Pacific Northwest NATIONAL LABORATORY

DOE Hydrogen and Fuel Cell Office
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Electrolyzer Stack Development and Manufacturing

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Pacific Northwest National Laboratory

DOE Project Award 34094

Project ID # T043

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Project Goal

- Support the US manufacturers in identifying and understanding manufacturing and operational issues by
  - Performing extensive commercial stack post-test characterization
  - Integrating predictive modeling to guide manufacturers in choosing conditions for operational stack durability and assembly process to prevent cell damage yet assuring good contacts and sealing
  - Assessing available diagnostic tools and QA/QC approaches applicable for large scale SOEC manufacturing at high production rates and identifying QC gaps as well as the points of mandatory inspection
  - Developing in operando stack health diagnostic module to detect faults during testing

Overview

Timeline

- Project Start Date: 10/01/2019
- End: Project continuation and direction determined annually by DOE

Key Barriers Addressed

- Hydrogen Cost
- System Efficiency
- Manufacturing
- Renewable Electricity Generation Integration

Budget

- FY24 DOE Funding received: $800,000
- FY24 Planned DOE Funding: $800,000
- Total DOE Funds Received to Date: $3,800,000 (since the project started)

Partners

- Pacific National Laboratory
- Idaho National Laboratory
- National Renewable Energy Laboratory

Potential Impact

- Support Hydrogen Earthshot's $1/kg H_2 target by performing R&D to understanding large scale stack manufacturing requirements and failure mechanisms to improve stack durability and efficiency, thus reducing hydrogen cost and increasing hydrogen production
- High level electron microscopy near nm level is imperative to understanding stack degradations and failures
  - Accelerates industrial development and production of SOEC stacks by providing in depth analysis at near nm scale across mm length scales
  - Industry often does not have capital to purchase next generation microscopic instrumentation
  - Unique chemical signatures assist in determining diffusion profiles of various components across mm length scales
- Advanced operando stack diagnostics and characterization help identify potential failures before they occur
- Advanced diagnostic tools and QA/QC approaches becomes critical for large scale SOEC manufacturing at high production rates

Approach

Address Important Electrolyzer Manufacturing Issues that Adversely Affect Stack Lifetime

- Provide high level analytical analyses to accelerate industrial research and design of commercial stacks and support by predictive modeling
- Protect industry proprietary information via NDAs and additional internal firewalls
- High level meetings are conducted with commercial partners to understand parameters and analysis needs
- Sample preparation for microstructural/microchemical examination including cutting, sectioning, impregnating with epoxy, polishing understanding specific details with respect to processing conditions. CT/SEM/S-TEM examination of stack cross-sections
- Survey the state-of-the art in QA/QC by reaching out to diverse industries and conducting literature search. Determine critical defects dominant in rejected cells. Identify knowledge gaps in existing QA/QC procedures. Identify improved methods to detect known defects

