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

Contract Number: DE-FE0032105

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Support: NASA, DOE

Pacific Northwest NATIONAL LABORATORY

23rd Annual Solid Oxide Fuel Cell (SOFC) Project Review Meeting
Pittsburgh, PA
April 18 - 20, 2023

DOE Project Manager: Drew O'Connell

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Beyond Current Potential

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

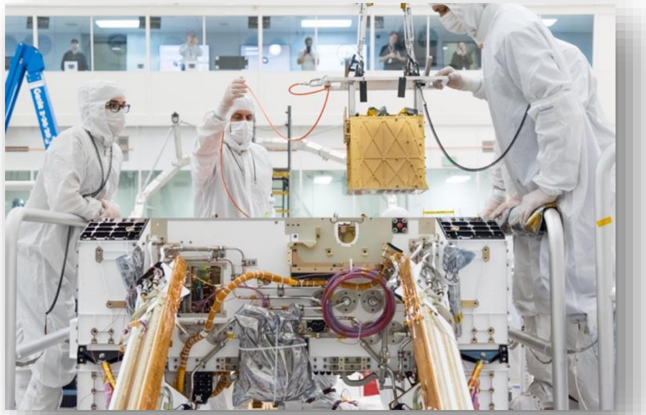


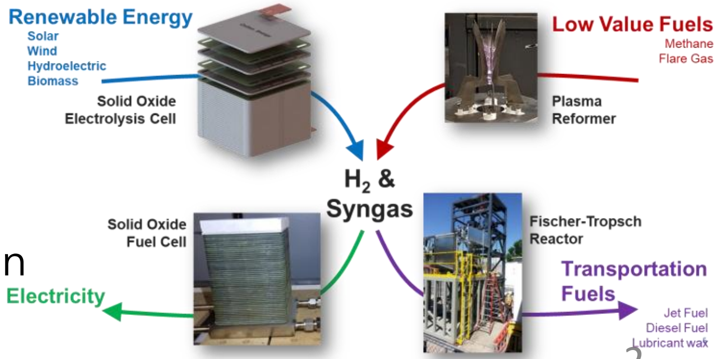
Image credit NASA/JPL-Caltec

Solid Oxide Fuel Cell and Electrolysis Stacks

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

Fuel Reformation and Generation

- Plasma Reformer – H_2 or syngas from flare gas; digester gas conversion; clean-up bio-gasification
- Fischer-Tropsch Reactors – Modular design for sustainable fuel production from H_2 and syngas



Project Objective

- **A solid oxide electrolysis cell (SOEC) stack in a laboratory test bed**
  - show improved performance over baseline stacks
    - robustness,
    - reliability,
    - endurance,
    - hydrogen purity, and
    - produce hydrogen at elevated pressure of 2 to 3 bar.

Project Goals

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

Robustness

Redox Tolerant Fuel Electrode - Background

Background: MOXIE Program Overview

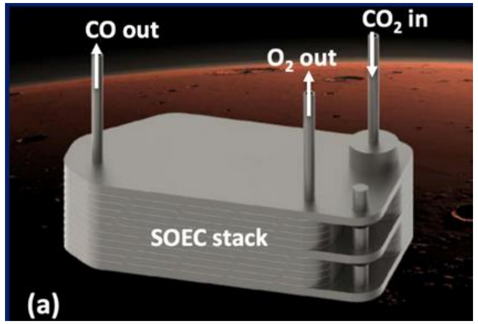
Mars **OX**ygen **I**SRU **E**xperiment aka "The Oxygenator"

CO_2 to $CO + O_2$

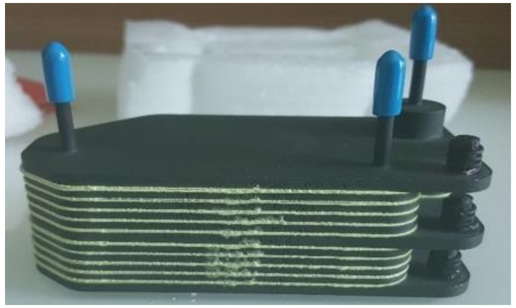
First of any kind of demonstration of **In-Situ Resource Utilization (ISRU)** technologies to enable propellant and consumable oxygen production from the Martian atmosphere – Currently onboard Perseverance Rover

