High rate capability of 5 V $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ cathode material synthesized via an microwave assist method

Xu-Yong Feng^{a, b}, Chen Shen^b, Hong-Fa Xiang^a, Han-Kang Liu^a, Yu-Cheng Wu^{a*}, Chun-Hua Chen^{b*}

^a School of Materials Science and Engineering, Hefei University of Technology, Anhui 230009, PR China.

^b CAS Key Laboratory of Materials for Energy Conversions, Department of Materials Science and Engineering & Collaborative Innovation Center of Suzhou Nano Science and Technology, University of Science and Technology of China, Anhui Hefei 230026, China

Abstract

$\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ spinel samples are synthesized through a citric acid method using either microwave heating or oven heating to prepare the precursor gel. Contrast to the oven heating, the microwave heating is a fast way to obtain a homogenous precursor gel and remove all the crystal water with a higher heating temperature. The product synthesized with this method is of different fine structure and higher purity of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$. As a result, the $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ obtained from microwave heating shows better rate performance with a high capacity of about 122 mAh g⁻¹ at 10C, which is 92% of the capacity at 1C (131 mAh g⁻¹). The cycling performance of this product at the rate of 1C is also impressive with about 95% of its initial capacity retained after 200 cycles (125 mAh g⁻¹). When the current increases to 10C rate, the cycling performance becomes worse due to the increase in impedance during cycling. At the same time, part of the sample is over-discharged and transformed to a tetragonal phase at a high discharge current of 10C and become electrochemical irreversible, resulting to the capacity loss. The energy density (521 Wh/kg) of the microwave assist sample is also pretty high at high rate of 10C, due to the good rate

* Corresponding author Tel.: Tel.: +86 551 2905413.

E-mail address: ycwu@hfut.edu.cn (Yu-Cheng Wu); cchchen@ustc.edu.cn (Chun-Hua Chen)

performance.

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1. Introduction

High energy lithium ion batteries are increasingly demanded for electric vehicles and portable electronic devices in the past decades. There are two ways to increase the energy density, i.e. increasing the voltage or the capacity, since the energy density of a cell is voltage times capacity. Besides, higher voltage is also benefit to high power (power equals to voltage multiplied by current), which makes high voltage cathode materials become popular in the past decades. Doped spinel $\text{LiM}_x\text{Mn}_{2-x}\text{O}_4$ ($0 < x < 1$, $\text{M}=\text{Ni}, \text{Cr}, \text{Co}$) is one of the choices for high potential cathodes since their working potential is as high as 4.7 V ($\text{Ni}^{2+}/\text{Ni}^{3+}$, $\text{Ni}^{3+}/\text{Ni}^{4+}$, $\text{Cr}^{3+}/\text{Cr}^{4+}$, $\text{Co}^{3+}/\text{Co}^{4+}$) vs. Li^+/Li [1-6], which is higher than that of most cathode materials like LiMn_2O_4 (4 V vs. Li^+/Li [7, 8]), LiCoO_2 (4 V vs. Li^+/Li [9, 10]) and LiFePO_4 (3.4 V vs. Li^+/Li [11, 12]). On the other hand, the stability of LiMn_2O_4 is worse due to the disproportionate of Mn^{3+} ($2\text{Mn}^{3+} = \text{Mn}^{2+} + \text{Mn}^{4+}$) and dissolve of Mn^{2+} in the electrolyte, $\text{LiM}_x\text{Mn}_{2-x}\text{O}_4$ with low valence ions doping can efficiently decrease the concentration of Mn^{3+} and thus enhance the cycling performance. Among these doped spinel lithium manganese oxides, the nickel doped $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ [2, 4-6] with no Mn^{3+} exhibits better cycle performance and rate performance and attracts more attention. In the past decade, $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ with good electrochemical performance was prepared via different methods, like solid-state reaction [13-15], sol-gel process [16, 17], co-precipitation [18, 19] and so on. The electrochemical performance is much different by using different raw material, different method, and different temperature. For the solid state reaction at a higher temperature than 700°C, a rock salt phase (NiO or $\text{Li}_x\text{Ni}_{1-x}\text{O}$) separated from $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ as an impurity [13-15], due to the decomposition of Mn^{4+} to Mn^{3+} and oxygen loss reaction at high temperature, Better mix with the raw materials is helpful to reduce the amount of impurities and improve the performance of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$. A citrate-based sol gel method is a common way to synthesize $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ with better performance [20-23].

