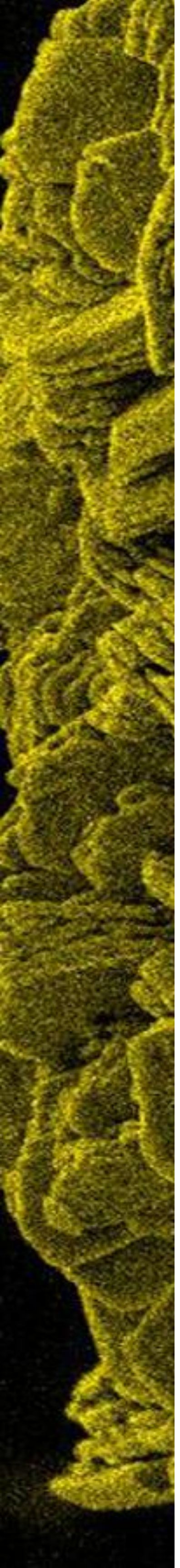


DOE Hydrogen Program
2022 Annual Merit Review and Peer Evaluation Meeting

Benchmarking Advanced Water Splitting Technologies: Best Practices in Materials Characterization

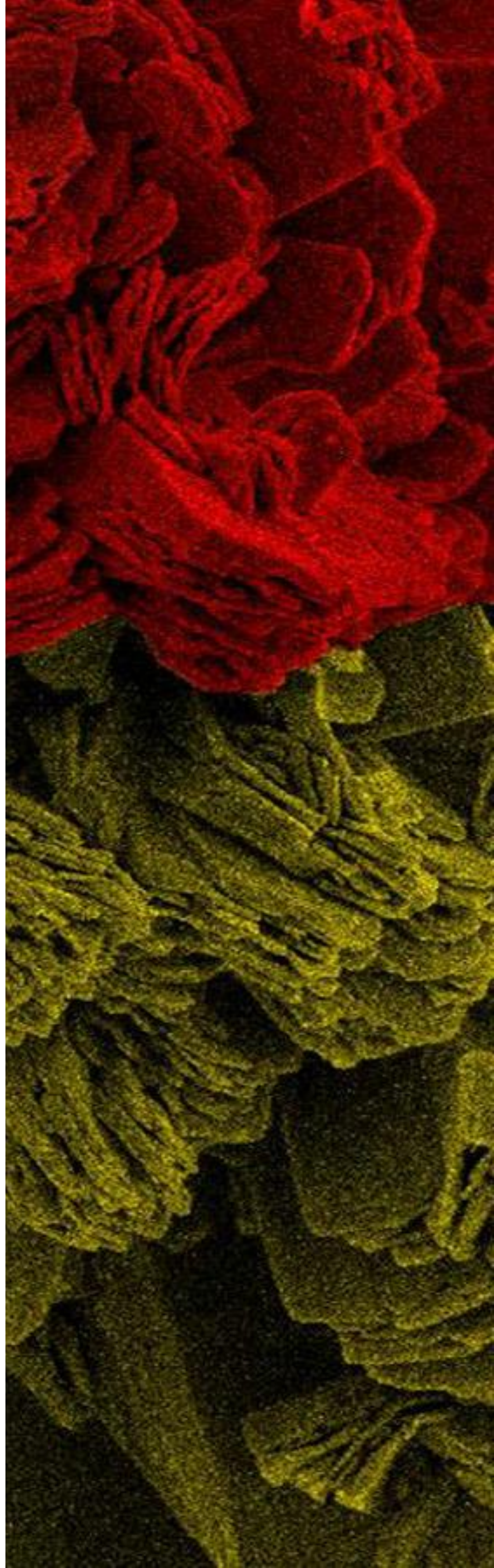
Olga A. Marina[1], Kathy Ayers[2], Ellen Stechel[3], CX Xiang[4], Karl Gross[5]

