H2NEW
Hydrogen from Next-generation Electrolyzers of Water
U.S. DEPARTMENT OF ENERGY

Membrane Pretreatment and Cell Conditioning for Proton Exchange Membrane Water Electrolysis

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Presenter: Elliot Padgett

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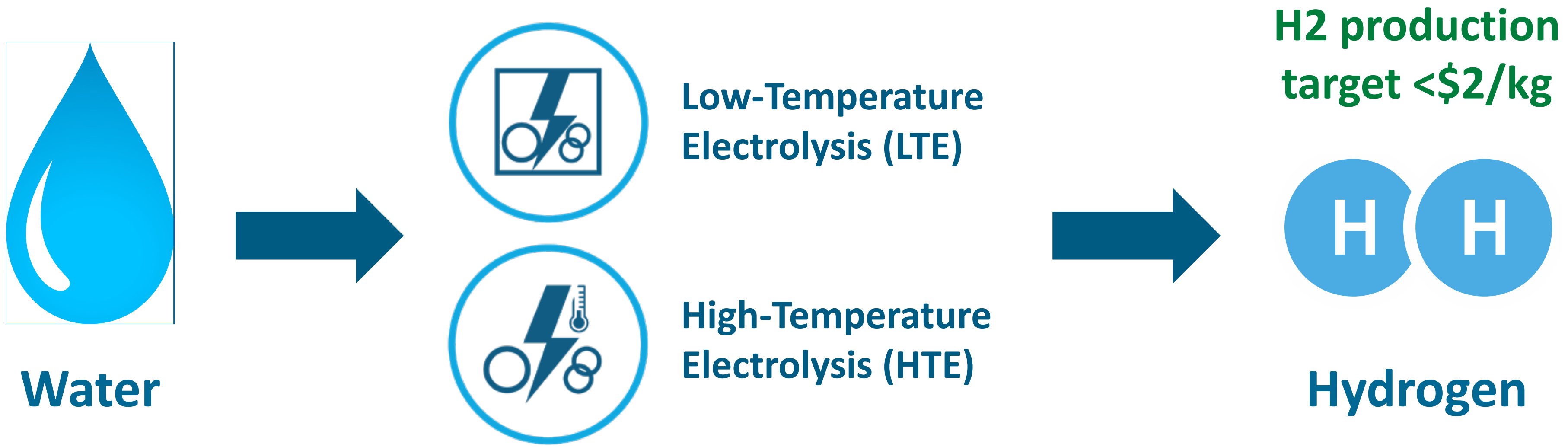
H2NEW Consortium Overview

Goal: H2NEW will address components, materials integration, and manufacturing R&D to enable manufacturable electrolyzers that meet required cost, durability, and performance targets, simultaneously, in order to enable \$2/kg hydrogen.



H2NEW has a clear target of establishing and utilizing experimental, analytical, and modeling tools needed to provide the scientific understanding of electrolysis cell performance, cost, and durability tradeoffs of electrolysis systems under predicted future operating modes

Electrolysis cell "conditioning" or "break-in"

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- Membranes and CCMs may receive chemical "pretreatments" before cell assembly and testing. E.g. soaking in DI water or acid, flat drying on a vacuum table.
- Before performance testing, electrolysis cells are typically operated using an initial "conditioning" or "break-in" protocol. Two examples used at NREL:
  - 0.2 A/cm^2 for 30 min; 1.0 A/cm^2 for 30 min; 1.7 V until current varies <1% per hour (~8-12 hr). [1]
  - 0.2 A/cm^2 for 1 hr; 1.0 A/cm^2 for 1 hr; 2.0 V for 30 min; 1.7 V for 2 hr; 2.0 V for 30 min. [2]
- Various other protocols are in use in the community.

1. G. Bender et al., *International Journal of Hydrogen Energy*, **44**, 9174–9187 (2019).
2. S. M. Alia, S. Stariha, and R. L. Borup, *J. Electrochem. Soc.*, **166**, F1164–F1172 (2019).

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- Various other protocols are in use in the community.
- The mechanisms of conditioning and impacts on cell performance and stability are not well know.
- **Performance stability of the cell after conditioning is not commonly reported**.
- Inconsistent conditioning can contribute to reproducibility challenges in the electrolysis community.

