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Mixed salts for lithium iron phosphate-based batteries operated at wide temperature range

Zhi-an ZHANG, Xing-xing ZHAO, Bo PENG, Yan-qing LAI, Zhi-yong ZHANG, Jie LI

School of Metallurgy and Environment, Central South University, Changsha 410083, China

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Abstract: To achieve good performance for $LiFePO_4$-based batteries operated at a wide temperature range, mixed salts of $LiBF_4$−LiBOB (lithium bis(oxalato)borate) and LiTFSI (lithium bis(trifluoromethanesulfonyl)imide)−LiBOB were investigated as alternative lithium salts to $LiPF_6$ in non-aqueous electrolytes. $LiFePO_4$/Li half cells using $LiPF_6$, $LiBF_4$−LiBOB and LiTFSI−LiBOB slats as lithium salts were investigated by galvanostatic cycling, cyclic voltammetry, thermogravimetric analysis. The results show that $LiBF_4$−LiBOB and LiTFSI−LiBOB mixed salts are much more thermally stable than $LiPF_6$. Corrosion of Al foil in the LiTFSI-based electrolytes can be suppressed successfully by the addition of LiBOB as a co-salt. The electrochemical performance of $LiBF_4$−LiBOB and LiTFSI−LiBOB mixed salts based cells are both better than that of $LiPF_6$-based cell. LiTFSI−LiBOB mixed salt based electrolyte is considered to be a very promising electrolyte candidate for Li-ion batteries that will be used in wide-temperature applications.

Key words: lithium iron phosphate-based batteries; lithium salts; $LiBF_4$−LiBOB; LiTFSI−LiBOB; wide temperature range

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1 Introduction

Lithium ion batteries have attracted significant attention in modern electrochemistry and are widely used in many applications, such as electric vehicle, aviation and spaceflight. However, the narrow operating temperature range of the state-of-art systems is one of the technical barriers associated with their use in plug-in-hybrid-vehicles (PHEVs), which has been identified by the Department of Energy (DoE) of China [1,2].

Electrolytes are often considered the key components to improve the temperature performance of lithium ion batteries. $LiPF_6$ is the most common lithium salt used in commercial Li-ion batteries, however, it is moisture sensitive and thermally unstable [3]. $LiPF_6$ will react with trace amount of water ($LiPF_6 + H_2O \rightarrow 2HF + LiF + POF_3$) and decompose into LiF and PF_5 in battery electrolyte solutions ($LiPF_6 \rightarrow LiF + PF_5$), which will be accelerated at elevated temperatures. The products generated from the side reactions corrode the solid electrolyte interphase (SEI) layers on anodes and react with EC-containing solvents. The series reactions generated from $LiPF_6$ could cause the following effects: 1) Losing charge carriers due to Li^+ entrapment in an insoluble salt, LiF; 2) Increasing solvent viscosity due to the formation of oligomeric compounds from reaction between solvent and PF_5; 3) Increasing cell resistance that may be due to the precipitation of polymer and insoluble salt on battery component surfaces; 4) Solid electrolyte interphase (SEI) layers damage due to HF corrodes. All the effects lead to capacity fading and poor cycle life [3,4]. Therefore, it is important to find alternative lithium salts or salt mixtures that are stable both against water and elevated temperatures, while still providing good low temperature performance.

Significant efforts have been made in the past to develop alternative salts, such as $LiClO_4$ [5], $LiBF_4$ [6], $LiN(SO_2CF_3)_2$ [7], $LiN(SO_2F)_2$ [8], LiODFB [9] and $LiAsF_6$ [10]. $LiBF_4$ is more thermally stable and less sensitivity to water than $LiPF_6$. It was reported that a Li-ion cell with a $LiBF_4$-based electrolyte has excellent low temperature performance, in addition to having better high temperature performance. This is because at low temperature, the $LiBF_4$ electrolyte has low viscosity and more importantly such a cell presents lower charge-transfer resistance. However, the application of $LiBF_4$ in Li-ion cells is limited mainly by its poor ability

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Corresponding author: Zhi-an ZHANG; Fax/Tel: +86-731-88830649; E-mail: zza75@163.com