Milestones

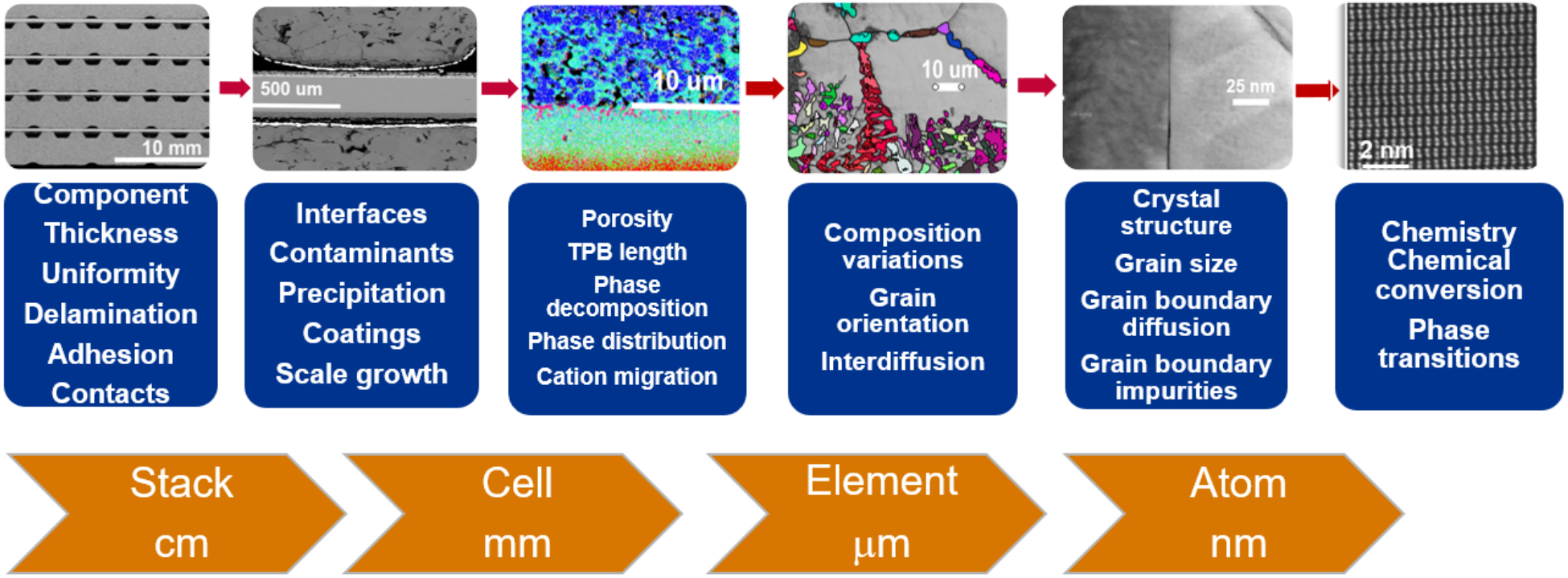
Date	Milestone/Deliverable	Complete
12/31	Complete packaging Gen1 in operando stack health monitoring module and deliver to INL for testing	100%
3/30	Complete post-mortem analysis of 2 commercial stacks and prepare technical report for each industry client	100%
6/30	Complete post-mortem analysis of at least 1 more commercial stacks and prepare a technical report and a journal publication	30%
7/31	QA/QC for high production rate cell manufacturing: identify mandatory points of inspection and validate at least 2 diagnostic tools	50%
Go/ noGo	Complete post-mortem analysis of 3 commercial stacks and prepare 1 technical report per stack for each industry client summarizing findings	50%

Approach: Safety Planning and Culture

- Project was not required to submit a safety plan to HSP
- Project follows strict PNNL safety requirements
  - Approved SOPs must be created, approved, and followed
  - Lab Assist activities must be created, approved, and followed for any work in the lab
  - All staff is required to take safety training related to the specific Lab Assist activities they are involved
  - Safety training must be completed to gain active access to the lab space
  - Safety training needs to be retaken following specific class requirements
- Specific to active work with hydrogen, all staff is also recommended to take AIChE Hydrogen Laboratory Safety class

Accomplishments and Progress: Partnered with Industry to Identify Manufacturing Issues, Degradation Mechanisms, Effect of Operating Conditions, Critical Micro and Nanostructural Parameters

Continuum Scale Commercial Stack Analysis



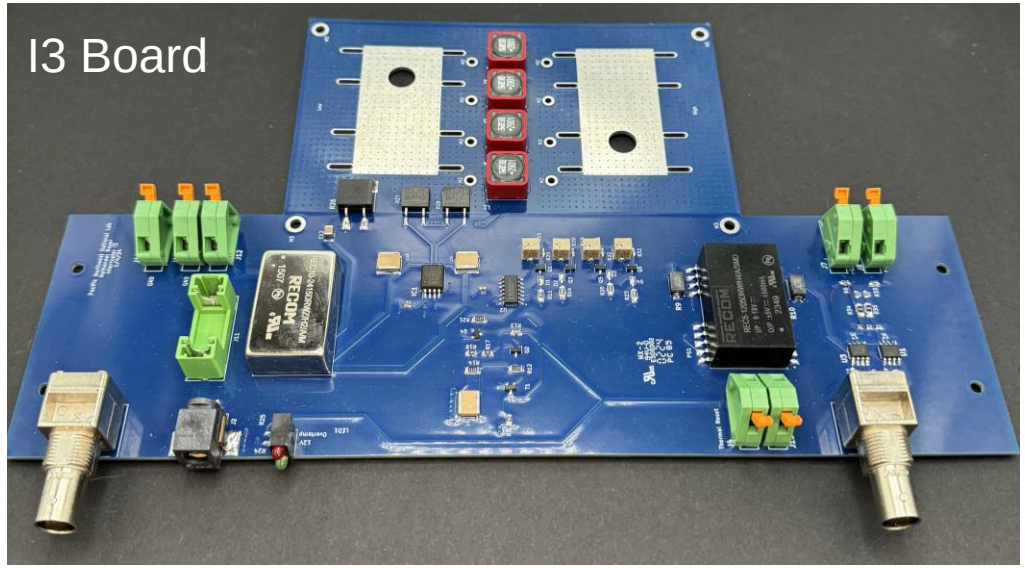
Further improvements of electrochemical performance and long-term durability is critical for scale-up and integration