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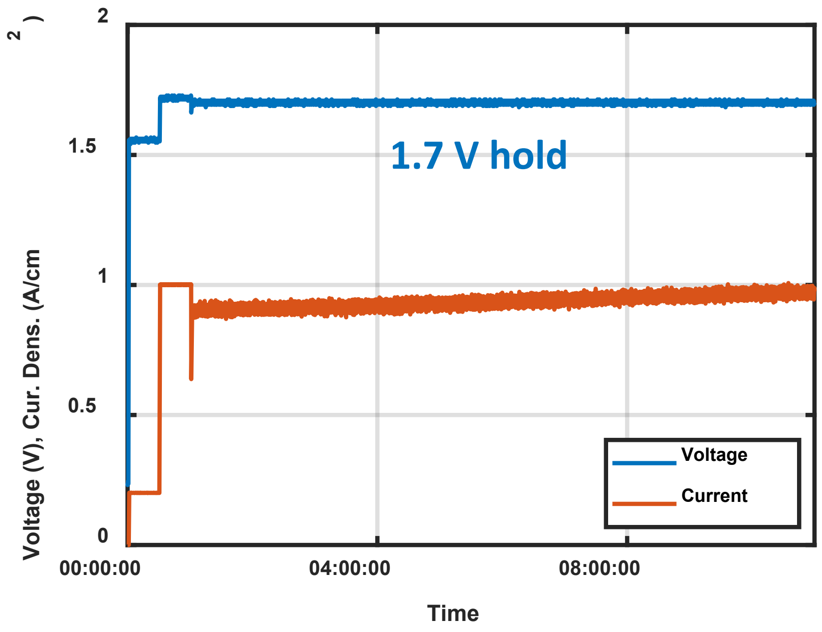
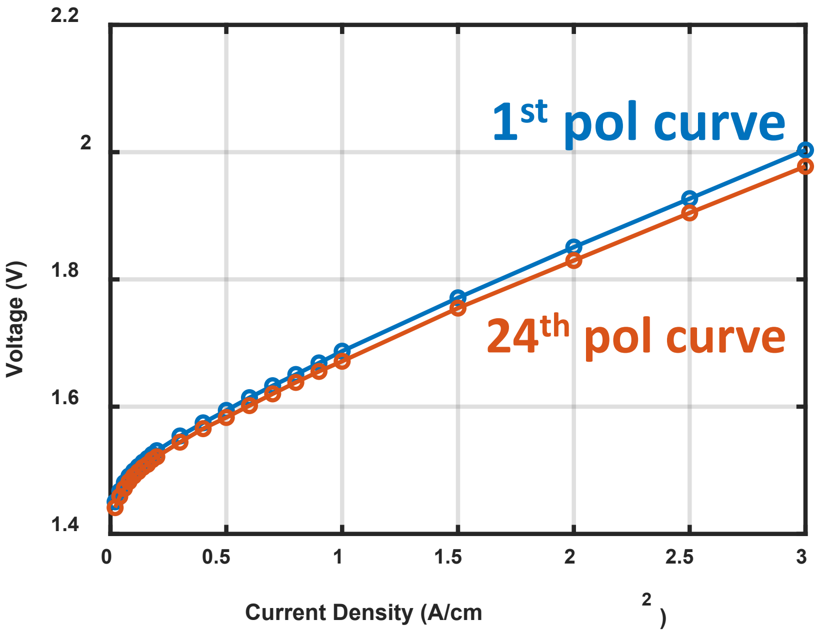
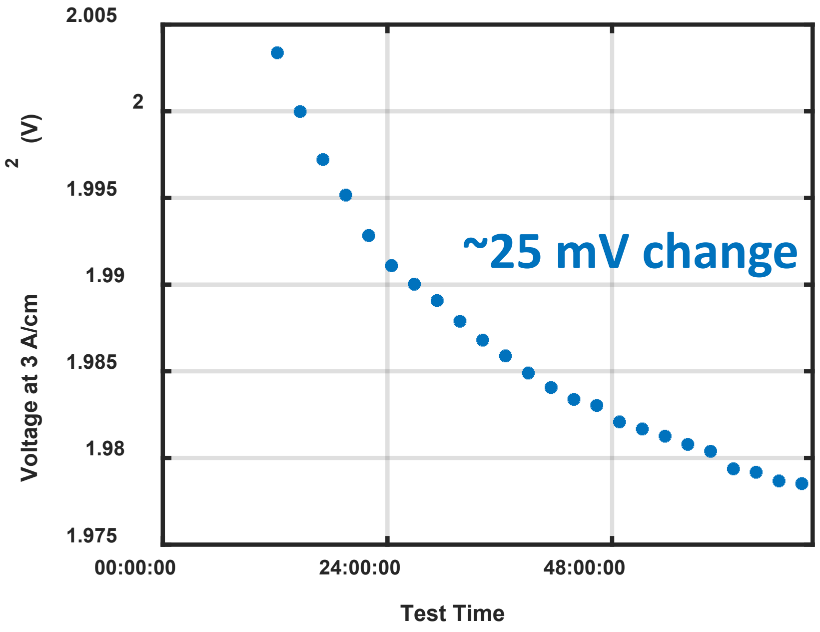
Slow conditioning processes

