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ARTICLE

# Electrochemical Properties of PP13TFSI-LiTFSI-P(VdF-HFP) Ionic Liquid Gel Polymer Electrolytes

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*N*-Methyl-*N*-propylpiperidiniumbis(trifluoromethanesulfonyl)imide (PP13TFSI), bis(trifluoromethanesulfonyl)imide lithium salt (LiTFSI), and poly(vinylidene difluoride-co-hexafluoropropylene) (P(VdF-HFP)) were mixed and made into ionic liquid gel polymer electrolytes (ILGPEs) by solution casting. The morphology of ILGPEs was observed by scanning electron microscopy. It was found that the ILGPE had a loosened structure with liquid phase uniformly distributed. The ionic conductivity, lithium ion transference number and electrochemical window were measured by electrochemical impedance spectroscopy, chronoamperometric and linear sweep voltammetry. The ionic conductivity and lithium ion transference number of this ILGPE reached 0.79 mS/cm and 0.71 at room temperature, and the electrochemical window was 0 to 5.1 V *vs.* $Li^+$/Li. Battery tests indicated that the ILGPE is stable when being operated in Li/$LiFePO_4$ batteries. The discharge capacity maintained at about 135, 117, and 100 mAh/g at 30, 75, and 150 mA/g rates, respectively. The capacity retentions were almost 100% after 100 cycles without little capacity fading.

**Key words:** Lithium ion battery, Ionic liquid, Polymer electrolyte, *N*-Methyl-*N*-propylpiperidiniumbis(trifluoromethanesulfonyl)imide, Poly(vinylidene difluoride-co-hexa fluoropropylene)

## I. INTRODUCTION

The rechargeable lithium polymer battery is the most attractive candidate as the power sources for portable electronic devices and electric vehicles because of its high energy density, long cycling life, safety, assembling flexibility, *etc.* [1, 2]. Compared with organic liquid electrolytes, polymer electrolytes can improve the safety of lithium ion batteries to some extent. However, the ionic conductivity of the polymer electrolytes still limited the performance of lithium ion batteries and safety problems still exist owing to the use of highly flammable organic solvents as plasticizers, which is the main cause of fire and explosion in short circuit or abuse conditions [3−5]. The research and application of ionic liquids provide a way to solve the safety problem of the lithium ion batteries. Ionic liquids are not only nonvolatile and nonflammable, but also have a high conductivity, a wide electrochemical stability and a high thermal stability [6]. Ionic liquids are usually used as a plasticizer to incorporate the polymer electrolyte to enhance the ionic conductivity. After Fuller using ionic liquids for polymer electrolyte in 1997, researchers have conducted a lot of research in ionic liquid gel polymer electrolytes [7−11].

Because $EMI^+$ is easily synthesized, research on EMITFSI used in ILGPEs is relatively more, and it possesses many desirable properties such as a wide electrochemical window at anode, low viscosity and high ionic conductivity, and ionic liquids based in $TFSI^-$ anion were hydrophobic, chemically stable against moisture and could provide rather stable lithium deposition/dissolution [12−15]. However, there are still some problems such as low cathodic stability, irreversible decomposition on the negative electrode surface and the competition of $EMI^+$ with $Li^+$, which decrease the lithium ions transference number $t_{Li^+}$ of the obtained polymer electrolyte and causes irreversible capacity and poor properties [16]. To find a practical electrolyte of the present Li-ion battery, new type RTILs are researched. Sakaebe *et al.* found out that quaternary ammonium-imide salts have been the most attractive due to the cathodic stability of the salts against lithium metal [17, 18]. They also found that *N*-methyl-*N*-propylpiperidiniumbis(trifluoromethanesulfonyl)imide (PP13TFSI) is the most promising candidate as the electrolyte base. PP13TFSI has a high ionic conductivity although somewhat lower than that of EMITFSI, a wide electrochemical window, and especially a good stability to the Li metal. A lot of work about PP13TFSI is focused on the liquid electrolyte [19−21], and only a little on the ionic liquid gel polymer

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electrolyte, such as PEO-PP13TFSI based electrolyte [22−24].

