Project ID: p152

Energy Materials Network
U.S. Department of Energy

HydroGEN
Advanced Water Splitting Materials

Proton-Conducting Solid Oxide Electrolysis Cells for Large-scale Hydrogen Production at Intermediate Temperatures

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Olga Marina, Jeff Stevenson - Pacific Northwest National Laboratory

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NREL NATIONAL RENEWABLE ENERGY LABORATORY · BERKELEY LAB · Sandia National Laboratories · INL Idaho National Laboratory · Lawrence Livermore National Laboratory · SRNL

Technical Contributors

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	Michael Reisert	Graduate Student
	Junsung Hong	Graduate Student
PNNL	Jeffery Stevenson	Laboratory Fellow
	Olga Marina	Chief Scientist
INL	Dong Ding	Materials Engineer
	Henping Ding	Postdoctoral Fellow
NREL	Andriy Zakutayev	Staff Scientist
	Zhiwen Ma	Staff Scientist
	Su Jeong Heo	Postdoctoral Fellow

Program Manager: Dr. David Peterson, Department of Energy

Project Overview

Project Partners

UConn: Prabhakar Singh (PI), Boxun Hu, Ugur Pasaogullari (Co-PI)

PNNL: Olga Marina and Jeff Stevenson (Co-PI)

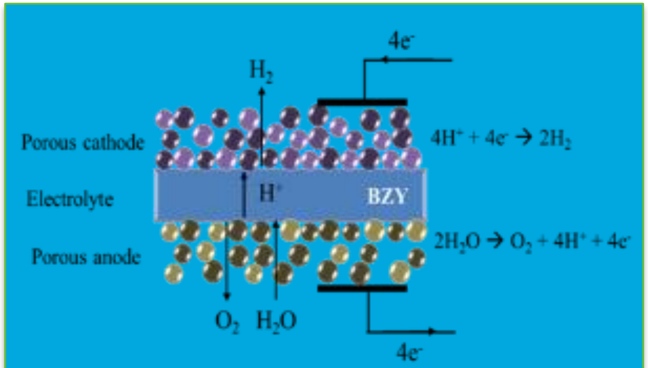
Project Vision

Identify novel materials and processing techniques to develop cost effective and efficient proton-conducting solid oxide electrolysis cells (H-SOECs) for large-scale hydrogen production at intermediate temperatures (600-800˚C) to meet DOE cost and performance targets.

Project Impact

(a) Innovation in materials chemistry – electrolyte and electrode formulations
(b) Use of non-noble and non-strategic cell and stack component materials
(c) Bulk, interface, and surface optimizations to achieve low ASR
(d) High proton-conductivity with a low sintering temperature (<1450˚C)
(e) Operating current density (>1 A/cm^2) with the performance degradation rate not to exceed the DOE performance metric (< 4 mV/1000 h)

Award #	EE0008078
Start/End Date	10/1/2017–9/30/2020
Total Project Value Cost Share %	$1.25M (DOE + Cost Share) 20%



- ➢ Higher conductivity (>0.01 s/cm) obtained
- ➢ Densification at <1400˚C achieved
- ➢ Uniform bulk phase composition obtained

Approach- Summary

Project Motivation

- Extensive background in HT/IT electrochemical systems
- Experience with functional ceramics, electrodics, electrochemical testing, performance degradation and data analysis
- Well established laboratory capabilities in materials processing and characterization
- On going research in SOFC,SOEC and H-SOEC

Key Impact

Metric	State of the Art	Proposed	Actual
Conductivity *(S/cm)*	*~10^{-3}*	*10^{-2}*	*4 x 10^{-2}*
Sintering temp. (˚C)	*>1450*	*≤1350*	*1350*
Thickness (µm)	>25	~15-20	20

Barriers

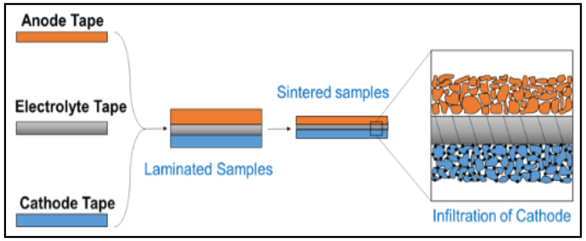
- High sintering temperature for electrolyte densification (>1400˚C)
- Decrease in conductivity during processing and operation
- High temperature gas sealing and operation with thermal cycling
- Complex processing and fabrication techniques
- Chemical and structural instability in presence of Cr, and Si contaminants

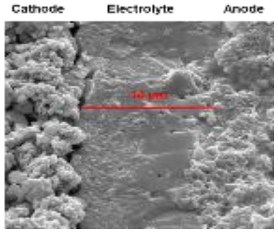
Partnerships

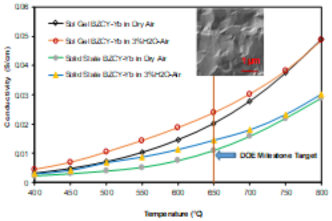
The research team collaborates with PNNL in developing and testing H-SOEC. Team will heavily leverage EMN network. We will work with NREL, INL and LBNL for the optimization of electrolyte chemistry.

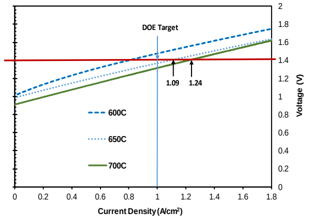
Approach- Innovation

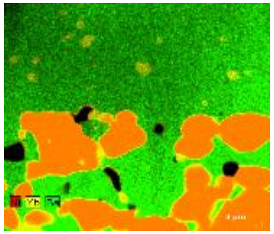
- Our approach for H-SOEC development leading to large scale manufacturing and commercialization will rely on utilizing EMN Network and core experimental and computational capabilities at NREL, INL and PNNL.
- Materials and Processes: Innovation in materials and processing techniques are anticipated to develop electrolyte formulations capable of densification (96-98%density) below 1400˚C in oxidizing atmospheres, meet electrical conductivity target (>0.01S/cm) and demonstrate bulk structural and chemical uniformity.
- Synthesis and fabrication processes: Cells utilizing tape cast multi-layer laminated electrolyte (10-20 μM) and electrode (integrated backbone, infiltration, thin film processing) will be sintered and electrically tested. Process will be optimized to achieve target ASR and current density to meet the overall project goals (1 A/cm^2 @1.4 V, 700˚C).
- Computational analysis: Electrolyte and electrode materials composition will be optimized for densification, proton conductivity and structural stability. Select electrode and electrolyte materials will be synthesized and electrochemically tested.
- Electrode poisoning and performance degradation mitigation: Electrode delamination and Cr assisted poisoning mechanisms will be developed. Mitigation approaches will be identified.











Relevance & Impact

- The proposed research program will develop cost effective and efficient solid oxide electrolysis cells consisting of novel thin film proton conducting electrolyte and tailored electrode materials for large-scale direct production of dry and pure hydrogen at intermediate temperatures using renewable energy. Developed materials will be electrically tested under SOEC systems operating conditions and mechanistic understanding for the performance degradation will be obtained.

- The research effort will benefit the global hydrogen producers, users and SOEC materials manufacturers. The developed technology will help the systems integrators to develop cost effective and stable materials, processing techniques and architectures for demonstrating and deployment of the SOEC technology for sustainable hydrogen production.

- Our approach for H-SOEC development leading to large-scale manufacturing and commercialization will rely on utilizing EMN Network and core experimental and computational capabilities at NREL, INL and PNNL.

- Key innovations include

  a) Computational materials design and optimization for chemically and structurally stable ceramic electrodes and electrolyte (NERL)

  b) Synthesis and fabrication processes for tailoring active components with enhanced catalytic sites and reduced area specific resistance (INL)

  c) Modelling and optimization of proton-conducting SOEC cell/stacks (NERL &PNNL).

  d) Mitigation of electrode delamination and chromium assisted poisoning (UConn).

Accomplishments

- Compositions and formulations of electrolyte and electrode materials have been developed using experimental and theoretical methods. Scale up of the powder synthesis process to produce 50-gram batch materials.
- Nanosized sintering aids have been selected and synthesized for dense electrolyte at low temperature (<1400°C).
- Highly conductive dense proton electrolyte has been sintered at low temperature (<1350°C). Contributions from proton, oxygen ion and electronic conductivities have been identified.
- Successful Fabrication of 1-inch diameter H-SOEC button cells using our developed electrolyte and electrode materials.
- Design experiment setup and utilize advanced characterization techniques. Structural and chemical degradation mechanisms operating under real-world steam electrolysis have been established.
- Graduate students were trained in the area of experimental methods and analytical tools. Post-doctoral fellows and the undergraduate students are also learning the SOEC technology, proton conducting oxide chemistry.
- Effectively utilized EMN Network and core experimental and computational capabilities at NREL, INL and PNNL. The overall program goals (M4-1 and GNG-BP1) of the Budget Period-2 and Go/No-Go Decision have been achieved.

Collaboration: Effectiveness

Multi-layer processing for large area cell fabrication (Node: Advanced Materials for Water Electrolysis at Elevated Temperature, Expert: Dr. Dong Ding)

The INL-UConn collaboration , INL spanned over the topics for the development of dense electrolyte and performance improvement of the anode. Technical discussions have been held with Dr. Ding with focus on materials selection, processing techniques and electrochemical performance evaluation.

The tasks for BP1 include:

Task 1: Development of electrolyte densification technique and determination of corresponding ionic conductivities (Q1-Q2).

INL also provided large size full cells (1.3 cm) and electrode materials for SOEC testing.

Task 2: Anode microstructural modification for performance improvement (Q3-Q4).

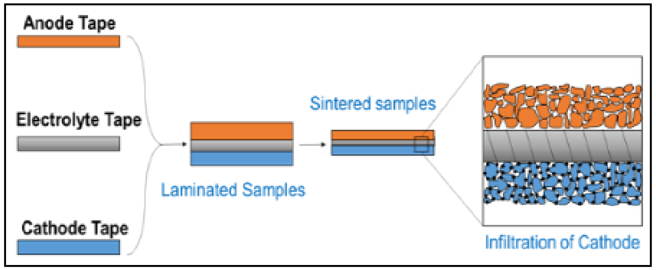
UConn have utilized INL capability to fabricate full cells with porous electrodes and thin electrolyte using HT-R2R technique with UConn-made electrode and electrolyte materials. The samples fabricated by (High Temperature Roll-to-Roll, HT-R2R) have been sintered and tested, and meet with project goals of ASR and current density.

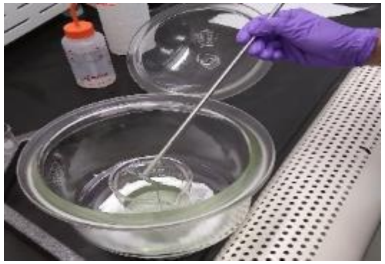
The tasks for BP2 include:

Task 3. Fabricate multilayer cells using roll to roll tape casting and sintering (2”X2”).

Task 4. Conduct quality assurance.

Task 5: Optimize electrode chemistry and structure.





Prepare BZCYYb slurry

roll to roll tape casting

Collaboration: Effectiveness

NERL High-Throughput Experimental Thin Film Combinatorial Capabilities (Experts: Drs. Andriy Zakutayev, John Perkins, David Ginley)

Technical discussion held with Dr. Andriy Zakutayev has identified scope of work for the development of electrolyte materials for sintering and conductivity optimization.

The tasks for BP1:

Task 1: The HTE combinatorial node at NREL is responsible for the investigation of combinatorial libraries of Y-substituted $BaZrO_3$ (BZY). Other minor additives (e.g. transition metals, alkali earth, rare-earth) that have a potential to improve BZY's sinterability without inducing secondary phases or impeding protonic and electronic charger transport.