Recently, microwave-assisted sintering has been developed to prepare $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ cathode materials due to the advantages of uniform and rapid heating

[24-26]. When sintering under the microwave, the reaction time can be shortened to 7 min at 700°C [25] and the electrochemical performance is much better. Even so, the microwave sintering at high temperature is hard to be widely applied due to a special device in need. In this paper, we introduced the microwave heating to prepare a uniform gel of Li-Ni-Mn precursor for $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, using a household microwave oven. The Li-Ni-Mn precursor obtained by microwave heating is different in appearance, homogeneity and composition, thus to reduce the impurity and change the fine structure, which results to a better electrochemical performance of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$.

2. Experimental

The spinel $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ sample was synthesized by a citric acid method with the drying step completed in a microwave oven or in an electric oven. All chemicals were purchased from Sinopharm Chemical Reagent Co., Ltd. Lithium acetate ($\text{LiAc} \cdot 2\text{H}_2\text{O}$, 10.5 mmol), nickel acetate ($\text{Ni}(\text{Ac})_2 \cdot 4\text{H}_2\text{O}$, 5 mmol) and manganese acetate ($\text{Mn}(\text{Ac})_2 \cdot 4\text{H}_2\text{O}$, 15 mmol) were first dissolved in 120 ml deionized water, then 20 mmol citric acid was added to the solution as a chelating agent. After a mixing by magnetic stirring, the solution was put in a microwave oven (G7D23AP-TD, Galanz) and heated at high power for 30 min to obtain a homogeneous xerogel. As a comparison, another precursor solution was put in an electric oven at 80°C to remove water. These two kinds of xerogels were calcined at 500°C in air for 5 h. After being ground, the as-calcined powders were sintered at 900°C for 10 h. Then the temperature was lowered to 700°C (1°C/min) and annealed for 10 h, after that, the samples were natural cooled to room temperature to obtain $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ products. These two kinds of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ were marked as M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ and O- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, corresponding to a microwave oven heating and an electric oven heating respectively. The obtained xerogels are correspondingly called O-gel and M-gel.

The crystalline structures of the $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ sample were characterized by X-ray

diffraction (XRD) using a diffractometer (Rigaku TTR-III, Cu K α radiation). The diffraction patterns were recorded at room temperature in the 2θ range from 10° to 80° . Raman spectra of these samples were measured on a LABRAM-HR Confocal Laser Micro-Raman spectrometer using an argon laser (wavelength 514.5 nm) at room temperature. The powder morphology and particle size were characterized with a scanning electron microscope (JSM-6390LA, JEOL).

The electrochemical properties of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ were measured with CR2032 coin-type half-cells. The working electrode consisted of a mixture of 84 wt% $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, 8 wt% carbon black and 8 wt% PVDF binder and each electrode film contains around 5 mg active material. The cells were assembled in an argon-filled glove-box (MBraun Labmaster 130) with a porous polypropylene membrane (Celgard 2400) as separator and 1.0 M LiPF_6 in EC:DMC (1:1 w/w) as electrolyte. These half-cells were cycled in the voltage range from 3.5 to 5.1 V on a multiple cycler (Neware). A.C. impedance of the cells were measured on a CHI 604 Electrochemical Workstation in the frequency range from 10^{-2} Hz to 10^5 Hz.

