Advanced Water-Splitting Technology Pathways Benchmarking & Protocols Workshop

Breakout Session Summaries
Low Temperature Electrolysis (LTE)

June 11 – 12, 2024

This presentation does not contain any proprietary, confidential, or otherwise restricted information

Pacific Northwest NATIONAL LABORATORY

nel

ASU Arizona State University

Caltech

LTE Breakout Sessions

Session ID	Topic	Lead	Note Taker
L1-A	PEM: Priority research opportunities: Components	Serafina Fortiner (Nel Hydrogen)	Nick Oliveira (Nel Hydrogen)
L1-B	Alkaline: Priority research opportunities for separators/membranes	Anthony Ekennia (U of Oregon)	Manasa Rajeev (U of Oregon)
L2-A	PEM: Durability and AST test protocols	Rangachary Mukundan (Mukund) (LBNL)	Shaun Alia (NREL)
L2-B	Alkaline: Priority research opportunities for catalyst/electrode materials	Melissa Kreider (NREL)	Emily Volk (NREL)
L3-A	PEM & Alkaline: Priority Research Opportunities - Stack & Cell Testing Gates for Qualification	Balsu Lakshmanan (Versogen)	Nadia E. Tolouei (UC Irvine)
L3-B	Alkaline: Standardization needs and test protocols to be written	Grace Lindquist (HGen)	Manasa Rajeev (Univ of Oregon)
L5-A	AEM & PEM: Validation of protocols	Su Min Ahn (LANL)	Minkyoung Kwak (U of Oregon)
L5-B	AEM & PEM: System materials compatibility	Nick Oliveira (Nel Hydrogen)	Serafina Fortiner (Nel Hydrogen)

Session Summary

Session ID: L1-A

Title: PEM Priority Research Opportunities: Components

Summary of Discussion

- **Membrane**: A standardized membrane that is available to all is needed. This needs to go through round robin testing. We need to be able to characterize this, so not protected by IP or NDAs.
- **PTLs**: No reason to standardize how coatings are made, but characterization should be standardized. It should lend itself to fast tests and high throughput. Things to characterize: Continuity of the coating, thickness, quality, contaminations, and how these impact performance. Should also have a standardized contact resistance protocol.
- **Catalyst/Electrode**: Need standard ways to characterize electrodes after fabrication, to understand how fabrication methods impact performance. Standardization is easy for some easily accessible techniques (spraying) but others may be more complicated. What is the optimal structure (catalyst, ionomer pore structure) relationship?

Consensus & Dissenting Opinions

- Strong consensus that a list of "worst practices" should be put together to point field in the correct direction
- In this vein, NREL was asked if they could provide insight into how to standardize electrode fabrication processes. This may be hard, because NREL changes ink rheology depending on purpose.
- Some debate on whether a consensus on HOW PTL coatings are made: Most people will just be buying them. Standardizing how to characterize them would help unify the field
- A standardized membrane is needed, thin, reinforced, grc.

Key Takeaways

- **Membrane**: A standardized that can be characterized is needed
- SOP's for characterizing these are needed (compressive creep for example)
- **PTL**: Standardized PTL coating characterization methods for contaminations, coating thicknesses, continuity and contact resistance measurements
- **Electrode:** Fabrication is greatly varying amongst groups and will be hard to standardize, especially with ongoing efforts to optimize electrodes. A list of "worst practices" gives researchers key parameters to watch out for. I.e. if x,y,z is observed, the electrode will perform poorly
- NREL has a great deal of data on electrode fabrication methods and performance to contribute.

Action Items

- **Membrane**: Compressive Creep: Adam Weber/Ahmet Kusoglu from LBNL. Trent Simonetti from Hyaxiom can work together to develop a protocol for how to measure under different conditions
- **Membrane**: Mukund (H2New) Identify standard membrane(s) – thinner, reinforced, GRC
- **Membrane**: Su Min Ahn LANL: Draft list of necessary protocols for ionomers
- **PTL**: Haoran Yu: Develop protocol for characterizing PTL coating continuity and thickness
- **PTL**: Rajib Das from ACS Industries Corroborate contact resistance measurements, esp for PTL
- **Electrode**: Shaun Alia NREL: Delegate within H2New: Create best/worst practices list and/or troubleshooting guide for electrode fabrication

