All-Solid-State Rechargeable Lithium Metal Battery with a Prussian Blue Cathode Prepared by a Nonvacuum Coating Technology

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ABSTRACT: A Prussian blue LiFeFe(CN)₆ thin-film cathode is fabricated by a nonvacuum coating technology without post-annealing process. The thin film of the solid electrolyte lithium phosphorus oxynitride (LiPON) is deposited onto the cathode by using radio-frequency magnetron sputtering. Then, the lithium metal anode is deposited on the LiPON film by the thermal evaporation method to fabricate the all-solid-state LiFeFe(CN)₆/LiPON/Li battery with a thickness of 16 μm and a size of ~10 cm². Electrochemical properties of LiFeFe(CN)₆/LiPON/Li battery are first investigated at various temperatures from −30 to 80 °C. Our results demonstrated that the all-solid-state LiFeFe(CN)₆/LiPON/Li battery exhibits a discharge capacity of 82.5 mA h/g for the third cycle at 60 °C and shows stable cyclic performance within 200 cycles. These results provide the feasibility to assemble an all-solid-state lithium-ion battery by combining nonvacuum and vacuum techniques through an environmentally friendly process at low temperature.

INTRODUCTION

With the growing energy demand and increasing social enhancement in environmental consciousness, the secondary chemical power system developing rapidly and gradually replaced the primary energy system. Lithium-ion battery (LIB) as the high-energy chemical power source has been used in a large number of applications in laptop computers, digital cameras, and other electronic products. However, the commercial LIB with an organic electrolyte always has several drawbacks such as safety issue, insufficient lifetime, high cost, and so forth. For example, the battery internal combustion to explosion is caused by short-circuit problem, and it has no regular outbreak and unpredictable happen. The development of all-solid-state thin-film battery with inflammable solid electrolytes is an important strategy to solve those problems, in particular, the safety issue. Therefore, the all-solid-state battery has become an exigent topic for a new generation battery system with high energy density.¹² The glassy lithium phosphorus oxynitride (LiPON) ion conductor has been the subject of increasing interest because of its isotropic ionic conduction, no grain boundaries, and ease of fabrication as an electrolyte for all-solid-state rechargeable ion batteries.³ LiPON has a stable electrochemical window at potential range from 0 to nearly 5.5 V in contact with metallic lithium and high conductivity close to 2 × 10⁻⁶ S cm⁻¹.¹³,⁴ The all-solid-state batteries with various cathodes such as LiCoO₂,⁴ LiMn₂O₄,⁵ LiCoPO₄,⁶ LiFePO₄,⁷ and FePO₄⁸ were widely investigated. Those cathodes were usually prepared by vacuum deposition techniques such as sputtering or evaporation.⁹

However, these film cathodes with crystalline or nanocrystalline need further high-temperature annealing; thus, the typical silicon or stainless steel substrates were used as an electric collector. Prussian blue iron hexacyanoferrate (FeHCF) and its analogues (AₓM[Fe(CN)₆]ₙ⁻·xH₂O, A and M are alkali metal and transition metal, respectively) have received considerable attention as attractive cathode materials for LIBs because of their low cost, environmental friendliness, and easy synthesis process. In addition, the FeHCF cathode material has a stable open-framework structure and delivers a high capacity of 195 mA h/g (the specific gravimetric and volumetric energy density: 624 W·h·kg⁻¹ and 1123 W·h·L⁻¹) with an average voltage of 3.2 V).¹⁰⁻¹³ Previous studies about the electrochemical activity of FeHCFs in LIBs in nonaqueous electrolyte reported that their electrochemical properties were strongly dependent on synthesis methods. For example, Shen et al. reported the electrochemical performances of cubic FeHCF synthesized by a coprecipitation method as cathode materials.¹⁰ Wu et al. reported high-quality and high-purity nanocrystal FeHCF electrode by using Fe(CN)₆⁶⁻ as a single iron source for a high-capacity and long-life LIB.¹¹ Recently, a high-quality-sized FeFe(CN)₆ as a cathode material for nonaqueous potassium ion battery was reported in our group.¹⁴ However, there is no available report on the electrochemical characterization of all-solid-state lithium...
battery based on Prussian blue LiFeFe(CN)$_6$ cathode because it is very difficult to prepare the thin-film electrode by vacuum deposition techniques. Here, we report the fabrication process and electrochemical performance of LiFeFe(CN)$_6$/LiPON/Li.
all-solid-state battery for the first time. All-solid-state cells are assembled with LiPON film as an electrolyte and metal lithium film as an anode, and LiFeFe(CN)₆ thin film on the aluminum foil fabricated by coating technology as a cathode. The nonvacuum coating technology is employed for cathode film fabrication from the perspective of large-scale preparation of current LiBs with liquid electrolyte. Our purpose is to examine the electrochemical feature of a novel all-solid-state LiFeFe(CN)₆/LiPON/Li battery system as well as to explore the new assembling methods for all-solid-state battery.

