



# Li-ion Soft Short Test & Cell Defect Correlation

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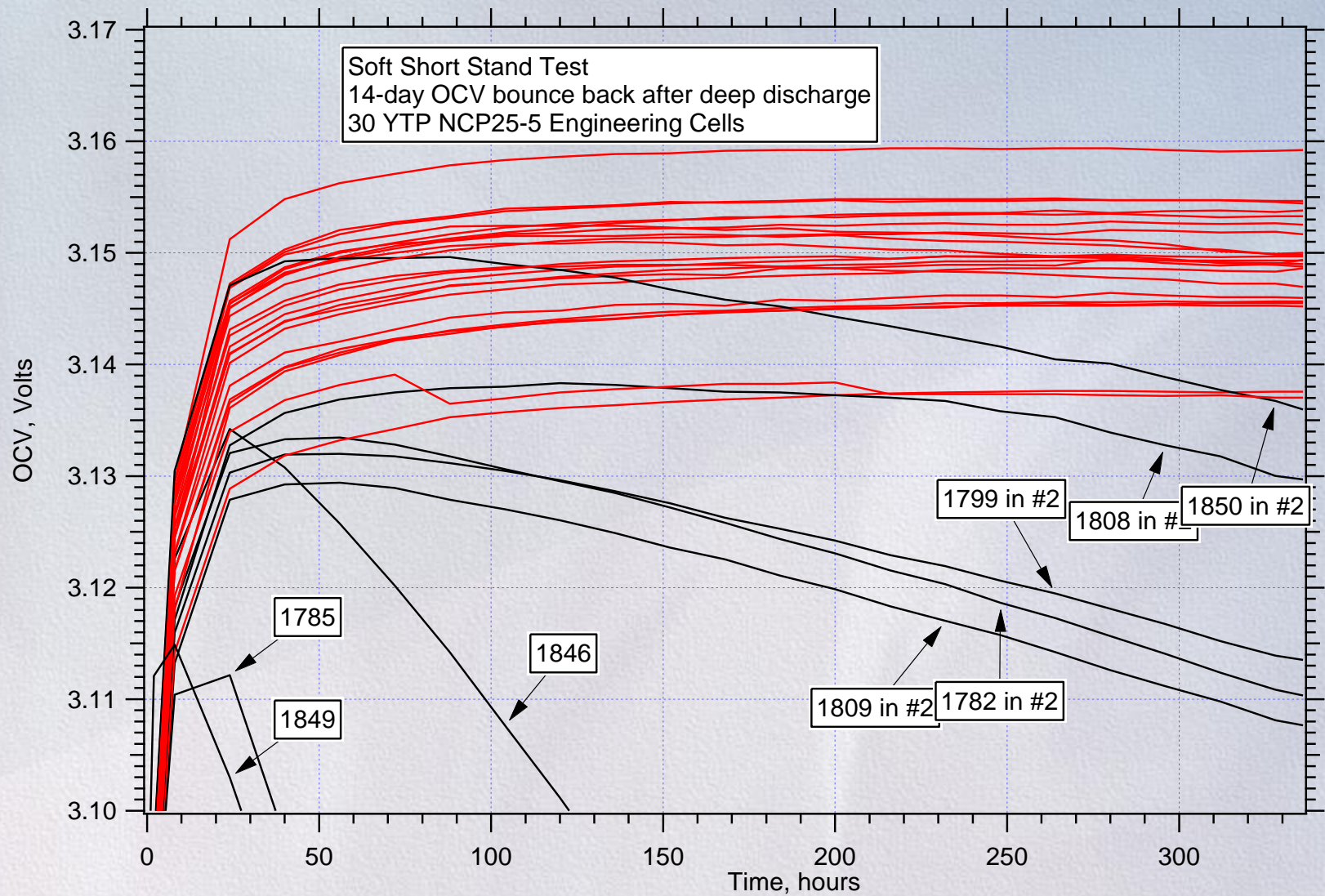
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Workshop  
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# Motivation

- Recent lot of YTP 30Ah flight cells are failing the soft short test at an alarming rate
  - 41/156 cells or 26% had  $\Delta V > 2\text{mV}$
  - 31/156 cells or 20% had  $\Delta V > 5\text{mV}$
  - 23/156 cells or 15% had  $\Delta V > 10\text{mV}$
  - Test was repeated twice on cells with suspect curves
- Questions
  - Is the test validly identifying cells with shorting defects?
  - Are some cell designs predisposed to fail this test?
  - Should it be included in our revised battery safety standard

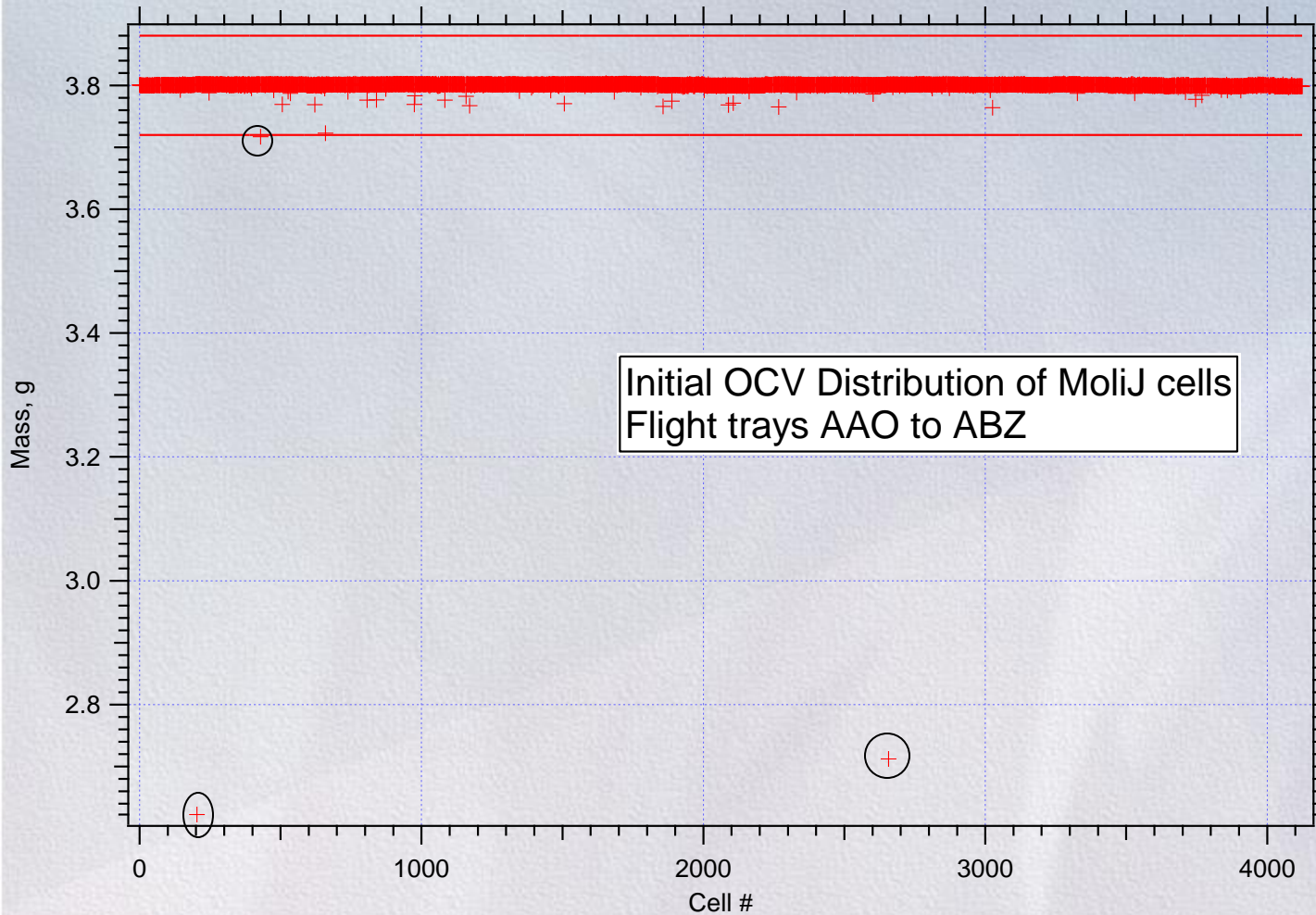
# YTP 14-day Soft Short Test Results



# Correlating Soft Short Rejects

- Objective
  - Determine how good the correlation is between OCV retention outliers and cell defects
  - Establish basis for soft short methodology using 18650 cells
- Plan
  - Use cells (Moli ICR18650J) that were rejected during acceptance OCV testing back in 2008-2009 for the EMU LLB project
    - Apr 2007 date code to Jul 2009 OCV test = 27 months of storage
    - 40 outliers out of 3641 kept in controlled storage since
  - For those that will accept a charge, cycle them and perform deep discharge, OCV bounce back soft short testing
    - CC discharge at C/10 to 3.0V
    - CV discharge at 3.0V to C/100 taper
    - Record OCV bounce back over 14 days
  - Perform cell DPA on the worst and best performers