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in facilitating formation of a stable solid electrolyte interface (SEI). Lithium bis (trifluoromethanesulfonyl) imide [LiTFSI, $LiN(CF_3SO_2)_2$] is also a promising alternative electrolyte salt to replace the classical $LiPF_6$ owing to its higher thermal stability and better tolerance to water [11]. So far, LiTFSI has not been used in commercialized rechargeable lithium ion cells owing to the strong corrosion behavior at the Al current collector. In this sense, the suppression of the corrosion of this salt to the Al current collector is of great significance for its successful applications. MATSUMOTO et al [11] found that Al corrosion was prevented at higher LiTFSI concentrations due to a passivation film composed of LiF formed on the Al surface. The addition of $LiPF_6$ also could lead to the formation of AlF_3-containing passivation layers [12]. Effective passivation films on Al foil have also been reported when LiBOB [lithium bis(oxalato)borate, $LiB(C_2O_4)_2$] is added to the electrolytes [4]. Unlike LiTFSI, lithium bis-oxalato borate (LiBOB) has been investigated for more than 10 years and its good passivating properties are well recognized. It also has the ability to form a stable solid-electrolyte interface.

In this work, mixed salts of $LiBF_4$−LiBOB and LiTFSI−LiBOB were studied. As LiBOB has the good SEI forming properties and passivating properties, it will cover the shortage of $LiBF_4$ and LiTFSI, so we speculate that an optimized electrolyte can be formulated for the $LiFePO_4$ cathode by using a $LiBF_4$−LiBOB and LiTFSI−LiBOB salt mixture. Based on our previous work [13], a mixed solvent of PC/EC/EMC was employed to formulate the electrolyte that enabled $LiFePO_4$ cathode to operate in a wide temperature range.

2 Experimental

2.1 Electrochemical measurements of $LiFePO_4$-based batteries

Battery-grade $LiBF_4$, LiBOB, EC, PC and EMC were purchased from Guangzhou Tinci Materials Technology Co., Ltd. (China). LiTFSI was purchased from Sigma-Aldrich (99.95% trace metals basis). All liquid reagents were dried using 4 Å molecular sieves. In an argon-filled glove box (Universal 2440/750, Mikrouna Mech. Tech. Co., Ltd.) with both oxygen and water contents less than 1×10^{-5}, electrolyte solutions were prepared by adding the lithium salt mixture to the PC/EC/EMC (1:1:3 V/V) mixture. Water and free acid contents in these electrolytes were controlled below 2×10^{-5}.

The $LiFePO_4$ cathodes containing 80% $LiFePO_4$, 10% carbon black and 10% PVDF (mass fraction), were fabricated by coating the slurry of $LiFePO_4$ active material, carbon black, and PVDF on aluminum foil collector. To evaluate the electrochemical behavior of these electrolytes in $LiFePO_4$/Li half cells, 2025-coin type cells with lithium foil as counter and reference electrodes were assembled in the glove box using Celgard® 2400 membrane as the separator and filled with these liquid electrolytes.

A high−low temperature test-chamber (GDH-2005C) was used to provide a constant temperature environment for test. The charge/discharge performance of $LiFePO_4$/Li half cells were evaluated on a Land LAND CT2001A charge−discharge system. The half cells were cycled 3 times at 0.1*C* (2.5−4.2 V) at room temperature (25 °C) to obtain a stable state before other evaluations. Then the charge/discharge capacity, electrochemical impedance spectroscopy (EIS) and other tests at different temperatures were conducted. In order to reach equilibrium of the cell temperature and the oven preset temperature for evaluations at different temperatures, the cells were kept at the specified temperature for at least 4 h.

2.2 Characterization of electrodes

Thermogravimetric analysis (TGA) of $LiPF_6$, $LiBF_4$−LiBOB and LiTFSI−LiBOB was measured on SDT-Q600 apparatus in the temperature range of 25 to 450 °C at a heating rate of 10 °C/min in air. Solartron 1470E Multi-Channel potentiostats was employed to determine Al passivation behavior and to measure EIS of the half cells. AC impedances of the cells were measured by applying a DC bias with its value equal to open circuit voltage (OCV) of the cell and an AC oscillation of 10 mV over the frequencies from 100 kHz to 0.01 Hz. The obtained EIS spectra were fitted by using ZView software (Scribner and Associates).

