Enhanced Performance of Li|LiFePO₄ Cells Using CsPF₆ as an Electrolyte Additive

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Abstract

The practical application of lithium (Li) metal anode in rechargeable Li batteries is hindered by both the growth of Li dendrites and the low Coulombic efficiency (CE) during repeated charge/discharge cycles. Recently, we have discovered that CsPF₆ as

an electrolyte additive can significantly suppress Li dendrite growth and lead to highly compacted and well aligned Li nanorod structure during Li deposition on copper substrates. In this paper, the effect of CsPF₆ additive on the performance of rechargeable Li metal batteries with lithium iron phosphate (LFP) cathode is further studied. Li|LFP coin cells with CsPF₆ additive in electrolytes show well protected Li anode surface, decreased resistance, enhanced rate capability and extended cycling stability. In Li|LFP cells, the electrolyte with CsPF₆ additive shows excellent long-term cycling stability (at least 500 cycles) at a charge current density of 0.5 mA cm⁻² without internal short circuit. At high charge current densities, the effect of CsPF₆ additive becomes less significant. Future work needs to be done to protect Li metal anode, especially at high charge current densities and for long cycle life.

Keywords: Lithium metal battery; Lithium dendrite suppression; Lithium protection; CsPF₆ additive; Li|LiFePO₄ cells

1. Introduction

The energy densities of the state-of-the-art lithium (Li)-ion batteries still cannot meet the increasing demand of the portable electronic devices and long range electric vehicles. Therefore, there is an increasing need on the alternative electrochemical cells with high-capacity electrode materials and high cell voltages. This is one reason that Li metal anode has been extensively reinvestigated in recent years after it has been replaced by graphite-based anodes since early 1990s [1-3]. Li metal has an

extremely high theoretical capacity (3860 mAh g^{-1}), the lowest negative electrode potential (-3.040 V vs. standard hydrogen electrode), and has been widely used as the anodes in the investigation of the next generation rechargeable batteries, such as Li-sulfur batteries [4, 5] and Li-air batteries [6, 7]. In addition, rechargeable Li metal batteries using intercalation compounds or high-voltage conversion compounds as cathode materials could be another alternative approach to meet the increasing need of the electrochemical energy storage systems with high energy densities [8].

The impacts of using Li anode to replace graphite in Li-ion batteries have been analyzed by Gallagher et al. recently [9]. For instance, batteries with Li metal anode and Li-/manganese-rich nickel-manganese-cobalt (LMRNC) cathode has a theoretical energy density of ca. 900 Wh Kg^{-1} , which is more than twice that of Li-ion batteries with graphite anode and NMC cathode (ca. 400 Wh Kg^{-1}) [9]. However, the practical application of Li metal anode in rechargeable Li batteries is hindered by both the growth of Li dendrites and the low Coulombic efficiency during repeated charge/discharge cycles. Although the low Coulombic efficiency in Li metal batteries can be partially compensated by the use of excess amount of Li metal, the dendrite-growth related safety issues are the major concerns of rechargeable Li metal batteries. Most solutions for Li dendrite prevention focus on improving the stability and uniformity of the solid electrolyte interphase (SEI) layer on the Li surface by adjusting electrolyte components and optimizing SEI formation additives [10-12 and references therewith]. Using mechanical barriers to block dendrite penetration and adding a second metal cation in electrolytes to form alloys or mixed metal deposits

during Li deposition are other two promising approaches [12 and references therewith]. Recently, we have found that CsPF_6 as an additive in the LiPF_6 -based electrolytes can significantly suppress Li dendrite growth and lead to smooth, highly compacted and well aligned Li nanorod structures during Li deposition on copper substrates [13-15]. In the present work, we used CsPF_6 as the electrolyte additive to improve the performance of Li metal batteries with lithium iron phosphate (LFP) as cathode. LFP is chosen because it exhibits excellent long-term cycling stability in Li-ion batteries [16]. The effect of CsPF_6 on suppressing Li dendrite formation and improving the cycling performance of Li|LFP cells has been demonstrated.

