The energy density of lithium-ion cells can be significantly increased by the use of silicon-containing negative electrodes. However, the long-term performance of these cells is limited by the stability of the silicon-electrode–electrolyte interface, which is continually disrupted during electrochemical cycling. Therefore, the development of electrolyte systems that enhance the stability of this interface is a critical need. In this article, we examine the cycling of ~20 mAh pouch cells with lithium bis(fluorosulfonyl)imide (LiFSI)-containing carbonate-based electrolytes, silicon-graphite negative electrodes, and Li_{1.03}(Ni_{0.5}Co_{0.2}Mn_{0.3})_{0.97}O_{2} based positive electrodes. The effect of fluoroethylene carbonate (FEC) and vinylene carbonate (VC) addition on cell performance is also examined and compared to the performance of our baseline LiPF_{6}-containing cells. Our data show that cells containing only LiFSI show rapid loss of capacity, whereas additions of FEC and VC significantly improve cell capacity retention. Furthermore, the performance of LiFSI-FEC and LiPF_{6}-FEC cells are very similar indicating that the electrolyte salts play a much smaller role in performance degradation than the electrolyte solvent. Future efforts to enhance longevity of cells with silicon-graphite negative electrodes will thereby focus on developing alternative solvent systems.

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electrolytes. Shkrob et al. have indicated that reduction of the FSI− anion results in inorganic SEI phases without the concurrent generation of organic radicals and gaseous products. This mineralized SEI continually cracks and reforms during Li+ deposition causing uniform growth on the Li metal surface instead of the metal whiskers that form in the SEI of conventional carbonate electrolytes.23

The research literature contains numerous half-cell studies of silicon-based electrodes with small mass loadings; the cyclability of electrodes with high mass loadings in a full-cell configuration is rarely described. Therefore in this article, we report the performance of full cells with LiFSI-bearing carbonate-based electrolytes, silicon-graphite negative electrode (also referred to as Si-G) and Li1.03(Ni0.5Co0.2Mn0.3)0.97O2-based positive electrodes (also referred to either as oxide or NCM523). The silicon-graphite electrode was developed at Argonne as a drop-in replacement for conventional graphite-based electrodes; it is physically robust, highly uniform, and very flexible even at capacity loadings exceeding 3 mAh/cm². The NCM523 electrode and the LiFSI-based electrolytes were prepared at Argonne from commercially-available constituents. The performance of LiFSI-containing cells is compared to performance of our baseline LiPF6-containing cells. Such investigations are important steps toward developing electrolytes that enable high-performance, high-energy-dense, long-lasting silicon-containing batteries.

### Experimental

**Materials.**—All electrodes in this study are from Argonne’s Cell Analysis, Modeling, and Prototyping (CAMP) Facility; details on constituents and composition of these electrodes are shown in Table I. The positive electrode contains Li1.03(Ni0.5Co0.2Mn0.3)0.97O2 as the active constituent; the C45 carbon, added to enhance the electrode’s electronic conduction, displays negligible electrochemical activity at voltages < 4.5 V vs. Li/Li+. In contrast, the C45 carbon is electrochemically active at potentials experienced by the negative electrode.24 However, the negative electrode capacity is mainly determined by the graphite (73 wt%) and nanosilicon (15 wt%) content. The positive electrode binder is PVdF (polyvinylidene fluoride), which provides both coating cohesion and adhesion to the current collector. The negative electrode binder is partially-lithiated polyacrylic acid (Li-PAA), prepared by titrating polyacrylic acid (Sigma Aldrich, average Mv ~450,000) with LiOH (Sigma Aldrich). This Li-PAA binder enables the fabrication of mechanically robust electrodes, even at relatively high active-material loadings, as indicated earlier.

All electrolytes (see Table II) were prepared in-house in a glove box filled with nitrogen (< 1 ppm O2, H2O). To prepare the formulations, ethylene carbonate (EC) and ethyl methyl carbonate (EMC), both from BASF, were pre-mixed in 30/70 w/w% ratio. Then the salt, either LiFSI (Sarchem Laboratories) or LiPF6 (Strem), was added to the solvent, the mixture was stirred 1 h at room temperature, filtered through 0.2 μm PTFE filter and the volume of the filtrate was adjusted to obtain 1.2 M salt concentration. FEC (Solvay) or VC (BASF) was added, if applicable, to the above mixture.

