Energy Materials Network
U.S. Department of Energy

HydroGEN
Advanced Water Splitting Materials

Advanced Water-Splitting Technology Pathways Benchmarking & Protocols Workshop

Breakout Session Summaries
Low Temperature Electrolysis

October 24 - 25, 2018

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

PROTON ON SITE
ASU Arizona State University
Caltech
Pacific Northwest NATIONAL LABORATORY

LTE Breakout Sessions

Breakout Session #	Session ID	Technology	Topic	Lead
2	L2-A	LTE	PEM: Membrane Physical Requirements/Tests	Bryan Pivovar (NREL)
2	L2-B	LTE	Non-PGM Catalyst: OER Stability & Activity	Alexey Serov (Pajarito Powders)
3	L3-A	LTE	AEM: Membrane Physical Requirements/Tests	Yu Sueng Kim (LANL)
3	L3-B	LTE	PGM Catalyst: OER Stability & Activity	Nemanja Danilovic (LBL)
4	L4-A	LTE	PTL: Characteristics & Characterization Tools	Adam Weber (LBL)
4	L4-B	LTE	MEA Device Level Protocols: Criteria/Tests	Guido Bender (NREL)
5	L5-A	LTE	Carbon GDL: Physical Requirements/Tests	Chris Capuano (Proton Onsite)
5	L5-B	LTE	Full Stack Level Protocols: Criteria/Tests	Corky Mittelsteadt (Giner)
6	L6-A	LTE	Accelerated Stress Tests	Chris Capuano (Proton Onsite)

Session Summary

Session ID: L2-A

Title: PEM Membrane

Summary of Discussions

- Industry can provide guidance on prioritization of characterization methods
- Need to look at this in two aspects
  - Standardizing material baseline
  - Benchmarking/Characterization to assess alternates and how changes impact durability/performance

Consensus and/or dissenting opinions

- For standardization, N117 can likely serve as the PEMWE baseline, as well as the 1100 dispersion
  - Some offered we should consider N115
- An area that needs to be further developed is in the post-mortem. As important in the initial benchmarking/characterization

Key Take-Aways

- Can't just take what is available for FC and think they will work in WE. Have significantly different mechanical and performance requirements.
- Understanding thermal mobility/stability of membrane is important in predicting long-term creep.
- Need to understand from industry what we can expect from field operating conditions

Action Items

- Identify degradation mechanisms
- Develop a tiered list of characterization methods to rank importance and requiring further development
- Explore AST for evaluating thinner membrane to understand risk

Session Summary

Session ID: L2-B

Title: Non-PGM Catalyst

Summary of discussion

- Methods on measuring Activity and Durability of PGM-free (OER) catalysts: main discussion on RDE experiments; benchmarking IrO_2; Round Robin RDE (6 labs agree)

Consensus and/or dissenting opinions

- Cons: RDE is a right tool for screening of catalysts performance and can be used to compare performance of new PGM-free materials (after establishing benchmarking performance – IrO_2)

Key Take-Aways

- Realistic measurements of MEA performance for PGM-free catalysts are complicated (especially in AEM case)
- Electroconductivity of PGM-free catalysts should be measure (pellets) and reported

Action Items

- Establish benchmarking test for comparison of PGM-free catalysts performance to IrO_2 (RDE). Requires agreements on unified batch of IrO_2 available for testers (HydroGEN), a testing protocol (NREL PGM protocol as a starting point), potentially “open source” of glass-ware setup for RDE tests

Session Summary

Session ID: L3-A

Title: AEM Membrane

Summary of discussion

- Hydroxide conductivity: Counter ion: chloride conductivity easier? By factor of 3 or 3.5 of chloride vs hydroxide. With hydroxide issues with carbonate/membrane issues. In liquid water or under humidity.
- IEC: Tool for degradation.
- 2D change: x and y since can be measured easier and can be minimized by support materials. 100% not an issue since electrolyzer is not an issue since it always humidified.
- Gas permeability: measure hydrogen current, hydrogen at one electrode vs nitrogen and another electrode.
- Chemical stability: Sealed vessels under 1 or 2 M NaOH, IEC easiest to keep track of, also conductivity. Stress strain curves before and after NaOH soak. Most accurate method spectroscopic method since conductivity could be due to morphology. Spectroscopic (NMR and FT IR) in addition to IEC and conductivity.
- Carbonate still an issue in LTE.