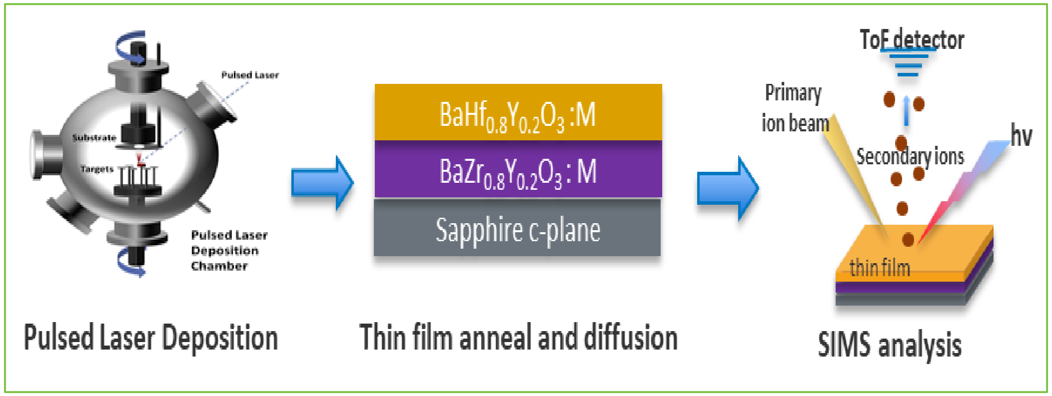
Task 2: The films are characterized at NREL for composition, structure, morphology, and electrical properties at room temperature. These NREL thin film results have been compared to the UConn bulk synthesis results, in order to determine how thin film morphology and ceramic sinterability correlate with each other. At later phases of the project, optimized thin film compositions may be deposited at NREL on ceramic or metallic supports provided by UConn.

The tasks for BP2:

Task 3: Using NREL combinatorial node, develop sintering aids to decrease sintering temperature.

Task 4: Conduct diffusion couple study to establish interfacial stability.

Task 5: Measure ASR using EIS technique and characterize phase stability and crystal structure using XRD.



Collaboration: Effectiveness

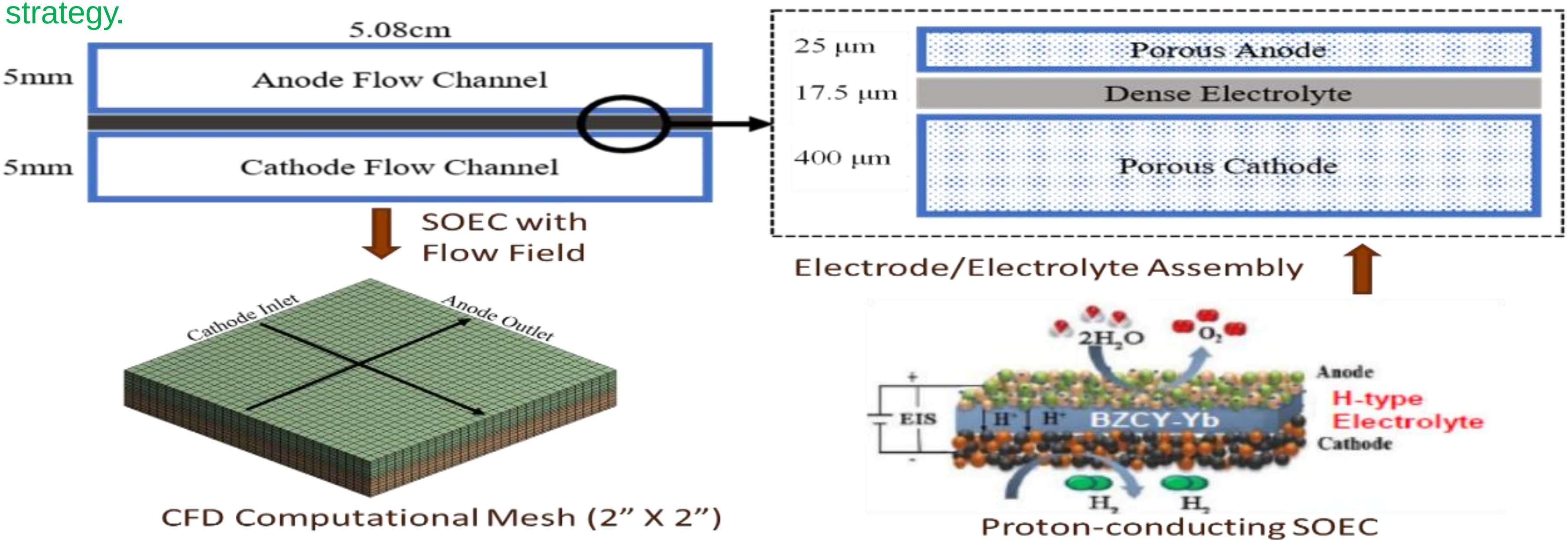
NERL: “Multi-Scale Thermochemical and Electrochemical Modeling for Material Scale-Up to Component and System Design” and “Techno-Economic Analysis of Hydrogen Production” (Expert: Dr. Zhiwen Ma)

The tasks for BP2:

Task 1. Develop and calibrate a SOEC electrochemical model based on cell materials and single cell testing data.

Task 2. Verify performance and economic model by predicting performance parameters including temperature, current density, and species distributions.

Task 3. Study various cell configurations and investigate scaling up approaches of the SOEC and operation strategy.



Synthesis of Electrolyte/Electrode Materials

Method A: Sol-gel synthesis

Method B: Solid-state synthesis

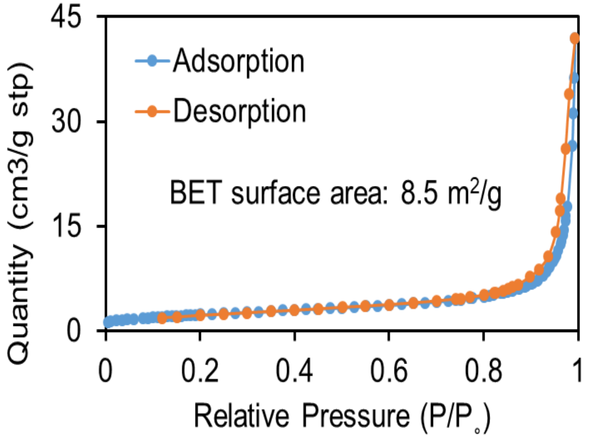
As-synthesized products

	Method A	Method B
Precursors	Nitrates of Ba, Zr, Ce, Y, and Yb	$BaCO_3$, ZrO_2, CeO_2, Yb_2O_3, and Y_2O_3
Temperature	25-85˚C	1100˚C
Reaction time	Sol-gel: 4 h, combustion<5mins	Ball mill >24 h
Total time for batch	Sol-gel-combustion<8 h	milling-drying-calcination>48 h
Sample quality	Small size, higher conductivity	Large particle size, lower conductivity
Energy/cost savings	Less energy consumption and cost	High energy ball mill and longer sinter time

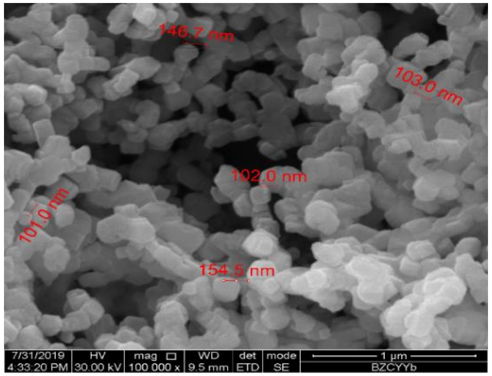
50-gram batch synthesis of BZCY-Yb proton-conducting materials has been validated using Sol-gel method. Quality has been ensured for different batch products.

Characterization: Electrolyte/Electrode Powder

BET Results of BZCYYb

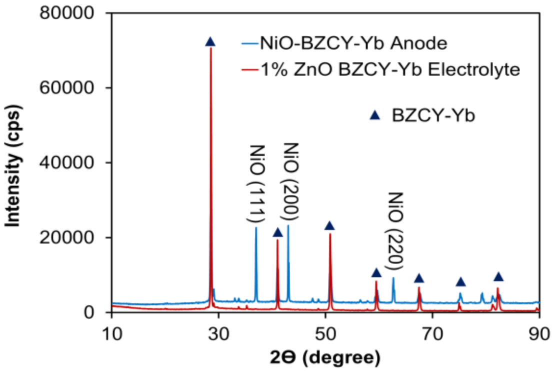


SEM image of BZCYYb

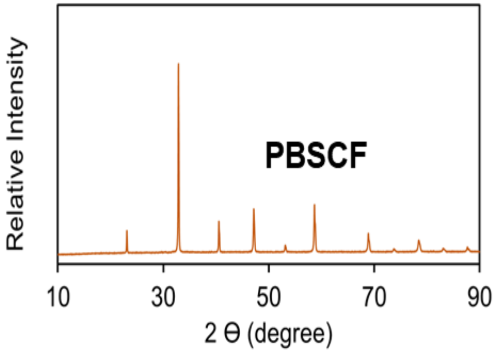


Instrument for BET

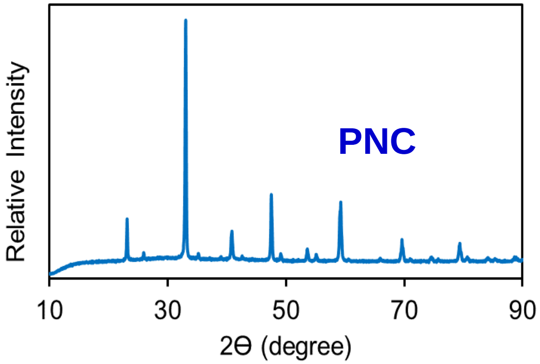
XRD Patterns of (NiO) BZCYYb



XRD pattern of PBSCF



XRD Pattern of PNC powder

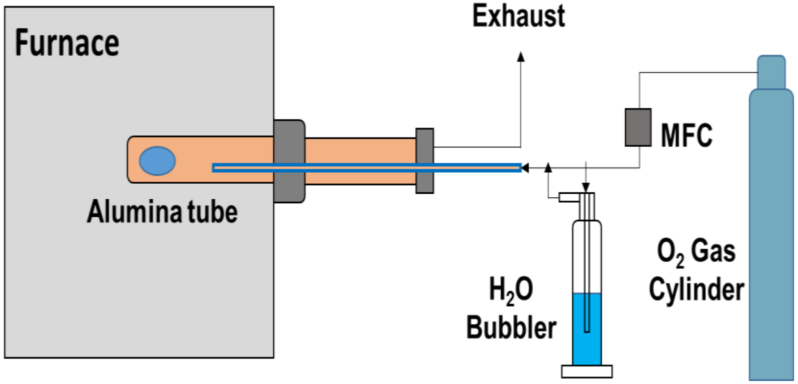


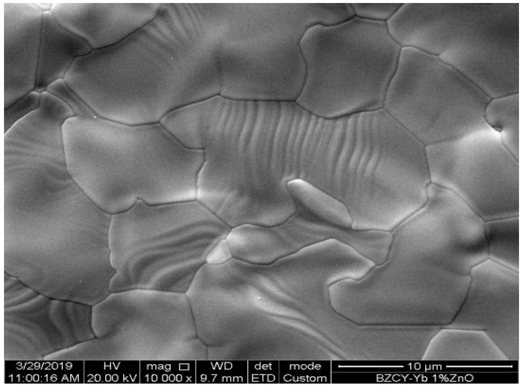
- **Morphologies and surface areas of cell components have been analyzed.**
- **Structure and phase purity of cell components have been determined.**

$BaZr_{0.1}Ce_{0.7}Y_{0.1}Yb_{0.1}O_{3-\delta}$ (BZCY-Yb, 1711), $PrBa_{0.5}Sr_{0.5}Co_{1.5}Fe_{0.5}O_{5+\delta}$ (PBSCF), PrNiCoOx (PNC)

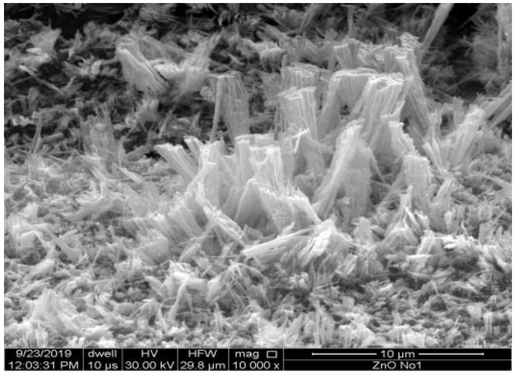
Nanowire Sintering Aids for Dense Electrolyte