**Pouch cell assembly and testing setup.**—The full cell tests were conducted in single-layer xx3450-type pouch cells (see Figure 1) assembled in a dedicated climate-controlled dry-room with a dew point less than −42℃ (< 100 ppm moisture).26 These stack-type cells consist of two manually-aligned electrode layers (one single-sided positive and one single-sided negative electrode) separated by a single sheet of Celgard 2325 (PP/PE/PP). The electrodes are dried overnight at 120℃ (positive) and 150℃ (negative) prior to cell assembly. The positive electrode has an average coating area of 14.1 cm² and the negative electrode has an average coating area of 14.9 cm²; the larger negative electrode area reduces the probability of lithium plating during electrochemical cycling. An aluminum tab is welded to the positive and a nickel tab is welded to the negative electrode for connection to the external circuit; both tabs are 7 mm wide and 100 μm thick. The electrode stack is placed in a pouch container and a side area heat sealer is used to seal 3 sides of the pouch; a tab area heat sealer is used to prevent leaks at the tab locations. The assembly is then dried overnight at 60℃.

The electrolyte of interest is added by pipette to the single open edge of the pouch in 0.5 mL aliquots. The pouch is then subjected to two 18 second vacuum and air-refill stages in a vacuum sealer to minimize air pockets and enhance electrode wetting by the electrolyte. After these soak and degassing steps, the final side of the pouch is heat-sealed while the cell is under vacuum. At this stage one side of the pouch contains an extra region that can fill with gases that may be generated during formation cycling.

For the electrochemical tests, the cells are fixed to rigid, nonconductive boards with female banana plugs in 2 rows of 2 cells per fixture layer. Standard stainless steel plates, xx3450-sized acrylic sheets, indicate areas heat sealed

**Table I. Electrode composition and nomenclature used in this article.**

| Electrolyte Composition | Nomenclature |
|-------------------------|--------------|
| (Solvent + 1.2 M LiFSI) | LiFSI        |
| (Solvent + 1.2 M LiFSI) + 10 wt% VC* | LiFSI-VC |
| (Solvent + 1.2 M LiFSI) + 10 wt% FEC* | LiFSI-FEC |
| (Solvent + 1.2 M LiPF6) + 10 wt% FEC* | LiPF6-FEC |

*VC or FEC was added after preparation of solvent/salt mixture.

**Table II. Electrolyte composition and nomenclature used in this article. For all electrolytes the solvent used is EC:EMC (3:7 w/w).**

| Electrolyte Composition | Nomenclature |
|-------------------------|--------------|
| (Solvent + 1.2 M LiFSI) | LiFSI        |
| (Solvent + 1.2 M LiFSI) + 10 wt% VC* | LiFSI-VC |
| (Solvent + 1.2 M LiFSI) + 10 wt% FEC* | LiFSI-FEC |
| (Solvent + 1.2 M LiPF6) + 10 wt% FEC* | LiPF6-FEC |

*VC or FEC was added after preparation of solvent/salt mixture.

### Figure 1. Schematic of pouch cell used in our studies. An “X-ray” view of the electrodes is shown for clarity. The negative electrode is 32.4 mm wide and 46.0 mm high; the positive is 31.3 mm wide and 45.0 mm high.
springs, and wing nuts are used to apply added localized stack pressure to the cells; the initial stack pressure is estimated to be 15 kPa for each pouch cell. All cycling tests are performed at 30°C using a MACCOR Series 4000 Test System; the cycling procedures are detailed in Table III. In essence, the cells initially undergo formation cycling and then are subjected to Performance Test A or B as shown in Table III to obtain capacity retention and impedance rise information. The extra region of the pouch cells is sometimes inflated after formation because of gases generated during cycling. Such inflation was not observed in our cells probably because of the small electrode area-to-cell volume ratio of the single-layer pouch cell design. Therefore, the extra region was left intact and the cells proceeded with performance testing. The electrochemical cycling was conducted in the 3.0–4.1 V range. The upper cutoff voltage (UCV) was limited to 4.1 V to minimize the likelihood of Al current-collector pitting that is known to occur at 4.2 V. The lower cutoff voltage (LCV) was set at 2.5 V; however, as indicated previously, cell lifetimes decrease when the LCV is reduced to 2.5 V. Higher efficiencies, around 90%, are obtained when the LCV is lowered to 2.5 V; however, as indicated previously, cell lifetimes decrease when the LCV is reduced to 2.5 V.

