

Battery Cell Leak Testing

Multiple testing methods are herein presented to quantitatively, deterministically and non-destructively leak test prismatic or cylindrical lithium-ion battery cells. **At this time no test method has been codified for finding small leak channels in the battery cells.** While the minimum detection limit of the available test methods has yet to be determined, the methods detailed in this paper have been demonstrated to effectively find leaks that are common in automated cell manufacturing lines. **The nature, cause and approximate size of the defects are described in this paper along with the methods shown to be effective in testing for them.**



Die idealen Turbop
für leichte Gase

Introduction

The rapid growth in demand in the hybrid and EV automotive market has sparked the pressing need for high performance batteries. Rechargeable lithium-ion batteries are currently the most established and widely used battery technology for this market. Lithium-ion batteries are also being used for solar energy storage in homes and in the electrical grid, in industrial machinery, in aerospace, and in consumer goods.

The risk of leaking batteries causing fires due to moisture ingress or electrolyte leakage presents a significant safety and financial risk to the public and battery producers, and is one of the remaining hurdles to more rapidly integrating the technology into the automotive and in-home electricity storage markets. Due to the increasing size and energy density of next-generation lithium-ion batteries, the consequences of battery fires to life and property are increasingly becoming headline news, so steps are necessary to identify and mitigate these risks as early in the production process as possible.

Safety concerns aside, the costs of cell failures to battery manufacturers can be exorbitant. Scrapped battery cells and lost production resulting from shutting down entire production lines to locate and resolve the root cause of the leaks is a major concern. There are currently no standardized methods used in the battery industry to find electrolyte leaks at the cell level. Anecdotal reports from several large-scale battery producers indicate that these leaks are frequently found when the electrolyte is seen leaking from the cell, or when the distinctive smell of electrolyte is noted in the production facility.

A properly designed leak detection system enables the manufacturer to minimize both modes of lost revenue by quickly performing in-line vacuum testing of cells to check for the presence of leaks, and verifying that the leak is an electrolyte egress leak rather than a leak in the test system.

The study which yielded the results described below was performed in cooperation with the University of Michigan Battery Lab.

Introduction to University of Michigan Battery Lab provided by proprietor Greg Less

The University of Michigan Battery Lab is a campus research core offering academic and industrial users from around the globe the expertise and resources required to prototype, test and analyze batteries and the materials that go into them. The lab's aim: work with the industrial and academic energy storage community to bring together scientists and engineers, as well as suppliers and manufacturers, to ease a bottleneck in battery development near the nation's automotive capital. The lab is available for any firm or researcher to use, and is a safe zone for IP-protected discovery, scale 10^{4-6} , and testing of next-generation batteries and battery materials.

The Battery Lab, part of the Michigan Materials Research Institute, was developed by U-M in cooperation with the Michigan Economic Development Corporation and Ford Motor Company.

For the leak testing experiment, a series of 30 pouch cells were made; ten were made without deliberate flaws, ten were made with bad tab seals, and ten were made with incomplete side seals. The cells (72 x 110 x 2.5 mm, approximately 3.5 Ah) were representative of current state of the art pouch cells, manufactured in a Z-fold stack with 8 NMC 111 cathode layers and 9 graphite anode layers, separated with Entek EPH 16 μm polyethylene separator film. The electrodes were stock electrodes coated and calendered on roll-to-roll processing machines. As the cells were not intended for cycling, the electrode loadings were not precisely balanced.

The “good” cells were made with no purposefully introduced defects during the tab welding or bag sealing steps. The “bad tab seal” cells were made to represent a cell in which the ultrasonically welded, 15 mm x 0.1 mm extension tab is misaligned with the heat-sealed bag edge, causing an incomplete or missing seal around the tabs. This was achieved by loading the tabs into the ultra-sonic welder in reverse so that the thermoplastic strip (which assures a good seal between the tab and the pouch) was outside of the heat-seal area. A seal can be achieved between the pouch material and the tab directly, but is much more prone to leaks than a seal that has been made between the pouch and the thermoplastic strip.

