Current Battery Safety Research Projects at Underwriters Laboratories



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Background / Overview

- Lithium-ion cells and batteries today, provide power in a wide variety of applications from consumer electronic, automotive and aerospace to stationary grid energy storage.
- With the increase in demand, millions of cells and thousands of batteries are manufactured every month.
- The challenge is to screen and match every individual cell.
 ✓ Typical COTS and some custom battery manufacturing process does not include cell screening and matching (aerospace may be a small exception)
- ✓ Cells are assembled into batteries in the 'as received" condition at lower SOC (typically 30 40%)
- Questions such as, "Are assembled batteries **tested under relevant stringent** conditions before they are sent out into the field?", arise.
- This has been a major concern for certain sectors such as the shipping and transportation industry.
 - IATA estimated approximately 3 billion cells (Li-ion and Li-metal) were shipped by air in 2008
- This led the International Civil Aviation Organization (ICAO) to set temporary bans in transporting Li-based cells and batteries as cargo in passenger and cargo aircraft, with a restriction on the state-of-charge (SOC) of a lithium-ion cell or battery to not exceed 30%.
- Projects carried out at UL relate to understanding the safety of li-ion cells that relate in many ways to understanding the worst case results under off-nominal conditions and related to the various standards working groups that we are part of.



Thermal Runaway Propagation Study of Li-ion Modules

Carried out at Stress Engineering and NASA-JSC



Background on ICAO/SAE Efforts

The Council of the International Civil Aviation Organization (ICAO) established a prohibition on the transport of lithium batteries as cargo on passenger aircraft as a temporary measure until controls were put into place which establish an acceptable level of safety. A performance-based packaging standard was identified as one of the controls.

- ICAO's intent to write a performance based packaging standard was declared in late 2015; SAE International was chosen to lead this effort as an SAE standard.
- G-27 Committee formed (>250 participants).
- Standard writing team formed in early 2016 (20 members); started effort in March 2016; now continuing with larger team of interested members of up to 50 per meeting.

"Performance based package standard for lithium batteries as cargo on aircraft" (AS6413)

This SAE Aerospace Standard (AS) specifies a minimum performance package standard that supports the safe shipment of lithium batteries as cargo on aircraft.

Technical Approach

- The intent of this test is to **severely abuse a single cell** such that it is most likely to enter **thermal runaway** with the presumption that a single cell may enter thermal runaway during transport.
- This test describes a method to **evaluate the package capability to limit the consequences of a single cell in thermal runaway** from becoming hazardous. The pass/fail criteria establishes a minimum performance requirement for the package.
- The test method and pass/fail criteria identified to **demonstrate acceptable performance** was chosen to minimize the number of tests needed for a particular cell/packaging combination (package).
- Under certain severe failure conditions, lithium-based rechargeable cells can **emit gases which may be harmful to humans and/or may form a combustible mixture in sufficient concentrations**. Examples may include, but are not limited to, carbon monoxide (CO), CO₂, organic solvent vapors and hydrogen fluoride (HF).
- The pass test criteria shall include evidence of **non-hazardous flame**, **non-hazardous fragment**, **non-hazardous vapor**, **package integrity and repeatability** as described in this section (the team is still working on allowable max external surface temperature and duration also)

Sub-assembly test for large batteries is being considered.



Sample Test Setup at FAA Tech Center

Current Study

Tested single (3.4 Ah 18650) Li-ion cells in 25 cell (5X5) arrangement with the following variables

- SOC: 100 % SOC; 3 % SOC
- Heater dimensions: 2"X2"; 1"X2"
- Heater thickness: Thick heater (0.014"); Thin heater (0.012")
- Location of trigger cell Center, corner and side wall (edge)
- Temperature:
 - Max temp. 392 °F (200 °C)
 - Temperature held at 392 °F for one hour
 - Temperature increased until thermal runaway
- Heating Rate
 - 19 20 °F / min rate of heating
 - 7 8 °F / min rate of heating
 - 3 4 °F / min rate of heating
- Package/Box Configuration
 - Lid Open and Closed Box



- Witness panels were made of cardboard covered with cheesecloth placed 1 inch away from each side of the box.
- Total of 35 tests (33 on 3.4 Ah 18650 and 2 on 3.4 Ah pouch format li-ion cells) performed to date. Judith Jeevarajan / Underwriters Laboratories Inc.