Coin cell assembly and testing.—The full cell data were complemented by half-cell capacity data obtained in 2032-type coin cells (1.6 cm² area electrodes). These cells contained the Si-Gr electrode, Celgard 2325 separator, and a Li-metal counter electrode. Each electrolyte listed in Table II was examined mainly to determine features associated with solid electrolyte interphase (SEI) formation. These cells were cycled in the 1.5–0.0 V vs. Li/Li⁺ range with a 0.085 mA/cm² current at room temperature.

Results and Discussion

As indicated previously, multiple cells containing identical chemistries were tested under the conditions shown in Table III. Negligible performance differences were seen for cells with identical chemistries. Therefore, only representative data are shown in this article to highlight aging trends and differences between various cell chemistries.

Initial cycles.—The first cycle capacity-voltage profiles for the NCM523/Si-Gr pouch cells containing the LiFSI, LiFSI-VC, LiFSI-FEC, and LiPF₆-FEC electrolytes are shown in Figure 2. At first glance, the cycling behavior for the various cells appears similar. A closer look at the 1.8–3.4 V charge curve (see inset, Figure 2) reveals distinct inflections in the profiles, especially for the FEC-containing cells. More specifically, the LiPF₆-FEC cell shows a brief drop in voltage at ~2.64 V before the voltage starts rising again. The LiFSI-FEC cell does not show this voltage drop; however, its profile deviates from that of the LiFSI cell around 2.4 V. The LiFSI-VC cell profile also shows a small deviation from that of the LiFSI cell around 2.8 V. The inset table shows that charge capacity of the LiFSI cells decreases slightly with VC and FEC addition. However, the discharge capacity and coulombic efficiency (CE) are marginally better for the LiFSI-VC cell. Note that the coulombic efficiencies for all cells are around 80%. Higher efficiencies, around 90%, are obtained when the LCV is lowered to 2.5 V; however, as indicated previously, cell lifetimes decrease when the LCV is reduced to 2.5 V.

Figure 3 shows differential capacity profiles for the data shown in Figure 2. The peaks observed in the 3.3–4.1 V range are mostly associated with lithiation and delithiation of the various electrode components in both the positive and negative electrodes. The peaks in the 2.2–3.2 V range (see inset, Figure 3) corresponds to the early onset voltage-profile inflections discussed in Figure 2, and their peak

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**Table III. Cycling procedures. Indicated C-rates are approximate.**

| Test Parameters | Initial Cycles | Performance Test A | Performance Test B |
|----------------|----------------|--------------------|--------------------|
| Formation Cycling | 3 cycles, 3.0–4.1 V, 0.07 mA/cm² | 94 cycles, 3.0–4.1 V, 0.47 mA/cm² | HPPC – 10s pulses, 4.26 mA/cm² |
| Tap Charge | *0.14 mA/cm² (C/10) to 1.5 V, then 15 minute hold at 1.5 V | (C/3) | charge, 3.19 mA/cm² (2.25C) charge |
| Rest | Open circuit, 12 h | discharge | (3C) |
| Aging Cycles | 3 cycles, 3.0–4.1 V, 0.07 mA/cm² | 94 cycles, 3.0–4.1 V, 0.47 mA/cm² | HPPC – 10s pulses, 4.26 mA/cm² |
| Final Cycles | 3 cycles, 3.0–4.1 V, 0.07 mA/cm² | 3 cycles, 3.0–4.1 V, 0.47 mA/cm² | (C/3) |

*cm² refers to area of positive electrode.
Our experiments show that this sign-change is seen for all LiPF_{6}-FEC cells containing negative electrodes with >5 wt% silicon, but is not observed for electrodes based solely on graphite; (iv) additional peaks at 0.85 V and 0.92 V are seen in the LiFSI-FEC and LiPF_{6}-FEC data, which suggests further solvent reduction; (v) additional peaks (not shown), with intensities that are two orders of magnitude smaller than the main peaks, are seen in the 1.8–2.8 V range. These initial reduction reactions likely contribute to SEI structure on the electrodes, but are not explored here because SEI formation mechanisms are beyond the scope of this manuscript.

Additional details from cycling the Si-Gr/Li cells in the 1.5–0.0 V range are shown in Table IV. It is evident that the irreversible capacity (lithiation minus delithiation) for cycle 1 is greater for the cells containing VC and FEC, which is consistent with greater lithium trapping during SEI formation in the presence of these additives. On the other hand, the irreversible capacity for cycle 2 is similar for all cells; in addition, the values are much smaller than those for cycle 1 indicating that the major SEI formation processes occur during the 1st cycle, as expected. The coulombic efficiency, which is also a measure of lithium trapping, also increases for cells from ~91–93% in cycle 1 to ~99% in cycle 3 (data not shown).