**RESULTS AND DISCUSSION**

Figure 1a shows X-ray diffraction (XRD) pattern of as-prepared LiFeFe(CN)₆. All of the diffraction peaks can be well indexed to that of the PB analogues with a space group of Fm3m (JCPDS no. 01-0239). No obvious peak separation is observed in the XRD pattern, indicating the pure phase of as-prepared LiFeFe(CN)₆. As shown in the Fourier transform infrared spectroscopy (FTIR) spectrum of the as-prepared LiFeFe(CN)₆, (Figure 1b), strong peak at 2086 cm⁻¹ can be attributed to the characteristic of the stretching vibration of C=N bonds in the [Fe³⁺=(CN)₆⁻] structure. Two peaks at 1632 and 3447 cm⁻¹ are assigned to the O=H stretching vibration and H=O–H bending mode, respectively, indicating the existence of crystal water in the open-framework structure of LiFeFe(CN)₆. This is further proved with thermogravimetric analysis (TGA) result, as shown in Figure 1c, in which a negative peak of differential thermal analysis (DTA) curve at 97.65 °C represents the loss of crystal water (18.76 wt %). In addition, two positive peaks at 322.67 and 410.42 °C represent the oxygenolysis of LiFeFe(CN)₆. The synthesized particles with sizes around 1 μm can be observed from scanning electron microscopy (SEM) image, as shown in Figure 1d. The structure of LiFeFe(CN)₆ is illustrated in Figure 1e, consisting of a three-dimensional network of Fe–N≡C–Fe chains along the edges of the unit cell cube. Fe₁ and Fe₂ are surrounded octahedrally by C and N atoms, respectively. This induces different spin configurations in Fe₁ and Fe₂ atoms: high spin configuration of t²g⁶ e⁰ in Fe₁ (FeHS) and low spin configuration of t²g⁶ e⁰ in Fe₂ (FeLS). The different redox behaviors of FeHS and FeLS have been investigated by Goodenough and co-workers by using soft X-ray absorption technique. Li⁺ ions can intercalate into or deintercalate from this three-dimensional framework.