Sintering BZCY-Yb at different gases/PO_2



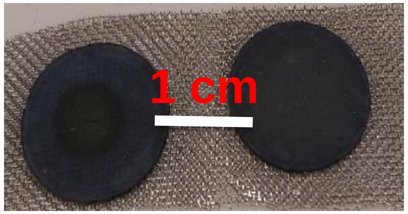


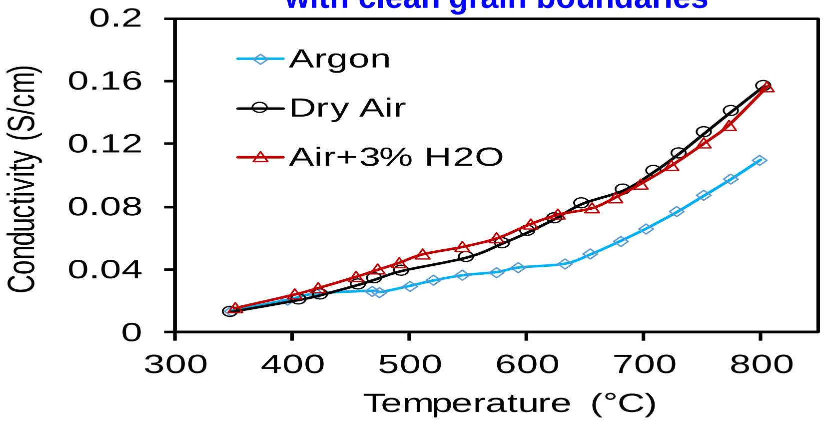
BZCYYb sintered at 1350C with clean grain boundaries



Nanosize ZnO synthiszed at UConn

Sintered Dense electrolyte with ZnO

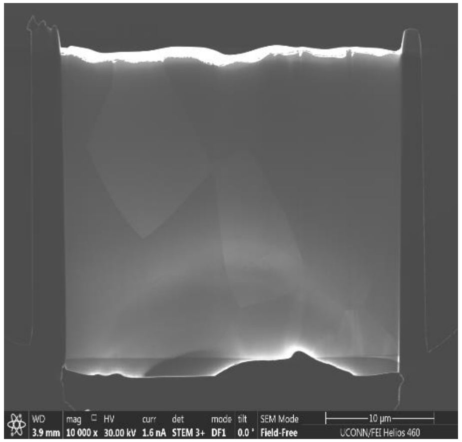




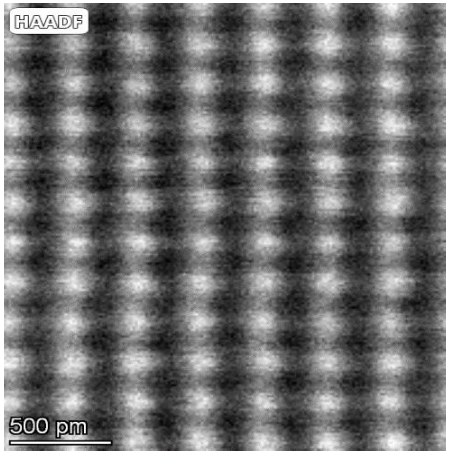
BZCY-Yb conductivity measured by 4-probe method

Select BZCY-Yb powder from large scale (>50 g/batch) synthesis was mixed with 1 wt% of nanosized ZnO and isostatically pressed, then sintered at temperatures ≤1350˚C. Sintered BZCYYb disc shows high total conductivity (>0.02 s/cm, 700˚C) meeting with Millstone M5-1. The contributions from proton, oxygen ion and electronic conductivities are identified in different gases/PO_2.

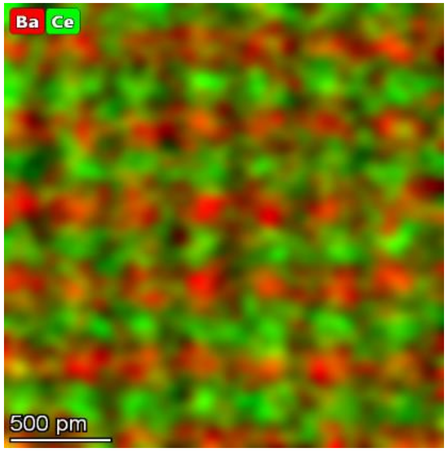
BZCYYb Structure Characterized by FIB-STEM



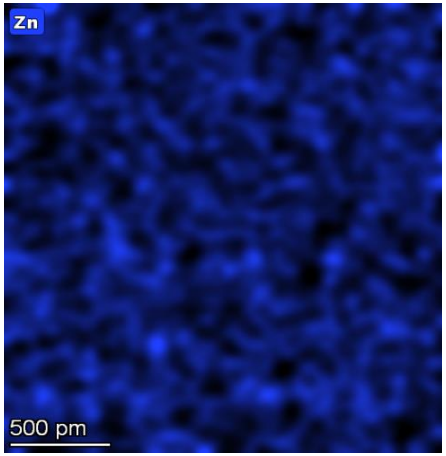
An FIB sample of BZCYYb



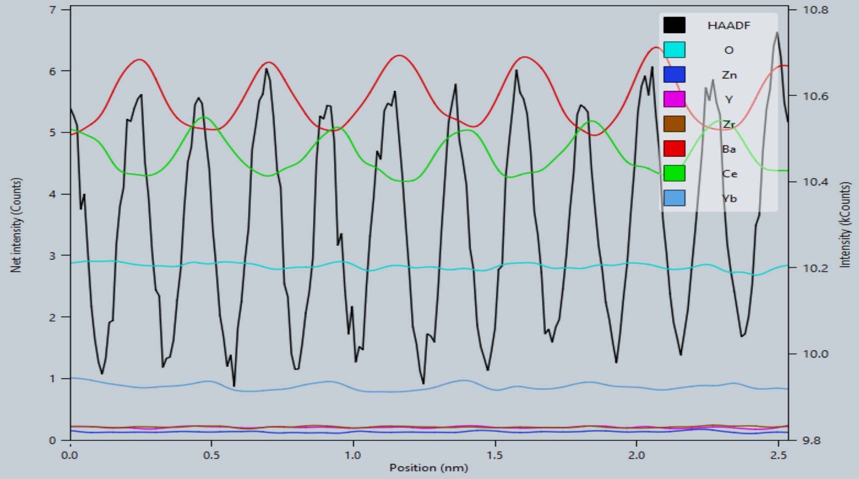
Atomic STEM-EDX image



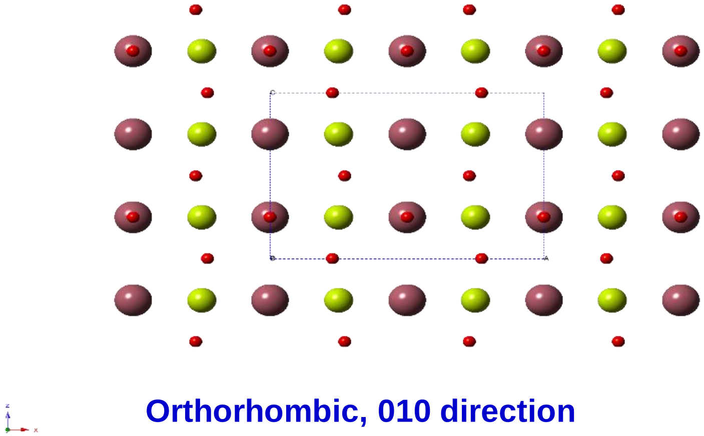
Combined maps of Ba & Ce



STEM-EDX map of Zn



Simulated Diffraction pattern by Crystal Maker



Orthorhombic, 010 direction

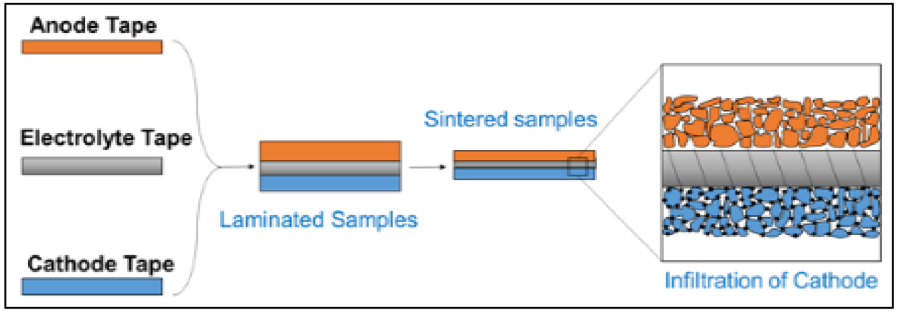
Select area TEM/EDS maps show a uniform and pure perovskite phase (ABO_3) formation with uniform elemental distributions of Ba, Zr, Ce, Y, Yb, and Zn in entire select area. Atomic resolution STEM maps confirm that barium occupies A sites and other cations (Ce, Zr, Y, Yb and Zn) occupy the B sites.

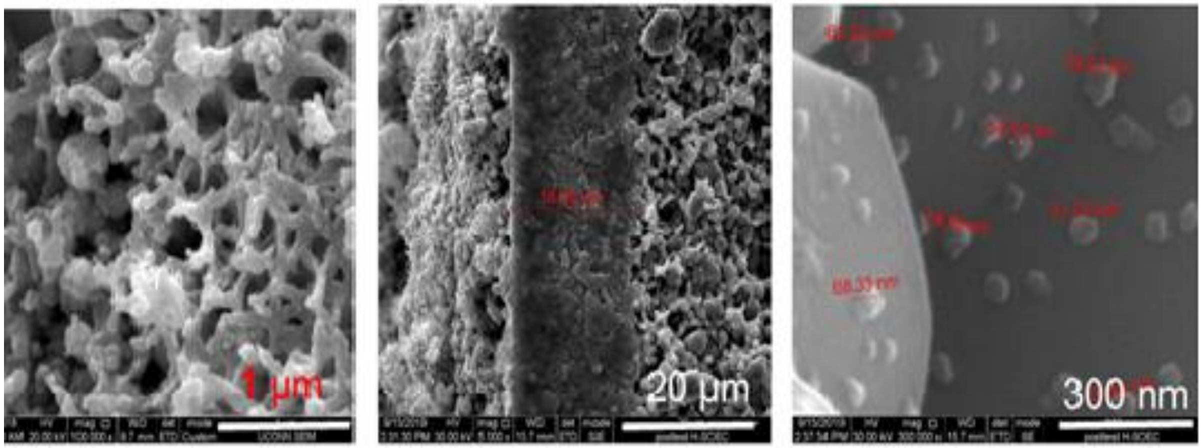
Fabrication and Characterization of Full Cells

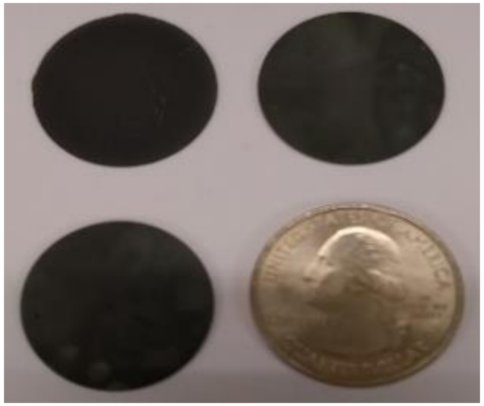
Processes of ½-Inch Diameter H-SOEC fabricated at UConn

Hydrogen electrode	BZCYYb Electrolyte	Oxygen electrode
Co-pressing & pre-sintering	drop coating & sintering	Screen-printing & sintering

