Polyethylene Separators Must Survive High Temperature Endurance (HTE) Testing

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September 6, 2023

Outline

01 Introduction to High Temperature Endurance Testing (HTE)

02 Failure Modes at HTE Testing

03 HTE Test Results (8 batteries/different producer)

04 Polyethylene Separator Oxidation Resistance

05 HTE Test Results (1 battery)

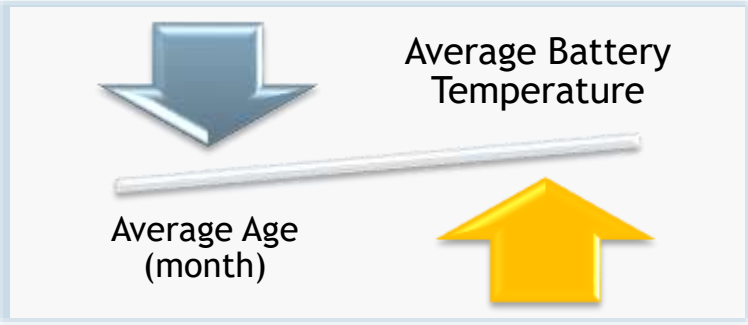
06 Conclusions

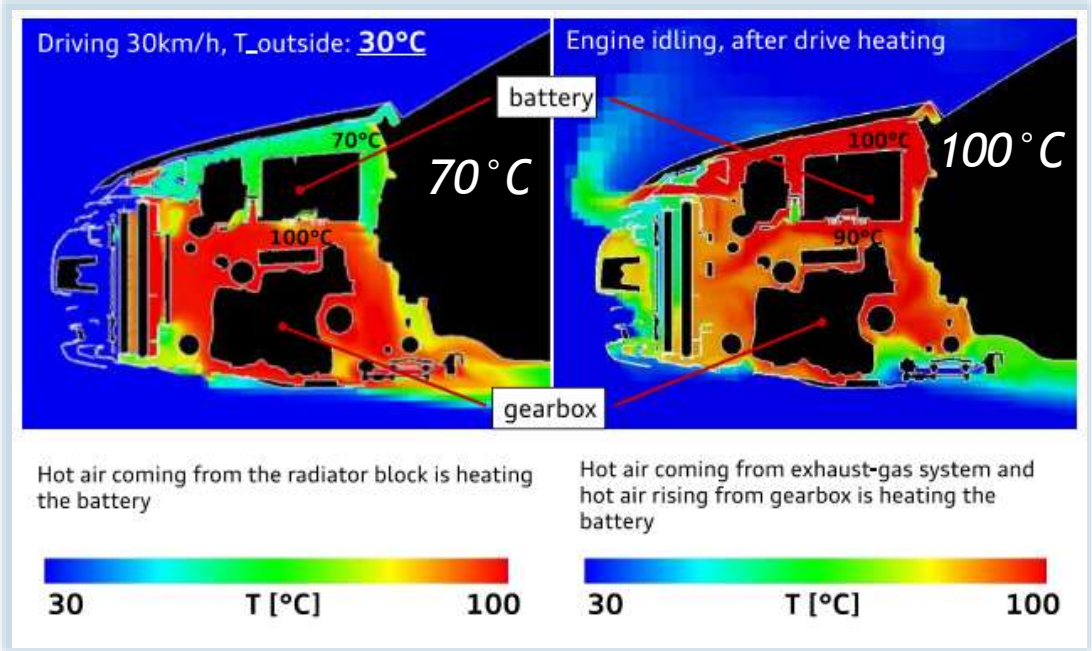
Batteries Are Getting Hotter

- **Polyethylene (PE) separators contributed significantly to the improvement of battery life. PE separators have also helped improve cold crank performance at low temperatures**

- **Batteries must survive under <u>hotter</u> environments today compared to years past, due to:**
  - Under-hood temperature increases
  - Under-hood crowding
  - Climate change

- **Heat influence on battery life:**





Source: Audi - E. Lodowicks 2018

High temperature is causing stress to the positive grid and PE separator

Battery Testing at High Temperatures

1995

- Water Consumption
  - Temperature 40°C
  - IEC 60095-1:2006
- Endurance Testing
  - Temperature 25°C
  - DIN EN 50342:2005
- DARAMIC **Separator** Testing
  - Temperature 60°C
  - Rapid Overcharge Test

2012

- Water Consumption
  - Temperature 60°C
- Endurance Testing
  - Temperature 40°C/75°C
  - EN 50 342-1:2015
  - SAE J240:2012
  - SAE J2801:2013

Today

- Water Consumption
  - Temperature 60°C
- **Endurance Testing**
  - Temperature 75°C

NEW STANDARD(s)

➔ Need of a new high temperature durability test for automotive lead-acid batteries

High Temperature Endurance test*

RWTH AACHEN UNIVERSITY

Ford

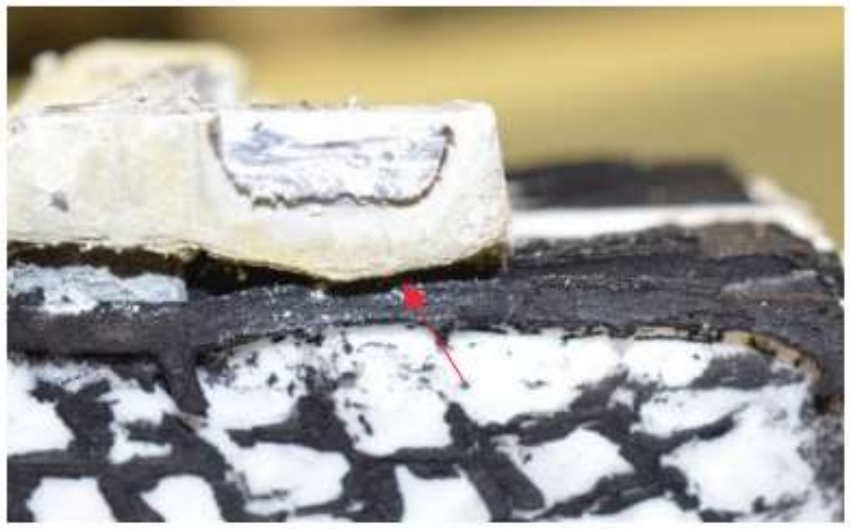
Searching for a laboratory battery test that reflects real world failure modes of batteries in actual applications

