HydroGEN
Advanced Water Splitting Materials

Advanced Water-Splitting Technology Pathways Benchmarking & Protocols Workshop

Breakout Session Summaries
High Temperature Electrolysis (HTE)

October 29 - 30, 2019

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Executive Summary

The HTE breakout session discussed cell components, testing approaches and the need to move towards device testing in order to better understand materials performance and degradation under realistic operating conditions and realistic steam utilizations. There was strong agreement that scaling cell size up should become a priority. For solid oxide electrolyzers, performance maps and TEA mapping and operating boundaries were discussed, and a strategy for choosing operating parameters was suggested. The consensus was that proton conductors are at a very early stage with many unknowns; for proton conducting cells the faradaic efficiency must always be measured and reported. For protocol development, the focus would remain both on materials characterization and button and large cell testing. In addition, protocols on durability testing, cycling testing, thermal and mechanical stress testing are urgently needed. Accelerated stress testing was agreed to be important, but would require a high throughput testing system and a good understanding of each degradation mechanism.

HTE Breakout Sessions

Breakout Session #	Session ID	Technology	Topic	Lead
1	H1-A	HTE	HTE Technology Roadmap Review & Discussion - Materials, Components	Olga Marina
1	H1-B	HTE	HTE Technology Roadmap Review & Discussion - Devices, Testing	Mark Williams
2	H2-A	HTE	Best Methods and Practices for Characterizing SOEC Materials	Joseph Barton
2	H2-B	HTE	Best Methods and Practices for Characterizing H^+-SOEC Materials	Dong Ding
4	H4-A	HTE	Cell Performance Measurements: Standards, Calibrations, Protocols, Validation	Jim O'Brien
4	H4-B	HTE	SOEC and H^+-SOEC Operating Conditions and Boundaries	Joseph Hartvigsen
5	H5-A	HTE	Materials and Device Lifetime Testing Protocols	Xingbo Liu
5	H5-B	HTE	Terminology and Units	Neal Sullivan
6	H6-A	HTE	Wrap-up/Action Item Assignment: HTE Materials, Components	Ani Kulkarni
6	H6-B	HTE	Wrap-up/Action Item Assignment: HTE Cells, Stacks	Jamie Holladay

Session Summary

Session ID: H1-A

Title: Roadmap – materials, components

Summary of discussion

- Economic viability
- Impact of stack life
- What cell design is better
- Where are main degradation
  - Materials: O2-conductors - need to understand materials performance better and what is the main degradation; H+ conductors - very early stage with many unknowns and no standard available.
  - Cells: need realistic values of steam utilization and operating conditions; need standards for large single cell test
  - Benefit of high pressure (up to 10bar)

Consensus:

- Testing large cells would allow meaningful comparisons between cell test results from different groups
- Need to identify sets of operating conditions for standardized testing
- Scale up to actual stack conditions is highly needed

Key Take-Aways

- Need to scale up testing from button cells to larger cells to operate at realistic conditions and realistic values of steam utilizations
- Need standards for large single cell test
- Pressurized conditions should be tested (up to 10 bar) because of system advantages, but challenges arise

Action Items

- Review and revise existing protocols – assignments made
- Identify a manufacturer of standard cells
- Assess where to get standards for single cell testing including contacting, sealing, etc.

Session Attendee List

Session ID: H1-A

Title: HTE Roadmap: Materials, Components

Name	Affiliation
Ani Kulkarni	CSIRO
Olga Marina	PNNL
Scott Barnett	Northwestern
Dong Ding	INL
Xingbo Liu	WVU
Neal Sullivan	CSM
Gary Groenewold	INL
Hanping Ding	INL
James O’Brien	INL
David Ginley	NREL

Session Summary

Session ID: H1-B

Title: Roadmap – cells, stacks

Summary of discussion

- Discussed TRL's and adjusted them
- Cost goals
- Performance goals
- Durability goals
- Manufacturability goals
- SOEC – intermittent operation so control issues
  - R&D area: what happens when go from FC to EC mode
- Economics- want high steam utilization so reversibility is desirable.