Processes of 1-inch Diameter H-SOEC Fabricated by Tape Casting at INL





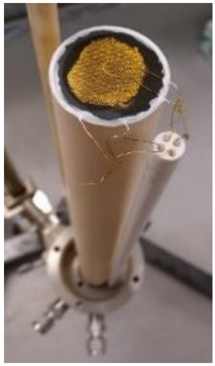


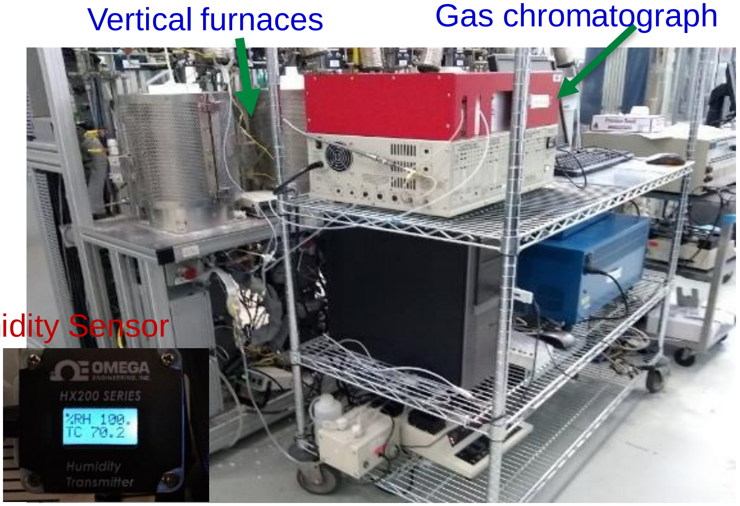
1-inch H-SOEC full cells

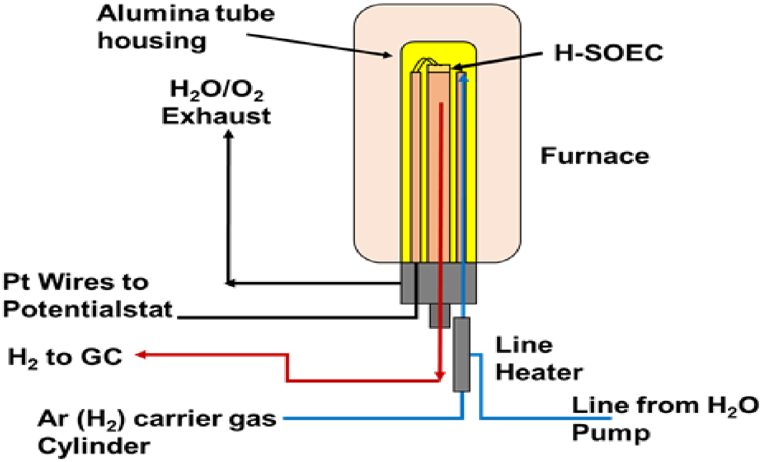
- **Large size H-SOEC full cells have been fabricated with thin dense electrolyte (15-20 µm).**
- **Structure and composition of full cells have been characterized by XRD, SEM, Raman and TEM.**
- **Reproducible materials composition have been obtained during synthesis.**

Experimental Design for Steam Electrolysis Testing

Reactor for 1-inch cell





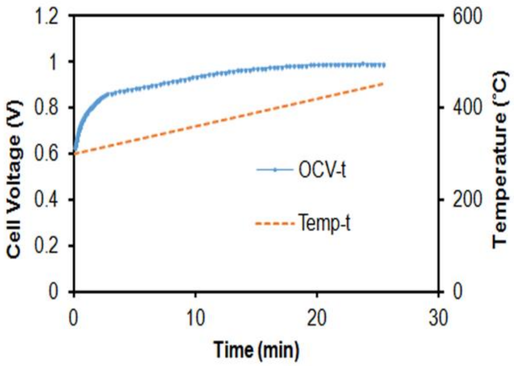


Performance Analysis → Materials Stability → Mechanisms

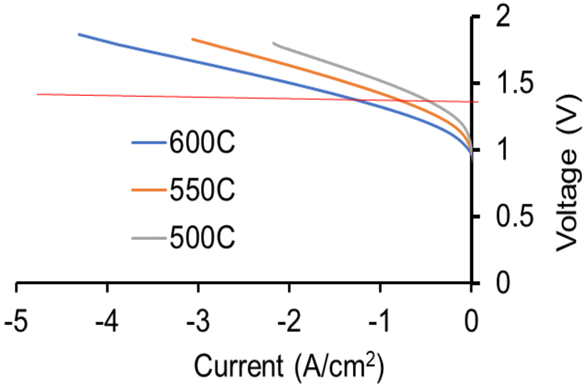
Testing Condition Matrix for H-SOEC Cell

Cell No.	Type of Electrolyte	Type of O-electrode	Operating Temperature	Steam%	Voltage(v)	Status
1	YSZ	LSM	800˚C	10%	1.3	Complete
2-10	BZCY-Yb	PBSCF	550-750˚C	3-50%	1.2-1.8	Complete
11-20	BZCY-Yb-ZnO	PBSCF	550-750˚C	3-75%	1.2-1.8	Complete
21-22	BZCY-Yb-ZnO	PLBC	550-750˚C	3-75%	1.2-1.8	In process
23-30	BZCY-ZnO	PNC	550-750˚C	3-75%	1.2-1.8	In process

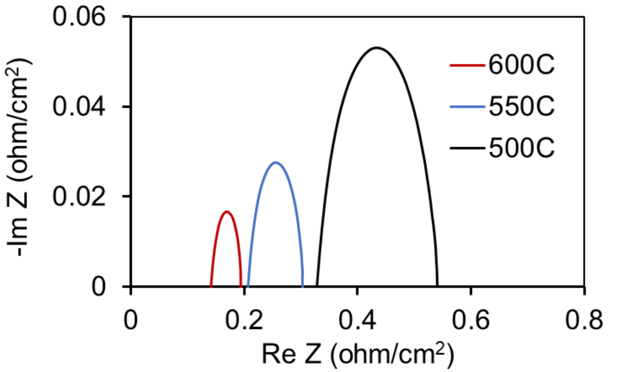
Performance-Stability: 1-Inch Diameter H-SOECs



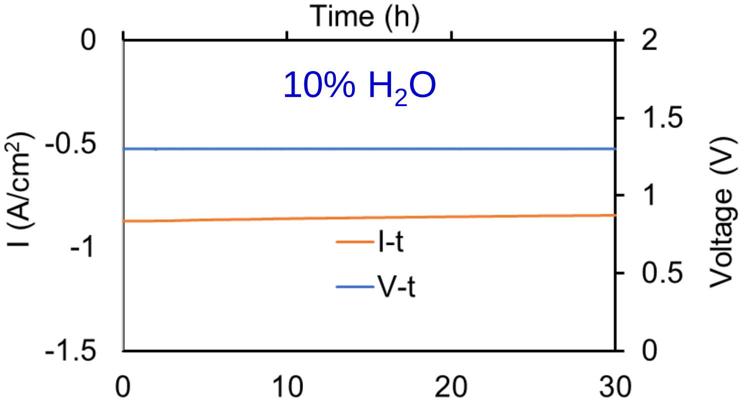
OCV >1.05 V at 500˚C



I >1 A/cm^2 at 600˚C



Low ASR at low/intermediate temperature

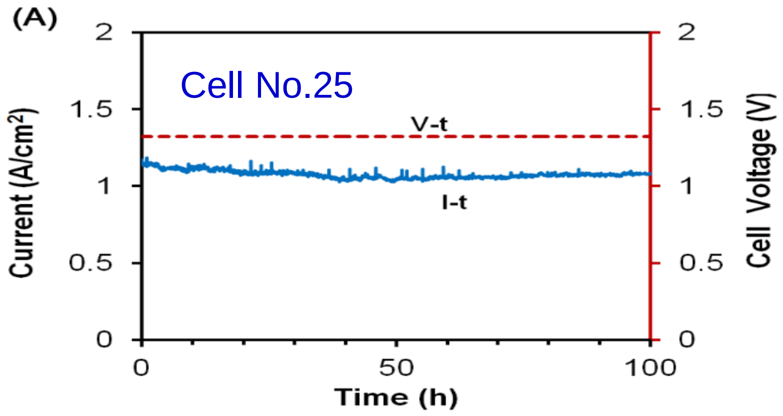


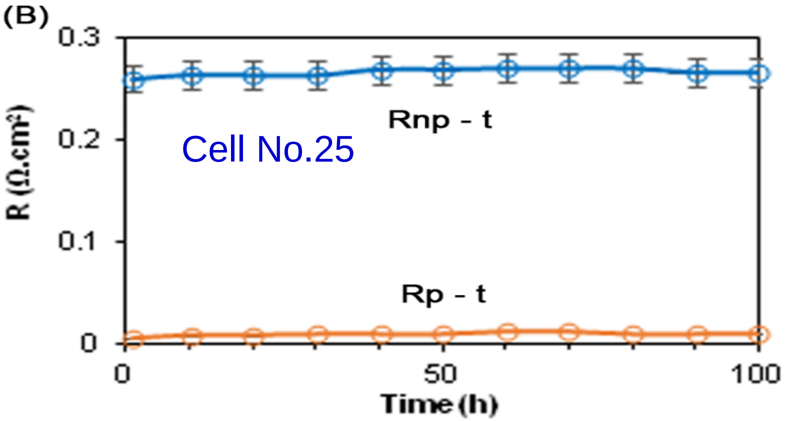
Ni-BZCY-Yb‖BZCY-Yb‖PNC, H electrode: 60 sccm H_2, O electrode: 120 sccm H_2O/air

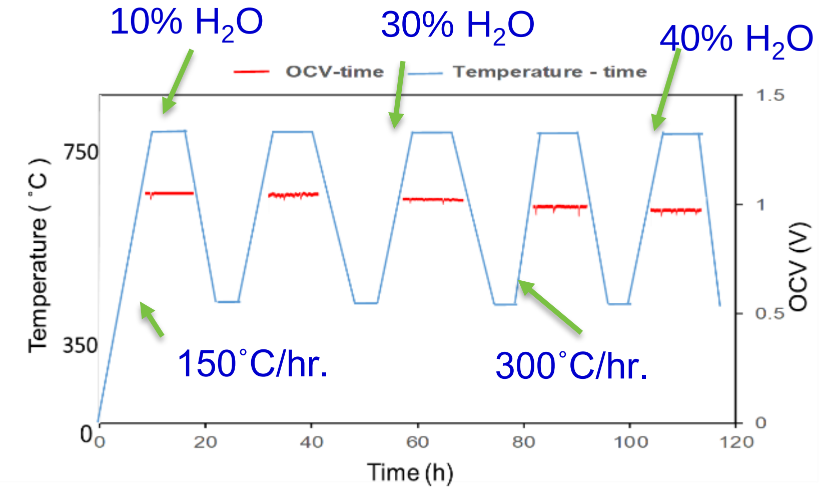
Relatively stable performance observed in 30-h test. Long term chemical/ structural stability in higher H_2O% needs to be investigated.

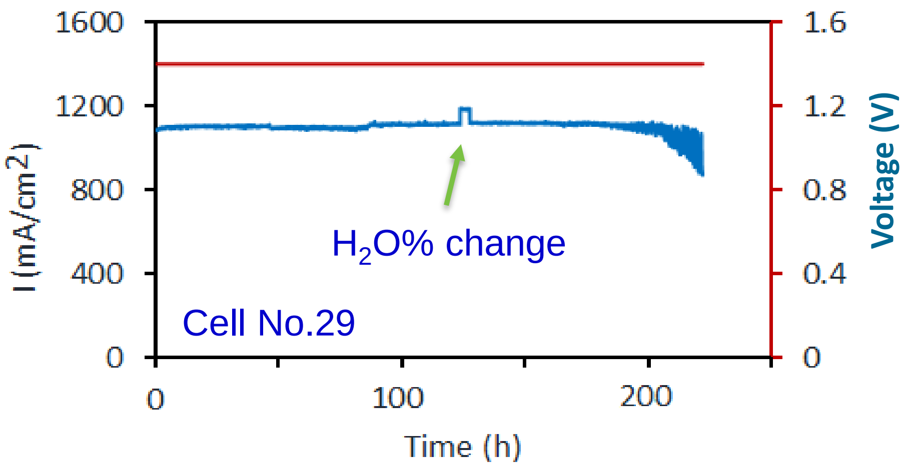
Current density of 1-inch diameter H-SOECs reaches above 1 A/cm^2 at ≤ 1.4 V and temperature of ≤ 650 ^{o}C.