The large open space in the framework of LiFeFe(CN)₆ with Fe sites as the active redox center, which can be provided for lithium storage. Figure 2a shows the first three cyclic voltammetry (CV) curves for the LiFeFe(CN)₆ electrode in 1.0 mol/L LiPF₆-ethylene carbonate (EC)/diethyl carbonate (DEC) electrolyte between a voltage range of 1.5 and 4.0 V and at a scan rate of 0.1 mV/s. In the first charging process, a large peak appears at 3.82 V, indicating the deintercalation process of Li⁺ from the LiFeFe(CN)₆ electrode. In subsequent scans, the main features in the CV curves exhibit two pairs of well-defined symmetric redox peaks at 3.82/3.73 V and 3.07/2.98 V. These peaks should correspond to the oxidation−reduction of the low spin Fe³⁺/Fe²⁺ couple coordinated to the C atoms and the high spin Fe³⁺/Fe²⁺ couple coordinated to the N atoms. The intensities and shapes of these two pairs of CV peaks were maintained in the subsequent cycles, indicating a highly reversible and stable oxidation−reduction process of the LiFeFe(CN)₆ cathode. If compared with CV curves of FeFe(CN)₆ electrode previously reported, the shapes of the redox peaks at 3.82/3.73 V are sharper and more symmetrical and their relative intensities to the redox peaks at 3.07/2.98 V are larger than those of FeHCFC electrodes. This result suggests that the LiFeFe(CN)₆ electrode exhibits a well electrochemical activity than that of the FeFe(CN)₆ electrode. The occupation of Li in the LiFeFe(CN)₆ electrode may help the structural stability of its framework to ensure the high electrochemical activity of the high spin Fe³⁺/Fe²⁺ couple. It should be noted that LiFeFe(CN)₆ as a lithium-contained Prussian blue electrode could exhibit a well electrochemical performance.

The galvanostatic charge/discharge curves of the LiFeFe(CN)₆ electrode in the first 200 cycles at 0.5 C (1 C = 142 mA/g) was calculated based on the first discharge capacity) are shown in Figure 2b. The initial charge process delivers a specific capacity of 90 mA h/g, which is close to one Li⁺ extracted from LiFeFe(CN)₆ whereas the first discharge capacity reaches to 142 mA h/g and two voltage plateaus at around 3.76 and 3.10 V can be observed clearly, exhibiting a two-electron redox process. The charge/discharge curves can almost overlap without any polarization in the subsequent processes, and voltage profiles are in good agreement with the CV data.

The well cyclic performance of the LiFeFe(CN)₆ electrode is also clearly seen from Figure 2c. After 200 cycles at 0.5 C, the electrode still retains a discharge capacity of 120 mA h/g and the Coulombic efficiencies from the second cycle can maintain a high level of nearly 100%. Besides, the capacity fade is calculated to be 0.1% per cycle after 200 charge/discharge cycles. Figure 2d shows good rate performance of the LiFeFe(CN)₆ electrode, in which the LiFeFe(CN)₆ electrode delivers discharge capacities of 142, 135, and 96 mA h/g at 0.5, 1, and 20 C, respectively. Such a good electrochemical cyclic and rate performances should be related to large interstitial sites that allow fast insertion/extraction of Li ions in the open-framework crystal structure of LiFeFe(CN)₆. It seems that water molecules tend to a stable structure of LiFeFe(CN)₆ with a lower water content. If the as-prepared LiFeFe(CN)₆ material is dried in a vacuum oven over 150 °C to remove the crystal water, it resulted in the low discharge capacities less than 70 mA h/g, and low Coulombic efficiency, and electrochemical performance deterioration for LiFeFe(CN)₆ electrode in 1.0 mol/L LiPF₆-EC/DEC electrolyte. It should be noted that the discharge capacities from lower plateaus (3.25−1.5 V) are 94, 93, and 87 mA h/g at 0.5, 1, and 20 C, respectively. The capacities from the lower plateaus due to the high spin Fe³⁺/Fe²⁺ are not changed by increasing currents, and the main capacity loss at higher rates should be attributed to the higher plateaus with the low spin Fe³⁺/Fe²⁺. It indicates that the oxidation−reduction of the low spin Fe³⁺/Fe²⁺ is kinetically limited and is strongly depend on the current rates from 0.5 to 20 C. In order to investigate the structural modification of LiFeFe(CN)₆ electrode during electrochemical process, ex situ XRD at charge and discharge states is carried out and is shown in Figure 3. It could be found that all of these diffraction patterns exhibit a typical face-centered cubic structure, whereas the shift of the diffraction peaks from (200) and (400) is observed clearly. The cubic lattice parameters were calculated as a₀ = 10.20 ± 0.01 Å, 10.17 ± 0.01 Å, and 10.23 ± 0.01 Å for the pristine and the LiFeFe(CN)₆ electrode after the charging to 4.0 V and discharging to 1.5 V, respectively. It means that the parameter a₀ decreases after charge and increases after...
The unit-cell volume changes of LiFeFe(CN)$_6$ after the full delithiation are ca. 0.88%. Such a negligible volume change during the charge/discharge processes of the LiFeFe(CN)$_6$ cathode is very attractive to all solid-state thin-film batteries.

