Energy Materials Network
U.S. Department of Energy

HydroGEN
Advanced Water Splitting Materials

Advanced Water-Splitting Technology Pathways Benchmarking & Protocols Workshop

Breakout Session Summaries
PEC

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PEC Breakouts

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P2-A	Protocol development in a half cell vs. a full cell	Todd Deutsch
P2-B	In situ/operando methods for PEC interfaces and devices	Shu Hu & Walter Drisdell
P3-A	Protocols for PEC stability testing	Kimberly Papadantonakis
P3-B	PEC electrolytes	Adam Weber
P4-A	Prototype formats and key metrics for benchmarking	James Young
P4-B	Protocol development on OER/HER activity benchmarking at intermediate/dynamic current density	Nemanja Danilovic
P5-A	PEC Nodes capabilities and gaps assessment	Tadashi Ogitsu
P6-A	PEC Workshop Action Items	CX Xiang

P2-A Protocol development in a half cell vs. a full cell Todd Deutsch

Summary of Discussion

- What leads to different results in half vs. full-cell is unknown
- Full cell testing should be done if it is possible
- The field needs to be made aware of these differences
- A study to quantitatively evaluate the differences should be done

Consensus or Dissenting Opinions

- Consensus: Diagnostic half-cell testing is useful but should not be used to predict full-cell efficiency or durability

Key Take-Aways

- The shortcoming of half-cell test is not widely recognized by the fields
- More groups reporting their failed test would be useful to raise awareness of this issue

Action Items

- The PIs of groups that have full-cells capable of spontaneous water splitting (JCAP, NREL, Zetian) should discuss 2E vs. 3E testing and consider writing a perspective piece.

P2-B In situ/operando methods for PEC interfaces and devices — Shu Hu & Walter Drisdell

Summary of Discussion

- Goal: how operando analysis help address the trade-off between efficiency and stability?
- Degradation occurs at various time-scales; how to use in-situ analysis to develop Accelerated Stress Testing (AST) protocols?

Consensus or Dissenting Opinions

- Delineate different interfaces that's important for PEC.
- All interfaces are important, don't know where the failure took place, but definitely has priorities.
- One emphasis mentioned here is the dynamics between material and electrolytes (corrosion, deposition of impurities, morphology changes
- In situ and operando methods, especially synchrotron facilities, useful to look at device level performances, but low throughput.
- Other in situ methods, ICP-MS or ATR-IR or microscopy, instead of synchrotron to look at device level performances.

Key Take-Aways

- In situ and operando measurements maybe categorized for different purposes:
- 1) Performance based -> benchmarking protocols
- 2) Discover based -> theory
- Framework: Theory in the center; feed to Benchmarking protocols, underlies the best practice suggested by experts; Degradation of catalysts, absorbers; interfaces need theory inputs;
- Benchmarking protocols should not be restrictive to certain materials, but a general guidance.

Action Items

- Sharing information/Database
- Establish a flow chart/staged pyramids to understand the need of synchrotron measurements,
- Simplest/minimal measurements needs to do
- Strategic: match making between nodes and PEC community to understand the critical needs; improving durability beyond 100 hours.
- Degradation mechanism for photoelectrochemical interfaces; A mini review paper, what are the compromises?(Shu Hu et al.)

P3-A Protocols for PEC stability testing — Kimberly Papadantonakis

Summary of Discussion

- How to relate three-electrode to two-electrode stability experiments? What are the right test conditions (potential, current density)?
- How can we understand degradation mechanisms for complex interfaces ? Mechanistic studies are time-consuming.
- Current best practices include chronoamperometry or chronopotentiometry, analysis of electrolyte for dissolved species, and before/during/after imaging or spectroscopy.
- Tests only detect certain mechanisms, current focus is on electrochemical mechanisms, but other pathways (e.g. chemical or erosion) may be important at longer timescales.

Consensus or Dissenting Opinions

- 3-electrode experiments are important tools for testing component stabilities, but these tests are typically not performed at the operating points of a 2-electrode device.
- If we can define "standard" operating characteristics for a counter/partner electrode and cell resistances etc, then we can relate 3-electrode to 2-electrode measurements.
- We do not need to define a minimum electrode area for testing, but do need to provide statistically representative results, not just champions.
- PEC community is not well aware of practices in corrosion science/engineering and might learn a lot from that field.

Key Take-Aways

- PEC stability testing is complex and challenging.
- There is a need to understand how to relate 3-electrode and 2-electrode stability testing.
- PEC community would benefit from increased knowledge of mechanisms of degradation in the systems we study.