- Slow conditioning processes can occur that cause continued performance improvements over multiple days.
  - Observed in low loaded (<0.4 mg_{Ir}/cm^2) CCMs fabricated by ultrasonic spray coating.
  - Example: record repeated polarization curves after completing standard conditioning protocol

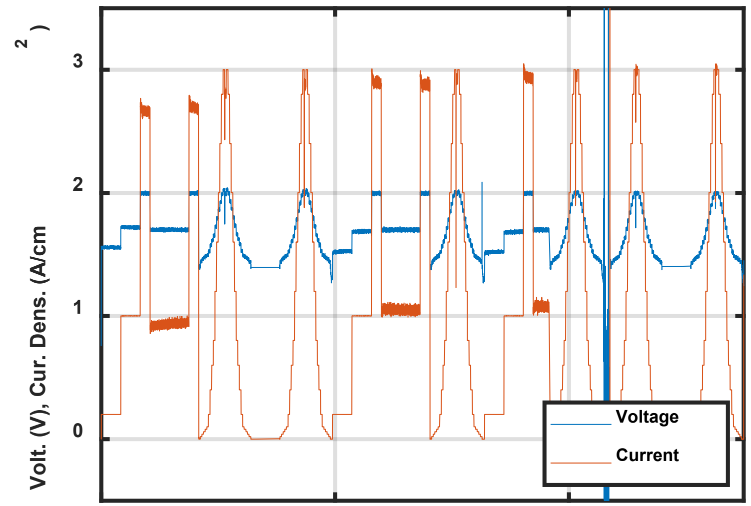






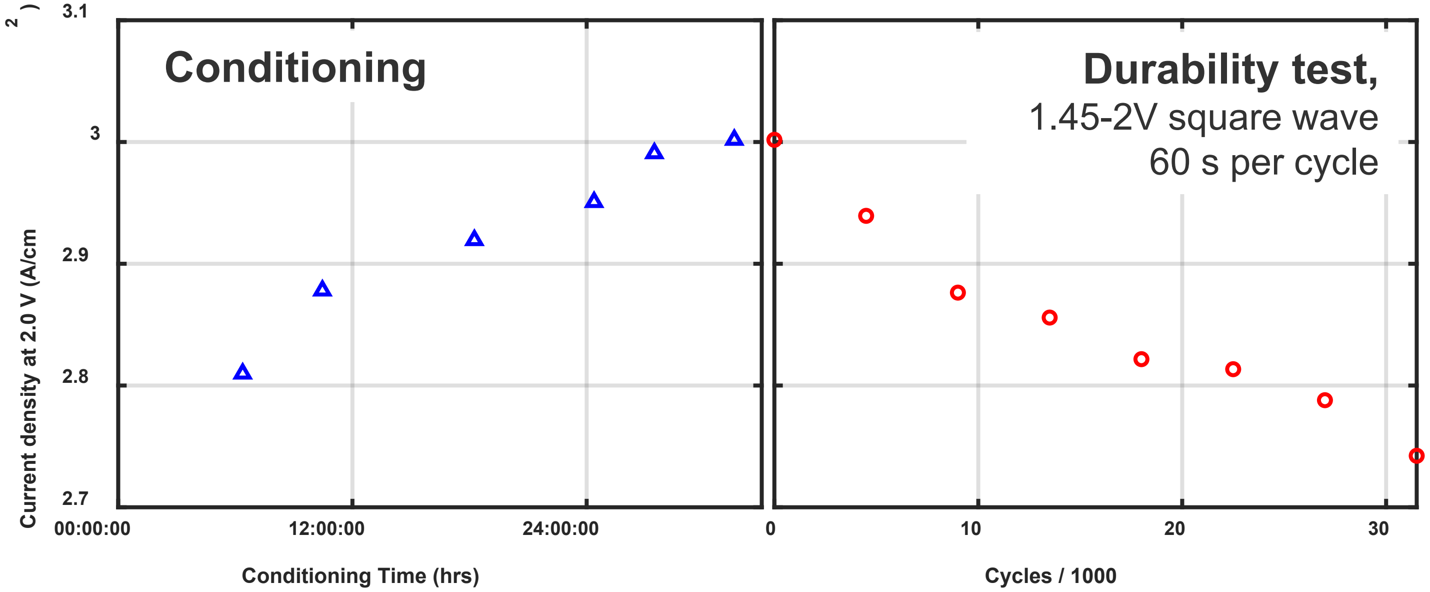
Anode: 0.34 mg_{Ir}/cm^2 IrO_2 (Alfa Aesar). I:Ir = 0.27. 250 um Giner Pt/Ti sintered PTL
Cathode: 0.11 mg_{Pt}/cm^2 46wt% Pt/HSC (TEC10E50E). I:C = 0.45. MGL 370 carbon paper PTL
Membrane: N117, no pretreatment