MOXIE is a ~0.5% scale prototype of expected final O_2 production rate

TRL 9 SOEC unit



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



Flight Qualification

Baseline Performance

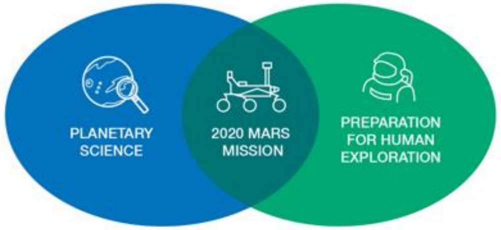
- **21 consecutive stacks** built with *aerospace quality standards and traceability* having a maximum baseline performance of 1.6 ohm-cm^2 dry CO_2 and 99.9%+ O_2 purity

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 O_2/hr production and 99.8% purity – Targets exceeded
- 1 stack to **cycle 61** with >99.6% purity at a controlled production rate of 6 g/hr at 55g/hr 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!**

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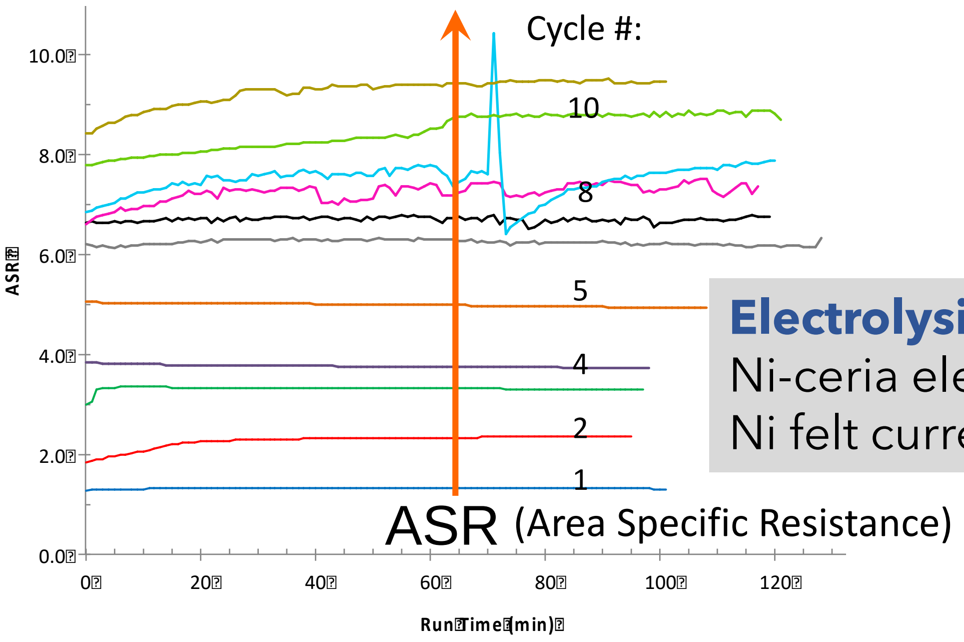
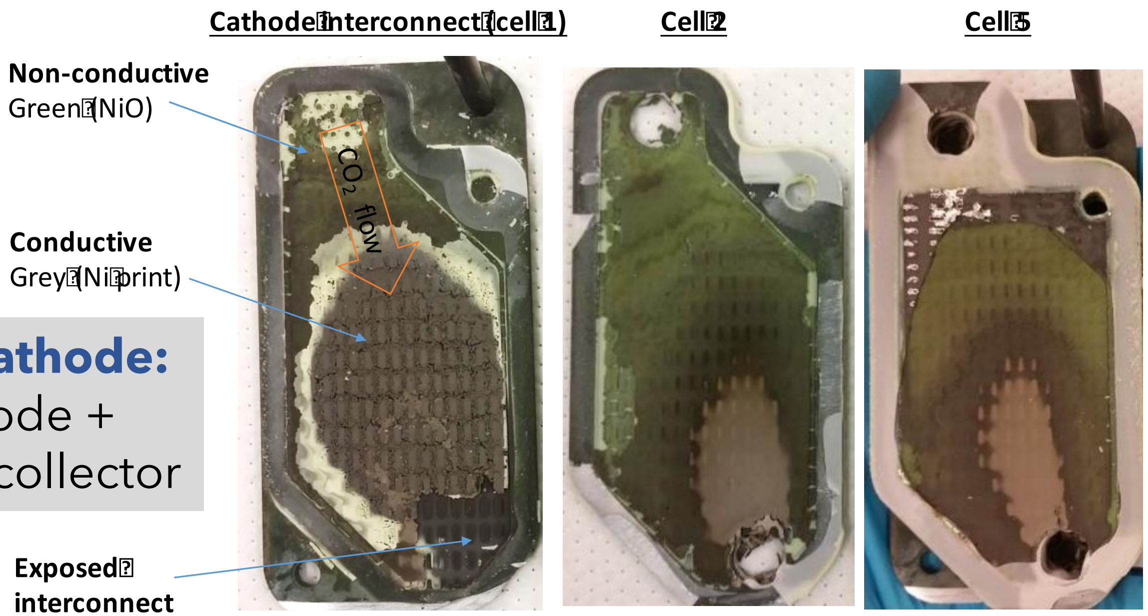
Cryo-Cycling

