Methods to Expedite Automotive Battery Development

Section 1C0: Test Cells and Cell Testing

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Agenda

- **The pros and cons of sub-scale battery research**
- **How sub-scale cells are deployed today:**
  - Which parameters are "trusted" on the sub-scale level? → **1C2** – *Sophia Bauknecht*
  - High throughput testing
  - Academic and mechanistic analysis
- **Key Cell-Based Research Considerations**
  - Globally recognized best practices → **Panel** – *Matt Raiford*
  - Cell sealing and the tools to accomplish it → **1C3** – *Nate Leibensperger, Nicolas Clemens*
  - Compression and saturation → **1C1** – *Shane Christie, Shawn Peng, Ian Wolfe, Nicolas Clement, Daniel Charles*
  - Vacuum filling → **1C4** – *Daniel Charles, Phil Jeyes*
  - Methods: bath control, gas measurement, half-cell potentials → **1C5** – *Abderrezak Hammouche*
- **Future pursuits**
  - Which applications benefit from the most from sub-scale data?
  - SLI/EFB, VRLA, AUX?

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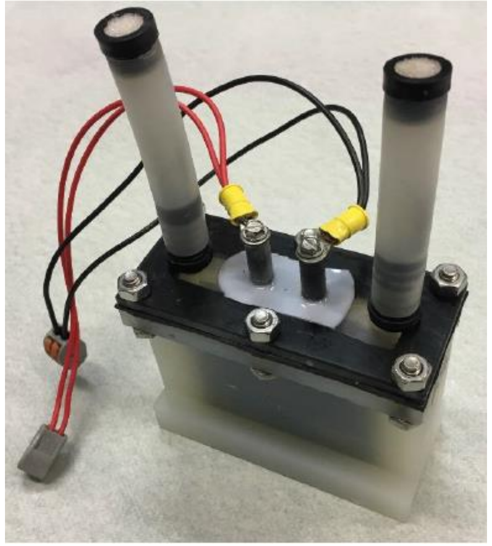
The Pros and Cons of Sub-Scale Battery Research

- **PROS**
  - Cheaper than working with Ton-scale battery builds
  - High-precision recipe control
  - High-throughput screening applicable
  - Does not require access to industrial equipment
  - Deep technical support in articles, community, and CBI
  - Permits heavy weighting of electrode of interest

- **CONS**
  - Higher variability between batches (needs strict QC)
  - Cast-on-straps / posts are hard to make, change results
  - Low number of cell replicates per pasting
  - Often hard to achieve application relevant AM:H2SO4
  - Lab-to-Lab variability due to differences in cell design



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HOW SUB-SCALE CELLS ARE DEPLOYED TODAY

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Which Parameters are "Trusted" on the Sub-Scale?

It is easy to make a cell; it is not easy to make robust, reliable test vehicle…

- **Parameters which can be…**
  - **…Absolutely trusted:**
    - Tafel slopes → response to CC or CV interrogation at top of charge
    - Reference electrode analysis → voltage response during work, related to a standard electrode
  - **…trusted, but with caveats:**
    - C_{20} → Can be trusted when NAM:PAM:electrolyte ratio is application-relevant, and when formation is effective
    - DCA → Can be trusted to give trends between variables in a well-built cell, but I_{DCA} magnitude will not scale smoothly**
    - Water consumption → Can be trusted when seals are well made
    - High-temperature testing → Can be trusted when seals are well made, and cell temperature is well controlled
    - Cycling → Can be trusted when NAM:PAM:electrolyte ratio is application-relevant in well-built cells
  - **…unreliable:**
    - High-Temperature Endurance (HTE/nKLT) → Can be trusted only in cells with specialty connections and seals**
    - CCA/HRD → Can be trusted only in cells with specialty connections

