Investigation of Gas Evolution from Li₄Ti₅O₁₂ Anode for Lithium Ion Batteries

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Lithium titanate (LTO) has been investigated as one of the leading anode materials for lithium ion batteries in grid storage and automotive applications. However, one of the primary challenges is cell gassing which can significantly limit life of cells despite the excellent lifetime performance of LTO anodes. Gas evolution has been previously attributed to water impurities from the electrolyte, moisture trapped in the electrode, the breakdown of lithium salt forming hydrofluoric acid (HF), and/or solvent reactions with the surface of the electrode. The role of electrolyte in gas evolution has been investigated during formation, high temperature storage, and high temperature cycling. The effect of LiTFSI, LiFSI, EC-free formulations, and a novel LTO electrolyte additive (tris(trimethylsilyl)borate) have been investigated. Incorporation of an EC free electrolyte or a novel electrolyte additive result in a significant reduction in gas generation. Analysis of the gas composition suggests that the majority of the gas results from solvent reactions with the LTO surface while the reactions of the residual water are a secondary source of gassing.

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Lithium-ion batteries have garnered significant attention for use in electric and hybrid electric vehicles due to their high energy density. While lithium titanate (Li₄Ti₅O₁₂, LTO) has a lower theoretical capacity of 175 mAh g⁻¹ compared to graphite (372 mAh g⁻¹), it is an attractive anode material for vehicle applications due to its long cycle life performance, superior safety due to the 1.55 V working potential, and very small volumetric lattice variation during charging and discharging.1–4 While being regarded as an interesting anode material,5 LTO suffers from excessive gas evolution at elevated temperatures resulting in premature cell failure.5–8

Many investigations of gas evolution by LTO have been conducted and there are three primary sources proposed for gas evolution. First, water contamination from electrolyte contamination or residual hydroxyl groups on the surface of the LTO from production have been suggested to be a source for hydrogen. Finally, interfacial reactions between the surface of the LTO and the electrolyte solvents has been suggested to be a source for hydrogen. Interfacial reactions between the surface of the LTO and the electrolyte solvents has been reported to be another source of gas evolution for LTO electrodes.6–15 Recent reports suggest that the majority of gas formed from LTO results from the solvents reacting with the surface of the LTO particles resulting in decarboxylation, decarbonylation, and dehydrogenation of the electrolyte solvents.15 While previous reports have provided some insight into gas evolution in LTO systems, the effect of electrolyte formulation on gas evolution in LTO/lithium manganese oxide (LMO) pouch cells has not been thoroughly investigated. Therefore, a detailed investigation of the effects of different electrolyte formulations of cell gassing has been conducted. The investigation includes electrolyte formulations replacing the LiPF₆ salt with imide salts, introducing an acid scavenging additive, testing an EC-free formulation, and introducing a novel LTO SEI additive, in an effort to better understand the gassing mechanism in LTO/LMO pouch cells at high temperature.

**Experimental**

**Materials.**—The cathode active material is commercially available BTBM lithium manganese oxide and the anode is commercially available POSCO Li₄Ti₅O₁₂. The 920 mAh dry cells, multilayer pouch cells (MLPC), were assembled by SKC Powertech. Battery grade ethylene carbonate (EC), propylene carbonate (PC), diethyl carbonate (DEC), lithium hexafluorophosphate (LiPF₆), lithium bis(trifluoromethanesulfonyl)imide (LiTFSI), and lithium bis(fluorosulfonyl)imide (LiFSI) were obtained from BASF and used as received. Electrolyte additives dimethylacetamide (DMAc) and tris(trimethylsilyl)borate (TMSB) were obtained from Sigma Aldrich and used as received. All electrolyte formulations were battery grade with water and HF concentration below 30 ppm.