Session Attendee List

Session ID: **L1-A**
Title: PEM Priority Research Opportunities: Components

Name	Affiliation
Bilal Iskandarani	UC Irvine
Su Min Ahn	LANL
Nadia E. Tolouei	UC Irvine
Haoran Yu	ORNL
Rangachary Mukundan	LBNL
Shaun Alia	NREL
Andrew Boudreau	NREL
Ai-Lin Chan	NREL
Adam Weber	LBNL
Shuang Ma Andersen	SDU
Mason Jang	Caltech
Earl H Wagener	Tetramer
Chris Topping	Tetramer
Trent Simonetti	HyAxiom

Session Attendee List 2

Name	Affiliation
Max Thouin	AvCarb
Rajib Das	ACS Industries
Flavio da Cruz	SoCalGas
George Roberts	Nel Hydrogen
Karl Gross	H2 Tech Consulting
Nick Oliveira	Nel Hydrogen
Serafina Fortiner	Nel Hydrogen

Session Action Item Assignments

Session ID: L1-A
Title: PEM Priority Research Opportunities: Components

Name	Affiliation	Action Item	Target Due Date
Trent Simonetti, Ahmet Kusoglu	HyAxiom, LBL	Create protocol for membrane compressive creep	
H2New (Mukund)	LBL	Identify standard membrane(s) – thinner, reinforced, GRC	
Haoran Yu	ORNL	Develop protocol for characterizing PTL coating continuity and thickness	
Rajib Das	ACS Industries	Corroborate contact resistance measurements, esp for PTL	
Su Min Ahn	LANL	Draft list of necessary protocols for ionomers	
Shaun Alia	NREL	Delegate within H2New: Create best/worst practices list and/or troubleshooting guide for electrode fabrication	

Session Summary

Session ID: L1-B

Title: Alkaline: Priority research opportunities for separators/membranes

Summary of discussion.

1. Developing separators and electrode/catalyst engineering with robust properties are essential to achieve the 1.7V @1A/cm^{-2} target in LWE.
2. Encourage field data sharing among the community.
3. Reducing the thickness of a separator alone isn't sufficient; it must also be more conductive to decrease the mass transfer resistance of OH through it while maintaining minimal gas crossover.

Consensus

The need for thinner / more conductive separators for AWE.

Versogen volunteered their membrane as an AEM baseline.

Dissenting opinions

1. There is disagreement on whether ADT (Accelerated Durability Testing) is an appropriate representation for long-term durability testing.

2. There is disagreement on selecting a baseline separator. Thickness may not be as important as structure.

Key Takeaways

1. Zirconium-based separators have mechanical stability and porosity loss issues after long-term testing.
2. It is crucial to understand the reversible degradation and the initial high degradation in AEMWE.
3. Unlike Nafion, in AEMWE, the conductivity, water uptake, and faradaic efficiency of AEMs vary as a function of thickness.

Action Items

1. Understand the failure mechanisms for separator degradation and develop standard test protocols and characterization techniques addressing the reported failure mechanisms. (Versogen and Nel Hydrogen volunteered to write the validation protocol for membrane durability testing)
2. Engineer more robust polymer designs that are KOH stable and can be made into more conductive, thinner separators with minimal gas crossover.
3. Develop a reproducible validation protocol and criteria for selecting baseline materials for AEMWE.

Session Attendee List

Session ID: L1-B

Title: Alkaline: Priority research opportunities for separators/membranes

Name	Affiliation
Grace Linquist	Hgen
Daniel Leonard	LANL
Isabela Rios Amador	SLAC
Michaela Burke Stevens	SLAC
Ethan Simonoff	SoCal Gas
Minkyoung Kwak	Univ. of Oregon
Sarah Park	LANL
Jong-Ho Chot	LANL
Shujin Hou	UCB
Melissa Kreider	NREL
Emily Volk	Mines/NREL
Duha Syar	UCB/LBNL
Balsu Lakshmanan	Versogen
Kathy Ayers	Nel Hydrogen
Ahmet Kusoglu	LBNL
Jeff Martin	Shell
Vinh Nguyen	TDA research Inc.
Anthony Ekennia	Univ. of Oregon
Manasa Rajeev	Univ. of Oregon

Session Summary

Session ID: L2-A
Title: PEM: Durability and AST Test Protocols

Summary

- Develop better understanding of failure mechanisms. Current understanding is not adequate to develop ASTs
- Focus on catalysts and membranes taking into account the effect of PTL
- Need better operando techniques to track degradation. E.g. quantification of Anode catalyst surface area, oxidation state etc.

