



Effect of capacity matchup in the $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4/\text{Li}_4\text{Ti}_5\text{O}_{12}$ cells

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ABSTRACT

The effect of the capacity matchup between cathode and anode in the $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4/\text{Li}_4\text{Ti}_5\text{O}_{12}$ cell system on cycling property, choice of electrolyte, high voltage and overcharge tolerances was investigated by comparing the cells with $\text{Li}_4\text{Ti}_5\text{O}_{12}$ limiting capacity with the cells with $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ limiting capacity. The former exhibits better cycling performance and less limitation of electrolyte choice than the latter. Furthermore, the $\text{Li}_4\text{Ti}_5\text{O}_{12}$ -limited cell exhibits better tolerance to high voltage and overcharge than the $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ -limited cell, owing to taking advantage of the extra capacity of $\text{Li}_4\text{Ti}_5\text{O}_{12}$ below 1 V. It is thus recommended that the $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4/\text{Li}_4\text{Ti}_5\text{O}_{12}$ cell whose capacity is limited by $\text{Li}_4\text{Ti}_5\text{O}_{12}$ anode should be used to extend the application of the state-of-the-art lithium-ion batteries.

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1. Introduction

Lithium-ion batteries are now widely used as energy storage devices for portable electronic devices such as laptop computers, cellular phones and digital cameras due to their high energy density. Carbonaceous anode is always used in the lithium-ion batteries mentioned above, along with lithiated metal oxide cathodes, i.e. LiCoO_2 , LiMn_2O_4 and LiFePO_4 , because carbonaceous anode has better safety characteristic and long cycle life compared with lithium metal anode [1]. Yet the operating potential plateau of the carbonaceous anode is so close to that of metal lithium that “dendrite” could still be unavoidable, especially at high rates. And the solid electrolyte interface (SEI) layer on the carbonaceous electrode, which is usually formed at the potential below 0.8 V versus Li^+/Li and accompanied over time with active lithium loss, an increase in impedance and a decrease in specific capacity, limits the lifetime and rate capability of the lithium-ion batteries [2]. Furthermore, the state-of-the-art lithium-ion batteries with carbonaceous anode have some other drawbacks, such as thermal stability concerns [3], and the bad compatibility with propylene carbonate-based electrolytes and some functional electrolytes, i.e. the flame-retarded electrolytes containing phosphates or phosphonate [4,5]. For a wide

range of applications, including electric vehicles (EVs) and hybrid electric vehicles (HEVs) batteries, some candidates for the replacement of carbonaceous anodes exhibiting better cell performances and safety characteristic must be found.

The spinel $\text{Li}_4\text{Ti}_5\text{O}_{12}$ (denoted as LTO) has been demonstrated as an alternative anode material because it has a very flat potential plateau at around 1.5 V versus Li^+/Li and displays excellent reversibility and structural stability as a zero-strain insertion material in the charge–discharge process [6]. The stable spinel structure will seldom change in the PC-containing electrolyte, which is different from the graphite anode with the brittle layer structure that can be easily exfoliated for co-interaction of the solvent with lithium ion. Moreover, the 1.5 V voltage plateau also can avoid PC and some additives decomposing reductively, and even SEI would be not found easily to limit the rate of lithium insertion and extraction [3,7]. So the lithium-ion batteries with LTO anodes is applicable in more circumstances, with important advantages in terms of cycling performance and thermal stability, although the output voltage of lithium-ion batteries with LTO anodes is lower than that of lithium-ion batteries with carbonaceous anodes.

In recent years, many works about the full lithium-ion cells with LTO anode have been reported. Many 2 V lithium-ion battery systems have been studied continually, such as $\text{LiCoO}_2/\text{LTO}$, $\text{LiMn}_2\text{O}_4/\text{LTO}$, $\text{LiFePO}_4/\text{LTO}$, $\text{LiNi}_{1/3}\text{Co}_{1/3}\text{Mn}_{1/3}\text{O}_2/\text{LTO}$, etc. [8–12]. To obtain an operating voltage of more than 3 V, the so-called 5 V spinel $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$ (denoted as LNMO) cathode will be chosen to couple with the LTO anode and fabricate the lithium-ion battery. LNMO is one of the most promising and attractive cathodes