# As Received OCV Distribution



Cell ID	OCV @ BOC
PD10SAAR110	2.62256809
PD10SAAT113	3.71686885
PD10SABL117	2.71236744

Average	3.8000
sdev	0.0267
sdev%	0.704%
Min	2.6226
Max	3.8036
Range	1.1810
Range%	31.081%
-3sigma	3.7197
+3sigma	3.8802
3sRange	0.1605
3sRange%	4.224%
Low Rejects	3
High Rejects	0
Total Rejects	3
Count	3641
Reject%	0.08%

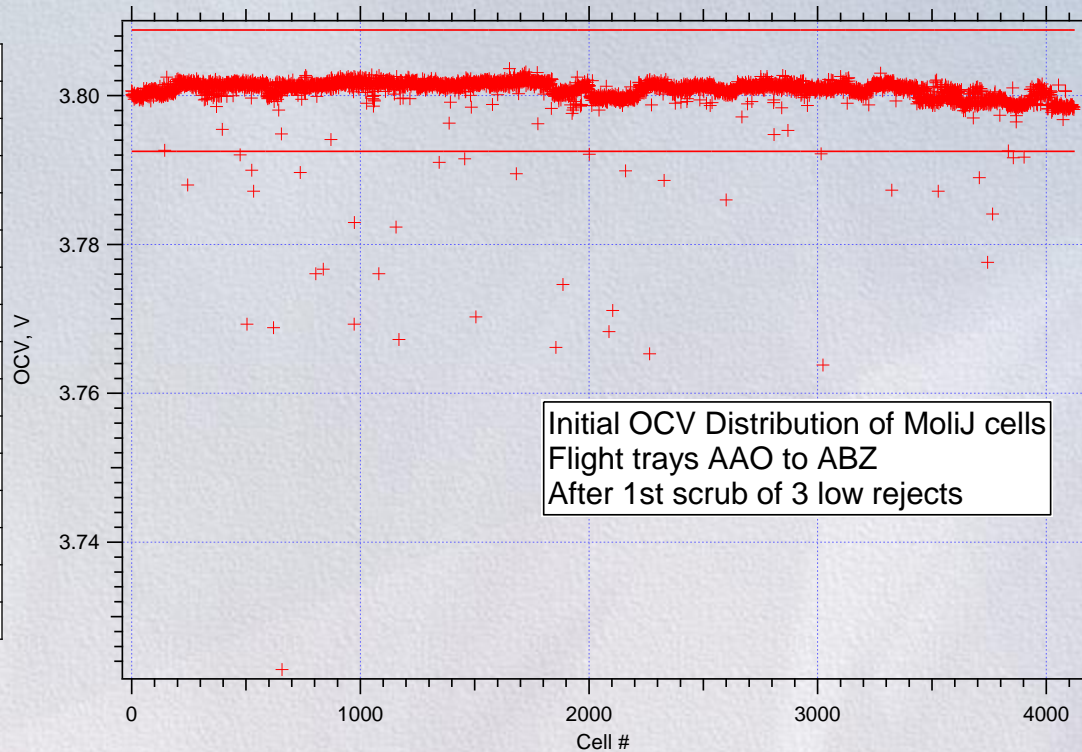
The 3 circled data points are the 3 outliers, 2 of them are severe outliers driving a  $6\sigma$  range of **161 mV (4.2%)**

11-24-09

“MoliJ” is short hand for E-one Moli Energy’s ICR18650J cell design

# As Received OCVs after 1<sup>st</sup> scrub

Average	3.8006
sdev	0.0027
sdev%	0.071%
Min	3.7229
Max	3.8036
Range	0.0807
Range%	2.124%
-3sigma	3.7925
+3sigma	3.8088
3sRange	0.0163
3sRange%	0.429%
Low Rejects	37
High Rejects	0
Total Rejects	37
Count	3638
Reject%	1.02%

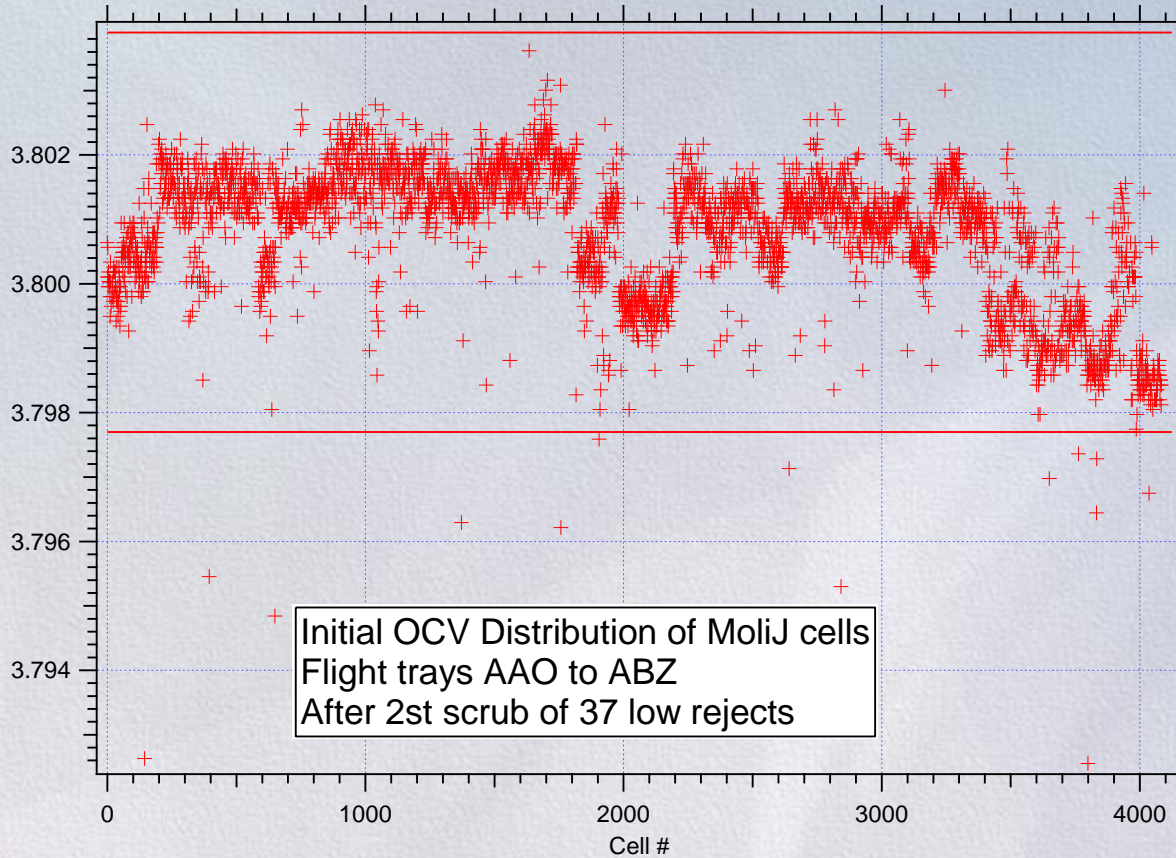


Cell ID	OCV @ BOC
PD10SAAV120	3.722896
PD10SABP045	3.76379
PD10SABI040	3.765316
PD10SABG075	3.766155
PD10SABA058	3.767224
PD10SAAO084	3.768292
PD10SAAV083	3.768826
PD10SAAU062	3.769284
PD10SAAY085	3.769284
PD10SABD076	3.770275
PD10SAAP005	3.771115
PD10SABH010	3.774624
PD10SAAX044	3.776074
PD10SAAZ098	3.776074
PD10SAAX078	3.776684
PD10SABV106	3.7776
PD10SABA045	3.78233
PD10SAAY086	3.78294
PD10SABV128	3.784085
PD10SABL062	3.785992
PD10SAAU091	3.787137
PD10SABT110	3.787137
PD10SABR127	3.787289
PD10SAAS025	3.787976
PD10SABJ008	3.788586
PD10SABV070	3.788968
PD10SABF029	3.789502
PD10SAAW072	3.789654
PD10SAAP062	3.789883
PD10SAAU083	3.78996
PD10SABC011	3.791028
PD10SABD026	3.791485
PD10SABW092	3.791638
PD10SABX045	3.791714
PD10SAAU032	3.79202
PD10SABH126	3.792096
PD10SABP037	3.792172

37 new outliers found outside new -3 sigma range of 16.3 mV (0.43%)  
 Note that none +3 sigma outliers suggests capacity cyclers errors are small