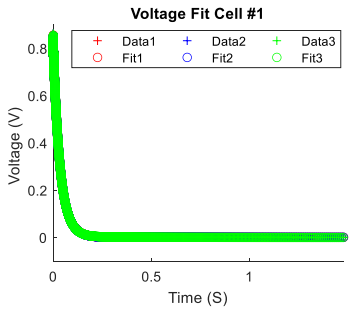
Accomplishments and Progress: Performing Analyses of Commercial Stacks

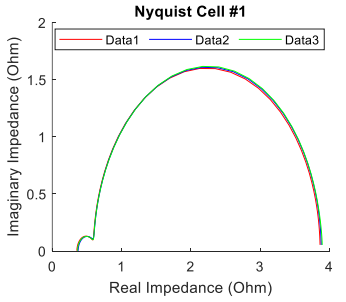
- Two stack samples have been received and processed to prepare SEM samples
- Samples were cross-sectioned and prepared for analysis
- SEM/EDS data collection has been completed and the initial reports have been sent to the client
- The follow-up analyses are being discussed
- Third stack has been shipped to PNNL

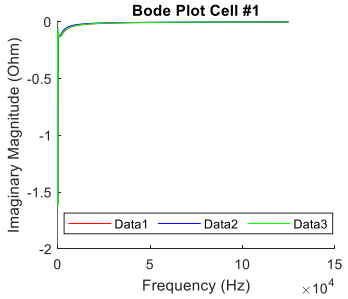
Accomplishments and Progress: Complete Packaging of a Current Interrupt Module Including Hardware and Software for end users

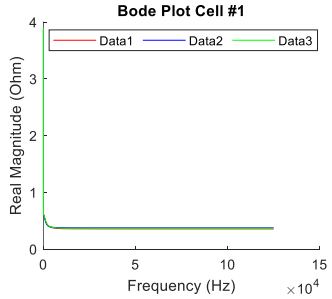
- I3 Board was developed, and functional checks were performed
- DAQ modules capable of safely handling high voltages from stack were added
- Digital “handshake” between the current interrupter and INL test system to ensure both systems are ready for interruption were completed