Consensus and/or dissenting opinions

- Water flux (water permeability) is key metric in LTE. Water flux supports electrolysis in flooded cell. Cathode feed does not help. At high current operation, mass transport limitation.
- Water content: Does it need to be taken at 80 C, can room temperature be ok. 80 C why, since it is a target temperature. Issue since most membranes cannot survive under this condition.
- Tensile strength: Nafion 95% RH modulus 34 MPa. How to relate modulus to actual physical metrics. Burst measurement good metric but not a common piece of equipment. Reinforcement or cross linking can be used to improve mechanical properties.
- Creep rate: Also key issue, more important than tensile strength. What the membrane see in the cell, understanding the compression and the creep rate. Difficult to measure
- Hydrocarbon ionomer is being oxidized/reduced in catalyst layer. Need ionomer that does not absorb on catalyst layer. Need to address this in the future.

Key Take-Aways

- Water flux (water permeability) is key metric in LTE. Water flux supports electrolysis in flooded cell. Cathode feed does not help. At high current operation, mass transport limitation.
- Hydrocarbon ionomer is being oxidized/reduced in catalyst layer. Need ionomer that does not absorb on catalyst layer. Need to address this in the future.
- Carbonate still an issue in LTE.

Action Items

- NREL and Los Alamos agree to evaluate the AEMs to propose standard materials. → This is critical component as the major HydroGen LTE R&D focuses on alkaline membrane electrolysis (PEM electrolysis is matured).
- Scalability (mass production) and cost analysis needs to be done (Tetramer).

Session Summary

Session ID: L3-B

Title: PGM Catalyst

Summary of discussion

- Benchmark materials and protocols
- Crosscut with non-PGM session
- Cathode Pt/C consensus
- Anode Ir? Much harder to get consensus

Consensus and/or dissenting opinions

- RDE is a tool that has application but must be understood
- Translation to MEA is not guaranteed
- Consistency in standard materials is very important
- Durability testing steady state vs cycling vs pure dissolution
- Inks interactions are very important

Key Take-Aways

- Need reproducible, consistent and precisely defined composition of IrO2 as baseline
- Need a durability protocol
- Living document can be modified in the future
- Need an ECSA protocol, ASTs?

Action Items

- Near term use IrO2 from AA
- Medium term Pajarito to have non-proprietary product IrO2 available
- NREL-LBNL to initiate protocols
- ECSA and AST development is required

Session Summary

Session ID: L4-A

Title: PTL: Characteristics & Characterization Tools

Summary of Discussion

- There was discussion concerning the importance of the PTL and also its standardization in terms of materials versus protocol benchmarking for its characterization
- Need to understanding coatings as well as PTL base material
- Define porosity and mesh spacing
- IEA has done and discussed a lot on this
- Need to understanding compliance

Consensus and/or dissenting opinions

- Need AST but not sure of the form of it in terms of property changes (e.g., oxide film)
- Balance between passivation and contact resistance
- Need vendor
- Cannot disassemble and do characterization

Key Takeaways

- Cathode: Carbon paper (e.g., SGL)
- Anode: need Ti
  - Availability?
  - Coating?
    - Perhaps can do limited studies and performance with just uncoated substrates

Action Items

- Define corrosion protocols including perhaps ASTs
  - High potential (perhaps with non aqueous)
- Define mechanical testing protocols
- Define resistance measurements and water properties
  - Bubble management
- Detailed characterization of baseline materials
  - Done for carbon GDLs

Session Summary

Session ID: L4-B

Title: MEA Device Level Protocols: Criteria/Tests

- Introductions
- High level discussion about the intent to develop framework to the next step
- What content is addressed by this group
- What is an MEA => 3 layer or 5 layer?
- Do we need to determine benchmarking/testing devices and materials
- How can IEA work be leveraged
- Review of the current framework / experimental list and setting of priorities