# As Received OCVs after 2<sup>nd</sup> scrub

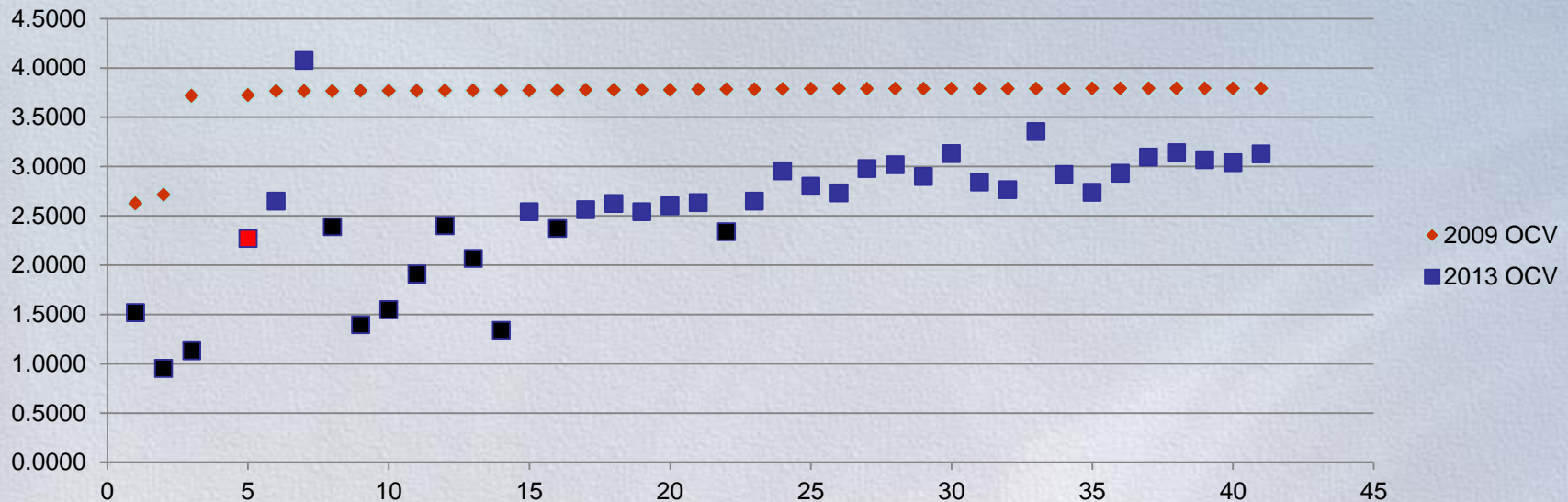
Cell ID	OCV @ BOC
PD10SABW070	3.792554
PD10SAAR050	3.79263
PD10SAAAX111	3.79408
PD10SABN051	3.794766
PD10SAAV118	3.794842
PD10SABN113	3.7953
PD10SAAT081	3.795453
PD10SABF124	3.796206
PD10SABC054	3.796292
PD10SABX008	3.796445
PD10SABY089	3.79675
PD10SABV043	3.796979
PD10SABM004	3.797131
PD10SABX009	3.797284
PD10SABW032	3.79736
PD10SABH050	3.797589



Average	3.8008
sdev	0.0010
sdev%	0.027%
Min	3.7926
Max	3.8036
Range	0.0111
Range%	0.291%
-3sigma	3.7977
+3sigma	3.8039
3sRange	0.0062
3sRange%	0.162%
Low Rejects	16
High Rejects	0
Total Rejects	16
Count	3601
Reject%	0.44%

Another 16 outliers found outside of recalculated  $6\sigma$  range of 6.2 mV (0.16%)  
 These 16 were rejected, but a new  $6\sigma$  range was not recalculated. In all, a total of 56 OCV outliers were removed from flight cell population

# OCV History

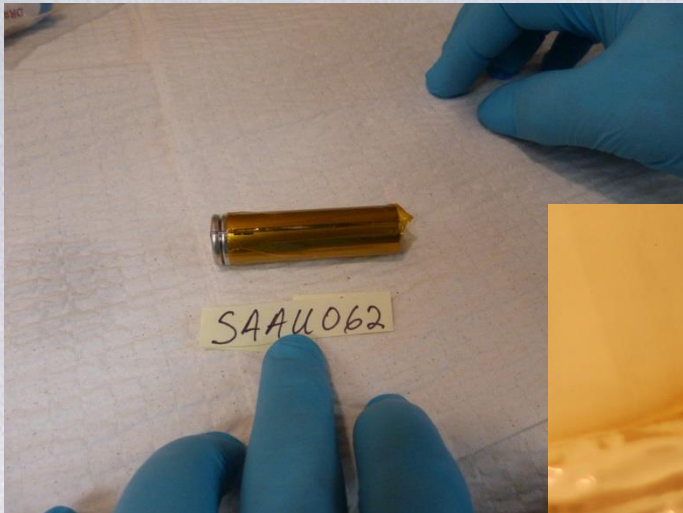


- After the 2009 OCV measurement, cells were capacity cycled and stored at 0% SOC
- Squares are the 2013 OCVs on the 40 cells
- 12 cells with OCVs  $< 2.5V$  (shown as black squares) would not accept a charge
  - Exception was cell #120 in red whose OCV was 2.2719V and it cycled



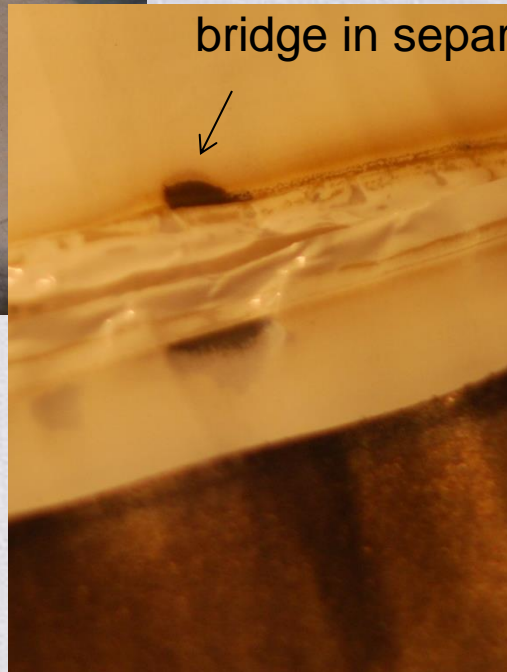
# Cells that wouldn't cycle

- 12 of 40 cells would not cycle
- All their OCVs had degraded  $< 2.5V$
- Five of the 12 have been DPA'ed so far



PD10SAAU062

Possible active material bridge in separator



Cell ID	2009 OCV	2013 OCV
PD10SAAR110	2.6226	1.5186
PD10SABL117	2.7124	0.9531
PD10SAAT113	3.7169	1.1313

PD10SAAV120	3.7229	2.2719
PD10SABP045	3.7638	2.6484
PD10SABI040	3.7653	4.0751
PD10SABG075	3.7662	2.3907
PD10SABA058	3.7672	1.396
PD10SAAO084	3.7683	1.549
PD10SAAV083	3.7688	1.909
PD10SAAU062	3.7693	2.4004
PD10SAAV085	3.7693	2.0677
PD10SABD076	3.7703	1.338
PD10SAAP005	3.7711	2.5417
PD10SABH010	3.7746	2.3714
PD10SAAV044	3.7761	2.5599
PD10SAAZ098	3.7761	2.6227
PD10SAAV078	3.7767	2.5401
PD10SABV106	3.7776	2.6015
PD10SABA045	3.7823	2.633
PD10SAAV086	3.7829	2.3404