3. Results and discussion

3.1 Structure and morphology of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$

The obtained gel for $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ is different when the water solvent evaporated through different process. Citric acid is helpful to form a uniform gel when removing the water solvent in the solution. Still, there are some dissolved salts separating from the solution prior to others, especially when dried slowly. The color of O-gel on the beaker (red arrow, Fig.1b) is a little light, which may contain less Ni. A faster evaporation of water can suppress the separation of different raw materials effectively (Fig.1a) and prepare homogenous gel. The thermal analysis results of the obtained xerogels are shown in Fig.1c. For O-gel, there is an endothermic peak at around 150°C , which is related to a dehydration process. In addition, another strong exothermic peak appears at around 320°C , which corresponds to the decomposition of the precursor. According to our previous work [27], $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ is formed at about

300°C with the raw materials of acetates, which means the spinel phase $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ is already formed along with the decomposition of organics. Nevertheless, the crystallinity of obtained samples at such a low temperature is poor and high temperature post-treatment is necessary to enhance the crystallinity. For M-gel, there is no dehydration reaction at around 100°C, suggesting that the crystal water in the acetates is lost during the heating process in the microwave oven. It is worth noting that the M-gel blots easily and needs to put into the furnace as soon as possible. With absent of crystal water, the decomposition temperature of M-gel and formation of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ is slightly higher than that of O-gel (362°C). The different uniformity and chemical composition (with or without crystal water) of gels may lead to the different fine structure and electrochemical performances of their derived $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ samples.

The scanning electron microscopy of the $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ powders is shown in Fig. 2. For both M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ and O- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, the particles are of regular morphology with a size of 1~5 μm . The particle size is much related to the sintering procedure at high temperature that there is no obvious difference between these two samples sintered at same temperature.

The X-ray diffraction patterns of the prepared $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ samples exhibit strong peaks that present a cubic spinel structure of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ (Fig.3a, JCPDS 51-0537). These sharp peaks indicate the highly crystalline feature of the synthesized $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ powders. According to previous works [25, 28-29], the ratio value of the $I(311)/I(400)$ peaks reflects the structural stability of the $[\text{M}_2]\text{O}_4$ spinel framework. Here, the $I(311)/I(400)$ ratio of the M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ and O- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ is 1.00 and 0.97 respectively, indicating better stability of M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ sample. In addition to the $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, rock salt phase (NiO) appears in the XRD pattern as impurity, especially for the O- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ (around 44° and marked with *). Fast drying with microwave is helpful to get homogenous gel (Fig. 1a, b) and restrain the formation of impurity. On the hand, formation of NiO impurity changes the ratio between Ni and Mn in $\text{LiNi}_{0.5-x}\text{Mn}_{1.5+x}\text{O}_4$ and thus the peaks shift. The peaks of M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ shift to the right (Fig. 3b) for more Ni in the structure [7] and less

impurity. There are two different space groups of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, $Fd\bar{3}m$ and $P4_332$, with similar XRD pattern and hard to distinguish [30-32]. So Raman spectra are necessary to distinguish these two different space groups. From the Raman spectra (Fig. 3b), there is some difference between these two samples, especially for the peaks around 200 cm^{-1} , which are characteristic peaks of $P4_332$ [30]. With higher intensity of the peaks around 200 cm^{-1} , the sample of $\text{O-LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ should contains more $P4_332$ and the main space group of $\text{M-LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ is $Fd\bar{3}m$. From previous work [30, 32], the electrochemical performance including cycle and rate performance of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ is highly related to the structure, $Fd\bar{3}m$ shows better performances.

Since the existing of Mn^{3+} is very important for the electrochemical performance of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, X-ray photoelectron spectra of the Mn $2p_{3/2}$ peak are measured and shown in Fig. 4 to confirm the coexisting of Mn^{3+} and Mn^{4+} . For these two different samples, two peaks at 643 and 642 cm^{-1} , corresponding to Mn^{4+} and Mn^{3+} coexists [26]. The ratio of Mn^{3+} calculated from the XPS spectra is 58% for $\text{M-LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ and 35% for $\text{O-LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, $\text{M-LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ contains more Mn^{3+} than $\text{O-LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, indicating more oxygen defect. XPS of Ni 2P was also measured and shown in Fig. 4 and no Ni^{3+} is found in both two samples.