Potential Failure Modes at High Temperature Battery Testing

Note

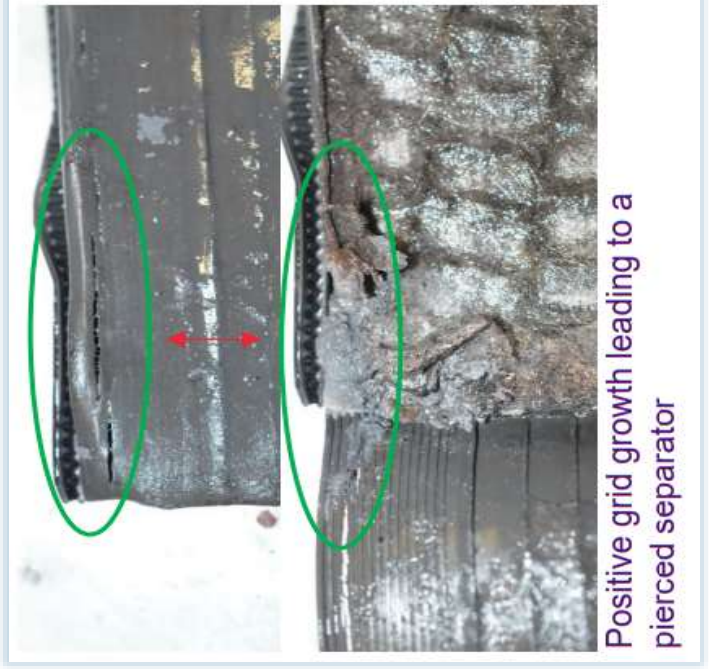
Grid growth of positive plate

in vertical direction



Positive grid growth leading to a connection with negative COS

in horizontal direction



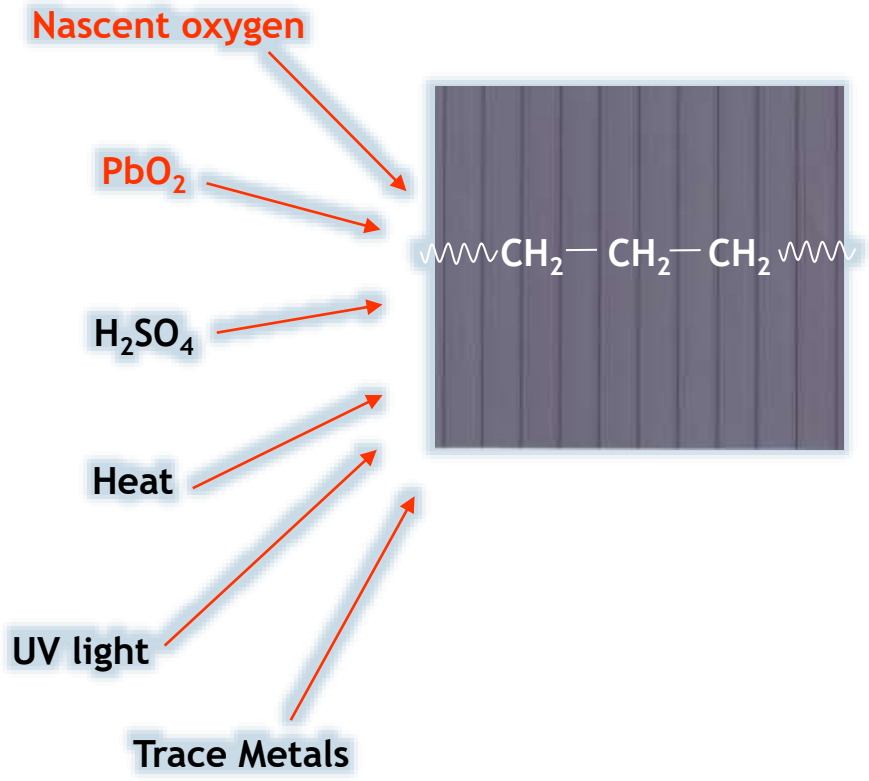
COS = Cast on Strap

Source: Pictures from ALBA workshop in Bergamo 2022

Positive grid growth is the expected failure mode

Potential Failure Modes at High Temperature Battery Testing

- PE separator strength/robustness

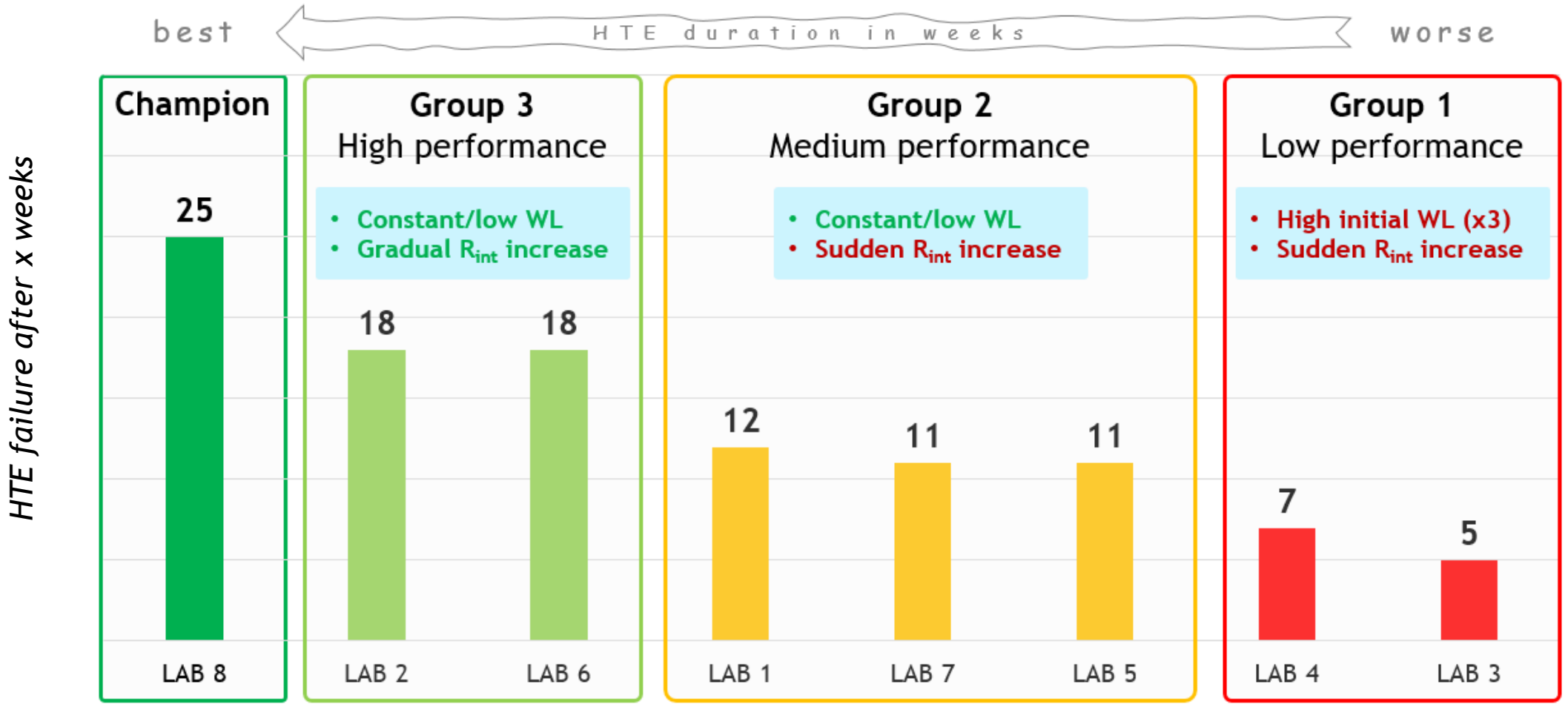


Note: Oxidation stability of PE separators is high…

Note: but not independent on the degree of positive grid growth

Daramic's target: The PE separator should not be the original failure mode at 75°C HTE testing

