HydroGEN
Advanced Water Splitting Materials

Advanced Water-Splitting Technology Pathways Benchmarking & Protocols Workshop

Breakout Session Summaries
Low Temperature Electrolysis (LTE)

October 29 - 30, 2019

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Executive Summary

The LTE breakout sessions focused on soliciting feedback on each of the released protocols and prioritization of the development of new protocols. Sessions were also held to review the technology roadmap with the goal of gaining agreement. General themes include:

- Technology Roadmaps
  - Need a consistent AEM membrane
  - HER catalysts need attention and should likely be more of focus (low and non-PGM)
  - Efforts should be increased on the development of a PTL
  - Corrosion resistant coatings should be developed to allow the use of lower cost materials in bipolar plates and PTL

- Current Test Protocols
  - A concerted, funded effort for validation is required and encouraged
  - Open access to test protocols will be important on an easily accessible online platform
  - Need a marketing strategy to communicate new protocols to broad water splitting community
  - Leverage international efforts to reduce duplication and conflicting protocols
  - Include technical rationale for prescribed test conditions/parameters and guidance of things to watch out for to prevent errors in testing

- New Test Protocols
  - Accelerated Testing- Need for 'EC-PAD' equivalent of FC-PAD
  - Need standard postmortem analysis

LTE Breakout Sessions

Breakout Session #	Session ID	Technology	Topic	Lead
1	L1-A	LTE	LTE Technology Roadmap Review & Discussion- Membrane, Catalyst	Chris Capuano (Nel Hydrogen)
1	L1-B	LTE	LTE Technology Roadmap Review & Discussion- Components, Stack	George Roberts (Nel Hydrogen)
2	L2-A	LTE	PEM Membrane: IEC, Thermal Stability, Water Uptake	Chulsung Bae (RPI)
2	L2-B	LTE	AEM Membrane: Conductivity, Gas Permeability, Chemical Stability	Yushan Yan (U of Del)
4	L4-A	LTE	PGM Catalyst: RDE	Marcelo Carmo (Julich)
4	L4-B	LTE	Non-PGM Catalyst: Electroconductivity	Guido Bender (NREL)
5	L5-A	LTE	Porous Transport Layer (PTL): Characterization Protocols	Nem Danilovic (LBNL)
5	L5-B	LTE	Gas Diffusion Layer (GDL): Characterization Protocols	Cortney Mittelsteadt (Giner)
6	L6-A	LTE	Wrap-up/Action Item Assignment: LTE Membrane, Catalysts	James Vickers (DOE)
6	L6-B	LTE	Wrap-up/Action Item Assignment: LTE Components, Stacks Technology	Ahmet Kusoglu (LBNL)

Session Summary - PEM

Session ID: L1-A
Title: LTE Technology Roadmap Review & Discussion- Membrane and Catalyst

Summary of discussion

- Roadmap appears to make sense based on current state of technology
- Status of membrane and PGM catalysts in fairly good shape

Consensus and/or dissenting opinions

- Consensus agrees non-PGM materials should be pursued
- Possibly pull in on timeline since it will require more development time

Key Take-Aways

- Industry is actively pursuing development of electrolyzer specific membranes
- Still PFSA based, should hydrocarbon membrane be part of the plan

Action Items

1. Update roadmap with more overlap of phased in changes instead of linear path

Session Summary - AEM

Summary of discussion

- Progress has been slowed due to lack of consistent (commercial) source of membrane/ionomer
- Require electrolyzer specific materials. Mostly using fuel cell membranes due to availability
- Roadmap direction makes sense

Consensus and/or dissenting opinions

- Agreed increase mechanical stability improvements needed along with water uptake management
- Chemical stability needed and discussed exposure to superoxides as a requirement

Key Take-Aways

- OER catalysts, both low and non-PGM materials are in a good position
- HER really needs attention and should likely be more of focus (low and non-PGM)

Action Items

1. Update the roadmap for more overlap of activities
2. Review methods (Vijay Ramani) for membrane degradation in the presence of oxygen as a way to further understand chemical degradation

