

Development of Stable Solid Oxide Electrolysis Cells for Low-cost Hydrogen Production

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**Pacific
Northwest**
NATIONAL LABORATORY

Annual Merit Review and Peer Evaluation Meeting
U.S. Department of Energy Hydrogen Program

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Beyond Current Potential

This presentation does not contain any proprietary, confidential, or otherwise restricted information.

Company Background

Utah, USA R&D/Manufacturing - 2017

- Office, laboratory, and manufacturing facility (24,000 ft²)
- NASA, DOE, DOD and commercial contracts
- Tape casting, cell and stack production, and testing
- End-to-end power to synfuels pilot plant

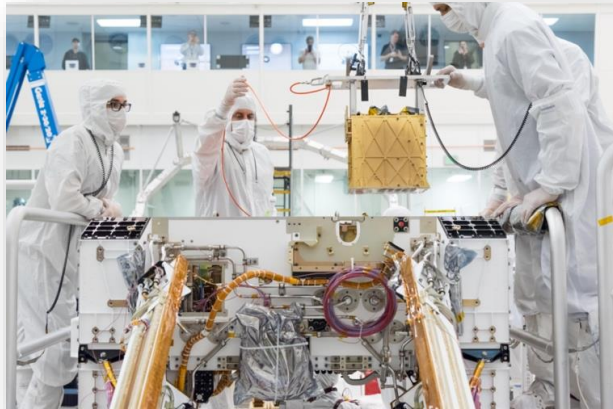


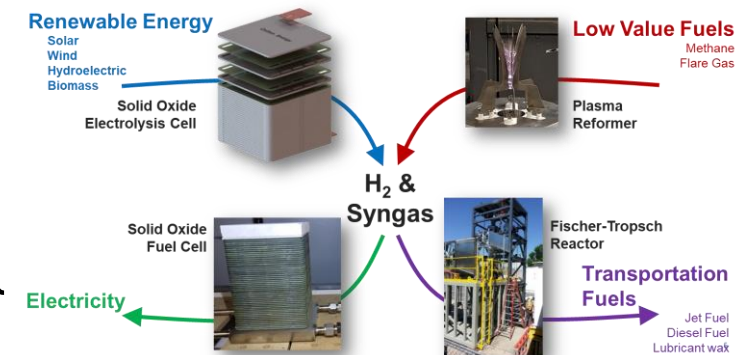
Image credit NASA/JPL-Caltec

Solid Oxide Fuel Cell and Electrolysis Stacks

- Longest running solid oxide fuel cell & electrolysis group
- Only flight qualified, TRL 9 SOEC unit with active NASA demonstration on Mars
- 30kW/10kW and 20kW/10kW reversible SOC system test programs

Fuel Reforming and Generation

- Plasma Reformer - H₂ or syngas from flare gas; digester gas conversion; clean-up bio-gasification
- Fischer-Tropsch Reactors - Modular design for sustainable fuel production from H₂ and syngas



DOE Award Announced (BIL funding) \$36 million: SOEC Pilot Manufacturing Plant

Test solid oxide electrolysis cell (SOEC) stack in a laboratory test bed

- Show improved performance over baseline stacks
 - Robustness,
 - Reliability,
 - Endurance,
 - Hydrogen purity, and
 - Hydrogen production at elevated pressure of 2 to 3 bar.

- **Robustness**
 - Capability for thermal cycling of a stack
 - Redox cycling of fuel electrode in a stack
 - Production of hydrogen at elevated pressure
- **Long term stability**
 - Projected lifetime of > 40,000 hours
- **Improved performance over baseline**
 - Reproducibility and lower polarization by electrode modification

Robustness

Redox Tolerant Fuel Electrode - Background

Background: MOXIE Program Overview



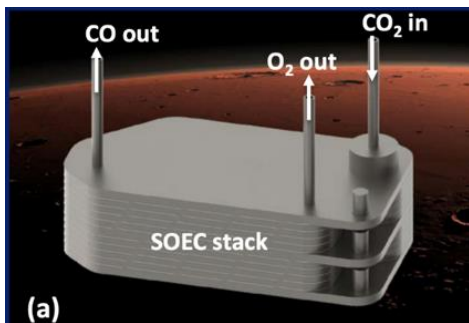
Mars OXygen ISRU Experiment for electrolysis of Mars atmosphere CO₂ to CO + O₂

First of any kind of demonstration of In-Situ Resource Utilization (ISRU) technologies to enable propellant and consumable oxygen production from the Martian atmosphere

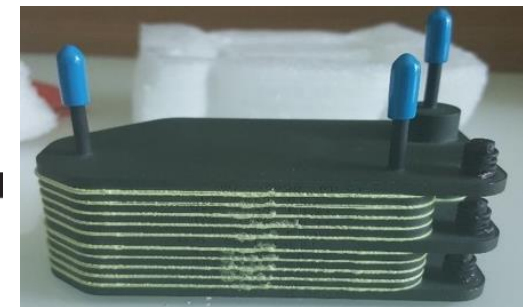
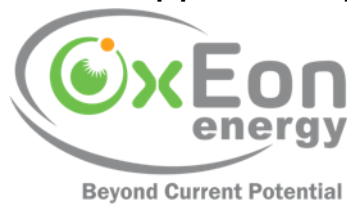
Successfully completed on Perseverance Rover mission

MOXIE is a ~0.5% scale prototype of expected final O₂ production rate

TRL 9 SOEC unit



Solid Oxide Electrolysis (SOXE) Development Team
Supported by NASA through the Jet Propulsion Laboratory (JPL)



Baseline Performance

- **21 consecutive stacks** built with *aerospace quality standards and traceability* having consistent baseline performance on dry CO₂ and 99.9%+ O₂ purity

Cycling Performance

- 3 stacks with **21 cycles** of identical test procedure having varying cycle-to-cycle flow rates and final cycle averages of 10.11 g O₂/hr production and 99.8% purity - Targets exceeded
- 1 stack to **61 cycles** with >99.6% purity at a controlled production rate of 6 g/hr at 55g/hr CO₂ feed

Structural Stability Testing

- **No leak or significant performance change after 10kN crush testing**
- Stacks tested to 25kN force with no crossover or external leakage
- *Load to failure required 62.2kN (>30 margin of safety from design)*

Shock/Vibe Testing

- Stacks vibrated at JPL and post vibe tested at OxEon
- **No leak or significant performance change post vibe!**
- **No leak after shock testing, no significant performance change!**