Importance of conditioning for performance and durability testing



Change in performance from extending conditioning can have similar magnitude to performance loss in durability tests.

Incomplete conditioning can complicate interpretation of performance changes due to degradation.



Anode:
- 0.20 mg_{Ir}/cm^2 IrO_2. I:Ir = 0.27.
- 250 um Giner Pt/Ti sintered PTL

Cathode:
- 0.12 mg_{Pt}/cm^2 46wt% Pt/HSC. I:C = 0.45.
- MGL 370 carbon paper PTL

Membrane:
- N117
- no pretreatment

Testing:
- 25 cm^2 cell, 80 C, ambient pressure
- 50 mL/min water feed both electrodes

Initial holds are a problematic stability metric

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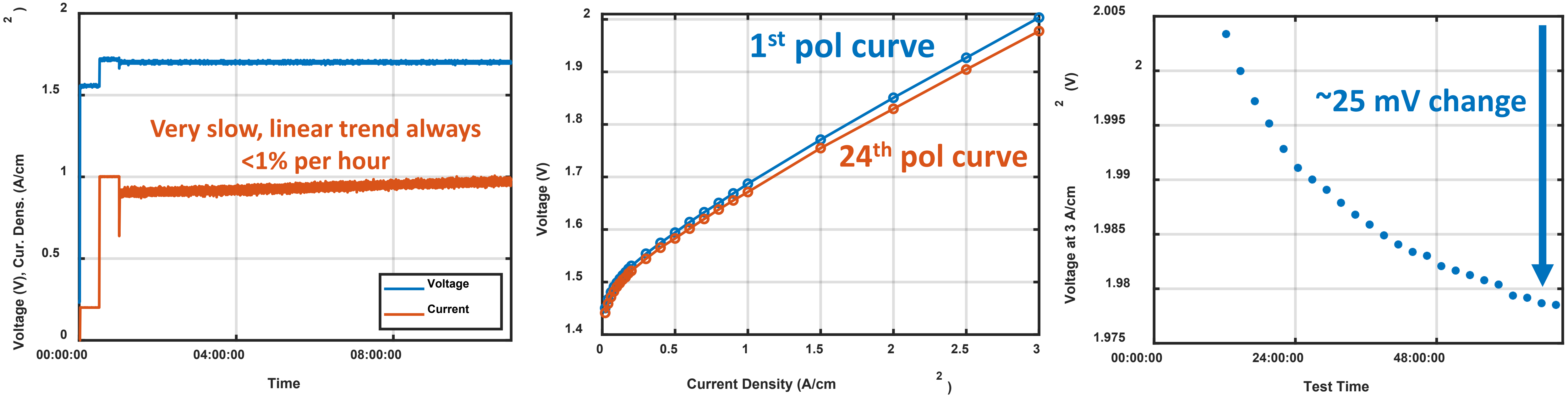
- Performance changes during a long hold are sometimes used to gauge stability and in criteria for ending conditioning, e.g. current varies <1% per hour. [1]
- Performance trends during initial holds vary significantly between component sets, and **do not effectively predict performance or stability later.**

Example 1: Materials from prior NREL durability work.