Session Attendee List

Session ID: L1-A

Title: LTE Technology Roadmap Review & Discussion- Membrane and Catalyst

Name	Affiliation
Christopher Capuano	Nel Hydrogen
Yushan Yan	University of Delaware
Hector Colon-Mercado	SRNL
Ehren Baca	SNL
Huyen Dinh	NREL
Cy Fujimoto	SNL
Hoon Chung	LANL
Sangwoo Lee	RPI
Sarah Eun Joo Park	LANL
Chulsung Bae	RPI
Josh Spurgeon	University of Louisville

Session Summary

Session ID: L1-B

Title: LTE Technology Roadmap Review & Discussion- Components, Stack

- Summary of discussion
  - MEA – fabrication methods such as roll to roll coating or sheet by sheet spray coating and issues around scale up
  - Bipolar Plate – Coating types for corrosion resistance and possible unique approaches to lower expensive metal content
  - PTL – What characteristics should a PTL have, lack of commercially available option
  - GDL – hydrophilic vs hydrophobic GDL and carbon free MPL lack of availability
  - Accelerated Testing- Need for 'EC-PAD' equivalent of FC-PAD, need for a funded focused effort

- Consensus and/or dissenting opinions
  - Each component needs a wholistic characterization i.e. can't sacrifice membrane resistivity to improve gas purity
  - Moving from lab scale to pilot scale can be an awkward transition, a need for accessible pilot scale facility
  - Corrosion of materials particularly in PEM requires an ideal coating solution

- Key Take-Aways
  - The PTL is currently underdeveloped and without any commercially available options
  - A hydrophilic and carbon free anode options for an MPL are lacking
  - Accelerated testing difficult to advance without a focused and funded research effort

- Action Items
  - Agree on a cell design for AEM and PEM systems and what it's capabilities should include
    - i.e. difficult to include all metrology such as gas purity into a single affordable test cell design
  - Focused effort on PTL research required
  - What approaches/facilities/commercial partners are available for scaling up MEA fabrication processes
  - Separate cost saving measures into short term (i.e. faster coating method) vs long term (i.e. new types of materials)

Session Attendee List

Session ID: L1-B

Title: LTE Technology Roadmap Review & Discussion- Components, Stack

Name	Affiliation
George Roberts	Nel Hydrogen
Katherine Ayers	Nel Hydrogen
Guido Bender	National Renewable Energy Laboratory
Shannon Boettcher	University of Oregon
Marcelo Carmo	Forschungszentrum Julich
Nemanja Danilovic	Lawrence Berkeley National Laboratory
Adrian Gestos	AquaHydrex
Ahmet Kusoglu	Lawrence Berkeley National Laboratory
Alexey Serov	Pajarito Powder. LLC

Session Summary: IEC

Session ID: L2-A

Title: PEM: IEC, Thermal, water uptake

Summary of discussion

- For PFSA type-membranes, industry-provided IEC value is available, therefore the protocol are mostly designed for mostly hydrocarbon-based membranes
- Error bar of IEC measurements should be reported
- Samples size: 100–200 mg; NaOH 0.01 M
- Titration either by Mettler Toledo T90 and internal indicator (phenolphthalein)

Consensus & dissenting opinions

- How to measure accurate weight of dry membrane? Should we include significant values (e.g. up to 0.1 mmg)?
- "Fresh" definition of titrating 0.01 M NaOH solution (prepared in 1 day?)
- Adding a picture of the end point of phenolphthalein indicator (pale pink color)
- How to measure weight of dry membrane after drying under ambient condition (e.g., 50% RH)

Key Take-Aways

- Avoid applying high temperature when drying; because drying membrane with vacuum and high temperature for a long time may affect the membrane solubility and change polymer structure
- Drying membrane with vacuum is sufficient (no heating); vacuum/pressure level needs to be specified, e.g., 1 mmHg)
- Blank titration using a known amount/ concentration of H_2SO_4

Action Items

- Off-line review of revised protocols by multiple organizations should be done to get more feedback/validation from people who actually doing measurement using the similar protocol
- The same IEC measurement by multiple labs using the same protocol might help to validate the reproducible protocol

Session Summary: Thermal & Water Uptake

Session ID: L2-A

Title: PEM: IEC, Thermal, water uptake

Summary of discussion

- **Thermal**: a typical straight polymer characterization method, not much for variation in protocol
- **Water uptake**: Pretreatment by immersing membrane in hot water is suggested but too high temperature might affect/damage the membrane property for certain types of hydrocarbon PEMs