Consenting dissenting opinions

- Focus on catalyst layers and membranes
- Consider impact of PTL. Example: Larger pores in PTL can lead to greater mechanical stress of the membrane. Use thin membranes to accelerate degradation rates. Avoid artificial edge effects in small cells that may not be relevant to larger area stacks.
- Need discussion on GDL materials properties. Especially change under compression, explore efforts to control materials properties more precisely.

Key takeaways

- Standardized protocols with standardized materials can really advance the field by generating durability data ripe for modern data analytic tools.
- H2NEW has AST working group, coordination with IEA.
- EU put out a harmonization document (stack focus).
- Harmonization protocols are more geared to a qualification procedure, not a fundamental understanding of degradation behind them.
- Drive at the true degradation mechanism.

Action items

- Develop model systems that can be used for degradation studies. Maybe adopt H2NEW baseline materials.
- Understand current degradation to guide AST development.

Session Attendee List

Session ID: L2-A
Title: PEM: Durability and AST Test Protocols

Name	Affiliation
Rangachary Mukundan	LBNL
Shaun Alia	NREL
Haoran Yu	ORNL
Ai-Lin Chan	NREL
Andrew Boudreau	NREL
Serafina Fortiner	Nel Hydrogen
Bilal Iskandarani	UC Irvine
Nadia E Tolouei	UC Irvine
Su Min Ahn	LANL
Micha Be-Naim	Clean Energy Ventures
Tom Jaramillo	Stanford
Adam Weber	LBNL
Chris Topping	Tetramer
Earl Wagener	Tetramer

Session Attendee List 2

Session ID: L2-A
Title: PEM: Durability and AST Test Protocols

Name	Affiliation
Isabela Rios Amador	Stanford
Trent Simonetti	HyAxiom
Max Thouin	AvCarb
Kathy Ayers	Nel Hydrogen
Balsu Lakshmanan	Versogen

Session Summary

Session ID: L2-B

Title: Alkaline: Priority research opportunities for catalyst/electrode materials

Summary

- We discussed catalyst types and standards, transport layers (metals, morphologies), catalyst/ionomer integration, electrode durability, and characterization techniques to understand degradation

Consenting dissenting opinions

- Ionomers in AEM catalyst layers – yes or no? If without, will alkaline and AEM unify?
- Standard durability protocols – what KOH concentration, catalyst loading, temperature, current, etc?

Key takeaways

- Ni is a good standard material for PTLs
- Standard tests are needed to understand the consequences of intermittent operation
- Well-defined durability target is needed

Action items

- Develop ASTs for intermittent operation
- Standardize durability protocols
- Standardize post-test characterization of electrodes

Session Attendee List

Session ID: L2-B

Title: Alkaline: Priority research opportunities for catalyst/electrode materials

Name	Affiliation
Emily Volk	Mines/NREL
Melissa Kreider	NREL
Grace Lindquist	Hgen
Nick Oliveira	Nel Hydrogen
Daniel Leonard	LANL
Michaela Burke Stevens	SLAC
Ethan Simonoff	SoCalGas
Sarah Park	LANL
Shuang Ma Andersen	SDU
Minkyoung Kwak	University of Oregon
Sujin Hou	UC Berkeley
Manasa Rajeev	U Oregon
Anthony Ekennia	U Oregon
Rajib Das	ACS Industries
Ahmet Kosoglu	LBNL
Jon-Ho Choi	LANL
George Roberts	Nel Hydrogen

Session Action Item Assignments

Name	Affiliation	Action Item	Target Due Date
All	N/A	Develop ASTs for intermittent operation	2025
All	N/A	Standardize post-test characterization of electrodes	2025
All	N/A	Standardize durability protocols	2025

Session Summary

Session ID: L3-A

Title: Stack & Cell Testing Gates for Qualification

Summary of discussion

- Degradation rate has to be specified on which operation condition; depends on several reasons including BOL cell voltage which has to be the voltage after it hits the steady state not the first point.
- Should have safety standard for test stands.
- How critical is faradaic efficiency ? cell leak test to confirm no big leaks.
- How to measure crossover? do standard diffusion test. use the flowmeter for cathode exhaust and compare that to current density.
- Pick your EOL voltage at customer requirement (balance EOL voltage based on efficiency and power supply/rectifier limit.)

Consensus

- Everyone agreed on these.