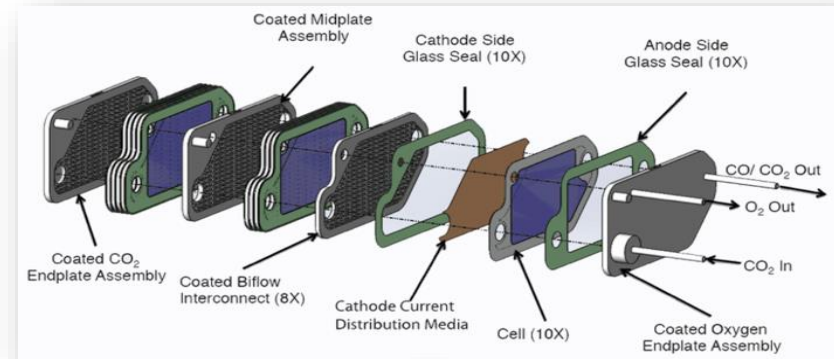
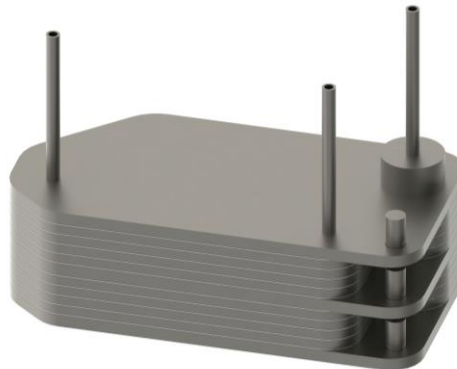
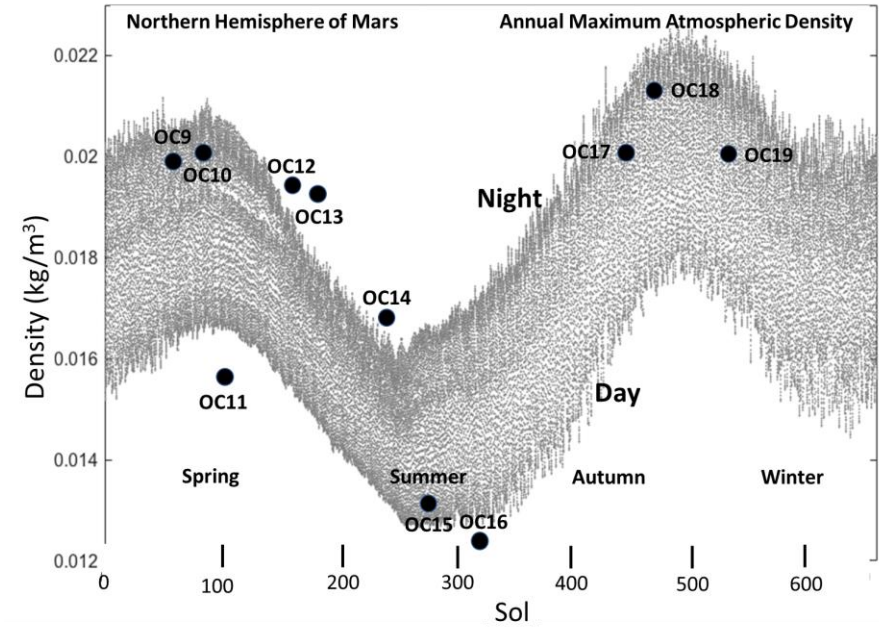
Cryo-Cycling

- Vibe stack cryo-cycled to -40°C (40 cycles), -55°C (3 cycles), -65°C
- **Stack performance and purity unchanged in operational cycling post test**

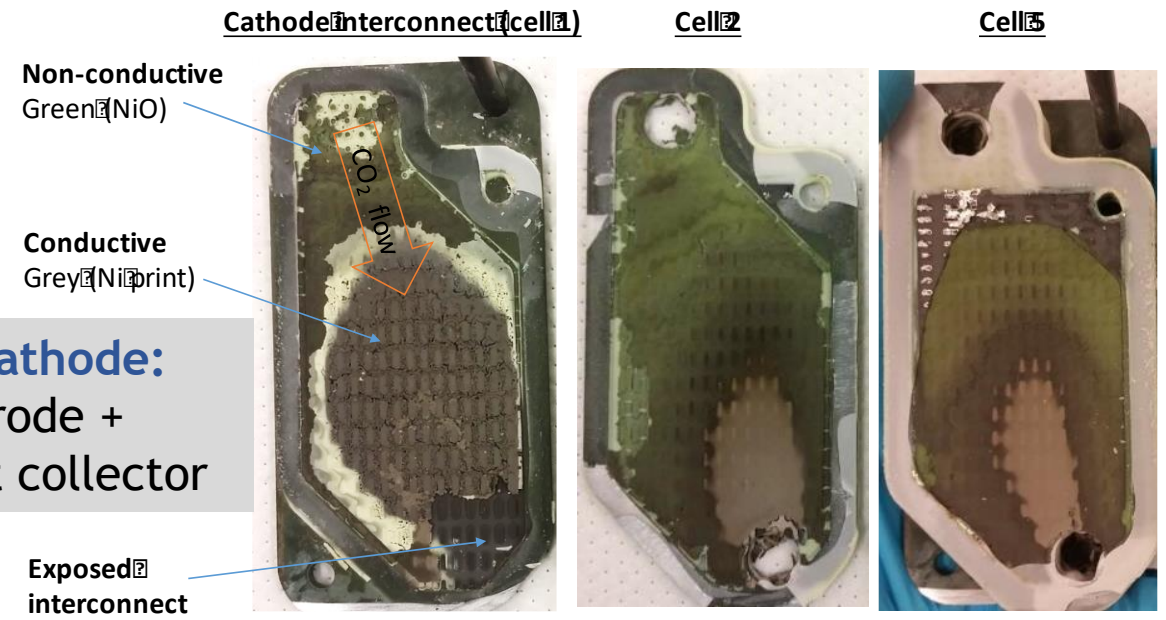
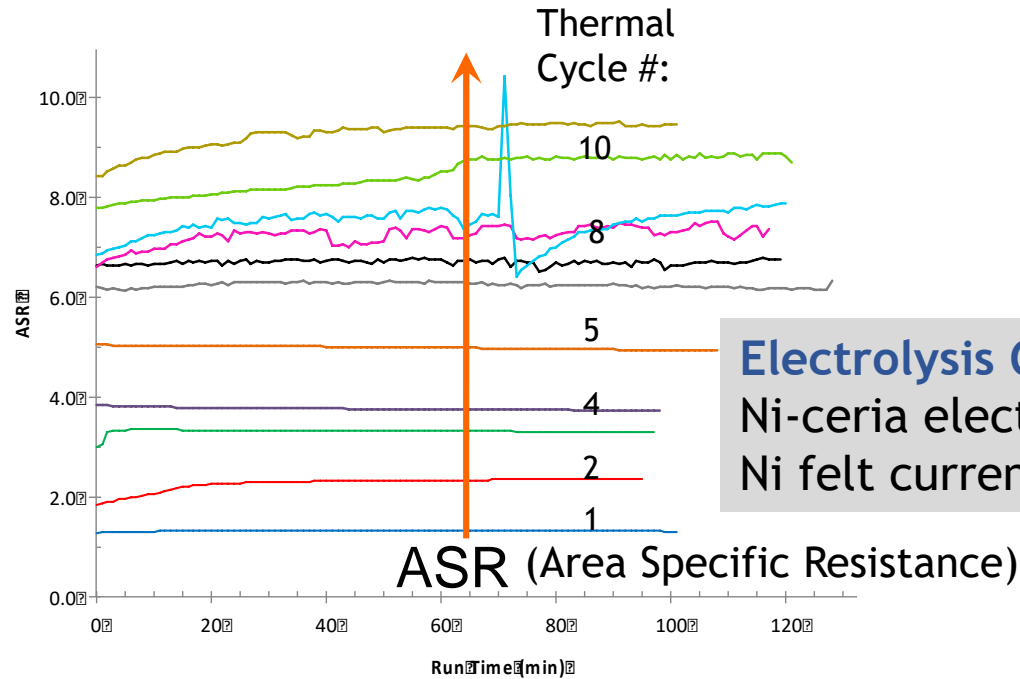


Flight Test Success - First Ever ISRU Demonstration

- 16 total operation cycles completed on Mars
- Pressure external to stack ~7 millibar; internal ~1 bar
- >99.6% Oxygen purity
- Operations have spanned the climactic extremes of the Mars year.
- All cycles performed as predicted: lab & models
- The MOXIE Mission completed in Sept 2023
- Basis for a Lunar and a Martian ISRU Demonstration System



Cathode Challenge: Oxidation in Dry CO₂



- Early MOXIE Test Stack:
 - 15 operational cycles - full thermal cycle with 120 min operation on dry CO₂
 - Dry CO₂ → O₂ production ~12% of initial