2. Experimental

Lithium hexafluorophosphate (LiPF_6), propylene carbonate (PC), ethylene carbonate (EC), ethyl methyl carbonate (EMC) were purchased in battery grade from BASF Battery Materials and were used as received. Cesium hexafluorophosphate (CsPF_6 , $\geq 99.0\%$, anhydrous) was ordered from SynQuest Laboratories and was dried at 60°C for four days under vacuum inside the antechamber of an argon-filled MBraun glove box. The electrolytes of 1 mol L^{-1} LiPF_6 in a mixture of EC, PC and EMC at a weight ratio of 5:2:3 without and with 0.05 mol L^{-1} CsPF_6 were prepared in the argon-filled MBraun glove box and denoted as E1 and E1Cs, respectively. For comparison, a conventional electrolyte used in state-of-the-art Li-ion batteries, 1.0 mol L^{-1} LiPF_6 in a mixture of EC and EMC at a volume ratio of 3:7 was also prepared and marked as E2.

The laminated lithium iron phosphate (LFP) electrodes were made of LFP powder (Süd-Chemie AG), Super P carbon (Timcal) and polytetrafluoroethylene (PTFE, from DuPont) in the weight ratio of 8:1:1, where the LFP powder and Super P carbon were first mixed well on a planetary miller before adding the PTFE binder. The typical active material loading of the LFP electrodes was around 2.4 mAh cm^{-2} . After drying at 80°C under vacuum, the LFP electrodes were punched into disks in the diameter of 1.4 cm. The experimental Li|LFP batteries were assembled in the argon-filled MBraun glove box by crimping an LFP electrode disk, a Celgard polyethylene separator, a Li metal chip (99.9%, with dimension of 15.6 mm in diameter and 0.45 mm in thickness, from MTI Corporation) and an 80 μL electrolyte into a CR2032 coin cell with aluminum-clad can (MTI Corporation).

The cycling performance and rate capability of Li|LFP coin cells were measured at room temperature using an Arbin BT-2000 battery tester (Arbin Instruments). All coin cells were first conducted two formation cycles between 2.8 and 3.9 V vs. Li/Li^+ at a charge and discharge current density of 0.24 mA cm^{-2} (i.e. C/10 rate). The electrochemical impedance spectroscopy (EIS) of fresh and cycled cells was measured at open circuit potential (OCP) with a 10 mV perturbation in the frequency range of 10^6 - 10^{-3} Hz on a Solartron Electrochemical Station SI-1287 coupled with a Solartron Frequency Analyzer SI-1255.

The cycled Li anode electrodes were obtained from disassembled cells, washed three times with anhydrous EMC, and dried in the antechamber of the MBraun glove box under vacuum. The Li electrodes were transferred in airtight containers filled with

argon to a nitrogen-filled glove box for scanning electron microscope (SEM) studies. SEM images of the surface and the cross section of cycled Li electrodes were taken on a JEOL 5900 scanning electron microscope.

3. Results and discussion

The first cycle voltage profiles of the Li|LFP cells with the three electrolytes (E1Cs, E1 and E2) investigated in this work are compared in Figure 1. All three electrolytes exhibit similar charge and discharge capacities ($143\text{-}144\text{ mAh g}^{-1}$ for charge and $141\text{-}143\text{ mAh g}^{-1}$ for discharge) and their first cycle efficiencies are in the range of 98-99%. The minor difference among the samples is that the sample containing conventional $\text{LiPF}_6/\text{EC-EMC}$ electrolyte (E2) has slightly higher overvoltage than those containing $\text{LiPF}_6/\text{EC-PC-EMC}$ electrolytes without or with CsPF_6 additive (E1 and E1Cs), meaning the voltage polarization of Li|LFP cells slightly decreases in the order of E2, E1 and E1Cs. It is indicated that the electrolyte formulations have slight effect on the cell polarizations.