The “bad side seal” cells were made by placing an 8 mm x 0.1 mm aluminum shim between the layers of the pouch material during heat sealing, which was subsequently removed, leaving an \sim 8mm unsealed area along the side seal. This defect was made to simulate a failing heater cartridge in the sealing machine which may result in a partial complete seal, with partial open areas.

The cells were filled with an electrolyte solution of 1M LiPF₆ in 1:1 ethylene carbonate: ethyl, methyl carbonate from Soulbrain USA. The ten good cells and ten bad tab seal cells were filled with 10 g of electrolyte solution each, the bad side seal cells were filled with 7.5 g of electrolyte solution each, before vacuum and final sealing operations. The trimmed cells were leak checked prior to any sort of tap charge, or formation cycling.

Test Technologies Used

Mass Extraction- The preliminary leak test is a mass extraction test using a Pfeiffer Vacuum ME4 mass extraction instrument. Mass extraction utilizes a vacuum pump to evacuate a chamber containing the test article, and measures the flow rate from the chamber to a vacuum reservoir. Flow rates can be correlated to determine the size of the defect in the unit under test. In this study, the flow values from the bad samples very quickly exceeded the full scale of the sensor, so no correlation was possible.

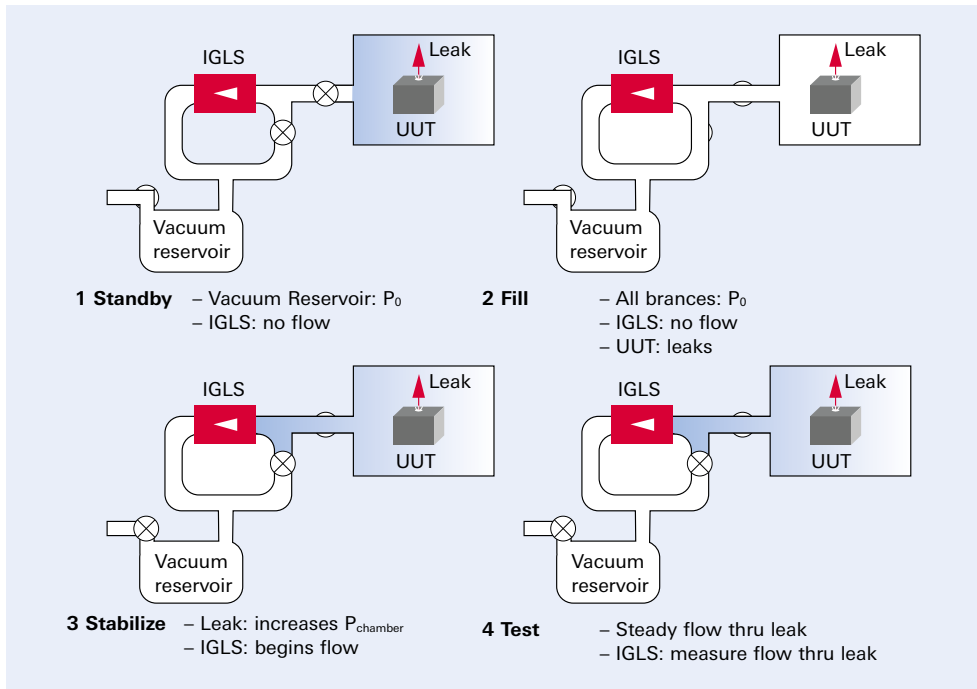


Figure 1: Mass Extraction Diagram

Omnistar

The OmniStar is a self-contained gas analysis system able to measure gasses and vapors up to 300 amu. This compact mass spectrometer-based analyzer includes a 1 or 2 meter heated sampling probe to enable gas analysis from atmospheric pressure down to 10 Torr. Results are available in real-time with continuous measurement speeds in the sub 1 second range.