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because of its acceptable stability, good cycling performance and high dominant potential plateau at around 4.7 V [13,14]. Compared with LiCoO_2 , LNMO also has the obvious advantage of low cost. By the partial substitution of Mn with Ni, the better cycling performance and higher energy density have been found for LNMO than plain LiMn_2O_4 , even though the spinel LNMO has a limitation of requiring an electrolyte for the high operating potential. It has been validated that the 3 V LNMO/LTO cells exhibited good cycling performance, flatness in operating voltage and high rate capability [15]. Recently we reported the nonflammable electrolyte could be used in the LNMO/LTO cell systems to fabricate high safety lithium-ion batteries [7]. However, before the commercialization of this LNMO/LTO cell system, there are still many factors that should be investigated. In this study, we focus on the capacity matchup issue between the positive electrode LNMO and the negative electrode LTO.

Commercial lithium-ion batteries with carbonaceous negative electrode are typically assembled with excess negative electrode capacity to prevent lithium deposition during charging process. In this case, the capacity of a cell is limited by the positive electrode. As for the LTO-based lithium-ion batteries, the cell capacity limited by the positive electrode is unnecessary owing to the relatively high voltage plateau of LTO. In fact, it may be desirable to design LTO-based cells with some excess positive capacity to alleviate the degree of electrolyte decomposition at the high potential region by means of keeping a relatively large value of x in $\text{Li}_x\text{Ni}_{0.5}\text{Mn}_{1.5}\text{O}_4$ when the cell is near or at its fully charged state. The main purpose of this paper is to investigate the effect of the capacity ratio of cathode and anode in the LNMO/LTO cell system on the cell performance in order to boost the development of the LNMO/LTO cell systems or other similar cell systems.

2. Experimental

LTO was purchased from BTR Energy Materials Co., Ltd. As previously disclosed, LNMO was prepared by a radiated polymer gel (RPG) method [7,16]. Composite electrodes consisting of LTO or LNMO (80 wt.%), acetylene black (10 wt.%) and poly(vinylidene fluoride) (PVDF) (10 wt.%) were made by a tape-casting process on aluminum and copper foils, respectively. The electrolytes were 1 M LiPF_6 dissolved in carbonate solvent mixtures including ethylene carbonate (EC), PC, dimethyl carbonate (DMC) and diethyl carbonate (DEC). All solvent ratios indicated later in this paper were in

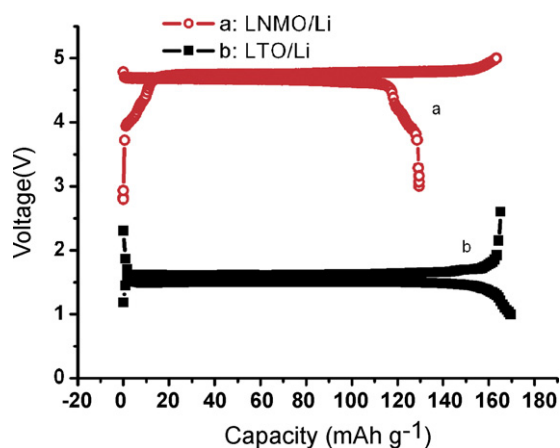


Fig. 1. The first charge–discharge voltage profiles of LNMO/Li (a) and LTO/Li (b) half-cells with the electrolyte of 1 M $\text{LiPF}_6/\text{EC} + \text{DMC}$ (1:1). The cells were operated at the rate of 0.2 mA cm^{-2} with the cutoffs of 3.0–5.0 V for LNMO/Li half-cell and 1.0–2.5 V for LTO/Li half-cell, respectively.