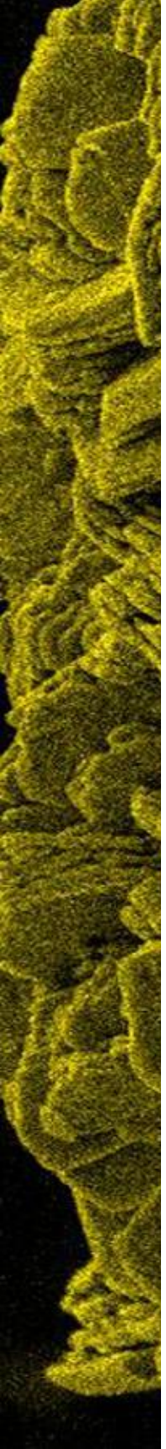
[1] Pacific Northwest National Laboratory; [2] Nel, [3] ASU, [4] Caltech, [5] H2tech

WBS 2.3.0.708
June 6–8, 2022

Project ID # p170

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





Project Goals

- Create a balanced portfolio of standards and protocols for Advanced Water Splitting Materials testing, from screening of fundamental material characteristics, to device or system level benchmarking in controlled and real-world operating conditions
- Engage the community and create more awareness and dialogue between the technology areas and for better leveraging of technical advancements and understanding
- Work on protocol development for bench-scale, sub-scale and higher levels, and on protocol verification and validation
- Develop a Round Robin testing verification plan.
- Work with H2NEW Consortia to assist in accelerated aging protocol development

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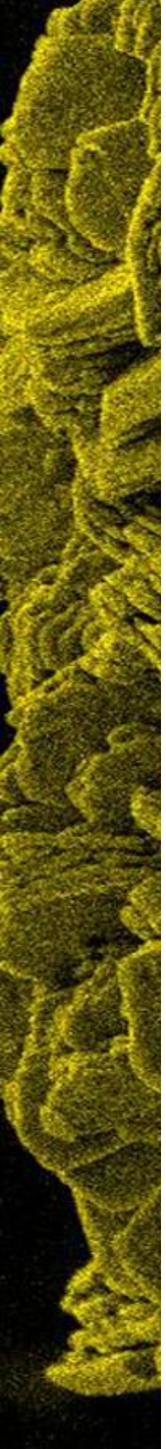


Overview

Timeline

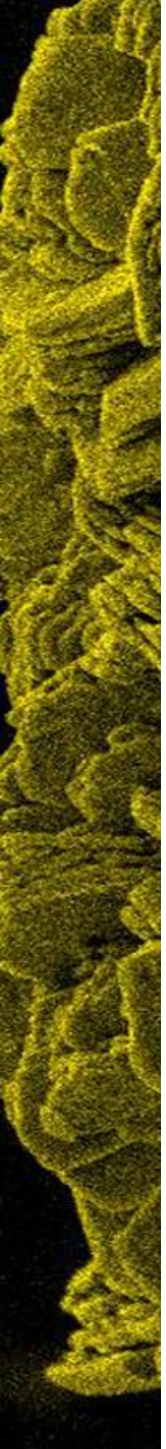
- Project Start Date: 10/20/2021
- Project End Date: 9/30/2022

Budget

- FY22 Planned DOE Funding: $1M
- Total DOE Funds Received to Date: $1M

Project Partners

- Pacific Northwest National Laboratory, Project Lead
- Nel
- Arizona State University
- Caltech
- H2TechConsulting



Innovation

- Develop a framework of protocols/standards for testing performance of materials, components, devices, and systems
- Facilitate community-wide acceptance of protocol and benchmarking
- Establish an annual International Workshop to; share learnings, stimulate participation, and develop recommendations within/across technology areas
- Assess capabilities and identify gaps for development of advanced water splitting technologies
- Develop and implement a plan for Protocol Validation
- Promote acceptance of protocols and methodologies including cost and performance assessments and database comparisons
- Assemble roadmaps to further development of each technology pathway

