Benchmarking Advanced Water Splitting Technologies: Best Practices in Materials Characterization

Katherine Ayers (Primary Contact), George Roberts
Proton OnSite
10 Technology Drive
Wallingford, CT 06492
Phone: (203)-678-2190
Email: KAyers@protononsite.com

DOE Manager: Katie Randolph
Phone: (720) 356-1759
Email: Katie.Randolph@ee.doe.gov

Contract Number: DE-EE0008092

Subcontractors:

- Caltech-Joint Center for Artificial Photosynthesis, Pasadena, CA
- Pacific Northwest National Laboratory, Richland, WA
- Arizona State University, Tempe, AZ

Project Start Date: September 1, 2017
Project End Date: October 31, 2020

Overall Objectives

- Develop a framework of protocols/standards for testing performance of materials, components, devices, and systems.
- Facilitate acceptance of community-wide technology.
- Establish an annual project meeting to share learnings and develop recommendations within and across technology areas.
- Assess capabilities and identify gaps for development of advanced water splitting technologies.
- Promote acceptance of protocols and methodologies including cost and performance assessments and database comparisons.
- Assemble roadmaps to further development of each technology pathway.

Fiscal Year (FY) 2018 Objectives

- Hold a Year 1 workshop to present output of capabilities, assess gaps, and solicit input to define details of bench-scale protocol development based on initial framework.
- Prepare important questions and parameters for each technology area and surveys for dissemination.
- Complete and synthesize capabilities assessments including surveys of each node with 80% response rate.
- Complete and synthesize gap assessment including questionnaires with a goal of 50% response rate.
- Compile and publish workshop results and outcome report.

Technical Barriers

This project addresses the following technical barriers from the Systems Analysis section of the Fuel Cell Technologies Office Multi-Year Research, Development, and Demonstration (MYRDD) Plan[1]:

C. Inconsistent Data, Assumptions and Guidelines

D. Insufficient Suite of Models and Tools.

Technical Targets

This project is focused on developing standards and test protocols that result in technologies to produce hydrogen consistent with the following DOE technical targets:

- Support DOE Hydrogen and Fuel Cells Program goals to sustainably produce hydrogen for <$2/kg.

FY 2018 Accomplishments

- A framework and questionnaire were developed with input from node experts at

[1] https://www.energy.gov/eere/fuelcells/downloads/fuel-cell-technologies-office-multi-year-research-development-and-22

national labs for each technology in a common format.

- A questionnaire for each technology was distributed to the broad community for input and responses were collected.
- The framework was reviewed by questionnaire respondents that "opted in" to provide feedback.
- The assessment of node capabilities was completed and a single table to summarize capabilities and readiness was developed.
- Quarterly newsletters were sent out to the advanced water splitting technologies community.
- The fall community-wide meeting was held at Arizona State University on October 24–25, 2018.

INTRODUCTION

The high-level project goal is to create a comprehensive best-practices benchmarking framework at the materials, component, device, and systems levels for advanced water splitting technologies. All advanced water splitting pathways covered under the HydroGEN Energy Materials Network (EMN) Consortium, which include advanced high- and low-temperature electrolysis of water, photoelectrochemical water splitting, and solar thermochemical hydrogen, need these best practices to advance materials discovery. These practices will also aid the H2@Scale DOE initiative to accomplish their goals of large-scale hydrogen production.

The overall objective for this effort is to guide development of a water splitting roadmap across the different technologies, based on the varying maturity levels and challenges of each approach, to assist DOE in maintaining a balanced R&D portfolio. To support this objective, the team is working with the HydroGEN EMN Consortium and water splitting community to assess and document current best practices and material standards, assess existing capabilities and needs, and recommend next steps and priorities.