**Extended cycles – Capacity fade.**—The behavior of the NCMS523/Si-Gr full cells, containing the LiFSI, LiFSI-VC, LiFSI-FEC and LiPF_{6}-FEC electrolytes, over 100 cycles is shown in Figure 5. In this figure cycles 1–3 and 97–100 are at a ~C/20 rate, where C refers to the initial C/1 capacity of the cells, cycles 4–94 are at a ~C/3 rate. The initial discharge capacities of the various cells are similar; at the 3rd cycle, the capacities are between 136 (LiFSI-FEC) to 140 (LiFSI-VC) mAh/g, where g refers to oxide content of the positive electrode. The CE’s at the 3rd cycle are 98.6% (LiFSI-VC), 98.4% (LiFSI-FEC), 98.3% (LiPF_{6}-FEC data) and 97.1% (LiFSI); i.e., the efficiencies are lowest for LiFSI, but are in the same range for the other cells.

On further C/3 cycling, the capacity of all cells decline (see Figure 5a). The fastest and slowest capacity decline is observed for the LiFSI and LiFSI-VC cells, respectively. The LiFSI-FEC and LiPF_{6}-FEC cells show an intermediate capacity decline; the capacities for these cells at the 100th cycle are similar. Table V shows capacity retention at the 100th cycle relative to the 1st cycle. The retention decreases as follows: 61.4% (LiFSI-VC), 58.1% (LiFSI-FEC), 56.1% (LiPF_{6}-FEC data), 18.7% (LiFSI), which is similar to the 3rd cycle CE trend shown above. The capacity retention is consistent with the CE values (at C/5 rate) with cycle number (see Figure 5b). In all cases, the CE is highest initially, shows a steady decrease, then rises again, i.e., it shows a “hammock” trend. For example, CE values for the LiFSI-VC cell are 99.5%, 99.2% and 99.5% at cycles 4, 50 and 97 respectively. The corresponding values for the LiFSI-FEC and LiPF_{6}-FEC cells are 99.4%, 99.0%, 99.1% and 99.2%, 98.9% and 99.1% respectively. In contrast, the values for the LiFSI cell are 98.5%, 94.5% and 97%-

### Table IV. Capacity data from Si-Gr/Li coin cells cycled in the 1.5–0.0 V range.

| Electrolyte | Cycle 1 | Cycle 2 |
|-------------|---------|---------|
| LiFSI       | 98.2    | 98.4    |
| LiFSI-VC    | 98.6    | 98.4    |
| LiFSI-FEC   | 98.3    | 97.1    |
| LiPF_{6}-FEC| 97.1    | 99.0    |

* g refers to grams of active material in the Si-Gr electrode.
The expansion/contraction of the Si-Gr electrode during lithiation/delithiation results in breakdown/reformation of the SEI layer thereby increasing the amount of lithium immobilized by these SEI-forming side-reactions. Electrolytes that yield SELs with higher inorganic (more mineralized) content are expected to show a lower CE because of continual cracking and reformation of the SEI. On the other hand, electrolytes that form an elastomeric SEI, which is more resistant to cracking, will show higher CE values. This explanation is in agreement with the above CE data, which is lowest for the LiFSI (89.4% at C/20) electrolyte, which is expected to form a more mineralized SEI. The values are higher for the LiFSI-FEC (98.3% at C/20), LiPF<sub>6</sub>-FEC (98.4% at C/20) and LiFSI-VC (99.0% at C/20) cells because the VC and FEC compounds are expected to form cross-linked elastomers in the SEI during electrolyte reduction.<sup>22</sup>

In addition to lithium-consuming side reactions in the negative electrode SEI, cell capacity fade can result from factors that include phase changes in the electrode active materials, isolation of active material particles through loss of electronic or ionic conductivity, and depletion of electrode active material through dissolution into the electrolyte. The NCMS23 electrode is not expected to be a major contributor to capacity fade because it does not display any major phase changes, or dissolve to any significant extent, in the voltage range examined. In contrast, nanosilicon particle loss and a reduction in electronic connectivity are known contributors to capacity fade in the Si-Gr electrode, especially in cells that lack VC or FEC in the electrolyte. Taking all factors into consideration we surmise that capacity fade in our cells arises at the negative electrode. Capacity retention is enhanced by the electrolyte additives (VC, FEC) that improve coating cohesion and lessen lithium-consuming side reactions at the negative electrode.