After being successfully fabricated the LiFeFe(CN)$_6$/LiPON/Li battery, the electrochemical performance was investigated. The all-solid-state battery with a size of ~10 cm$^2$ (3.1 × 3.2) is shown in Figure 4a. Figure 4b shows cross-sectional SEM image of a typical all-solid-state battery with a LiFeFe(CN)$_6$ cathode. Each layer of the battery fabricated by different techniques can be clearly observed. The SEM image clearly shows that the thickness of the LiFeFe(CN)$_6$ electrode prepared on the aluminum substrates by a nonvacuum coating technology is about 16 μm. Thickness of the LiPON layer deposited on the LiFeFe(CN)$_6$ electrode by radio-frequency (rf) sputtering is about ~3 μm. Thickness of Li metal deposited by thermal evaporation is about 1–2 μm, and such a film battery can light a blue light-emitting diode (LED) in pouch condition (Figure 4c).

The electrochemical charge/discharge profiles of an all-solid-state LiFeFe(CN)$_6$/LiPON/Li battery are shown in Figure 5a. The galvanostatic discharge/charge curves of LiFeFe(CN)$_6$/LiPON/Li are examined at 10 μA/cm$^2$ at 60 °C. The open-circuit voltage lies at 2.6 V. The charge and discharge profiles include a slope region from 3.4 to 4.0 V for the first cycle and a corresponding slope region from 2.8 to 1.5 V in the second cycle, respectively. In the subsequent cycles, the charge and discharge curves differ considerably from the first cycle and both the charge/discharge voltage increases during the activation process of electrode. Apparently, one plateau on the charge and discharge processes appears in the all-solid-state battery, whereas two plateaus in the liquid electrolyte are observed, as shown in Figure 2b. The higher plateau at 3.8 V in liquid electrolyte disappears in the solid-state electrolyte because of the slow kinetic oxidation—reduction of the low spin Fe$^{3+}$/Fe$^{2+}$, and this will result in the lower discharge capacity in solid-state electrolyte than that in liquid electrolyte. This difference should be associated with the different interfaces between the electrode—liquid electrolyte and the electrode—solid electrolyte. The closer contact at the solid—solid interface with lower interfacial resistance should be needed to improve the capacity of LiFeFe(CN)$_6$/LiPON/Li in the future work. The discharge capacities of 61.5, 60.5, and 82.5 mA h/g are obtained, respectively, in the first, second, and third cycles. Afterward, the cycling becomes more and more stable, as shown in Figure 5b. The discharge capacities of the 50th, 100th, and 200th cycles are 53, 52, and 51 mA h/g, respectively, indicating highly reversibility and stability of all-solid-state battery.