# Moli Cell #PD10SABD076



Active material bump on anode coating

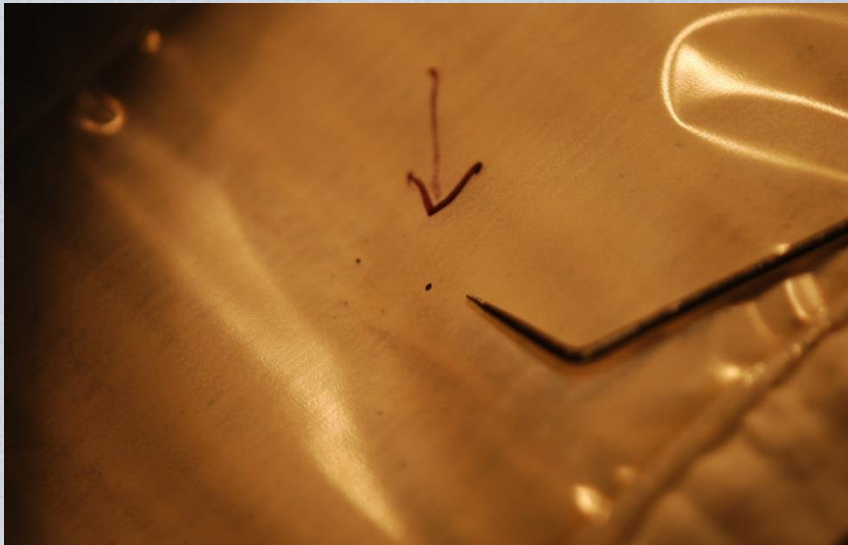


Active material deposit on anode side of separator

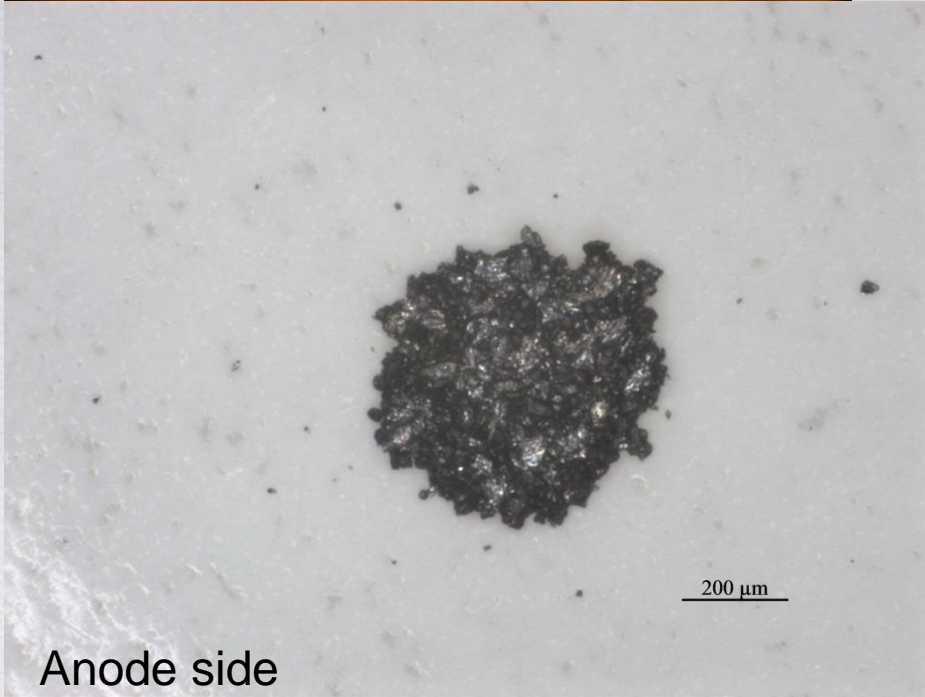
Active material deposit on cathode side of separator



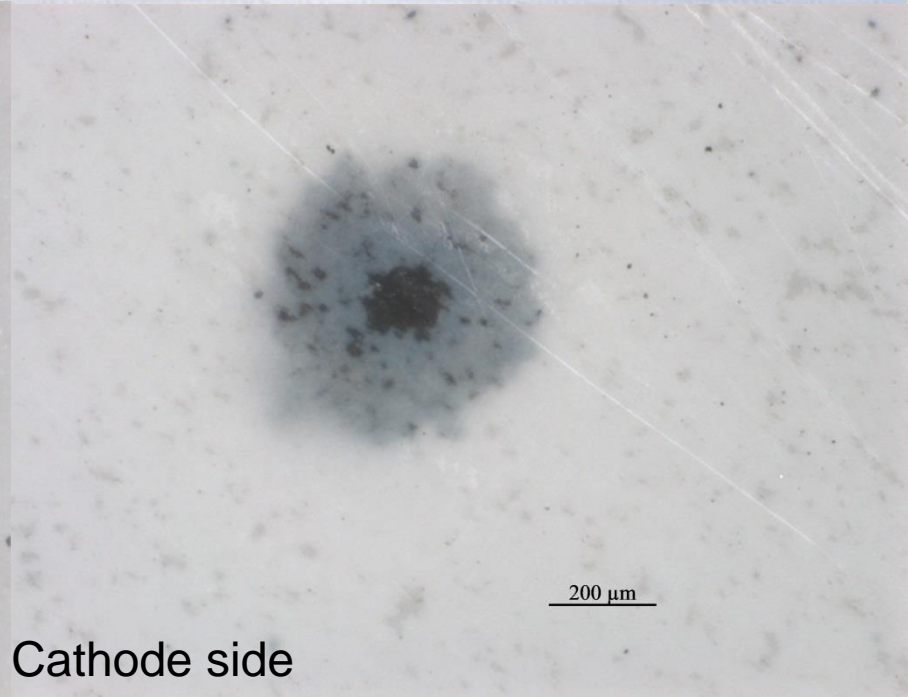
# Moli Cell #PD10SABL117



- Found active material deposit on both side of separator
- Most likely cause of the soft short

