H2NEW
U.S. DEPARTMENT OF ENERGY
Hydrogen from Next-generation Electrolyzers of Water

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

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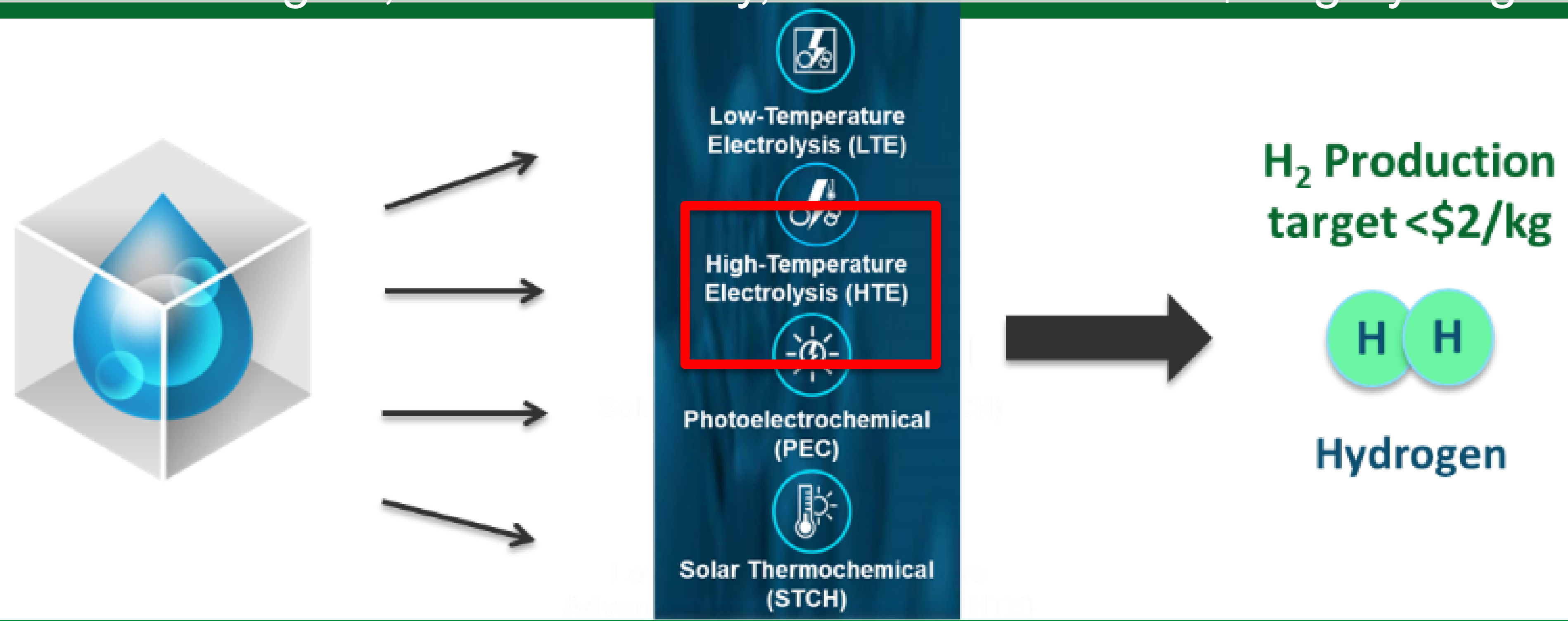
NREL Transforming ENERGY · INL Idaho National Laboratory · Argonne National Laboratory · Berkeley Lab Bringing Science Solutions to the World · Lawrence Livermore National Laboratory · Los Alamos National Laboratory EST. 1943 · National Energy Technology Laboratory · Oak Ridge National Laboratory · Pacific Northwest National Laboratory

Project Goals

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

H2NEW HTE Task 7: Advanced Characterization – Relevance and Impact

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Electrolyzer Stack Goals by 2025		
	LTE PEM	HTE
Capital Cost	$100/kW	$100/kW
Electrical Efficiency (LHV)	70% at 3 A/cm^2	98% at 1.5 A/cm^2
Lifetime	80,000 hr	60,000 hr