weight ratios. CR2032 coin cells were assembled in an argon-filled glove box (MBrumn Labmaster 130) and then galvanostatically cycled on a multi-channel battery cycler (Neware BTS2300, Shenzhen). In order to simulate the real load pattern of HEV batteries, accelerated cycle tests were designed and carried out to evaluate the cycle lifetime. In these tests, all of the cells were cycled alternately at the constant voltage at 3.5 V (charge) and at 2.0 V (discharge) for 2 min, respectively. Cyclic voltammetry (CV) was carried out at a CHI 604 Electrochemical Workstation to examine the high voltage retention of the cell systems. The ac impedance was also measured with the Electrochemical Workstation above, with the frequency range and voltage amplitude set as 100 kHz to 0.01 Hz and 8 mV, respectively. For most of the tests, the limited capacity and the excess capacity of the two electrodes in the LNMO/LTO full-cells were about 1.0 and 1.2 mAh, respectively, whether the cell capacity was determined by the LNMO cathode or the LTO anode (hereinafter designated as the LNMO-limited cell and the LTO-limited cell).

3. Results and discussion

As shown in Fig. 1, LNMO and LTO have the flat operating voltage plateaus of ~ 4.7 and ~ 1.5 V, respectively. On the LTO anode side, nearly all of common carbonate-based electrolytes have good stability. But most of the electrolytes are apt to be oxidized on the high-voltage LNMO cathode side, especially near the full charge state (100% SOC). Thus, for the LTO-limited cells, at the upper limit of 2.0–3.5 V cutoff voltage, the potential of LNMO cathode still remains at 4.7 V versus Li/Li^+ , while that of the LTO drops down from 1.5 V. However, when the cell capacity is limited by the LNMO cathode, under the same conditions, the potential of the LTO anode remains at 1.5 V, but that of the LNMO cathode rises to about 5 V where many adverse side reactions could occur between the electrolyte and the strong oxidative cathode. Results of the accelerated cycle tests of two kinds of full-cells above are compared in Fig. 2, in order to validate the inference above. To accelerate cell cycles, all the cells were cycled under constant voltage charge at 3.5 V for 2 min and discharge at 1.5 V for 2 min. Maximum charging current at 3.5 V was observed to be 10 mA and the maximum discharging current was 12 mA in this case. The cell stored and delivered about 10% of theoretical capacity on charge and discharge for 2 min. By cycling, the capacity decreased gradually to $\sim 5\%$ at the 2900th cycle. Fig. 2a and b shows the voltage profiles of the LTO-limited cells and the LNMO-limited cells at different cycles when they are operated galvanostatically between 3.5 and 1.0 V at the current of 0.2 mA cm^{-2} . Depending on which electrode is the limiting one, the specific capacities of the cells are calculated based on the mass of LTO anode and LNMO cathode, respectively. For a clear comparison, the capacity retention with the capacity normalized to the discharge capacity at the third cycle and coulombic efficiency for the two kinds of cells at different cycles are shown in Fig. 2c. After 2900 cycles, the LTO-limited cell shows higher capacity retention of 85% compared with only 56% for the LNMO-limited cell. And also the former usually has the higher coulombic efficiency for the same cycle than the latter. The ac impedance spectra of the two kinds of full-cells after different cycles are shown in Fig. 3. It can be seen that for both cells the impedance increases with cycle number, but the impedance of the LNMO-limited cell is greater than that of the LTO-limited cell at the same cycle, which is probably due to some adverse side reactions between the electrolyte and the LNMO cathode at a high cell voltage, and the passivation film with the high impedance formed further. These results indicate that the LNMO/LTO cell system with the capacity limited by LTO has the better cycling performance than that limited by LNMO, just as we forecast.