Six primary formulations were used for cell testing with three alternate formulations used during the electrolyte storage screening process. The following are the formulations used for cell testing:

- The baseline electrolyte (Baseline) is 1.0 M LiPF₆ EC/PC/DEC (15/20/65, v).
- Acid Scavenging electrolyte (DMAc): Baseline + 0.25% DMAc.
- LTO Surface film electrolyte (TMSB): Baseline + 1% TMSB.
- LiTFSI Salt Replacement electrolyte (LiTFSI): 1.0 M LiTFSI in EC/PC/DEC (15/20/65, v) + 5% LiPF₆.
- LiFSI salt replacement electrolyte (LiFSI): 1.0 M LiFSI in EC/PC/DEC (15/20/65, v) + 5% LiPF₆.
- EC-free electrolyte (PC): 1.0 M LiPF₆ in PC/DEC (35/65, v)

During the electrolyte storage test, three other formulations were investigated to determine the best acid scavenger formulation. The following formulations are the alternate acid scavenging formulations that were screened:

- 1% LiTFSI: Baseline + 1% LiTFSI.
- LiTFSI + DMAc: Baseline + 1% LiTFSI + 0.25% DMAc.
- LiTFSI: consisted of 1.0 M LiTFSI EC/PC/DEC (15/20/65, v)

**Electrolyte storage testing.**—Storage experiments were conducted on the electrolytes to investigate the thermal stability of the electrolyte. Each formulation was prepared in a large batch and transferred into small aluminum air tight bottles in a nitrogen filled glove box. Triplicates from each batch were stored and analyzed to ensure reproducibility. The baseline was established by testing the first sample directly after blending.

The HF was measured using a Metrohm titrator with Tiamo software and a Metrohm double platinum wire 6.0341.100 pH electrode. About 50 grams of crushed ice formed from deionized (DI) water and 60 grams of DI water were added to a Teflon beaker. The ice was used to delay the hydrolysis of LiPF₆ to generate additional hydrofluoric acid (HF) and other acidic species, which is accelerated in acid formation, high temperature storage, and high temperature cycling. The effect of LiTFSI, LiFSI, EC-free formulations, and a novel LTO electrolyte additive (tris(trimethylsilyl)borate) have been investigated. Incorporation of an EC free electrolyte or a novel electrolyte additive result in a significant reduction in gas generation. Analysis of the gas composition suggests that the majority of the gas results from solvent reactions with the LTO surface while the reactions of the residual water are a secondary source of gassing.

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- 1% LiTFSI: Baseline + 1% LiTFSI.
- LiTFSI + DMAc: Baseline + 1% LiTFSI + 0.25% DMAc.
- LiTFSI: consisted of 1.0 M LiTFSI EC/PC/DEC (15/20/65, v)
Electrolyte storage at 45 °C.—The results of screening electrolytes for HF inhibition via 4 weeks of storage at 45 °C are provided in Figure 1. The concentration of HF in ppm is depicted as a function of time. The fresh, initial levels of HF show that the carbonate solvent blend without LiPF₆ salt has no HF present while the baseline electrolyte has 25 ppm of HF present indicating that LiPF₆ is the main source of HF. The DMAc electrolyte and the LiTFSI + DMAc electrolyte have no initial HF present suggesting that the DMAc successfully inhibits HF generation in the baseline due to LiPF₆ decomposition.18,19 Compared to the baseline, the HF content for 1% LiTFSI electrolyte decreased, but did not inhibit the HF step at 60 °C for 24 hours at 100% SOC. After the second aging, cells were discharged and allowed to reach 25 °C before measuring the gas volume. Cells were placed into the 60 °C chamber under tight clamping to be cycled between 2.8 V and 1.7 V at 1C with a C/10 cycle every 50th cycle. Every 300 cycles the cycling was suspended and the volume of the cells were measured at room temperature. After the volume measurements, cells were returned to the same cycling procedure for another 300 cycles.

All cells were prepared in triplicates to confirm reproducibility for all tests conducted.