APPROACH

The overall project is divided into two major phases, Budget Period (BP) 1 and BP 2, across 3 years. In BP 1, the team will focus on development of the database framework and proposed bench-scale protocols to be introduced within the community. BP 2 will pursue validation/revision of the proposed protocol through user experience and development of a vision for the "subscale" category. Strategies for stakeholder engagement throughout the project will include annual cross-technology workshops as described below, as well as organization of conference symposia for specific technology areas for more focused, in-depth discussions and monthly virtual meetings per approach to gather stakeholder input and feedback.

RESULTS

HydroGEN Node Capabilities Assessment

A common table of node readiness levels was developed to identify capability gaps in the nodes with respect to the test frameworks developed for each water splitting technology. Updates were made to the HydroGEN advanced water splitting node capabilities website (https://www.h2awsm.org/capabilities) to incorporate agreed-upon changes to readiness levels from the assessment.

Each technology area principal investigator then assessed each of the test methods/protocols in their respective framework documents against the current node readiness levels. The goal of this exercise was to identify shortfalls in the node capabilities required to support the technology frameworks and make recommendations for augmenting the capabilities. Each test in the framework was reviewed and a relevant node was assigned where there was a clear match. In cases where gaps were identified, suggestions were made for labs that may be better suited for the testing (sometimes outside of the EMN). The details of this assessment were reviewed with DOE and an action plan will be developed to close the gaps.

Questionnaire

A questionnaire was created and distributed for each of the water splitting pathways. Distribution lists were developed to include EMN project leads, national lab node leads, and industry, academic, and international experts. The goal of this effort was to collect broad feedback across the water splitting community with a specific target of obtaining at least a 50% response rate from EMN Level 1 node leads and project principal investigators. These inputs are archived on the HydroGEN data hub (https://datahub.h2awsm.org/project/about/benchmarking). An example of feedback received is shown in Figure 1.