Performance Stability of 1-Inch Diameter H-SOECs









Tested 1-inch diameter cells demonstrate stable electrolysis performance (<25 mV/1000 h) for 200 hours. Thermal cycle shows that sealing is relatively stable in five thermal cycles.

Hanping Ding, Dong Ding, Boxun Hu, Prabhakar Singh, et al. Self-Sustainable Protonic Ceramic Electrochemical Cells Using A Triple-Phase Conducting Electrode for Hydrogen and Power Production, manuscript submitted to *Nature Communication*.

Summary of Phase II Work

- Scale up of the powder synthesis process to produce 50-gram batch proton-conducting electrolyte (BZY and BZCY-Yb) and electrode materials by sol-gel method at UConn with conductivity of 0.065 S.cm^{-1} at 650° C (greater than the target: 0.02 S.cm^{-1}). Electrolyte powder has been sent to PNNL for characterization.
- Examination of the sintering effect of ZnO and MnO on BZY: M (M: Zn, Mn) film samples by pulsed laser deposition at NERL using XRD and SEM techniques.
- FIB-STEM analysis confirming a uniform and pure perovskite phase (ABO_3) formation and atomic resolution STEM maps showing barium occupying A site and other cations (Ce, Zr, Y, Yb and Zn) occupying the B site.
- Performance evaluation of 1-inch H-SOEC full cell at a current density of 1.1 A/cm^2 @ 1.4 V and achieving low area specific resistance (0.2 ohm/cm^2, 600˚C) along with chemical/structural stability for 200 hours in high temperature steam electrolysis condition.
- The degradation mechanisms have been developed by operando analysis (EIS) and post-characterization (SEM, XRD, and FIB-TEM), meeting with the Milestone M6-1
- Electrochemical modeling of the H-SOEC cell performance conducted by NERL and PNNL using UConn experimental data incorporated in the CFD software Fluent able to model the SOEC performance on cell and stack level. The model simulates scaling-up cell performance and parameter distributions, and has good agreement with experimental data.
- The overall program goals (M5-1, M6-1, and M7-1) of the Budget Period-2 and Go/No-Go Decision have been achieved.

Future Work

The overall objective of the proposed development efforts under phase II includes

(a) Optimize materials chemistry and processing conditions utilizing the NERL node.
  - Lower electrode polarization and resistive losses (ASR< 0.4 Ω/cm^2 at 650ºC).
  - Increase proton conductivity(>0.02 $S.cm^{-1}$ at 650ºC)
  - Identify contributions from proton, oxygen ion and electronic conductivities, measure transference number.
  - Decrease the cell degradation rate using Cr getters and advanced oxygen electrode materials.

(b) Fabricate large button cells(25 cm^2) and 3 cell stacks using tape casting utilizing the INL node.

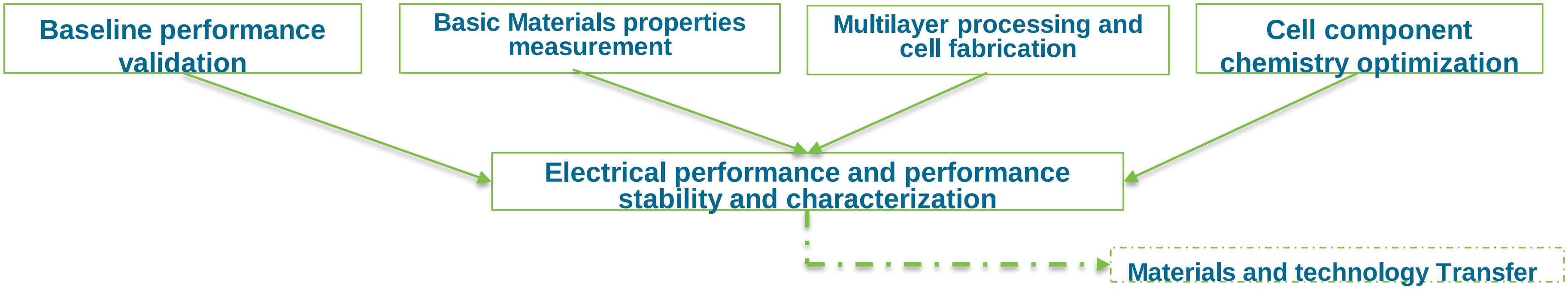
(c) Examine and validate long term (>500 h) chemical and structural stability of cell component materials, large cells, and 3 cell stacks in the high temperature steam electrolysis.

(a) Demonstrate performance stability and project target: degradation rate <4 mV/1000 h at 1 A/cm^2, electrical efficiency >95%, and cost of hydrogen production < \$2/gge H_2.

(b) Characterize cell components (as fabricated and post tested) for degradation mechanisms.

(c) Develop mechanistic understanding of electrochemical / electrodics processes.

(d) Modelling and economic analysis of large-scale hydrogen production using NERL node (Dr. Zhiwen Ma).
  - Develop and calibrate a SOEC electrochemical model based on cell materials and single cell testing data.
  - Economic model by predicting performance parameters including temperature, current density, and species distributions.



Baseline performance validation

Basic Materials properties measurement

Multilayer processing and cell fabrication

Cell component chemistry optimization

Electrical performance and performance stability and characterization

Materials and technology Transfer

Publications & Presentations

List of publications and presentations that have resulted from this project:

Paper submitted and in preparation:

- Hanping Ding, Dong Ding, Boxun Hu, Prabhakar Singh, et al. Self-Sustainable Protonic Ceramic Electrochemical Cells Using A Triple-Phase Conducting Electrode for Hydrogen and Power Production, manuscript submitted to *Nature Communication*, accepted.
- Boxun Hu, Michael Reisert, Ashish Aphale, Seraphim Belko, Olga Marina, Jeff Stevenson, Dong Ding and Prabhakar Singh, "Hydrogen Production by intermediate temperature steam electrolysis using Proton-conducting Solid Oxide Electrolysis Cells Sintered at Low Temperatures with nanosized Sintering Aids", manuscript in preparation.

Presentations:

- Boxun Hu, Olga Marina, Seraphim Belko, Michael Reisert, Ashish Aphale, Junsung Hong, Dong Ding, Hanping Ding, Zhiwen Ma, Prabhakar Singh, Stability of Proton-conducting Solid Oxide Electrolyzers for Hydrogen Production and Energy Storage, Electrosnthesis of Fuels, 237th ECS Meeting and Exhibit May 10-15, 2020, abstracts accepted.
- Boxun Hu, Olga A Marina, Ashish N Aphale, Dong Ding, Hanping Ding, Andriy Zakutayev, Jeffry Stevenson, Prabhakar Singh, Stable Proton-conducting Solid Oxide Electrolysis Cells for Pure Hydrogen Production at Intermediate Temperatures, 2019 Materials Research Society Spring Symposia on Advanced Water Splitting, April 22–26, 2019, Phoenix, Arizona.
- Boxun Hu, Michael Reisert, Ashish Aphale, Seraphim Belko, Olga Marina, Jeff Stevenson, Dong Ding and Prabhakar Singh, Barium Zirconate Based Electrolyte Densification Using Reactive Sintering Aids, 43rd International Conference and Exposition on Advanced Ceramics and Composites (ICACC 2019), Daytona FL, Jan. 25-31, 2019.
- Boxun Hu, Ashish N Aphale, Michael Reisert a, Seraphim Belko, Olga A Marina, Jeffry Stevenson, and Prabhakar Singh, Solid Oxide Electrolysis for Hydrogen Production: From Oxygen Ion to Proton Conducting Cells, 233rd ECS Meeting, Seattle, WA, May 13 -17, 2018.

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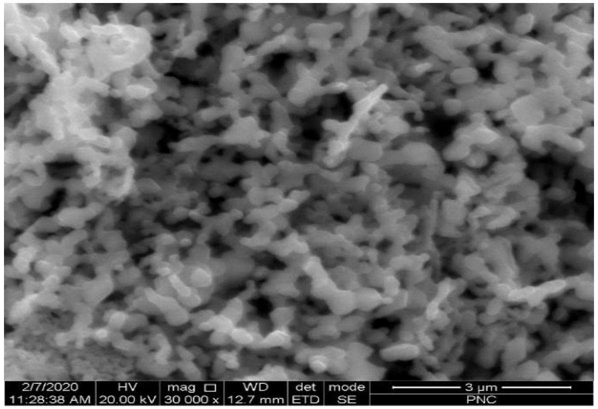
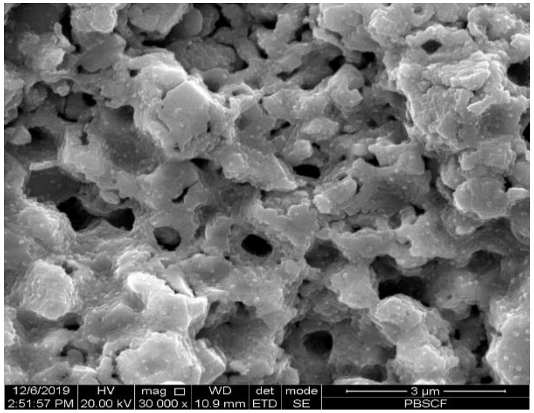
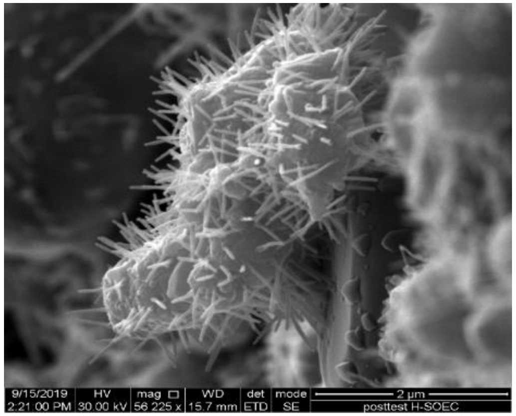
Degradation Mechanisms: Oxygen Electrode



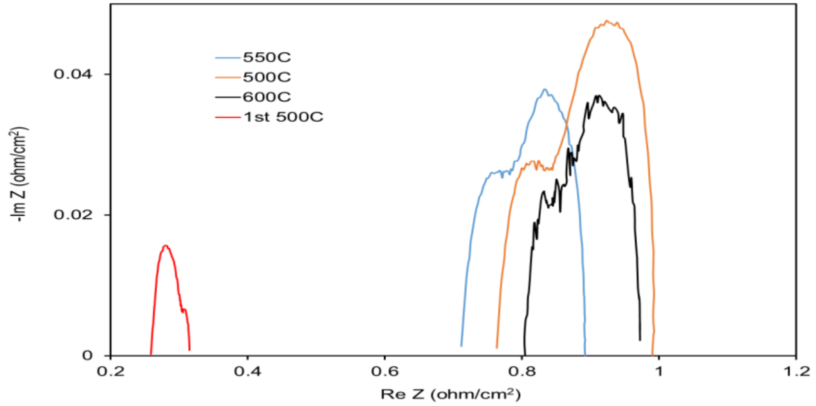
Posttest PNC Electrode

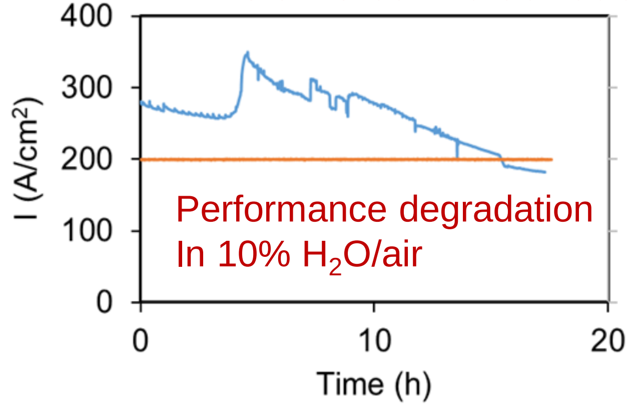


Posttest PBSCF Electrode (3% H2O/air)



SrO segregation on PBSCF steam electrode





Performance degradation In 10% H_2O/air

PBSCF is stable at low steam concentrations (~3% H2O).
PNC is more stable in high steam concentration than PBSCF.