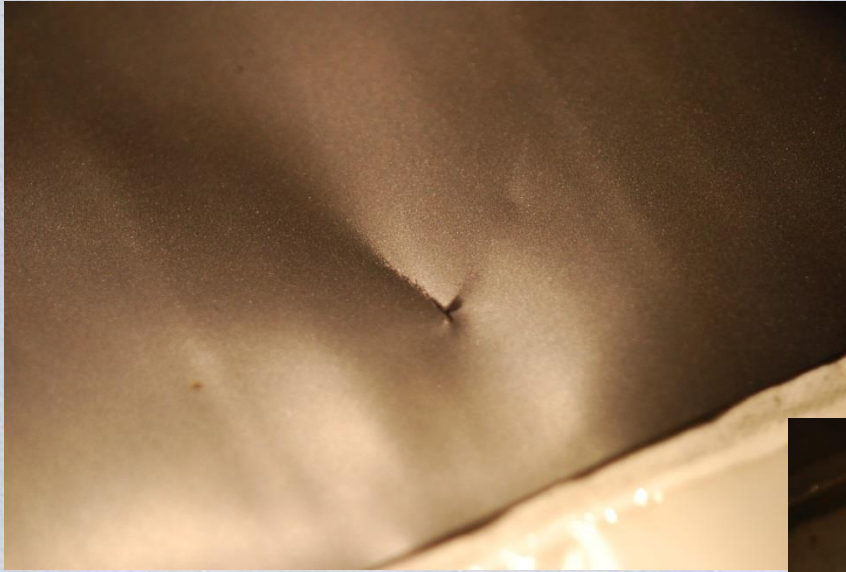


Anode side



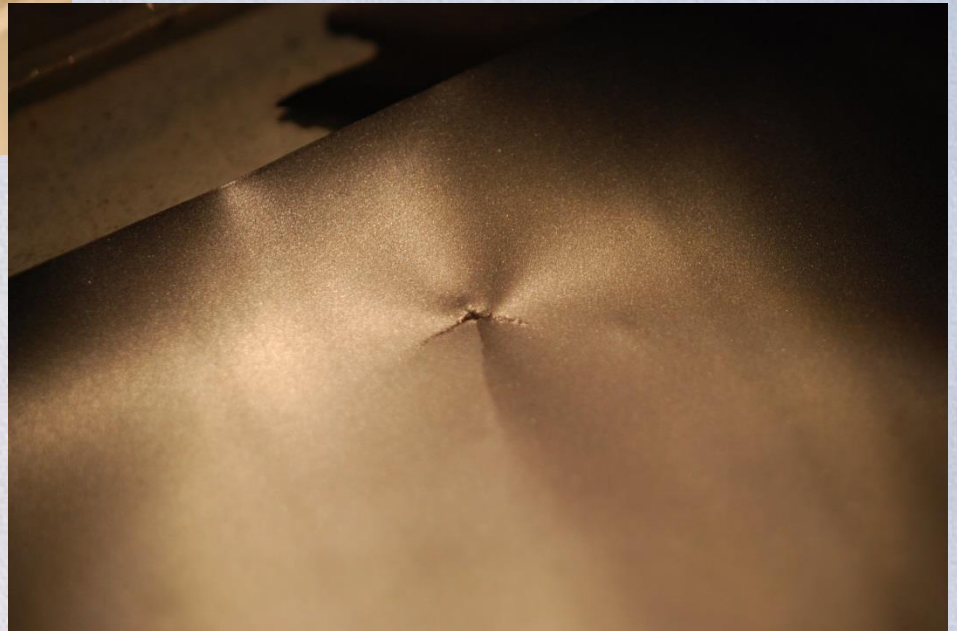
Cathode side

# Moli Cell #PD10SABL117 (cont)

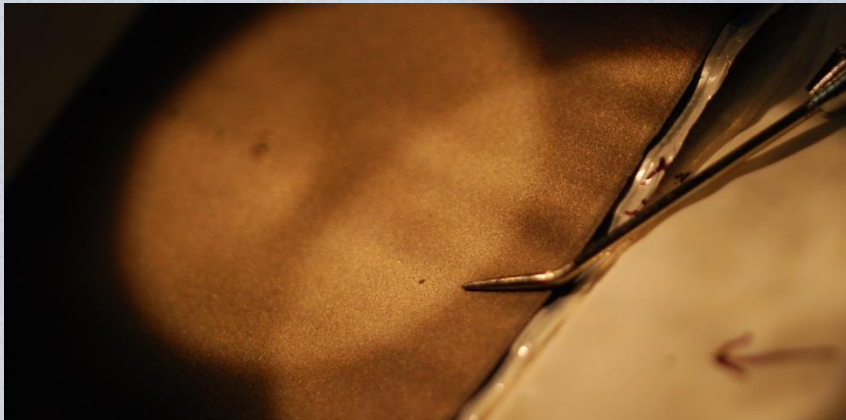


- Found crease in cathode, but no separator mark

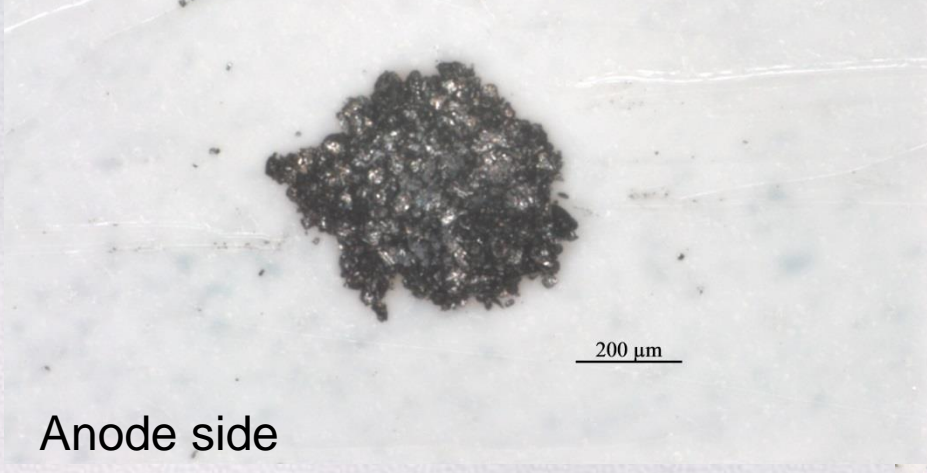
This defect may not have caused the soft short



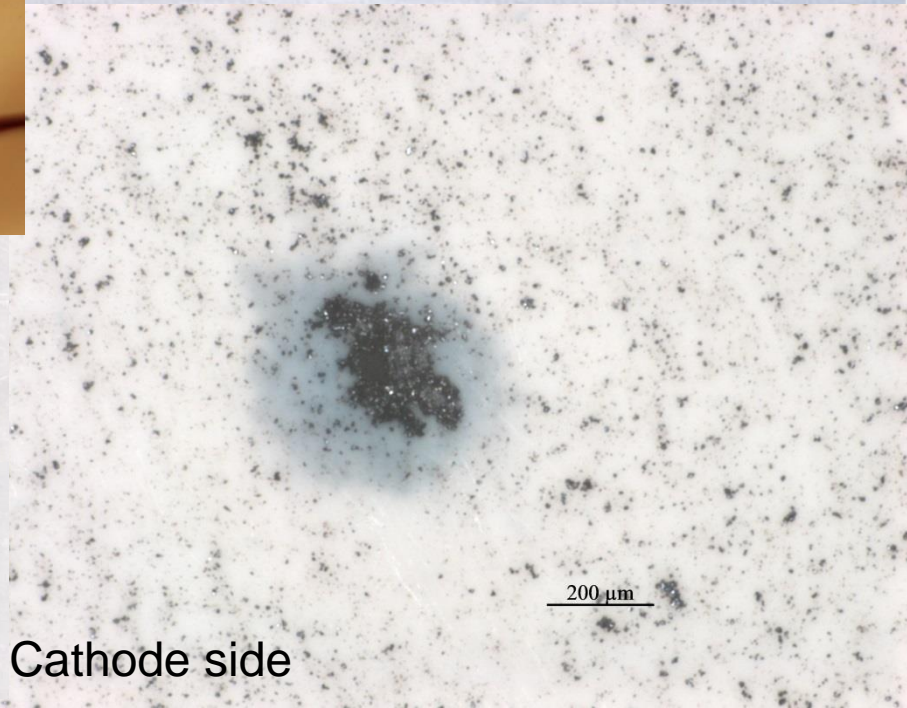
# Moli Cell #PD10SAAT113



- Found active material deposit on both side of separator
- Most likely cause of the soft short