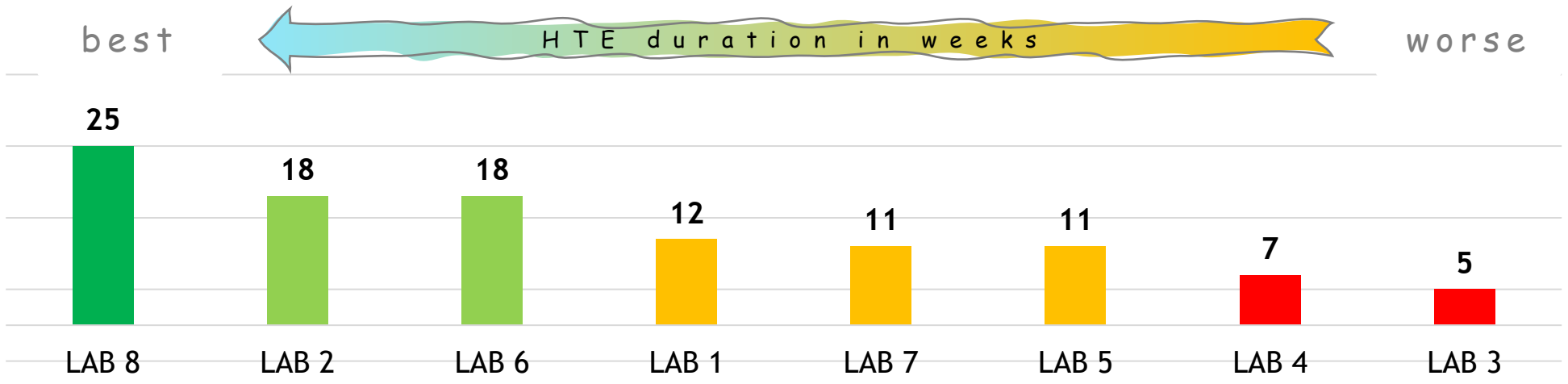
HTE - First Round of Testing Various Lead Batteries (70 Ah)



***LAB** = Lead-Acid Battery* ***WL** = Water Loss* R_{int} *= Internal Resistance*

What was the battery failure mode and our learnings in respect to the PE separator?

HTE - Time Until Failure (weeks) and Failure Modes



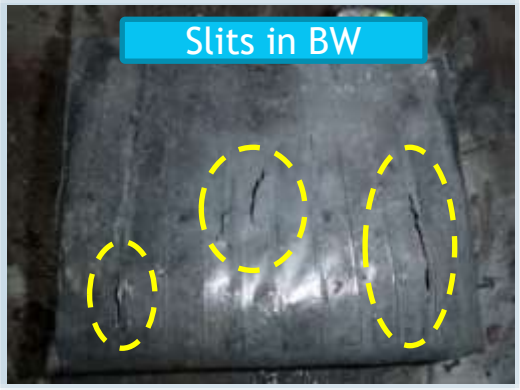
(+) Average grid growth (mm)								
• *vertical*	+ 7	+ 5	*)	+ 5	+ 8	+ 9	+ 11	+ 7
• *horizontal*	+ 5	+ 6	*)	+ 6	+ 6	+ 7	+ 10	+ 5
Sep. condition	n.a.	++	o (splits)	++	+	o	o (splits)	+
Battery Failure Mode	Side shorts	Vertical grid growth	Grid corrosion	Corroded strap	Vertical grid growth	Vertical grid growth	Horizontal grid growth	Manufacturing defect