Figure 2: Omnistar

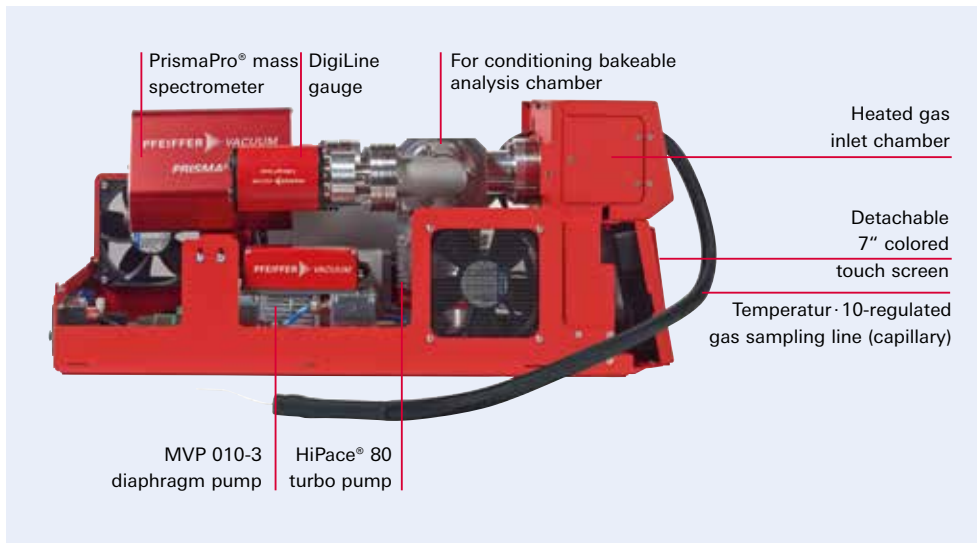


Figure 3: Omnistar explosion illustration

Samples Tested

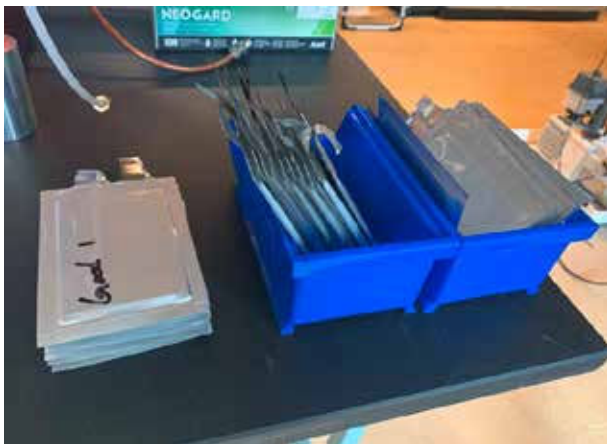


Figure 4: Test Samples: Good, Bad Tabs, Bad Seals

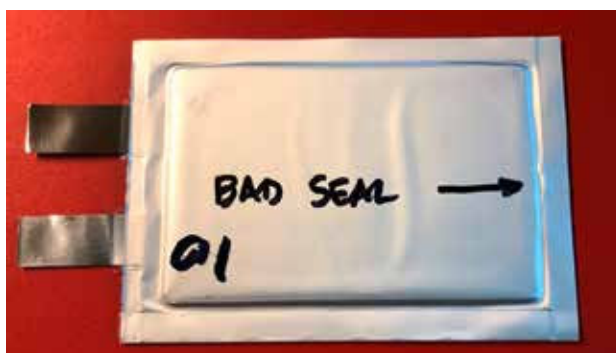


Figure 5: Bad Seal Sample

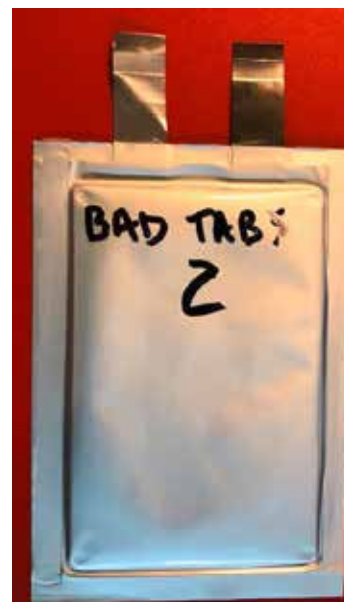


Figure 6: Bad Tab Sample



Figure 7: Test Apparatus

Preliminary Findings:

- Sniffing bad cells at barometric condition did not detect the presence of electrolyte in known leaking samples.
- Placing the cells inside a chamber and evacuating causes the electrolyte to evaporate and leave the cell through the defect.
- This evaporated electrolyte was then detectable with the instruments described above.