3 Results and discussion

3.1 Thermal stability of $LiPF_6$, $LiBF_4$−LiBOB and LiTFSI−LiBOB slats

In order to investigate the thermal stability of $LiPF_6$, $LiBF_4$−LiBOB and LiTFSI−LiBOB slats, thermo gravimetric analysis was conducted in the temperature range of 25 to 450 °C. Figure 1 displays the TGA profiles of the three kinds of salts. $LiPF_6$ salt got a 10% mass loss from 70 °C to 80 °C and its mass decreased sharply when temperature was above 150 °C. It indicated that $LiPF_6$ started to decompose at 70 °C and completely decomposed when temperature was above 200 °C, which was consistent with the result of SLOOP's research [14]. The LiTFSI−LiBOB mixed salt did not get any mass change until the temperature was up to 300 °C. It started to decompose at 300 °C and completely decomposed when temperature was above 450 °C. As to $LiBF_4$−LiBOB slat mixture, it kept stabilization till to 250 °C and got completely decomposed above 250 °C. Comparing the TGA curves of LiTFSI−LiBOB and $LiBF_4$−LiBOB mixtures, it can be easily found that both mixtures had a mass loss between 300 °C and 350 °C. The thermal stability followed: $LiPF_6$<$LiBF_4$−LiBOB< LiTFSI−LiBOB.

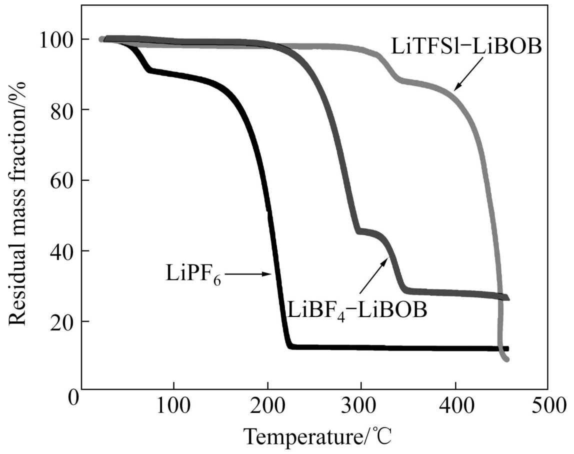

Fig. 1 TGA profiles of three kinds of salts

3.2 Corrosion behaviors of electrolytes with $LiPF_6$, LiTFSI−LiBOB and $LiBF_4$−LiBOB salts on aluminum foil

The corrosion behaviors of electrolytes with $LiPF_6$, LiTFSI−LiBOB and $LiBF_4$−LiBOB salts on aluminum foil are exhibited in Fig. 2, respectively. Figure 2(a) shows that a slow increase of current occurred at 3.25 V and 4.5 V (vs Li^+/Li) in the first positive sweep for $LiPF_6$-based electrolyte. The curve of $LiPF_6$-based electrolyte show more than one peak during the positive sweep, which indicated that the electrolyte decomposed and the products reacted with aluminum [15]. Figures 2(b) and (c) showed that a slow increase of current occurred at 4.0 V and 4.25 V (vs Li^+/Li) in the first positive sweep for $LiBF_4$−LiBOB−based and LiTFSI−LiBOB-based electrolyte, respectively. The corrosion current density peak of LiTFSI−LiBOB-based electrolyte was about 15 μA/cm^2, which was much smaller than the result of 0.1 mA/cm^2 in Ref. [16]. The peak height got lower and the oxidation peak occurred at higher potential in the second and third cycle for all the electrolytes with different salts, which indicated that aluminum trended to react with the lithium salts contained electrolytes, resulting in the formation of a thin film on the electrode surface to protect aluminum from further corrosion. As shown in Fig. 2, in the $LiFePO_4$ cathode operating window, i.e., 2.5−4.2 V, the corrosion current density was approximately 0 μA/cm^2 in the third cycle for all the electrolytes. So it can be concluded that LiTFSI−LiBOB and $LiBF_4$−LiBOB mixtures have good Al passivation behavior and are suitable as lithium salts of lithium battery. The result also confirms the positive effect of LiBOB on suppressing the corrosion of Al in LiTFSI-based electrolytes.