Key Takeaways

- Crossover measurement is possible through GC intermittent gas sampling.
- Turn-down ratio can be relaxed based on measured X-over BoL & EoL Voltages for MEA qualification
- There is TRL4.5 where a company has something they want to test but they don't have enough infrastructure to test it (need open-source controls for low-cost power supply)

Action Items

- None assigned

Session Attendee List

Session ID: L3-A

Title: Stack & Cell Testing Gates for Qualification

Name	Affiliation
Shuan Alia	NREL
Haoran Yu	ORNL
Andrew Boudreau	NREL
Ai-Lin Chan	NREL
Serafina Fortiner	Nel Hydrogen
Bilal Iskandarani	UC Irvine
Adam Weber	LBNL
Rangachary Mukundan	LBNL
Shuang Ma Andersen	SDU
Trent Simonetti	HyAxiom
Max Thouin	AvCarb
Rajib Das	ACS industries
George Roberts	Nel Hydrogen
Kathy Ayers	NEL Hydrogen
Nadia E. Tolouei	UC Irvine

Session Summary

Session ID: L3-B
Title: Alkaline: Standardization needs and test protocols to be written

Summary of discussion

1. Discussed standard components (anode/cathode catalyst, electrode/PTL, and membrane/separator) for AEM and liquid alkaline
2. Discussed needs for test protocols at cell/stack level for AEM and liquid alkaline
3. Discussed current status and needs for individual component test protocols for AEM and liquid alkaline

Consensus

- AEM has progressed enough for standard protocol
- Protocol should apply to both KOH and pure water
- Standard materials: Bekaert Ni fiber anode/Pt/C Toray cathode; Versogen AEM

Dissenting opinions:

- Should the anode baseline material be IrOx or NiFe?
- Should baseline include an uncoated anode reference?
- Should other AEMs beyond the Versogen material (other backbone chemistries/counter ions) be included?

Key Takeaways

1. Liquid alkaline standard components are Ni foam anode/cathode and Zirfon 500. AEM standard components are beginning to center to 1 materials set but some dissenting opinions remain (see consensus/dissenting opinions)
2. Action items were assigned to write cell-level AEM test protocol
3. For individual component protocols (e.g. conductivity, gas permeability, etc.) most translate well to liquid alkaline and just need to add increased concentration KOH - no action items were assigned

Action Items

1. Ahmet Kusoglu (LBNL) has agreed to extend the mechanical studies (water uptake and swelling) for PEM to AEM
2. NREL, UO, Versogen, and SLAC have agreed to draft standardized AEM single cell test protocol
3. It was noted there are currently separate efforts ongoing at NREL to adapt the current PEM electronic conductivity catalyst protocol for AEM catalyst powders - no additional action items assigned

Session Attendee List

Session ID: L3-B
Title: Alkaline: Standardization needs and test protocols to be written

Name	Affiliation
Vinh Nguyen	TDA Research Inc.
Jeff Martin	Shell
Daniel Leonard	LANL
Nick Oliveria	Nel Hydrogen
Isabela Rios Amadar	SLAC
Michaela Bruke Stevens	SLAC
Ethan Simonoff	SoCal Gas
Emily Volk	Mines/NREL
Melissa Kreider	NREL
Sarah Park	LANL
Sumin Ahn	LANL
Minkyoung Kwak	Univ. of Oregon
Shujin Hou	UCB
Duha Syav	LBNL
Anthony Ekennia	Univ. of Oregon
Ahmet Kusoglu	LBNL
Jong Ho Chot	LANL
Grace Lindquist	Hgen
Manasa Rajeev	Univ. of Oregon

Session Summary

Session ID: L5-A

Title: AEM & PEM: Validation of protocols

Summary of discussion

- Validation of LTE-P-7 and LTE-P-22 was reviewed
- When validating protocols, duration and cost wouldn't be compromised with accuracy and reproducibility of measurements
- Coupling related protocols and validating them together will be more efficient
- Validation of protocols from multiple groups can be done when writing protocols
- Getting people being aware of protocols and adapting those in research will be important

Consensus and/or dissenting opinions

- Inclusion of alternative methods and equipment is encouraged
- Accuracy and reproducibility of protocols are important general consensus on protocols of LTE-P-7 and LTE-P-22
- People agreed that protocols should be more accessible and adopted
- For time dependent characterization, people have different opinions whether characterization should be done after sample acquisition or it's okay to collect all samples and characterize later
- For LTE-P-22 (Alkaline Stability): Potassium Hydroxide used for the alkaline condition should be pure for the validation. (99.99%)