**Subject of ALBA R+D Workgroups from 2019-2022

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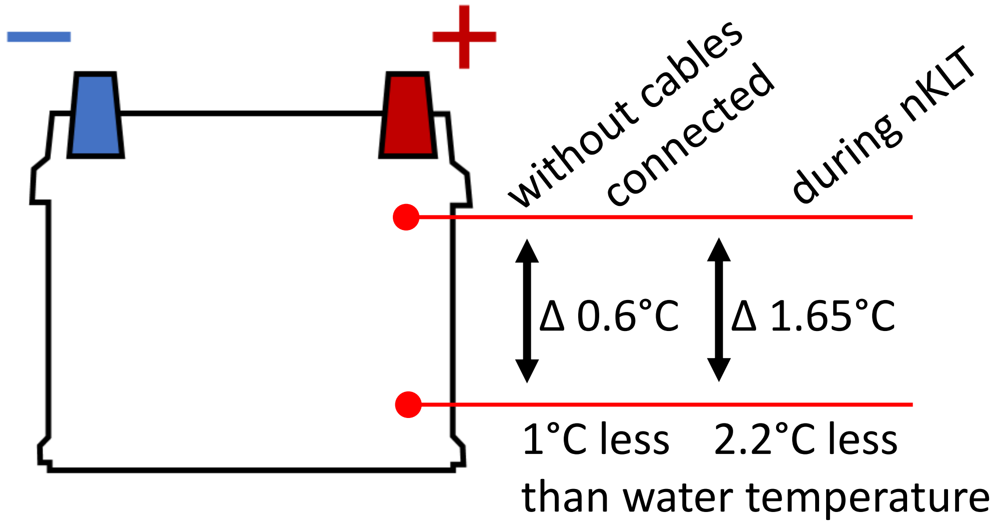
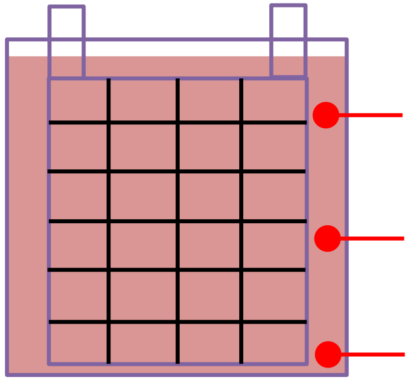
Example: Thermal Gradients in Sub-Scale Testing

1C2

battery temperature during hot temperature tests:



cell temperature ???



- There is a temperature gradient in batteries during hot temperature testing!
- How big is this temperature gradient it in small test cells?
- Do we need to & How can we diminish the temperature gradient?

Technische Universität Berlin EET Fraunhofer ISC
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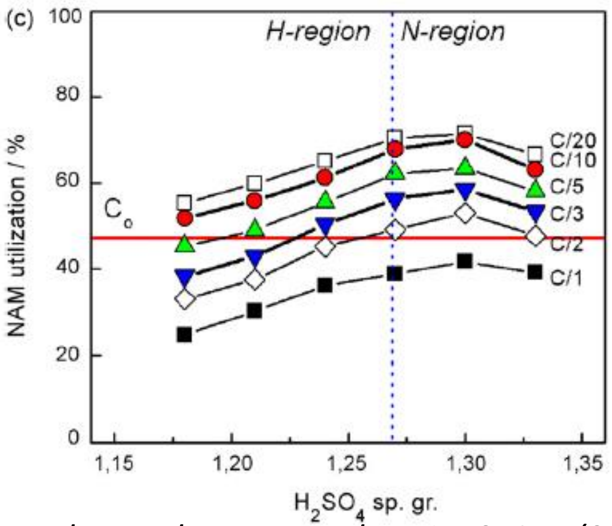
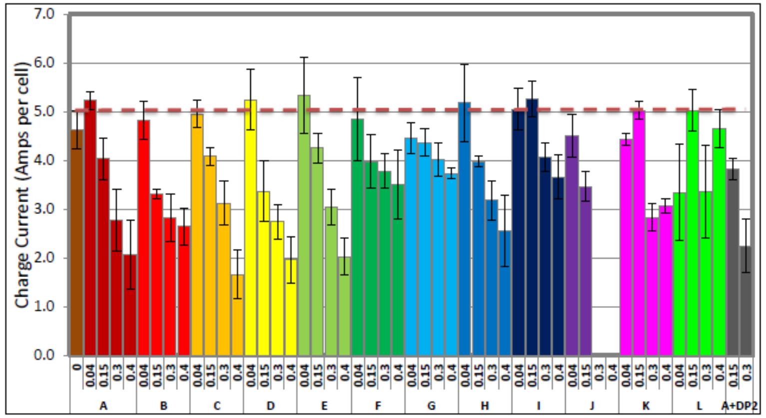
High-Throughput Testing

- **Paste rheological variables**
- **Expander components**
  - Ratios
  - Loadings
- **Electrolyte additives + concentrations**
- **Separator permutations**
- **Grid alloy development**
- **Other…**