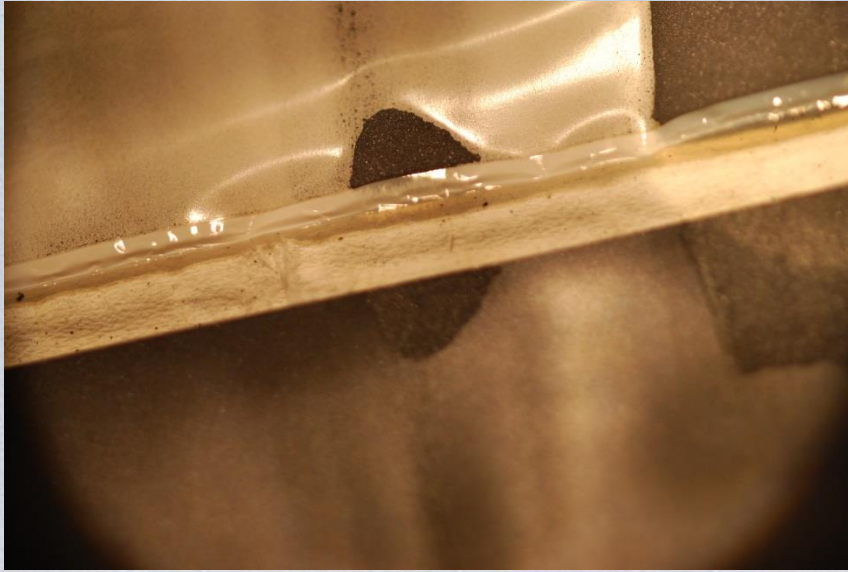


Anode side

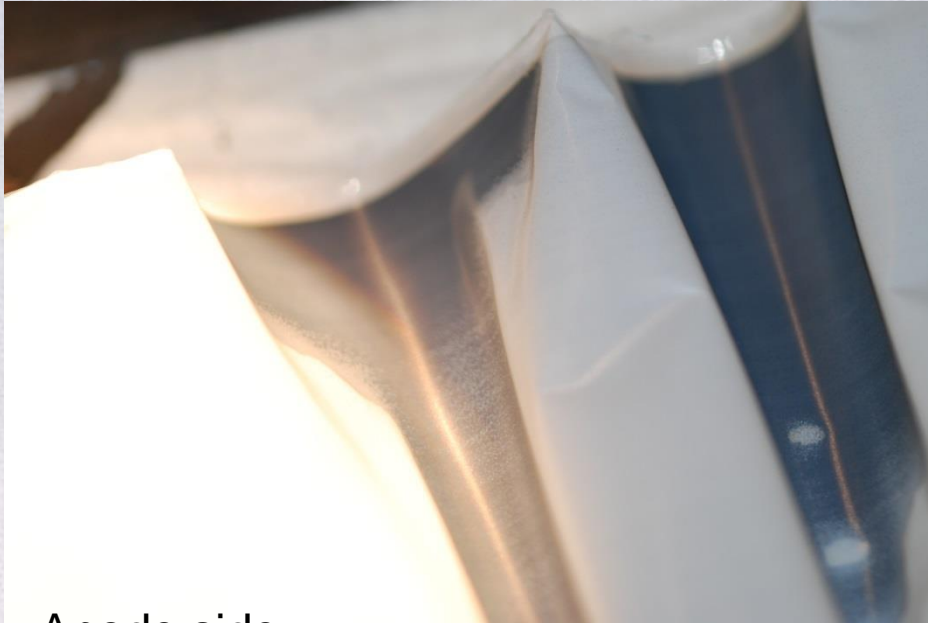


Cathode side

# Moli Cell #PD10SAAR110



- Heavy delamination of cathode active material found in separator

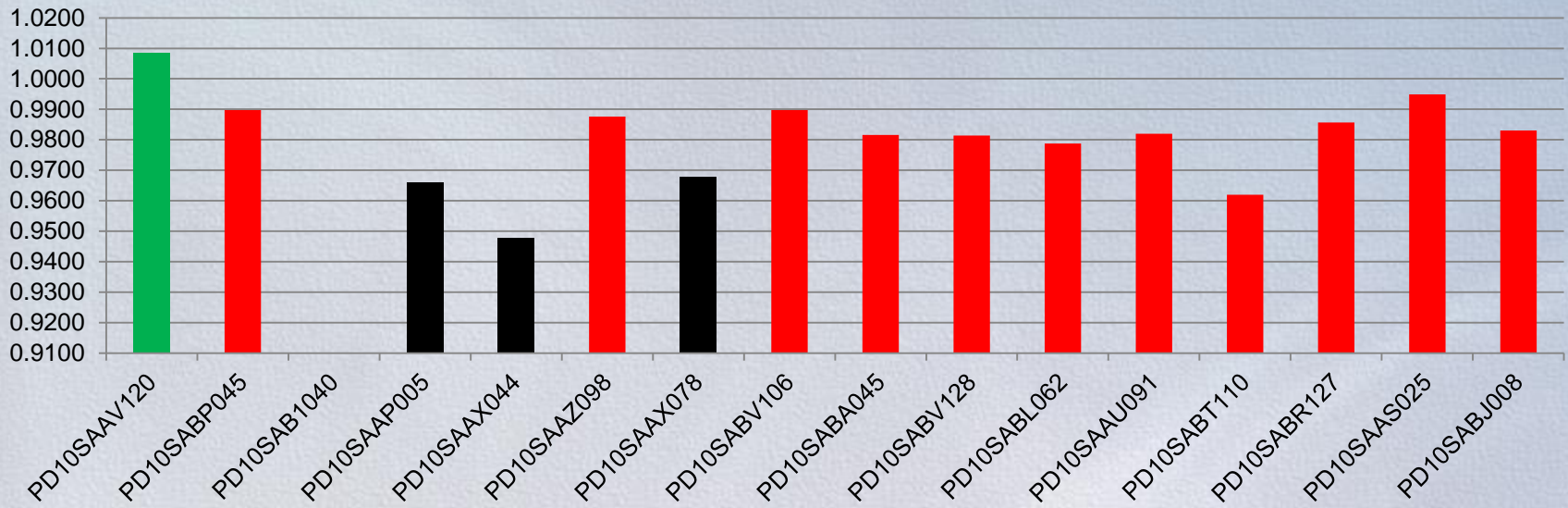


Anode side

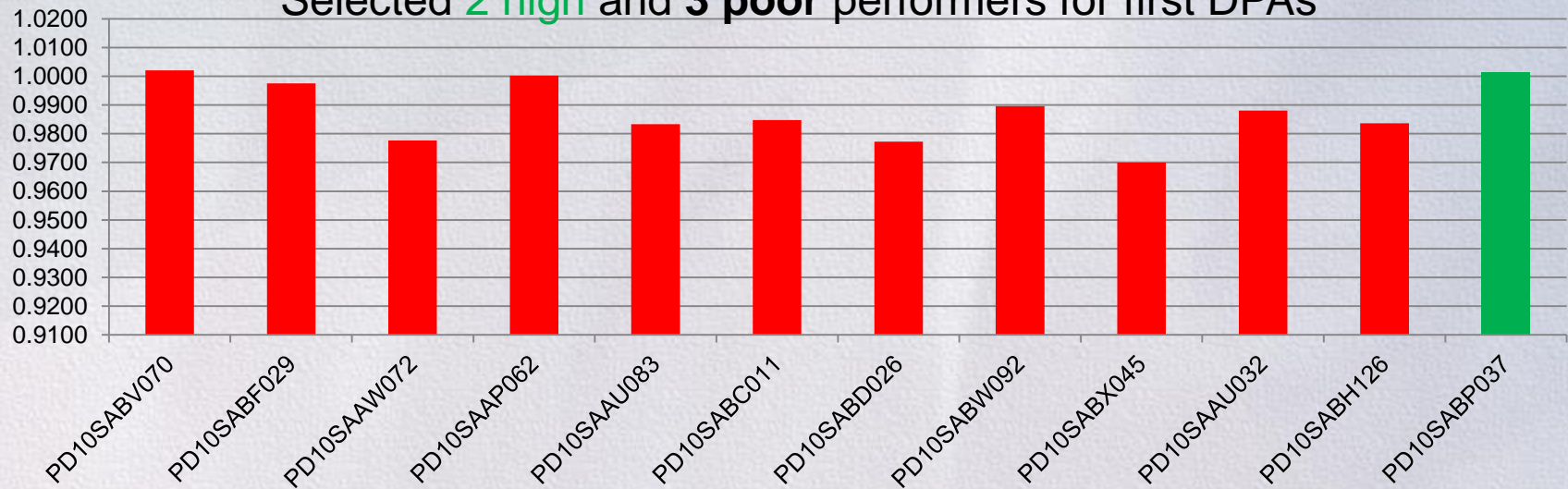


Cathode side

# Coulombic Efficiency of the 28 cells



## Selected 2 high and 3 poor performers for first DPAs

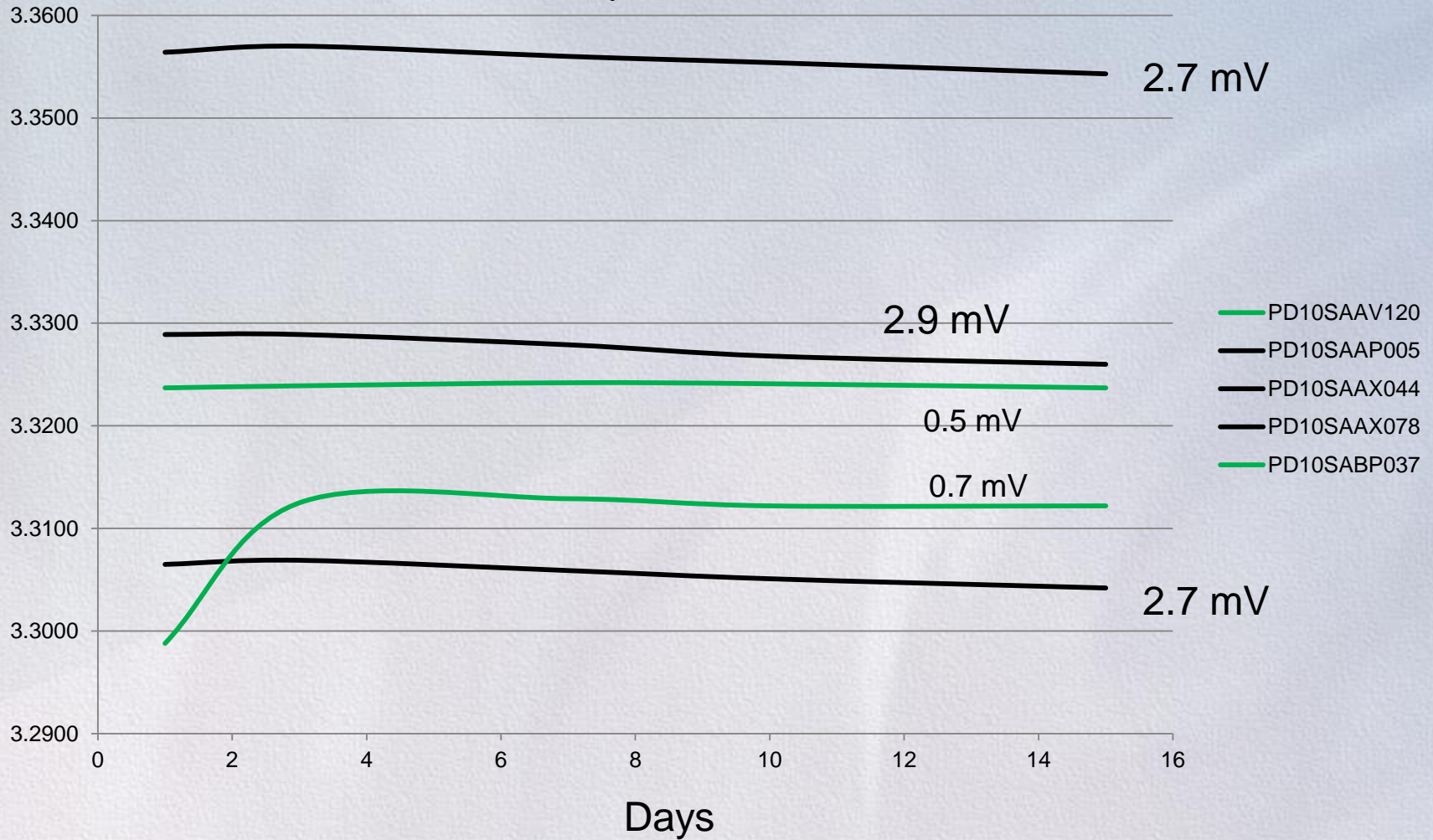


# OCV Bounce Back of 5 Selected Cells

2 cells with nominal Ah eff

3 cells with poor Ah eff

OCV decline from maximum





# Soft Short Results on all 28

ID#	OCV ~Day 1 Date 8-20-13	OCV ~Day 3 Date 8-22-13	OCV ~Day 7 Date 8-26-13	OCV ~Day 10 Date 8-29-13	OCV ~Day 16 Date 9-4-2013	$\Delta V$ drop
PD10SABL062	3.2990	3.2954	3.2950	3.2940	3.2934	0.0056
PD10SAAX078	3.3289	3.3289	3.3279	3.3268	3.326	0.0029
PD10SAAP005	3.3564	3.3570	3.3560	3.3554	3.3543	0.0027
PD10SAAX044	3.3065	3.3069	3.3059	3.3051	3.3042	0.0027
PD10SABV128	3.3000	3.2992	3.2985	3.2979	3.2973	0.0027
PD10SABV106	3.3122	3.3122	3.3112	3.3104	3.3096	0.0026
PD10SABP045	3.3327	3.3326	3.3317	3.3306	3.3302	0.0025
PD10SAAZ098	3.3012	3.3016	3.3007	3.3001	3.2991	0.0025
PD10SABA045	3.3112	3.3111	3.3105	3.3095	3.3087	0.0025
PD10SABT110	3.2970	3.2975	3.2967	3.2954	3.295	0.0025
PD10SAAU091	3.2947	3.3438	3.3432	3.3423	3.3418	0.0020
PD10SABJ008	3.3300	3.3299	3.3294	3.3283	3.3281	0.0019
PD10SABC011	3.2695	3.2805	3.2806	3.2797	3.2789	0.0017
PD10SABW092	3.2923	3.3073	3.3075	3.3066	3.3058	0.0017
PD10SAAU032	3.3234	3.3478	3.348	3.3471	3.3463	0.0017
PD10SAAW072	3.3432	3.3812	3.3852	3.3845	3.3837	0.0015
