation of LiNi$_{0.5}$Mn$_{1.5}$O$_4$. To illustrate the effect of the prepared O-LNMO, bulk LNMO (B-LNMO) was also prepared by a sucrose-assist combustion method. As is shown in SFig.1, the B-LNMO exhibits irregular polyhedron shape with multiply facets and severe aggregation after annealing at 800 °C, indicating that it is difficult to control the morphology of the LNMO materials in terms of such method. As a comparison, XRD patterns of O-LNMO and B-LNMO are shown in Fig. 4, all peaks of the two samples can be assigned to the spinel structure with a space group of Fd3m. When all the peaks in the XRD patterns are normalized by the (311) peak, the (111) and (400) peaks of O-LNMO are more intensive than that of B-LNMO. The relative peak intensities reflect the dominant surface orientations of the products. This result suggests both the prepared samples have the same spinel structure but different crystallographic facets, which might result in different electrochemical performance.

To study the electrochemical properties of the prepared LNMO, half cells were assembled using Li metal as reference and counter electrodes. The cyclic voltammetry (CV) curves of B-LNMO and O-LNMO were shown in Fig. 5a, for the both samples, there two pairs of redox peaks centered in the high voltage of 4.6 ~ 4.9V, relating to the reversible reactions of Ni$^{3+}$/Ni$^{4+}$ and Ni$^{2+}$/Ni$^{3+}$ couples [30], while a pair of broad and weak peak due to the Mn$^{3+}$/Mn$^{4+}$ couple could be also seen around 4.0V [18]. The O-LNMO sample exhibits slight higher peak current density and more symmetrical redox than the B-LNMO, indicating enhanced electrode reactivity. Figure 5b shows the cycling performance of the O-LNMO and B-LNMO, the O-LNMO delivers a discharge capacity of about 117 mAh g$^{-1}$ after 300 cycles, and maintains 94% of its initial discharge capacity (124.7 mAh g$^{-1}$) at 1C, while in the B-LNMO case, the discharge capacity of the cell with B-LNMO electrode decreases from 122.3 to 108 mAh g$^{-1}$ with a capacity retention of 88%, which suggests the O-LNMO electrode shows better cycling stability. To evaluate the rate capability of the prepared samples, the cells were charged at a rate of 0.5C and discharged at different rate currents
ranging from 0.5C to 40C between 3.5 and 4.9 V, Fig. 5c shows the obtained results, both the O-LNMO and B-LNMO electrodes suffer from a substantial decrease with increasing the rate, it is obvious that the O-LNMO has a better rate capacity than the B-LNMO counterpart, even at a rate of 40C, a specific capacity of 99.2 mAh g\(^{-1}\) could be still obtained for the O-LNMO, while it is only 26.7 mAh g\(^{-1}\) for the B-LNMO. Moreover, O-LNMO exhibits obviously slower capacity decay with increasing discharge rates. These suggest that the O-LNMO shows better rate capacity and cyclability than that of B-LNMO. The remarkable improvement of rate capacity of the O-LNMO can be ascribed to nanosized octahedral LiNi\(_{0.5}\)Mn\(_{1.5}\)O\(_4\) with exposed (111) facet, which can facilitate Li\(^{+}\) insertion/deinsertion in the LNMO electrode. The long-term cycling performance of the O-LNMO electrode is shown in Fig. 5d, after cycling at 5C for 2000 cycles, the O-LNMO can still deliver a discharge capacity of 86.7 mAh g\(^{-1}\) with 73% capacity retention, meaning only 0.0135% capacity decay per cycle. The excellent cycling stability and rate capability from the O-LNMO indicates its potential application for the high performance lithium-ion batteries.

Surface morphology of the O-LNMO and B-LNMO electrodes after 300 cycles are observed by SEM. It can be observed from Fig. 6b that some of the B-LNMO particles suffered destruction after cycling, which might be attributed to the transition metal dissolution during cycling. On the contrary, the structure of the O-LNMO particles maintained well (Fig. 6a), the octahedral morphology can be identified clearly. Moreover, the surface of the B-LNMO electrode is covered with nonuniform and thick surface layer, which can be ascribed to the severe decomposition of the electrolyte cycling upon high voltage. Different from the case of B-LNMO, the O-LNMO shows uniform and thin deposit on the surface layer, indicating there is less electrolyte decomposition on the O-LNMO particle, which results from the formation of the stable SEI due to the exposed (111) facet.

To further investigate the effect of the O-LNMO, the images of the cycled Li anodes was shown in Fig. 7. The smooth and clean surfaces of the Li anodes no longer exist, and significant changes can be seen on both cycled Li anodes. It can be observed that much more slurry precipitated on the Li anode extracted from the cell with B-LNMO cathode than that taken from cell with O-LNMO cathode. Therefore, ICP-MS was used to further analyze the chemical composition of the precipitated slurry on the cycled Li anodes. The two cycled Li anodes were first washed with DMC for several times, and dissolved in 2% HNO\(_3\) solution, followed by adding deionized water to 25 mL. As is shown in Table S1, it can be seen that the content of Mn and Ni for the Li anode extracted from the O-LNMO cell are much lower than that with B-LNMO cell. The results of ICP-MS experiment suggests that dissolution of Mn and Ni from the LNMO cathode can be reduced, which might be ascribed to the structural stability and the stable SEI formed on the (111) facet resulting from the nanosized octahedral LNMO.

### 4. Conclusions

In this work, we have synthesized nanosized octahedral LiNi\(_{0.5}\)Mn\(_{1.5}\)O\(_4\) with predominant (111) facet using Mn\(_3\)O\(_4\) nanoparticles precursor. The crystal structure, morphology characteristics, and
electrochemical performance of the LNMO were thoroughly investigated. The resultant octahedral \( \text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4 \) with nanosized structure can buffer the lattice stress caused by John-Teller distortion and favour fast \( \text{Li}^+ \) intercalation kinetics. Moreover, stable and protective SEI formed on the LNMO can be obtained due to the exposed (111) facet. As a result, the LNMO exhibits excellent cyclic stability and rate capability. We think that the results reported here may shed more light on the practical application of LNMO material, which would promote the development of the high energy density LIBs.

5. Declarations

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