- 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**

Cathode Challenge: Oxidation in dry CO_2



Electrolysis Cathode:
Ni-ceria electrode +
Ni felt current collector



Dramatic degradation resulted from progressive oxidation front

- Early MOXIE Test Stack:
  - 15 operational cycles - full thermal cycle with 120 min operation on dry CO_2
  - Dry CO_2 → O_2 production ~12% of initial

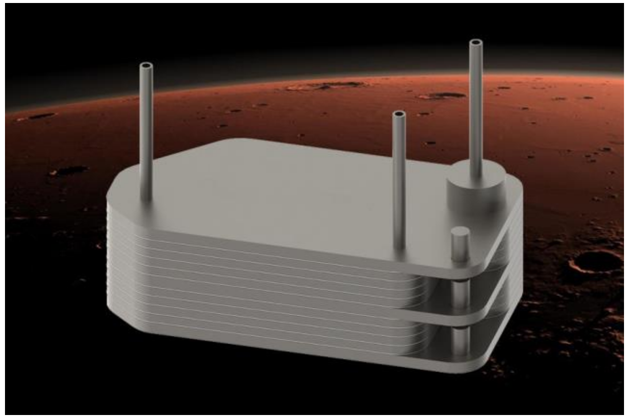
Oxidation of Ni to NiO causes ~24% vol expansion, and in this case, irreversible damage to the electrode & current collector

MOXIE implemented recycle of produced CO to prevent cathode oxidation

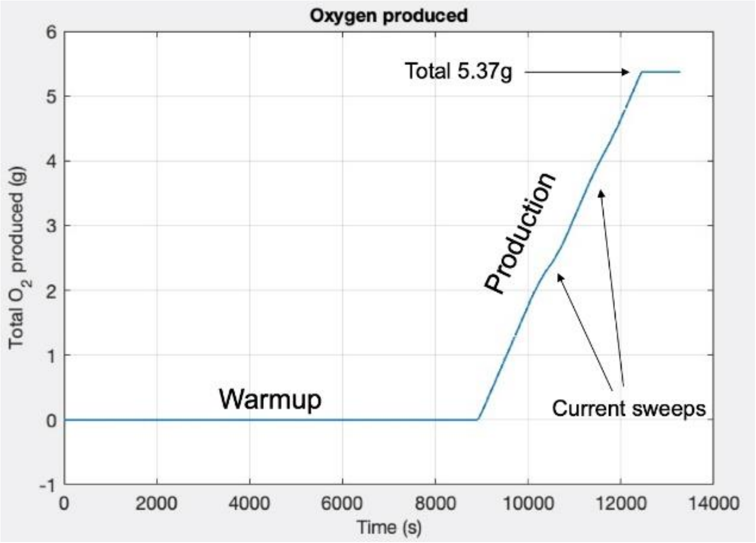
NASA Support through JPL

Flight Test Success – First Ever ISRU Demonstration!

First 100 Sols!