Figure 2 shows the rate capability of the Li|LFP cells with the three electrolytes, where the charge was conducted at C/5 rate (i.e. 0.48 mA cm^{-2}) while the discharge was carried out at different rates from C/5 to 5C (i.e. 12.0 mA cm^{-2}). It is found that Li|LFP cells with all three electrolytes have very similar rate capability at the discharge current density lower than 1C rate (i.e. 2.4 mA cm^{-2}). However, with the increase of the discharge rate from 1C to 2C (i.e. 4.8 mA cm^{-2}) and even to 5C, the Li|LFP cell with CsPF_6 -containing electrolyte (E1Cs) demonstrates much higher

capacity than those with the two control electrolytes without CsPF₆ additive. This result is consistent with the reduced voltage polarization in the samples with CsPF₆ additive as shown in Fig. 1. It is also consistent with the rate performance of the Li|Li₄Ti₅O₁₂ cells containing the LiPF₆-PC electrolytes with and without CsPF₆ additive as shown in the Supplemental figure of ref. [14]. This is due to the suppressed Li dendrite growth via CsPF₆ as reported in our previous works [13-15].

Figure 3 shows the cycling stability of Li|LFP cells with the three electrolytes at a charge current density of 1.6 mA cm⁻² (i.e. 2C/3 rate) and discharge current density of 2.4 mA cm⁻² (i.e. 1C rate) after two formation cycles at C/10 rate. The capacities of Li|LFP cells start to decrease after 50, 78 and 100 cycles for the electrolytes E2, E1 and E1Cs, respectively, and the related capacity retention at the 100th cycle are 54% for E2, 72% for E1 and 85% for E1Cs. The capacity fading speed is fast for the electrolytes without CsPF₆ additive. The cells with the CsPF₆-containing electrolyte E1Cs show relatively slower capacity fade, which is mainly from the protection of Cs⁺ on the Li metal anode. The electrolyte E1 shows slightly better cycling stability than the conventional electrolyte E2. It is probably beneficial from the much higher concentration of solvent EC in E1 (44.1wt%) than in E2 (32.1wt%) because EC forms good SEI layers on graphite and Li anode surfaces and protects further decompositions of electrolytes on these anode surfaces [10 and references therewith].

Furthermore, the cycling stability of Li|LFP cells with the three electrolytes at other charge current densities (from 0.48 to 1.2 mA cm⁻² (i.e. C/5 to C/2 rate)) was also investigated. The same discharge current density of 2.4 mA cm⁻² (i.e. 1C rate)

was used in these investigations. To make the figures simple, we normalized the discharge capacity at the 100th cycle at different charge rates over the discharge capacity at the first formation cycle (at C/10 rate) as the nominal capacity, and compare the nominal capacities at the 100th cycle for Li|LFP cells containing the three electrolytes at different charge current densities (from 0.48 to 1.6 mA cm⁻²) in Figure 4. For the conventional LiPF₆/EC-EMC electrolyte E2, the nominal capacity at the 100th cycle drops sharply when the charge current density is increased from 0.8 to 1.6 mA cm⁻², indicating the poor cycling stability of this electrolyte at high charge current densities. The electrolyte E1 also shows a quick fade of the nominal capacity at the 100th cycle when the charge current density increases from 0.80 to 1.20 mA cm⁻², but it seems to have similar cycling stability in the charge current density range from 1.2 to 1.6 mA cm⁻². The reason is probably associated with the good SEI layer from the high EC content in this electrolyte. After CsPF₆ is added into the E1 electrolyte, the E1Cs electrolyte exhibits much higher nominal capacities than the E1 electrolyte without CsPF₆ at almost all charge current densities. The E1Cs electrolyte also shows similar nominal capacities in the charge current density range from 0.8 to 1.6 mA cm⁻², suggesting the notable effect of CsPF₆ additive on the Li surface protection at different charge current densities.

Li metal has been extensively used as the counter and reference anode in half cells on the performance studies of electrode materials for Li-ion batteries. The electrochemical polarization of Li metal anode electrodes in half cells is usually ignored due to the excess amount of Li metal and the relatively low mass loading of