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Relevance and Impact

Standardized Test Methods and Benchmarks

- Decrease development cycle times through common comparison
- Allow for direct comparisons of materials and water splitting technologies
- Revisions to draft test protocols with feedback from EMN and International experts
- Released finalized version of protocols and prioritized plans for validation
- Initiation of new protocol drafts

Community Engagement and Outreach

- Annual workshop with international participation
- Publications of protocols in open journals and on the NREL Hydrogen-AWSM openpoint website.
- Presentations at scientific conferences

Test Protocol Table of Contents

1.Procedures
- a. Scope and Applicability
- b. Summary of Method
- c. Definitions
- d. Health & Safety Warning
- e. Cautions
- f. Interferences
- g. Personnel Qualifications / Responsibilities
- h. Equipment and Supplies
- i. Step by Step Procedure
  - Instrument or Method Calibration and Standardization
  - Sample Collection
  - Sample Handling and Preservation
  - Sample Preparation and Analysis
  - Troubleshooting
  - Data Acquisition, Calculations & Data Reduction Requirements
  - Computer Hardware & Software
- j. Data and Records Management

2.Quality Control and Quality Assurance Section

3.General Notes

4.Reference Section

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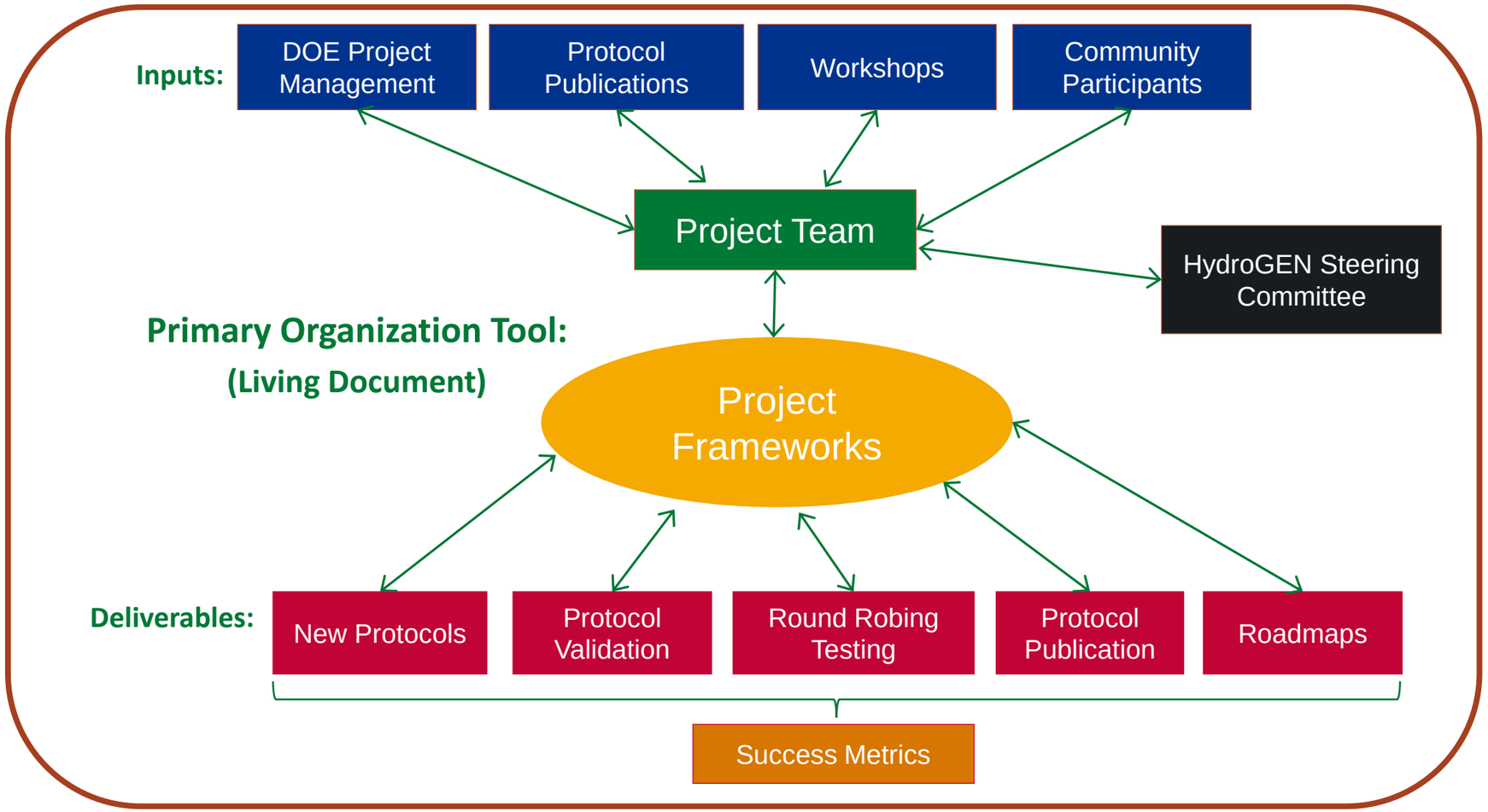
Approach