In recent years, poly(vinylidene fluoride-co-hexafluropropylene) (P(VdF-HFP)) polymer electrolyte containing ionic liquid has aroused much attention due to their higher ionic conductivities [25−27]. Though the ionic conductivity reaches $10^{-3}$ S/cm at room temperature, the electrochemical performance of cathode or anode materials using PP13TFSI-LiTFSI-P(VdF-HFP) gel polymer as electrolyte has not been reported in details elsewhere. In this work, LiTFSI-PP13TFSI-P(VdF-HFP) polymer electrolytes were prepared and characterized in terms of the basic electrochemical properties, the cycling performance, and reversibility of Li/$LiFePO_4$ batteries.

## II. EXPERIMENTS

### A. Preparation of ionic liquids gel polymer electrolytes

PP13TFSI was prepared by mixing two aqueous solutions of equimolar *N*-methyl-*N*-propylpiperidinium bromide (PP13Br, Acros Organics, USA) and lithium-bis(trifluoromethanesulfonyl)imide (LiTFSI, Acros Organics, USA) at room temperature for 6 h to get a phase separated solution with hydrophobic ionic liquid PP13TFSI and aqueous phases. The resulting PP13TFSI was repeatedly washed with deionized water, and then dehydrated under vacuum at 120 °C for 48 h. The final product PP13TFSI was a clear colorless liquid at room temperature. Water content was measured to be below 50 ppm using the standard Karl Fischer method. The titrations were performed with an automatic Karl Fischer coulometer titrator (SFY-3000, Haifen Instrument Ltd. Co., China).

Ionic liquid gel polymer electrolyte was prepared by the solution casting method as follows. 1 g P(VdF-HFP) ($M_w$=5×$10^5$, Aldrich) was dissolved in 6 g *N*-methyl pyrrolidone (NMP) and stirred for 24 h to get a homogeneous gel solution. Then PP13TFSI and LiTFSI were added into the solution and stirred for 12 h and finally a transparent gel solution was obtained. Then the solution was cast into the polytetrafluoroethylene molds and dried at 80 °C for 36 h under vacuum. After cooling down naturally, the desired P(VdF-HFP)-LiTFSI-$x$PP13TFSI ($x$=PP13TFSI/LiTFSI in weight ratio, P(VdF-HFP)=LiTFSI) ILGPE was got and stored in a glove box filled with argon.

### B. Characterization of ionic liquid gel polymer electrolyte

The morphology of polymer electrolytes was characterized by scanning electron microscopy (SEM, KYKY-EM3200). The samples were sputter-coated with approximately 10 nm of gold before analysis.

The ionic conductivity ($\sigma$) of the ILGPEs was determined by electrochemical impedance spectroscopy (EIS) with SS/ILGPE/SS cell by Potentio-Galvanostat (EG&G Princeton Applied Research, M273). The frequency range was 100 kHz to 100 mHz and an AC signal of 5 mV in amplitude as the perturbation. The value of $\sigma$ was calculated from Eq.(1):

$$\sigma = \frac{d}{R_\mathrm{b} S} \tag{1}$$

where $d$ is the thickness of the membrane, $S$ is the area of the stainless steel (SS) electrode and $R_\mathrm{b}$ is the bulk resistance.