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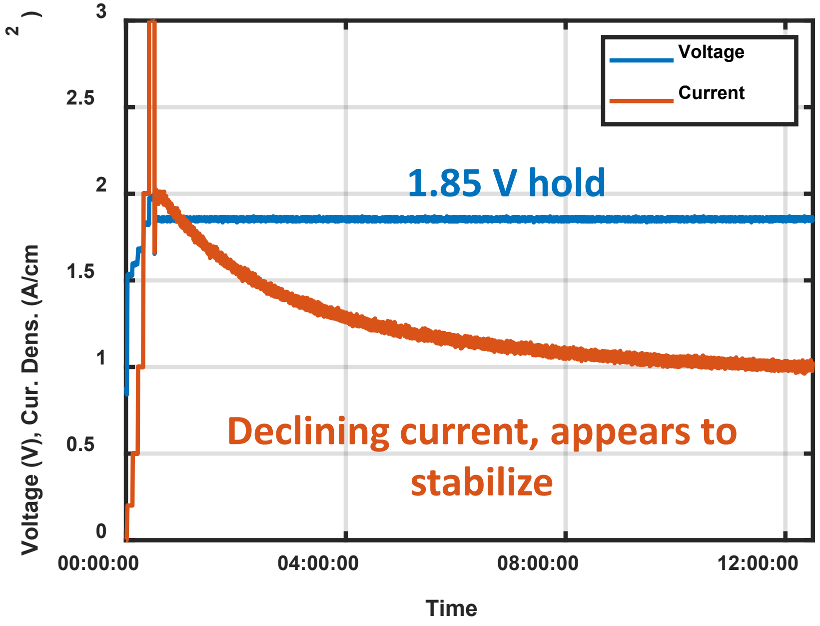
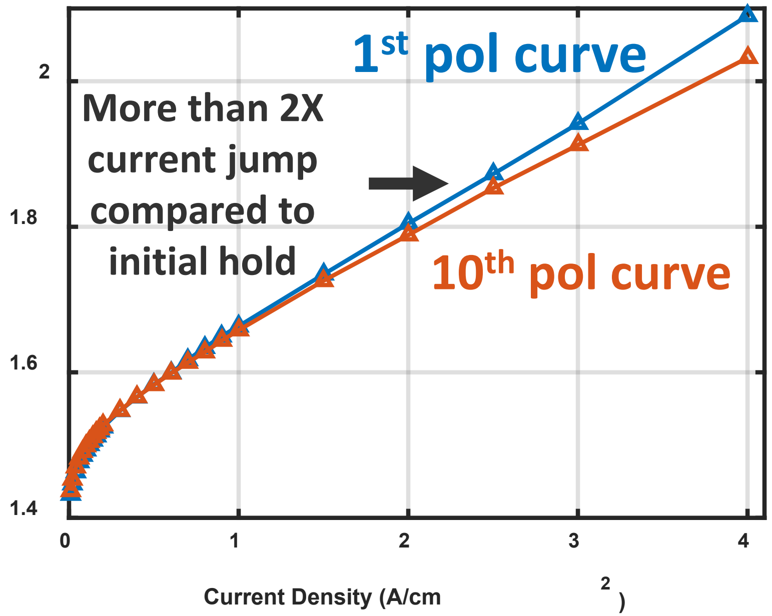
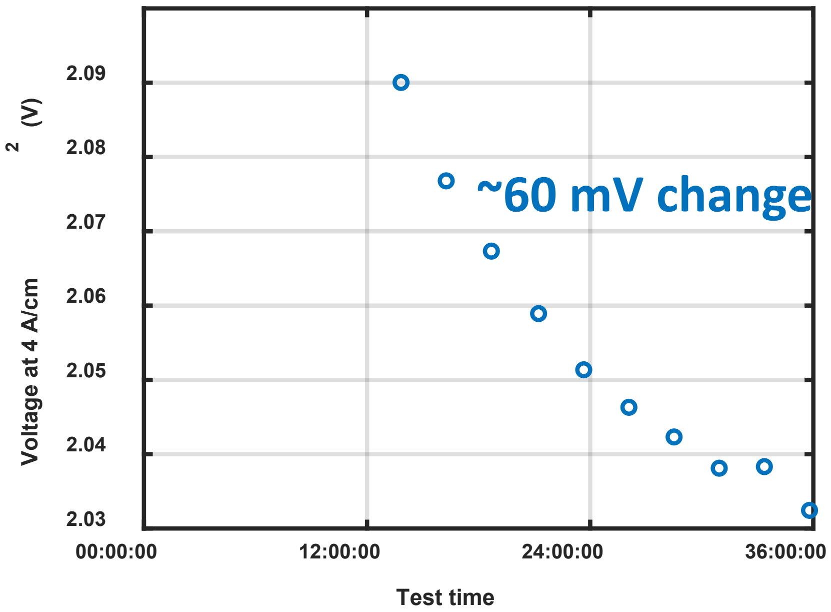
Example 2: H2NEW "Future Generation MEA" (FuGeMEA) baseline materials