Figure 3 compares the capacity retention of the Li/$LiFePO_4$ cells with different electrolytes when discharged at 0.1*C*, at 0 °C, −20 °C and −40 °C respectively, in which the capacity retention is expressed as the ratio of discharge capacity at a specific temperature to that at 25 °C. A general trend is that the discharge capacity is reduced with the decrease of the temperature. This phenomenon is attributed to the

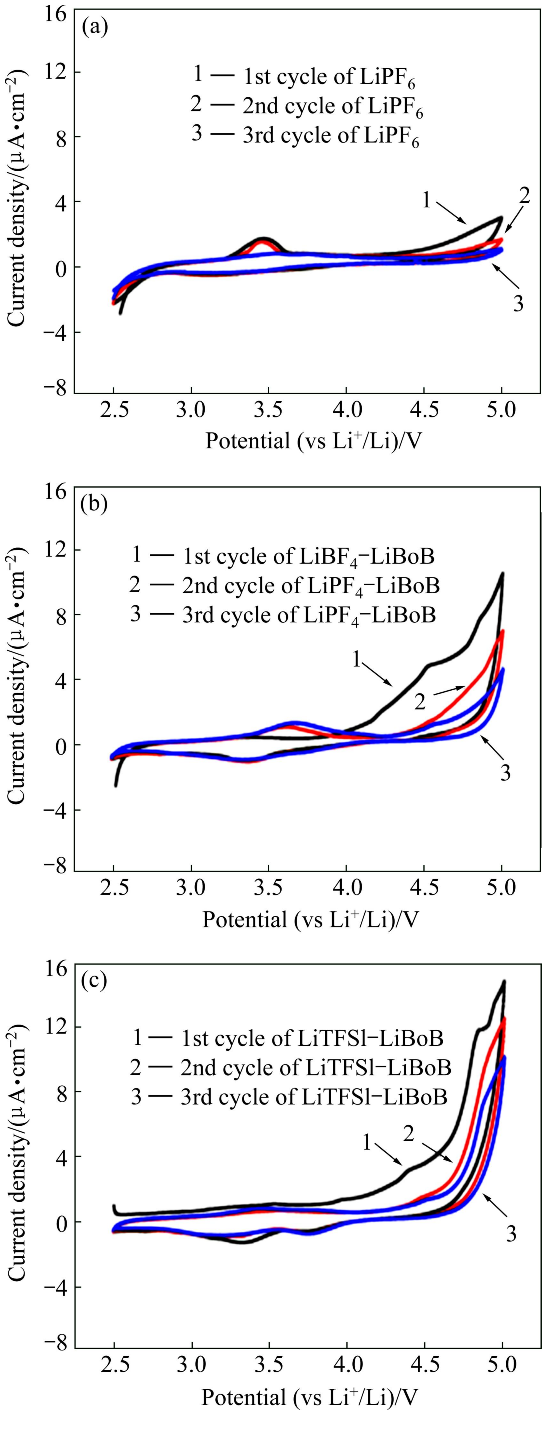

Fig. 2 Corrosion behaviors of electrolytes with different slats: (a) $LiPF_6$; (b) LiTFSI−LiBOB; (c) $LiBF_4$−LiBOB