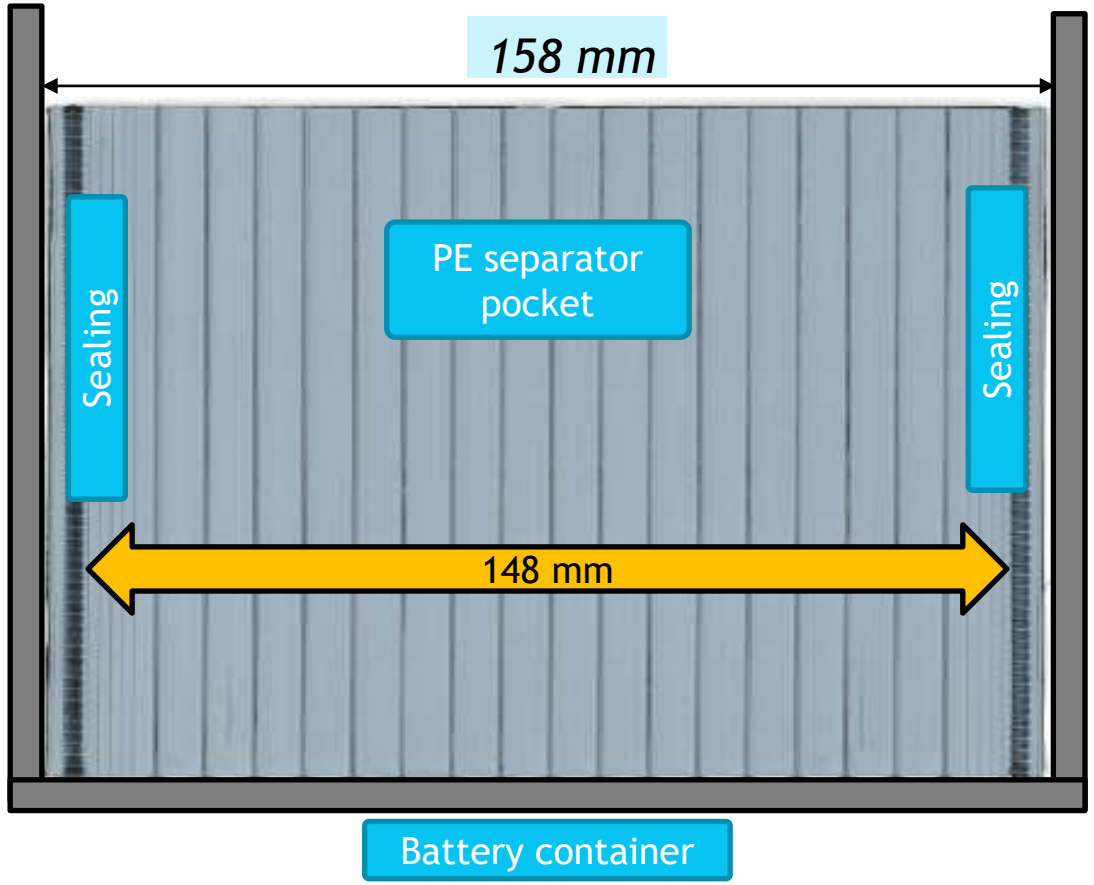
**) positive grid completely corroded - not measurable* *++ = very good* *+ = good* *o = acceptable*

What happens first? Grid growth causing PE separator degradation or PE degradation causing shorts with subsequent grid corrosion?

Failure Mode: Horizontal Grid Growth (schematic)

- **Pocket size** (typical)
  - Width: 158 mm
  - Height: 119 mm
- **Plate size** (typical)
  - Width: 143 mm
  - Height: 113 mm
  - Thickness: 2 mm





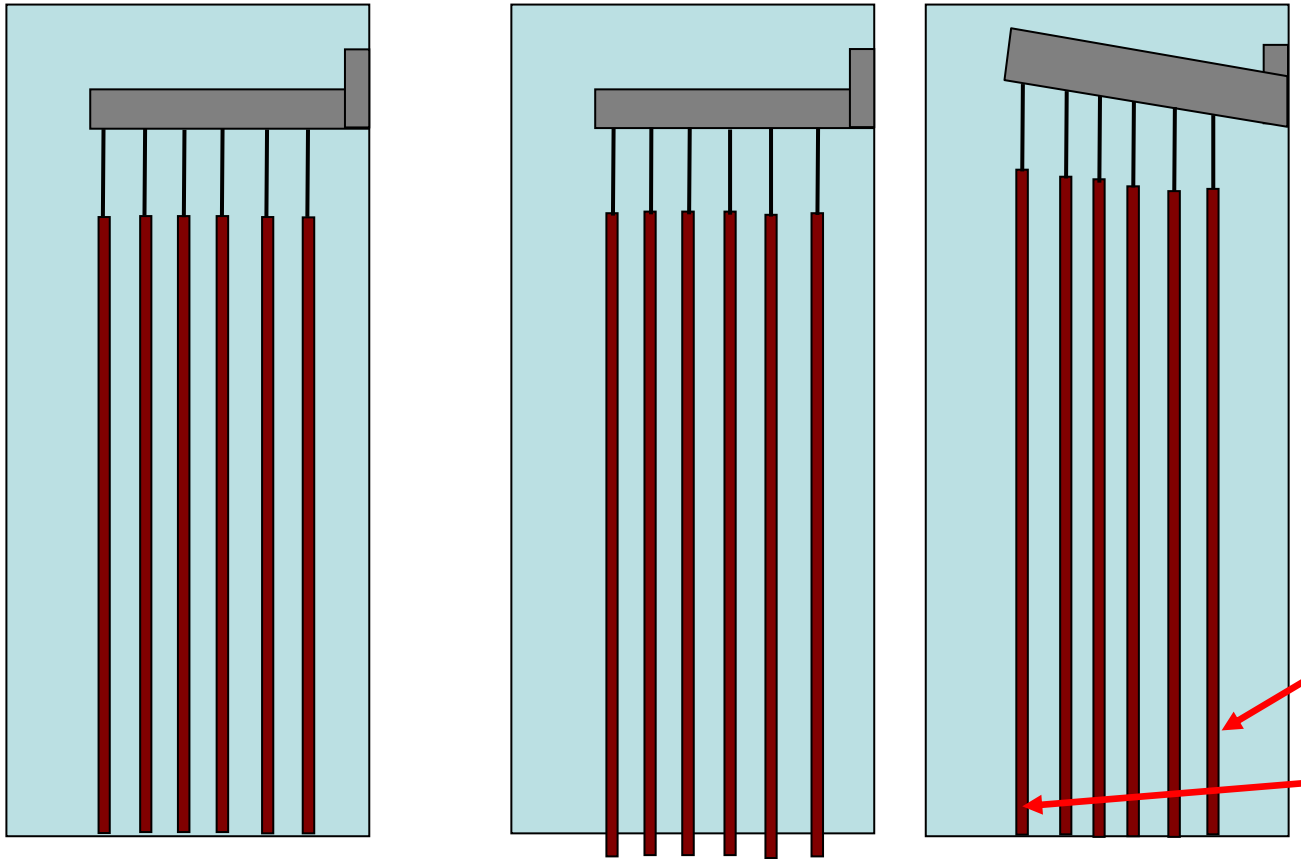
BW = Back Web *PAM* = Positive Active Material