- **Sol 5 "Aliveness Test" Mon Feb 22, 2021**
- **Sol 13 First run with Run Control Table (RCT)**
- **Sol 14 "Health Check" of heaters and compressor**
- **Sol 59-60 April 20, First Oxygen**
  - Produced 5.4 g O_2 pre-dawn, peak rate of 6 g/h (2 A current)
- **Sol 81 May 12, 2nd Oxygen**
  - Nighttime (early AM) operation
  - Produced 7 g O_2, 8 g/h peak
- **Sol 100 May 31 3rd Oxygen**
  - Mid-day operation with lower atmospheric density
  - Extended 8 g/h operation



First Run

13 Successful high purity (>99.6%) oxygen production runs on Mars to-date

NASA Support through JPL

Redox Tolerance for CO_2 Electrolysis (NASA SBIR)

Ni-based electrode

Button Cell: Redox Cycling and Long-Term Stability for CO_2 Electrolysis

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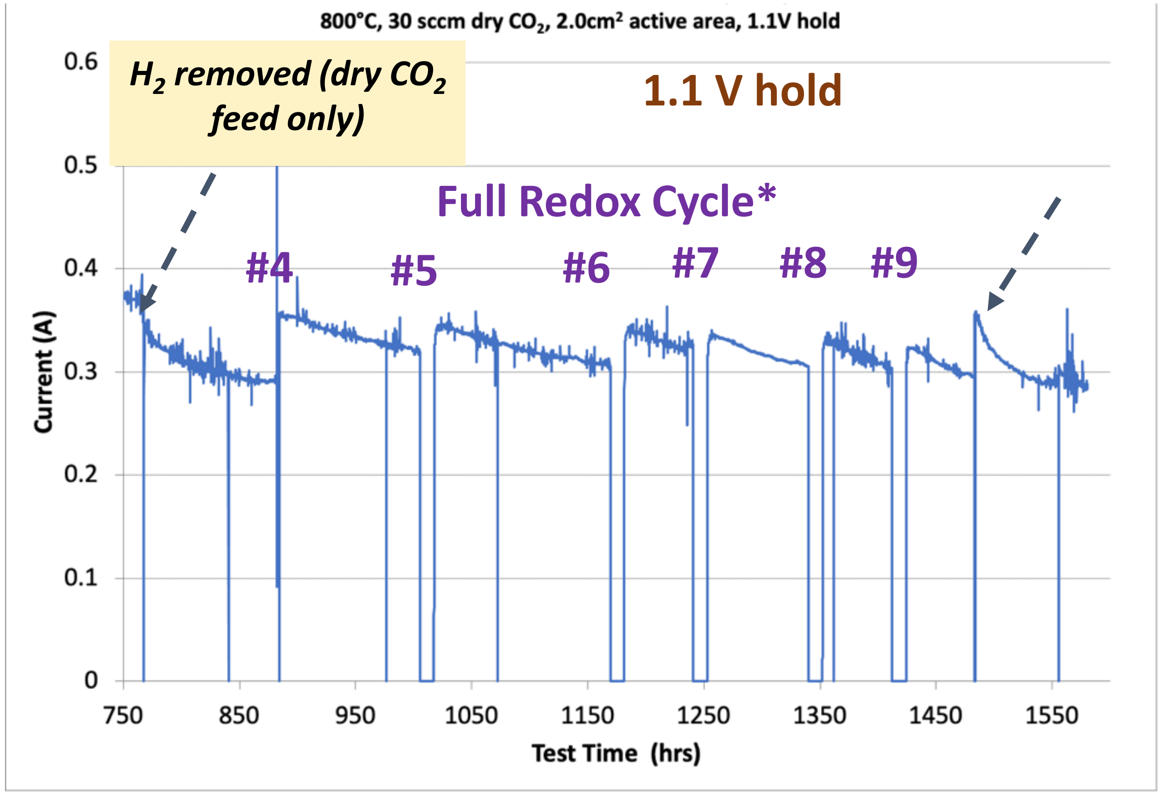
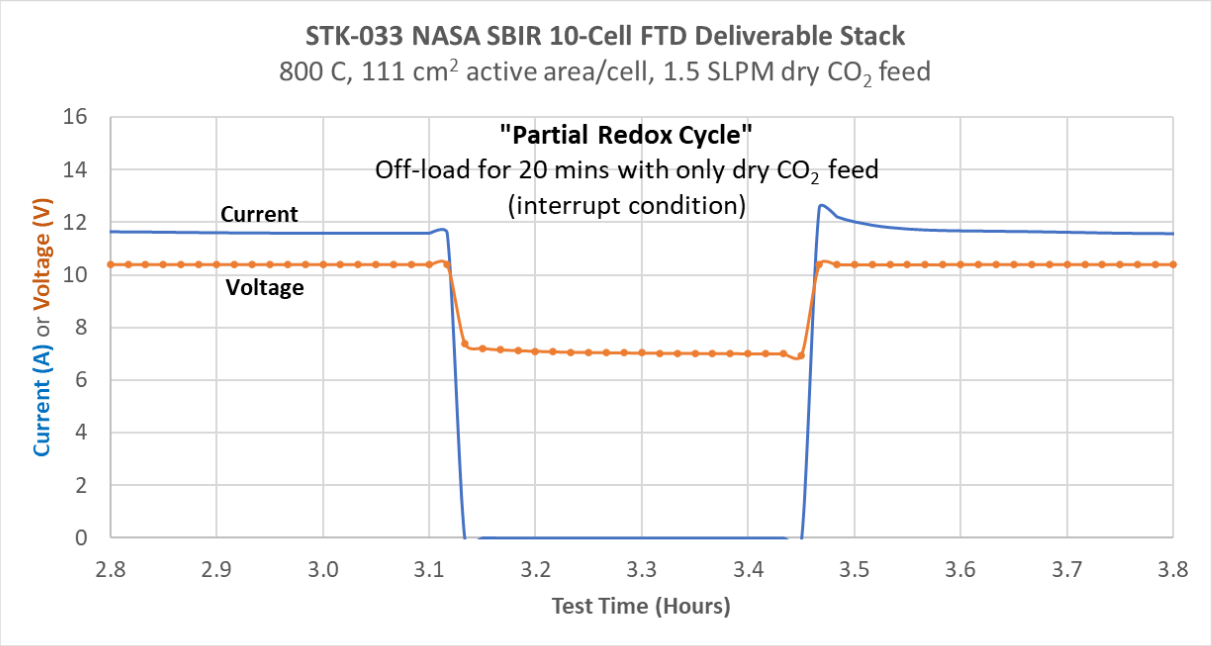