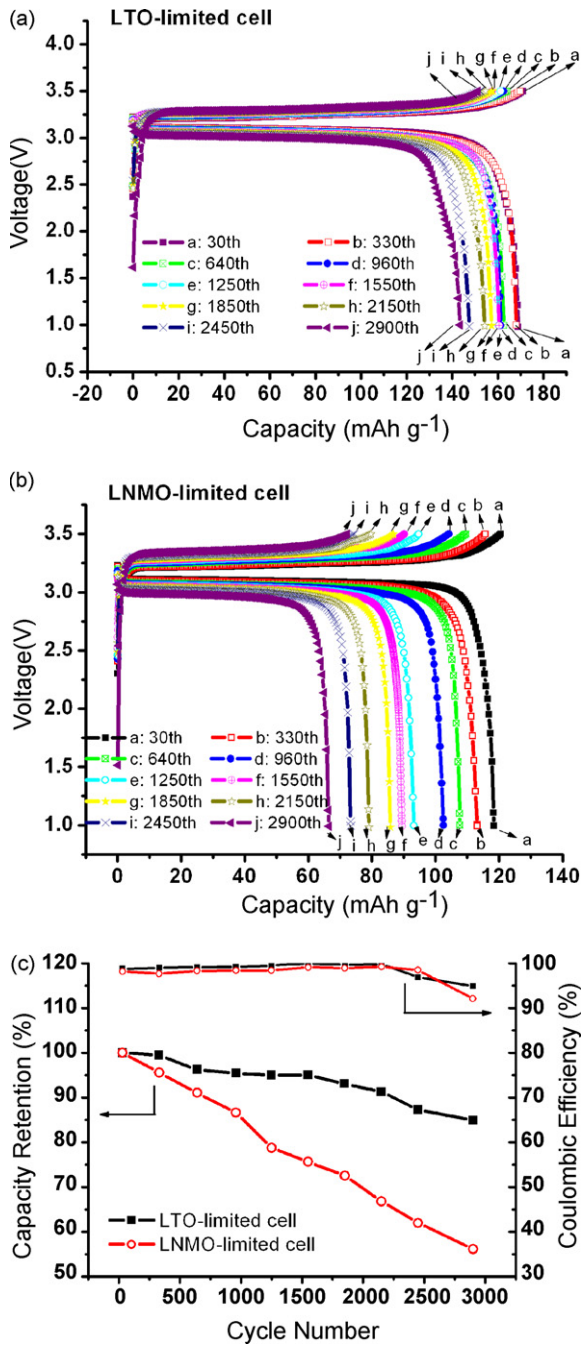


Fig. 2. Results on the accelerated cycling tests of the LNMO/LTO cells with capacity limited by LTO anode (a) and capacity limited by LNMO cathode (b), and their comparison on the capacity retention and coulombic efficiency (c). The cells were cycled at constant voltage charge at 3.5 V for 2 min and discharge at 2.0 V for 2 min. The voltage profiles were obtained at different cycles when they were operated galvanostatically between 3.5 and 1.0 V at the current of 0.2 mA cm^{-2} , with a few cycles discarded.

On the other hand, the application of a 5 V cathode material, i.e. $\text{LiNi}_{0.5}\text{Mn}_{1.5}\text{O}_4$, LiCoPO_4 or $\text{LiMn}_{1.9}\text{Co}_{0.1}\text{O}_4$, etc., usually encounters a barrier of limited electrolyte option. The $\text{LiPF}_6/\text{EC} + \text{DMC}$ system is a typical choice for the 5 V cathode materials mentioned above. Here, we examine whether the limitation could be alleviated or even released for the LTO-limited cells in comparison with the LNMO-limited cells. Two electrolytes, 1 M $\text{LiPF}_6/\text{EC} + \text{DEC}$ (1:1) and 1 M $\text{LiPF}_6/\text{PC} + \text{DEC}$ (6:5), have been chosen. Figs. 4 and 5