Any horizontal grid growth larger than 3 to 5 mm will results in mechanical stress to the PE pockets. The envelope separator will stretch and may rupture (= risk of splits) when exceeding 5 mm growth.

Failure Mode: Vertical Grid Growth (schematic)

Pos. plate height
113 mm (original)

Pos. plate growth to + x mm due to grid corrosion



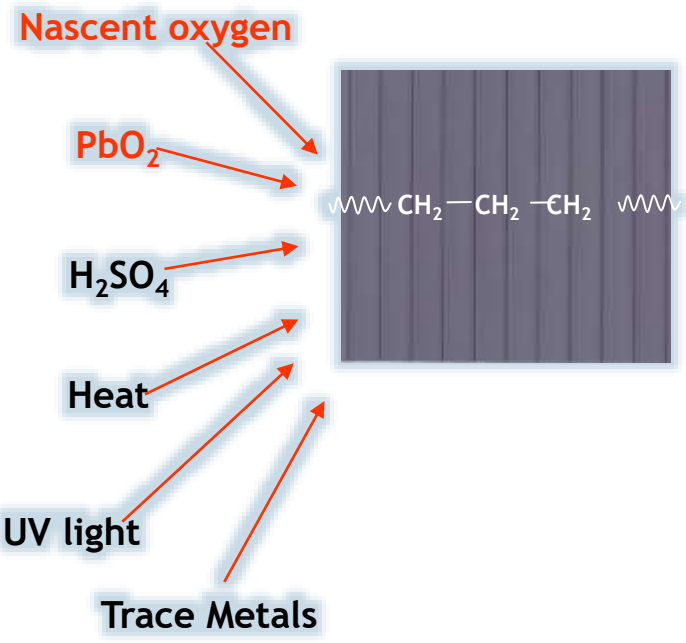
Deformation of bridge

Positive grid growth higher than 5 to 6 mm creates top shorts between pos. and negative plate and a damaged pocket crease area (& PAM shedding). The separator crease area is particularly vulnerable.

Separator Oxidation Resistance

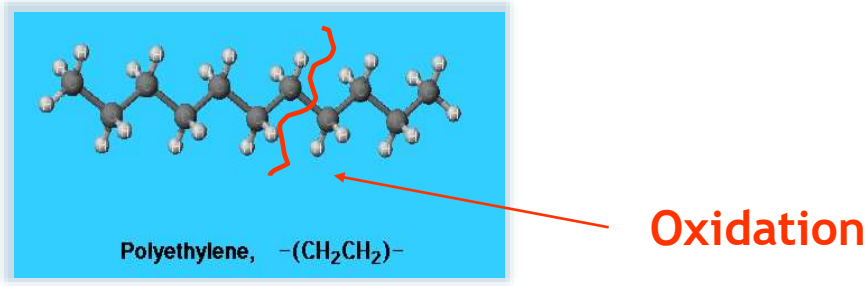
Definition

- Oxidation resistance or oxidation stability is the separator ability to withstand the aggressive medium of the lead battery, namely sulfuric acid, positive active material (PAM) and extremely reactive nascent oxygen and peroxides



What does it mean?

- During battery life, the PE separator UHMWPE[1)] molecular weight is reduced, which results in a reduction of the separator elongation (elasticity)