studied electrode materials. However, in practical rechargeable Li metal batteries with high-loading sulfur, oxygen or Li-intercalation materials as cathode, the repeated Li dendrite growth and consequent polarization of Li metal anode must be considered, especially at high charge current density during long-term cycling [17]. Recently, Lv et al. studied the failure mechanism of fast-charged Li metal batteries with carbonate-based electrolytes and found that the quick capacity fade at high current densities is due to the fast formation of a highly resistant SEI layer on Li metal surface causing the dramatically increase in cell impedance [17]. This is easily understandable. During charging process, Li^+ in the electrolyte passes through the ion-conductive SEI layer on Li surface, gets the electron, reduces to Li metal and deposits on Li anode beneath the SEI layer. At a low charge current density, the Li deposition speed is slow and the original SEI layer on Li surface will be pushed up gently and slowly so it will not break, thus leading to a higher Coulombic efficiency and a longer cycle life of the battery. However, at a high charge current density, a lot of Li is deposited beneath the original SEI layer in a short time and at an uneven pace (due to the non-uniformly distributed electric field on the electrode surface), the old SEI layer is easily broken, then more fresh and porous-structured Li is exposed to the electrolyte and new SEI layers form via the reactions between Li and the electrolyte, resulting in a higher cell resistance. With cycling, more and thicker SEI layers will build up on Li anode leading to a significant increase in cell resistance, and some of the deposits will form the so-called “dead” Li and cannot be electrochemically utilized, therefore, fast capacity decay and cell failure are observed. This behavior can

be demonstrated by the microscopic morphologies of the Li anodes and the impedance changes of the Li|LFP batteries.

Figure 5 shows the SEM morphologies of the surface and cross-section views of cycled Li electrodes in the three electrolytes with and without CsPF₆ additive after 150 cycles at 1.2 mA cm⁻². Although no dendrite on Li surface is observed for all the three samples, the surface of the Li electrode cycled in electrolyte with CsPF₆ additive (i.e. E1Cs, see Figure 5c) is much smoother than the other two in the electrolytes without CsPF₆ additive (i.e. E2 and E1, see Figures 5a-b). In the electrolytes without CsPF₆, the cross-section images of the cycled Li electrodes (Figures 5d-e) clearly show the severe corrosions of Li metals and the formation of porous Li layers due to the reactions of Li metal with electrolytes and the growth of SEI layers. In contrary, the cross-section image of Li cycled in E1Cs electrolyte exhibits much lesser corrosion and thinner SEI layer (Figure 5f), demonstrating less SEI formation and well protection of Li metal anode by the Cs⁺ additive in Li|LFP coin cells. This is consistent with what we previously reported about the dendrite prevention and smooth Li deposition in CsPF₆-containing electrolytes [13-15].

Figure 6 compares the EIS curves of Li|LFP cells containing the three electrolytes with and without CsPF₆ additive before and after 200 cycles at a charge current density of 1.6 mA cm⁻². After 200 cycles, the cells containing the electrolyte with CsPF₆ additive, i.e. E1Cs in Figure 6c, have much lower cell resistance than the cells containing the electrolytes without CsPF₆ additive (Figures 6a for E2 and 6b for E1) demonstrating that CsPF₆ additive can protect the Li metal anode. It is interesting to

note that the cells with E1Cs electrolyte show even reduced total impedance after long-term cycling compared to the impedance before cycling. This indicates that the Cs^+ -induced SEI layer has a higher ionic conductivity as compared to those without Cs^+ additive. The trend of the cell impedance change in Li|LFP coin cells with E1Cs electrolyte is different from that in Li|Cu beaker cells in our previous work [15], which is probably because of the differences in cell protocols, substrates on which Li is deposited, the electrolyte solvent systems, and the electrolyte amounts in the two studies.

The impedance at the high frequency end (i.e. the left side) of the semicircle of the a.c. impedance curve is for the resistance of the bulk electrolyte in the cells. After 200 cycles at such a high charge current density, all the three electrolytes lead to an increase in the impedance of the bulk electrolyte, which should be caused by the consumption of electrolyte via chemical reactions with Li and electrochemical decompositions. For the electrolyte E2, though it leads the cells to have much lower total resistance than the electrolytes E1 and E1Cs before high rate cycling (i.e. the curves in red color for the after formation), it results in significantly increased bulk electrolyte resistance and consequently high cell impedance after 200 cycles at such a high charge current density, indicating the continuous reactions of this electrolyte with Li metal during cycling. Meanwhile, the cells containing the electrolyte with CsPF_6 additive, i.e. E1Cs in Figure 6c, have the lowest bulk electrolyte resistance after 200 cycles among all the cells with and without CsPF_6 as an additive demonstrating that CsPF_6 additive can protect the Li metal anode further.