$t_{\mathrm{Li}^+}$ was determined using a symmetric Li/ILGPE/Li cell [28]. When a constant polarization potential (DC bias potential) of 10 mV was applied to the cell, a current was measured that fell from an initial value $I_0$ to a steady-state value $I_\mathrm{s}$. With time going, ions except $\mathrm{Li}^+$ accumulated at the electrodes and a concentration gradient was formed. At the steady state, only $\mathrm{Li}^+$ ions carry the current. So $t_{\mathrm{Li}^+}$ could be evaluated from the ratio $I_\mathrm{s}/I_0$. Because the thickness of the passivating film on the electrode would vary over the time required to reach a steady-state current, the values of the intrinsic resistance must be measured shortly before the application of the DC bias potential and immediately after the attainment of steady state to determine the correct $t_{\mathrm{Li}^+}$. Thus, $t_{\mathrm{Li}^+}$ was calculated using the Eq.(2):

$$t_{\mathrm{Li}^+} = \frac{I_\mathrm{s}(V - I_0 R_0)}{I_0(V - I_\mathrm{s} R_\mathrm{s})} \tag{2}$$

here the subscripts 0 and s indicate initial values and steady-state values, respectively, $R_i$ ($i$=s and 0, respectively) is the interfacial resistance which is approximately equal to sum of charge transfer resistance and passivating film resistance, and can be achieved by recording two impedance spectra on the cell in the frequency range between $10^{-2}$ and $10^5$ Hz before the application of the bias potential, and after steady-state has been reached and the DC bias potential has been removed. $V$ is the applied potential 10 mV, and $I$ is the current.

Evaluation of the electrochemical window of the electrolyte was carried out by linear sweep voltammetry (LSV, scan rate of 2 mV/s) using an electrochemical analyzer (CHI604b) in two-electrode cells with stainless steel (SS) as the working electrode ($\Phi$=16 mm), and lithium as both counter and reference electrode.

Cyclic voltammetry (CV) measurements of a Li/ILGPE/$LiFePO_4$ cells were performed at 25 °C with a scan rate of 0.1 mV/s from 2.5 V to 4.5 V, using an electrochemical analyzer (CHI604b).

The $LiFePO_4$ electrodes were prepared by casting slurry containing 80%$LiFePO_4$, 10%acetylene black (AB) and 10%poly (vinylidene difluoride) (PVDF) on aluminum foil and dried at 120 °C for 12 h under vacuum. The electrodes were punched into disks

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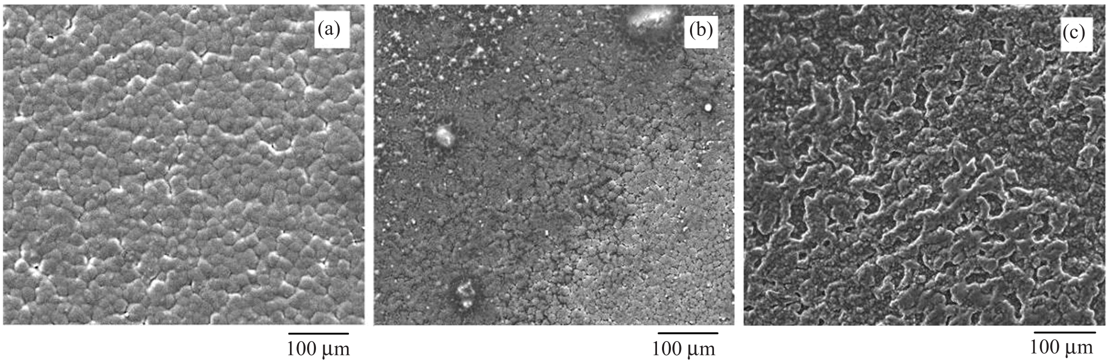

FIG. 1 SEM images of polymer electrolytes. (a) Pure P(VdF-HFP), (b) LiTFSI-P(VdF-HFP), and (c) PP13TFSI-LiTFSI-P(VdF-HFP).