Extended cycles – Impedance rise.— For vehicular applications, in addition to excellent capacity retention, battery cells need to sustain high current pulses under rapid discharge and charge conditions without degrading significantly. This ability is often determined by the hybrid pulse power characterization (HPPC) tests, in which cell impedance is determined over its useable charge and voltage range.<sup>23</sup> In a typical HPPC test, the cell is charged to its UCV (4.1 V, here), then subjected to repetitions of a pulse profile that contains constant-current discharge (3C, here) and charge (2.25C, here) pulses, followed by 10% depth of discharge (DOD) constant-current C/1 discharge segments, each followed by a 1h rest period.

The area specific impedance (ASI) data for the various pouch cells, obtained by using the HPPC protocol, is shown in Figure 6. ASI values at 3.5 V and 3.7 V, interpolated from the measured data, are listed in Table V. The data indicate that initial cell ASI's are quite similar, especially in the voltage range of 3.4–4.0 V. For example, at 3.7 V, the ASI values range from 32 (LiFSI) to 31 (LiFSI-VC); i.e., the values are the lowest for LiFSI and highest for the LiFSI-VC cell. Cell impedances are higher after the 100 charge-discharge cycles. We were unable to determine ASI values for the LiFSI cell because of its excessive capacity loss. For the other cells, however, the ASI values at 3.7 V are 24 (LiFSI-VC), 47 (LiFSI-FEC), 44 (LiPF<sub>6</sub>-FEC data). Although these data lie within a relatively narrow ASI band, Table V indicates the impedance increase is lowest for LiFSI-VC and highest for the LiFSI-FEC cell.

The causal mechanisms leading to impedance rise in these cells are yet to be explored. However, initial experiments in cells containing similar electrolytes and a Li-I-Sn reference electrode indicate that cell impedance rise mainly arises at the positive electrode; contribution of the negative electrode to impedance rise is, surprisingly, small.<sup>22</sup> The impedance rise at the positive electrode can result from various factors consistent with its rapid capacity decline; the sharp peaks in this dataset, which indicates a higher charge-to-discharge ratio probably results from occasional fracturing and reformation of the negative electrode SEI layer. Finally, for all cells, the CE for the 100th cycle, which is at ~C/20 rate, is lower than the CE for the prior C/3 cycles, and is likely related to the longer measurement times for the C/20 cycles compared to that for the C/3 cycles.
to enhance cell lifetimes will focus on designing alternative solvent systems.

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Conclusions

The electrochemical performance of cells containing Li1.03(Ni0.5Co0.2Mn0.3)0.97O2–based positive electrodes, 15 wt% nanosilicon–bearing negative electrodes, and LiFSI–based electrolytes were examined in the 3.0–4.1 V cycling range. The resulting area specific impedance (ASI) vs. cell voltage for NCM523//Si–Gr pouch cells containing the LiFSI, LiFSI-VC, LiFSI-FEC, and LiPF6–FEC electrolytes, before (filled symbols) and after (open symbols) the aging cycles. The ASI data shown were obtained with a 5C discharge pulse, where C refers to the initial C/10 capacity of the cells.

Figure 6. Area specific impedance (ASI) vs. cell voltage for NCM523//Si–Gr pouch cells containing the LiFSI, LiFSI-VC, LiFSI-FEC, and LiPF6–FEC electrolytes, before (filled symbols) and after (open symbols) the aging cycles. The ASI data shown were obtained with a 5C discharge pulse, where C refers to the initial C/10 capacity of the cells.

that hinder the motion of lithium ions. These factors include the following: (a) microcrack development in the oxide particles; (b) crystal structure changes at the oxide-electrolyte interface; (c) decrease in ionic conductivity of electrode surface films; (d) degradation of electronic pathways between the carbon matrix and the oxide active material; and (e) deterioration of electrolyte transport properties within the pores of the electrode coating. Prior experiments have suggested that the oxidation products of VC create a barrier layer that minimizes degradation of the positive electrode by the electrolyte. On the other hand, FEC is highly resistant to oxidation and is not likely to form a passivation layer at the positive electrode-electrolyte interface. This difference may explain the lower impedance rise for the LiFSI-VC cell. Further electrochemical and physicochemical experiments are needed to identify the appropriate mechanistic scenarios that lead to cell performance degradation. These experiments are currently in progress and their results will be detailed in future articles.

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