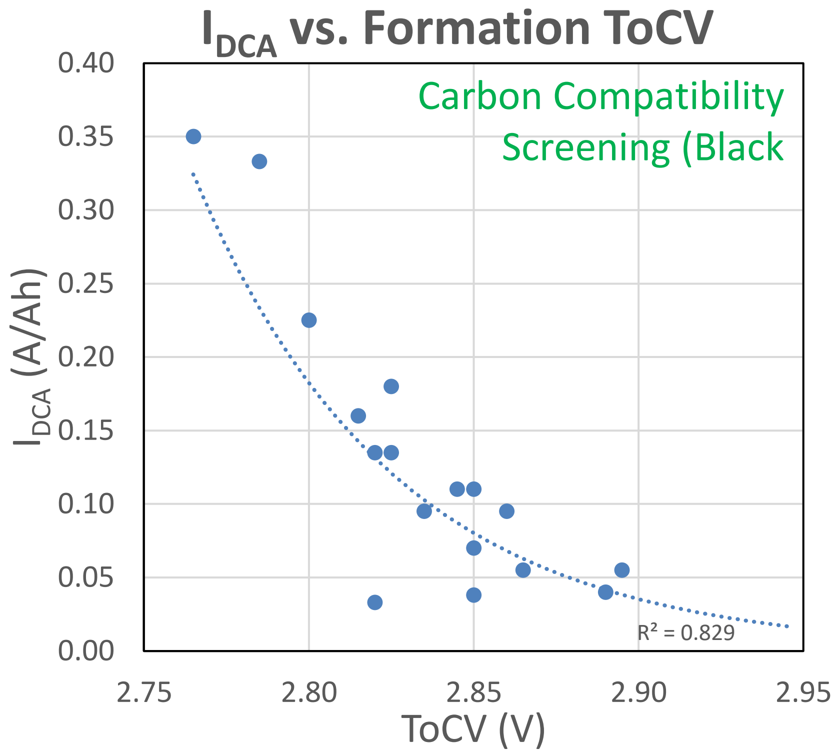


Pavlov, Petkova, Rogachev. JoPS. 175 (2005)

Figure 20: Charge acceptance current measured after 10 minutes (2.40V, 0°C) average of 4 cells per variable error bars show ±1σ.



Hammond, Lignotech, Moura. ALABC Project #1012J. May 2012



Black Diamond Structures, LLC. Internal Data 2019

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Academic and Mechanistic Understanding

- **Example**
  - Review and mechanism of action of new additives (ex. Poly(aspartate) – "DS")
  - Chemical surface groups as performance levers for advanced carbons(O, S, N)
  - How/Do carbons change the NAM in ways which drive charge acceptance?

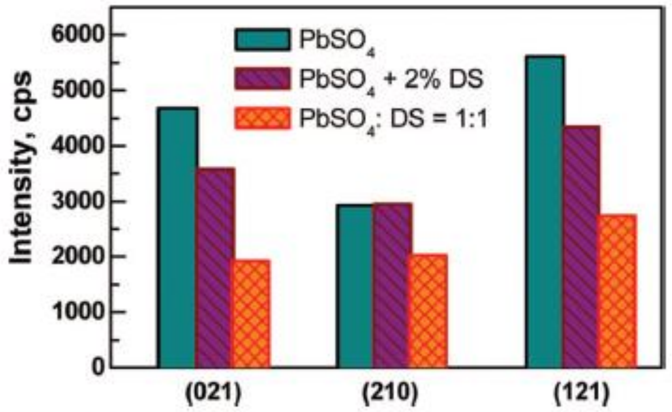

Figure 20. Intensity of the diffraction peaks at 2θ = 26.71° (021), 2θ = 27.69° (210) and 2θ = 29.68° (121) for $PbSO_4$ crystals treated in H_2SO_4 solutions with or without addition of DS.

Pavlov + Nikolov. J. Electrochem. Soc. 159(8) A1215. 2012

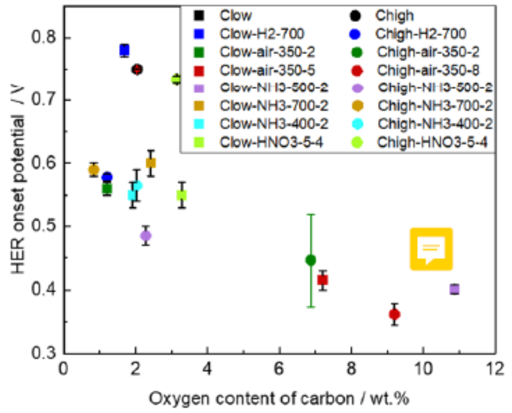

Figure 22. Relationship between the HER onset potential of carbon electrodes and the oxygen content of carbon.

