HydroGEN
Advanced Water Splitting Materials

Advanced Water-Splitting Technology Pathways Benchmarking & Protocols Workshop

Breakout Session Summaries
Cross Cutting Topics

October 29, 2019

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Executive Summary

The Cross-Cutting breakout sessions focused on common materials shared between water splitting technologies, best practices for developing and executing test protocols and alignment with international protocol development. General themes include:

- Hybrid Thermal Chemical Cycle (Electrochemical Step)
  - Many LTE protocols can be leveraged
  - Leverage other electrochemical system test methods (Fuel cells, flow batteries)
  - Improve linkage/focus of Hybrid Thermochemical Cycle to existing water splitting technology projects to accelerate development
- PEC/LTE Common Materials
  - Non-PGM Catalyst
    - Dissolution rates and how they depend on potential and environment important.
    - Most important: liq. Electrolyte, solid electrolyte, current density, activity across time, ICP-MS, QCM, XRF
    - AEM vs. PEM and Trad. Alkaline. PEC optical properties important.
  - Membrane
    - For PEC: membrane requirements are device architecture specific and often very different to LTE (could be PEM, AEM or bipolar)
    - For LTE: chemical and mechanical durability, good conductivity and low gas permeability all important
- International Alignment
  - There's a need for coordination and harmonization in and of the four pathways, irrespective of the differing stages of progress in both the research/development
  - Continue to organize coordination meetings in association to well-matched conferences.

Cross Cutting Breakout Sessions

Breakout Session #	Session ID	Technology	Topic	Lead
3	C3-A	Cross Cutting	Hybrid Thermochemical Cycle Material Screening- Electrochemical step	Hector Colon-Mercado (SRNL)
3	C3-B	Cross Cutting	PEC/LTE Cross-cutting: Non-PGM Catalysts	Shannon Boettcher (U of Oregon)
3	C3-C	Cross Cutting	PEC/LTE Cross-cutting: Membrane Requirements & Tests	Chris Topping (Tetramer)
3	C3-D	Cross Cutting	Cross-Cutting Elements of Protocol Development (calibration, null measurements, etc)	Karl Gross (H2 Technology Consulting)
3	C3-E	Cross Cutting	Cross-Cutting Elements of Protocol Development	Guido Bender (NREL)
3	C3-F	Cross Cutting	International alignment on Benchmarks, Protocols, and Roadmaps	Ivan Ermanoski (Arizona State Univ)

Session Summary

Session ID: C3-A

Title: Hybrid Thermochemical Cycle Material Screening- Electrochemical step

Discussion:

- Many LTE protocols applicable to HTC, especially catalyst characterization
- Specific conditions will be different or change with time – e.g. ECSA
- Crossover issues are somewhat similar but for multiple species (e.g. SO2)
- Conductivity more complex due to conductive electrolyte
- May want to go to H2 pressure later

Consensus:

- Sampling solution could be useful for multi-component catalyst to measure degradation (constant V)
- Going too high in acid reduces conductivity
- Need to watch crossover for toxicity rather than flammability

Key Take Aways:

- Not sure if there is a need for improved carbon paper or not? Will set differently from water

Actions:

- Should consider similar activity on high temp side
- Should look at other electrochem technologies (e.g. flow batteries) as well
- Link to fuel cells e.g. high temp membranes
- Need better linkage/focus to make progress (more projects would likely help)

Session Attendee List

Session ID: C3-A

Title: Hybrid Thermochemical Cycle Material Screening- Electrochemical step

Name	Affiliation
Hoon Chung	LANL
James Vickers	DOE
Kathy Ayers	Nel Hydrogen
Hector Colon-Mercado	SRNL
Nemanja Danilovic	LBNL

Session Summary

Session ID: C3-B
Title: PEC/LTE Non-PGM Catalysts

Summary of discussion:

Parameters that are important to know:

- Conductivity (in situ, as conductivity can change with E)
- Surface area (BET, electrochemically accessible)
- Need effective normalization of activities (via mass, mol of catalyst etc.)
- Dissolution rates and how they depend on potential and environment important.
- Most important: liq. Electrolyte, solid electrolyte, current density, activity across time, ICP-MS, QCM, XRF
- AEM vs. PEM and Trad. Alkaline. PEC optical properties important.