3.2 Electrochemical performance of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$

The galvanostatic charge-discharge curves of the synthesized $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ samples at different current rates are shown in Fig.5. The specific capacity of $\text{M-LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ is about 131 mAh g^{-1} at the rate of 1C (Fig. 5a). The overall capacity can be divided into those at three plateaus of 4.7 V, 4.6 V and 4.0 V, which correspond to the conversions of $\text{Ni}^{4+}/\text{Ni}^{3+}$, $\text{Ni}^{3+}/\text{Ni}^{2+}$ and $\text{Mn}^{4+}/\text{Mn}^{3+}$, respectively. For $\text{M-LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, the capacity at the 4.0 V plateau is higher than that of $\text{O-LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, indicating higher ratio of Mn^{3+} in the structure, which is consistent with the XPS results. Both of specific capacity and discharge voltage decrease with increase of current. When the current increases to 10C, the specific capacity decreases to 122 mAh g^{-1} (93% of the specific capacity at 1C) and the discharge voltage

decreases to about 4.4 V. Eventhough, the performance is much better than that of O-LiNi_{0.5}Mn_{1.5}O₄ (Fig. 5b). The capacity of O-LiNi_{0.5}Mn_{1.5}O₄ decreases to 69 mAh g⁻¹ at 10C, although its specific capacity at 1C current density (about 132 mAh g⁻¹) is close to that of M-LiNi_{0.5}Mn_{1.5}O₄. The difference of electrochemical performance between these two samples is highly related to their structures, such as Mn³⁺ content, space group, et al. For example, the 4 V plateau is related to the existing of Mn³⁺ and the rate performance is related to the different space group, LiNi_{0.5}Mn_{1.5}O₄ with $Fd\bar{3}m$ structure always show better rate performance, due to the higher conductivity [32].

The specific energy density of M-LiNi_{0.5}Mn_{1.5}O₄ is appreciable because of its high discharge voltage and specific capacity, even at high rates. It can be seen in Fig. 6 that the specific energy density of M-LiNi_{0.5}Mn_{1.5}O₄ at 1C is about 602 Wh kg⁻¹, which is higher than that of LiMn₂O₄ (419 Wh kg⁻¹) [33]. When the current density increases to 10C, the specific energy decreases to 521 Wh kg⁻¹, which is still higher than that of LiMn₂O₄.

The cycling performance (Fig. 7a) of M-LiNi_{0.5}Mn_{1.5}O₄ at 1C is very good that almost 95% of the initial capacity is retained after 200 cycles, although there are some trivalent manganese ions existing in the sample. The good cycling performance suggests better stability of M-LiNi_{0.5}Mn_{1.5}O₄, for the higher ratio of $I(311)/I(400)$, as shown in Fig. 3a. For the sample of O-LiNi_{0.5}Mn_{1.5}O₄, only 123 mAh g⁻¹ (or 89% of the initial capacity) is retained after 120 cycles (since the capacity decayed fast, we stopped the test at 120 cycles). The sample of M-LiNi_{0.5}Mn_{1.5}O₄ is also cycled at higher rates (Fig. 7b) for more critical test. The initial specific capacity of M-LiNi_{0.5}Mn_{1.5}O₄ at 10C is about 129 mAh g⁻¹, which is very close to that at 1C and higher than the value after 21 cycles (Fig.6) due to its lower impedance in the first several cycles. The cycling performance at 10C becomes much poor with a capacity of only 88 mAh g⁻¹ retains after 200 cycles. To find the reason for the fast fading of the specific capacity at 10C, the half-cell of this sample is tested at the alternating rates of 1C and 10C (Fig.6b, red). It is found that its specific capacity at the rate of 1C

after 200 cycles is almost equal to that cycled constantly at 1C (Fig.6a, black). This result suggests that very little active material is consumed at the current density of 10C, and most of the capacity fading at 10C should be due to the increasing impedance. The impedance spectra of a Li/M-Li_{0.7}Ni_{0.5}Mn_{1.5}O₄ cell at different cycles (cycled at 1C) and charge to 50 mAh g⁻¹ are measured and shown in Fig.8. The impedance at the 3rd cycle is much lower than that at the 23rd cycle. The increase of the impedance may come from the continuous decomposition of the electrolyte at high voltage at the surface of M-LiNi_{0.5}Mn_{1.5}O₄ [34]. Therefore, the cycling performance should be better even at the current density of 10C if the electrolyte is stable at high voltage. Also, other kinds of structure change may lead to the increasing of impedance (discussions later on). Actually, similar increasing of impedance happens in the Li/O-Li_{0.7}Ni_{0.5}Mn_{1.5}O₄ cells (not shown here) and it is common for most high voltage cathodes.