Test Procedure:

1. Place cell in test chamber
2. Evacuate test chamber for 15 seconds
3. Dwell 30 seconds after evacuation ends
4. Record sniffer measurement
5. Purge vacuum
6. Record sniffer measurement

Test Results

The initial test was a measurement of the control negative samples. The mass extraction test of the control samples showed an average background flow measurement of 16.7 micrograms per minute. A retest of the same samples with a 5-micron calibrated leak added to the test yielded an average of 42.0 micrograms per minute of flow. These were the results of the fine leak measurement at the end of a 40 second test. The control positives that were manufactured with the tab and seal defects all either failed the gross leak check or very quickly went to the full scale of the sensor and the tests were ended in 15 seconds.

The Omnistar background measurement of the control negatives averaged an ion current of $4.8 \cdot 10^{-12}$ A of the 59 AMU signal, corresponding to a constituent of the electrolyte solvent 30 seconds after the conclusion of the mass extraction test, and $3.6 \cdot 10^{-11}$ A immediately after the vacuum in the test chamber was purged. The control positives with the bad tabs averaged $8.2 \cdot 10^{-10}$ A 30 seconds after the conclusion of the Mass Extraction test and $6.8 \cdot 10^{-8}$ A immediately after purging the vacuum. The control positives with the bad seals averaged $7.5 \cdot 10^{-10}$ A 30 seconds after the conclusion of the mass extraction test and $5.5 \cdot 10^{-8}$ A immediately after purging the vacuum chamber.

Control Negatives	Fine Leak Measurement (µg/min)
1	19.1
2	16.8
3	15.8
4	19.8
5	15.5
6	16.7
7	17.1
8	15.4
9	15.5
10	15.2
Ave	16.7

Table 1: Mass Extraction Control Negatives

Negatives + 5u	Fine Leak Measurement (µg/min)
1	41.4
2	40.2
3	40.4
4	42.4
5	39.6
6	41.0
7	55.6
8	39.3
9	40.4
10	39.9
Ave	42.0

Table 2: Mass Extraction Control Negatives + 5-micron calibrated leak

Positives Bad Tab	Test Result (µg/min)
1	Gross Leak
2	Gross Leak
3	Gross Leak
4	Gross Leak
5	Gross Leak
6	Gross Leak
7	Gross Leak
8	Full Scale
9	Gross Leak
10	Gross Leak

Table 3: Mass Extraction Test of Bad Tab Control Positives

Positives Bad Seal	Test Result (µg/min)
1	Gross Leak
2	Gross Leak
3	Gross Leak
4	Gross Leak
5	Gross Leak
6	Gross Leak
7	Gross Leak
8	Gross Leak
9	Gross Leak
10	Gross Leak

Table 4: Mass Extraction Test of Bad Seal Control Positives

Negatives + 5u	Ion Current (A), 30s delay	Ion Current (A), after purge
1	$3.0 \cdot 10^{-11}$	$1.0 \cdot 10^{-10}$
2	$6.0 \cdot 10^{-12}$	$3.0 \cdot 10^{-11}$
3	$3.0 \cdot 10^{-12}$	$3.0 \cdot 10^{-11}$
4	$2.0 \cdot 10^{-12}$	$3.0 \cdot 10^{-11}$
5	$2.0 \cdot 10^{-12}$	$5.0 \cdot 10^{-11}$
6	$1.0 \cdot 10^{-12}$	$3.0 \cdot 10^{-11}$
7	$1.0 \cdot 10^{-12}$	$3.0 \cdot 10^{-11}$
8	$1.0 \cdot 10^{-12}$	$2.0 \cdot 10^{-11}$
9	$1.0 \cdot 10^{-12}$	$2.0 \cdot 10^{-11}$
10	$7.0 \cdot 10^{-13}$	$2.0 \cdot 10^{-11}$
Ave	$4.8 \cdot 10^{-12}$	$3.6 \cdot 10^{-11}$
ST Dev	$9.0 \cdot 10^{-12}$	$2.4 \cdot 10^{-11}$
Ave+6 STDEV	$3.2 \cdot 10^{-11}$	$1.1 \cdot 10^{-10}$