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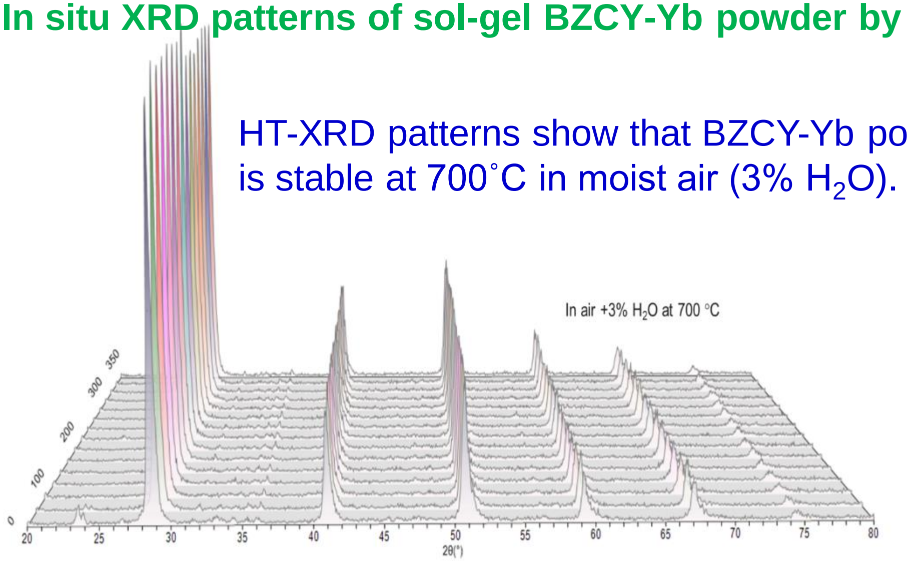
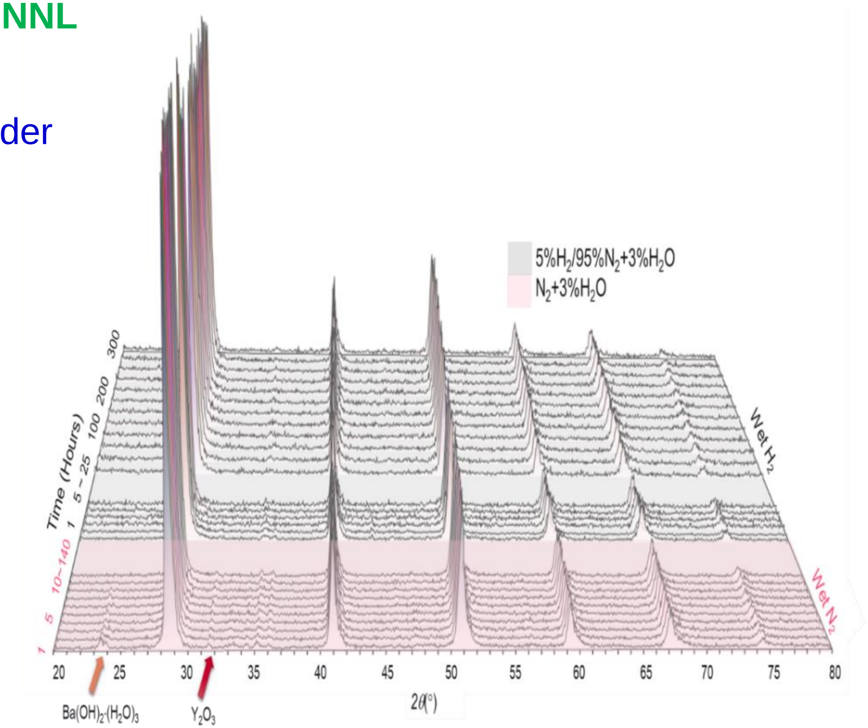
Degradation Mechanisms: Electrolyte

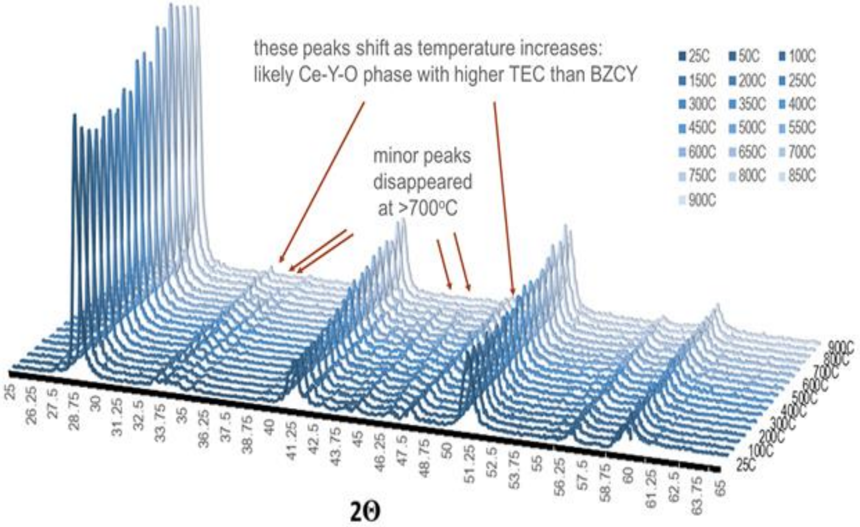
In situ XRD patterns of sol-gel BZCY-Yb powder by PNNL



HT-XRD patterns show that BZCY-Yb powder is stable at 700˚C in moist air (3% H_2O).



Hydroxide/oxide may presence in N_2/3% H_2O. But they disappear in 5% H_2/3% H_2O.



Sol-gel BZCY-Yb powder remains stable in reducing (H_2) atmosphere under SOEC operating conditions

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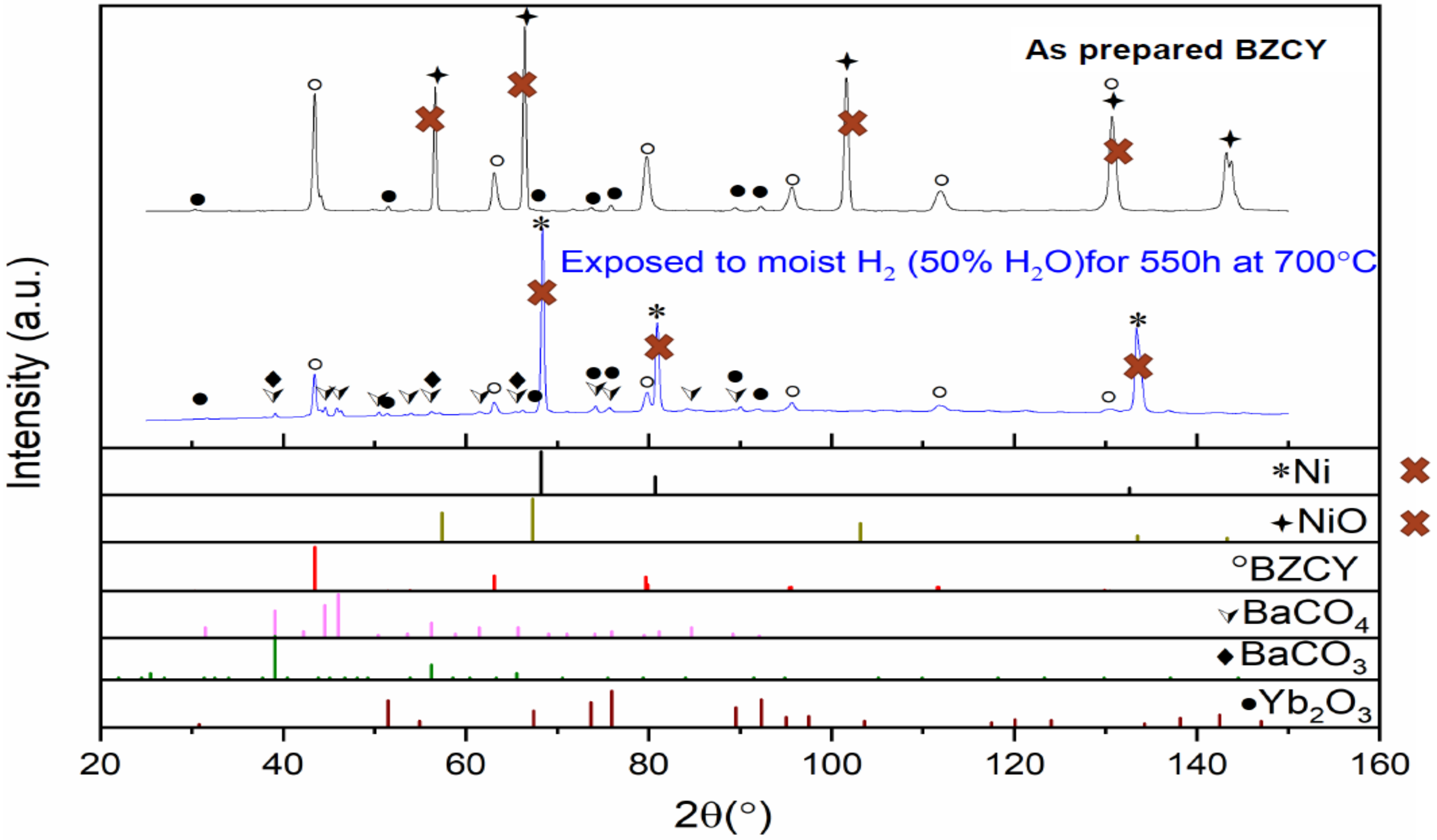
Degradation Mechanisms: Hydrogen Electrode

In situ XRD patterns of sol-gel BZCY-Yb powder by PNNL



Stability of NiO-BZCYYb in 50% Steam

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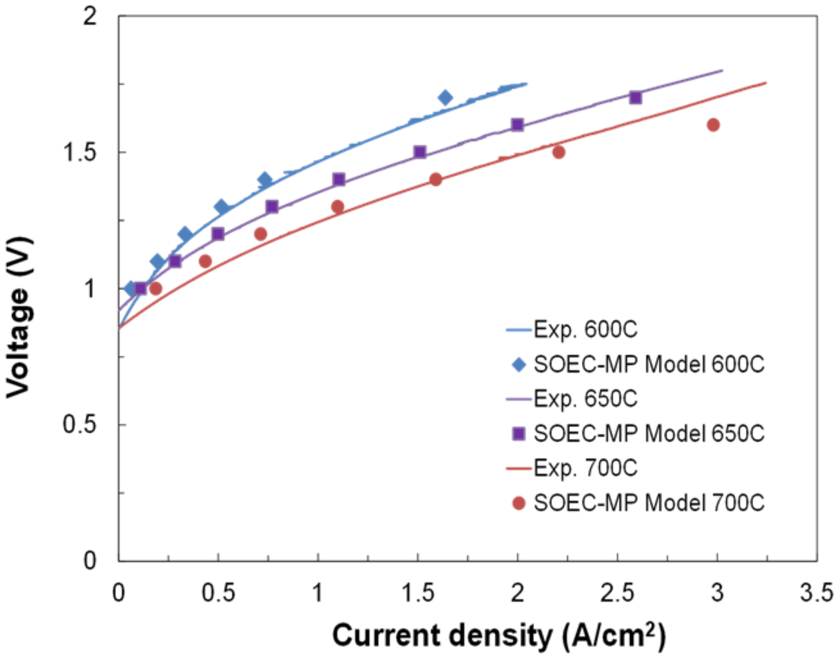
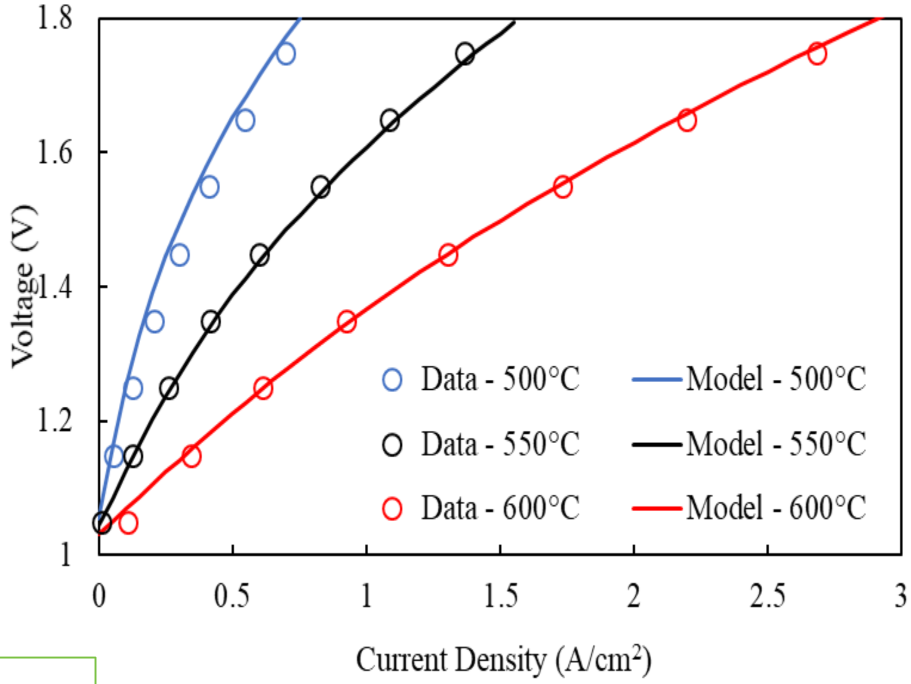
Comparison: Experimental/Modelling Results