After 200 cycles at 1C, the structure of M-LiNi_{0.5}Mn_{1.5}O₄ sample remains the same as before (Fig. 9a, JCPDS 51-0537). That is why the cycling performance is very good at low rate. On the other hand, when the current rate increases to 10C, a new phase is detected by XRD (Fig. 9b). The new phase is of a tetragonal structure with the space group of I4₁/amd, which should be over-discharged spinel phase, like Li₂Mn₂O₄ (JCPDS 32-0581). When discharged at this high rate, part of LiNi_{0.5}Mn_{1.5}O₄ samples is likely over-discharged to Li_{1+x}Ni_{0.5}Mn_{1.5}O₄, due to the inhomogeneity of the electrode film and high potential gradient at high rate. When over discharged, the part irreversibility of Li_{1+x}Ni_{0.5}Mn_{1.5}O₄ during the cycling makes the cycle performance at a high rate much worse (Fig. 7b).

The cycling performance of LiNi_{0.5}Mn_{1.5}O₄ samples is tested more strictly at 55°C (Fig. 10). The specific capacity of M-LiNi_{0.5}Mn_{1.5}O₄ decreases faster than that at room temperature with only 93% of the initial capacity is retained after 100 cycles. The worse cycling performance at the elevated temperature is probably due to the dissolution of manganese, which is easier at a high temperature. The O-LiNi_{0.5}Mn_{1.5}O₄ shows an even worse cycling performance at high temperature, indicating a worse stability.

4. Conclusions

A $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ spinel powder with a main phase of $Fd\bar{3}m$ is synthesized by a citric acid method with a drying step in a microwave oven. It is clear that fast and high temperature heating by microwave is helpful to get a homogenous gel precursor with no crystal water. The final structure and thus electrochemical performances of final products are highly related to the gel precursor that the M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ is more pure (less NiO) and stable (higher $I(311)/I(400)$). This sample of M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ shows good rate performance with a specific capacity of 122 mAh g^{-1} at 10C and good cycling performance with only 5% capacity loss after 200 cycles at 1C. But the impedance of bare $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ without modification on the surface increases continuously and lead to a dramatically capacity loss at high rate (68% capacity left after 200 cycles at 10C). In addition, part of the $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ sample is over-discharged to $\text{Li}_{1+x}\text{Ni}_{0.5}\text{Mn}_{1.5}\text{O}_4$ and its space group is converted to $I4_1/amd$, which also leads to worse cycle performance.

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Figure captions:

Fig. 1 Xerogel of microwave heated sample (a) and oven heated sample, DTA (c) of the xerogels for $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ samples.

Fig. 2 SEM image of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ samples.

Fig. 3 XRD pattern (a), zoomed XRD pattern (b) and Raman spectra (c) of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ samples.

Fig. 4 X-ray photoelectron spectra of the Mn $2p_{3/2}$ and Ni $2p$ peak of (a) M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ and (b) O- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$.

Fig. 5 Galvanostatic charge and discharge curves of a $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4/\text{Li}$ cell at 1C charge and various C-rates of discharge.

Fig. 6 Rate performance of M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ sample.

Fig. 7 Cycling performance of $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ sample at different conditions: a) M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ and O- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ at 1C; b) M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ at 1C (charge)/10C (discharge) or 10C/10C.

Fig. 8 Ac impedance of M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ samples at 50 mAh g^{-1} state-of-charge in the 3rd (a) and 23rd (b) cycle.

Fig. 9 XRD patterns of M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ samples after 200 cycles: a) discharged at 1C; b) discharged at 10C.

Fig. 10 Cycling performance at 55°C of M- $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ sample at 1C.

a)

b)

M-LiNi_{0.5}Mn_{1.5}O₄

20kV X10,000 $\overline{1\mu\text{m}}$

14 40 SEI

O-LiNi_{0.5}Mn_{1.5}O₄

20kV X10,000 $\overline{1\mu\text{m}}$

14 40 SEI