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A low temperature electrolyte for primary Li/CF_x batteries

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ABSTRACT

In this work, a 1:1 by weight blend of acetonitrile (AN) and γ -butyrolactone (BL) was studied as the solvent of low temperature electrolyte for high energy density Li/CF_x batteries. Both visual observation and impedance analysis show that metallic Li is kinetically stable in a 0.5 m LiBF₄ 1:1 AN/BL electrolyte. This property is attributed to the formation of a protective passivation film on the surface of metallic Li, and it has been successfully used to develop the low temperature electrolyte for Li/CF_x cells. It is shown that the cell with such an electrolyte outperforms the control cell with 0.5 m LiBF₄ 1:1 (wt.) propylene carbonate (PC)/1,2-dimethoxyethane (DME) electrolyte in both power capability and low temperature discharge performance. Impedance analyses reveal that the improved discharge performance is attributed to the high ionic conductivity of the AN/BL electrolyte. Due to the chemical incompatibility between metallic Li and A xt high temperatures, the storage and operation temperature for the Li/CF_x cells with 0.5 m LiBF₄ 1:1 AN/BL electrolyte is limited to or below ambient temperature (30 °C).

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1. Introduction

Lithium/carbon monofluoride (Li/CF_x with x = 1) batteries were first introduced by Matsushita in the 1970s as a primary lithium battery known to have a higher specific energy than Li/MnO₂, $Li/SOCl_2$ and Li/SO_2 batteries [1,2]. Currently, Li/CF_x batteries are mainly used in computer memory/clock backups and implantable medical devices such as cardiac pacemakers, where long operation time is essential at relatively low power. Major limitations to the large-scale application of Li/CF_x batteries are the low power capability and the poor low temperature performance, both of which are known to be associated with the low electrical conductivity and slow cell reaction kinetics of the CF_v cathode. Previous efforts to improve power capability were focused on the processing of the CF_x cathode, including reduction of the electrode thickness [3,4] and the use of subfluorinated carbon that has better rate capability [5–7]. However, the improvement gained by these approaches was achieved often in exchange for reduced energy density of the battery. Without exception, the electrolytes used in commercial Li/CF_x batteries are composed of LiBF₄ salt dissolved in a single organic solvent or an organic solvent blend, such as a propylene carbonate (PC)/1,2-dimethoxyethane (DME) mixture or a γ -butyrolactone (BL)/DME mixture [8–11]. This is probably because LiBF₄ is superior to LiPF₆ in thermal stability and in providing low charge-transfer resistance at low temperatures [12,13].

Previous attempts to improve low temperature performance were focused on the formulation of organic solvent blends with a significantly high fraction of DME, low salt concentration, or through the addition of fluorinated ethers [8,11]. All of these formulations lead to low viscosity and offer high ionic conductivity at low temperatures. More recently, the addition of a boron-based anion acceptor such as tris(1,1,1,3,3,3-hexafluoroisopropyl) borate and tris(pentafluorophenyl) borane into the electrolyte has been proposed to dissolve LiF, one of the discharge products of Li/CF_x batteries [11,14]. This approach has proven effective in increasing the utilization of the CF_x active cathode while providing little enhancement in the power capability. To meet the requirements for military batteries capable of operating at low temperature and at high power, the current electrolyte formulations need to be improved further.

Acetonitrile (AN) is an organic solvent with one of the highest dielectric constants, lowest melting points ($-45 \,^{\circ}$ C) and lowest viscosities (0.343 mPs at 25 $^{\circ}$ C), and it has been widely used as the solvent in the electrolytes for double-layer capacitors [15–17]. AN is chemically incompatible with metallic lithium where upon contact it immediately reacts to form hydrogen as shown by the reaction:

$$2\text{Li} + 2\text{CH}_3\text{CN} \rightarrow 2\text{Li}\text{CH}_2\text{CN} + \text{H}_2 \tag{1}$$

Surprisingly, we have found that metallic lithium becomes kinetically stable in AN when AN is mixed with an appropriate ratio of BL. This feature makes it possible to develop a low temperature electrolyte for Li/CF_x batteries. We studied the stability of metallic lithium in AN/BL electrolyte and examined the low temperature performance of Li/CF_x cells with such an electrolyte. In this paper

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we will report and discuss the enhanced power capability and low temperature performance of Li/CF_x cells.