Thermal Runaway Propagation Test 25 Cell (3.4 Ah) at 100 % SOC and Center Cell Trigger







Observations: Trigger cell vented, laterPost-Testwent into thermal runaway with fire and
propagated, all cells went into thermal
runaway; No smoke was observed outsideudith Jeevarajan / Underwriters
Laborable box until thermal runaway6

Thermal Runaway Propagation Test 25 Cell (3.4 Ah) at 3 % SOC and Center Cell Trigger









Observations: Trigger cell vented and later underwent thermal runaway, no propagation, moderate damage. Trigger cell voltage:0V, adjacent damaged cell:0.737V, rest of the cell voltages:3.36V A lot of smoke was observed outside

the box, but no fire.

Thermal Runaway Propagation Test 25 Cell (3.4 Ah) at 3 % SOC and Corner Cell Trigger

Open Box

Test Stopped after temp reached 392 ° F and was held for one hour





Test on PTCs from Various Lots



Modeling studies with Prof. Jain at UT-Arlington to understand the Anomalous behavior

Random cells from each lot were tested. Other cells from problematic lot are being tested now.

ID#	LDC	Pre-OCV	Weight (g	Resistance (mohms	Capacity (Ah)	Manufacturing Country		
2_1	P-LDC3A-561 50W	3.62	45.63	58	3	China		
2_2	P-LDC3A-561 50W	3.62	45.75	45	3.02	China		
2_3	P-LDC3A-561 50W	3.62	45.57	53	3.02	China		
2_4	P-LDC3A-561 50W	3.62	45.68	47	3.05	China	Range	45-70
2_5	P-LDC3A-561 50W	3.62	45.71	. 70	3.06	China		
2_6	P-LDC3A-561 50W	3.62	45.6	64	3.08	China		
3_1	P-LDC3B-56190W	3.62	45.63	46	3.01	China		

Judith Jeevarajan ésistan dévalues indicating issues with the cells 9 Laboratories Inc.



Test Setup for G27 tests using similar packaging configurations







Summary

- No difference in performance of thick or thin heaters; thick heater did not require additional tape to hold it in place, hence all tests were carried out with thick heater.
- The 2"X1" heater not different from the 2"X2" heater tape but the 2"X1" heater simulated local heating better. Questions arose as to whether the larger heater caused additional heating of adjacent cells. The smaller heater allows for cell heat to radiate to adjacent cells in a more consistent manner.
- 7-8 ° F / min heating rate was found to be optimal for consistent test results.
- Lot to lot variation observed especially with cells made by another manufacturer (in a different country) for the OEM.
 - Cells should be screened well before testing to remove cell lots (batches) that could result in anomalous results.
- The Open box test showed that during the thermal runaway process, electrolyte is released for cells at low SOC, which can then catch on fire or smoke heavily, causing the propagation to the inserts in the box as well as the other cells.
 - Previous studies have indicated that once-activated PTCs can be the cause of failure if activated a second time. Data from Purdue Univ testing shows problematic PTCs.
 - Studies by JHUAPL have also indicated that the presence of gases such as dimethyl carbonate over hot cells can lead to ignition and cell thermal runaway
- This type of thermal runaway propagation was only observed with the third lot of cells which were made for the OEM in a different country.
- For the configurations studied, the center cell seemed to be the worst location for the first batch but for the second and third batch of cells, the corner and side wall location seemed worse. This may depend on the arrangement of the cells, the inserts, spacing between the cells (with the inserts), etc.
- Testing should be done in the relevant configuration and may need to be performed independently when cells with the same model number are manufactured in different manufacturing facilities, especially different countries.

Working with Prof. Ankur Jain at UT-Arlington on modeling the thermal runaway propagation tests.