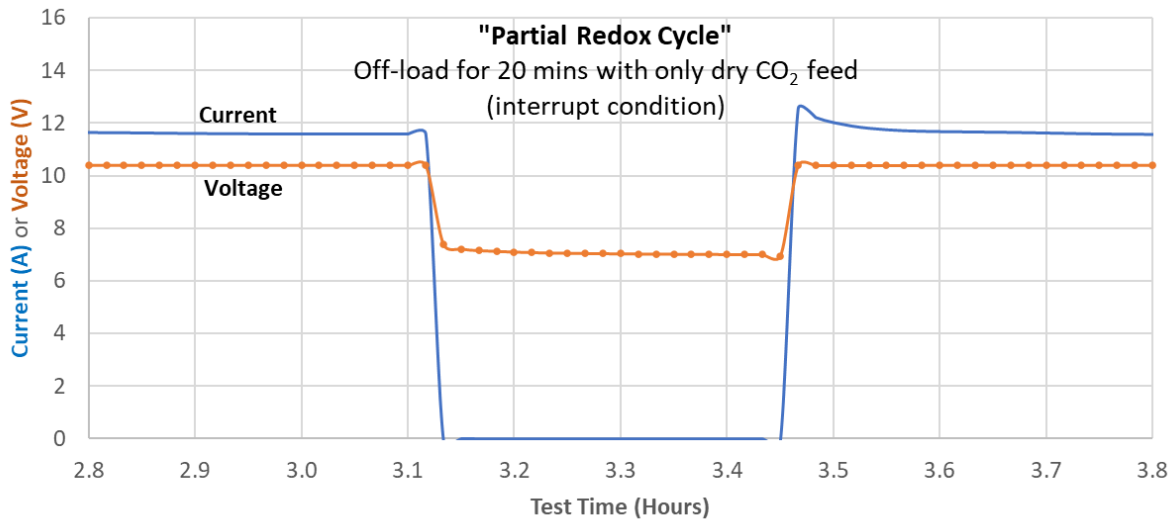
Dramatic degradation resulted from progressive oxidation front

Oxidation of Ni to NiO causes ~24% vol expansion, and in this case, irreversible damage to the electrode & current collector

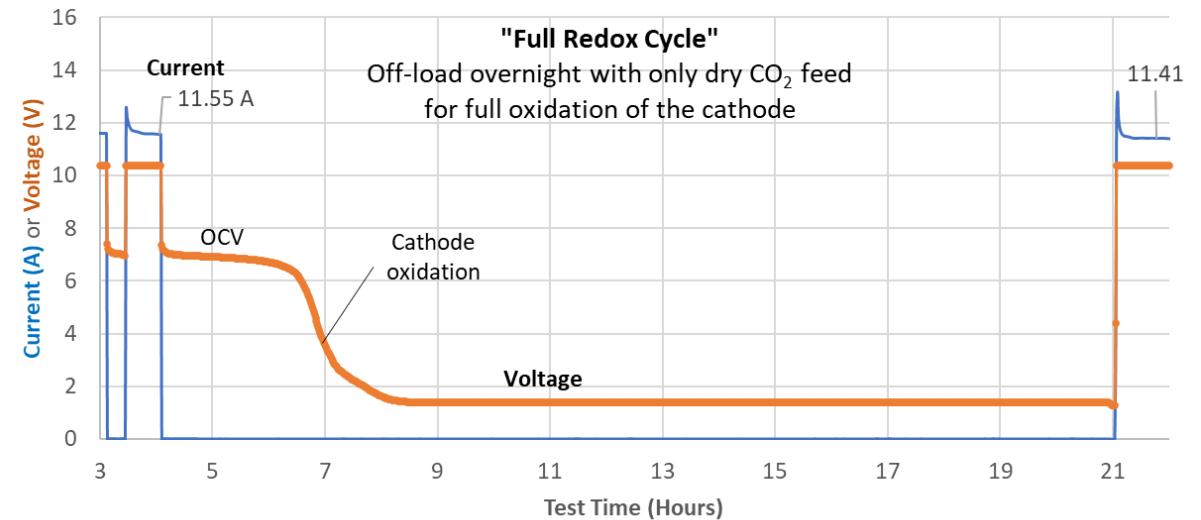
MOXIE implemented recycle of produced CO to prevent cathode oxidation

Redox Tolerance: Partial and Full Redox Cycles in CO₂ Electrolysis

STK-033 NASA SBIR 10-Cell FTD Deliverable Stack
800 C, 111 cm² active area/cell, 1.5 SLPM dry CO₂ feed



STK-033 NASA SBIR 10-Cell FTD Deliverable Stack
800 C, 111 cm² active area/cell, 1.5 SLPM dry CO₂ feed



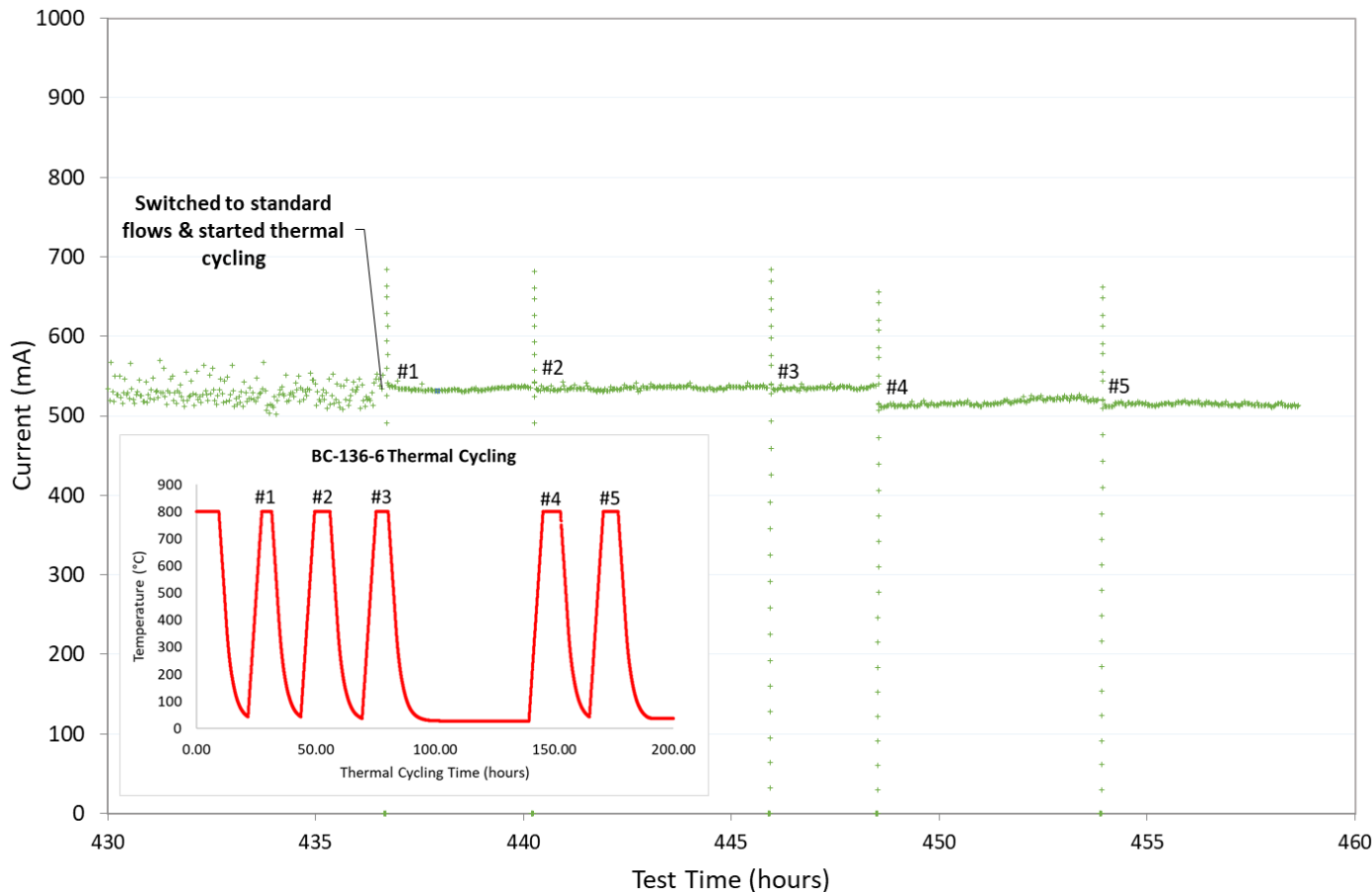
- Stack: Short (20 min) and long (12 hrs) exposure to CO₂
- Application of voltage → Full and immediate recovery of performance