The discharge/charge capacities of solid-state battery are found to be dependent on the ambient temperature and
current density. As shown in Figure 6, at the room temperature, the all-solid-state cell only delivers a capacity of 54.5 mA h/g at the current density of 10 μA/cm². The discharge capacity could be increased to 73.3 mA h/g at a current density of 10 μA/cm². The discharge polarization of LiFeFe(CN)₆/LiPON/Li battery obviously increases at a larger current density of 20 μA/cm² at 50 °C, which results in the reduced discharge capacity of 66.0 mA h/g. When the temperature is increasing, the capacity of solid-state battery increases at the current density of 20 μA/cm². When the temperature is increased to 60, 70, and 80 °C, higher discharge capacities of 76.4, 81.6, and 85.1 mA h/g are obtained. This indicates the utilization of about 90% of the active material in the thickness of about 16 μm if one Li⁺ is deintercalated from LiFeFe(CN)₆ in all-solid-state cell. These results suggest that LiFeFe(CN)₆/LiPON/Li battery could work at 80 °C and the high temperature is beneficial in the improvement of the capacity of solid-state battery. The low-temperature characteristic of the solid-state battery is a very important point of concern. Such an all-solid-state battery can charge or discharge in −30 °C at a reduced rate. Figure 7 shows the discharge and charge curves of all-solid-state LiFeFe(CN)₆/LiPON/Li battery at −30 °C and at a smaller current rate of 0.1 μA/cm². It can be clearly observed that the polarization of the cell sharply increases when the working temperature is decreased to −30 °C, indicating that the internal resistance of all-solid-state battery rises at low temperature. The discharge capacity of the cell significantly decreases, and the discharge capacity of 0.056 mA h (0.8 mA h/g) even at 0.1 μA/cm² is obtained for the second cycle; this value is almost 2 orders of magnitude smaller than the specific discharge capacity of 54.5 mA h/g delivered at room temperature at the 10 μA/cm². These measurements can carry out up to 40 cycles with a capacity loss of less than 2.0% per cycle. In order to clarify the possible origin for the limited capacity of all-solid-state LiFeFe(CN)₆/LiPON/Li battery at low temperature, ac impedance spectra of the LiPON thin film and the all-solid-state cell were measured at −30 °C. As shown in Figure 8a, it can be found that the electrolyte resistance acutely increases with the temperature decreasing and its values change from 2 kΩ at room temperature (see the inset) to about 400 kΩ at −30 °C. Ionic conductivity of the LiPON electrolyte film could be obtained from an ac impedance measurement of the Au/LiPON/Au sandwich structure on a glass substrate, and this type of sandwich structure has been described elsewhere. According to the following equation

\[
\delta = \frac{d}{RA}
\]

(1)

where \(d\) is the film thickness, \(A\) is the area of the Al contact, and \(R\) is the film resistance, which can be estimated by the ac impedance spectra analysis. The ionic conductivity (\(\delta\)) of LiPON can be obtained to be 2.0 × 10⁻⁶ S/cm at the room temperature and 1 × 10⁻⁸ S/cm at −30 °C. Figure 8b shows the impedance spectra of the cell. Data at the room temperature are included for comparison. It can be seen that the resistance of the cell increases from 2330 Ω at the room temperature to more than 13,500 Ω at −30 °C. According to the area of the full cell and ionic conductivities mentioned above, the impedance of LiPON between Li and LiFeFe(CN)₆ electrodes in the all-solid-state cell is calculated to be 8 and 250 Ω at the room temperature and −30 °C, respectively. Apparently, a low ion conductivity at −30 °C is not the origin of the limited discharge capacity of LiFeFe(CN)₆/LiPON/Li battery at low temperature. The interfaces impedance between LiFeFe(CN)₆/LiPON and LiPON/Li as well as the slow diffusion of Li ion in LiFeFe(CN)₆ at low temperature should mainly be responsible for the kinetics limitation of the all-solid-state cell.

**CONCLUSIONS**

LiFeFe(CN)₆ was synthesized as a cathode material for all-solid-state thin-film batteries. The electrochemical feature of
LiFeFe(CN)$_6$/LiPON/Li battery with 16 μm cathode layer was presented at measured temperatures from 80 to −30 °C. In the liquid cell, two pairs of well-defined symmetric redox peaks at 3.82/3.73 V and 3.07/2.98 V were clearly observed in the CV curves of LiFeFe(CN)$_6$ electrode. The discharge/charge curves of LiFeFe(CN)$_6$/LiPON/Li are different from those of the LiFeFe(CN)$_6$/LiPON/LiPON/Li battery. The higher plateau at 3.8 V in liquid electrolyte disappears in solid-state electrolyte because of the slow kinetic oxidation–reduction of the low spin Fe$^{3+}$/Fe$^{2+}$. The cell could exhibit a discharge capacity of 51 mA h/g after 200 cycles at 10 μA/cm$^2$. Our results have demonstrated the feasibility to assemble an all-solid-state battery by a nonvacuum coating technology for the cathode film, which is very important for grid-scale batteries because of high energy density, low cost raw materials, and an environmentally friendly process.