Fraunhofer ISC, Wroclaw University. ALABC Project #1012J. Feb 2022 (Ongoing)

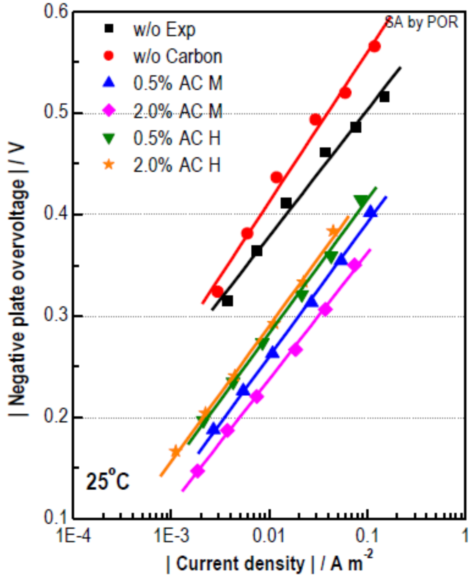

Figure 8. Tafel plots (at temperature of 25°C) for cells with different activated carbon additives to NAM and the respective linear regression trend lines

BAS. ALABC Project #1618. Sep 2017

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KEY CELL-BASED RESEARCH CONSIDERATIONS

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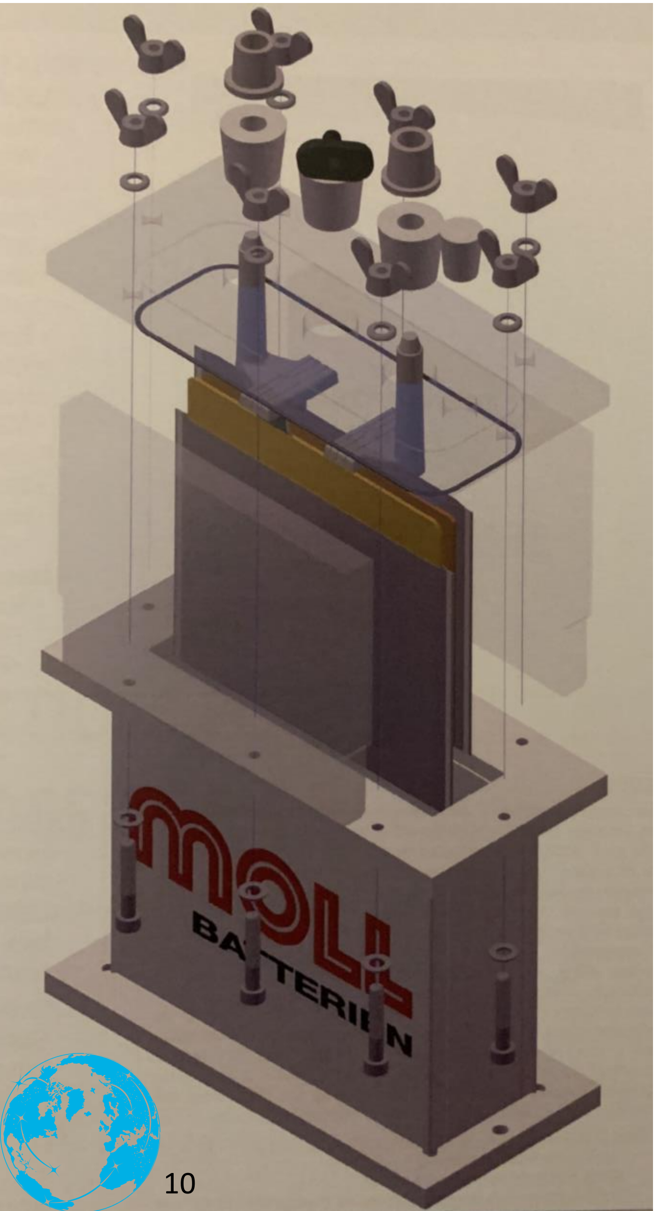
Globally Recognized Best Practices – A History

PNL



- NAM cell testing was discussed during the 1st ALBA at Kloster Eberbach
- Initial discussion featured several methods and challenges associated with the construction of sub-scale cells
  - Up until then, research cells "Best Practices" had not been brought together.
- The topic has grown year to year, gathering new info and presenting on relevant topics of study.
- DCA has been a key variable in tests run with cells, also a testing vehicle for current CBI projects.
- The current Best Practices began to focus on elements of DCA
  - Before proper discussion of DCA scaling factors and cell specific precautions, the link between cells and 12 V batteries needed to be discussed and studied further

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Globally Recognized Best Practices Embodied

PNL

Best Practices for Cell Based Research

Revision 2.5 From 6 June, 2022

Matthew Raiford, Ph.D.