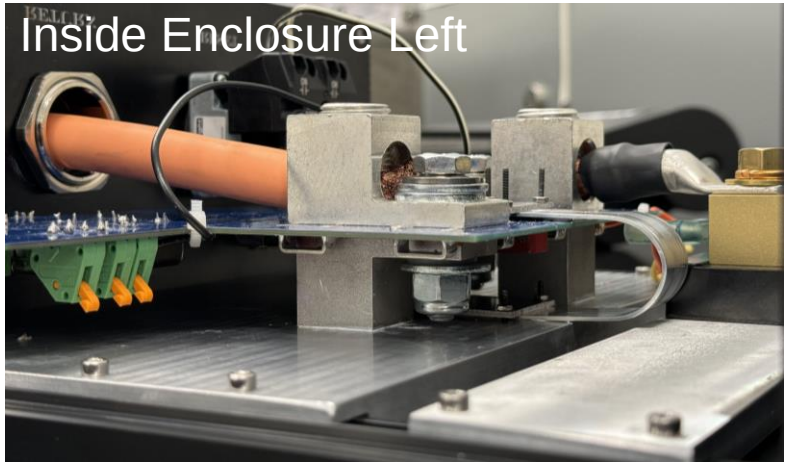


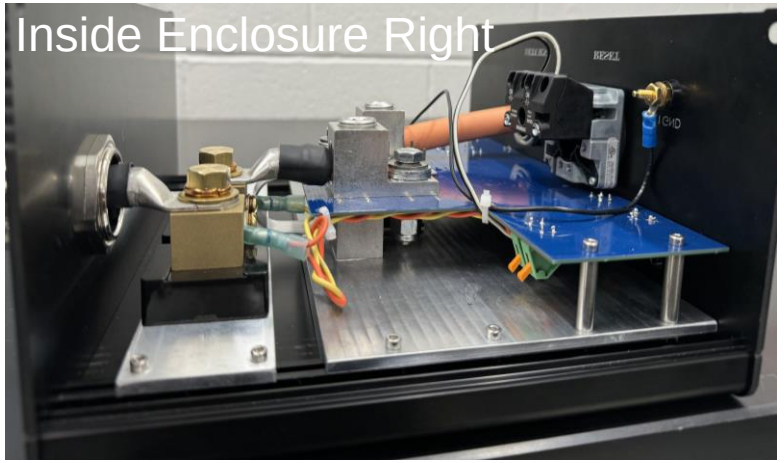




Accomplishments and Progress: Enhancements Made to Software and Hardware to Improve Integration with INL Test Capability

- I3 board was packaged in an enclosure
- I3 device, voltage sense DAQ, cables, and software were shipped to INL for independent validation