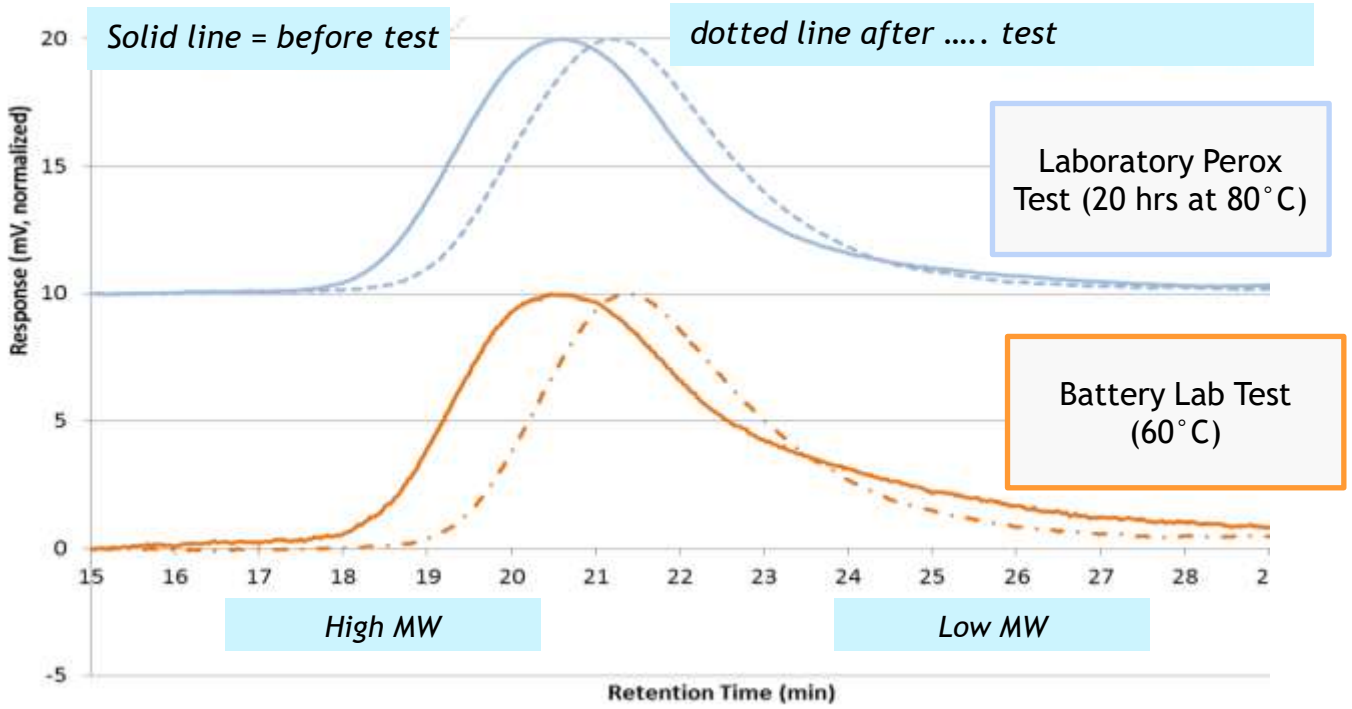


- Gel Permeation Chromatography (GPC) studies have proven that the loss of elasticity is caused more by chain scission than by PE crosslinking

1) UHMWPE = Ultra High Molecular Weight Polyethylene

How can you evaluate PE separator oxidation resistance?

Gel Permeation Chromatography (GPC)



High Molecular Weight (MW)
= High CMD Elongation = Sep. is very flexible

Low Molecular Weight (MW)
= Less CMD Elongation = Sep. might become brittle

CMD = Cross Machine Direction

- PE separator oxidation is resulting in lower PE molecular weight
- Perox 80 test (20 or 40 hours) is a good laboratory test
- Measurement of remaining separator elongation is an indirect measurement, but a faster test

GPC allows direct measurement of oxidation, while elongation loss is an indirect measurement

What is Really the Life Limiting Factor at 75°C Battery Testing?

DARAMIC

Positive grid corrosion/growth or a failed PE separator pocket, due to poor oxidation resistance?

A second HTE test was conducted with same EFB batteries lasting around 18 weeks.

Separator Properties of DARAMIC HP 250:

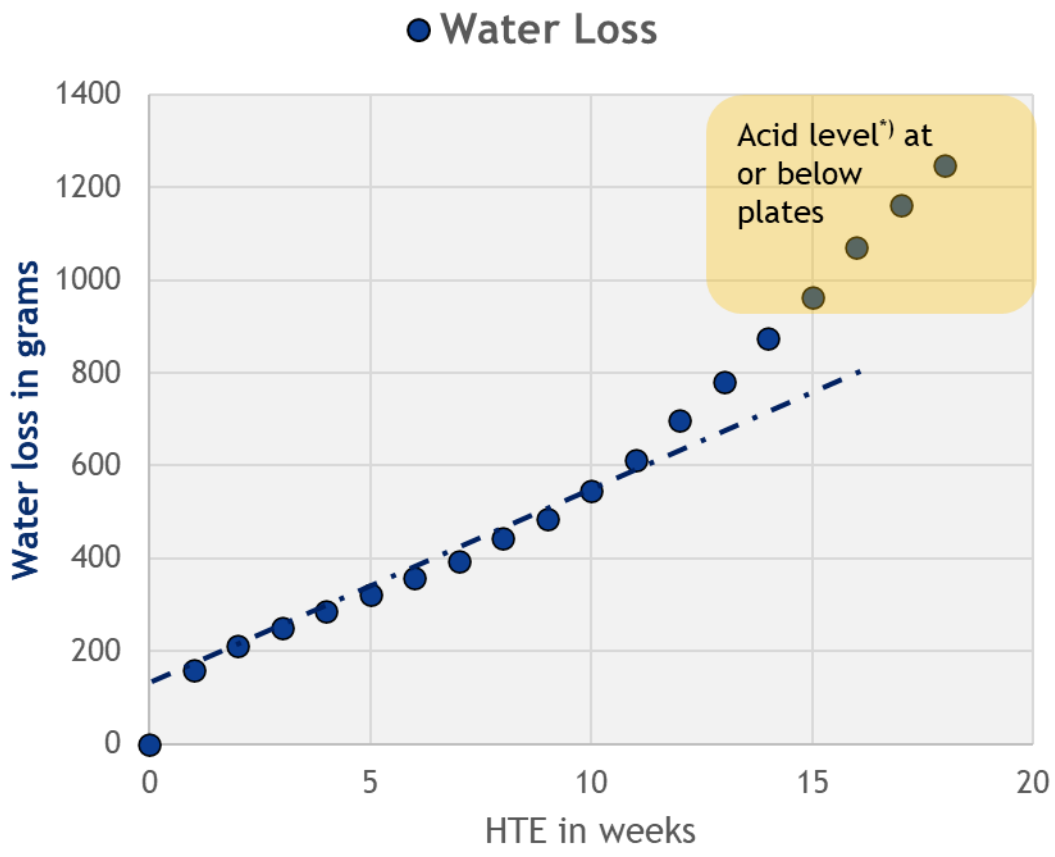
Overall Thickness:	*0.80 mm*
Backweb Thickness:	*0.250 mm*
Pocket Width:	*160 mm*
Pocket Height:	*119 mm*
Electrical Resistance:	*60 mΩ.cm²*
Puncture Resistance:	*13.4 N*
Total Oil content:	*17.7 %*