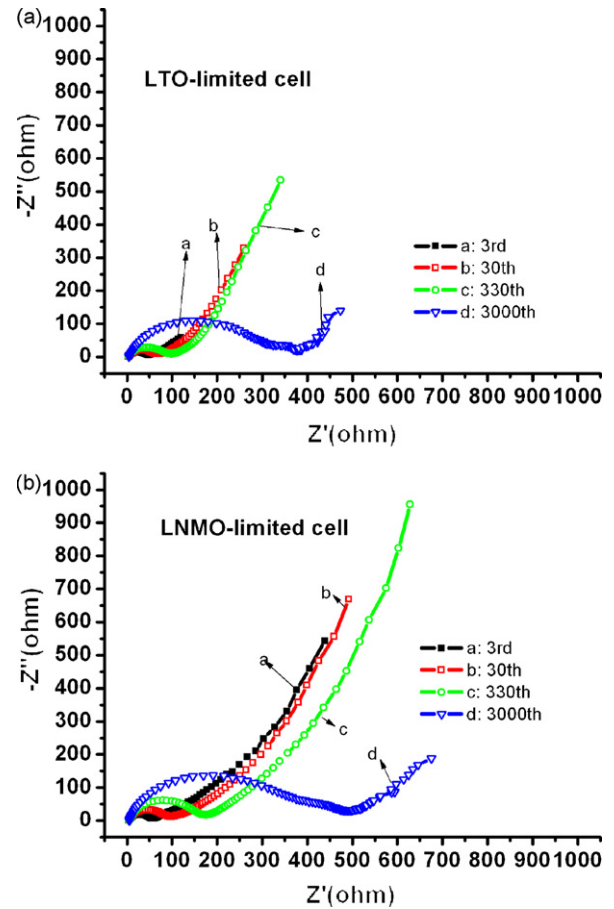


Fig. 3. ac impedance spectra of a LTO-limited cell (a) and a LNMO-limited cell (b) at different cycles.

show the electrochemical performance of two kinds of LNMO/LTO full-cells containing the electrolytes above, respectively. There is similar character between the two kinds of electrolytes for the first charge–discharge cycle voltage profiles (Figs. 4a and 5a). For both of the two kinds of electrolytes, the LTO-limited cells exhibit the flatter operating voltage and higher coulombic efficiency (CE) than the LNMO-limited cells. Moreover, the LTO-limited cells containing the two electrolytes have a relatively distinct change in voltage at the end of charge, which can be considered as an end-of-charge indicator. As for the LNMO-limited cells, the voltage at the end of charge rises slowly, thus it can not indicate clearly the state of full charge. Fig. 4b shows the cycling performance of two kinds of cells containing 1 M $\text{LiPF}_6/\text{EC} + \text{DEC}$ (1:1). It is obvious that the LTO-limited cells have higher CEs and slower capacity fading (0.20%/cycle) than the LNMO-limited cells whose capacity fading is 0.50%/cycle. Similar results can be found in Fig. 5b for the cells with 1 M $\text{LiPF}_6/\text{PC} + \text{DEC}$ (6:5) electrolyte system. The LTO-limited cells also have higher CEs and slower capacity fading (0.11%/cycle) than the LNMO-limited cells with the capacity fading of 0.26%/cycle. Based on the results above, it can be concluded that the electrolyte limitation of the LNMO/LTO cell system can be alleviated when the cell capacity is limited by the LTO anode.

Recently, Amine and his co-workers reported that a new phase generated after the spinel/rock-salt phase transition of LTO with extra lithium insertion [17]. Our recent work also indicated that the extra capacity is highly reversible and useful during long-term charge/discharge process between 0 and 3 V [3]. Now we propose that the extra capacity could be used to withstand high voltage