Button Cell Thermal Cycling in Steam Electrolysis

Button cell thermal cycled 5 times → stable performance

BC Thermal Cycling: BC-136-6 (Sr-Free)

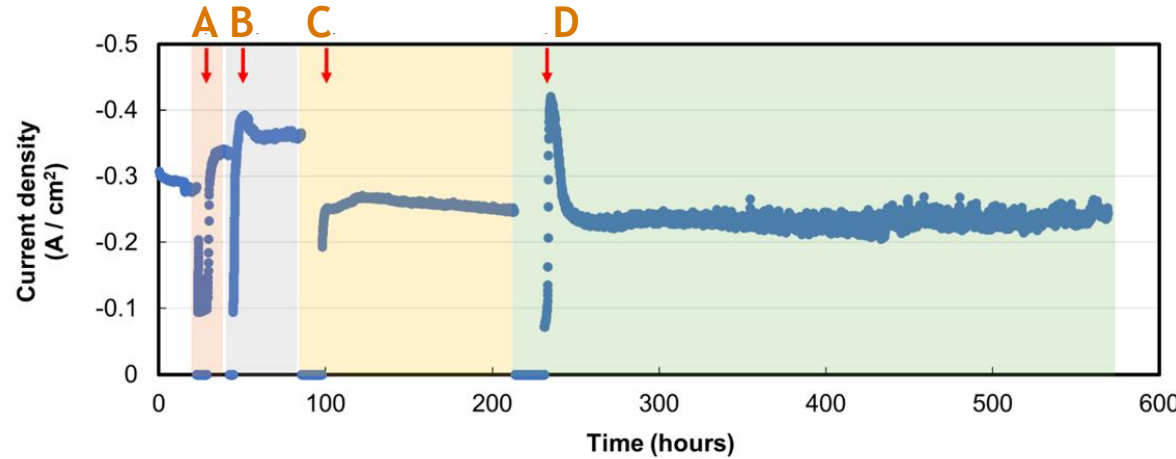
800 °C, 2.0 cm² active area, 30 sccm H₂, 30 N₂, 82°C bubbler, 1.3 V hold



- Sr-free air electrode composition
- Thermal cycle steps:
 - Remove 1.3 V load.
 - Cool to RT, reheat to 800 °C (2°C/min ramps).
 - Reapply voltage.
- Current density
 - Before thermal cycles: 0.266 A/cm²
 - After 5 thermal cycles: 0.259 A/cm²

Button Cell Redox Cycling in Steam Electrolysis

Button cell (BC-109-4) performance recovered in redox cycling



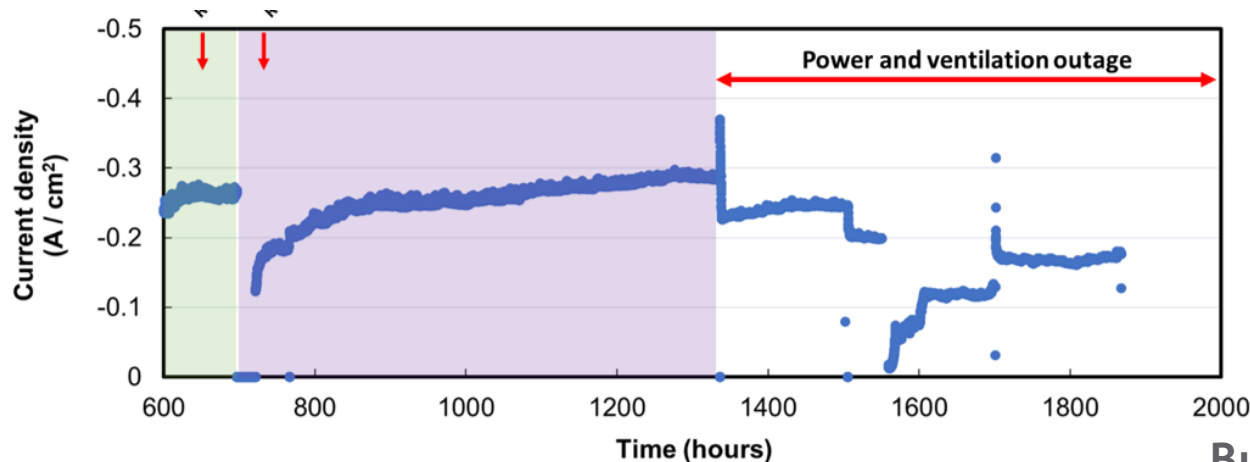
A - Initial performance, 6 30-min redox cycles - **exposure to steam at OCV for 30 minutes; no H2 in feed; apply voltage**

B - 2-hr redox cycle

C - 12-hr redox cycles

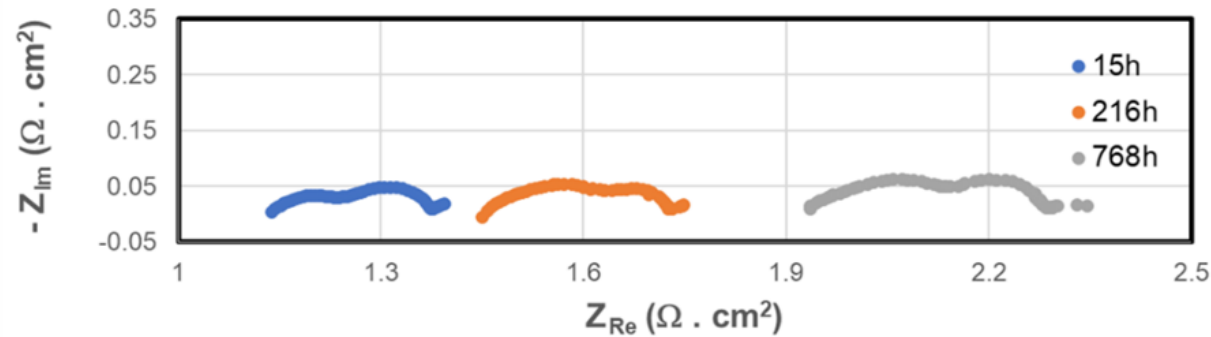
D - 16-hr redox cycle

Button cell (BC-109-4) performance continually improved over 600 hours after 16 hr oxidation cycle (~700 hrs)



Ohmic resistance dominates performance loss in redox cycles. Likely results from button cell current collector delamination.