Modelling data of H-SOEC I-V (conducted by PNNL team)



Modelling data of H-SOEC I-V (conducted by NREL team)



Modelling Methodology

- Describe SOEC current-voltage (I-V) electrochemical performance with expression that characterizes button cell data.
- Calibrate the I-V relationship to match the experimental data from different sources.
- In the future, it would be possible to modify the stack modeling tool SOFC-MP (including I-V relation) to simulate SOEC stack operation.

Experimental I-V curves at intermediate temperatures (500-700°C) match well with modelling I-V curves conducted at PNNL and NREL.

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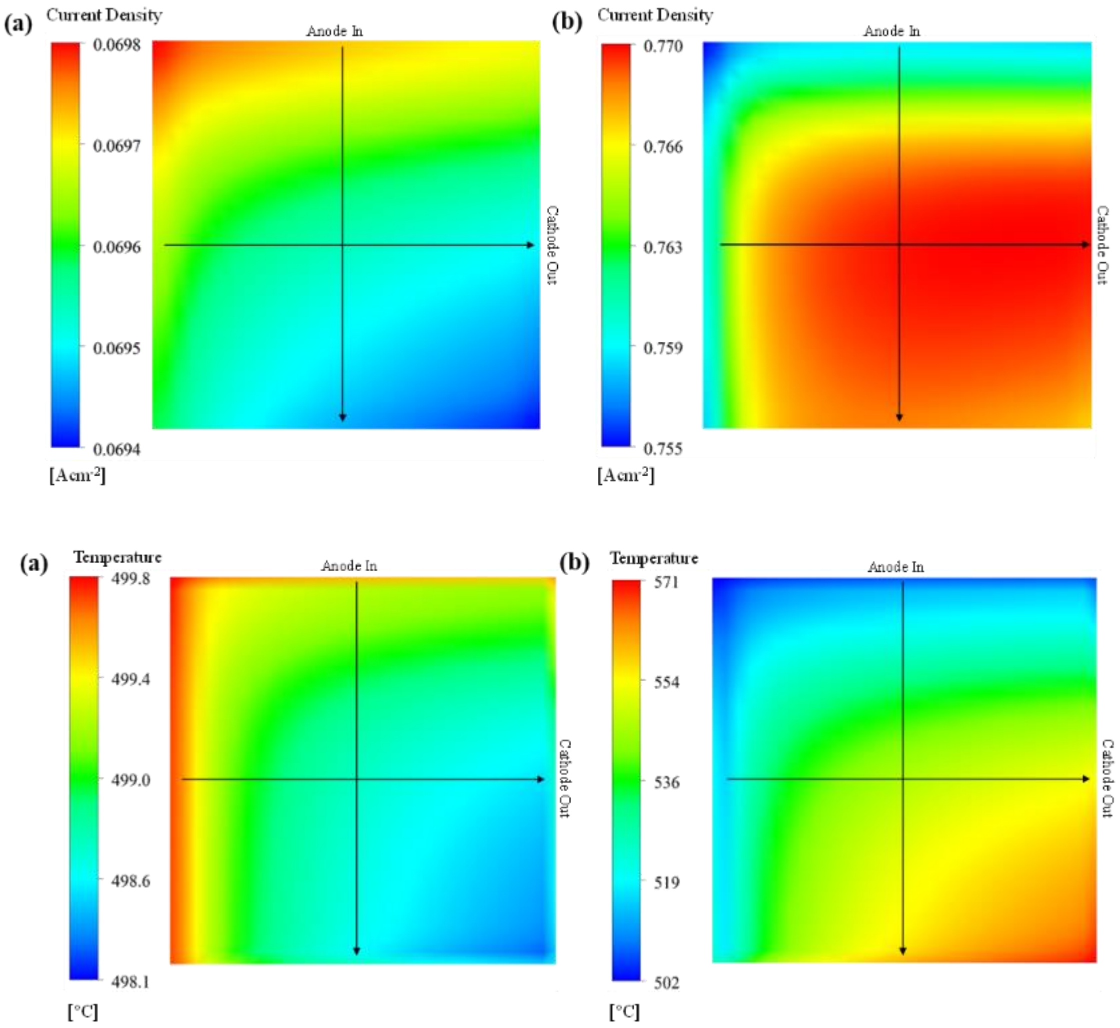
Modeling of H-SOECs for Steam electrolysis



From NREL

- 2” X2” cross flow cell configuration.
- Two cell applied voltages: 1.2 V and 1.8 V at 500°C:
  - Endothermic process shown in low current case.
  - High current case shows exothermic cell heating.

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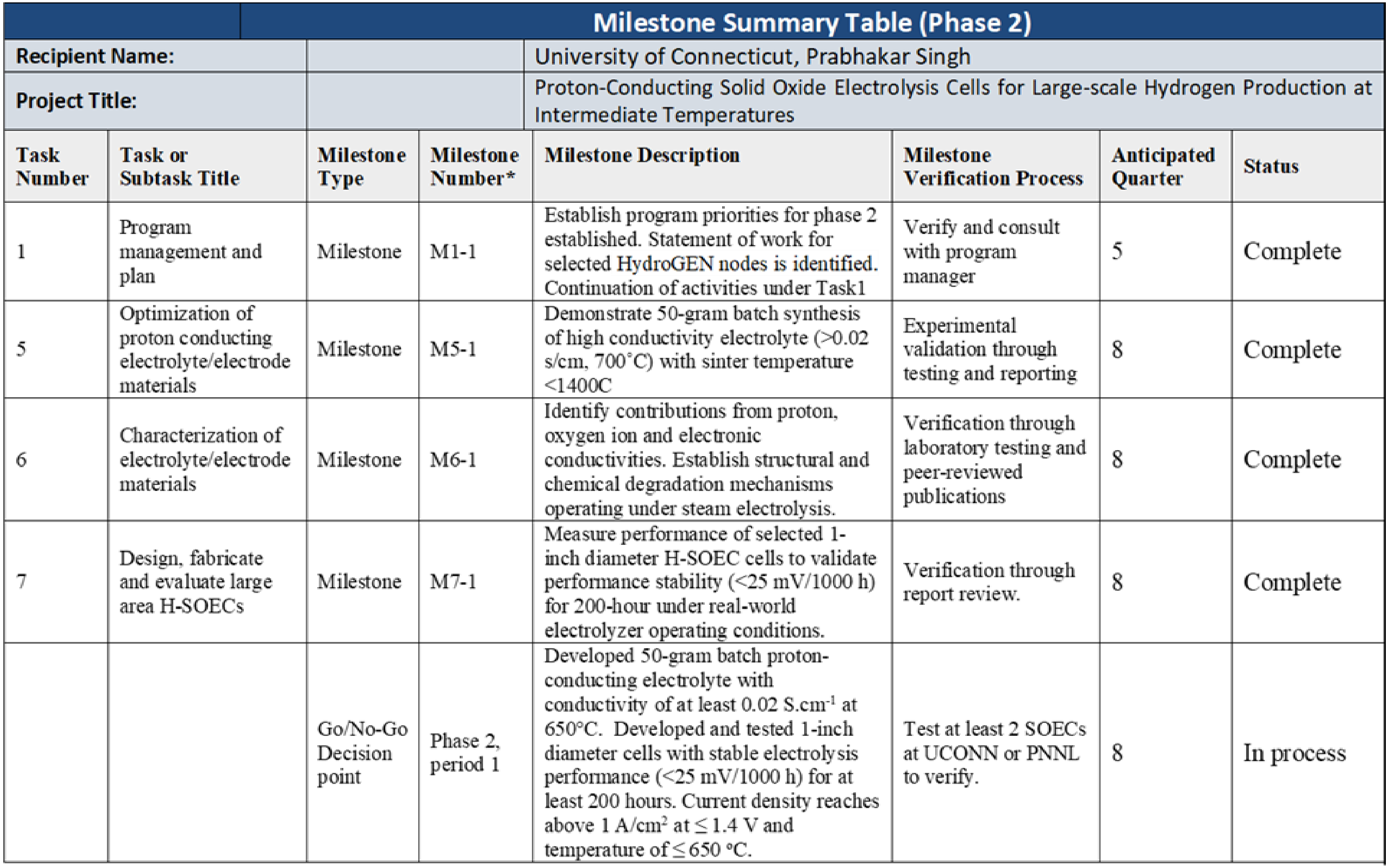
Milestones and Status



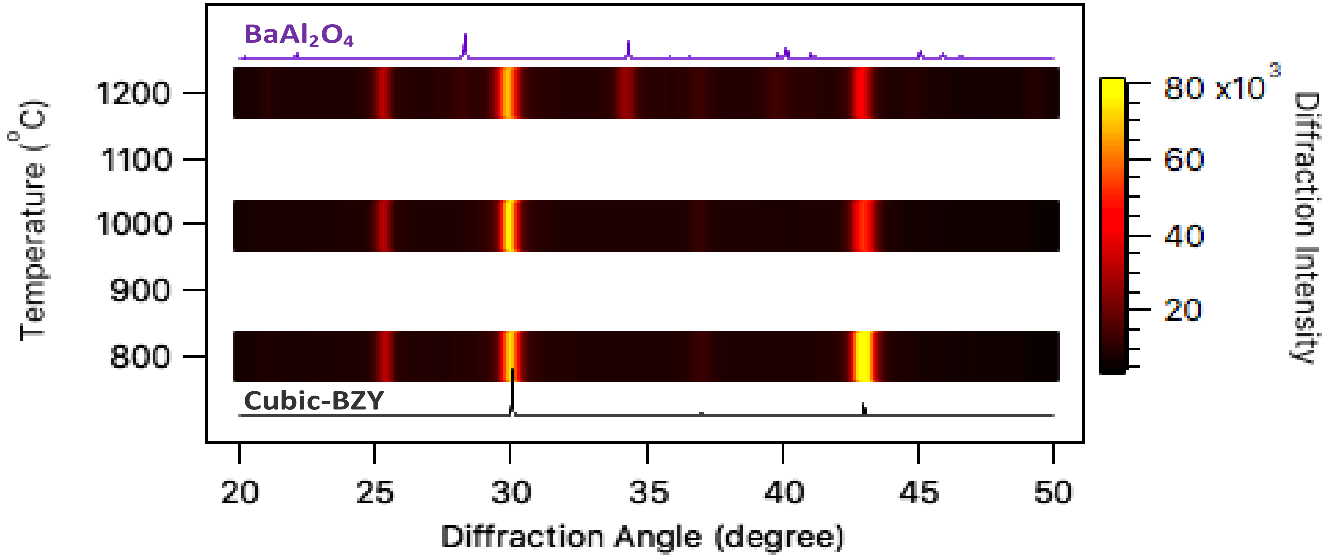
Milestone Summary Table (Phase 2)