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Research and development of lead batteries c
as the basis. This document will outline a se
research to insure and provide higher quality a

Appendix 7.3: These pages are copied from material prepared at and distributed to all participants of the **CBI/CENELEC workshop "EFB&Heat", Bruges, 22/23 May 2019**

Appendix:
Draft Instructions for DCA test according to EN 50342-6:2015 when performed on test cells

Eckhard Karden, Sophia Matthies, Begüm Bozkaya, Shane Christie, Jonathan Wirth

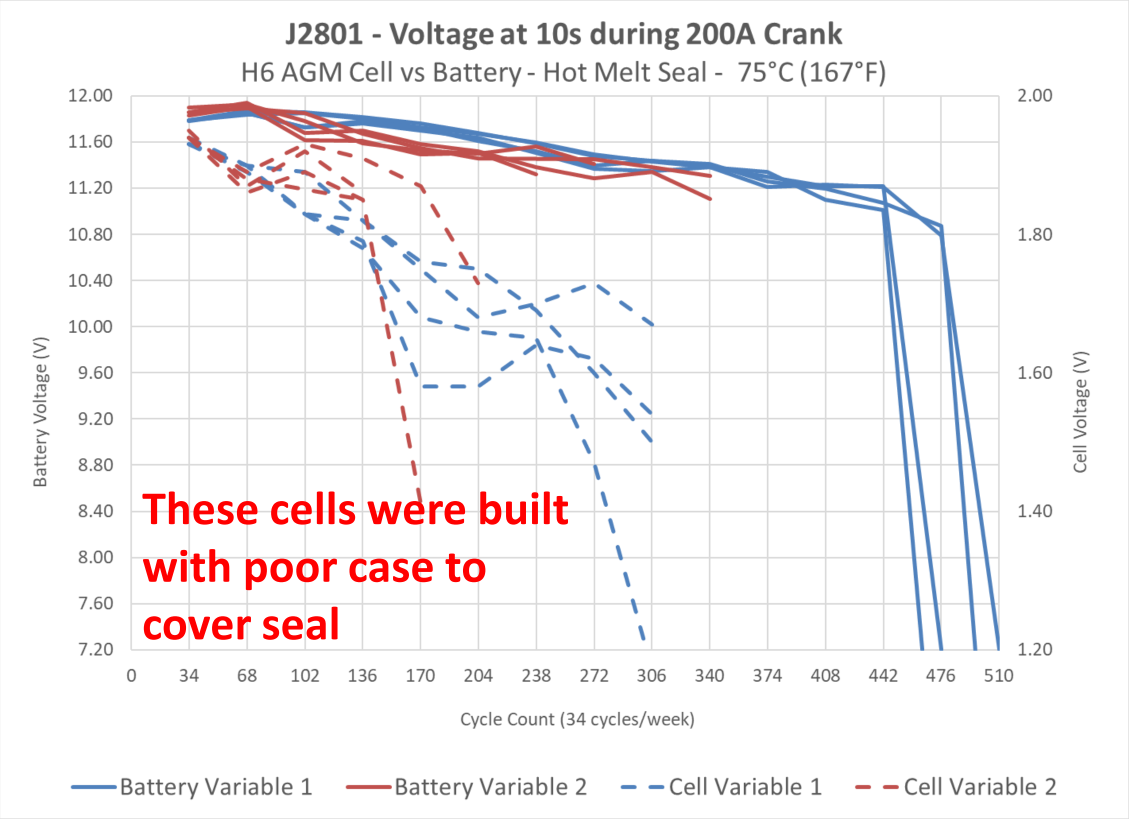
Several participants of the workshop requested detailed instructions for performing a complex DCA test procedure on laboratory test cells, typically 2P1N or 3P2N configurations consisting of automotive (~10 Ah) or significantly smaller (often ~2 Ah) plates. The following draft summarizes experience collected by the authors and suggests a modified version of EN 50342-6:2015, §7.3, for laboratory implementation with test cells.

- **From the breakout, came several outputs**
- **Best Practices Guide covering:**
  - Cell construction and sealing
  - Materials and methods
  - Examples of cells
  - High temperature considerations
  - Troubleshooting and more…
- **EN 50342-6:2015 DCA specific instructions for cells**
  - Sizing the test to small Ah 2V cells
  - Construction considerations
  - Connection considerations
  - Data analysis homologation and possible issues