UN Informal Working Group

- Tests are being carried out by various test research organizations using an agreedupon test plan
- Single cell triggered to thermal runaway; six cells in a row; voltage and temperatures
 monitored and recorded
 UN Series 1, Test 1, Six (6) 18650s, 100% SOC, 9-11-19

Voltage (+)

/oltage (-)

• All cells at 100% SOC Top View

Side View

Test 1





Cylindrical Cells

UN Informal Working Group – contd.. Pouch Cells



State of Charge vs Safety Study



Thermal Runaway using Heating Method

- 40W Kapton heater was used to initiate thermal runaway
- 1" x 2" 20W/in² or
- 2" x 2" 10W/in²
- Heating rate was maintained at 10 °F/min until thermal runaway occurred or until the cell voltage fell to 0V
- Cells were subjected to thermal runaway test at 6 different states-of-charge -100%, 50%, 40%, 30%, 15%, and 0% (3 samples under each test condition)
 Cells from 6 different manufacturers were tested
 - Manufacturer: A, B,C, D, E, and F
 - Label on cells indicate that manufacturer for A & B were the same, however, A was purchased at the nominal cost from trusted vendor and B cells were inexpensive and purchased online (~\$2/ cell)
- Pouch cells were restrained during the heating tests

Cell Design	Cathode Chemistry	Rated Capacity (mAh)	Actual Capacity (mAh)	Internal Resistance (mΩ)
18650	NCA	3200	3250	45
18650	NCA	3200	1850	35
26650	NMC	5000	5011	<20
Pouch	NMC	3300	3220	<15
26650	LFP	2500	2500	6
Pouch	LFP	10000	10300	<10
	Cell Design 18650 18650 26650 Pouch 26650 Pouch	Cell DesignCathode Chemistry18650NCA18650NCA26650NMCPouchNMC26650LFPPouchLFP	Cell DesignCathode ChemistryRated Capacity (mAh)18650NCA320018650NCA320026650NMC5000PouchNMC330026650LFP2500PouchLFP10000	Cell DesignCathode ChemistryRated Capacity (mAh)Actual Capacity





Heating Method - Manufacturer A (18650, 3.2 Ah, NCA)



Heating Method - Manufacturer A (18650, 3.2 Ah, NCA)



Summary of Results for Manufacturer A (18650, 1.8 Ah, NCA)

State of Charge (%)	Venting Temperature (°F)	Venting Time (min)	Thermal Runaway Onset Temperature (°F)	Onset Time (min)	Maximum Temperature (°F)	Observations
100	245	16.5	335	23	1095	Fire and smoke
50	270	21	365	29	1200	Fire and smoke
40	240	15.5	275	19	1325	Fire and smoke
30	265	19	380	25	875	Fire and smoke
15	265	19	415	33	800	Smoke
0	290	22	N/A	N/A	455	No fire or smoke







100%SOC

15%SOC

Summary of Safety vs SOC using the Heating Method

- Voltage behavior
 - Cell voltage drops incrementally (first to ~2V and then to 0V at TR) with cylindrical cells that have a PTC except for the low-cost cell (Manuf B) that did not have a PTC or CID; although designated with same manufacturer name as type A.
- Venting temperature
 - Most of the cells followed similar trends, where temperature at which venting occurs increases (↑) as SOC goes down (↓)
 - Except for Manufacturer E and F (both LFP type) venting temperature is around the same value
- Thermal Runaway onset temperature increases (\uparrow) as SOC goes down (\downarrow) except for manufacturer B (low cost cells).

Cell Type	% SOC								
	100	50	40	30	15	0			
A – 18650 NCA	Thermal Runaway with Fire and Smoke	Thermal Runaway with Fire and Smoke	Thermal Runaway with Fire and Smoke	Thermal Runaway with Fire and Smoke	Thermal Runaway with Smoke	-			
B- 18650 NCA	Thermal Runaway with Fire and Smoke	Thermal Runaway with Smoke	Thermal Runaway with Smoke	Thermal Runaway with Smoke	-	-			
C – 26650 NMC	Thermal Runaway with Fire and Smoke	Thermal Runaway with Fire and Smoke	Thermal Runaway with Smoke	Thermal Runaway with Smoke	Thermal Runaway with Smoke				
D – Pouch NMC	Thermal Runaway with Fire and Smoke	Thermal Runaway with Smoke	Thermal Runaway with Smoke	-	-	-			
E – 26650 LFP	Thermal Runaway with Smoke	Thermal Runaway with Smoke	Thermal Runaway with Smoke	-	-	-			
F – Pouch LFP	Thermal Runaway with Smoke	-	-	-	-	-			