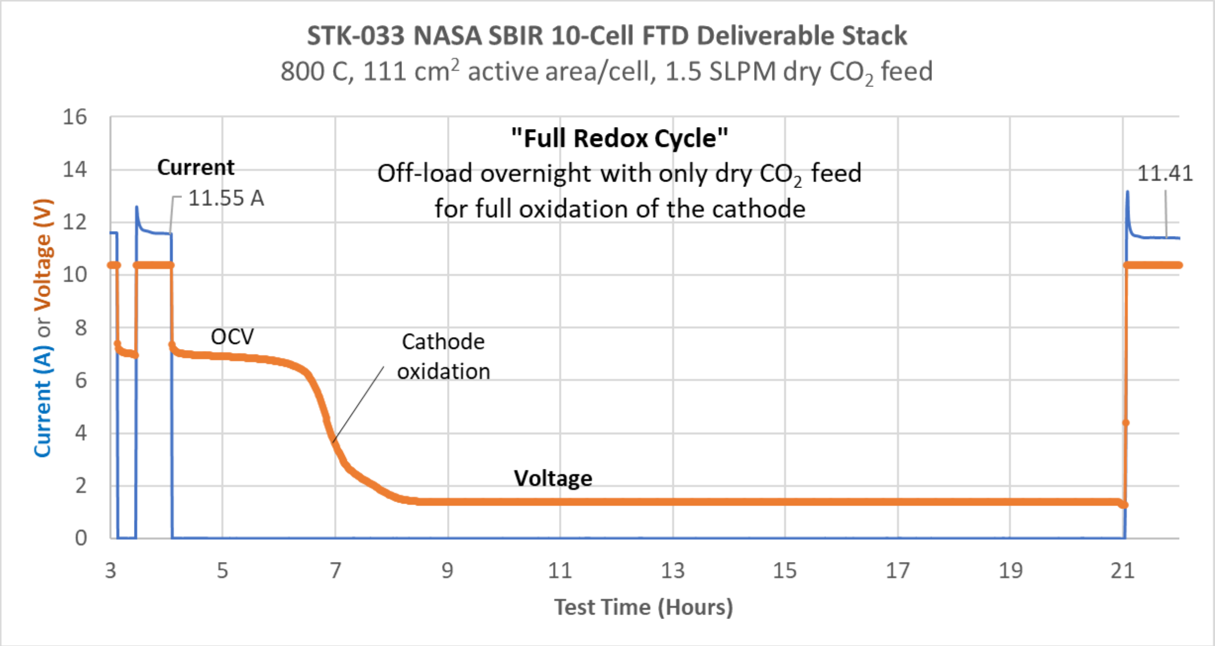
Down-selected Composition
Ni-Ceria based (N85)