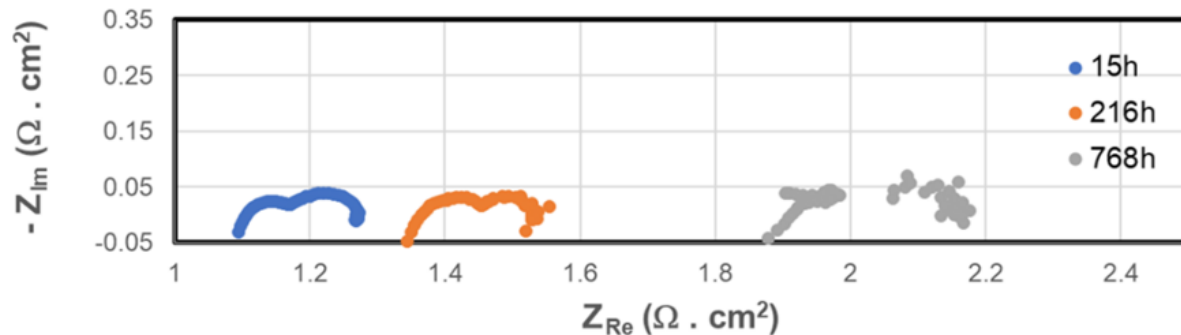
EIS at OCV



Ohmic resistance increased 28%

Polarization resistance increased 19%

EIS at 1.3V



Ohmic resistance increased 23%

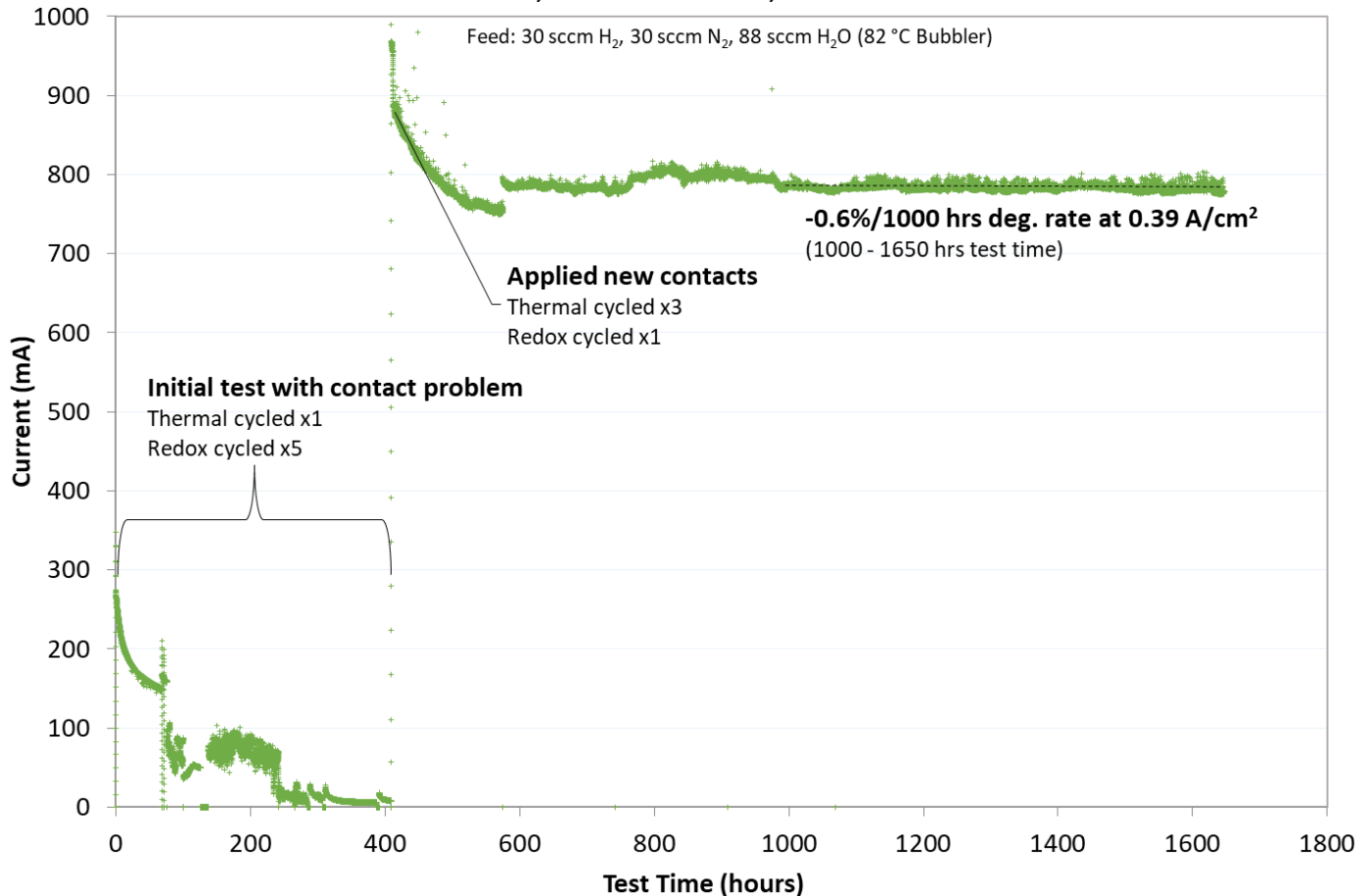
Polarization resistance increased 12%

Button cell thermal cycled 4 time, redox cycled 6 times → stable performance

BC Thermal & Redox Cycling: BC-136-2 Retest with new contacts

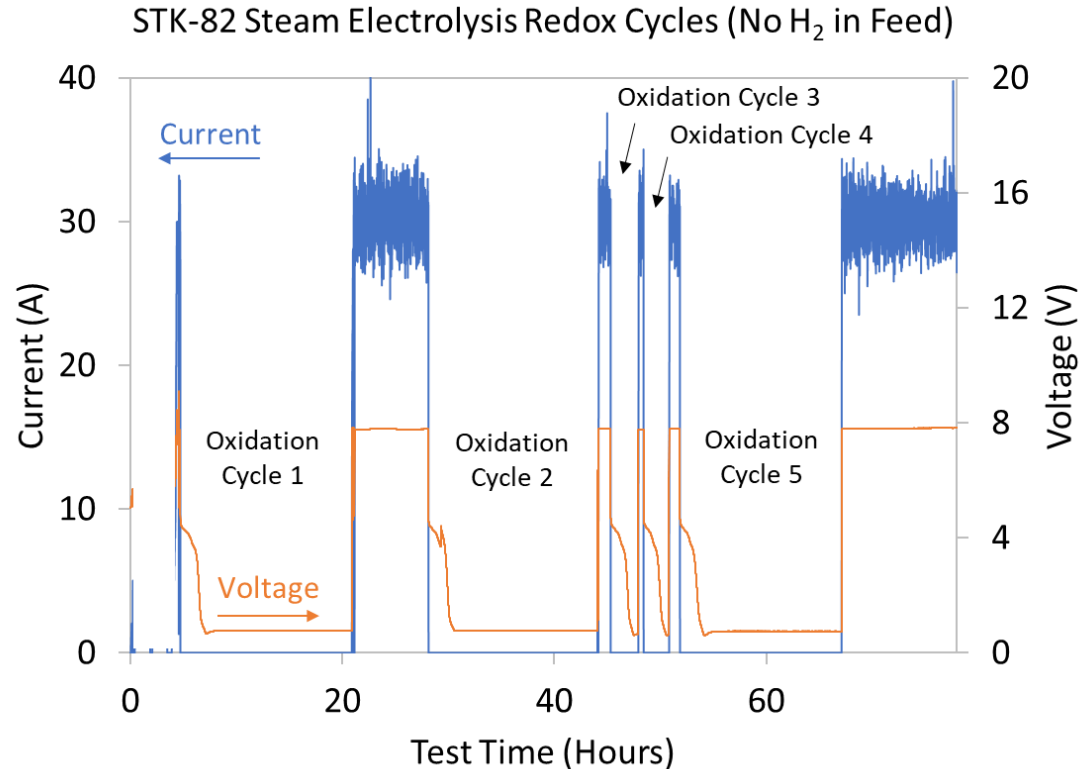
800 °C, 2.0 cm² active area, 1.3 V hold

Feed: 30 sccm H₂, 30 sccm N₂, 88 sccm H₂O (82 °C Bubbler)