TABLE I Composition of PP13TFSI-LiTFSI-P(VdF-HFP) ILGPEs and their ionic conductivities.

| Code name | Mass content/% | | | $\sigma$/(μS/cm) |
|---|---|---|---|---|
| | PP13TFSI | LiTFSI | P(VdF-HFP) | |
| ILGPE-0 | 0 | 50.0 | 50.0 | 28.7 |
| ILGPE-1 | 20.0 | 40.0 | 40.0 | 436 |
| ILGPE-2 | 33.3 | 33.3 | 33.3 | 793 |
| ILGPE-3 | 42.9 | 28.6 | 28.6 | 842 |
| ILGPE-4 | 50.0 | 25.0 | 25.0 | 883 |

($\Phi$=14 mm) and compressed at 10 MPa with a hydraulic press. The Li/$LiFePO_4$ half-cells (CR2025) were assembled in an argon-filled glove box and galvanostatically charged and discharged using BTS-510A (5 V-1 mA/3 mA) (Neware electronic Co., LTD, Shenzhen, China) at 25 °C in the voltage range of 2.7−4.2 V *vs.* $Li^+$/Li.

The electrochemical impedance spectroscopy (EIS) of Li/ILGPE/$LiFePO_4$ half-cells was measured from 100 kHz to 10 mHz with an AC signal of 5 mV in amplitude as the perturbation.

## III. RESULTS AND DISCUSSION

### A. Ionic conductivity

Ionic conductivity of electrolytes is the main factor influencing the performance of batteries. A higher ionic conductivity will make the polarization of cells lower and be good for cells to improve the rate capability. So the ionic conductivity of the ILGPEs was first studied. Table I shows the conductivity of different PP13TFSI-LiTFSI-P(VdF-HFP) electrolytes.

As can be observed in Table I, the ionic conductivity of the ILGPE without PP13TFSI was 28.7 μS/cm and the ionic conductivity increased obviously after PP13TFSI was added. When the amount of PP13TFSI was 33.3%, the ionic conductivity reached the value of 793 μS/cm at room temperature. Then it increased slower with the addition of PP13TFSI. An increase in conductivity with the increase of PP13TFSI at low concentration range probably resulted from the increased amount of charge carriers. At the higher concentration, the charge carriers and transmission channel of ions were already abundant. So the continuous addition of PP13TFSI would not lead to apparent increase of ionic conductivity. At the same time, the mechanical strength became worse.

Above all, the ILGPE with 33.3% PP13TFSI was the most suitable for Li-ion batteries and the following was studied on this type of ILGPE.

### B. Morphology

The obtained ILGPEs are free standing films and the SEM of the surface of ILGPEs is shown in Fig.1.

The pure P(VdF-HFP) membrane was subtransparent in appearance and Fig.1(a) shows a tightly packed structure with few cracks. It was seen that no phase separation occurred. With the addition of LiTFSI, the polymer membrane changed into transparent in appearance and unconsolidated with some cracks appearing (Fig.1(b)). After blending with PP13TFSI, there was a liquid phase uniformly distributed in the membrane [28, 29] and the polymer electrolyte became looser. This loosened structure was more beneficial for the ions diffusion and would certainly improve the ionic conductivity.

### C. Lithium ion transference number

During charge-discharge tests of rechargeable Li-ion batteries, the $Li^+$ and the other ions in the ILGPE such as $PP13^+$ and $TFSI^-$ are all mobile, but only the migration of $Li^+$ is effective. Therefore, $t_{Li^+}$ is also critical for high performance ILGPEs. In this work, the $t_{Li^+}$ values are obtained from polarization measurements. Figure 2(a) presents the impedance spectra of PP13TFSI-LiTFSI-P(VdF-HFP) ILGPE recorded before and af-