Consensus and/or dissenting opinions

- Consensus: AST is highly desirable as it will take many tests to develop and validate protocols. Long term and short term tests – (dozens)
- Dissent on value of SOEC/SOFC reversibility
  - Does increase utilization
  - Do we really want to put the H2 back to electrons back to GRID

Key Take-Aways

- Need to define scale up for both cell size and manufacturing
- Thermal management for stacks is a gap
- Steps being taken to improve quality of materials: processing of the materials is helping to deal with delamination, nickel migration, chromium contamination
- AST: depend on degradation mechanisms which are still being defined

Action Items

- Add $/kg as a metric
- Need to define scale up for both cell size and manufacturing
- Stacks: thermal management, changing operating mode

Session Attendee List

Session ID: H1-B

Title: HTE Roadmap: Devices, Testing

Name	Affiliation
Mark Williams	Key Logic
Nguyen Minh	University of California San Diego (UCSD)
Kevin Huang	University of South Carolina
Joseph Barton	FuelCell Energy
Thomas Malkow	JRC Petten
Joseph Hartvigsen	OxEon Energy
Scott Swartz	Nexceris
David Peterson	DOE
Richard Boardman	INL
Jamie Holladay	PNNL

Session Summary

Session ID: H2-A

Title: Best methods characterizing SOEC

Summary of discussion

- Reviewed practices/protocols and discussed importance
  - Ionic conductivity and transference number discussions
  - Electrode size, particle size, surface area, how materials were processed - important to be specified
  - Should characterize specific electrodes
  - Button cell testing is useful?
  - Validation standardization
  - Specific resistance unit and how to report degradation, i.e., mV/kh

Consensus and/or dissenting opinions

- Protocols needed on
  - Steam content quantification
  - Steam sensing techniques: water vaporization, H2 burners
  - Seals for H+ electrolytes
  - Button cell vs scaled up cell vs 2-cell stack
  - Leakage rate

Key Take-Aways

- Characterizing electrodes requires reporting starting powder as well as processing route
- Accurate steam measurement on inlet and outlet is vital for electrolysis; need to report utilizations
- Two protocols needed for cell testing:
  - Button cell testing important for QA/QC as well as material development
  - 5x5 cell testing required for system/degradation studies and separate protocols needed for both

Action Items

- ASR units in document was not correct.
- 5x5 cm testing protocol (J. Barton)
- Button cell testing protocol (S. Schwartz)
- Could be standard cells for testing be purchased from Fuel Cell Materials (S. Schwartz)

Session Attendee List

Session ID: H2-A

Title: Best Methods and Practices for Characterizing O-SOEC Materials

Name	Affiliation
Thomas Malkow	JRC Petten
Joseph Hartvigsen	OxEon Energy
Scott Swartz	Nexceris
Amin Nouri	Greenlight Innovation
Robert Bell	NREL
Olga Marina	PNNL
Kevin Huang	Un. of South Carolina
Mark Williams	Key Logic
Nguyen Minh	Un. of California, San Diego
Ani Kulkarni	CSIRO
Joseph Barton	FuelCell Energy

Session Summary

Session ID: H2-B
Title: Best Methods and Practices for Characterizing H+-SOEC Materials

Summary of discussion

- Faradaic efficiency measurement of electrolyte materials
- Transference number measurement metrics: effect of driving voltage, operating temperature, gas compositions
- Full cells necessary to apply real bias for faradaic efficiency (> $5 cm^2$)

Consensus and/or dissenting opinions

- Agree that Faradaic efficiency is important
- Dissenting opinions on which factors are dominating Faradaic efficiency: pO_2 vs pH_2O

Key Take-Aways

- Transference number measurement protocol is valuable; add T, pO_2
- Protocols should be reviewed by experts in the field

Action Items

- Complete the Faradaic efficiency measurement protocol (UTRC/INL, H. Ding)
- Add some metrics into exiting transference number measurement protocol (HTE-P-02): O_2 and H_2 partial pressures, temperature, gas compositions (PNNL)

Session Attendee List

Session ID: H2-B
Title: Best Methods and Practices for Characterizing H+-SOEC Materials

Name	Affiliation
Dong Ding	INL
Xingbo Liu	University of West Virginia
Neal Sullivan	Colorado School of Mines
Harping Ding	INL
Richard Boardman	INL
Jim O'Brien	INL
Scott Swartz	Nexceris
Gary Groenewold	INL
Jamie Holladay	PNNL
Scott Barnett	Northwestern

Session Summary

Session ID: H4-A

Title: Cell Performance Measurements: Standards, Calibrations, Protocols, Validation