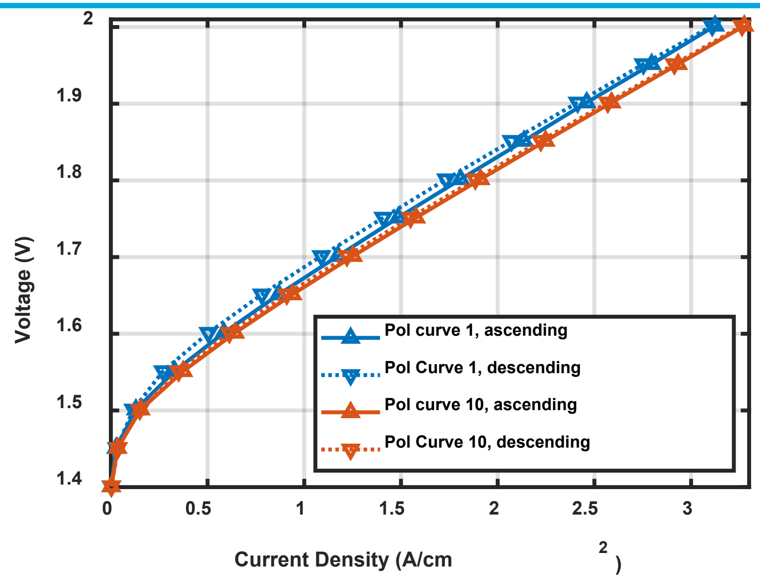




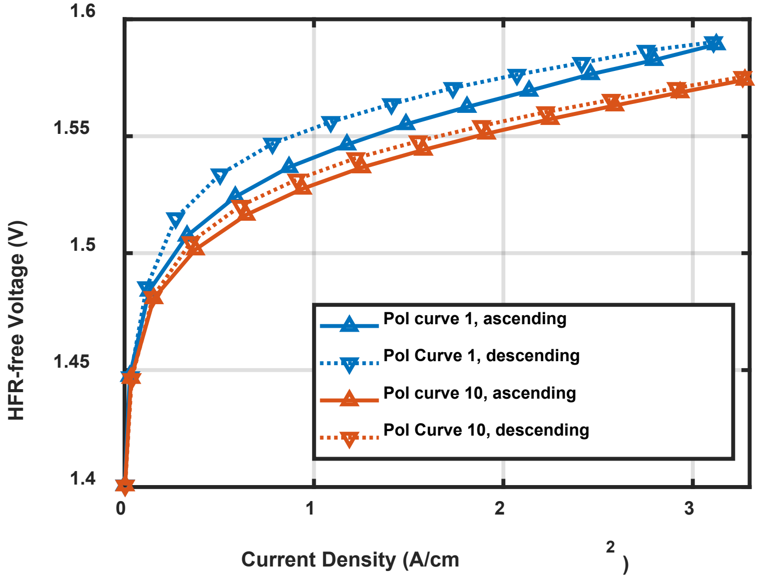
1. G. Bender et al., *International Journal of Hydrogen Energy*, **44**, 9174–9187 (2019).

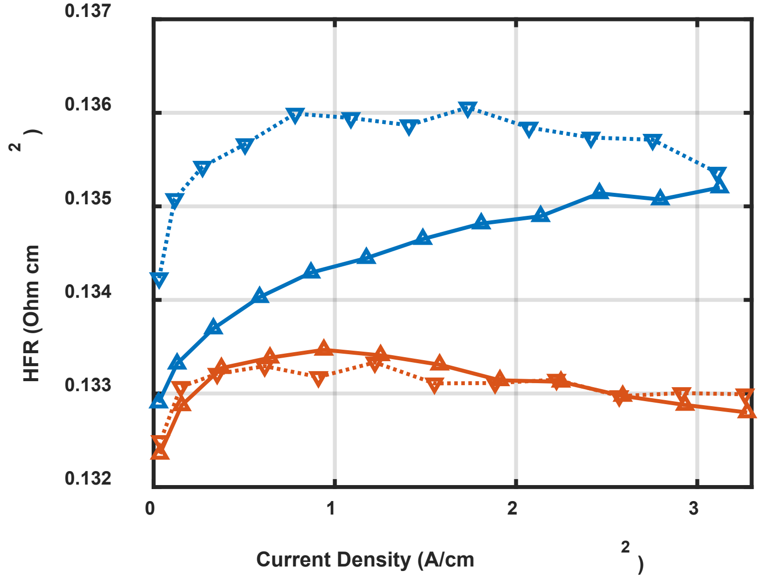
Anode: 0.16 mg_{Ir}/cm^2 IrO_2. I:Ir = 0.27. 250 um Bekaert Pt/Ti fiber PTL
Cathode: 0.08 mg_{Pt}/cm^2 46wt% Pt/HSC. I:C = 0.45. MGL 280 carbon paper PTL
Membrane: N115, no pretreatment