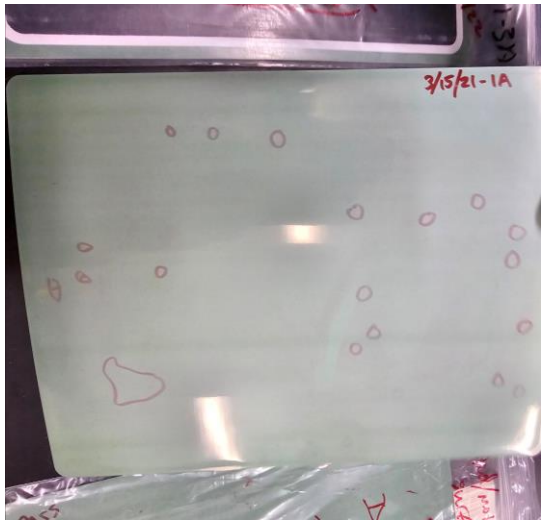




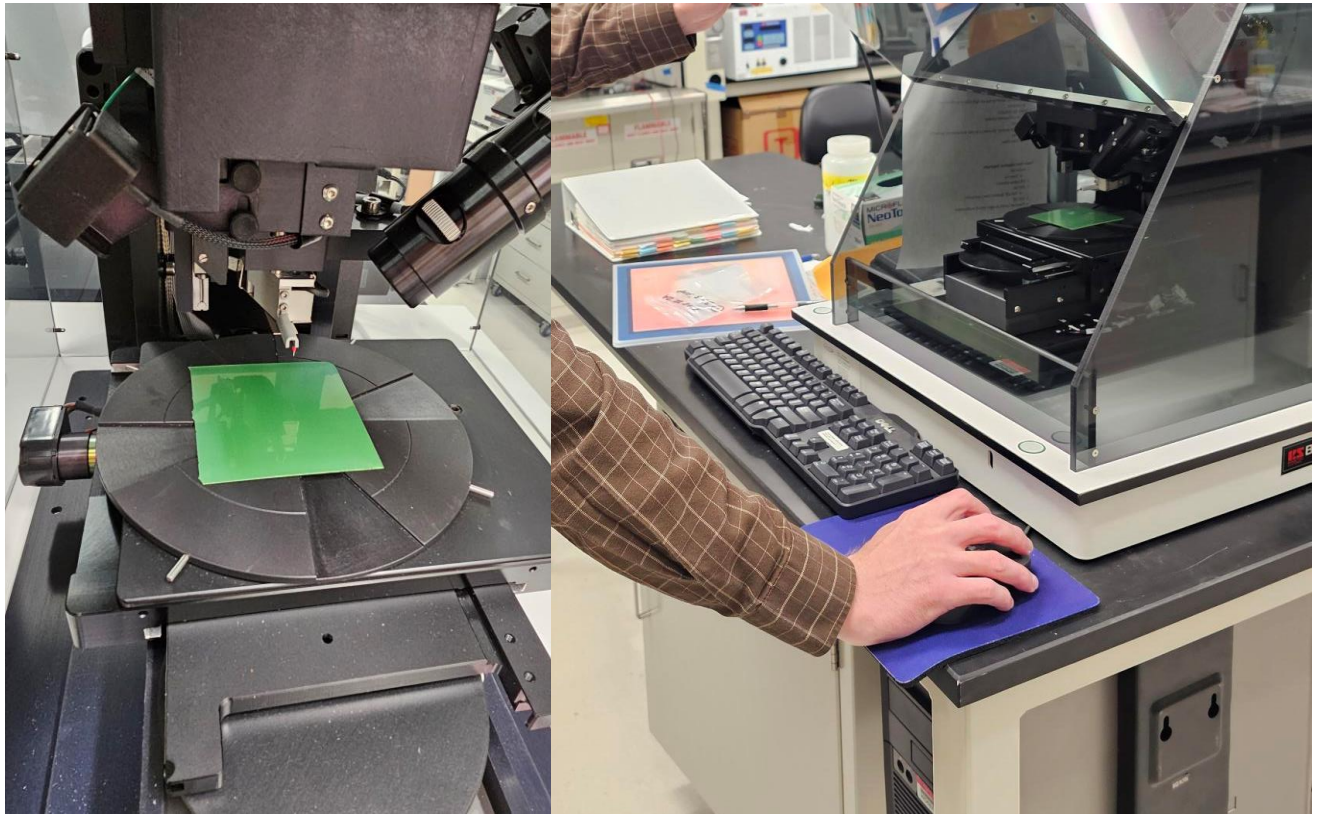
Accomplishments and Progress: Developed State-of-the-Art QA/QC Survey for Solid Oxide Cell Developers

- A web-based Questionnaire targeted at SOEC/SOFC industries has been created to evaluate the current status of QA/QC in cell and stack production
- Over 30 private companies and national laboratories in North America, Asia and Europe have been contacted with the request for QA/QC feedback; responses are being received and analyzed
- Physical and chemical diagnostic methods have been correlated to the types of defects they are used to monitor in SOEC and other industries with overlapping processes such as fuel cells and batteries

Accomplishments and Progress: Assessed Available Diagnostic Tools to Reject Cells



A large-area substrate rejected due to the identified defects



Evaluation of the surface uniformity of the film using a profilometer

Accomplishments and Progress: Assessed the Use of Potential Diagnostic Tools by Industry

Examples of questions in a QA Questionnaire

1.5. Do you test for different defects at different frequencies i.e., constant monitoring vs. occasional sampling?

- ◯ Yes
- ◯ No

4.2. Profilometry

- ◯ Yes
- ◯ No

If you use profilometry, for what defects and at what steps?

Your answer

4.3. Imaging (optical/thermal) with machine learning

- ◯ Yes
- ◯ No

Where are QA/QC gaps?

3.1. What else would you like to monitor and at what stage?

Your answer

3.2. If a non-destructive technique is unavailable, how many parts are you willing to sacrifice for QC?

Your answer

2.6. What needs to be controlled better than current approaches?

Your answer

2.5. What other inspection tools would like to use?

Your answer

4.8. Adhesion test

- ◯ Yes
- ◯ No

If you use adhesion test, for what defects and at what steps?

Your answer

4.9. X-Ray Fluorescence (XRF)

- ◯ Yes
- ◯ No

If you use Ellipsometry, at what steps?