Summary of discussion

- Review of testing metrics and existing protocols
- Cell, stack, coatings, seals, contact layers and interconnect specifications
- Testing methods/protocols depend on the scale:
  - Button cell testing yields relatively idealized performance
  - Single cell testing represents stack conditions
- Detailed diagnostics (EIS) are appropriate at the button cell or single cell scale, but not useful at the stack level
- Focus on stack testing

Consensus and/or dissenting opinions

- Consensus on need for more stack-level testing and scale-up
- Include stack operating conditions such as current density, per-cell voltage, steam utilization, constant current or constant voltage operation, hydrogen inlet flow rate, hydrogen recycle, and heat recuperation

Key Take-Aways

- Stack testing introduces numerous additional complications beyond cell testing
- Stacks often require mechanical compression.
- Scale-up to 10's or 100's of kW should be supported for technology development
- Intermittent operation should also be considered

Action Items

- Need to develop protocols for stacks testing
  - Cell reduction procedure
  - Mechanical compression (for cell-interconnect contact and seals) heatup profiles
  - Operating conditions
  - Long-term testing
  - Instrumentation
  - Safety

Session Attendee List

Session ID: H4-A

Title: Cell Performance Measurements: Standards, Calibrations, Protocols, Validation

Name	Affiliation
Jim O'Brien	INL
Scott Barnett	Northwestern
Kevin Huang	University of South Carolina
Dong Ding	INL
Xingbo Liu	University of West Virginia
Kevin Huang	University of South Carolina
Scott Swartz	Nexceris
Harping Ding	INL
Mark Williams	Key Logic

Session Summary

Summary of discussion

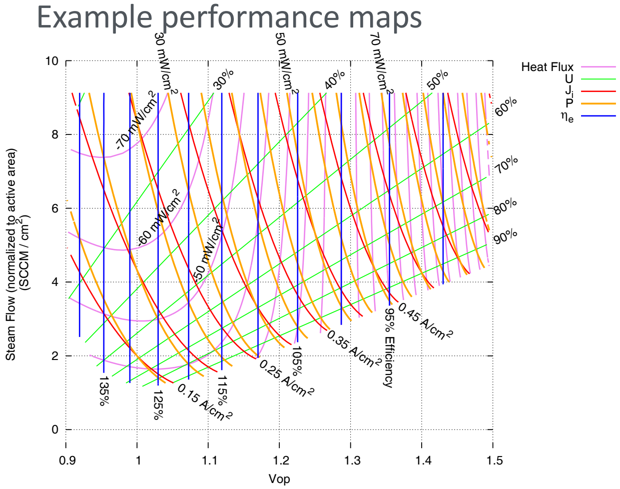
- Simple hydrogen production rate
  - I-V, composition dependence, temperature dependence, cycle dependence, time on stream dependence
- Strategy for choosing operating and boundaries parameters
- Methodologies to consolidate functionality:
  - Performance mapping, degradation mapping, TEA mapping
  - Projected stack lifetime and operating cost

Consensus and/or dissenting opinions

- Efficiency: use multiple metrics
  - η as %HHV, %LHV, kW-h/kg, kg/MWh, or operating voltage
- Need to measure H_2 production rate
- System integration matters

Key Take-Aways

- Target steam utilization is 50%
- Example performance maps



Action Items

- Protocol on defining ASR (J. Hartvigsen)
- Map operating conditions (N. Minh)
- Operating conditions/boundaries protocol (J. Barton)
- Pressurized operation (J. Hartvigsen)
- Thermocycling protocol

Session Attendee List

Session ID: H4-B
Title: SOEC and H+-SOEC Operating Conditions and Boundaries

Name	Affiliation
Olga Marina	PNNL
Neal Sullivan	CSM
Gary Groenewold	INL
Nguyen Minh	UCSD
Tianli Zhu	UTRC
Joseph Hartvigsen	OxEon Energy
Joseph Barton	FuelCell Energy
Ani Kulkarni	CSIRO
Jamie Holladay	PNNL

Session Summary

Session ID: H5-A
Title: Materials and Device Lifetime Testing Protocols

Summary of discussion

- How to track degradations; Main degradation mechanisms; Accelerated stress testing - pros and cons for each "accelerating condition"
- Tests needed at materials, cell, and stack levels; each level provides some useful information, but not all
- Fixed V, Fixed I, fixed H_2 production rate, are all relevant, yet hard to say one is better than the other.
- Faraday Efficiency for H+-SOEC