Table 5: OmniStar Test of Control Negatives

Positives Bad Seal	Ion Current (A), 30s delay	Ion Current (A), after purge
1	$1.5 \cdot 10^{-10}$	$6.0 \cdot 10^{-9}$
2	$1.0 \cdot 10^{-9}$	$7.0 \cdot 10^{-8}$
3	$1.0 \cdot 10^{-9}$	$8.0 \cdot 10^{-8}$
4	$1.0 \cdot 10^{-9}$	$7.0 \cdot 10^{-8}$
5	$1.9 \cdot 10^{-10}$	$2.0 \cdot 10^{-8}$
6	$7.0 \cdot 10^{-10}$	$5.0 \cdot 10^{-8}$
7	$1.0 \cdot 10^{-9}$	$7.0 \cdot 10^{-8}$
8	$7.0 \cdot 10^{-10}$	$5.0 \cdot 10^{-8}$
9	$8.0 \cdot 10^{-10}$	$6.0 \cdot 10^{-8}$
10	$1.0 \cdot 10^{-9}$	$7.0 \cdot 10^{-8}$
Ave	$7.5 \cdot 10^{-10}$	$5.5 \cdot 10^{-8}$
ratio	158	1517

Table 7: OmniStar Test of Bad Seal Control Positives

The very high ratios of measurement between the control negatives and the control positives provides confidence that bad parts will be failed correctly and that false failures will be eliminated.

Combining these two technologies provides a robust solution to the costly and unsafe problem of leaking battery cells, and minimizes assembly line down time. Contact Pfeiffer Vacuum today to discuss how these leak detection technologies can help prevent scrap and production downtime.

Positives Bad Tab	Ion Current (A), 30s delay	Ion Current (A), after purge
1	$7.0 \cdot 10^{-10}$	$6.0 \cdot 10^{-8}$
2	$8.0 \cdot 10^{-10}$	$7.0 \cdot 10^{-8}$
3	$9.0 \cdot 10^{-10}$	$7.0 \cdot 10^{-8}$
4	$9.0 \cdot 10^{-10}$	$7.0 \cdot 10^{-8}$
5	$5.0 \cdot 10^{-10}$	$6.0 \cdot 10^{-8}$
6	$1.0 \cdot 10^{-9}$	$7.0 \cdot 10^{-8}$
7	$1.0 \cdot 10^{-9}$	$8.0 \cdot 10^{-8}$
8	$6.0 \cdot 10^{-10}$	$6.0 \cdot 10^{-8}$
9	$8.0 \cdot 10^{-10}$	$6.0 \cdot 10^{-8}$
10	$1.0 \cdot 10^{-9}$	$8.0 \cdot 10^{-8}$
Ave	$8.2 \cdot 10^{-10}$	$6.8 \cdot 10^{-8}$
ratio	26	627

Table 6: OmniStar Test of Bad Tab Control Positives

Conclusion

Mass Extraction is a test method capable of identifying battery cells that have defects common in high-volume cell manufacturing. It should be noted that a failed test could be due to test chamber leakage, or moisture on the outside of the cell. False fails are undesirable because the cost of shutting down an assembly line due to a false failure is exorbitant.

The OmniStar gas analysis test eliminates concerns of unnecessary lost production by rapidly verifying that a failed mass extraction test is due to electrolyte leaking out of the

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