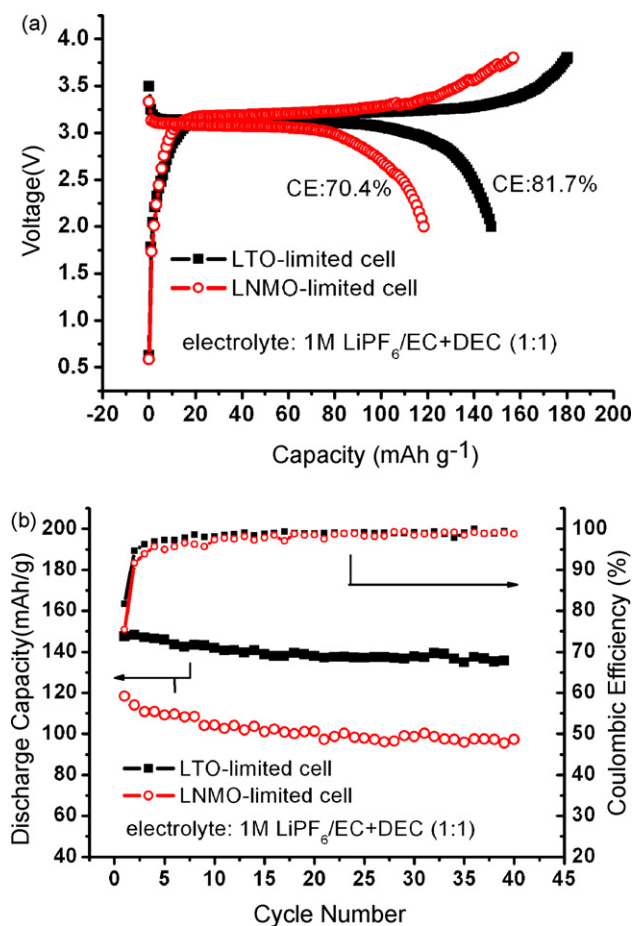


Fig. 4. Voltage profiles at the first cycle (a) and cycling performance (b) of a LTO-limited cell and a LNMO-limited cell with the electrolyte of 1 M LiPF₆/EC+DEC (1:1). The cells were cycled galvanostatically between 3.8 and 2.0 V at the current of 0.2 mA cm⁻².

and avoid overcharge if the cell capacity were limited by the LTO anode. The cyclic voltammetry (CV) results of a LTO-limited cell and a LNMO-limited cell are shown in Fig. 6. For the LTO-limited cell, the peak at 3.5 V during the positive scan and the peak at 2.6 V during the negative scan can be correlated to the spinel/rock-salt phase transition of the LTO anode and typical redox transition of the LNMO cathode. A pair of peaks above 4 V perhaps can be ascribed to the potential change mainly on the LTO anode due to the order/disorder transition of the LTO anode between the rock-salt Li₇Ti₅O₁₂ and the new phase reported by Amine and co-workers [17]. Therefore, the LTO-limited cell exhibits good reversibility between open circuit voltage and 5 V. However, an absolute irreversible phenomenon has been found for the LNMO-limited cell under the same conditions, because lots of side reactions could occur including electrolyte decomposition and electrode collapse when the cell voltage exceeds 4 V. The main reason is that when the potential of LNMO cathode arrives at a very high level of exceeding 5.5 V versus Li/Li⁺ with the cell voltage exceeding 4 V, the LNMO may be destroyed for overcharge and, more likely, the electrolyte begins fiercely to decompose on the LNMO electrode surface and becomes ineffective [2]. As for the LTO-limited cells, when the cell voltage exceeds 4 V, the LNMO cathode should still rest on the 4.7 V voltage plateau along with the LTO anode falling to 0.7 V where the extra capacity of LTO related to the Ti³⁺/Ti⁴⁺ transition and order/disorder transition is available, thus the stability and reversibility of the cell can remain nearly unchanged. Further, the cycling performance the

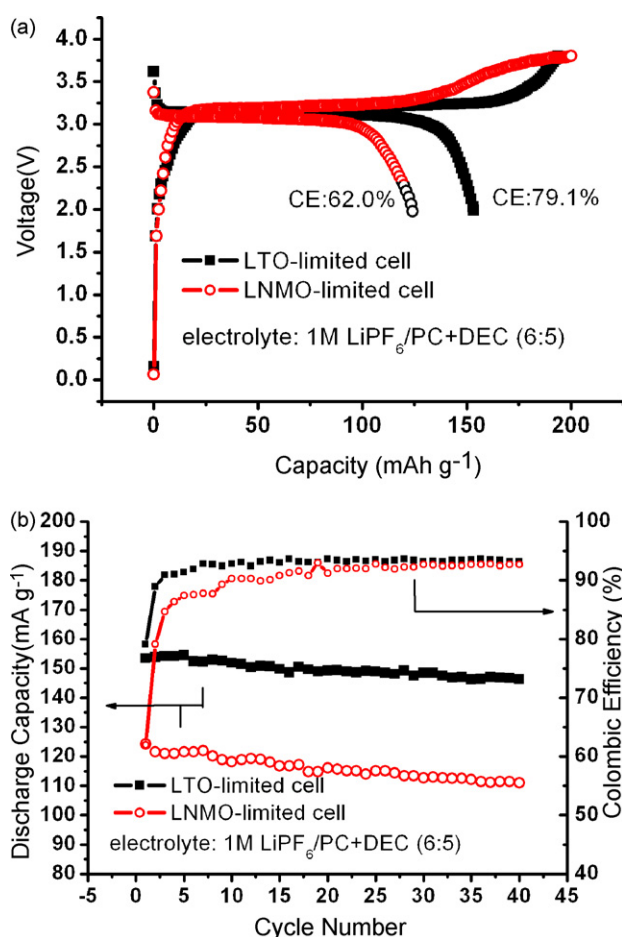


Fig. 5. Voltage profiles at the first cycle (a) and cycling performance (b) of a LTO-limited cell and a LNMO-limited cell with the electrolyte of 1 M LiPF₆/PC+DEC (6:5). The cells were cycled galvanostatically between 3.8 and 2.0 V at the current of 0.2 mA cm⁻².