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Budget Period Project Tasks

Task	Timing	Goal
1.0 Subcontracts	February	Finalized 4 PI subcontracts: Nel, ASU, Caltech, and H2TechConsultling
2.0 2022 Workshop	May	Completed with over 120 participants.
3.1 Bench Scale Protocols	January – October	Continue developing new-next level bench/sub-scale testing protocols for each water splitting pathway
3.2 Next Generation Protocols	January – October	Ensure protocols and Best Practices are created to meet higher levels of device development including accelerated ageing test protocols.
4.1 Protocol Verification & Validation	January – October	Apply protocols within the community to validate the approach and incorporate changes needed in protocols. This will include round-robin testing and identification of lessons learned.
4.2 Protocol Modification	January – October	Based on Validation results, updates to protocols will be made and published in an accessible forum.
5.0 Program Management	January – October	The team will manage the technical work within the program schedule, budget, and technical scope and will prepare and submit interim and final reports.

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Accomplishments and Progress

Conducted 2022 Workshop to Engage Technology Experts

- Updated the distribution list
- Distributed Save the date fliers
- Assembled agenda, breakout sessions, invited plenary speakers
- Community engagement in
- Strategy and plans on protocol validation and Round Robin testing
- Monitor synergistic international efforts in harmonization of protocols
- Identify new protocols to be written and revisions

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Accomplishments and Progress

12 Protocols Submitted for Publication in Frontiers in Energy

Test Protocol Development Process

- Materials level test protocols were prioritized
- Future protocols will include device level and accelerated testing

Thank you to our test protocol contributors and expert reviewers!

Thanks for workshop input from many national and international researchers!

Authors:

LTE	HTE	PEC	STCH
Shaun Alia	John Hardy	James Young	Andrea Ambrosini
Chulsung Bae	Jeff Stevenson	Todd Deutsch	Bob Bell
Chris Capuano	Yeong-Shyung Chou	Adam Weber	Eric Coker
Nem Danilovic	Fengyu Shen	Nem Danilovic	Dave Ginley
Kelly Meeks	Dong Ding	Charles Dismukes	Chris Muhich
Sarah Park	Mike Tucker	Shu Hu	Anthony McDaniel
Alexey Serov	Hanping Dong	Burt Simpson	Michael Sanders
Hui Xu	Ani Kulkarni	Jason Cooper	Jonathan Scheffe
		Dave Palm	

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Accomplishments and Progress

36 Test Protocols for Review and Validation

- **36 test protocols were drafted, reviewed and prioritized for validation**
- **40 additional protocols in drafting process**

LTE:

ID #	Protocol	Component	Status
PEC-P-1	Photoelectrode fabrication and area measurement protocol	component (photoelectrode)	completed
PEC-P-2	Illumination calibration prptocol	Device	completed
PEC-P-3	IPCE measurement protocol	component (photoelectrode)	completed
PEC-P-5	Product crossover measurement protocol	component (transport)	completed
PEC-P-7	Band energetics measurement protocol	Materials (protection layer)	completed
PEC-P-8	Through-Plan membrane conductivity measurement protocol	component (transport)	completed
PEC-P-9	On sun testing protocols	Device	completed
PEC-P-10	Fabrication and integration and scale up protocol	component (Auxiliary)	completed
PEC-P-11	Photoelectrodes stability testing protocols	Component (photoelectrode)	completed
PEC-P-12	Band-gap and light absorption measurement protocols	Materials (photoabsorber)	completed
PEC-P-13	OER and HER activity protocols for water splitting.	Materials (catalyst)	completed
PEC-P-14	Minority carrier diffusion length measurement protocols	Materials (photoabsorber)	completed
PEC-P-15	pH imaging/sensing protocols	Component (photoelectrode)	replaced by a review paper pending submission
PEC-P-16	Check list documents for beyond 1000h testings	Device	completed
PEC-P-17	Flat band potential measurements/protocols	Materials (photoabsorber)	completed
PEC-P-18	Spatially resolved PEC SECM protocol	component (photoelectrode)	completed
PEC-P-19	Protective layer conductivity measurement protocol	Materials (protection layer)	completed
PEC-P-20	Protective layer optical measurement protocol	Materials (protection layer)	completed
PEC-P-21	Doping type and doping density measurement protocol	Materials (photoabsorber)	completed

HTE:

ID #	Protocol	Component	Status
LTE-P-1	Compressibility of GDLs for Water Electrolyzers	GDL	Complete
LTE-P-3	PEM Ion Exchange Capacity	PEM	Complete
LTE-P-5	PEM Thermal Stability	PEM	Complete
LTE-P-6	AEM Conductivity	AEM	Complete
LTE-P-7	AEM Ion Exchange Capacity	AEM	Complete
LTE-P-8	Gas Permeability	AEM/PEM	Complete
LTE-P-10	Rotating Disk Electrode	PGM	Complete
LTE-P-13	3-Electrode Cell for Screening OER/HER Electrocatalysts Activity	Non-PGM	Complete
LTE-P-14	Electrical Conductivity of PGM-free OER Catalysts	Non-PGM	Complete
LTE-P-17	Resistance measurements and water properties	PTL	Complete
LTE-P-19	Comparison Metrics and Terms for LTE	General	Complete
LTE-P-20	Water Uptake Measurement	PEM/AEM	Complete
LTE-P-21	Develop standard post mortem analysis	General	in review
LTE-P-22	Alkaline Stabililty of AEM	AEM	Complete
LTE-P-23	AEM Oxidation resistance protocol	AEM	Complete
LTE-P-24	Sub-Scale Test Protocol	PEM	Draft complete. On Hold- pending round robin test results
LTE-P-25	AEM Creep rate protocol	AEM	Hold

PEC:

ID #	Protocol	Component	Status
HTE-P-01	Measurement of Bulk Conductivity	Electrolyte/Electrode	Complete
HTE-P-02	Ion Conductivity/Transference Numbers	Electrolyte	Complete
HTE-P-03	Mixed Ion Conductivity	Electrolyte	Moved to FY22; will be combined with P-02
HTE-P-04	Density Measurement	Electrolyte	Complete
HTE-P-05	Linear Thermal Expansion	Electrolyte	Complete
HTE-P-07	Leak Test	Cell/Stack	Complete
HTE-P-08	Cell Conditioning Protocol	Cell/Stack	Moved to FY22
HTE-P-09	Button Cell Performance	Cell	Complete
HTE-P-10	Polarization Resistance	Electrode	Complete
HTE-P-11	Impedance Spectroscopy Test	Electrode	Complete
HTE-P-12	HTE Definitions and Notations	General	Moved to FY22
HTE-P-13	Metal-Supported Cell Test	Cell	Complete
HTE-P-14	Bonding Strength	Contact Layer	Complete
HTE-P-15	Mechanical Strength	Electrolyte	Complete
HTE-P-16	Seal Stability in Dual Environment	Cell	Not written
HTE-P-17	Steam Content Measurement	Cell	Not written
HTE-P-18	Interconnect Resistance Measurements	Interconnect/Coatings	Complete
HTE-P-19	Area Specific Resistance	Cell/Stack	Complete
HTE-P-21	Cell Thermal Cycling	Cell	Complete
HTE-P-22	Measurement of Faradaic Efficiency	Cell	Complete
HTE-P-23	SOEC Stack Testing	Stack	Complete
HTE-P-24	Cell Durability Testing	Cell	Complete
HTE-P-25	Large Area Cell Testing	Cell	Complete
HTE-P-27	H2 Production Rate and Electronic Leakage	Cell	Complete