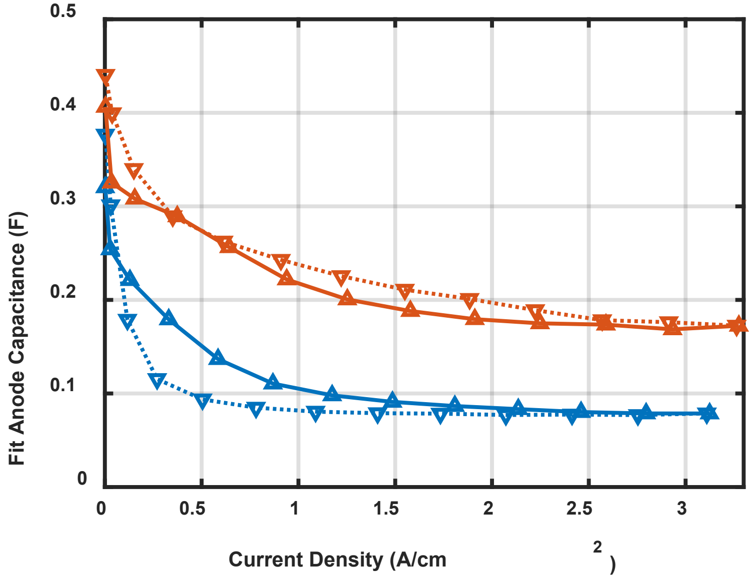
Mechanisms of slow conditioning processes



- Slow conditioning processes appear to involve change to catalyst layer structure and contact to PTLs
  - OER kinetics improve
  - HFR improves
  - Electrode capacitance increases
  - Polarization curve hysteresis decreases
  - Can be influenced by cell compression







Membrane and CCM pre-treatments

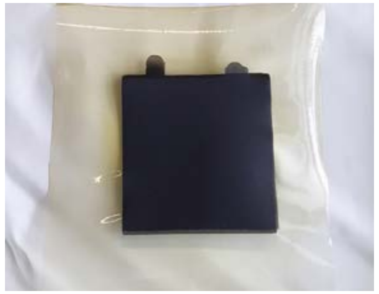
Membranes are sometimes pretreated to remove contaminants. Does this impact conditioning processes?

NREL membrane pre-treatment:

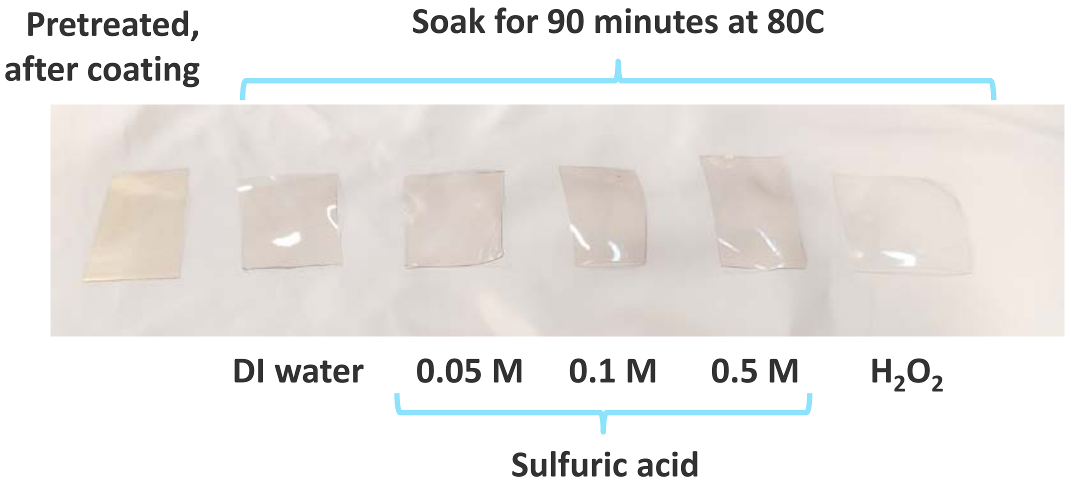
1. 1 hr soak in 3% H_2O_2, 80˚C
2. 1 hr soak in DI water, 80˚C
3. 1 hr soak in 0.5 M H_2SO_4, 80˚C
4. 1 hr soak in DI water, 80˚C
5. Flat dry on vacuum table, 2 hrs at 60˚C