2. Experimental

Carbon monofluoride (CF_x, x = 0.99-1.08, FluorStar Grade F, made from carbon fiber) was obtained from Lodestar, Inc., Howell, NJ. Solvents PC and DME (both electrolyte grade from Ferro), and salt LiBF₄ (Stella) were used as received. Prior to use, solvents AN and BL (both from Aldrich) were first dried over 4 Å molecular sieves for a week and then distilled by collecting fractions with the correct boiling point. In a glove-box, an electrolyte with a composition of 0.5 m LiBF₄ 1:1 (wt.) AN/BL was prepared along with the composition 0.5 m LiBF₄ 1:1 (wt.) PC/DME used as the control electrolyte.

A cathode film with a composition by weight of 85% CF_x , 9% carbon black (Aldrich), 1% carbon nanofiber (Pyrograf-III, Applied Sciences, Inc., Cedarville, OH) and 5% poly(vinylidene fluoride-cohexafluoropropylene)(Kynar FlexTM 2801, Elf Atochem North America) was prepared by making a slurry with N-methylpyrrolidone as the solvent and coating the slurry onto an Al foil. The coating was dried in an 80 °C-oven to evaporate solvent, and the resulting electrode film was punched into small disks with a diameter of 1.27 cm² for coin cell testing. Microstructure of the CF_x cathode film was observed using a Nikon Eclipse LV100 microscope equipped with a DXM1200F digital camera. The electrode disks were further dried at 100 °C under vacuum for 8 h, and then transferred into the glovebox for cell assembly. Li/CF_x coin cells were assembled by using Celgard[®] 3500 membrane as the separator and filled with 50 µL of electrolyte. Coin cells were discharged on a Maccor Series 4000 cycler with a 1.5 V cutoff voltage and a pre-determined C rate that was calculated from the weight and theoretical specific capacity $(865 \text{ mAh g}^{-1} \text{ for } x = 1) \text{ of } CF_x \text{ active material.}$

A Tenney Environmental Oven Series 942 was used to provide a constant temperature environment for the tests at various temperatures. An EG&G PAR Potentiostat/Galvanostat Model 273A was used to run cyclic voltammetry. To determine the electrochemical window of the electrolyte, a newly scratched Cu wire (for cathodic stability) and Al wire (for anodic stability), respectively, with a diameter of 1.0 mm was used as the working electrode by exposing a 1 cm length of wire to the electrolytic solution, and lithium foils were used as the counter and reference electrodes. The *I–V* response of the first two sweeps was recorded at a potential scanning rate of 5 mV s⁻¹. A Solartron SI 1287 Electrochemical Interface and a SI 1260 Impedance/Gain-Phase Analyzer was used for impedance measurements. Ionic conductivity of the electrolyte was calculated from the impedance of the solution measured using a two-platinum-electrode cell. Kinetic stability of metallic lithium in the AN/BL electrolyte was evaluated by monitoring impedance change of a three-electrode coin cell, in which three pieces of Li foil were used as the working, reference and counter electrodes. Details about the cell configuration and assembly were described elsewhere [18]. Impedance of the coin cells was potentiostatically measured at the open-circuit voltage (OCV) with an ac oscillation of 10 mV amplitude over the frequencies from 100 kHz to 0.01 Hz, in which the stable OCV was obtained by discharging the cell to a pre-determined depth-of-discharge (DOD) and then leaving the cell at open-circuit overnight.

3. Results and discussion

3.1. Electrode microstructure and electrolyte conductivity

It has been reported that the discharge of Li/CF_x cells is accompanied with serious cathode swelling, and that the expanded volume



Fig. 1. Microstructure of the CF_x cathode with a composition of 85% CF_x , 9% carbon black, 1% carbon filament, and 5% Kynar Flex 2801.

roughly equals the volume of LiF formed during discharge [19,20]. Although conductive carbon is formed in the discharge, this carbon is isolated or covered by very fine LiF particles and the electrode swelling inevitably causes the loss of electrode integrity. In order to increase electrical contact between the CF_x active material and conductive carbon and to retain electrode integrity, small amount of Pyrograf-III carbon nanofiber (having a nominal diameter of 70-200 nm and a length of $50-100 \,\mu\text{m}$) was added into the electrode composition. Shown in Fig. 1 is an optical microscope image of the CF_x cathode with a composition of 85% CF_x , 9% carbon black, 1% carbon nanofiber, and 5% binder. It can be seen that the thin and long carbon nanofibers are uniformly dispersed throughout the electrode. Such structures are helpful in remaining electrode integrity during discharge. We have found that the addition of carbon nanofibers increased the viscosity of the electrode slurry and enhanced the adhesion of the electrode layer to the current collector. Meanwhile, Li/CF_x cells with the additional 1–2% carbon nanofibers were shown to have less voltage delay at the beginning of discharge and had slightly higher utilization of the CF_x active material, as compared with the control cells.