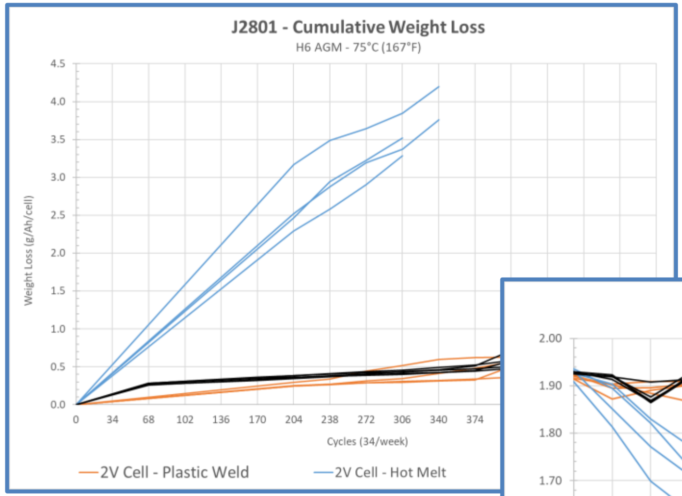
Importance of Cell Sealing

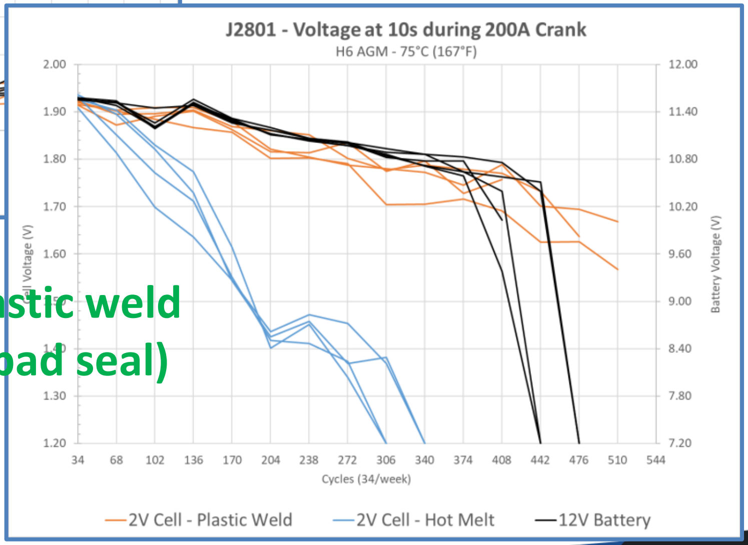
1C3

- **Analogous cell results can be produced with not optimal sealing**
  - Quicker failures
  - Maybe changes failure mode



- **However, a good seal is needed for cell data to represent battery data**
  - Similar voltage data and cycle count





These cells compared plastic weld (good seal) to hot melt (bad seal)

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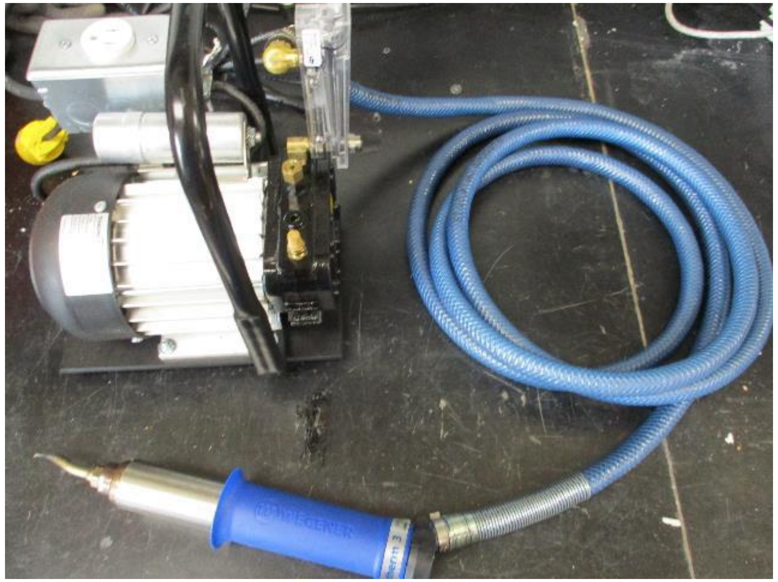
Importance of Cell Sealing – Correct Tooling

1C3

- **Heat sealing**
  - Its what battery do
- **Plastic welding**
  - Easier for small scale lab testing
  - As strong as heat sealing



