H2NEW: Hydrogen (H2) from Next-generation Electrolyzers of Water, Task 7: Advanced Characterization

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Goal: H2NEW will address components, materials integration, and manufacturing R&D to enable manufacturable electrolyzers that meet required cost, durability, and performance targets, simultaneously, in order to enable $2/kg hydrogen.

H2NEW has a clear target of establishing and utilizing experimental, analytical, and modeling tools needed to provide the scientific understanding of electrolysis cell performance, cost, and durability tradeoffs of electrolysis systems under predicted future operating modes.
H2NEW HTE Task 7: Advanced Characterization – Overview

Timeline and Budget

• Start date (launch): **October 1, 2020**
• Awarded through September 30, 2025
• FY21 DOE funding: **$175**
• Annual budget adjustments anticipated: *Increasing as planned once cell testing begins*

Barriers

• Durability – economic competitiveness hinges on longer o-SOEC lifetime, understanding and mitigating degradation mechanisms
• Realizing reduced o-SOEC cost, while maintaining or improving efficiency

National Lab Consortium Team

• Task 7: Advanced Ex-situ and In-operando characterization
• Coordination w/ Task 5: o-SOEC accelerated stress testing
• Coordination w/ Task 8: Atomistic / spectroscopy modeling, Microstructural / electrochemical degradation modeling
### Electrolyzer Stack Goals by 2025

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<tr>
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<th>LTE PEM</th>
<th>HTE</th>
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<tr>
<td><strong>Capital Cost</strong></td>
<td>$100/kW</td>
<td>$100/kW</td>
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<tr>
<td><strong>Electrical Efficiency (LHV)</strong></td>
<td>70% at 3 A/cm²</td>
<td>98% at 1.5 A/cm²</td>
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<tr>
<td><strong>Lifetime</strong></td>
<td>80,000 hr</td>
<td>60,000 hr</td>
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**Two key factors** holding HTE / SOEC technology at its current TRL

1. Cost, performance, durability limited by degradation, esp. fuel electrode
   - Ni migration, electrode breakdown near the electrolyte, impurity buildup
2. Recent performance improvements *via* optimization of fuel electrode
   - micro/nano-structure and chemistry

Catalyst infiltration, layer deposition, exsolution, etc

Potential HTE Cell Failure Mechanisms:
• Void formation at interfaces
• Migration of cations from the electrodes into the electrolyte – new phase formation
• Nickel coarsening
• Cation reduction/oxidation

Advanced Characterization Goal:
• Confirm failure mechanisms
• Discern order of failures – relative kinetics

Advanced characterization is required to gain a fundamental understanding of failure mechanisms at the atomic level for improved cell development
H2NEW HTE Task 7: Advanced Characterization – Approach

**Transmission X-Ray Microscopy**
- Identification of structural changes (i.e. void formation), cation migration, and chemical information
- 3D imaging /30 nm resolution
- X-ray absorption spectrum at each pixel/voxel provides element specific mapping and chemical information including oxidation state
- Ex-situ and in-situ experiments

**X-Ray Absorption Spectroscopy**
- Determination of cation oxidation states and changes in oxidation state after cycling
- Ex-situ and in-situ experiments

**X-Ray Diffraction**
- Identification of element speciation and migration
- Ex-situ and in-situ experiments

**Advanced characterization at a synchrotron source:**
- **High resolution data**
- **High signal to noise ratio**
- **Ex-situ and in-operando experiments**
H2NEW HTE Task 7: Advanced Characterization - Accomplishments & Progress

Accomplishments:

• Successful proposal submitted to SLAC for access to XRD, XAS, and TXM beam lines – beam time has been awarded

• Collaborations with beam line scientists have been established

• Initial XRD and TXM experiments have been carried out successfully

Continuing Progress:

• In-depth data analysis of previous work is on-going

• In close collaboration with beam line scientists, in-situ experimental tools are being developed – including partnership award with molecular foundry

• Beam time has been awarded for the next round of ex-situ experiments and experiments are being planned in collaboration with beam line scientists
TXM Results – cell cycled 300 hours

For TXM work, INL synthesized and cycled the HTE cells, SNL fibbed to appropriate size, NREL ran the experiments and carried out data analysis.