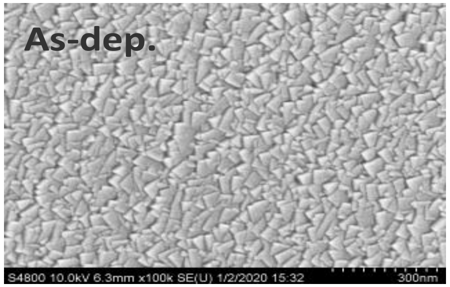
Recipient Name:	University of Connecticut, Prabhakar Singh
Project Title:	Proton-Conducting Solid Oxide Electrolysis Cells for Large-scale Hydrogen Production at Intermediate Temperatures

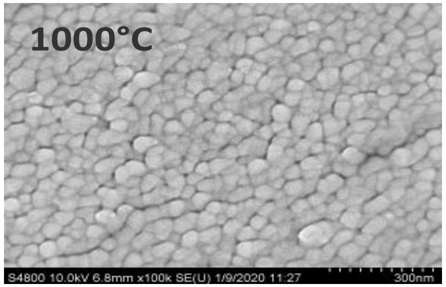
Task Number	**Task or Subtask Title**	**Milestone Type**	**Milestone Number***	**Milestone Description**	**Milestone Verification Process**	**Anticipated Quarter**	**Status**
1	Program management and plan	Milestone	M1-1	Establish program priorities for phase 2 established. Statement of work for selected HydroGEN nodes is identified. Continuation of activities under Task1	Verify and consult with program manager	5	Complete
5	Optimization of proton conducting electrolyte/electrode materials	Milestone	M5-1	Demonstrate 50-gram batch synthesis of high conductivity electrolyte (>0.02 s/cm, 700˚C) with sinter temperature <1400C	Experimental validation through testing and reporting	8	Complete
6	Characterization of electrolyte/electrode materials	Milestone	M6-1	Identify contributions from proton, oxygen ion and electronic conductivities. Establish structural and chemical degradation mechanisms operating under steam electrolysis.	Verification through laboratory testing and peer-reviewed publications	8	Complete
7	Design, fabricate and evaluate large area H-SOECs	Milestone	M7-1	Measure performance of selected 1-inch diameter H-SOEC cells to validate performance stability (<25 mV/1000 h) for 200-hour under real-world electrolyzer operating conditions.	Verification through report review.	8	Complete
		Go/No-Go Decision point	Phase 2, period 1	Developed 50-gram batch proton-conducting electrolyte with conductivity of at least 0.02 $S.cm^{-1}$ at 650°C. Developed and tested 1-inch diameter cells with stable electrolysis performance (<25 mV/1000 h) for at least 200 hours. Current density reaches above 1 A/cm^2 at $\leq$ 1.4 V and temperature of $\leq$ 650 °C.	Test at least 2 SOECs at UCONN or PNNL to verify.	8	In process

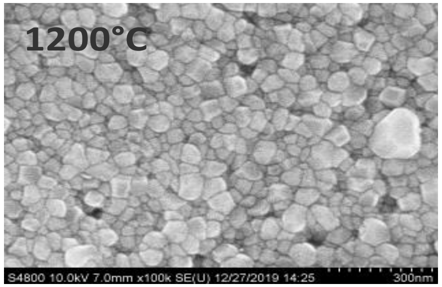
Sintering of Electrolyte – Baseline (on Sintering Aid)

Annealing of PLD deposited BZY film without sintering aids (NREL)







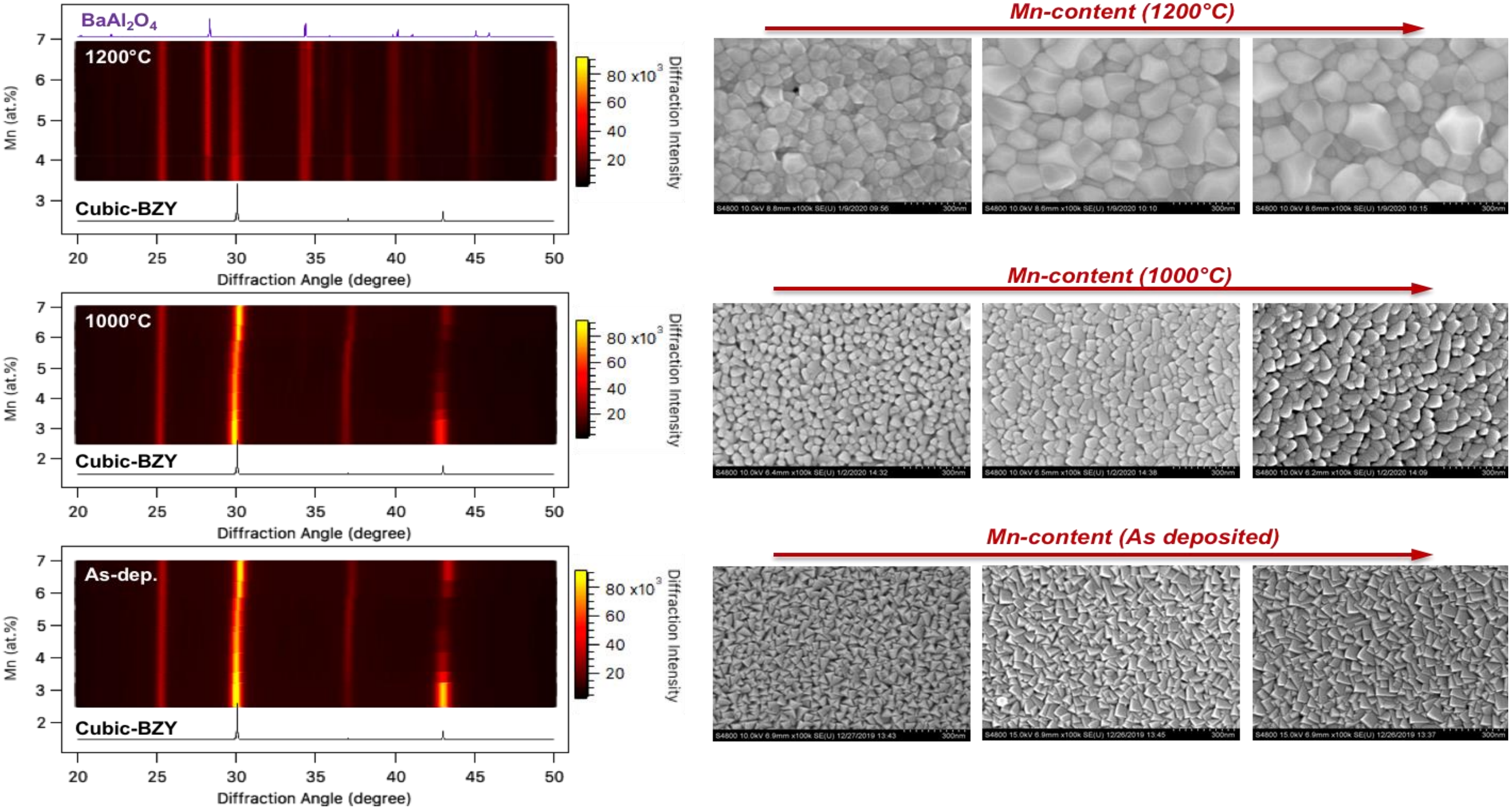


Sintering of electrolyte: MnO as Sintering Aids

Annealing of PLD deposited BZY film without sintering aids (NREL)

Zn substitutes Zr-site.

Dense films form with increased grain size.



Color scale maps of diffracted intensity (left) and surface morphologies (right) as a function of Mn-content of as-deposited BZY:Mn and after annealing at 1000 ºC and 1200 ºC

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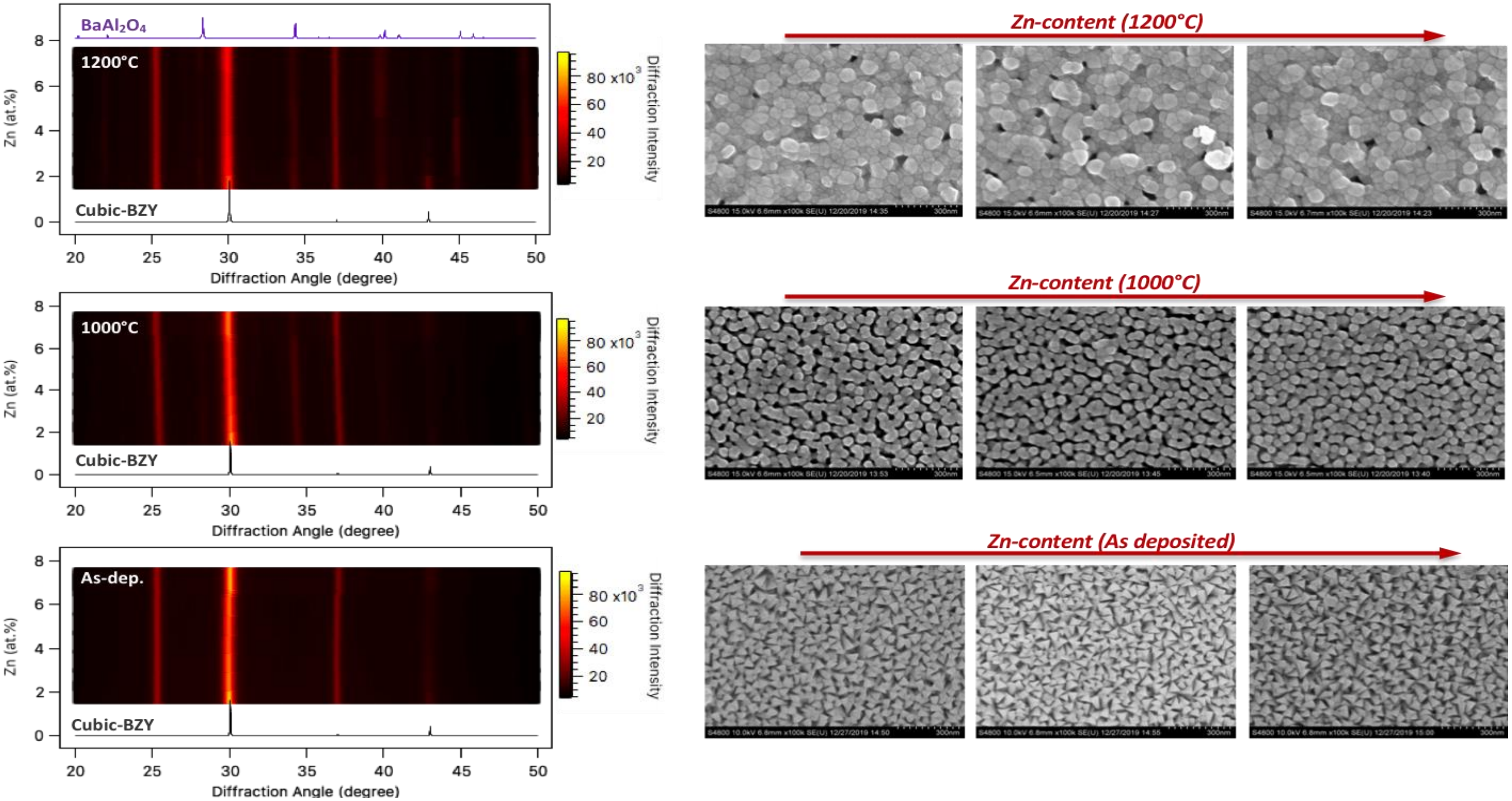
Sintering of electrolyte: ZnO as Sintering Aids

Annealing of PLD deposited BZY film with ZnO as sintering aid (NREL)



Color scale maps of diffracted intensity (left) and surface morphologies (right) as a function of Zn-content of as-deposited BZY:Zn film and after annealing at 1000 ºC and 1200 ºC