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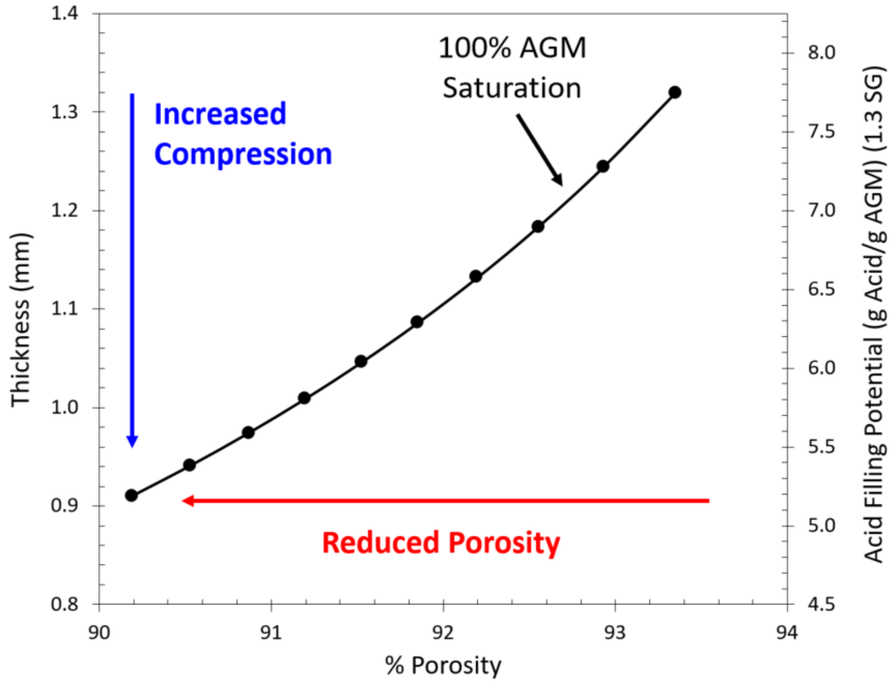
Compression and Saturation

- **Compression and Saturation are defining properties of an AGM battery design. A well functioning AGM cell/battery relies on consistent, well-regulated values for both.**

- **Why are compression and saturation important? How are they defined, and how are they measured?**

- **Compression**
  - Design/manufactured compression (DUF state) vs. Operating (wet) Compression – not the same!
  - Relationship with pore volume and impact on saturation
  - Variation with SoC, cycling, lifetime – compression is not a fixed value…



- **Saturation**
  - Definition of Saturation: Separator, Cell, Active Material, Other?
  - How to measure / calculate / estimate
  - Impact of State of Charge, Time, Charging State (steady state vs pulse), Temperature.
  - Impact on electrical/battery performance (resistance, cranking performance, cycling life, etc)

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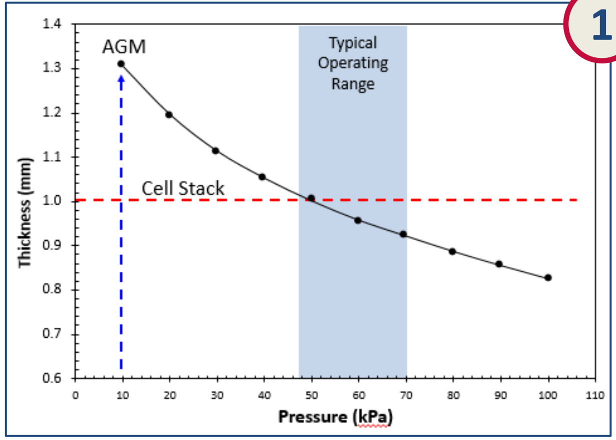
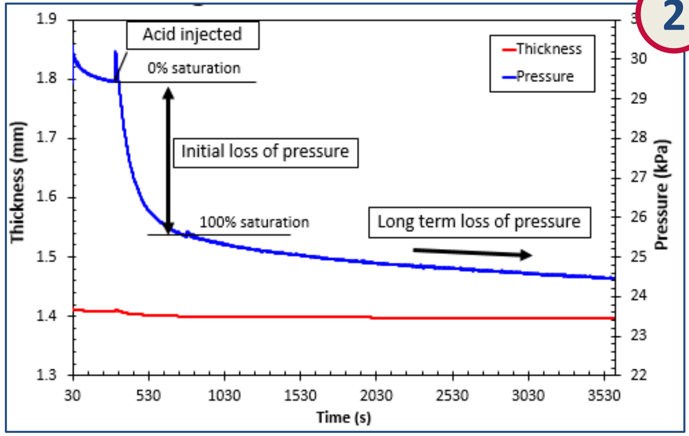
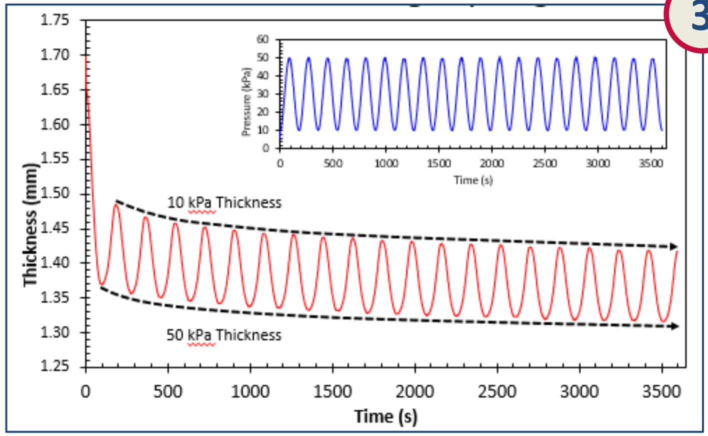
Compression / Vacuum Filling on Sub-Scale Level