STCH:

ID #	Protocol	Status
STCH-P-1	Metrics, Units, Definitions	In progress
STCH-P-2	Ceria Standard and Material Specs	In progress
STCH-P3	ABO3 Standard and Material Specs	Dropped
STCH-P4	Detailed Thermodynamic Screen	Submitted and second one in progress
STCH-P5	Hydrogen spike Thermodynamic Screen	TBD
STCH-P6	Extracting the Thermodynamics Measurables (ΔH and ΔS) from the measurements	In progress
STCH-P8	Detailed Kinetic Screen	In progress
STCH-P10	Durability Level 1 Screen	In progress
STCH-P11	Durability Level 2 Screen	Delayed
STCH-P12	Durability Level 3 Screen	Delayed
STCH-P13	DFT Best Practices - Lessons learned	TBD
STCH-P16	Operating Limits	TBD
STCH-P17	Spectroscopic Tools	Target Complete by 1/31/2021

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Collaboration and Coordination

- Wide-ranging and collaborative effort within and beyond the Advanced Water Splitting Community.
  - LTE, HTE, STCH, and PEC technologies
  - Significant engagement from universities, national labs, and international subject matter experts
  - Feedback has been positive and enthusiastic throughout
- Workshops, newsletters, symposia, and now publications used to spread information and solicit input
- Continue engaging with world experts and new project teams as they participate in creating new protocols and validating current protocols

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Milestones

Milestone Name/Description	Criteria
Publish Protocols	Publish at least 2 full articles per each of 4 AWS technologies, each containing 1-4 material testing protocols. Engage technology experts to prepare and assemble protocols into manuscripts for publishing in Frontiers in Energy. Initiate manuscript submission into a journal issue. Identify reviewers. Assist with fee coverage, when needed.
Finalize subcontracts	Finalize 4 sub-contracts: Nel, ASU, Caltech, and h2techconsulting.
Hold 4th Annual Workshop	Complete 4th annual Workshop in one of the three formats: in-person, hybrid, virtual participation. Secure 4 guest speakers from parallel international HTE, LTE, STCH, PEC programs.
Complete Annual Report	Annual technical report will establish criteria for sub-scale test hardware and facility requirements for testing protocols; assess the existing expert sites within DOE and international programs; and identify at least 2 potential validation centers per technology.

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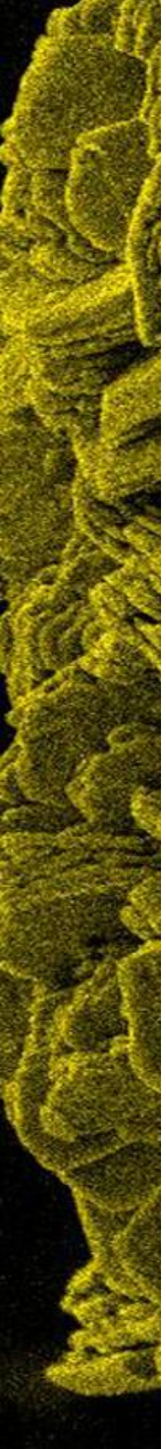
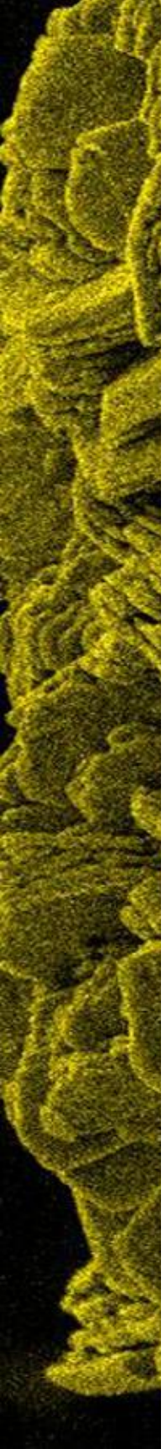