Priority of Experimental List	PEM	AEM
Cell Voltage / Overpotential	High	High
H2 permeation	Medium	Medium
Water Flux	Low	High
Durability: Steady State	High	High
Durability: Cycling	High	High
Short Circuit Resistance	Low	Medium to Low
Catalyst Layer Thickness analysis	Take out, this is a post mortem	
Catalyst Layer Porosity analysis	Take out, this is a post mortem	
High Frequency Resistance	High	High
Impurity	Medium	Medium
Electrolyte Concentration	-	
Effluent Water Analysis	Medium	Medium

Key Takeaways

- Terminology used, such as for example “MEA” needs to be defined
- AEM and PEM systems have different needs and should be treated separately
- Community sees the benefit and is willing to participate in smaller working groups to address the need

Action Items

- Working groups were created for AEM & PEM to define:
  - What belongs to the expression MEA.
  - What benchmark materials could be used.

For AEM & PEM:

- Define measurement system
- Review experiment list with specific working group
- Perform round robin testing to identify spread

Session Summary

Session ID: L5-A

Title: Carbon GDL: Physical Requirements/Tests

Summary of Discussions

- Most available GDLs (carbon paper) are primarily for fuel cells
  - Difficult to get alternatives that might work better for electrolysis unless you have a project or development effort with supplier
- Tests identified in characterization spreadsheet look generally accurate for characterization
  - Potential to add a few more and prioritize the list

Consensus and/or dissenting opinions

- For standardization, Sigracet is being viewed as an option
  - Available to research community
  - Supplier well-established
- GDL identified for PEMWE is likely not the same used for AEMWE
- Thickness will be determined by cell configuration being used

Key Take-Aways

- Most GDLs have been made for fuel cells, so optimization for WE needs to happen
- In-plane resistance measurements not viewed as important as others
  - Compressibility and strength is viewed as more relevant and should be added to tests.
- Post mortem testing of carbon layer needs definition
  - Corrosion/Oxidation
  - Wettability before and after?

Action Items

- Identify degradation mechanisms
- Develop a tiered list of characterization methods to rank importance and requiring further development
- Evaluate potential methods of post mortem and critical criteria for measure

Session Summary

Session ID: L5-B

Title: Stack Protocols

Summary of discussion

- Lots of talk about difference between types of stacks, and needs for different standards for each.
- Discussion for need of long term testing protocol
- Where should efficiency be measured, stack or system
- Pol Curves should be taken periodically
- When does scale matter

Consensus and/or dissenting opinions

- Standard testing should be 60C and atmospheric pressure, lower temps are not of interest and pressurized operation excludes too many people.
- 15 uV/hr should not be a disqualifier over the first 1000 h, might disqualify something promising
- Efficiency should be measured at stack and system level
- Direct current cell testing needs to be done
- Larger are better, but small is ok for catalyst and membrane work.

Key Take-Aways

- kWh/kg much preferred over LHV or HHV % efficiency
- 1A/cm2 is fine baseline, but AEM needs to be different
- Degradation should be from hours 500-1000, do not include break-in
- 2% vol H2 in I2 is too high for crossover, should not get that close

Action Items

- Would be nice to have operating load profile for simulated wind or solar load, NREL or providers?

Session Summary

Session ID: L6-A

Title: LTE ASTs: Ad-Hoc Session

Summary of Discussions

- ASTs developed should differentiate if they are for assessment of chemical or mechanical degradation or both
- Possible/likely may have to develop different protocols for PEMWE and AEMWE
- Different ASTs will have to be developed for exploration of different mechanisms

Consensus and/or dissenting opinions -

- There was uncertainty that an AST could be developed which could provide predictability
- Interest was expressed on the recoverability from AST effects
- High temperature should have an accelerating effect on both membrane and catalyst degradation

Key Take-Aways

- Lower catalyst loadings can be used to drive up cell potential on a cell to accelerate some forms of degradation
- Water quality should be looked at for degradation effect
  - Impacts both catalyst and membrane
  - Is it recoverable and if so, what does this mean for remaining life of cell?
- Differential pressure stress was viewed as more than a membrane degrader
  - Assess quality of PTL
  - How does increase hydrogen cross-over degrade membrane

Action Items

- Incorporate water impurity characterization
- Identify what operating parameter being changed impacts what component