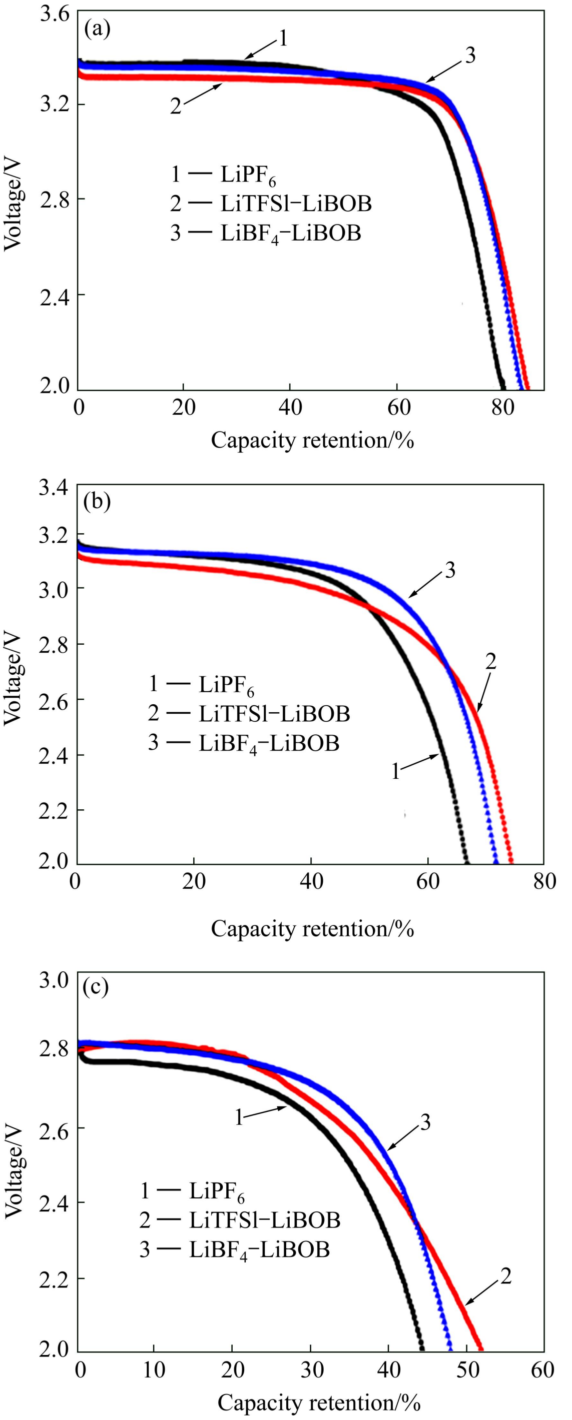

Fig. 3 Capacity retention of Li/LiFePO$_4$ cells with different electrolytes at low temperature: (a) 0 °C; (b) −20 °C; (c) −40 °C

decreased ionic conductivity of the electrolyte and to the slowed-down kinetics of cell reactions, which are suffered at low temperatures [17]. As shown on the discharge curves, cells with LiPF$_6$ based electrolyte provide ~80% of the room temperature capacity at 0 °C, ~66% and 45% at −20 °C and −40 °C respectively, similar to the research by SMART et al [18] and a little better than the work of LIAO et al [19]. LiBF$_4$−LiBOB and LiTFSI−LiBOB mixture salts containing cells have a better performance at low temperature than that of LiPF$_6$ salt. Cells with LiTFSI−LiBOB mixture salt exhibit the best low temperature performance, which provide approximately 85% of the room temperature capacity at 0 °C, 75% at −20 °C and 52% at −40 °C. It indicates that LiTFSI−LiBOB mixture salt has optimized performance at low temperature.

3.3 Electrochemieal measurements

Cycling performance of the Li/LiFePO$_4$ cells at room temperature was evaluated, and the results are shown in Fig. 4. It can be seen from Fig. 4 that cells with mixed salts can deliver a capacity around 130 mA·h/g at the initial cycle, which is similar to the cell with LiPF$_6$ salt. The LiTFSI−LiBOB and LiBF$_4$−LiBOB mixed salt can effectively stabilize the Li/LiFePO$_4$ cells for 100 cycles with nearly no capacity loss at room temperature, while cells with LiPF$_6$ salt lose 4.1% of initial capacity. The result indicates that the cells with LiTFSI−LiBOB and LiBF$_4$−LiBOB mixed salt have better cycle performance than that with LiPF$_6$. In the LiPF$_6$-based electrolyte, the capacity degradation is caused mainly by the dissolution of iron from the cathode active material into the electrolyte. This occurs because the formation of corrosive HF is almost unavoidable in LiPF$_6$-based electrolytes due to the hydrolysis decomposition of LiPF$_6$ in the presence of residual water as an impurity in the electrolytes [16]. As to the cells with LiBF$_4$−LiBOB-based electrolyte, LiBOB can substantially suppress the dissolution of Fe(II) ions from LiFePO$_4$ [26], and in the cells with LiTFSI-based electrolyte, there is no HF generated because LiTFSI has high thermal stability and good tolerance to water.