- Void formation in GDC observable in 3D tomography
- Iron oxides migration into GDC layer identified in 2D XAS imaging

3D Tomography of THE cell cycled 300 hours

2D XAS map at the Fe edge (top) and corresponding XAS spectra (bottom)
H2NEW HTE Task 7: Advanced Characterization – Key Results: XRD 0 hr cell

XRD Results – cell cycled 0 hours
- Synchrotron enables XRD through full cell
  - SLAC XRD able to see all layers of the cell
  - Bottom Ni/YSZ layers observed through full cell
- Detected secondary phases present in 0hr sample
  - Discovered Fe$_2$O$_3$ is left over from synthesis of GDC layer (red arrow)
  - Additional phase (blue arrow)
H2NEW HTE Task 7: Advanced Characterization
– Key Results: XRD 300 hr cell

XRD Results – cell cycled 300 hours
• Secondary phases identified
• Secondary phase from match expected pattern from Gd$_3$FeO$_6$ (Gd$_x$Fe$_y$O$_z$ family in blue)
  – Evidence suggests other Gd&Fe compounds forming at 300hrs
  – GdFeO$_3$/Gd$_3$Fe$_5$O$_{12}$
• Additional secondary phase peaks suggest additional decomposition reactions

![XRD Pattern](image1)

![XRD Pattern](image2)
Challenges

• Post-mortem processing can have unknown affects on cell structure.
  • Characterization of a single cell at different points in its lifetime is necessary for confirming degradation mechanisms requiring in-situ experiments.

Proposed Future Work

• Continuation of pristine and post-mortem cells synchrotron characterization:
  • XRD
  • XAS
  • TXM

• Development of cell test stands that can be run outside of a beam line and moved into the beam pathway/beam line hutch for characterization at multiple points in the cell life-time
  • The established collaborations between NREL and the beamline scientists will be integral

Any proposed future work is subject to change based on funding levels
Task 7 – Advanced Characterization Path Forward

• Q5 (October – December, 2021)
  – Produce 3-D, 2D microstructure of an electrode-supported YSZ cell capable of measuring changes in local elemental composition, crystalline grain size and structure, and material defects (NREL – QPM10)

• Q6 (January, 2022 and beyond)
  – In-operando test stand for button cells, large area cells, beam line compatible, replicating INL, PNNL and LBNL test stands (NREL – QPM12)
  – Concepts for improved cell performance: 10% at 1.28 V, w/ mitigated degradation (INL, PNNL, LBNL, NREL – M4).
  – AST protocols for SOEC HTE. 3 component-level HTE ASTs; compare cell and AST performance losses.
    Criteria: Use comparisons of spectroscopic, microscopic characterization and electrochemical performance to verify that AST effectively captures and accelerates relevant degradation observed under long term cell operation. (INL, PNNL, LBNL, LLNL, NREL, NETL, ANL – GNG1)
Task 7 – Advanced Characterization Path Forward, con’t

• **Q6 (January, 2022) and beyond, con’t**
  - Development and validation of AST protocols for PEM LTE/SOEC HTE. Propose at least 3 component-level LTE and HTE ASTs (each) and compare cell and AST performance loss. Criteria: Through characterization comparisons (using appropriate approaches including spectroscopic, microscopic and electrochemical signatures) verify that at least 1 AST effectively captures and accelerates the relevant degradation mechanisms observed under long term cell operation via achieving similar experimental and characterization results between accelerating and standard operating conditions. (All labs – GNG1)
  - Expanded test matrix: interconnects, current collectors representative of stack operations
Task 7 Advanced Characterization Summary

- Beamline experiment prep
- Beamline experiment status
- Other
  - Detailed understanding of degradation mechanisms, development of in operando characterization tools, and multiscale modeling, setting stage for transition to H2NEW
Technical Backup and Additional Information
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