Consensus

- Much foundational work needed to develop platform for materials testing and development

Key Take-Aways

- Oxide and other non-PGM catalysts much more complicated than metals
- Surface area, conductivity, surface phase changes etc. all difficult to track
- Connections between in-situ and ex-situ measurements need to be made
- Thickness, loading, ionomer or liq. electrolyte measurements all important
- Good ionomer/membranes are needed particularly for AEM, catalysts may be there?

Action Items-Protocols:

- Cleaning electrolyte (e.g. base)
- Inks for OER (non-carbon conductive support?)
- Mass normalization (make activity-stability measurement under *mass normalized* currents relevant to application?
- Nonaq. capacitance measurements to measure surface area? (https://pubs.acs.org/doi/full/10.1021/jacs.7b10966)
- In-situ/ex-situ conductivity measurement protocol
- Dissolution rate protocol (online ICP-MS?)

Session Attendee List

Session ID: C3-B
Title: PEC/LTE Non-PGM Catalysts

Name	Affiliation
Shannon Boettcher	U. Oregon
James Young	NREL
Ehren Baca	Sandia
Chris Capuano	Nel Hydrogen
Shinjae Hwang	Rutgers
Siwei Liang	LLNL
Alexey Serov	Pajarito Powder
Srinivas Vanka	U. Michigan
Guosong Zeng	LBNL

Session Summary

Session ID: C3-C

Title: PEC/LTE Cross-cutting: Membrane Requirements & Tests

Summary of discussion

- Discussed shared and unique membrane requirements of LTE & PEC
- Covered conductivity (various ions), permeability (O_2, H_2, hydrocarbons), mechanical stability (water uptake, pressure), chemical stability (oxidation, electrolyte impurities), durability & cost
- Role of electrolyte in PEC

Consensus and/or dissenting opinions

- Both LTE & PEC have a *lot* of variables
- PEC variables increased further by lack of single leading device design
- PEC & LTE have similar temperature requirements
- Mechanical stress (pressure) less of an issue for PEC
- So far most focus of PEC on H^+ / Na^+ (due to availability of Nafion)
- Chemical degradation should be less severe for PEC (membrane further from site of electrochemical reaction)

Key Take-Aways

- For PEC: membrane requirements are device architecture specific and often very different to LTE (could be PEM, AEM or bipolar)
- For LTE: chemical and mechanical durability, good conductivity and low gas permeability all important
- Ion conductivity less important for PEC (low current density) but low H_2/O_2 permeability more important
- Physical/chemical characterization database of leading membranes would be a useful
- UV, visible, IR degradation is unique to PEC (but could be mitigated by cell design)

Action Items

- Limit variables
- Down-select / prioritize leading PEC cell designs
- Prioritize general membrane requirements common to leading PEC devices
- Define PEC specific membrane stresses based on collective observations of failure mechanisms
- Aim to relate PEC & LTE membrane failures to fundamental physical/chemical properties

Session Attendee List

Session ID: C3-C
Title: PEC/LTE Cross-cutting: Membrane Requirements & Tests

Name	Affiliation
Chris Topping	Tetramer Technologies
Huyen Dinh	NREL
Corky Mittelsteadt	Giner
Sarah Eun Joo Park	LANL
Chulsung Bae	RPI
Josh Spurgeon	Univ. Louisville
Adam Weber	LBNL
Shane Ardo	UC Irvine
Cy Fujimoto	Sandia NL
Sangwoo Lee	RPI
Yushan Yan	Univ. Delaware
Ahmet Kusoglu	LBNL

Session Summary

Session ID: C3-D
Title: Cross-cutting Elements of Protocol Development

- Summary of discussion
  - Protocols avoid Mistakes: time, & and effort lost (vs. students training).
  - Discussed key elements of protocols across technologies using examples:

Clear Terms & Units , Calibrations, Null Measurements, Sample Prep & Measurement Conditions, include Common Issues, Standards for Validation, Round Robins.