External Short

- External short was carried out on cells that do not contain the internal PTC device. This includes pouch cells or high power cells (see table below).
- The load was held for 3 hours, or until thermal runaway.
- Shunt resistance used for the short was 8-10 mohms.
- Pouch cells were restrained and tabs reinforced with Ni tabs.
- Cells were subjected to external short at 6 different states-of-charge (SOC) -100%, 50%, 40%, 30%, 15%, and 0%. Three cells were tested under each condition.
- Cells from 4 different manufacturers were tested:
 - Manufacturer: B, D, E, and F

Manufacturer	Cell Design	Cathode Chemistry	Rated Capacity (mAh)	Actual Capacity (mAh)	Internal Resistance (mΩ)
В	18650	NCA	3200	1850	35
D	Pouch	NMC	3300	3220	<15
E	26650	LFP	2500	2500	6
F	Pouch	LFP	10000	10300	<10





Charge Retention

- All 6 manufacturers were subjected to charge retention test to characterize self-discharge.
- Cells were stored in ambient temperature (controlled) at 6 different SOC
 - 100%, 50%, 40%, 30%, 15%, and 0%
 - 2 cells are under test for each condition.
- OCV is recorded once every week for the first month and then once every month for up to 6 months. If there is no change in the OCV, storage time will be increased to 9 months – study is still under progress

566								
	100%	50%	40%	30%	15%	0%		
Manufacturer A	13	3	2	1	1	-2		
Manufacturer B	34	14	10	20	26	40		
Manufacturer C	9	1	1	0	-7	-12		
Manufacturer D	48	2	2	7	7	14		
Manufacturer E	26	4	2	4	5	31		
Manufacturer F	-26	1	1	2	-83	-3		

SOC

Voltage Drop (mV)

*based on 2 months of storage



Summary and Conclusions

Heating Method:

- Venting temperature
 - Most of the cells followed similar trends, where temperature at which venting occurs increases (↑) as SOC goes down (↓)
 - Except for Manufacturer E and F (both LFP type) venting temperature is around the same value
- Thermal Runaway onset temperature increases (↑) as SOC goes down (↓) except for manufacturer B (low cost cells).
- Cells with Ni based cathodes NMC and NCA display fire and smoke at high SOC but LFP cells display only smoke even at high SOC
- Cells with Ni based cathodes NMC and NCA display smoke even at SOC as low as 15%. This may be due to the leakage of electrolyte at low SOCs which can then cause smoke to be generated due to the high temperatures induced by the heater.

External Short:

- Maximum temperature recorded decreases with decreasing SOC.
- Tabs burn instantaneously for pouch cells at high SOCs and the tab that burns is typically the one that has the lower m.pt.
- Burning of tabs prevents cells from experiencing the short circuit load and hence they don't swell or experience thermal runaway at the high SOCs. Cells show swelling when tabs do not burn off.



Safety of Fresh and Aged Li-ion Cells and Modules



Introduction

- Lithium ion batteries are used in a variety of applications from consumer electronics, automotive and aviation to space applications.
- They have been introduced into the Stationary Energy Storage Systems in recent years
- Automotive batteries are being repurposed for use in utility/stationary energy storage applications
- Questions that arise on the safety of used batteries are
 - Does the safety change with cycle and calendar life (including storage life)?
 - What parameters need to be characterized after first life and before installation in second life?
 - What parameters need to be studied closely during usage in second life?





Aging Effects on Cell

• Lithiation and de-lithiation causes

- Anode electrode morphology changes and volume changes – surface can form cracks leading to electrical isolation; delamination from current collector; changes in intercalation kinetics; loss of active lithium inside anode, etc.
- Decomposition
 - Binder and electrolyte; SEI decomposition; HF production, li-ion side reaction with electrolyte; etc.
- Corrosion
 - Current collector, cell can materials, pouch cell swelling and shorting due to corrosion of pouch material, etc.
- Cathode changes
 - Structural disorder, metal dissolution, disproportionation, etc.