In the previous work, when we studied the effect of CsPF₆ additive on the suppression of Li dendrite formation during Li deposition in beaker cells (meaning no pressure effect) at different deposition current densities, it has been demonstrated that the electrolyte containing CsPF₆ additive could effectively smooth the Li surface at current densities less than 1.0 mA cm⁻² [14]. The above cell performance data of Li|LFP cells at different charge current densities also suggest that the effect of CsPF₆ additive may decrease with increasing charge current density. This effect can be attributed to larger increase in SEI thickness at higher current densities. Therefore, more pronounced effect for CsPF₆ can be expected if the charge current density is not too high. Figure 7 shows the excellent cycling stability of the Li|LFP cells with the E1Cs electrolyte for 500 cycles when the charge current density was 0.48 mA cm⁻² (i.e. C/5 rate). The cycling efficiency is close to 100%. However, the capacities of Li|LFP cells using electrolytes without CsPF₆ additive start to decrease quickly after about 100 cycles even at such low charge current density. This demonstrates the dendrite suppression via CsPF₆ additive is more dramatic at relatively low charge current densities.

4. Conclusions

Addition of CsPF₆ as an electrolyte additive improves both cycling performance and rate capability of Li|LFP cells. CsPF₆ protects Li metal anode by suppressing Li dendrite growth, reducing the chemical reactions between Li and electrolytes and the electrochemical decompositions of electrolytes, and retarding the increase of the cell

impedance during long-term cycling, which have been confirmed by SEM and a.c. impedance analyses. The Li|LFP coin cells with a CsPF₆-containing electrolyte can be stably cycled for at least 500 cycles with negligible capacity fading when the charge current density was 0.48 mA cm⁻², while the capacities of Li|LFP cells start to decrease quickly after about 100 cycles in electrolytes without Cs⁺ additive due to the growth of porous and thick SEI layers. With the increase of the charge current density, the effect of CsPF₆ additive is less dramatic in Li|LFP cells. Future work needs to be done in optimizing the electrolyte compositions for Li anode protection during long-term cycling and at high charge current densities.

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Figures captions

Figure 1. Voltage profiles of Li|LFP coin cells with the three different electrolytes (E1, E1Cs and E2) during the first formation cycle from 2.8 V to 3.9 V at C/10 rate.

Figure 2. Rate capability of Li|LFP cells with three different electrolytes (E1, E1Cs and E2) after 2 formation cycles at C/10 rate. The charge rate is the same (C/5) and the discharge rate is from C/5, to C/3, C/2, 1C, 2C, 5C then back to C/5, each with 5 cycles.

Figure 3. Cycling performance of Li|LFP coin cells with three different electrolytes (E1, E1Cs and E2) at charge current density of 1.60 mA cm^{-2} (2C/3) and discharge current density of 2.40 mA cm^{-2} (1C) after two formation cycles at C/10.

Figure 4. Nominal discharge capacity at the 100th cycle for Li|LFP cells containing three different electrolytes (E1, E1Cs and E2) at different charge current densities (from 0.48 to 1.6 mA cm^{-2}) and the same discharge current density (2.4 mA cm^{-2}).

Figure 5. SEM images of the surface (a, b and c) and cross-section (e, d and f) of Li metal anodes after 150 cycles at 1.2 mA cm^{-2} (C/2) charging and 2.4 mA cm^{-2} (1C) discharge in E2 (a, d), E1 (b, e) and E1Cs (c, f), respectively.

Figure 6. A. C. impedance plots of fresh and cycled Li|LFP cells using electrolytes with and without CsPF₆ additive: (a) E2, (b) E1 and (c) E1Cs. The cells were cycled from 2.8 V to 3.9 V at discharge current density of 2.4 mA cm^{-2} and charge current density of 1.6 mA cm^{-2} .

Figure 7. Long-term cycling performance of Li|LFP coin cells with different electrolytes (E1, E1Cs and E2) at charge current density of 0.48 mA cm^{-2} (C/5) and discharge current density of 2.4 mA cm^{-2} (1C) after two formation cycles at C/10.

Figure 1

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Figure 4

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Figure 7