- Redox cycle steps:
 - Switch to oxidizing feed (N₂/ H₂O).
 - Remove 1.3 V load to stop H₂ generation.
 - Wait for full oxidation, then reapply load.
 - Add H₂ for reduction recovery if needed.
- Thermal cycle steps:
 - Remove 1.3 V load.
 - Cool to RT, reheat to 800 °C (2 °C/min ramps).
 - > 800 °C for new contacts application
 - Reapply load.
- Current density
 - Initially: 0.174 A/cm²
 - At 1,300 hrs: 0.394 A/cm²

STK-82: Full Redox Cycling in Steam Electrolysis



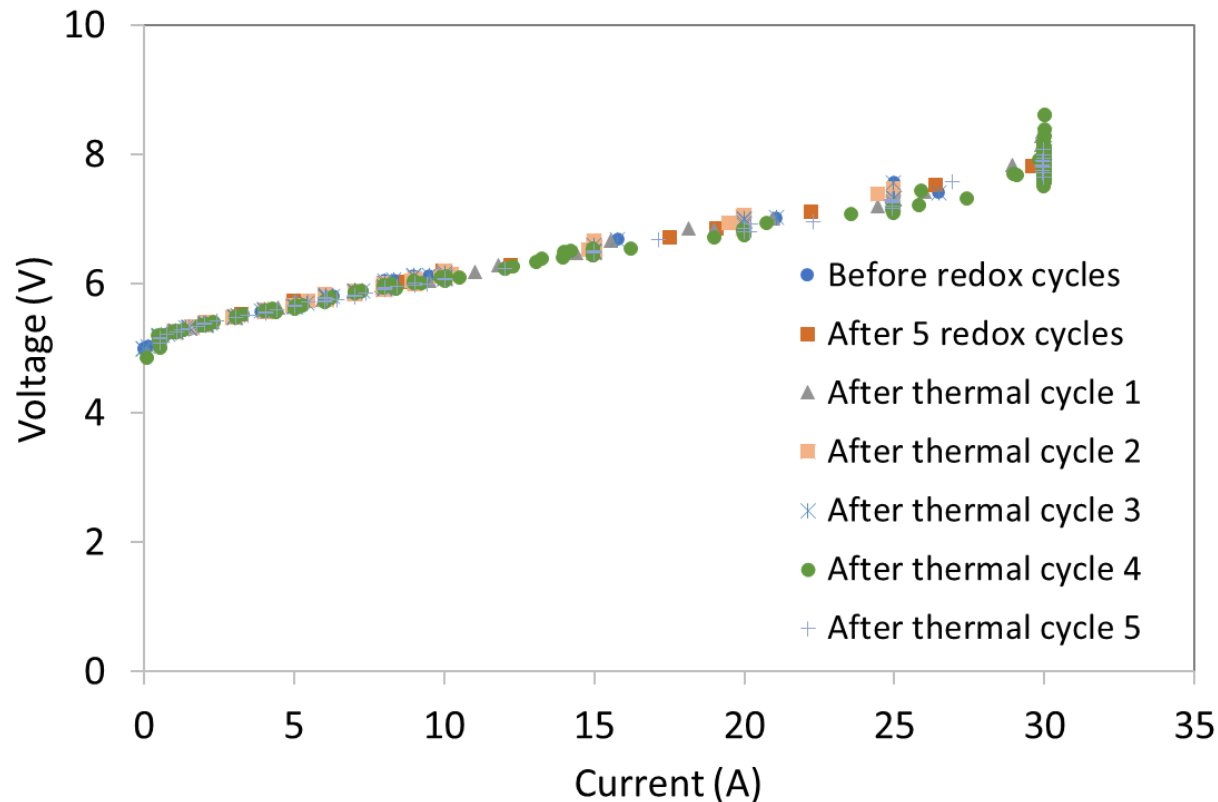
STK-82 (6-cell stack) recovered performance after 6 redox cycles

STK-82 Redox Cycle Comparison			
Cycle	Current (A)	Voltage (V)	Power (W)
1	28.04	7.82	219
2	31.12	7.78	242
3	31.33	7.77	243
4	31.49	7.8	245
5	31.03	7.8	242
6	31.09	7.8	242

- Fuel electrode was fully oxidized by exposure to steam only
- Application of voltage → Full and immediate recovery of performance
 - Self-generated hydrogen at fuel electrode
 - No external reducing gas!

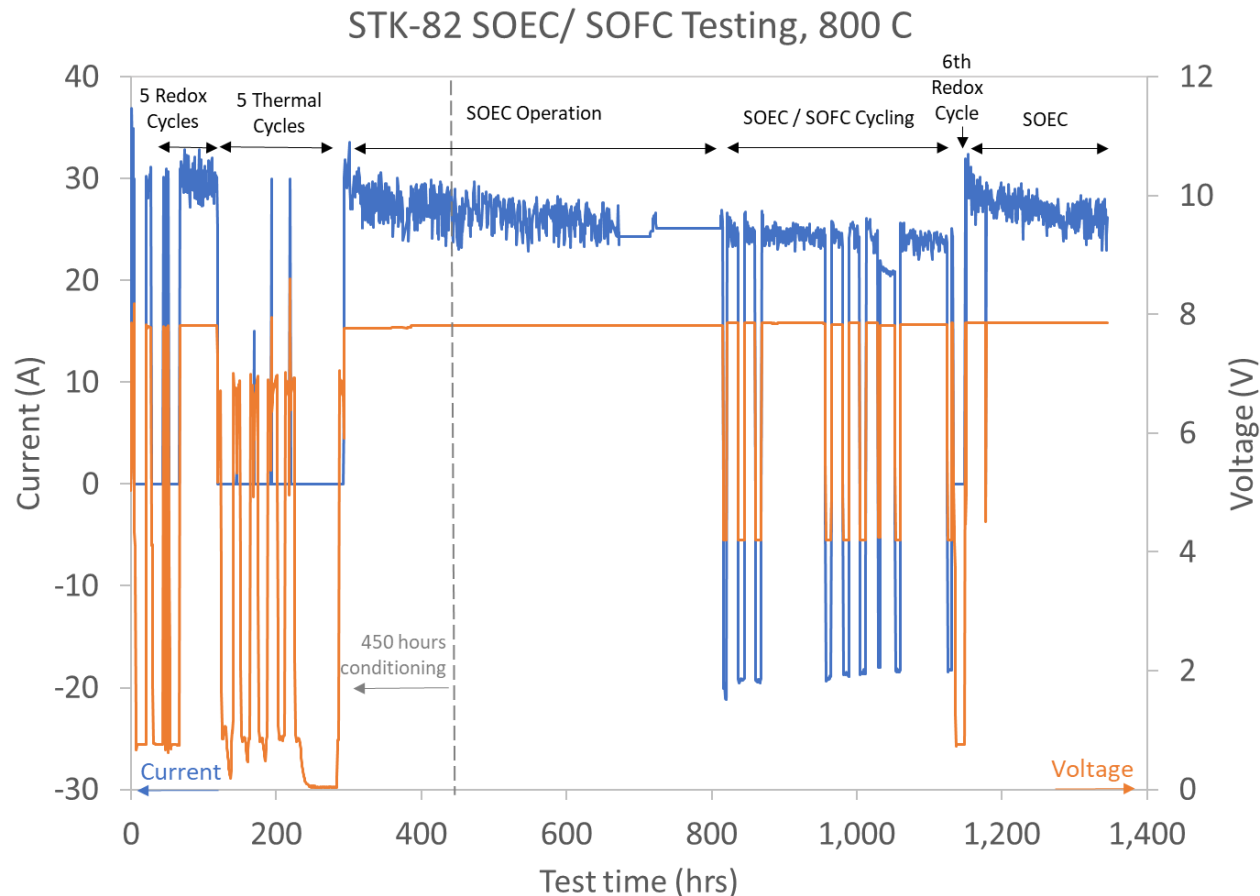
STK-82: Thermal Cycling - 800 °C to Room Temperature