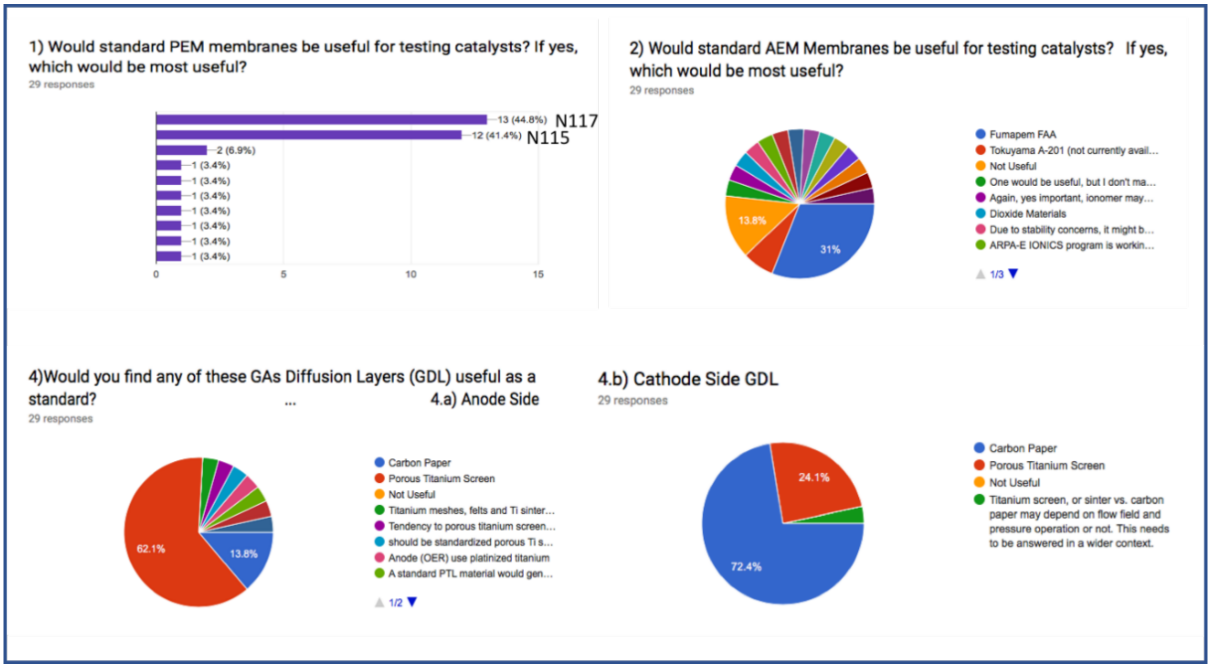

Figure 1. Example low-temperature electrolysis questionnaire results

Framework

A standardized framework was developed that could be applied across all water splitting technology pathways for developing comprehensive best practices of benchmarking methods. It was decided to focus on two primary categories: ex situ materials testing and in situ testing. The ex situ testing can be performed on individual materials using standard test methods and phased to allow for rapid evaluation. In situ testing targets the evaluation of materials in representative system operation and conditions for a given water spitting technology.

Standards for use in calibrating test equipment and test methods were identified along with minimum performance criteria for comparison based on inputs from subject matter experts and published literature. Individual frameworks have been developed for each technology and continue to be refined based on feedback from the questionnaire and workshop breakout sessions.

Workshop

A community-wide workshop was planned for October 24–25, 2018, at Arizona State University, Tempe campus.

Workshop objectives:

- Understand the needs of the community for effective comparison of results based on surveys
- Review and refine draft frameworks for standardized testing by component or configuration
- Hold face-to-face discussions about protocol development in breakout sessions
- Refine methods based on community engagement
- Leverage international efforts to increase harmony across the field
- Realize increased usage of database and Zotero capability for community benefit.

Representative intended outputs:

- Protocols Version 1.0
- Documentation on further needs/gaps in protocols and benchmarking in each technology pathway
- A perspective or viewpoint paper on crosscutting and/or overall advanced water splitting technology pathways.

The workshop comprised a plenary session and a series of breakout sessions (Figure 2) followed by a wrap up.

2018 HydroGEN Advanced Water Splitting Technology Pathways Benchmarking and Protocols Workshop - Breakout Sessions

Breakout Session #	Session ID	Technology	Topic
1	C1-A	PEC/LTE	Membrane operating at different regimes
1	C1-B	LTE, PEC	Theory on catalytic reactions with metal oxides and other materials
1	C1-C	PEC, LTE, HTE, STCH	Standards development and crosscutting measurement issues
1	S1-A	STCH	Performance Metrics - units, system boundaries
2	H2-A	HTE	Electrolyte: oxygen and proton conductors
2	L2-A	LTE	PEM: Membrane Physical Requirements/Tests
2	L2-B	LTE	Non-PGM Catalyst: OER Stability & Activity
2	P2-A	PEC	Protocol development in a half cell vs. a full cell
2	P2-B	PEC	In situ/operando methods for PEC interfaces and devices
2	S2-A	STCH	Standard materials and form factors
2	S2-B	STCH	Detailed thermodynamics - operating conditions and methodology
3	H3-A	HTE	Electrode Activity & Stability
3	L3-A	LTE	AEM: Membrane Physical Requirements/Tests
3	L3-B	LTE	PGM Catalyst: OER Stability & Activity
3	P3-A	PEC	Protocols for PEC stability testing
3	P3-B	PEC	PEC electrolytes
3	S3-A	STCH	"Quick and dirty" thermodynamic screen
3	S3-B	STCH	Extracting thermodynamic variables from theory and experiment
4	H4-A	HTE	Cell test protocols
4	L4-A	LTE	PTL: Characteristics & Characterization Tools
4	L4-B	LTE	MEA Device Level Protocols: Criteria/Tests
4	P4-A	PEC	Prototype formats and key metrics for benchmarking.
4	P4-B	PEC	Protocol development on OER/HER activity benchmarking at intermediate/dynamic current density
4	S4-A	STCH	Detailed kinetic screening
4	S4-B	STCH	Systems analysis and TEA
5	H5-A	HTE	In situ methods for degradation studies
5	L5-A	LTE	Carbon GDL: Physical Requirements/Tests
5	L5-B	LTE	Full Stack Level Protocols: Criteria/Tests
5	P5-A	PEC	PEC Nodes capabilities and gaps assessment.
5	S5-A	STCH	"Quick and dirty" kinetics screening
5	S5-B	STCH	Durability protocols
6	H6-A	HTE	Full stack test protocols
6	C6-A	PEC/STCH, LTE/HTE	Comparative analysis on key cross-cutting metrics (definition and discussion of device efficiency, cost of hydrogen, etc)
6	L6-A	LTE	HOLD FOR AD-HOC SESSIONS
6	P6-A	PEC	HOLD FOR AD-HOC SESSIONS
6	S6-A	STCH	HOLD FOR AD-HOC SESSIONS