In order to obtain low viscosity and to avoid salt precipitation at low temperatures, electrolytes with low salt concentration were used preferably in Li/CF_x cells. Fig. 2 shows ionic conductivity of two electrolytes at various temperatures in which the one with the composition of 0.5 m LiBF₄ 1:1 (wt.) PC/DME is a common formulation used in the commercial Li/CF_x cells (to be used as the control in this work), and the other one with the composition of 0.5 m LiBF₄ 1:1 (wt.) AN/BL is the subject of the present work. The reason for choosing an AN/BL solvent blend is because this combination has been extensively used in the state-of-art double-layer capacitors that are known to provide excellent low temperature performance. It can be seen from Fig. 2 that the AN/BL electrolyte has overwhelmingly higher conductivity than the control electrolyte over the whole temperature range. This advantage is mostly attributed to the high dielectric constant of AN. Due to freezing point depression from salt-dissolution and solvent-eutectic, the electrolyte could go to a much lower temperature without freezing than the melting points that AN and BL would suggest.

3.2. Kinetic stability of lithium in AN/BL electrolyte

The critical challenge for the use of AN/BL solvent in lithium batteries is the stability of metallic Li against AN. It is known that Li and AN are chemically incompatible. Metallic Li instantly reduces the α -hydrogen on AN to generate H₂ as soon as it contacts the



Fig. 2. Ionic conductivity of liquid electrolytes at various temperatures. (1) 0.5 m LiBF₄ 1:1 PC/DME, and (2) 0.5 m LiBF₄ 1:1 AN/BL.

AN solvent. However, this reaction can be effectively suppressed in the presence of an appropriate portion of BL. We dipped a piece of shiny Li foil into the 0.5 m LiBF₄ 1:1 AN/BL electrolyte, and did not find any visible changes on the surface of Li foil after 1 year storage at room temperature. This phenomenon can be attributed to the formation of a dense passivation film on the surface of metallic Li. To examine the formation and stability of the passivation film, we ran impedance measurements on a Li-Li-Li three-electrode cell. Fig. 3 plots impedance spectra of two Li foils (both having an area of $1.27 \,\mathrm{cm}^2$) with different storage time at $30 \,^{\circ}\mathrm{C}$ and $60 \,^{\circ}\mathrm{C}$, respectively. The impedance spectra are typically composed of two overlapped semicircles. The one in the high frequency region relates to passivation film and the other one in the low frequency region links to charge-transfer process occurring on the interface between the Li and the electrolyte. Fig. 3a shows that at 30 °C the size of both semicircles is slowly increasing with storage time. This behavior is similar to that observed in other Li-ion battery electrolytes [21], and it reflects the formation and growth of a passivation film on the surface of the Li metal. In the case of storage at 60 °C, as shown in Fig. 3b, a different behavior was observed. First, the spectra for 1 h and 2 h show an inductive characteristic in the low frequency end, which is an indication of chemical reaction on the Li-electrolyte interface. Second, the charge-transfer resistance becomes too large to present



Fig. 4. Discharge curves of Li/CF_x cells after 18 days storage (1) at room temperature and (2) at 60 °C, respectively. Electrolyte: 0.5 m LiBF₄ 1:1 AN/BL; discharge conditions: C/50 at 20 °C.

a semicircle for storage times of 5 h and 10 h. These results indicate that at $60 \,^{\circ}$ C the passivation film, although formed and grown as shown by the increase in the size of the high frequency semicircle in Fig. 3b, was unable to protect Li from the reaction with AN. Therefore, it may be said that metallic Li is kinetically stable in AN/BL electrolyte around ambient temperature.

The kinetic stability of metallic Li in AN/BL electrolyte was further confirmed by the storage testing of Li/CF_x cells. Fig. 4 compares discharge performance of two Li/CF_x cells that before test had been stored for 18 days at room temperature and at 60 °C, respectively. As seen in Fig. 4, Cell 1 that had been stored at room temperature released 883 mAh g⁻¹ capacity with the normal voltage range of a Li/CF_x cell, whereas Cell 2 that had been stored at 60 °C suffered significantly higher polarization and provided less capacity (785 mAh g⁻¹). This observation is in agreement with the results of impedance analyses, and again it can be attributed to the instability of metallic Li with AN at 60 °C.