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

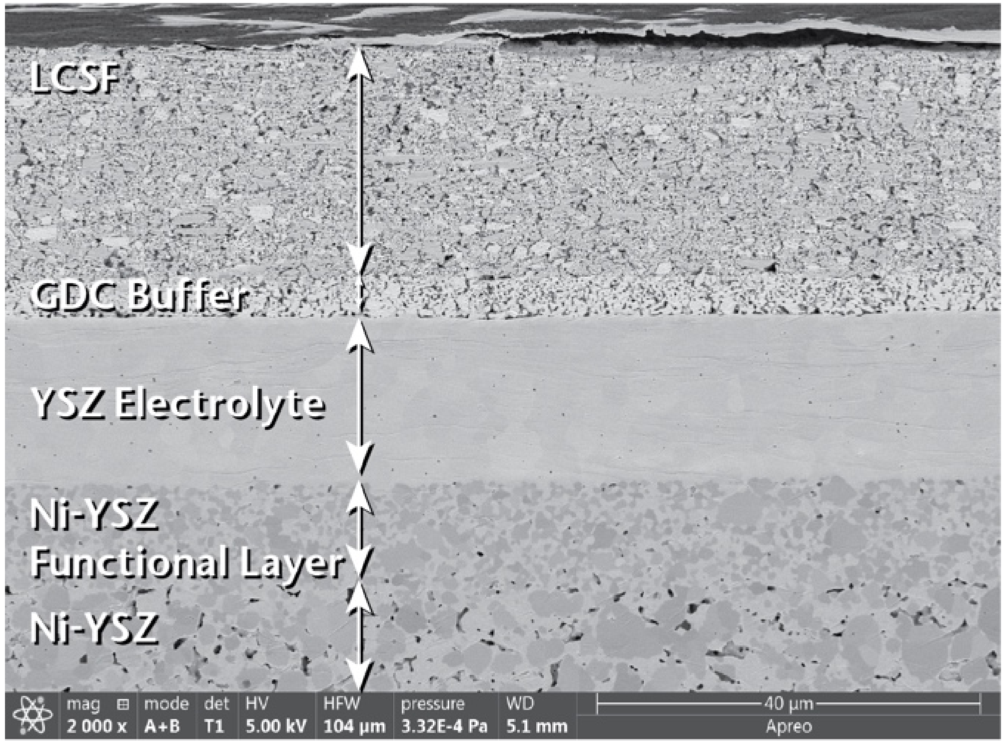
Hauch et al (2020). Recent advances in solid oxide cell technology for electrolysis. *Science*, *370* (6513)

H2NEW HTE Task 7: Advanced Characterization – Relevance and Impact

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

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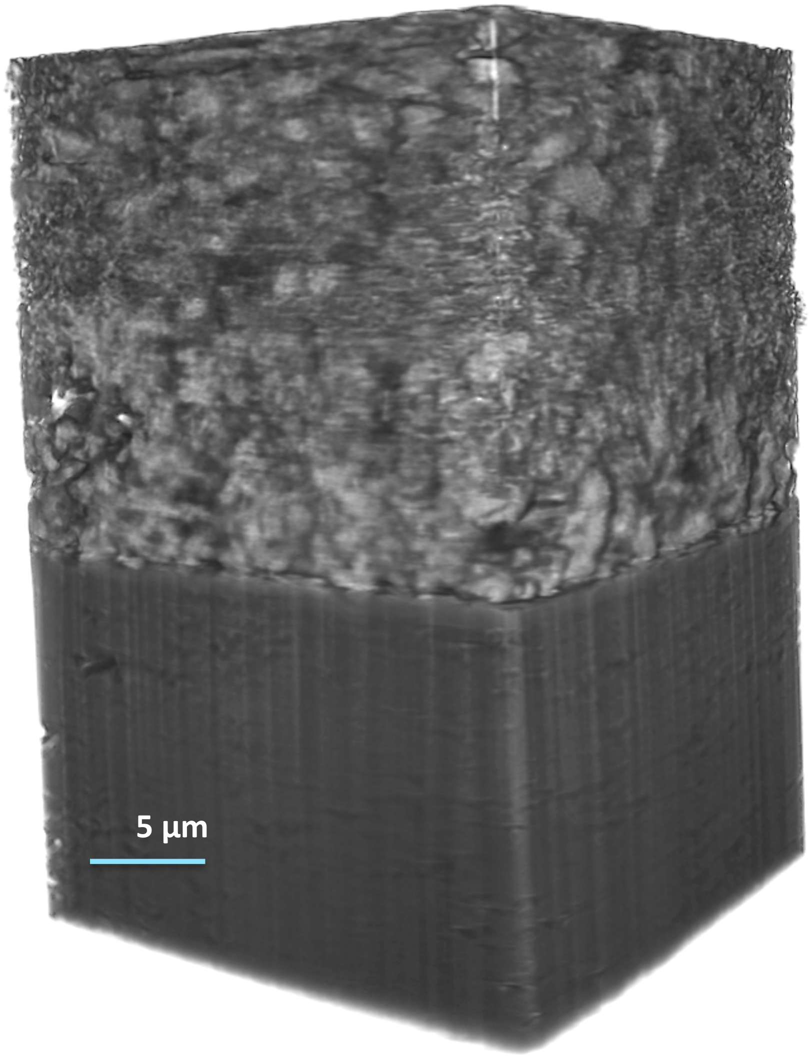
Accomplishments:

- 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

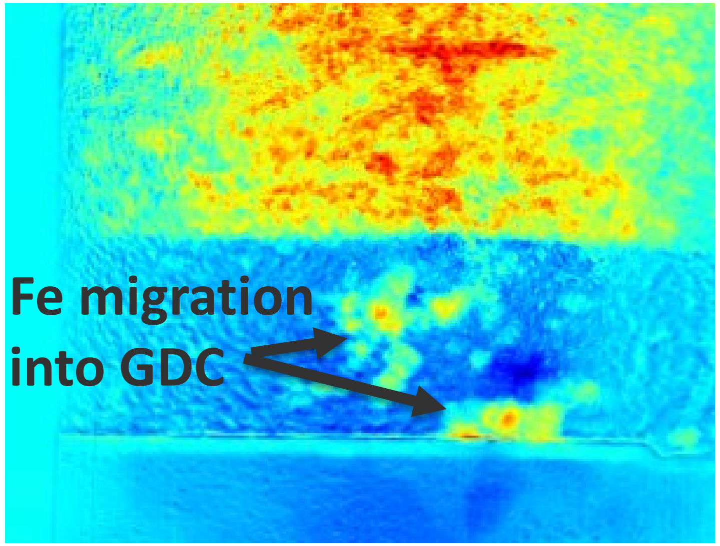
Continuing Progress:

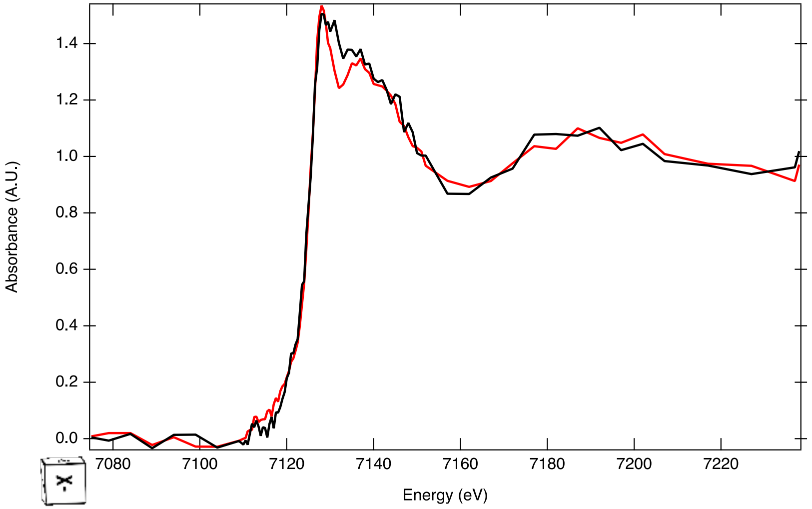
- In-depth data analysis of previous work is on-going
- In close collaboration with beam line scientists, in-situ experimental tools are being developed – including partnership award with molecular foundry
- Beam time has been awarded for the next round of ex-situ experiments and experiments are being planned in collaboration with beam line scientists