Figure 2. HydroGEN advanced water splitting workshop breakout sessions

The breakout sessions encompassed the majority of the workshop and were intended to dig deeper into each section of the testing frameworks for each water splitting technology. Specific emphasis was placed on establishing test methods, standards, and performance criteria. A session leader was identified for each session who was responsible for leading the discussion and summarizing the outputs. Following the workshop, the outputs from each of the sessions are being integrated into their respective testing frameworks and will serve as the basis for test method development.

CONCLUSIONS AND UPCOMING ACTIVITIES

Upcoming activity will focus on integrating all the outputs of the workshop breakout sessions into a revised test framework. Each test will be reviewed, and assignments will be made to various stakeholders to write detailed test methods. Test protocols will be developed and validated and will include accelerated testing and the definition of degradation mechanisms. A strategy for closing the gaps in node capabilities will be developed.

There are no issues expected to affect project progress at this time.

FY 2018 PUBLICATIONS/PRESENTATIONS

1. E.B. Stechel, "HydroGEN: Advanced Water Splitting Materials," Invited presentation at the International Workshop on Solar Thermochemistry, Julich, Germany, September 12–14, 2017.
2. HydroGEN AWSM Benchmarking Meeting (PEC Working Group Meeting), organized by CX Xiang, T. Deutsch, T. Ogitsu and H. Dinh, Seattle, WA, May 13, 2018.
3. J. Holladay (Pacific Northwest National Laboratory), B.S. Pivovar (National Renewable Energy Laboratory), K.E. Ayers (Proton OnSite), O.A. Marina (Pacific Northwest National Laboratory), E.B. Stechel (ASU-LightWorks), and C. Xiang (California Institute of Technology), "An Overview of H2@Scale and Water Splitting Protocol Development," Invited, 233rd ECS, Seattle, WA, May 14, 2018.
4. K.E. Ayers (Proton OnSite), "Low Temperature Electrolysis for Hydrogen and Oxygen Generation—A Tutorial on Catalyst and Electrode Development for Proton and Anion Exchange Membrane-Based Systems," Invited, 233rd ECS, Seattle, WA, May 14, 2018.
5. HydroGEN LTE/HTE Benchmarking Discussion, organized by K. E. Ayers, H. Dinh, and N. Danilovic, 233rd ECS, Seattle, WA, May 14, 2018.
6. Chengxiang ("CX") Xiang, "Development of Best Practices and Standard Protocols in Benchmarking Photoelectrochemical (PEC) Hydrogen Production," 233rd ECS, Seattle, WA, May 17, 2018.
7. K.E. Ayers, "Benchmarking Advanced Water Splitting Technologies," U.S. Department of Energy's Hydrogen and Fuel Cells Program 2018 Annual Merit Review and Peer Evaluation Meeting, Washington, DC, June 13, 2018.
8. K.E. Ayers, C. Capuano, and P. Mani, "High Efficiency PEM Electrolysis: Potential for H2@Scale," 234th ECS, Cancun, Mexico, October 2, 2018.