LTO-limited cells with three voltage cutoffs, i.e. 2.0–4.0, 2.0–4.5, 2.0–5.0 V, is shown in Fig. 7, while the LNMO-limited cells can not operate even at the cutoff of 2.0–4.0 V. When the cutoff is set at 2.0–4.0 V, the LTO-limited cell has good capacity retention (90.8% after 50 cycles). Even though the upper limit of cutoff is elevated to 4.5 and 5 V, the LTO-limited cells are still capable of working

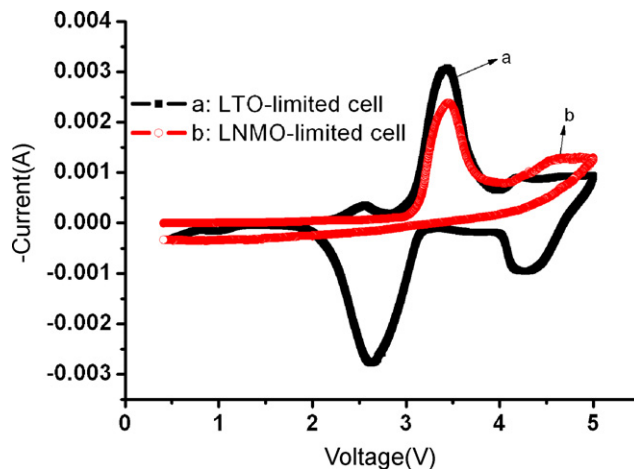


Fig. 6. Cyclic voltammetry of a LTO-limited cell (a) and a LNMO-limited cell (b) with the electrolyte of 1 M LiPF₆/EC+DMC (1:1). Scan rate $\nu = 0.5$ mV s⁻¹.

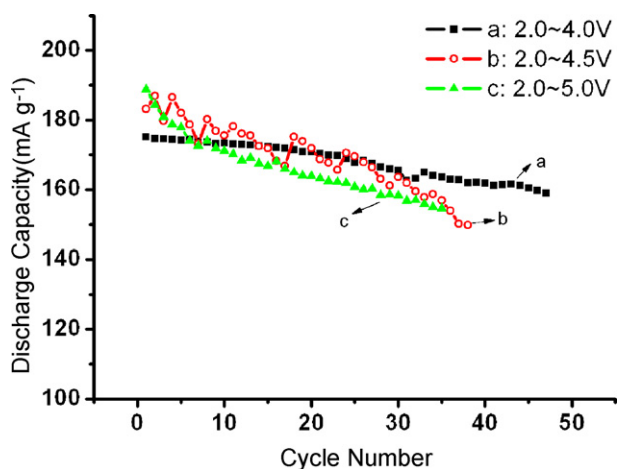


Fig. 7. The cycling performance of the LTO-limited cell with the electrolyte of 1 M LiPF₆/EC + DMC (1:1) at different voltage cutoffs: 2.0–4.0 (a), 2.0–4.5 (b), and 2.0–5.0 (c).

continually. So, it is concluded that the LTO-limited cells have a high degree of tolerance to the voltage, even to 5 V, but the LNMO-limited cells have no tolerance.