Oxidation Resistance

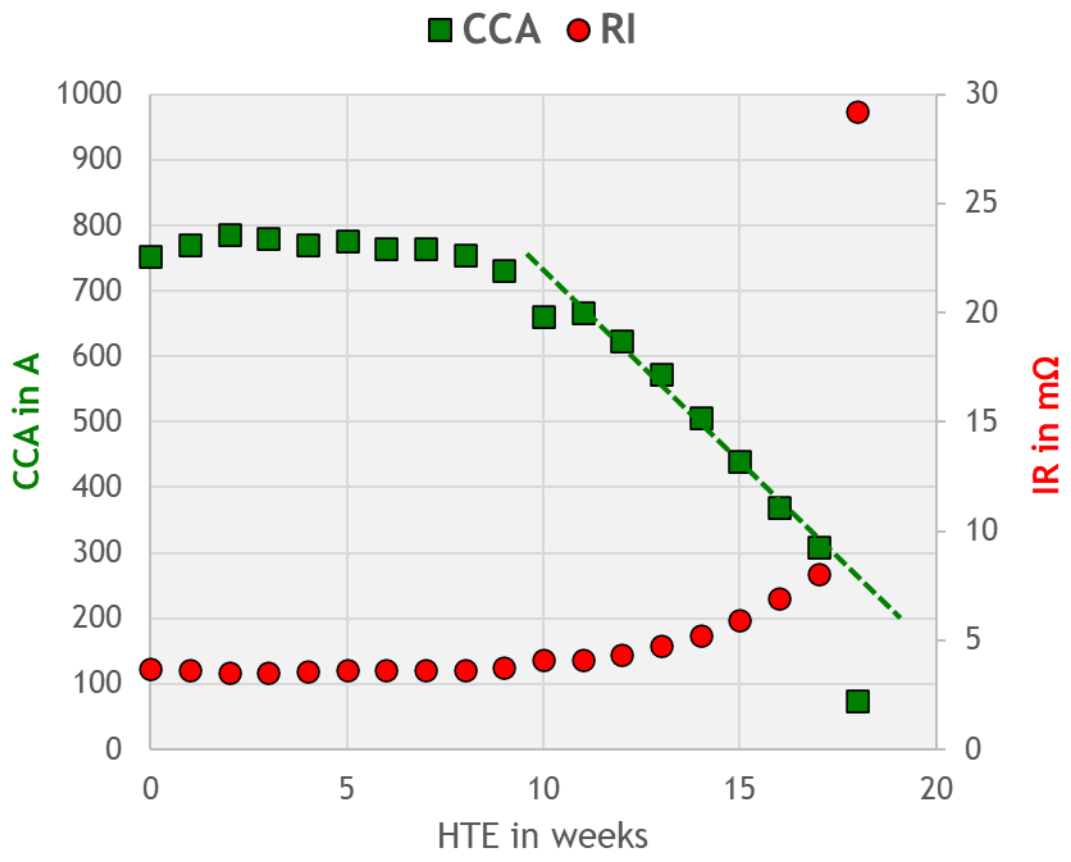
- *0 h*	*363 %*	*(100%)*
- *20 h*	*312 %*	*(86%)*
- *40 h*	*205 %*	*(56%)*

EFB batteries were torn down after various time intervals during the HTE test, for example, not only at the end (after failure)