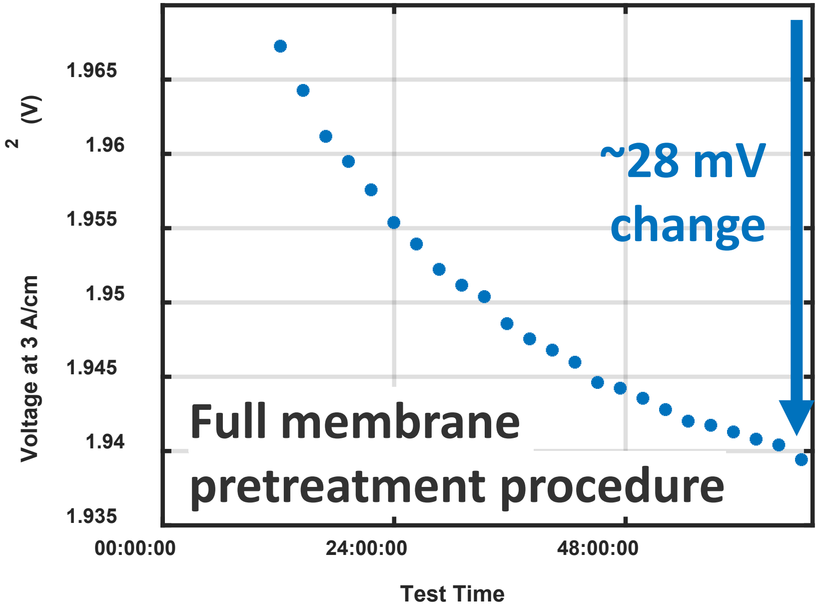
No pretreatments for membranes or CCMs were found to impact the slow conditioning process.

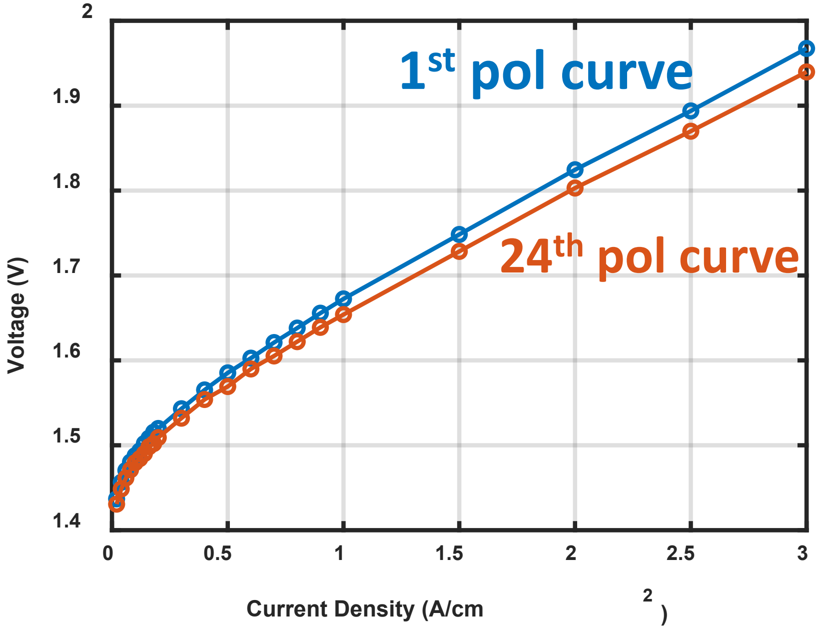
Soaking and flat drying can stretch and thin the membrane up to 30%, lowering HFR and loading. This makes other impacts ambiguous.



Discoloration in spray-coated CCM on N117.







Conclusions and Recommendations

- Incomplete conditioning of PEM electrolysis cells creates challenges for reproducibility and interpreting performance and durability.
- Slow conditioning processes appear related to restructuring of anode catalyst layer and interface to PTL. Varies with catalyst layer loading and coating method, PTL, and cell compression. Chemical pretreatments do not impact conditioning time but can thin the membrane.
- There may be no one conditioning protocol that is appropriate for all circumstances, making it important to measure and report cell stability.
- Recommended metric: Repeat polarization curve measurement at least 3 times, report change and trend.
- When complete conditioning is needed, repeat conditioning until the polarization curve stabilizes.

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Thank you!

We welcome questions.

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