Consensus and/or dissenting opinions

- Agreement on separate protocol development for H-SOEC and O-SOEC
- Agreement on operating conditions for short term testing with:
- O-SOEC: temperature 650-850°C; steam content 50%±20%; hydrogen content in steam is 1-5% (or use 50/50 =H_2/N_2 in steam); voltage 1.3-1.4V; current density 1±0.2 A/cm^2 (or 0.5 A/cm^2 for electrolyte-supported); test time is 1,500-5,000 hours
- H-SOEC: 600±100°C; steam content 50%±20%; voltage 1.2-1.4 V; current density 0.5-1.2 A/cm^2; use GC to monitor H_2 production; testing time is 500-2,000 hours

Key Take-Aways

- How to project realistic HTE performance: do real long-term testing
- Testing needs to be coupled with post-mortem analyses to understand degradation mechanisms and kinetics
- Faraday Efficiency needs to be better tracked, for proton-conducting SOECs

Action Items

- Develop draft testing protocols based on the discussion
- Re-visit the protocol after it is being implemented

Session Attendee List

Session ID: H5-A
Title: Materials and Device Lifetime Testing Protocols

Name	Affiliation
Xingbo Liu	WVU
Nguyen Minh	University of California San Diego (UCSD)
Kevin Huang	University of South Carolina
Joseph Barton	FuelCell Energy
Tianli Zhu	UTRC
Joseph Hartvigsen	OxEon Energy
David Peterson	DOE
Dong Ding	INL
Olga Marina	PNNL
Scott Swartz	Nexceris

Session Summary

Session ID: H5-B

Title: Definitions

Summary of discussion

- Reviewed definitions, terms, and metrics
- Thermal efficiency: vague, do we need it?
- Overall efficiency discussed
  - Cell Level: H2 HHV/(Vcell)/2F; Faradaic Efficiency, V_{OCV} vs E_{nernst},
  - Stack level
  - System : H2 HHV/ (electrical + thermal energy)
- Gas composition- discussion on the need to define this. It does need to be reported.
- Akin to efficiency metric on [prod rate]/[input power] – kg H2/MW_e-hr not tonne H2/MWhr

Consensus and/or dissenting opinions

- Dissenting opinion on need to define gas compositions. : Note "standard" is not necessarily optimal for a given technology
- Consensus on Electrical efficiency = LHV H2/Mw_ehr*100
- H2 production for a certain condition (V_{TN}) moles H2/cm^2/sec
- Consensus on advantages of HTE vs LTE

Key Take-Aways

- Additional definitions are needed (see action list)
- Important for measured V_{OCV} to be close to theoretical V_{OCV} to ensure cell is operating correctly (i.e. no mechanical leaks, etc.)

Action Items

- Define conditions to measure / calculate OCV versus Nernst (M. Williams)
- Protocol for Faradaic efficiency (D. Ding)
- Define ASR in electrolysis mode (N. Sullivan)
  - Define non-linearity with R^2 fit
  - Define ASR at thermoneutral
- Protocol to accurately measure H_2 production and gas (mechanical) leakage and electrical leakage – part of faradaic efficiency protocol (H. Ding)

Session Attendee List

Session ID: H5-B

Title: Definitions

Name	Affiliation
Jamie Holladay	PNNL
Nguyen Minh	University of California San Diego (UCSD)
Kevin Huang	University of South Carolina
Joseph Barton	FuelCell Energy
Tianli Zhu	UTRC
Joseph Hartvigsen	OxEon Energy
Ani Kulkarni	CSIRO
Scott Barnett	Northwestern
Harping Ding	INL
Mark Williams	Key Logic
James O'Brien	INL
Gary Groenewold	INL

Session Summary

Session ID: H6-A

Title: Wrap-up/Action Item Assignment , HTE Materials, Components

Summary of discussion

- Some of the already written protocols needs a revision to make it more generic (rather than specific to one lab)
- Need for protocols development reiterated

What are we missing?