PD10SABR127	3.2972	3.3587	3.3583	3.3573	3.3572	0.0015
PD10SABF029	3.2929	3.3109	3.3112	3.3105	3.3099	0.0013
PD10SABD026	3.2878	3.3085	3.3091	3.3083	3.3078	0.0013
PD10SABX045	3.2941	3.3081	3.3087	3.3078	3.3074	0.0013
PD10SAAS025	3.3480	3.3484	3.3480	3.3472	3.3471	0.0013
PD10SAAU083	3.2991	3.3174	3.3179	3.3172	3.3166	0.0013
PD10SABH126	3.2854	3.2984	3.2988	3.2978	3.2976	0.0012
PD10SABV070	3.2864	3.3027	3.3034	3.3026	3.3023	0.0011
PD10SABP037	3.2988	3.3125	3.3129	3.3122	3.3122	0.0007
PD10SAAV120	3.3237	3.3239	3.3242	3.3241	3.3237	0.0005
PD10SAAP062	3.3554	3.3974	3.4047	3.4052	3.4051	0.0001
PD10SAB1040						0.0000

- 14-day soft short test identified most of the 27 months OCV rejects
- 24 cells with  $\Delta V > 1$  mV (86%)
- 11 cells with  $\Delta V \geq 2$  mV (39%)

# Moli Cell #PD10SAAP005

OCV reject by 21.4 mV

Failed Soft Short Test ( $\Delta V = 2.7$  mV)



Active material bump on anode coating



Active material deposit  
on cathode side of  
separator



Active material deposit  
on anode side of  
separator

# Moli Cell #PD10SAAX044

OCV reject by 16.4 mV

Failed Soft Short Test ( $\Delta V = 2.9$  mV)

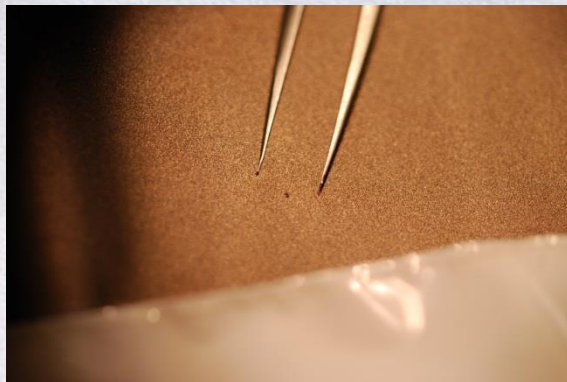


Defect 1

Active material spot on anode coating and adjacent spot on separator

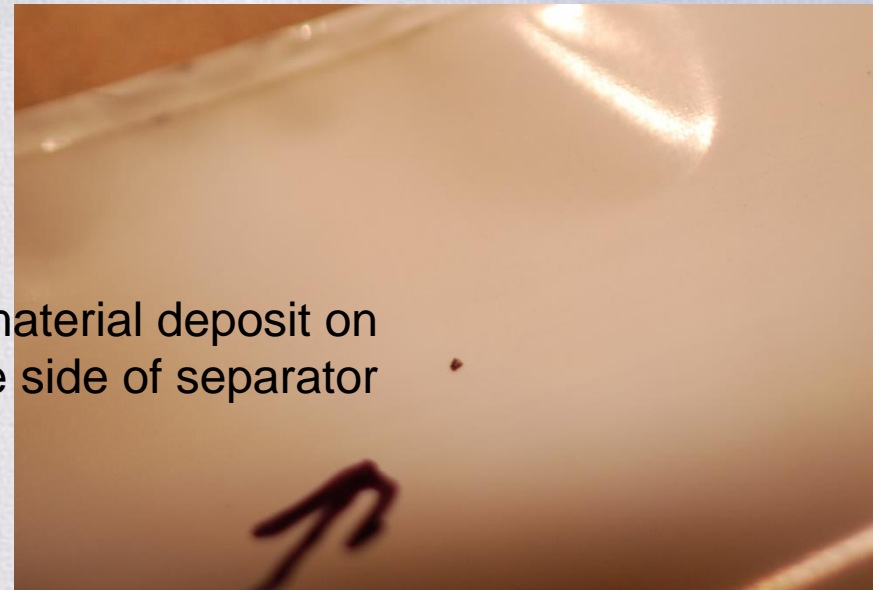


Active material deposit on cathode side of separator



Defect 2

Active material spot on anode coating

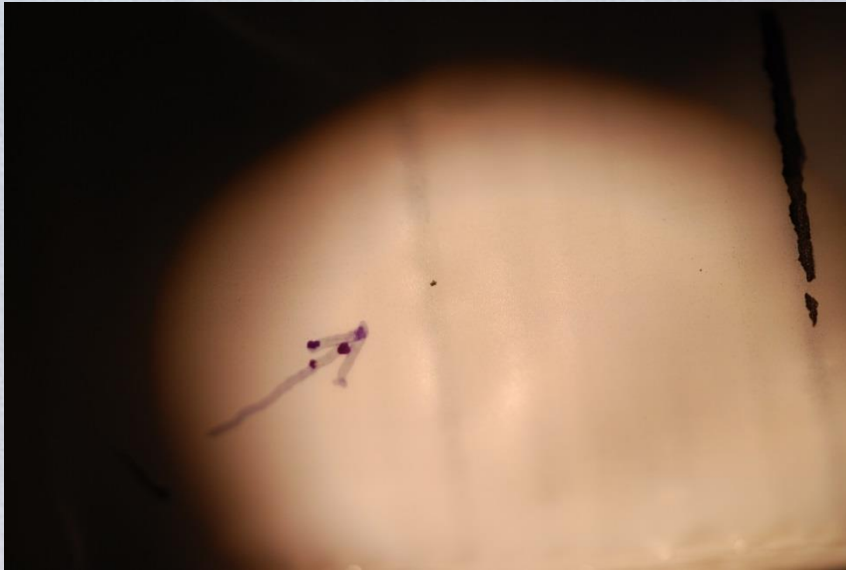


Active material deposit on anode side of separator

# Moli Cell #PD10SAAX078

OCV reject by 15.8 mV

Failed Soft Short Test ( $\Delta V = 2.7$  mV)