Your answer

Responses to 2023 Reviewers' Comments

- *Identifying gaps in reproducibility is rather challenging unless the stacks are tested in-house*
  - SOPs, test protocols, and software manuals have been developed and shared with the collaborators. Reproducibility issues have also been addressed by Benchmarking project suggesting to establish protocol validation sites.
- *Working with larger industrial partners on QA/QC cell and stack fabrication or consultants coming from the large industry is worth considering*
  - Indeed, although several industries responded to our Questionnaire and provided their valuable feedback, we would still need to gain trust of other industries to respond or convince that their time invested in responding is valuable.
- *Investigating accelerated stack tests (beyond single-cell tests) in order to effectively simulate long-term operation would be valuable. Further expanding on the matrix structure, including more on/off cycles, would be valuable*
  - Large stack effort has proved to be more complex, and testing short stacks using ASTs protocols developed by H2NEW would be of extreme value beyond H2NEW (that is focusing on cells and components).
- *PNNL's work on post-mortem characterization of tested stacks provided by industry is extremely valuable, as PNNL possesses characterization and analysis capabilities to which most industrial stack developers and manufacturers do not have access. PNNL's development of techniques for measuring impedance in stacks under load could be valuable in developing a real-time understanding of stack degradation. Predictive models established for glass sealing, temperature distributions, and thermo-mechanical stress–strain relationships can also provide value to stack developers.*
  - Industry partners have been very much interested in all 3 topics mentioned above and we believe this work should continue.
- *PNNL's support of stack developers by providing post-test characterization of stacks is exemplary.*
  - *Industry opened even more this year to a close collaboration; we will pursue further opportunities to support domestic industry*
- *The project should focus on improving performance and reducing degradation by addressing core stack technology issues (sealing, current collection, shorting, etc.) that have been encountered. The team might consider a smaller stack demonstration at INL so that more project resources can be devoted to the above.*
  - Focusing on short stacks and understanding manufacturing issues to suggest the solutions and reporting on degradations and mitigation strategies would be of high value to industry

Collaboration and Coordination

- Reached out and established collaboration with industry to assist in understanding stack issues by providing extensive post-test characterization; developed a firewall to protect proprietary information from internal work
  - 3 commercial stack samples were provided
  - Stakeholder meetings/engagement
    - ✓ Pre-shipping meetings to discuss needs/requirements
    - ✓ Understanding industrial needs
    - ✓ Post-sample analysis discussions/presentations
- INL will be testing the current interrupter device as an independent validator to provide feedback for future optimization and improvements
- Initiated collaboration with NREL to leverage their expertise in PEM QA/QC. Using similar approaches, NREL will assess the potential defects in large SOEC cells provided by PNNL

Remaining Challenges and Barriers

- Gaining industrial partners confidence is key to successful collaboration
  - Samples shipped by industrial partner on time
  - Clear/direct sample preparation guidance is key

- Barriers
  - Engaging new industrial partners takes time in legal departments
  - Access to equipment
  - Sample delivery with respect to deadlines
  - Information on QA/QC in industry can be proprietary; limited access to such information may result in duplicating efforts and in creation of incomplete view of the state of QA/QC

Proposed Future Work

- Continued industrial support to analyze and report on next generation/ exploratory designs likely to be accelerated by newly announced FOA awards
- Further extensions of this program will involve advertising capabilities to attract new companies to participate
- Analyze the feedback from industry and aggregate with the existing knowledge results to determine the existing QA/QC gaps
- Determine the critical defects dominant in rejected cells. Assess whether the existing information about the type of defects and their importance is adequate. If significant gaps exist, carry out in-house tests to define the critical defects
- Evaluate existing manual or visual tests with in-line and automated alternatives

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

Summary

- Industrial engagement has proven to be critical to this project success
  - In depth planning and establishing close collaboration with multiple partners is being pursued
  - Three stacks were shipped for post-test analyses
  - SEM analyses on two stacks achieved and chemical and microstructural analysis performed
  - Rigorous data analysis being performed
  - Crucial information related to stack integrity at the nm scale being provided
  - Reached out to over 30 private companies and national laboratories in North America, Asia and Europe with the request for QA/QC feedback using the specially developed web form
  - Tabulated physical and chemical diagnostic methods and the types of defects they are used to monitor in SOEC and other industries
  - Plan to identify significant knowledge gaps in SOEC QA/QC and propose two methods that exceed the capability of currently used manual or visual methods
- Delivered stack health monitoring system for determining operational issues on stacks to INL for independent validation