Gas analysis.—Gas volume was measured before the first formation, after the first formation, after aging, before storage and/or cycling, and after storage and/or cycling according to the procedure described by Aiken et al. The pouch cells were hung from the bottom of scale and tarred. After reaching a stable zero, the cells were submerged completely to a defined level in 25 °C deionized water. The recorded weight of the cell while submerged was then used along with the Archimedes’ principle to calculate the amount of gas evolved over time.17

To measure the composition of gasses, cells were brought into the argon dry box for extraction. A 0.5 mL Vici precision sampling analytical pressure-lok syringe was used to manually extract the gas sample from the cell under argon atmosphere. The sample was then manually injected into a Varian 450 gas chromatograph equipped with a 19808 ShinCarbon ST column, thermal conductivity detector (TCD), and an argon carrier gas.

Results and Discussion

Concentration of HF detected throughout the course of storage at 60 °C with the Baseline, baseline + 1% LiTFSI, baseline + 0.25% DMAc, baseline + 1% LiTFSI + 0.25% DMAc, and 1.0 M LiTFSI in EC/PC/DEC.

Figure 1. Concentration of HF detected throughout the course of storage at 60 °C with the Baseline, baseline + 1% LiTFSI, baseline + 0.25% DMAc, baseline + 1% LiTFSI + 0.25% DMAc, and 1.0 M LiTFSI in EC/PC/DEC.
formation entirely. In contrast, the DMAc and LiTFSI + DMAc electrolytes successfully inhibit HF formation for up to two weeks. After the initial two weeks, HF is observed suggesting that the DMAc is consumed and no longer inhibits HF formation from LiPF6 decomposition. Interestingly, a synergistic effect is seen when combining LiTFSI and DMAc allowing for an extra week of storage before HF generation is observed. As expected, there was no HF observed after 4 weeks of storage of the solvent blend without LiPF6, suggesting that impurities in the solvents are not a source of HF. The 60°C storage testing suggests that 0.25% DMAc can inhibit HF formation for several weeks.

Formation gas investigation.—Cells were filled with electrolyte formulations shown in Figure 2 to test inhibition of gas formation in LTO cells. The quantity and composition of gas evolved for cells containing the different electrolyte formulations during formation are provided in Figure 2. After formation, including the aging step, the volume of gas in the cells was measured. The baseline electrolyte evolved 3.7 mL of gas. Comparatively, the DMAc electrolyte evolved 4.9 mL, TMSB electrolyte evolved 2.5 mL, LiTFSI electrolyte evolved 3.4 mL, LiFSI electrolyte evolved 9.5 mL, and the PC electrolyte evolved 3.6 mL of gas. The baseline formation gas has a composition that contains 2.8 mL of H2, which accounts for 77% of the gas formed. The remaining components of the gas are carbon monoxide (15%, 0.5 mL), carbon dioxide (4.9%, 0.2 mL), ethylene (2.6%, 0.1 mL), and methane (1.1%, <0.1 mL). The DMAc electrolyte not only increases the total amount of gas formed, but it also increases the amount of hydrogen by 1.4 mL. However, the DMAc electrolyte does not generate any carbon dioxide or ethylene. The TMSB electrolyte reduces the amount of hydrogen by 0.5 mL, reduces carbon monoxide by 0.3 mL, and eliminates the carbon dioxide. The TMSB electrolyte is the only formulation that reduces the total quantity of gas and the amount of hydrogen gas evolved. While the LiTFSI electrolyte was able to reduce the total amount of gas evolved slightly, the composition is very similar to the Baseline. The LiTFSI electrolyte severely increases the gas evolved and increases the hydrogen gas produced by 4.9 mL. All other gases are also increased, but this is the only formulation which also produces ethane (0.2%, <0.1 mL). The PC electrolyte slightly reduces the total formation gas and eliminates carbon dioxide and ethylene, but produces more hydrogen than the baseline.