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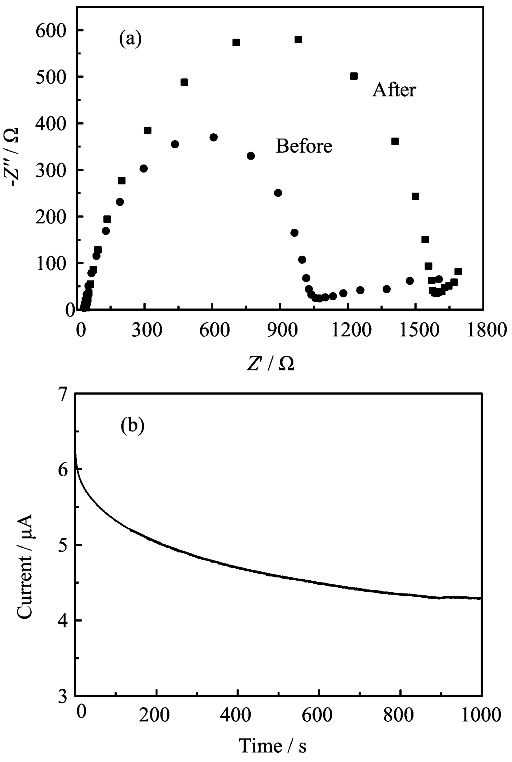

FIG. 2 (a) Impedance spectra of PP13TFSI-LiTFSI-P(VdF-HFP) ILGPE recorded before and after the polarization at room temperature. (b) Chronoamperometry of the Li/ILGPE/Li cell at room temperature.

ter the polarization at room temperature. Figure 2(b) presents the chronoamperometric curve.

The values were $I_0$ (6.28 μA), $I_\mathrm{s}$ (4.29 μA), $R_0$ (1042.94 Ω), and $R_s$ (1550.61 Ω), obtained from Fig.2. According to Eq.(2), the value of $t_{\mathrm{Li}^+}$ was calculated to 0.71, which could satisfy the demand of being used in lithium ion batteries.

### D. Electrochemical stability window

A wide electrochemical stability window makes electrolyte suitable for most electrode materials, so that the electrolyte can be used in wide application range. Linear sweep voltammetry (LSV) experiments are performed to investigate the electrochemical stability window of the P(VdF-HFP)-LiTFSI and P(VdF-HFP)-LiTFSI-PP13TFSI polymer electrolytes and the results are shown in Fig.3.

It could be seen from Fig.3 that the anodic stability limit (here the potential was set at a current density up to 0.1 mA/cm$^2$) of the P(VdF-HFP)-LiTFSI membrane was around 5.1 V *vs.* Li$^+$/Li. The anodic limiting current corresponded to the oxidation of the TFSI$^-$ anions. The cathodic current was observed around 0 V *vs.* Li$^+$/Li, which was associated with the deposition of lithium. With the addition of PP13TFSI, the electrochemical stability window changed little. The results indicated that PP13TFSI based ILGPE had a higher stability than EMITFSI based [16] and was satisfied to be used with most Li-ion battery electrode materials.

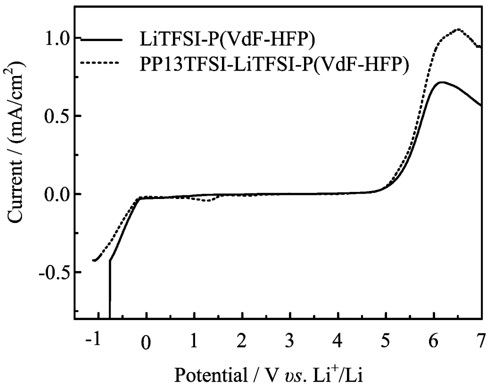

FIG. 3 Linear sweep voltammetry of polymer electrolytes at 2 mV/s.

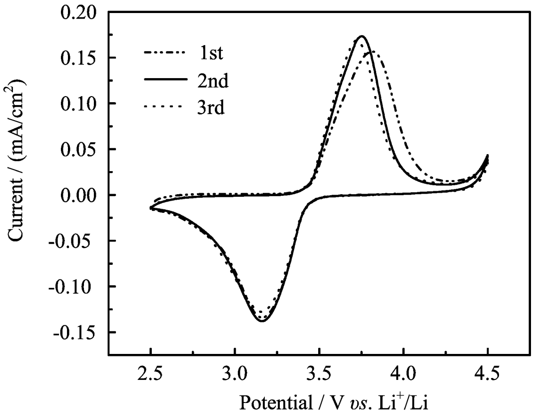

FIG. 4 Cyclic voltammetry curves of Li/ILGPE/LiFePO$_4$ cell at a scan rate of 0.1 mV/s.