Key Take-Aways

- Figuring out which protocols would be prioritized to validate will depend on current lab capabilities and how frequent protocols are used in the fields
- Involvement/volunteer from different organizations when writing protocols will be important. Validation can be done during initial protocol writing to minimize time and effort

Action Items

- LTE-P-7 (AEM IEC): LANL testing protocol in different temperatures (20 °C vs 30 °C)
- LTE-P-7 (AEM IEC): UO sending electrode to LANL to test electrode compatibility
- LTE-P-22 (AEM Alkaline Stability): UO analyzing collected samples without delay; NREL analyzing collected samples after preservation
- LTE-P-6 (AEM Conductivity): LANL/UO/NREL work on validating this protocol while working on LTE-P-22 testing

Session Attendee List

Session ID: L5-A

Title: AEM & PEM: Validation of protocols

Name	Affiliation
Sarah Park	LANL
Shuang Ma Andersen	SDU
Isabela Rios Amador	Stanford/SLAC
Rajib Dan	ACS Industries
Kathy Ayers	Nel Hydrogen
George Roberts	Nel Hydrogen
Anthony Ekennia	U of Oregon
Shujin Hou	UC Berkeley
Manasa Rajeev	U of Oregon
Andrew Boudreau	NREL
Shaun Alia	NREL
Ai-Lin Chan	NREL
Sian Sosa	So Cal Gas

Session Action Item Assignments

Session ID: L5-A
Title: AEM & PEM: Validation of protocols

Name	Affiliation	Action Item	Target Due Date
Su Min Ahn	LANL	LTE-P-7 (AEM IEC): LANL testing protocol in different temperatures (20 °C vs 30 °C)	7/31/2024
Anthony Ekennia	U of Oregon	LTE-P-7 (AEM IEC): UO sending electrode to LANL to test electrode compatibility	7/31/2024
Anthony Ekennia	U of Oregon	LTE-P-22 (AEM Alkaline Stability): UO analyzing collected samples without delay	12/31/2024
Andrew Boudreau	NREL	LTE-P-22 (AEM Alkaline Stability): NREL analyzing collected samples after preservation	12/31/2024
Su Min Ahn	LANL	LTE-P-6 (AEM Conductivity): LANL/UO/NREL work on validating this protocol while working on LTE-P-22 testing	12/31/2024
Anthony Ekennia	U of Oregon		
Andrew Boudreau	NREL		

Session Summary

Session ID: L5-B
Title: AEM & PEM: System Materials Compatibility

Summary of discussion

- Behavior of contaminants in AEM vs PEM systems
- H2 processing (separator, dryer) in AEM vs PEM vs AWE
- Scale-up considerations – footprint vs material cost
- Gasketing and NRHW different in KOH

Consensus and/or dissenting opinions

- AWE may be a better starting point for AEM system design than PEM (at least for materials)
- Near term AEM systems need to be designed for KOH operation, DI water still far in the future
- Dissent: Feed configuration still up for debate (pressurization, safety factors)

Key Take-Aways

- Different considerations for contaminants
  - KOH dissolves polishers – need different approach to keep contaminants down (i.e. dilution)
  - Electroosmotic drag is opposite in AEM vs PEM
  - Organic contaminants can cause frothing in KOH
- Stainless steel can likely be used in AEM systems, but will need treatment similar to AWE
- AEM is a “middle ground” between PEM and AWE – will need to borrow system design elements from both technologies

Action Items

- Improve efficiency of AEM membranes
- More detailed TEA considering different materials and degradation

Session Attendee List

Session ID: L5-B
Title: AEM & PEM: System Materials Compatibility

Name	Affiliation
Ethan Simonoff	SoCalGas
Trent Simonetti	HyAxiom
Haoran Yu	ORNL
Earl Wagener	Tetramer
Chris Topping	Tetramer
Grace Lindquist	Hgen
Mason Jang	Caltech
Rangachary Mukundan	LBNL
Duha Syar	UC Berkeley/LBNL
Daniel Leonard	LANL
Balsu Lakshmanan	Versogen
Michaela Burke Stevens	SLAC
Melissa Kreider	NREL
Jong-Ho Choi	LANL
Emily Volk	Mines/NREL
Serafina Fortiner (scribe)	Nel Hydrogen