Test Plan – UL/Texas A&M University

• Single Cell Studies – 18650 and pouch formats - ~3.4 Ah capacity

- Cycle life with continuous cycling at normal voltage range
- Cycle life with continuous cycling with reduced voltage range (200 mV less from both ends of voltage range)
- HEV profile at 3 temperatures (10 °C, 25 °C and 45 °C)
- Overcharge and external short test on fresh and cycled single cells
- Module (18650 3P9S: 33.3 V, 9.9 Ah ; pouch 5P5S:21V, 16.5 Ah) Studies
 - Cycle life with continuous cycling at normal voltage range
 - Cycle life with continuous cycling with reduced voltage range (200 mV less from both ends of voltage range (for 18650 cells and modules only)
 - Overcharge and External short test of fresh modules and overcharge test of cycled modules

Destructive Analysis:

Fresh cells : uncycled, externally shorted cell and overcharged cell Cycled cells (cells removed after 10%, 15% and 20% capacity loss), externally shorted cycled cells, overcharged cycled cells



Cycle Life Testing on Single 18650 Li-ion Cells- Capacity Trend



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Overcharge Test



Fresh cells take longer to exhibit CID activation Cycled cells exhibit shorter times to CID activation possibly due to formation of gases and accumulation of pressure as cells are cycled



Capacity and Internal Resistance Trend for HEV profile cycles



2-hr rest period between cycles; Normal capacity checks once a week 2.2

2.0

1.8 0 T = 10 °C

T = 25 °CT = 40 °C

10

15

Time [Weeks]

20

25

30

Cell at 10 °C: 25% Capacity Fade (13 weeks) (18% increase in internal resistance)

Cell at 25 °C: 25% Capacity Fade (29 weeks) (33% increase in internal resistance)

Cell at 40 °C: 25 %Capacity Fade (24 weeks) (29% increase in internal resistance)



DPA



Faces 6 and 3 of the separator that faces the cathode have ceramic coating

Anodes 10% 15% 20%

Cathodes



DPA of cells that underwent Cycle Life tests only

U

DPA after safety tests on cycled cells - Cathodes



Bb 20%

Overcharge DPA below shows both sides of electrode







20%

(ceramic coating is typically on surfaces of separator facing cathode – see graphic on slide 19); in cells that underwent₃₂ safety tests the coating is stuck on the electrode surface

	Cycling	Cycling	Cycling + Ex	ternal Short
		and the second difference of the second differ	Total AV	- HANNE
Section Ca (25 °C) Ambient Temperature	Cell Ca 2 CF = 23.11 % Weeks = 27 Cycles = 3126	Degradation pattern		
Section Cb (10 °C) Low Temperature	Cell Cb 1 CF = 24.56 % Weeks = 13 Cycles = 1347	Lack of Lithiation		
Section Cc	Cell Cc 1 CF = 21.52 %		Delamination	Lithium Plating
Temperature	Veeks = 25 Cycles = 2730	Lithiation		Electrodec
SEM of cycled cathode at ambient temperature	SEM of cyc anode at ambient temperatur (after solve	e ent		after Cycling + Overcharge

Module Cycle life and Internal Resistance Trend





Overcharge Test of Fresh Module



Overcharge Test of Cycled Module









Summary

- No difference in performance of thick or thin heaters; thick heater did not require additional tape to hold it in place, hence all tests were carried out with thick heater.
- The 2"X1" heater not different from the 2"X2" heater tape but the 2"X1" heater simulated local heating better. Questions arose as to whether the larger heater caused additional heating of adjacent cells. The smaller heater allows for cell heat to radiate to adjacent cells in a more consistent manner.
- 7-8 ° F / min heating rate was found to be optimal for consistent test results.
- Lot to lot variation observed especially with cells made by another manufacturer (in a different country) for the OEM.
 - Cells should be screened well before testing to remove cell lots (batches) that could result in anomalous results.
- The Open box test showed that during the thermal runaway process, electrolyte is released for cells at low SOC, which can then catch on fire or smoke heavily, causing the propagation to the inserts in the box as well as the other cells.
- This type of thermal runaway propagation was only observed with the second lot of cells which were made for the OEM in a different country especially if the temperature was held at 392 °F for one hour or if heater was left on until thermal runaway occurred.
- For the configurations studied, the center cell seemed to be the worst location for the first batch but for the second batch of cells, the corner and side wall location seemed worse. This may depend on the arrangement of the cells, the inserts, spacing between the cells (with the inserts), etc.
- Testing should be done in the relevant configuration and may need to be performed independently when cells with the same model number are manufactured in different manufacturing facilities, especially different countries.
- Working with Prof. Ankur Jain at UT-Arlington on modeling the thermal runaway propagation tests.