### E. CV curves for Li/LiFePO$_4$ cells

Figure 4 is CV curves for Li/LiFePO$_4$ cells using PP13TFSI-LiTFSI-P(VdF-HPF) ILGPE.

The first redox reactions of LiFePO$_4$ were observed at nearly 3.80 and 3.16 V *vs.* Li/Li$^+$. While the redox reactions of LiFePO$_4$ in the second and third cycles were almost the same, about 3.70 and 3.16 V *vs.* Li/Li$^+$, indicating that the Li/LiFePO$_4$ cell with PP13TFSI-LiTFSI-P(VdF-HPF) ILGPE had good cyclic stability. The areas of oxidation and reduction were about the same, which indicated that the redox reaction has good reversibility. The anodic peak of the first cycle was a little more positive than that of second and third cycles, showing a higher polarization during the first cycle. It meaned the internal resistance of the cells reduced after the first cycle.

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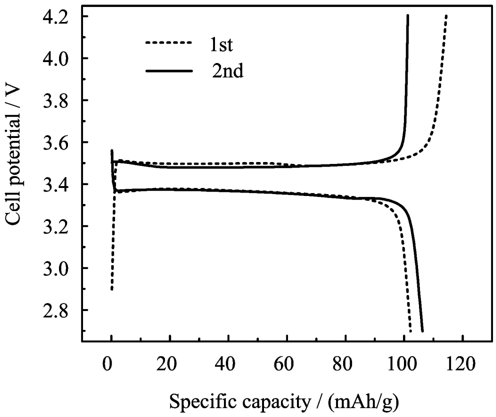

FIG. 5 Charge-discharge curves of Li/LiFePO$_4$ cells at 1 C rate.

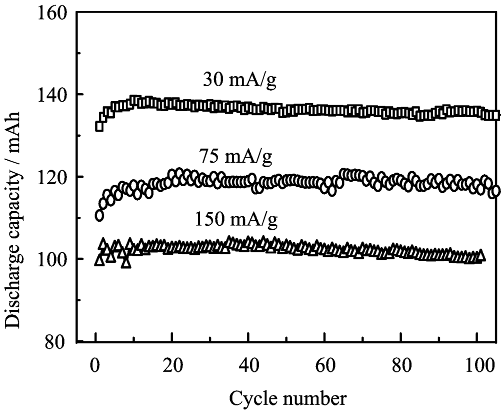

FIG. 6 Cycling performances of Li/LiFePO$_4$ cells at different rates at 25 °C.

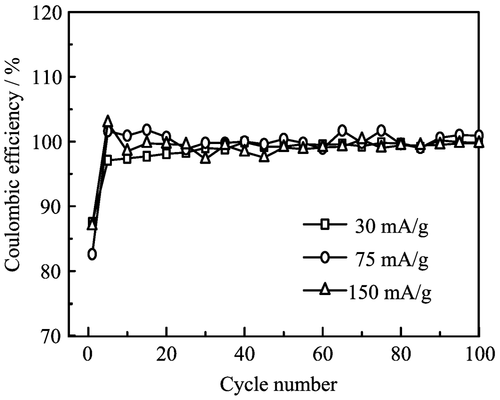

FIG. 7 Coulombic efficiencies of Li/LiFePO$_4$ cells at different rates at 25 °C.

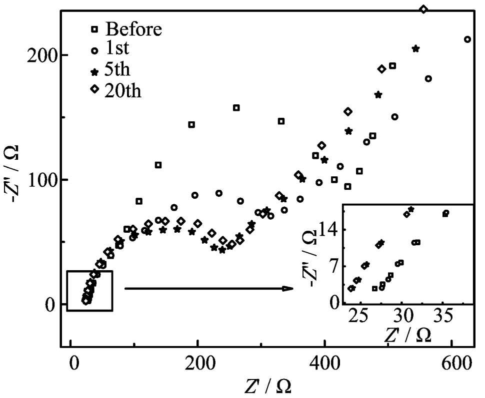

FIG. 8 Electrochemical impedance spectra of the Li/LiFePO$_4$ cell after selected cycles at 25 °C at 30 mA/g rate.