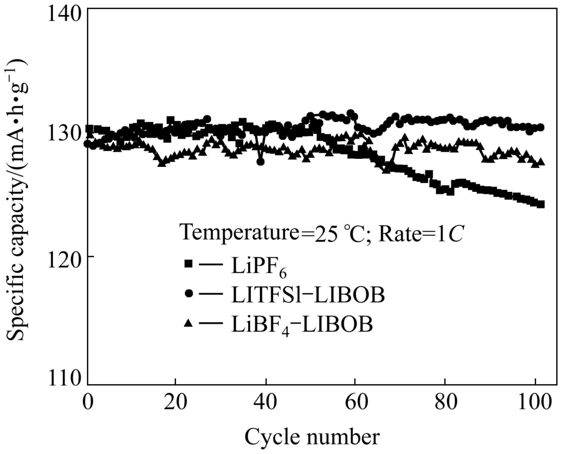

Fig. 4 Cycling performance of Li/LiFePO$_4$ cells with different electrolytes at room temperature

Cycling performance of the Li/LiFePO$_4$ cells at high temperature was also measured. After cycling 3 times at 0.1*C* at room temperature (25 °C) to make sure solid electrolyte inter-phase (SEI) films are formed stably,

$LiFePO_4$/Li cells were cycled at 1*C* at 65 °C for 100 times. The discharge capacity results are summarized in Fig. 5, in which a difference of the cycling behaviour in the electrolytes containing different lithium salts is observed. The discharge capacity at 65 °C is a little higher than that at 25 °C, due to the higher conductivity at high temperature. Cells with LiTFSI−LiBOB and $LiBF_4$−LiBOB mixed salts have relatively higher capacity compared with that with $LiPF_6$ salt. The cells with all the three kinds of salts cycle well with a limited capacity fade after 100 cycles. Cells with $LiPF_6$ based electrolyte have capacity retention of 79.3% after cycle, which is similar to the results of ZHOU et al [15]. Li/$LiFePO_4$ cells containing LiTFSI−LiBOB and $LiBF_4$−LiBOB mixed salt keep 85.1% and 87.5% of capacity retention respectively, which are considerably higher than that obtained in the $LiPF_6$ based cells. It seems that the better thermal stability of LiTFSI−LiBOB and $LiBF_4$−LiBOB mixture salts plays an important role in improving the high-temperature resilience of $LiFePO_4$ electrode.

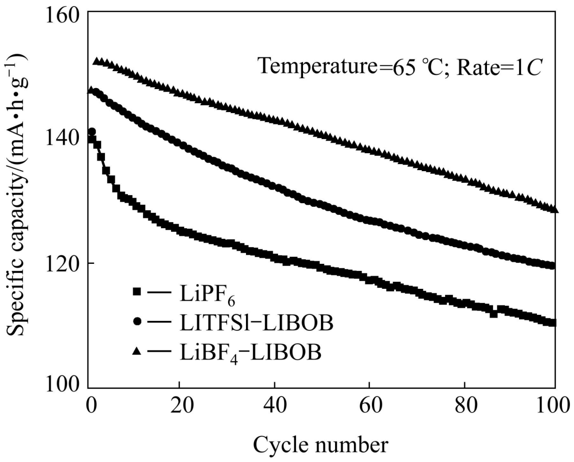

Fig. 5 Cycling performance of Li/$LiFePO_4$ cells with different electrolytes at high temperature

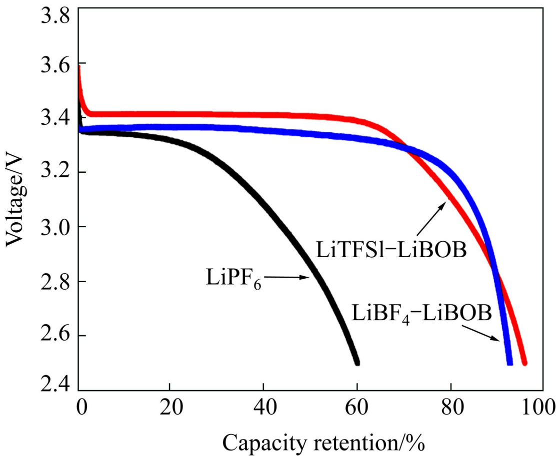

Fig. 6 Discharge curves of Li/$LiFePO_4$ cells with different electrolytes after storage at high temperature