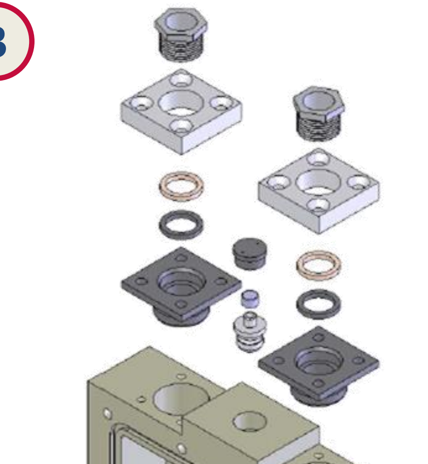
- Studying compression in AGM separator on cell assembly and pressure maintenance through filling and use

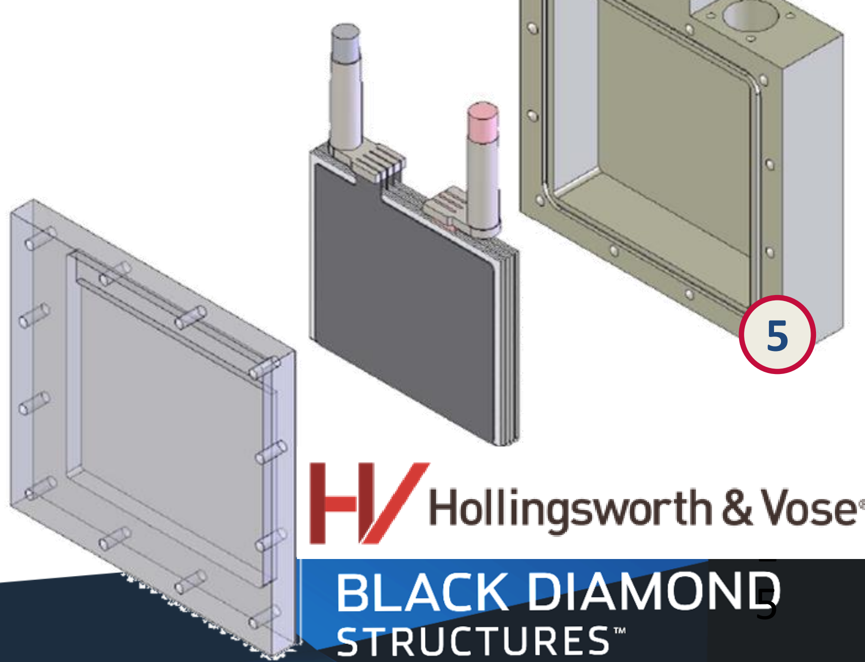






1. AGM compression, linking plate gap and pressure with nominal thickness
2. Measuring loss of pressure when adding acid to the AGM
3. AGM elasticity measurement, proxy pressure maintenance measure during cycling
4. H&V acid filling machine
5. H&V reusable 2V cell design





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Advanced Analysis Methods

- **Operative AGM single cell**
  - Made of a single plexiglas piece with thick, transparent walls and a side cover with a rubber gasket, preventing leakage of fluid and gas.
  - Enables specific investigations that are otherwise laborious and costly if conducted with full battery batches.
  - Enables the monitoring of various parameters (i, Ucell, U+/-, T, P, X, f…) without the effect of cell-to-cell inconsistency.

CLARIOS
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EFB → AGM → AUX/Storage?

FUTURE PURSUITS

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Which Applications Lend Themselves to Sub-Scale?

- **Automotive EFB?**
  - Relatively easy to reproduce on the sub-scale
  - With care, can mimic full-scale AM:acid and plate ratio
  - Decades of work poured into the designs
  - A wealth of community support

- **Automotive VRLA?**
  - Processes which work well for industrial scale, are hard to shrink to the sub-scale
    - Ex. Saturation, vacuum filling
  - More cost intensive, but front loaded

- **Automotive AUX?** → ***Discuss in Breakout session!!***
  - EFB or VRLA?
  - Is an existing EFB or VRLA test vehicle acceptable to evaluate AUX protocols and formulae?

- **Solar / Stationary?**

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Where Should Cell Development Groups Focus?

- **Automotive AUX**
  - Charge recovery over minutes (ex. Modified EN-50342-1, cold temperature performance)

- **Corrosion vs. water-loss for float and non-float applications**

- **Performance-relevant mechanistic exploration**
  - Ex. DCA Memory Experiments

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