### F. Li/LiFePO$_4$ cell performance

The Li/PP13TFSI-LiTFSI-P(VdF-HFP) ILGPE /LiFePO$_4$ cells was tested at 25 °C. Figure 5 shows the charge-discharge curves of Li/LiFePO$_4$ cell at 150 mA/g.

The discharge capacity of the first cycle was 102 mAh/g and the coulombic efficiency was 87%. For the second cycle, the discharge capacity was 106 mAh/g, a little higher than that of the first cycle, and the coulombic efficiency almost reached 100%, revealing good reversibility of Li/LiFePO$_4$ cells with PP13TFSI-LiTFSI-P(VdF-HFP) ILGPE. At the same time, the overlapped charge-discharge plateaus of the two cycles also showed good cycling stability.

The cycling performance of Li/LiFePO$_4$ cells at different rates is shown in Fig.6. The cells showed good cycling performance at each rate. The discharge capacity maintained at about 135, 117, and 100 mAh/g at 30, 75, and 150 mA/g, respectively, and the capacity retentions were almost 100% after 100 cycles with little capacity fading. The discharge capacities increased slowly and continuously during the first 10 cycles because of the activation of electrode materials. The discharge capacity declined with the discharge rate increase. This was mainly caused by the lower ionic conductivity of ILGPE compared with liquid electrolytes. The ions were not able to move fast enough, so the polarization during the charge-discharge tests increased with the discharge rate increase.

Figure 7 presents the coulombic efficiency of Li/LiFePO$_4$ cells at 25 °C at different rates, showing that the coulombic efficiency was about 97% or above. This also indicated the PP13TFSI-LiTFSI-P(VdF-HFP) ILGPE had good compatibility with LiFePO$_4$ electrode. The low coulombic efficiency of the first cycle was related to the irreversible capacity, which arised from the reductive decomposition of electrolyte.

To get more information about the interface of the cells, EIS of the cells after selected cycles were measured and results are shown in Fig.8. It was observed in Fig.8 that the impedance spectrum was composed of two partially overlapped semicircles at high frequency and medium frequency and a straight sloping line at the low frequency end. The bulk resistance reflecting the electric conductivity of the ILGPE and electrodes

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was about 25 Ω and almost keep constant during the charge-discharge test. The resistance, which reflected the solid-state interface layer formed on surface of electrode and charge-transfer resistance between electrode and electrolyte, decreased after the first cycle and then kept a constant value. This was similar to Fig.4. It is speculated that the solid-state interface layer formed on surface of electrode supplied channels for ions to migrate and reduced the internal resistance of cells. Meanwhile, it was also beneficial to keep good cycling stability.

## IV. CONCLUSION

A PP13TFSI-LiTFSI-P(VdF-HFP) ionic liquid gel polymer electrolyte was prepared by solution casting. The addition of PP13TFSI made the ILGPE unconsolidated and the uniformly distributed liquid phase improved the ionic conductivity of the ILGPE. When the amount of PP13TFSI was 33.3%, a self standing film was obtained, and the ionic conductivity and lithium ion transference number were 790 μS/cm and 0.71 at room temperature with a wide electrochemical window from 0 to 5.1 V *vs.* $Li^+$/Li. The tests of the ILGPE in Li/$LiFePO_4$ cells at different rates certified the cycling stability and reversibility. The discharge capacities maintained at about 135, 117, and 100 mAh/g at 30, 75, and 150 mA/g with very little capacity fading after 100 cycles.

## V. ACKNOWLEDGMENTS

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