Overcharge is one of most important reasons for the safety issue of lithium-ion batteries, which may usually cause the electrolyte to an oxidative decomposition and make the cathode deteriorate rapidly. LTO anode has been validated with better safety characteristic on the thermal stability than the graphite anode [3]. Here we investigate the effect of overcharge on the safety performance of LNMO/LTO lithium-ion batteries. In the cells prepared for overcharge tests, the ratio of the limited capacity and the excess capacity of the two electrodes is about 1:2. All the cells were firstly cycled between 2.0 and 3.5 V at a current of 0.2 mA cm⁻², and then charged to 6 V, followed by discharge to 2.0 V at the same rate. Fig. 8 shows the results of overcharge tests on the two kinds of LNMO/LTO cells. It can be seen that the electrolyte in the LNMO-limited cell decomposes ruinously when the voltage exceeds 4 V, thus the cell cannot be discharged any longer and it seems to be destroyed completely after overcharge to 6 V. Considering the fact that most of the electrolyte systems containing LiPF₆ and carbonates will not

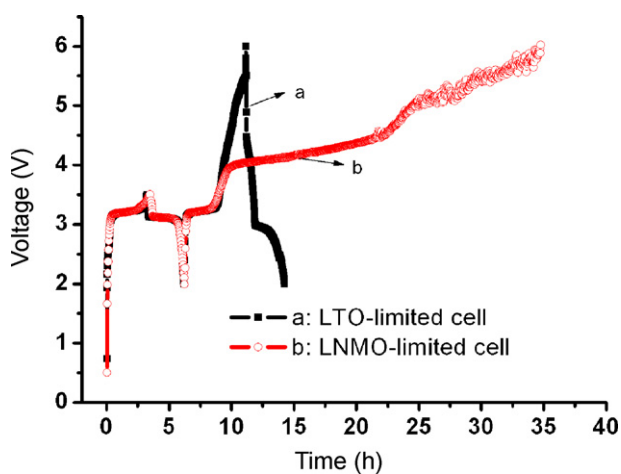


Fig. 8. The voltage–time profiles of two different capacity-limited cells during an overcharge up to 6 V. The electrolyte was 1 M LiPF₆/EC + DMC (1:1). The cells were first cycled normally between 2.0 and 3.5 V, followed by an overcharge to 6 V and discharge to 2 V at a rate of 0.2 mA cm⁻².

begin to decompose until 5.4 V versus Li⁺/Li. Thus the electrolyte above is stable for a cell with the carbonaceous anode when its voltage reaches 5 V, if the structure stability of cathode materials is ignored. However, the cells with the LTO anode whose capacity is determined by the cathode usually encounter more easily overcharge and oxidative decomposition for electrolyte, because the actual potential of the cathode exceeds 5.5 V when the voltage of the full-cells is 4 V. When the capacity is limited by LTO anode, the overcharge issue of a LTO-containing cell will be alleviated, because the extra capacity of the LTO anode below 1.0 V functions to postpone overcharge, and above all, the potential of the anode drops to 0 V along with the potential of cathode constant before the cell is charged to 4.8 V. From Fig. 8, perhaps the overcharge process for the LTO-limited cell is accompanied with the extra lithium inserting the Li₇Ti₅O₁₂ structure and a certain extend of decomposition for electrolyte, which is less violent than in the LNMO-limited cell. Thus, after overcharge the LTO-limited cell can still release certain capacity, although the discharge capacity is much lower than the theoretical capacity. Based on the facts above, we conclude that the LTO-limited cells have better overcharge tolerance than the LNMO-limited cell.

4. Conclusions

The LTO anode was combined with the LNMO cathode to assemble the 3 V LNMO/LTO cell system, and then two kinds of cell systems whose capacities are limited by the LTO anode and the LNMO cathode were compared. The results indicate that the cell limited by LTO exhibits better cycling performance and less limitation to applicable electrolytes than that limited by LNMO cathode. By taking advantage of the extra capacity related to lithium inserting Li₇Ti₅O₁₂ and the order/disorder transition, the LTO-limited cell has better tolerance to high voltage than the LNMO-limited cell. In addition, it is found that the LTO-limited cell is easier to encounter overcharge problem than the state-of-the-art cell system with carbonaceous anode, because of the 1.5 V plateau of LTO that needs a much higher potential on the cathode during the overcharge process. Fortunately, the LTO-limited cell shows much better overcharge tolerance than the LNMO-limited cell. Thus, the LNMO/LTO cell system can extend the application of lithium-ion batteries under the condition that the cell capacity is limited by the LTO anode.

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