Trend of CCA, Internal Resistance (RI) and Water Loss



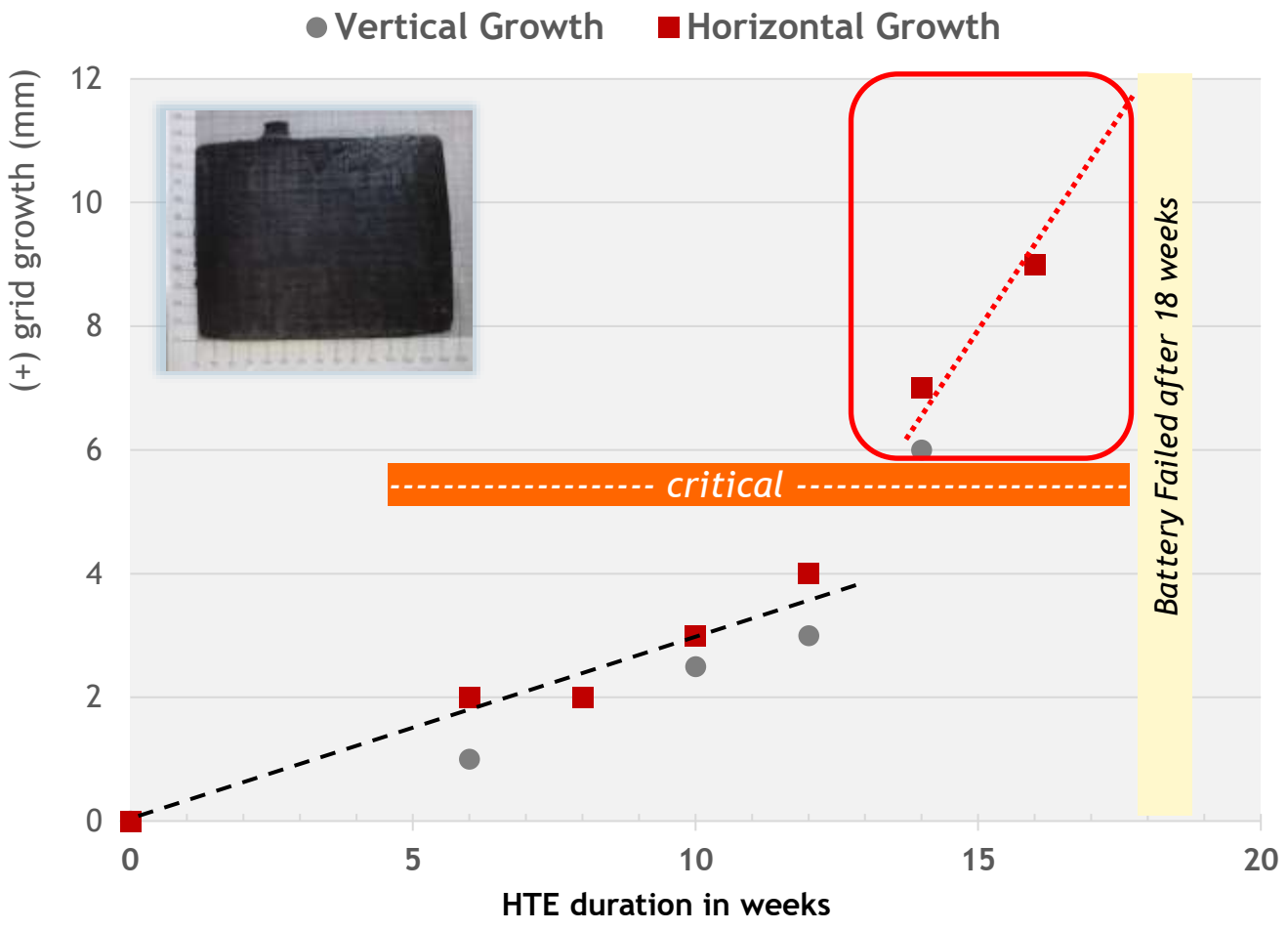
*) Acid densities > 1.3 g/cm³

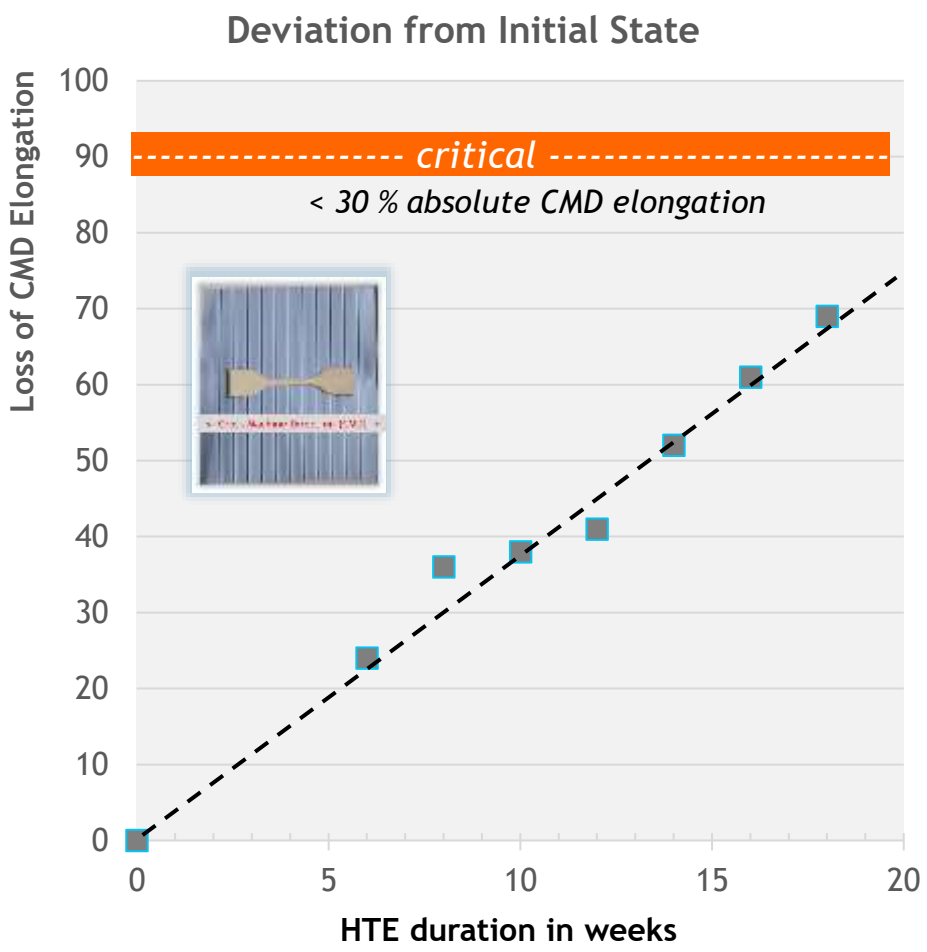


***CCA** = Cold Crank Amperage* ***RI** = Internal Resistance*

The last battery failed after 18 weeks

Learnings from HTE Test 2





CMD = Cross Machine Direction

Daramic PE separators survive operating temperatures at 75°C provided the other parts are also designed for high temperatures

Final Conclusions

01 DARAMIC PE separators can be used in batteries that need to pass High Temperature Endurance (HTE) testing

02 DARAMIC PE separators survive operating temperatures at 75°C, provided other parts of the battery are also designed for high temperatures

03 The lead alloy of the positive grid and the grid forming process plays an important role on grid corrosion and growth. Positive grid growth cannot be prevented but delaying and limiting plate growth is helping to extend battery life

04 In case HTE battery tests do not show expected results, we should work together and optimize the system

05 It is important to understand and determine the battery failure mode during the HTE test

06 Consider DARAMIC products that can slow positive grid growth and water loss

Thank You For Your Attention and Please...

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