Overdischarge Study on Li-ion Cell Modules



Objective

Objective: To investigate the interplay between aging and overdischarge degradation mechanisms. The aim is accomplished by discharging cells prior and after the onset of reverse potential. Results will provide an insight on the safety implications of this interaction and the main with behind failure



Overdischarge Depth

Reverse Potential or **Cell Reversal** is the condition were anode potential becomes larger than the cathode one resulting on a negative voltage on the full cell. Anode and cathode potential are decoupled via a three-electrode cell.



Overdischarge + Cycling



Fresh Cell DPA and SEM Images



Destructive Physical Analysis (DPA)



Wanma Fresh Cell



Fresh separator and anode during DPA



Open fresh cell showing clean separator



Remaged Cell



Burn from internal short circuit

Swelling of damaged cell due to electrolyte decomposition



DPA of Extreme Overdischarged Cell



Damaged Cathode



Anode with burn marks due to internal shorting



Point of failure

Separator damage

Separator burned through





Ovals show internal short circuit burns. In some cases, the short circuit burned completely through the separator

EDS of Cycled & Overdischarged Cells (Anode)



E	С	Cu	AI	Р	S	F	0	Со
0.0	95.9	0.0	1.4	0.9	0.2	1.6	0.0	0.0
2.7	84.1	0.0	0.7	0.8	0.0	3.5	10.8	0.0
1.5	66.5	0.0	0.8	1.0	0.0	5.2	26.2	0.3
0.0	58.8	0.7	1.0	1.2	0.2	17.8	20.2	0.0
-0.5	46.1	0.5	0.9	1.0	0.4	6.0	45.0	0.1
-1.5	86.1	2.3	1.7	1.6	0.2	8.1	0.0	0.0

EDS of Cycled & Overdischarged Cells (Cathode)



E	С	Cu	AI	Р	S	F	0	Со
0.0	40.1	0.0	4.0	0.0	0.0	5.3	18.7	31.9
2.7	30.8	0.0	3.1	0.8	0.0	5.7	20.6	38.5
1.5	31.5	0.0	3.2	0.7	0.0	8.0	18.8	37.9
0.0	36.4	0.0	1.9	0.5	0.0	10.2	22.8	28.1
-0.5	36.2	0.0	2.1	1.0	0.1	9.8	24.7	27.9
-1.5	0.0	30.1	13.1	0.6	0.0	4.9	12.6	38.8

Conclusions

- Three-electrode cell is a necessary tool in order to extend the analysis for the interplay between aging and other degradation phenomenon.
- Overdischarge is a benign degradation phenomenon when it happens one time even during an extreme overdischarge scenario.
- Cathode signature: Fracture Electrochemical stacked
- Anode signature: Li plating as well as cobalt presence
- Copper bridge happens at extreme overdischarge with the dissolution of the copper current collector and it's deposition mainly in separator.
- Lithium plating and copper deposition interaction leads to internal short circuit of the cell, swelling and failure of the cell.
- Degradation number (Φ_R) shows to be a good dimensionless descriptor of the State of Health of the cell.
- Li-ion cells aged near or after the reverse potential point are more prone to Indergo a fast degradation and they could pose a threat to safety.

Fire Research in Li-ion Batteries

In collaboration with UT Austin and NFPA

- DHS- FEMA funding to study fires in li-ion batteries for stationary grid storage systems
- Testing carried out at UT Austin
- Batteries procured and tested using a similar trigger method
- Cell, module and battery fires are being studied
- Some large scale rack testing has been carried out



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Modeling Studies on anomalous behavior of thermal runaway propagation

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THANK YOU.