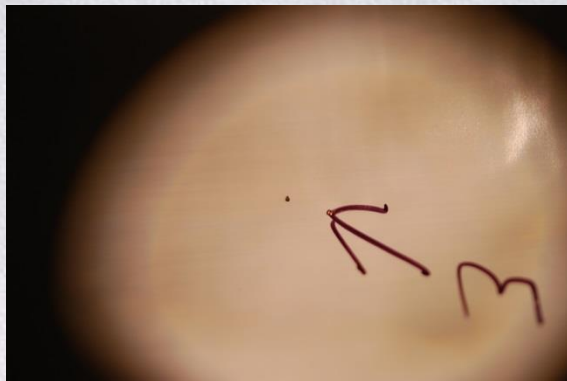


Active material bump on anode coating

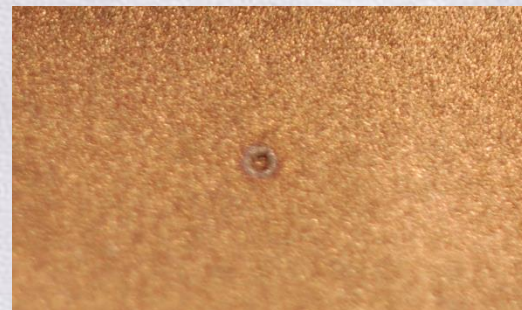
Adjacent



Active material deposit on cathode side of separator



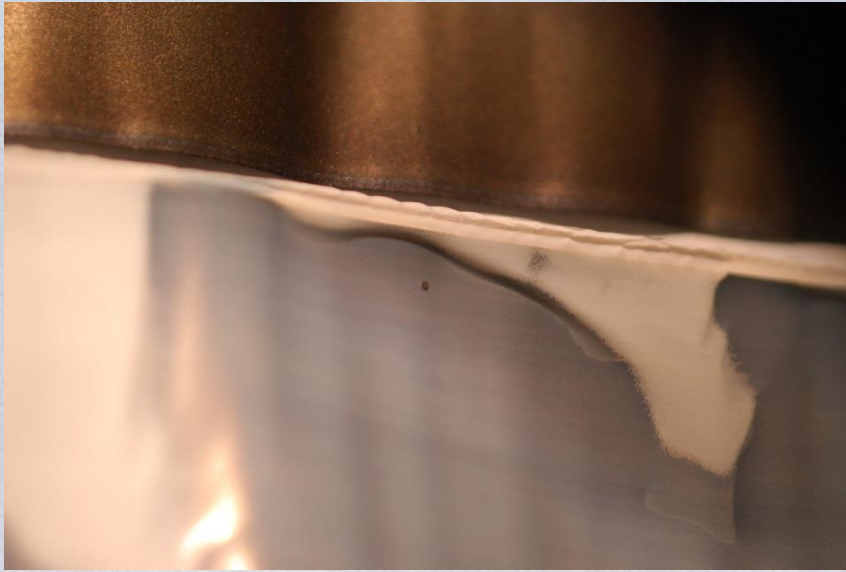
Adjacent



Anode deposit causing halo mark

Active material deposit on anode side of separator

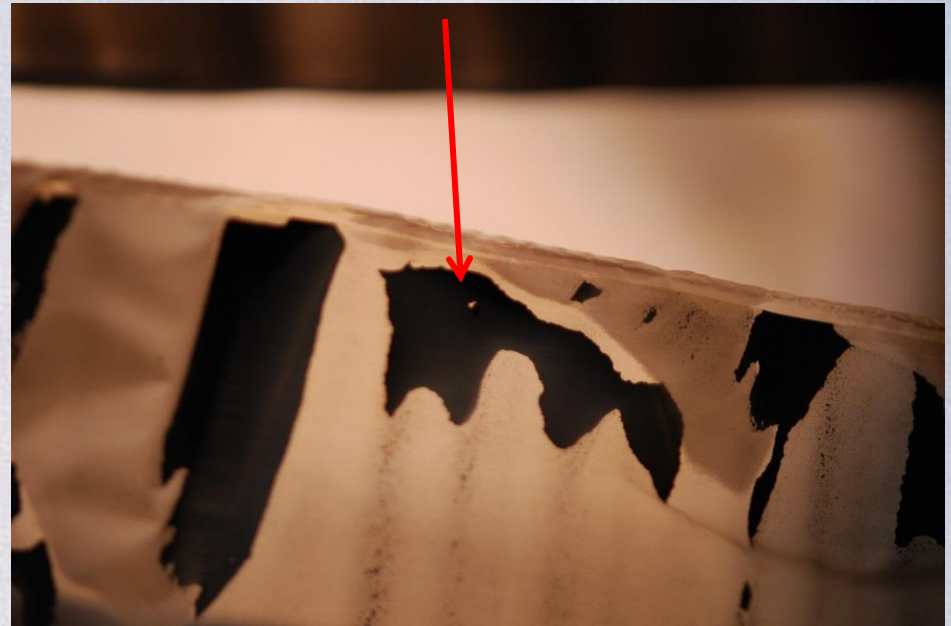
# Moli Cell #PD10SABP037



Active material deposit on anode side of separator with corresponding mark on anode

Marginal OCV reject by 0.3 mV  
Passed Soft Short Test ( $\Delta V = 0.7$  mV)

Hole in active material laminated one separator

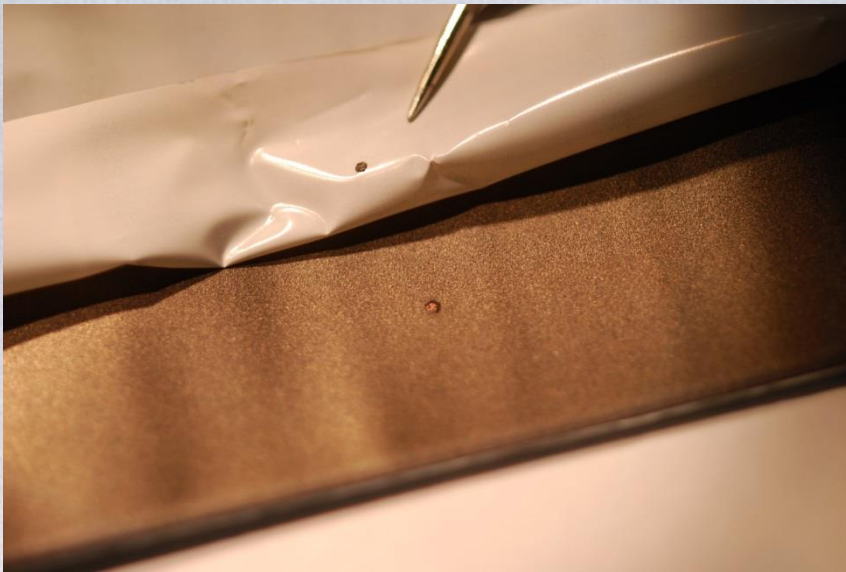


Separator does not appear to be bridged with active material  
Stereomicroscope analysis will be done for confirmation

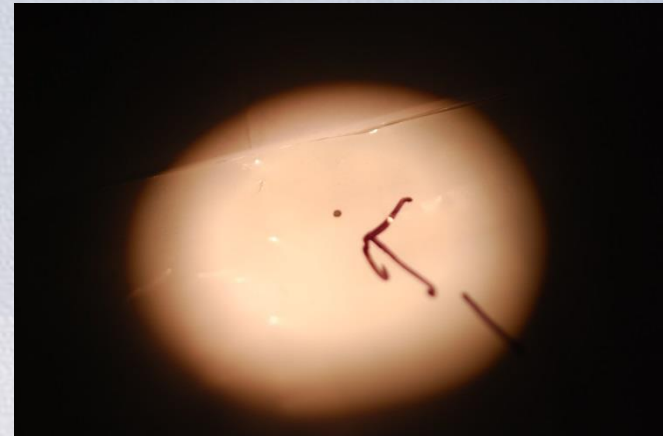
# Moli Cell #PD10SAAV120

OCV reject by 69.6 mV

Passed Soft Short Test ( $\Delta V = 0.5$  mV)



Active material bump on anode coating  
With corresponding deposit on separator



Shadow of spot on cathode  
side of separator

Stereomicroscope analysis will determine if  
material bridge exists

# Preliminary Conclusions

- 14-day soft short test identified as suspect 86% of the 27-month OCV storage rejects
- DPA of cells that were too discharged to cycle
  - Revealed suspected bridging defects in the separator of each cell
  - First two that were analyzed with stereomicroscope were confirmed as shorted separator spots
- DPA of cells that did accept cycling and were soft short tested
  - Suspected defects in cells with poor Ah efficiency and  $\Delta V \geq 1$  mV appear more severe than those in cells with good Ah efficiency and good OCV bounce back
- Remaining cell DPAs with stereomicroscope analysis pending
- Preliminary results indicate the value of 14-day soft short test
  - However, its resolution is not as good as received OCV measurement after long storage period (27 months)
- Test protocol may need to be adjusted with YTP cells
  - In 2010, 20% of heritage cells failed, but with lower  $\Delta V$  declines
  - vs 20% of latest Orion cells failed with  $\Delta V \geq 5$  mV, 2013
  - The discharge voltage point of test may need to be adjusted higher

# Acknowledgements

- Laura Baldwin/NASA-JSC for performing the cycling and soft short testing
- Chris Iannello/NASA Engineering Safety Center for funding the effort