Figure 6 shows the discharge curves of Li/$LiFePO_4$ cells after storage at high temperature. After cycling 3 times at 0.1*C* at room temperature (25 °C) to make sure solid electrolyte inter-phase (SEI) films are formed stably, Li/$LiFePO_4$ cells were fully charged and then stored at 65 °C for 7 d. The discharge experiment was conducted at 25 °C and 0.1*C*. The capacity retention is expressed as the ratio of discharge capacity to charge capacity before storage. As illustrated in Fig. 6, the capacity retention of $LiPF_6$ based cells is 60%, which is corresponded to the result of our previous research [20]. The capacity retention of LiTFSI−LiBOB and $LiBF_4$-LiBOB based cells are 96% and 92%, which are considerably higher than that obtained in the $LiPF_6$-based cells. Furthermore, the discharge voltage platform of LiTFSI−LiBOB based cell is a little higher than that of $LiPF_6$ and $LiBF_4$−LiBOB based cells. These results can be easily ascribed to the enhancing thermal stability of LiTFSI−LiBOB and $LiBF_4$−LiBOB mixture salts.

4 Conclusions

1) $LiBF_4$−LiBOB and LiTFSI−LiBOB mixed salts were investigated as an alternative lithium salt to $LiPF_6$ in electrolyte for Li-ion cells operated at a wide temperature range.

2) Thermogravimetric analysis shows that $LiBF_4$−LiBOB and LiTFSI−LiBOB mixed salts are thermally much more stable than $LiPF_6$.

3) Corrosion of Al foil in the LiTFSI-based electrolytes can be suppressed successfully by the addition of LiBOB as a co-salt.

4) The electrochemical performances of $LiBF_4$−LiBOB and LiTFSI−LiBOB mixed salts based cells are both better than that of $LiPF_6$ based cell. Li/$LiFePO_4$ cell with LiTFSI−LiBOB mixed salt is able to provide approximately 85% of the room temperature capacity at 0 °C, 75% at −20 °C and 52% at −40 °C, after being cycled 100 times with nearly no capacity loss at 25 °C and keep 96% capacity after 7 d storage at 65 °C.

5) The good low temperature performance and excellent high-temperature stability make the LiTFSI−LiBOB mixed salt based electrolyte a very promising electrolyte candidate for Li-ion batteries that will be used in wide-temperature applications.

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基于复合锂盐的宽温度磷酸铁锂电池的性能

张治安，赵星星，彭 波，赖延清，章智勇，李 劼

中南大学 冶金与环境学院，长沙 410083

摘 要：为了提高磷酸铁锂($LiFePO_4$)电池在宽温度范围内的性能，研究 $LiBF_4$−LiBOB 复合盐和 LiTFSI−LiBOB 复合盐用于替代 $LiPF_6$ 作为非水电解质的锂盐。分别通过恒流充放电测试、循环伏安测试和热重分析法研究使用 $LiPF_6$, $LiBF_4$−LiBOB 和 LiTFSI−LiBOB 作为锂盐的 $LiFePO_4$/Li 半电池的性能。结果显示：$LiBF_4$−LiBOB 和 LiTFSI−LiBOB 复合盐的热稳定性高于 $LiPF_6$；在 LiTFSI 基电解液中添加 LiBOB 可以提高铝箔的抗腐蚀性；使用 $LiBF_4$−LiBOB 和 LiTFSI−LiBOB 复合盐的电池电化学性能相对于使用 $LiPF_6$ 的电池有明显改善。因此 $LiBF_4$−LiBOB 和 LiTFSI−LiBOB 复合盐电解液可以用来改善锂电池宽温度范围下的性能。

关键词：磷酸铁锂电池；锂盐；$LiBF_4$−LiBOB；LiTFSI−LiBOB；宽温度性能

(Edited by Yun-bin HE)