STK-82 I-V Sweeps, Steam Electrolysis Testing (800C)



- Stack after 5 redox cycles was cooled to room temperature in dry H₂/N₂ at OCV, then returned to 800 °C (100 °C/min)
 - 5 thermal cycles
- Application of voltage → Full and immediate recovery of performance
- **Demonstrates robust stack and seals**

STK-82: Long Term Stack Stability Testing

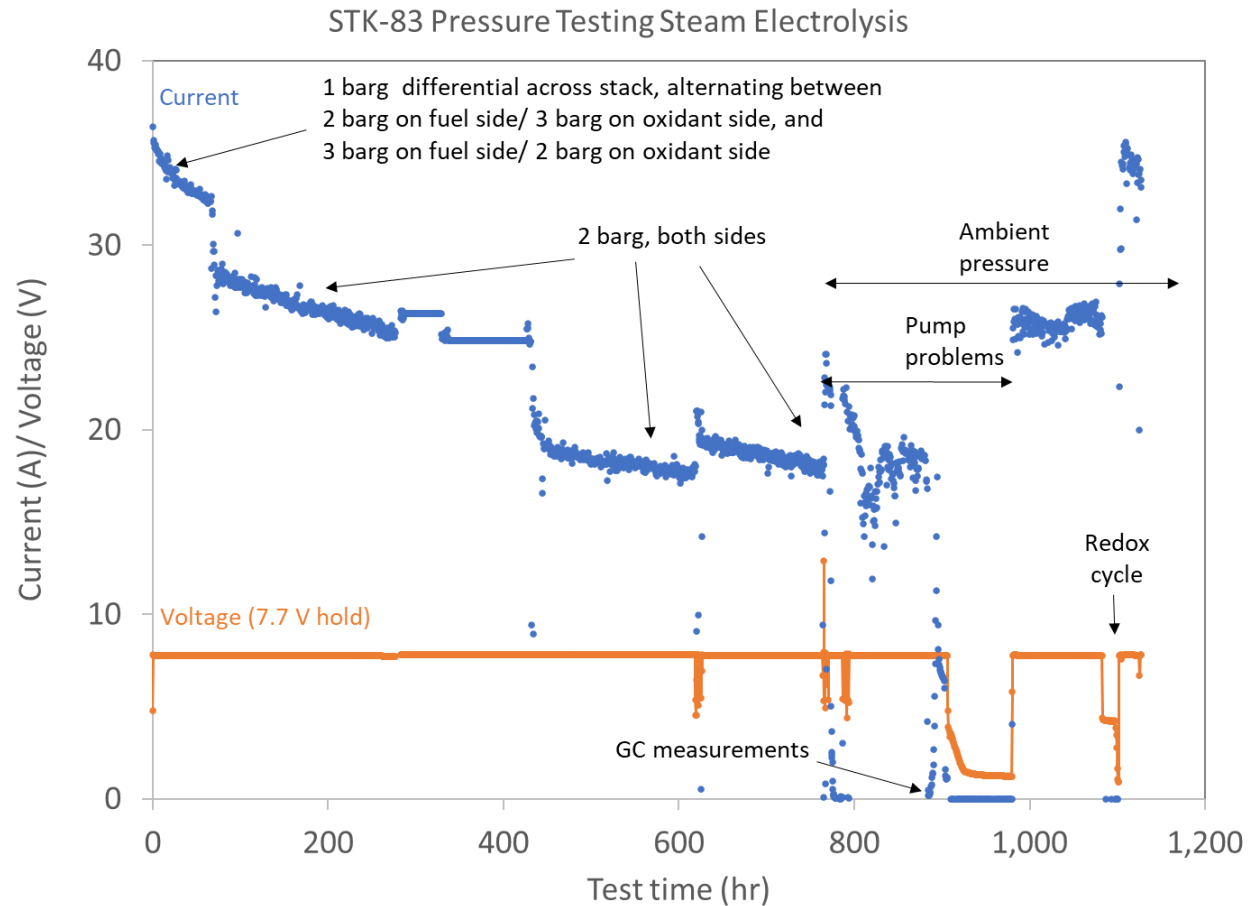


Stack test sequence

- 5 redox cycles
- 5 thermal cycles
- 500+ hour steam electrolysis
- 300+ hours SOEC/ SOFC cycling
- 6th redox cycle
- Steam electrolysis

Degradation = 1.8 %/ 1,000 hrs in SOEC operation after conditioning period.

STK-83: Pressurized Stack Operation



- Stack (6-cell stack) tested at moderate pressure for ~800 hours
 - 3 barg pressure during testing
 - 1 barg differential across stack
- Performance drop is likely caused by poor seal between stack and stack test assembly (external gasket)
- **Hydrogen production at pressure!**
- **Performance (at ambient pressure) recovered to initial value after a redox cycle**

STK-83: Pressurized Stack Operation

Gas chromatograph analysis of anode and cathode tail-gas composition, taken at ~775 hrs

GC Results (%) & Product Calculations (slpm)

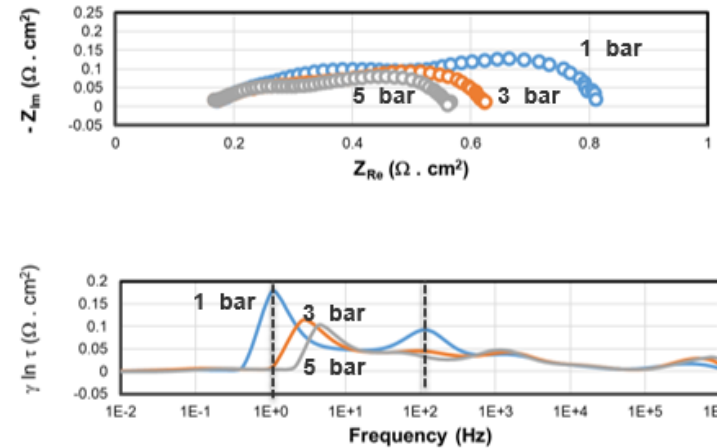
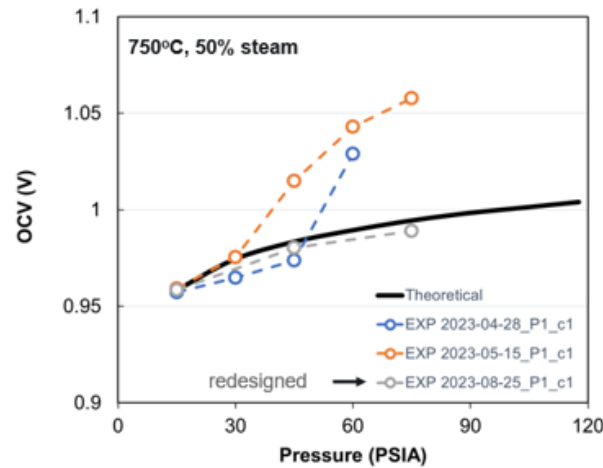
Pressure condition	2 barg O ₂ , 2 barg fuel		2.2 barg O ₂ , 2 barg fuel		2 barg O ₂ , 2.5 barg fuel		2 barg O ₂ , 3 barg fuel	
	O ₂	fuel	O ₂	fuel	O ₂	fuel	O ₂	fuel
H ₂		85.8		84.8		84.08	0.01	85.41
O ₂	99.84		98.58		99.46		99.58	
N ₂	0.16	14.03	1.39	15.03	0.54	15.77	0.41	14.36
H ₂ generated (slpm)		0.95		0.86		0.80		0.92
O ₂ generated (slpm)		0.47		0.43		0.40		0.46
H ₂ O Conversion (slpm)		84%		76%		71%		81%
Stack Current, A (calculated)		22.6		20.5		19.2		21.9

Robust seal: O₂ purity demonstrates lack of cross over leak

H₂ and O₂ generated at pressure!