Electrochemical stability of the AN/BL electrolyte was examined by cyclic voltammetry and linear potential sweep. The cathodic stability was determined by using a newly scratched Cu wire as the working electrode. Prior to Li plating at 0 V vs. Li⁺/Li, as shown in Fig. 5a, there are no major reduction current peaks except for some very small ones that are related to the formation of protective passivation film in the case of metallic Li. This means that the cathodic stability of AN/BL electrolyte is limited by the potential of Li plating, rather than by the reduction of AN. It should be noted that Coulombic efficiency for Li plating and stripping cycle was very low (i.e.,



Fig. 3. Impedance spectra of metallic lithium in a 0.5 m LiBF₄ 1:1 AN/BL electrolyte. (a) at 30 °C, and (b) at 60 °C. Li area = 1.27 cm².



Fig. 5. Electrochemical stability of a 0.5 m LiBF₄ 1:1 AN/BL electrolyte, which was measured at a potential scanning rate of 5 mV s⁻¹ (a) on Cu wire, in which the inset is an enlarged part of the 1st and 2nd cyclic voltammograms, and (b) on Al wire.



Fig. 6. Discharge performance of Li/CF_x cells with different electrolytes at C/50 and at 20 $^\circ$ C, (1) 0.5 m LiBF₄ 1:1 PC/DME, and (2) 0.5 m LiBF₄ 1:1 AN/BL.

2.4% in the 1st cycle and 6.7% in the 2nd cycle). This is probably because the newly platted Li immediately reacts with AN due to its high specific surface area. Since metallic Li is used as the anode in primary lithium batteries, this disadvantage would not affect the performance of Li/CF_x cells significantly. The anodic stability of the electrolyte was determined on an Al electrode, a material of the cathode current collector of Li/CF_x cells. Fig. 5b shows that the electrochemical (anodic) passivation of Al starts around 2.8 V vs. Li^+/Li , being lower than the open-circuit potential (normally 3.3–3.5 V) of CF_x cathode. This result reveals that Al current collector can be self-passivated in AN/BL electrolyte when it is used in the Li/CF_x cell. In addition, curve 2 in Fig. 5b indicates that the electrolyte is stable up to 4.5 V vs. Li^+/Li on the passivated Al surface. The results above



Fig. 7. Comparison of the discharge performance of Li/CF_x cells under different discharge conditions of C/10 at $20 \circ$ C (Region I), C/10 at $-50 \circ$ C (Region II), and C/50 at $-50 \circ$ C (Region III). (1) 0.5 m LiBF₄ 1:1 PC/DME in solid lines, and (2) 0.5 m LiBF₄ 1:1 AN/BL in dot lines.



Fig. 8. Temperature performance of Li/CF_x cells with 0.5 m LiBF₄ 1:1 PC/DME (1) and 0.5 m LiBF₄ 1:1 AN/BL electrolyte (2), respectively. In this experiment, the cell was discharged at C/20 for 5% theoretical DOD in each temperature step in the order of 20 °C, -50 °C, -40 °C, -30 °C, -20 °C, -10 °C, 0 °C, 10 °C, and 20 °C, and finally the cell was discharged to the end at 20 °C.

verify that AN/BL electrolyte is suitable for Li/CF_x cells if the cells are stored and operated at or below ambient temperature.

3.3. Enhanced discharge performance by AN/BL electrolyte

The effect of electrolyte on the discharge performance of Li/CF_x cells is shown in Figs. 6 and 7. At low discharge rate (*C*/50, as shown in Fig. 6), there were no differences in discharge voltage between the cells using AN/BL electrolyte and PC/DME electrolyte, and both cells approached the theoretical specific capacity (865 mAh g⁻¹ for CF_x with x = 1). When the discharge current was increased to C/10, however, the cell with AN/BL electrolyte provided higher voltage



Fig. 9. Dc-polarization curves of Li/CF_x cells with different electrolytes, which were measured at 20% theoretical DOD and at -50 °C by applying a 1.5 V dc-voltage to the open-circuit cells. (1) 0.5 m LiBF₄ 1:1 PC/DME, and (2) 0.5 m LiBF₄ 1:1 AN/BL.



Fig. 10. Impedance spectra of Li/CF_x cells with (1) 0.5 m LiBF₄ 1:1 PC/DME electrolyte, and (2) 0.5 m LiBF₄ 1:1 AN/BL electrolyte, which were measured at 20% theoretical DOD. (a) 20 °C, (b) -30 °C, and (c) -50 °C.

than the control cell as shown by Region I in Fig. 7. When the temperature was lowered to -50 °C with the discharge rate remaining unchanged, the AN/BL cell still showed some capacities while the control cell completely lost discharge capability (see Region II). In Region III the cells were discharged at C/50 and at -50 °C, in this case the AN/BL cell was obviously superior to the control cell in both the voltage and the capacity.