High temperature storage testing.—Cells were stored at high temperature (60°C) for one week followed by volume measurements and gas analysis. The gas measured and analyzed during this step results exclusively from storage since the cells were degassed after the formation phase. Pouch cells were degassed after formation and stored at 60°C for 1 week at 100% SOC. After storage, gas was measured, extracted, and injected into a GC-TCD for analysis. The quantity and composition of the gas evolved upon storage is depicted in Figure 3. The baseline produces 6.9 mL of gas composed primarily of hydrogen. The predominant components of the gas evolved from the baseline electrolyte include 4.7 mL of hydrogen, 0.9 mL of carbon monoxide, 0.7 mL of carbon dioxide, and 0.4 mL of ethylene. The gas evolved from the DMAc electrolyte has 4.1 mL greater volume than the baseline electrolyte indicating that inhibiting HF generation with DMAc does not solve the gassing issue. The DMAc electrolyte produces 7.8 mL of hydrogen. All other gaseous components were also increased compared to the baseline electrolyte. The TMSB electrolyte was the only formulation that reduced the gas evolved upon storage with only 6.5 mL of gas produced. The composition of the TMSB electrolyte gas has a large reduction in carbon dioxide and ethylene, however, the quantity of hydrogen remains the same and the carbon monoxide increases compared to the baseline. Both salt replacement formulations have increased gassing with the LiTFSI electrolyte displaying a 2.0 mL increase and the LiFSI electrolyte displaying a 4.2 mL increase in gas evolution. The PC electrolyte also increased the gassing, but the unique increase in propene gas suggests that PC is not an appropriate replacement for EC and was used in too high of concentration. Overall the only electrolyte to show gassing benefits upon storage at 60°C was the TMSB electrolyte containing the surface film forming additive. Removing the LiPF6 salt or inhibiting HF evolution were not effective methods to reduce gassing.

Understanding the source and reducing cell gassing in LTO systems is important, but cell performance cannot be sacrificed. The remaining and recovered capacity is provided in Figure 4 to depict the influence each electrolyte formulation has on performance compared to the baseline electrolyte. The baseline electrolyte has 91.7% remaining capacity and 96.5% recovered capacity. All electrolyte formulations show a reduction in remaining and recovered capacity other than the LiTFSI electrolyte. The DMAc electrolyte has 88.1% remaining and 95.5% recovered capacity, the TMSB electrolyte has 85.6% remaining and 91.1% recovered capacity, the LiTFSI electrolyte has 94.5% remaining and 99.0% recovered capacity, the LiFSI electrolyte has 78.8% remaining and 91.4% recovered capacity, and the PC electrolyte has 83.3% remaining and 94.4% recovered capacity. Overall,
Gas Analysis After 1 Week of 60°C Storage

![Graph showing volume of gas (mL) for different electrolytes.]

**Figure 3.** Average volume and gas composition of pouch cells after 1 week of storage at 60°C with the Baseline, 1.0 M LiTFSI in EC/PC/DEC + 5% LiPF₆, 1.0 M LiFSI in EC/PC/DEC + 5% LiPF₆, baseline + 1% TMSB, 1.0 M LiPF₆ in PC/DEC, and baseline + 0.25% DMAc.

Remaining and Recovered Capacity % After 1 Week 60° C HTS

![Graph showing remaining and recovered capacity percentage for pouch cells.]

**Figure 4.** Remaining and recovered capacity percentage for pouch cells after 1 week of storage at 60°C (100% SOC) with the Baseline, baseline + 0.25% DMAc, 1.0 M LiTFSI in EC/PC/DEC + 5% LiPF₆, baseline + 1% TMSB, 1.0 M LiPF₆ in PC/DEC, and 1.0 M LiFSI in EC/PC/DEC + 5% LiPF₆.

The recovered capacity for all formulations is above 90%, but the remaining capacity show steady losses with the LiFSI electrolyte being the worst. While the LiFSI electrolyte has the lowest remaining capacity and the most gassing, the TMSB electrolyte has the least gassing and third worst remaining capacity. Similarly, the PC electrolyte has the second lowest remaining capacity, but the third lowest gassing behind the TMSB electrolyte and the baseline. Comparing the performance to the gassing does not lead to a linear correlation, but rather indicates that LiFSI is not suitable for this system. In addition, TMSB is a good LTO surface film forming additive which inhibits gassing, but leads to some non-gas producing side reactions and reduced capacity. In contrast, LiTFSI increases the performance, but has the third worst gassing. Therefore, there is not direct correlation between gassing and reduced capacity in the LTO system, and gassing and non-gassing reactions should be seen as separate conditions to address. However, it is important to note that the capacity retention for all cells is reasonable for most applications and cell gassing remains the largest barrier for most LTO cells.