Consensus & dissenting opinions

- **Thermal:** suggest flow rate of N2 and air (50 mL/min)?
- **Water uptake**: Membrane treatment in hot water – temperature 30, 60, 80 C? how long?
- **Water uptake**: how to measure weight of wet membrane accurately?
- **Water uptake**: provide a picture of watch glass holding membrane's weight measurement

Key Take-Aways

- **Water uptake**: measurement should be repeated with multiple (2–3) times for each membrane (report an average value)
- **Water uptake**: measurement should be repeated at different temperatures

Action Items

Session Attendee List

Session ID: L2-A

Title: PEM: IEC, Thermal, water uptake

Name	Affiliation
Chulsung Bae	RPI
Sarah Eun Joo Park	LANL (note taker)
Kathy Ayers	Nel Hydrogen
George Roberts	Nel Hydrogen
Nem Danilovic	LBNL
Ahmet Kusoglu	LBNL
Marcelo Carmo	Julich
Guido Bender	NREL
Hector Colon-Mercado	SRNL

Session Action Item Assignments

Session ID: L2-A

Title: PEM: IEC, Thermal, water uptake

Name	Affiliation	Action Item	Target Due Date
Chulsung Bae	RPI	Off-line review of revised protocols by multiple organizations	03/31/2020
Chulsung Bae	RPI	Validation of IEC measurement by multiple labs	04/30/2020

Session Summary

Session ID: L2-B

Title: AEM Membrane: Conductivity, Gas Permeability, Chemical Stability

- Summary of discussion
  - Conductivity
  - Oxidation resistance
  - Creep rate

- Consensus and/or dissenting opinions
  - Protocols for AEMs need to consider other liquid containing platforms
  - Long term goal to use pure water

- Key Take-Aways
  - Oxidation resistance protocol not established, but needed
  - Conductivity protocol needs improvements
  - Creep rate protocol not established but needed

- Action Items
  - Update conductivity protocol
  - Initiate oxidation resistance protocol
  - Initiate the creep rate protocol

Session Attendee List

Session ID: L2-B

Title: AEM Membrane: Conductivity, Gas Permeability, Chemical Stability

Name	Affiliation
Jimmy Rojas	Stanford U
Adrian Gestos	AquaHydrex
Alexey Serov	Pajarito Powder
Shannon Boettcher	U Oregon
Ehren Baca	Sandia National Lab
Cy Fujimoto	Sandia National Lab
Sanwoo Lee	RPI
Chris Capuano	Nel Hydrogen
Yushan Yan	U Delaware

Session Summary

Session ID: L4-A

Title: PGM Catalyst: RDE

Summary of discussion

- Protocols are already defined, and next step before further work is the validation. Key here is to identify and create strong coordination + commitment so that the work can be finalized.

Consensus

- Start Round Robin Test ASAP

Dissenting opinions

- Is RDE suitable for PGM testing (LTE)
- The use of Au or GC RDEs

Key Take-Aways

Action Items

- Access/Decide on RDE substrate
- Access/Decide on CE
- Reach out int. community for protocol feedback
- Round robin testing RDE
  - National: NREL, LBNL, SRNL, ANL, Stanford
  - LBNL to coordinate
  - International: Jülich, TUB, TUM, Julich

Session Attendee List

Session ID: L4-A

Title: PGM Catalyst: RDE

Name	Affiliation
Kathy Ayers	Nel Hydrogen
Corky Mittelsteadt	Giner
George Roberts	Nel Hydrogen
Sara Park	LANL
Sangwoo Lee	RPI
Huyen Dinh	NREL
Ahmet Kusoglu	LBNL

Session Action Item Assignments

Session ID: L4-A

Title: PGM Catalyst: RDE

Name	Affiliation	Action Item	Target Due Date
Marcelo Carmo	Jülich	Distribute Protocols to other experts. Link to the Annex 30 RRT RDE activity.	
Nem Danilovic	LBNL	Coordinate RRT-RDEs	
NREL (Shaun Allia)		Access/define RDE substrate	

Session Summary

Session ID: L4-B
Title: Non-PGM Catalyst: Electroconductivity

Discussion:

- Protocol review on measuring PGM-free catalyst conductivity
- Detailed discussion on setup and procedure

Consensus:

- Procedure useful for any catalyst powder not just PGM-free
- Some verification of reproducibility is needed
- Safety and reproducibility can be improved by defining equipment
  - Max voltage needed, force sensor
- Protocol for measuring in-plane resistance of electrode also needed

Key Take Aways:

- Method can be improved with minimal effort
- Blueprints would support dissemination of method
- Reproducibility needs to be confirmed
- Funding would be useful for the verification/implementation

Actions:

- Identify funding for implementation
- Edit protocol
- Update schematic
- Create CAD drawings for dissemination
- Round robin test

Session Attendee List

Session ID: L4-B
Title: Non-PGM Catalyst: Electroconductivity

Name	Affiliation
Guido Bender	NREL
Alexey Serov	Pajarito Powder
Yushan Yan	Delaware University
Adrian Gestos	Aqua Hydrex
Chris Capuano	Nel Hydrogen
Cy Fujimoto	SNL
Ehren Baca	SNL
James Vickers	DOE
Jimmy Rojas	Stanford University
Hoon Chung	LANL
Chulsung Bae	RPI
Hector Colon-Mercado	SRNL

Session Summary

Session ID: L5-A

Title: PTL and Definitions

- Summary of discussion
  - No protocols
  - Went over background for developing protocols
    - Corrosion
    - Mechanical

- Consensus and/or dissenting opinions
  - Community lacking a reference material to baseline against
  - Can corrosion protocols from bipolar plate work be applied to PTLs?

- Key Take-Aways
  - Need to understand what pH is at PTL interface
  - Accelerating factors still need to be identified for corrosion/degradation mechanisms
  - Mechanical testing protocols should look to implement a tool that has differing channel and landing width
  - Protocol should include a part flatness and surface topography

- Action Items
  - PTL
    - Establish baseline PTL
  - Definitions
    - testing in-situ and cell is evolving gas, should include a safety warning about oxygen generation
    - When do targets get added and what are they?
    - In the SOP, add section on how components are classified

Session Attendee List

Session ID: L5-A

Title: PTL and Definitions

Name	Affiliation
Nemanja Danilovic	LBNL
Ahmet Kusoglu	LBNL
Adam Weber	LBNL
Chris Capuano	NEL
Kathy Ayers	NEL
Adrian Gestos	Aquahydrex
Thomas Malkow	European Commission, JRC
Hoon Chung	LANL
Guido Bender	NREL

Session Action Item Assignments

Session ID: L5-A

Title: PTL and Definitions

Name	Affiliation	Action Item	Target Due Date
Kathy Ayers	NEL	Revise definitions document	1/31/2020
Guido Bender	NREL	Test Ti mesh as “common PTL”	3/31/2020

Session Summary

Session ID: L5-B

Title: Gas Diffusion Layer: Characterization Protocols

Discussion:

- Key requirements for GDL
- Review of LTE-P-1 Compressibility Protocol

Consensus:

- Need to tailor hydrophilicity of GDL/MPL for LTE
- Establish a reference standard (i.e. SGL Sigracet)
- Different requirements for
  - AEM vs. PEM
  - Catalyst coated substrates (vs. catalyst coated membranes)

Key Take-Aways:

- GDL Key Requirements
  - Porosity
  - Tensile strength (bridging)
  - Hydrophilicity (contact angle)
  - Uniformity of properties (thickness, porosity) Thermal conductivity- not important
  - Defect characterization (fibers could poke through membrane)
  - Purity: Leachable impurities (possible contaminants to membrane/catalyst)
- Leverage GDL suppliers from PEM Fuel Cells

Actions:

- Update GDL Compressibility protocol with:
  - Increase maximum loading pressure to 500 psi (closer to stack loading pressure)
  - Explain justification for loadings selected
- Work directly with GDL supplier to adopt these protocols
- Use this compressibility method to also measure contact resistance (may need gold platens)

Session Attendee List

Name	Affiliation
Karl Gross	H2 Technology Consulting
Hector Colon-Mercado	SRNL
James Vickers	DOE
George Roberts	Nel Hydrogen
Chulsung Bae	RPI
Sangwoo Lee	RPI
Sarah Eun Joo Park	LANL
Yushan Yan	U of Del
Jimmy Rojas	Stanford
Alexey Serov	Pajarito Powder