Temperature performances of the AN/BL and control cells are compared in Fig. 8 by using a constant discharge rate of C/20, in which the voltage spikes show initial voltage of each discharge step. In this experiment, 5% theoretical capacity was discharged in each step starting with 20°C and then stepping from -50°C to 20°C in 10°C intervals, and finally the cells were discharged to 1.5V at 20°C. It is shown that the AN/BL cell had higher discharge voltages in all temperature steps except for the first step at 20 °C. It is interesting to note that in the last discharge step at 20°C, the AN/BL cell showed higher voltage than the control whereas these two cells had the same voltage in the first step at 20 °C. The reason for this discrepancy has been unclear, however, we consider that it may be associated with the slow OCV recovery of Li/CF_x cells, which partially reflects the progress in the decomposition of graphite intercalation compound intermediate with solvated lithium fluoride [22]. The time interval between two temperature steps may not be long enough for the control cell to get full OCV recovery. The similar phenomenon also has been reported by Smart et al. [8] who showed that the cell reaction resistance (R_{cr}) of Li/CF_x cells, staying at higher value, could not recover to the original value after low temperature characterization. The superior low temperature performance of the AN/BL cell also can be verified by the dc-polarization experiment. Fig. 9 shows the current-time response of the AN/BL and control cells when a 1.5 V dc-voltage was applied to the open-circuit cell at -50 °C. In Fig. 9 the highest current corresponds to the maximum power that the cell can achieve. It can be seen that the current of the AN/BL cell right away reached a 12.6 mA maximum and then steeply declined, showing a typical polarization characteristic of Li batteries. The current of the control cell showed a small delay at the beginning of polarization, and it remained constant at a much lower value (2.2–1.2 mA).

In order to understand the performance improvement in the AN/BL electrolyte, impedances of the Li/CF_x cells at 20° C, -30° C, and -50 °C were measured and plotted in Fig. 10, in which all measurements were made on the cells with a 20% theoretical DOD. According to previous analyses [22], the impedance of a Li/CF_x cell is prominently contributed by the CF_x cathode. The depressed semicircle reflects cell reaction resistance (R_{cr}) of the CF_x cathode that is affected by Li⁺ ion diffusion (conductivity) in the discharge product shell and by charge-transfer process of the cell reaction. Whereas the intercept between the semicircle and real resistance (horizontal) axes at high frequency end represents a bulk resistance $(R_{\rm b})$ of the cell, including those of the current collector, electrode, separator and electrolyte. It can be seen from Fig. 10 and the inset that the cell with AN/BL electrolyte has much lower R_b and R_{cr} than the control cell. By comparing the insets in Fig. 10a-c, we further find that the reduction in $R_{\rm b}$ by the AN/BL electrolyte becomes more significant with a decrease in the temperature. This observation is attributed to the high ionic conductivity of the AN/BL electrolyte. Since the $R_{\rm b}$ at low temperatures, for example at $-50\,^{\circ}$ C as shown in Fig. 10c, is substantially smaller than the R_{cr}, we may further contribute the performance improvement at low temperatures by the AN/BL electrolyte to the reduction in the R_{cr} , i.e., to the enhanced kinetics of the CF_x cell reaction.

4. Conclusions

The results of this work show that the kinetic stability of metallic Li with AN can be obtained by combining AN and BL, based on the formation of a protective passivation film on the surface of metallic Li. The high dielectric constant and low viscosity of AN makes it possible to formulate an electrolyte having high ionic conductivity at low temperatures. The use of AN/BL electrolyte

provides an excellent solution to the high power capability and improved low temperature performance of Li/CF_x cells. Impedance data shows that the Li/CF_x cells with AN/BL electrolyte had much lower bulk resistance and cell reaction resistance. Due to the chemical incompatibility of metallic Li and AN, the storage and operation temperature for the Li/CF_x cells using a 0.5 m LiBF₄ 1:1 AN/BL electrolyte are limited to or below ambient temperature (30 °C). Future work for increasing the storage and operation temperature of Li/CF_x cells can be focused on optimizing the ratio of AN to BL and on searching for other electrolyte solvents or additives that are more powerful in promoting the formation of a protective passivation film on the metallic Li surface.

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