- Consensus
  - Example of x-cutting metric that is not clear across fields: efficiency
  - Effect of conditioning on data.
  - Effect of local ambient temperature and pressure

- Key Take-Aways
  - Importance of instrument calibration: zero point, standard…
  - A baseline difficult to establish, regardless of the technology.
  - Include Lesson Learned and make sure that knowledge remains for long period of time

- Action Items
  - Find reference standards for all technologies: challenging but necessary.
  - Round Robin Testing: first standard then materials to be benchmarked. Recent testing with PEM encouraging

Session Attendee List

Session ID: C3-D

Title: Cross-cutting Elements of Protocol Development

Name	Affiliation
Robert Bell	National Renewable Energy Laboratory
Dong Ding	Idaho National Laboratory
Nicolas Gaillard	University of Hawaii
Adrian Gestos	AquaHydrex
Karl Gross	H2 Technology Consulting, LLC
Kevin Huang	University of South Carolina
Wei Li	West Virginia University
Xingbo Liu	West Virginia University
Samantha Millican	University of Colorado Boulder
Nguyen Minh	University of California San Diego
Amin Nouri	Greenlight Innovation
Tadashi Ogitsu	Lawrence Livermore National Laboratory
Jimmy Rojas	Stanford University
Jonathan Scheffe	University of Florida
Neal Sullivan	Colorado School of Mines
Scott Swartz	Nexceris, LLC
Alan Weimer	University of Colorado Boulder

Session Summary

Session ID: C3-E
Title: Cross-Cutting Elements of Protocol Development

- 4 technologies with different status on standard protocol development
  - STCH: Beginning of process
  - HTE: Wild West
  - LTE:
    - Formulating protocols and testing underway
    - International effort with Europe
    - In-Situ further developed than Ex-Situ
  - PEC:
    - Book published in past
    - Unknown if community is aware of the book
    - Is book up to date?

- National Labs can play a role in performing unbiased testing of results and upkeeping standards (3rd party verifications)
- A significant part of the community needs to participate to gain critical mass and make standards meaningful
- A common strategy for protocol development may exist
- Frequent protocol review (5 years?) desirable

- Culture shift is required for community to follow standard protocols
- Measures are needed that encourage participation
  - Funding sources? => key metrics, deliverables, procedures
  - Community? => Reviewers of peer reviewed journals

- Communicate with DOE:
  - Standard protocols reduce development times and accelerate research
  - Funding is needed to put them in place
- Study the PEC book case
  - Use lessons for implementation of standards in the other communities
- Develop high level format for all technologies
  - Material characterization protocols
  - Operating protocols

Session Attendee List

Name	Affiliation
Guido Bender	NREL
Joseph Barton	Fuel Cell Energy
Ani Kulkarni	CSIRO
Chris Muhich	ASU
Tianli Zhu	UTRC
Eric Coker	SNL-NM
Frances Houle	LBNL
Walter Drisdell	LBNL
Joseph Hartvigsen	OxEon
Jimmy Rojas	Stanford University
George Roberts	Nel Hydrogen
Jie Pan	MSU
Zetian Mi	Michigan University
Michael Sanders	CSM

Session Summary

Session ID: C3-F

Title: International Alignment on Protocols and Roadmaps

Summary of discussion

Alignment efforts in all hydrogen production pathways were discussed. Perspectives on priorities vary by region (e.g. cost in US, vs. environmental benefits in Europe). Priorities also matter for academia: harmonization does not produce high-impact publications. The need for alignment, harmonization, and benchmarking is well-appreciated in all pathways, and is to some extent ongoing. The effort is fragmented and largely operates on volunteered time. Funding agencies have generally not supported these activities at a level necessary for a timely and robust effort.

Consensus and/or dissenting opinions

- Protocols and roadmaps are absolutely necessary, and so is international coordination and alignment
- Dedicated funding to coordination activities is necessary
- Organizing coordination meetings in association with major conferences is an appealing path forward, but may or may not work out
- Connecting existing coordination activities is highly desirable

Key Take-Aways

Most importantly: there's a need for coordination and harmonization in and of the four pathways, irrespective of the differing stages of progress in both the research/development (e.g. low temperature electrolysis is commercial, whereas other technologies have not yet reached that level) and existing harmonization (much has already been done in protocols for LTE and PEC, not as much in STCH, for example).

Action Items

- Continue to organize coordination meetings in association to well-matched conferences.
- Connect existing coordination programs, such as those ongoing in DOE, SolarPACES, IEA
- Ellen Stechel: Report back from the MI-5 meeting later this year