H2NEW HTE Task 7: Advanced Characterization – Key Results: TXM



3D Tomography of THE cell cycled 300 hours





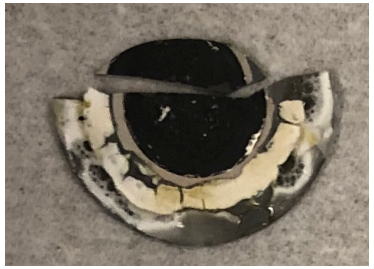
2D XAS map at the Fe edge (top) and corresponding XAS spectra (bottom)

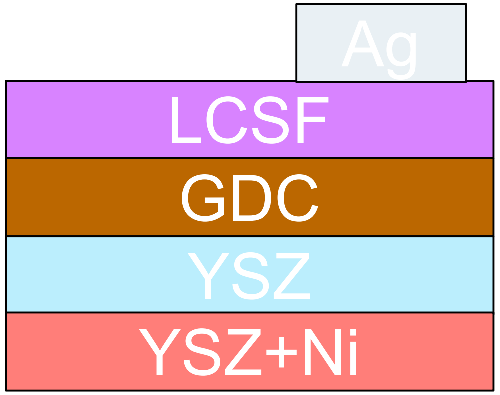
TXM Results – cell cycled 300 hours

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

H2NEW HTE Task 7: Advanced Characterization – Key Results: XRD 0 hr cell

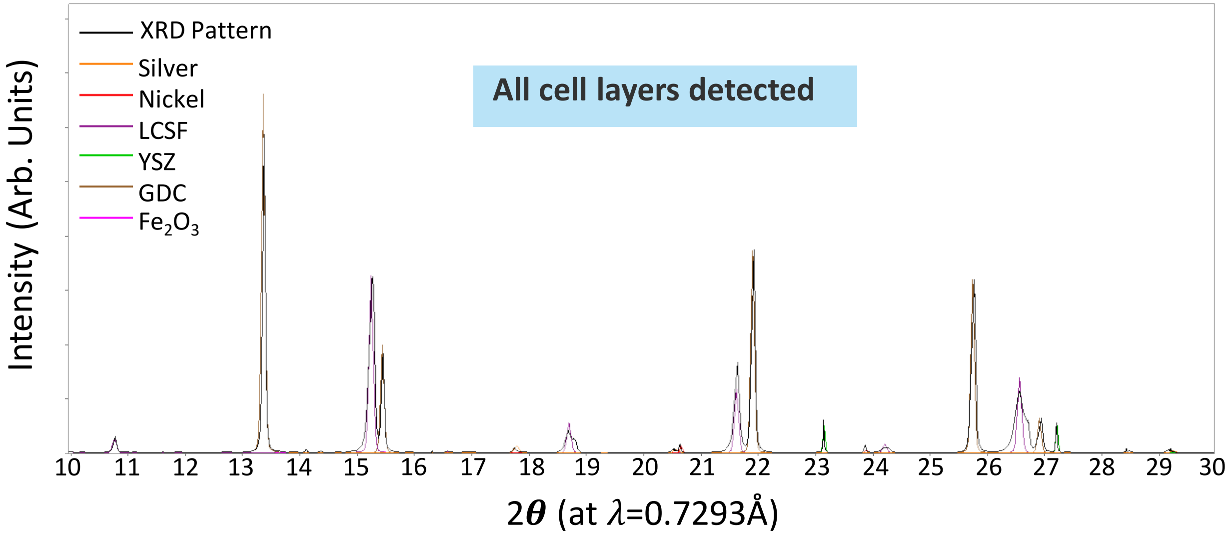




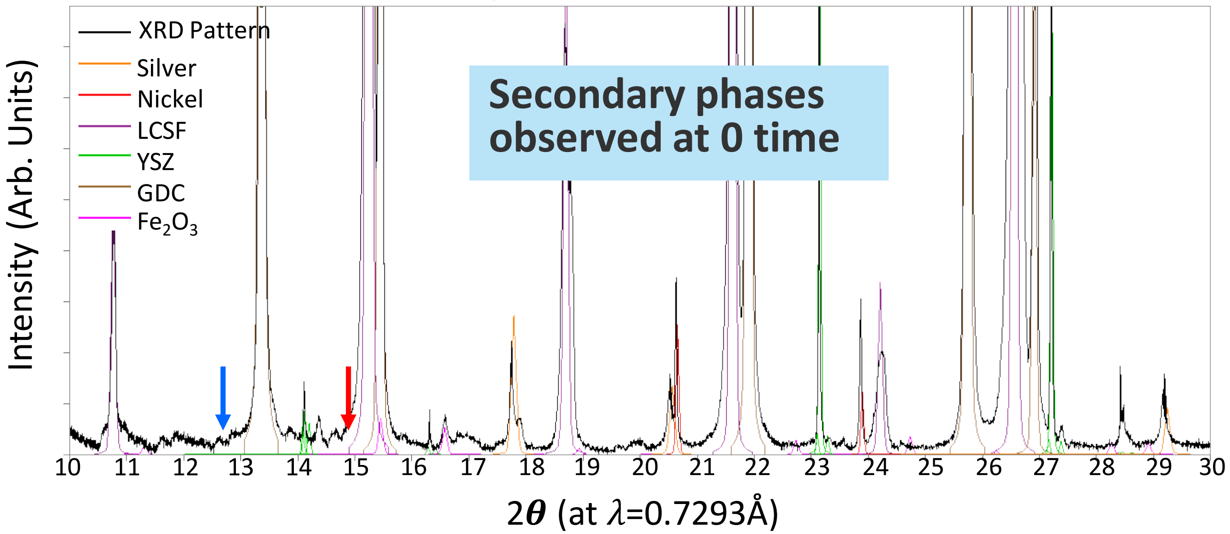
XRD Results – cell cycled 0 hours

- 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_2O_3 is left over from synthesis of GDC layer (red arrow)
  - Additional phase (blue arrow)