Action Items

- Comparison of stability from 3-electrode and 2-electrode experiments, at various operating points. This could include some modeling or providing "standard" operating characteristics to allow estimates of 2-electrode results from 3-electrode tests (CX and Shu) but also includes gathering and comparing actual data (James and Todd, as per ad hoc PEC session)
- Review and collect knowledge from corrosion field and see where that maps onto PEC stability. Progress talk at Spring MRS, and a review/perspective thereafter (Kimberly)

Summary of Discussion

- Should we standardize the electrolyte for PEC testing?
  - Suggest 3 electrolytes:
    - 0.5 M H2SO4
    - Phosphate vs. borate buffer
    - 1M KOH
  - Is it a system?
- What characterization should we use benchmark electrolyte?
- Discussed solid electrolytes as well

Consensus or Dissenting Opinions

- Need to worry foremost about safety and cost
- Ensure that electrolyte is not sacrificial
- Transport properties beyond conductivity could be important
  - Water transport, bubble management, gas solubility/permeation

Key Take-Aways

- Electrolyte choice should not be restrictive
- There could be effects due to spectator counterion
- Note that pH should be measured
- Local conditions are critical so stability by soaking is not enough, need to test in operating cell where pH gradients can form

Action items

- ***Suggest*** possible acid, neutral and alkaline electrolytes to use
  - Includes purity assessment
  - Understand interactions with light
  - Interaction with other components including both chassis and photoelectrodes

P4-A Prototype formats and key metrics for benchmarking — James Young

Summary of Discussion

- List metrics for benchmarking
- What makes a prototype?
- Feedback on current NREL photoreactor platform

Consensus or Dissenting Opinions

- Prototype – what makes a prototype?
- All necessary components present (e.g also membrane)
- Unit cell that is designed with ability to be scaled or tiled
- Too early in the field for a true prototype, but a standard platform would be valuable
- Particulate systems out of scope of platform designs

Key Take-Aways

- Efficiency, durability (other sessions), cost (out of scope)
- Efficiency (focus efficiency for this break-out)
  - JV (two-electrode)
    - Area definition
  - IPCE
  - Faradaic efficiency
    - H2 and O2, crossover measurement
- Durability-efficiency link
  - Total H2 produced metric
- Component level benchmarking, example
  - e.g. Could work with LTE for membrane benchmarking

Action items

- Initial RR with LBNL as part of PEC Supernode
- Initial hands-on exposure testing by other interested groups, eventually NREL/LBNL provide benchmarking measurements

P4-B Protocol development on OER/HER activity benchmarking at intermediate/dynamic current density — Nemanja Danilovic

Summary of Discussion

- Motivation behind intermediate current density/diurnal
- Crosscut with LTE both ways
- No existing protocols for either

Consensus or Dissenting Opinions

- Planar versus "roughened electrodes, liquid vs solid electrolytes
  - Lots of potential issues with bubbles current distribution etc
- Living document/protocol that is updated

Key Take-Aways

- LTE protocols exist for <20mA/cm2 can adapt these
- Turnover frequency for LTE is much lower than in PEC, can decrease loading to stress as protocol

Action Items

- Perform RDE (LTE) loading study for activity and stability with nanoparticles, repeat with sputtered film. Create baseline
- Flooded GDE type measurement would allow 100mA/cm2

P5-A PEC Nodes capabilities and gaps assessment Tadashi Ogitsu

Summary of Discussion

- How could HydroGEN Nodes can be useful or be more useful for broader community, other than the seedling project.
- Nodes don't exist that should exist.
- Nodes functioning so far? how's the interaction between nodes and PIs ?

Consensus or Dissenting Opinions

- Lots nodes seems comprehensive, there might still be lack of capabilities, but need to prioritize the nodes to make sure that top 80% of the needed nodes are up and running and operational.
- If we can define "standard" operating characteristics for a counter/partner electrode and cell resistances etc, then we can relate 3-electrode to 2-electrode measurements.
- More outreach to others through videos, conferences, etc.
- How do we recognize ourselves as a people in HydroGEN: PIs, Node experts, affiliates or others for potential node users, get some access to data?

Key Take-Aways

- There is a mechanism for outsider PIs, CRADA agreement, etc, already exist, supposedly fast interaction with Nodes. But needs improve kinetics of the process, to increase the awareness of this.
- People recognize that lots of barriers exists for this type of engagement: Researchers from NSF wont be sending money to labs
- post engagement was good, Science has been benefited greatly through interaction with nodes and others. (funding mechanism, bit strange process before FOA.)

Action Items

- A workshop for addressing Node usage, LTE&HTE joint session and a reception for other researcher to engage.
- Go beyond the current role for Nodes, which is a unique instrument combined with people with expertise. Some sort of consulting type of roles for nodes experts to interact with researchers, sorta like holding office hours once a month, or publish a webinar, or some tutorial videos for common question and issues.

- Literature review and synthesis on corrosion science and applicability to PEC (Kimberly Papadantonakis, talk scheduled at MRS Spring 2019)
- A working group and a mini review on PEC in situ/operando analysis (Shu Hu, Tony Van Buuren, Walter Drisdell, Tadashi Ogitsu, CX Xiang, talk scheduled at MRS Spring 2019)
- A mini review/view points on PEC 2-electrode vs. 3-electrode (Todd Deutsch, talk scheduled at MRS Spring 2019)
- NREL+NREL round robin testing PEC prototypes (James Young, Nemanja Danilovic, talk scheduled at MRS Spring 2019)