- More generic protocols
- Technical: Some of the missing protocols (assigned as action items)
- A Central repository (to enable accesses and make tracked changes) to the protocols during development stage
- Definition of thermal stability: only conductivity? Thermal shock resistance? How about other

Consensus and/or dissenting opinions

- Separate protocol for procedures already covered with ASTM (*yes and no from participants*)
- Protocol may be still written but can refer to ASTM standards (this is for sake of completeness of this whole exercise)
- Protocols for interconnect not required at this stage (*yes and no from participants*)
- Should EIS protocols include modelling? (*yes and no from participant*)
- All agree that protocols need revisions and refinement
- Some of the protocols can be merged into single protocol
- Agreed conditions for conductivity measurements benchmarking O-SOEC: 500-900C, Wet air (3%), H-SOEC wet H_2 (3%)

Key Take-Aways

- Revisit protocols P01, P02, P03, and combine into one major document covering all these aspects (total conductivity, mixed conductivity using four probe to be combined into one document)
- Instead of developing a separate “stability” (P06) document, we can modify measurement protocols by adding one sentence for other document, just mentioning expose to certain hours and measure the properties again
- EIS should be measured, and Rs & Rp should be extracted, but from benchmarking and protocol development points-of-view, we may not want to go detailed interpretations (modelling of EIS)

Action Items

- New (combined) four probe conductivity measurement protocol **Xingbo Liu**
- New protocol for conductivity test for interconnects **(Ani Kulkarni**)
- New Protocol for the TPB lengths measurement (**Scott Barnette**)
- Link for all current protocols or copies to be sent to all involved in the workshop/s (**Olga Marina**)
- Modify P10 (with current collector for button cells) (**Kevin Huang**)
- Confirm Benchmarking conditions for conductivity measurement mentioned above (**Neal Sullivan**)
- Track what next (**Olga Marina)**

Session Attendee List

Session ID: H6-A

Title: Wrap-up

Name	Affiliation
Scott Barnett	Northwestern
Kevin Huang	University of South Carolina
Ani Kulkarni	CSIRO
Harping Ding	INL
Dong Ding	INL
Xingbo Liu	WVU
Neal Sullivan	CSM
Gary Groenewold	INL
Thomas Malkow	JRC Petten
Amin Nouri	Greenlight Innovation

Session Summary

Session ID: H6-B

Title: HTE Cells and Stacks

Summary of discussion

- Potential gaps
- Lots of discussion on economics and balance-of-plant
- Discussed differences between small cells, large cells and stacks in terms of different needs
- Discussed need for accelerated stress testing of button cells, full size cells, and stacks (short and full)

Consensus and/or dissenting opinions

- Balance-of-plant: consensus that it is very important, but dissenting opinions on if HydroGEN should do it. The majority thought it was outside the scope of HydroGEN
- Consensus: HydroGEN should focus on materials which limits it to small cells. Large system testing should not be part of HydroGEN
- Consensus: Accelerated stress testing is important, but requires a lot of testing to develop
- Consensus: Round robin testing will be important, but not sure how it will be funded
- Consensus: Workshop in FY20 is important

Key Take-Aways

- Gap: durability protocol for oxygen and proton conductors
- Gap: stack durability protocol
- Roadmap: need to add scaling cell size up and new optimal stack design (not all SOFC can be adopted)
- Gap: define control (null) experiment for material testing
- Gap: What standards are we to use (ASTM, codes committees?)
- Gap: include why protocol methodology is recommended (lessons learned), how to analyze the data
- Gap: advanced manufacturing development to decrease costs
- Gap: Accelerated stress test will need to be developed for each degradation mechanism. This will take a lot of testing. A high throughput testing system is needed.

Action Items

- Priorities: define what is the standard cell? Who makes? How much will it cost to the users (universities)?
- Define what is the standard stack? What is the standard size?
- What is the standard operation conditions?
- Move from small button to single large cells
  - Round Robin testing – how this will be funded?
  - Accelerated stress testing: tests will need to be developed for each degradation mechanism. This will take a lot of testing. A high throughput testing system is needed.

Session Attendee List

Session ID: H6-B

Title: Wrap-up

Name	Affiliation
Mark Williams	Key Logic
Nguyen Minh	University of California San Diego (UCSD)
Olga Marina	PNNL
Joseph Barton	FuelCell Energy
Jim O’Brien	INL
Joseph Hartvigsen	OxEon Energy
Scott Swartz	Nexceris