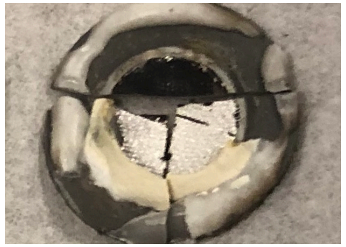
XRD of 0 hrs Cell

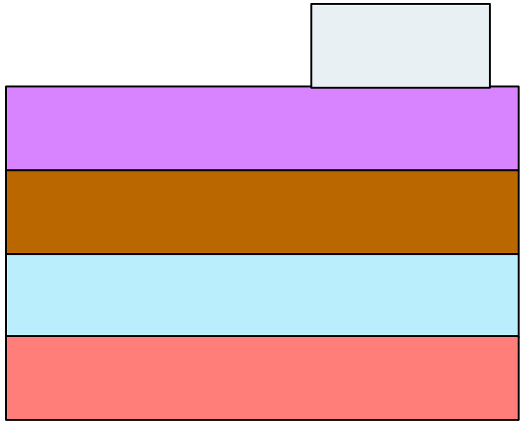


Magnified XRD of 0 hrs Cell



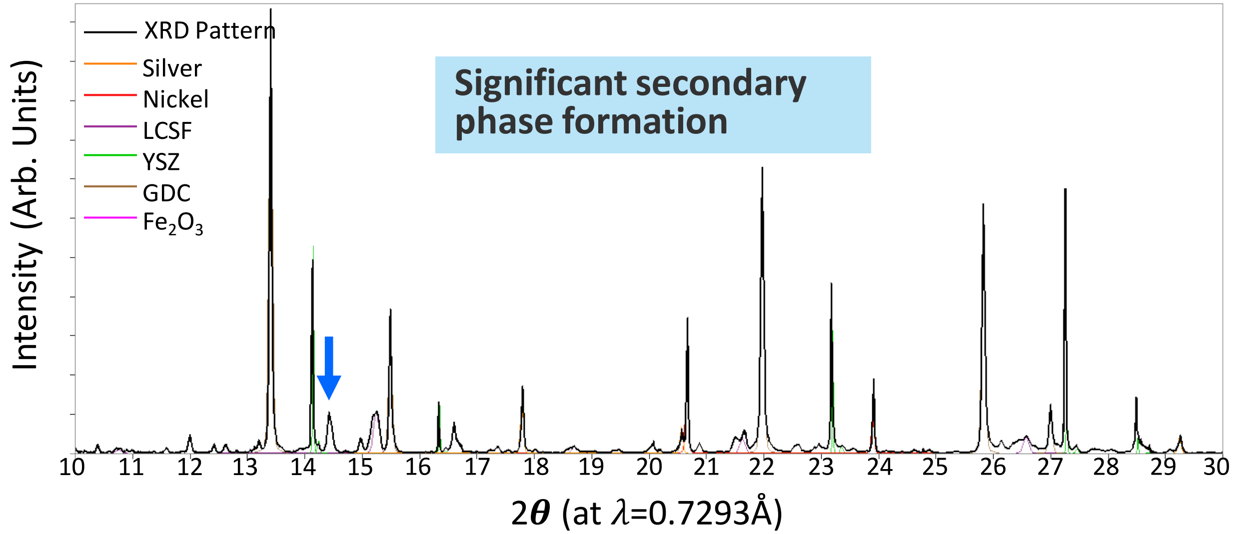
H2NEW HTE Task 7: Advanced Characterization – Key Results: XRD 300 hr cell

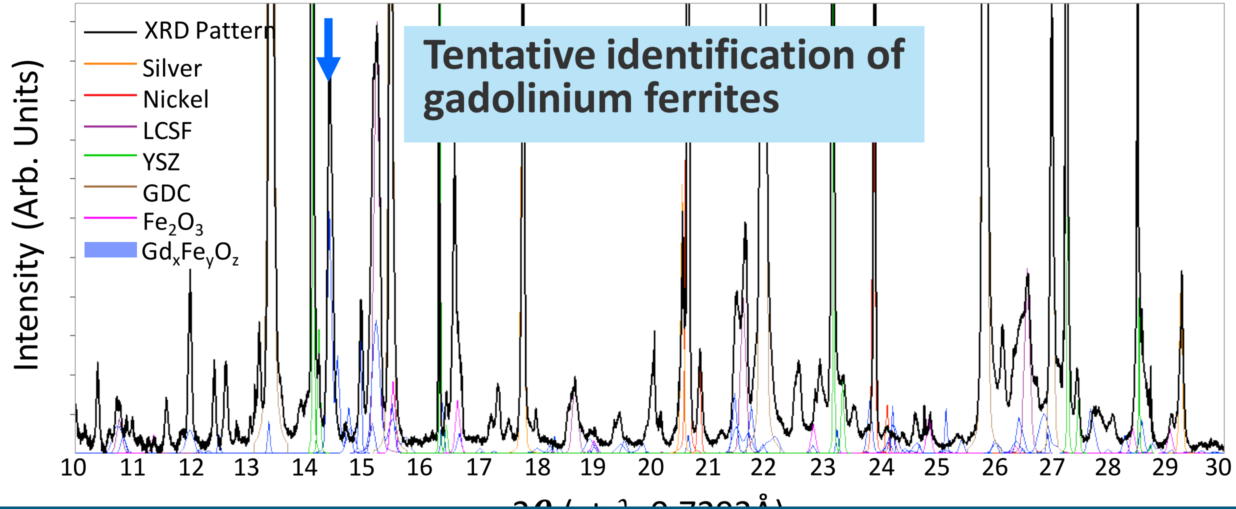




XRD Results – cell cycled 300 hours

- Secondary phases identified
- Secondary phase from match expected pattern from Gd_3FeO_6 ($Gd_xFe_yO_z$ family in blue)
  - Evidence suggests other Gd&Fe compounds forming at 300hrs
  - $GdFeO_3$/$Gd_3Fe_5O_{12}$
- Additional secondary phase peaks suggest additional decomposition reactions





H2NEW HTE Task 7: Advanced Characterization - Remaining Challenges, Future work

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Challenges

- Post-mortem processing can have unknow 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

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- 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

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- 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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