**EXPERIMENTAL SECTION**

**Synthesis of LiFeFe(CN)$_6$ Powder.** LiFeFe(CN)$_6$ powder was synthesized by simple aqueous reaction, 50 mL (0.05 mol/L) of Na$_2$Fe(CN)$_6$ aqueous solution was slowly added to 100 mL (0.1 mol/L) of FeCl$_3$ solution under the magnetic string and maintained for 6 h under 60 °C. The precipitate was soaked in excess LiCl solution and then was washed with deionized water. The molar concentration ratios of Na/Fe in these precipitates after each wash were examined by inductively coupled plasma emission spectroscopy (P-4010, Hitachi Co., Japan). All Na have been replaced with Li when the content of Na decreased from 1 to 0 during soaking and washing processes. Then, the green precipitate of LiFeFe(CN)$_6$ was collected by centrifugation and washed with deionized water several times and dried in a vacuum oven at 80 °C overnight.

**Characterization of LiFeFe(CN)$_6$ Powder.** The TGA/DTA (SDT Q600) was conducted in the temperature range from 20 to 600 °C under air atmosphere to calculate the number of the H$_2$O molecules in the as-prepared LiFeFe(CN)$_6$ material. The structure of pristine powder was examined by XRD (Bruker D8 ADVANCE, Germany) using Cu Kα radiation ($\lambda = 1.5415$ nm) at 1.6 kW. The morphology of LiFeFe(CN)$_6$ powder was measured with a SEM (Cambridge S-360). FTIR spectra were recorded on a Nicolet Nexus-470 spectrometer. The structure evolution of LiFeFe(CN)$_6$ cathode after initial charge/discharge process was also measured by ex situ XRD. The electrochemical cell was disassembled in the glovebox, and the electrode was washed by DEC three times to remove the residual electrolyte and salt.

**Assembling of Liquid Cell.** Active material (LiFeFe(CN)$_6$ powder, 80 wt %), electric conductor (acetylene black, 10 wt %), and binder (polyvinylidene fluoride, Sigma-Aldrich, 10 wt %) were mixed homogeneously and were dispersed into N-methyl-2-pyrrolidone (Sigma-Aldrich). LiFeFe(CN)$_6$ electrodes were prepared by coating the slurry onto an aluminum foil and drying in vacuum. The area mass loading of the electrode was approximately 2.3 mg/cm$^2$. A conventional coin cell was constructed in an argon ambient filled in the glovebox with LiFeFe(CN)$_6$ electrode as the cathode and one sheet of high-purity lithium foil as the anode. LiPF$_6$ (1 M) in nonaqueous EC/DEC (1:1) was used as a liquid electrolyte.

**Fabrication of All-Solid-State Battery.** The thin film of solid electrolyte LiPON was deposited (3 μm in thickness) onto the surface of LiFeFe(CN)$_6$ electrodes (the same electrodes as those used in liquid cell) by using rf magnetron sputtering in pure nitrogen atmosphere. Li$_2$PO$_4$ target was used for sputtering. The sputtering power was about 100 W. After that a metal lithium film (close to 1 μm in thickness) as an anode was deposited onto the surface of LiPON by the thermal evaporation method to form an all-solid-state battery.

**Electrochemical Measurement.** Galvanostatic charge/discharge cycles were carried out from −30 to 80 °C with a Land CT 2001A battery system. CV and ac impedance tests were performed on a CHI660A electrochemical working station (CHI instruments, TN). ac impedance spectra measurements were examined in the frequency range from 100 kHz to 0.01 Hz with an ac perturbation of 5 mV/s. The current densities and capacities of electrodes were calculated based on the weight of active materials.

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**Notes**

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