Publications and Presentations

- OA Marina, High Temperature Electrolysis: Challenges and Opportunities of H_2 Production, *Workshop: Where Is Energy Storage Headed? Challenges of Degradation in Long-term Operation, Boston University, February 11, 2022.*
- K Ayers, Challenges and Opportunities for Large Scale PEM Electrolysis, Invited lecture, *The H2 Economy Program*, University of Houston, March 7, 2022.
- OA Marina, High Temperature Electrolyzers for Hydrogen and Chemicals Production, Invited lecture, *The H2 Economy Program*, University of Houston, March 7, 2022.
- OA Marina, Current Status and Future Focus of SOEC Manufacturing, Plenary presentation at the *DOE SOEC Manufacturing Workshop*, March 8, 2022.



Remaining Challenges and Barriers

- Validation sites are likely to require financial support to cover the costs and labor

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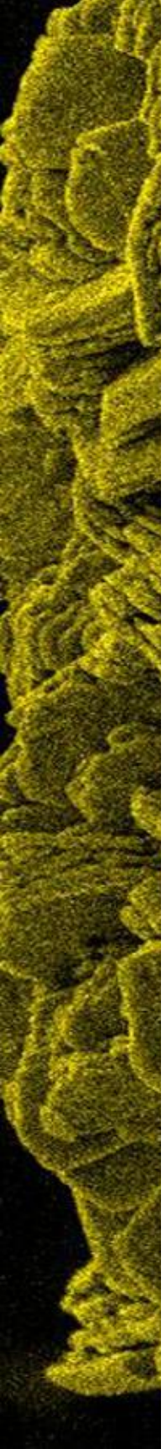

Proposed Future Work

- Continue engaging technology experts to create new high-priority protocols and prepare manuscripts for publishing in Frontiers in Energy
- Initiate manuscript submission into a journal issue; Identify reviewers; Assist with fee coverage, when needed
- Prepare technical report to establish criteria for sub-scale test hardware and facility requirements for testing protocols
- Continue engaging the community in identifying and writing advanced testing protocols for bench-scale, sub-scale and higher levels
- Assess the existing expert sites within DOE and international programs and identify at least 2 potential validation centers per technology
- Identify community accepted standards for round-robin/validation testing and potentially a curator for these standards

Any proposed future work is subject to change based on funding levels.

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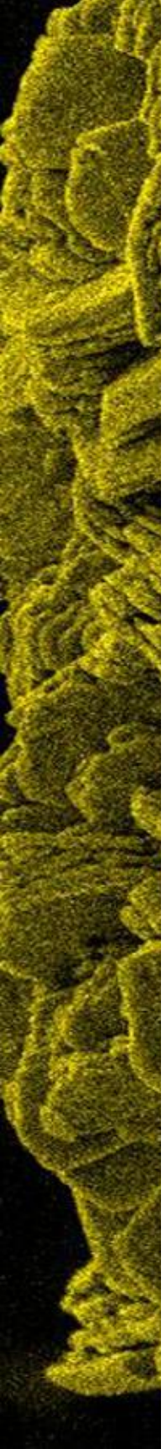

Response To Reviewer Comments

- This project was not reviewed last year

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