SOEC Performance Increases with Pressure

(PNNL-fabricated cells).



Pressurized button cell

- Low frequency (mass transport) and mid-frequency (diffusion) processes are suppressed
- Troubleshooting seals for long-term tests

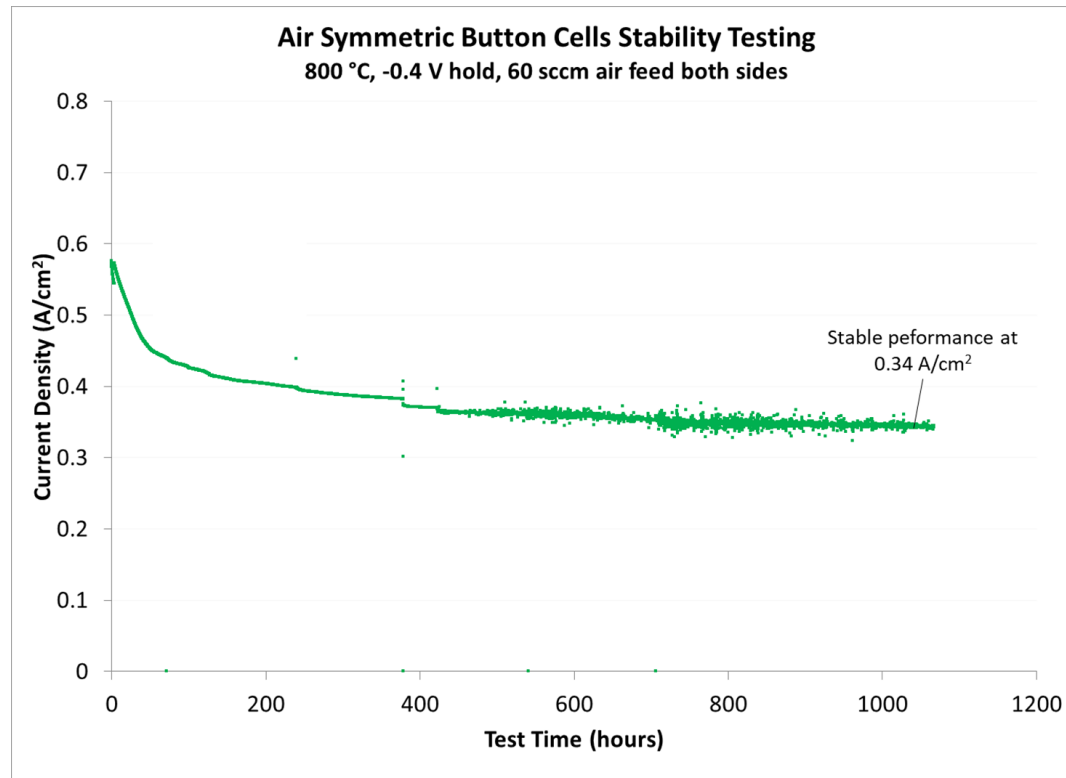
Test stand modifications on-going to meet 7 bar pressure target

- Good OCV match to the Nernst voltage, but there is a seal leak somewhere in the test system
- Reached 5 bar.

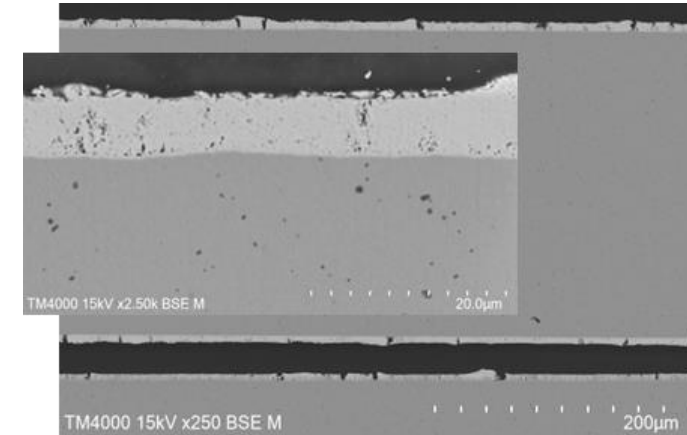
Oxygen Electrode Interface Improvement

Improvements to barrier layer density and continuity.

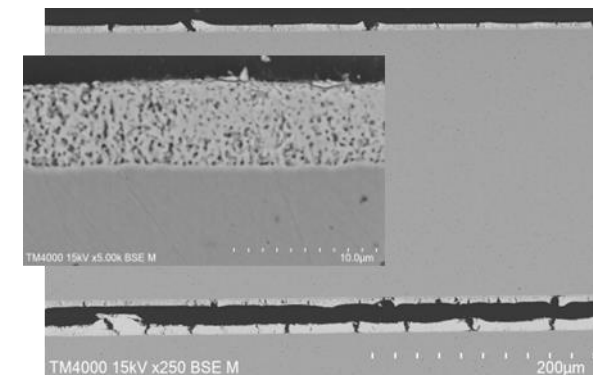
Stable performance at 0.34 A/cm² measured after 600 hours conditioning that continued through 1,000 testing



Barrier layer density increased using a modified process



Higher porosity with screen printed barrier layer



Eliminate Sr in current collector layer by replacing LSCF with Sr-free composition

- Ohmic resistance close to expected based on electrolyte thickness
- Polarization resistance indicates Sr-containing LSCF layer is not required for water splitting
 - Sr-free cell measured 58% lower initial polarization resistance relative to the LSCF cell

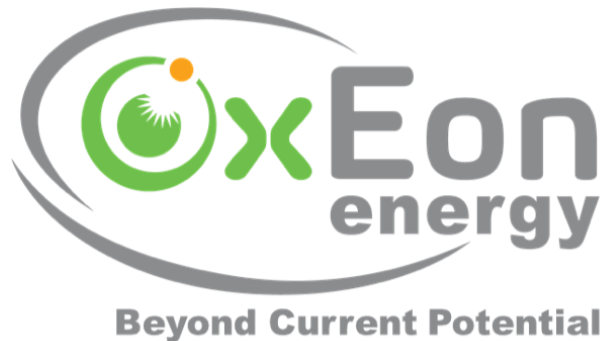
Electrode CC Layer	ASR ($\Omega\cdot\text{cm}^2$)		
	Total	Ohmic	Polarization
Sr-free composition	0.66	0.59	0.08
LSCF	0.71	0.52	0.19

- **Redox tolerance validated for steam electrolysis**
 - Stack test complete
 - Oxidized Ni electrode recovery without the need for hydrogen in inlet
- **Thermal cycle stability demonstrated (up to 5 cycles)**
- **Pressurized tests: steam electrolysis**
 - Stack showed high purity O₂ and H₂ at 3 barg pressure
 - Button cell test ongoing at PNNL
- **Electrode materials modification - validation in progress**
 - Composition to improve thermochemical stability
 - Surface/Interface modification for improving catalytic property and performance stability
- **Cation migration is still a challenge for long-term stability**
- **External influences (steam, air, BOP contaminants) still need evaluation and mitigation**

Thank you

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