**High temperature cycling.**—High temperature cycling was conducted at 45°C and the specific capacity is displayed in Figure 5. At 300 cycles, cells are paused and taken out for gas volume measurement before being restarted. The baseline electrolyte has 95.2% retention after 600 cycles. Performing slightly worse than the baseline electrolyte, the DMAc electrolyte has 92% retention and the TMSB electrolyte has 93.4% retention. The LiFST electrolyte and the LiFSI electrolyte have retentions of 93.4% and 87.1%, respectively. The LiFSI electrolyte is the worst of all formulations and the only formulation to go below 90% confirming that LiFSI electrolytes are not suitable for LTO cells. The only formulation to display better performance than the baseline is the PC electrolyte with 96.4% capacity.
retention after 600 cycles. Due to the strong performance of the PC electrolyte, we can conclude that EC is not necessary for this system.

The volume of gas has been measured for all cells after 300 and 600 cycles as displayed in Figure 6. After 300 cycles, the cell containing the baseline electrolyte produced 4.9 mL of gas. By 600 cycles, cells cycled with the baseline electrolyte generated a total of 6.6 mL of gas. The DMAc electrolyte produces significantly more gas than the baseline, 10.7 mL after 300 cycles. The quantity of gas generated for the DMAc electrolyte after 600 cycles is by far the most of all formulations precluding accurate measurement. The TMSB electrolyte again successfully reduces the evolved gas by 1.2 mL compared to the baseline electrolyte. This data also confirms that replacing the LiPF₆ salt or using DMAc to inhibit HF formation does not improve cycling performance or inhibit gas evolution. Analysis of the gas composition of the cycled cells has unfortunately been problematic and cannot be reported at this time.

Conclusions

The source of gas evolution from LTO electrodes in LTO/LMO pouch cells has been investigated via high temperature storage and high temperature cycling. The source of gas evolution is very complicated. The role of impurity levels of H₂O and HF have been investigated through use of DMAc as an acid inhibiting additive. The results suggest that LiPF₆ is the source of HF in electrolytes and that DMAc can inhibit HF generation from LiPF₆ electrolytes until the additive has been consumed. Despite HF inhibition by DMAc, electrolyte formulations containing DMAc have the most gas evolution performance limitation. This data also confirms the necessity of DMAc as an acid inhibiting additive.
during cycling and second most gas evolution during high temperature storage. Incorporation of DMAC also provides no performance benefits suggesting that HF and water impurities in the electrolyte are not the main source of gassing. The role of the electrolyte reactions with the surface of the LTO has been investigated through the LTO surface film forming additive, TMSB, and EC-free electrolyte formulations. Both formulations reduce the amount of gas generated upon formation and high temperature cycling. Electrolytes containing TMSB also reduce the gassing after high temperature storage. The results suggest that generation of a surface film on the LTO is an effective way of decreasing the gas formed in the LTO system, and that TMSB is an effective surface film forming additive. Along with the benefits of TMSB, EC-free electrolytes have superior cycling performance indicating that the presence of EC is detrimental to the performance of LTO anodes. The change in gas volume, gas composition, and cell performance suggests that the main sources of gassing in LTO are reactions between the electrolyte and the electrode surface. It is also important to note that a direct correlation between the gassing and performance is not clear suggesting a complex combination of gas forming and non-gas forming reactions. Ongoing work will focus on the individual solvent reactions with the LTO surface. In addition, new additives are being investigated and ex-situ surface analysis is being used to determine the optimal surface film for LTO electrodes to minimize gas evolution while maintaining capacity retention.

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