Session Action Item Assignments

Name	Affiliation	Action Item	Target Due Date
George Roberts	Nel Hydrogen	Update GDL Compressibility protocol with: • Increase maximum loading pressure to 500 psi (closer to stack loading pressure) • Explain justification for loadings selected	12/1/2019

Session Summary

Session ID: L6-A

Title: Wrap-up/Action Item Assignment: LTE Membrane, Catalysts

Discussion

- Technology Roadmaps
  - AEM
  - PEM
- Test protocols

Consensus and/or dissenting opinions

- Protocols for AEMs need to consider other liquid containing platforms
- Test Protocol should start ASAP

Key Take-Aways

- A concerted funded effort for validation is required and encouraged
- Need a consistent AEM membrane
- Accelerated stress tests should be developed with standard cell hardware

Action Items

- Begin round robin verification of test protocols
- Draft accelerated test protocols

Session Attendee List

Session ID: L6-A

Title: Wrap-up/Action Item Assignment: LTE Membrane, Catalysts

Name	Affiliation
Sangwoo Lee	RPI
Nemanja Danilovic	LBNL
Hector Colon-Mercado	SRNL
Alexey Serov	Pajarito Powder
Ehren Baca	SNL
Cy Fujimoto	SNL
Corky Mittelsteadt	Giner
Stephanie Byham	DOE
Chulsung Bae	RPI
Sarah Eun Joo Park	LANL
Christopher Capuano	NEL
Yushan Yan	U. Delaware

Session Action Item Assignments

Session ID: L6-A
Title: Wrap-up/Action Item Assignment: LTE Membrane, Catalysts

Name	Affiliation	Action Item	Target Due Date
Yushan Yan	Delaware	Reach out to journals to engaging on benchmarking effort	

Session Summary

Session ID: L6-B

Title: Wrap-up/Action Item Assignment: LTE Components, Stacks Technology

Discussion:

- Review of stack/component tests
- Gap in discussion on ASTs discussed further
- Impact of systems components on stack performance (e.g. patent from DeNora on iron protection)
- Use protocols as teaching tools with pitfalls and rationale

Consensus:

- Consider amplitude vs. duration; impact of ramp rates
- Need to be cautious about interacting variables (e.g. current density vs. water flow)
- Intermittent analysis (pre-failure) of structural changes with AST vs. baseline
- Need standard materials/baseline test to verify setup

Key Take Aways:

- Need to relate tests to real systems; get industry input, post operational diagnostics
- Need to compare lab data with real field data
- Need to understand translation/interacting variables to accelerate
- Need for break in procedures
- Open access important – e.g. ECS Archive

Actions:

- Need to get the group that has done cycling together and look for common operating conditions
- Need a marketing strategy – lack of awareness of parallel efforts/past publications
  - Improved access to broader community for DataHub etc.
  - Archives/open access articles
  - Series in relevant journal
  - Working group to look at best options
- Leverage JRC work – look for agreement to 5%
- Need standard post mortem analysis

Session Attendee List

Session ID: L6-B

Title: Wrap-up/Action Item Assignment: LTE Components, Stacks Technology

Name	Affiliation
George Roberts	Nel Hydrogen
Guido Bender	NREL
Kathy Ayers	Nel Hydrogen
Ahmet Kusoglu	LBNL
Amin Nouri	Greenlight Innovations
Shannon Boettcher	U. of Oregon
Jimmy Rojas	Stanford
Adrian Gestos	Aquahydrex
Dave Peterson	DOE

Session Action Item Assignments

Session ID: L6-B
Title: Wrap-up/Action Item Assignment: LTE Components, Stacks Technology

Name	Affiliation	Action Item	Target Due Date
Adrian Gestos	Aquahydrex	Interest in AST definition	
Shannon Boettcher	U of Oregon	Degradation mechanisms	
Ahmet Kusoglu	LBNL	Coordination of membrane PEM/AEM protocols, validation, publication compilation	
Guido Bender	NREL	What components can we find ASTs for? Post characterization	
Shaun Alia (via Guido)	NREL	Leverage catalyst work; connect Ahmet with Kelly at NREL	
Ahmet/Kathy/Shannon		Communication with publications and context to drive purpose	
Amin Nouri	Greenlight	Standardization of polarization curves and analysis/diagnostics	