- **Full Redox Cycle = 12 hours off load with dry CO_2 only feed**
- **Kept at 800 °C to nearly fully oxidize the cathode material (Ni metal → Nickel-oxide)**
- **Load is reapplied**
- **No external reducing gas**

NASA SBIR Funded

STK-033 Partial and Full Redox Cycles



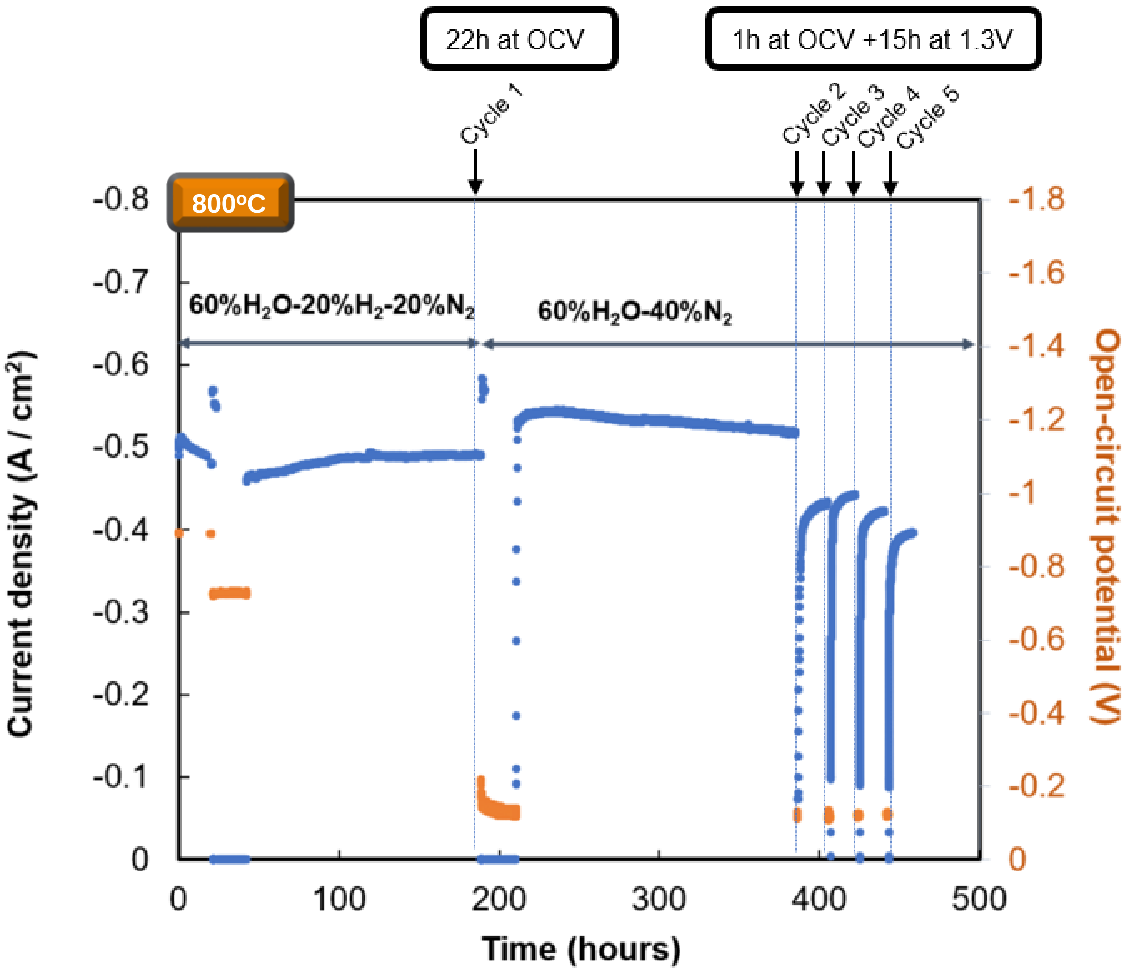


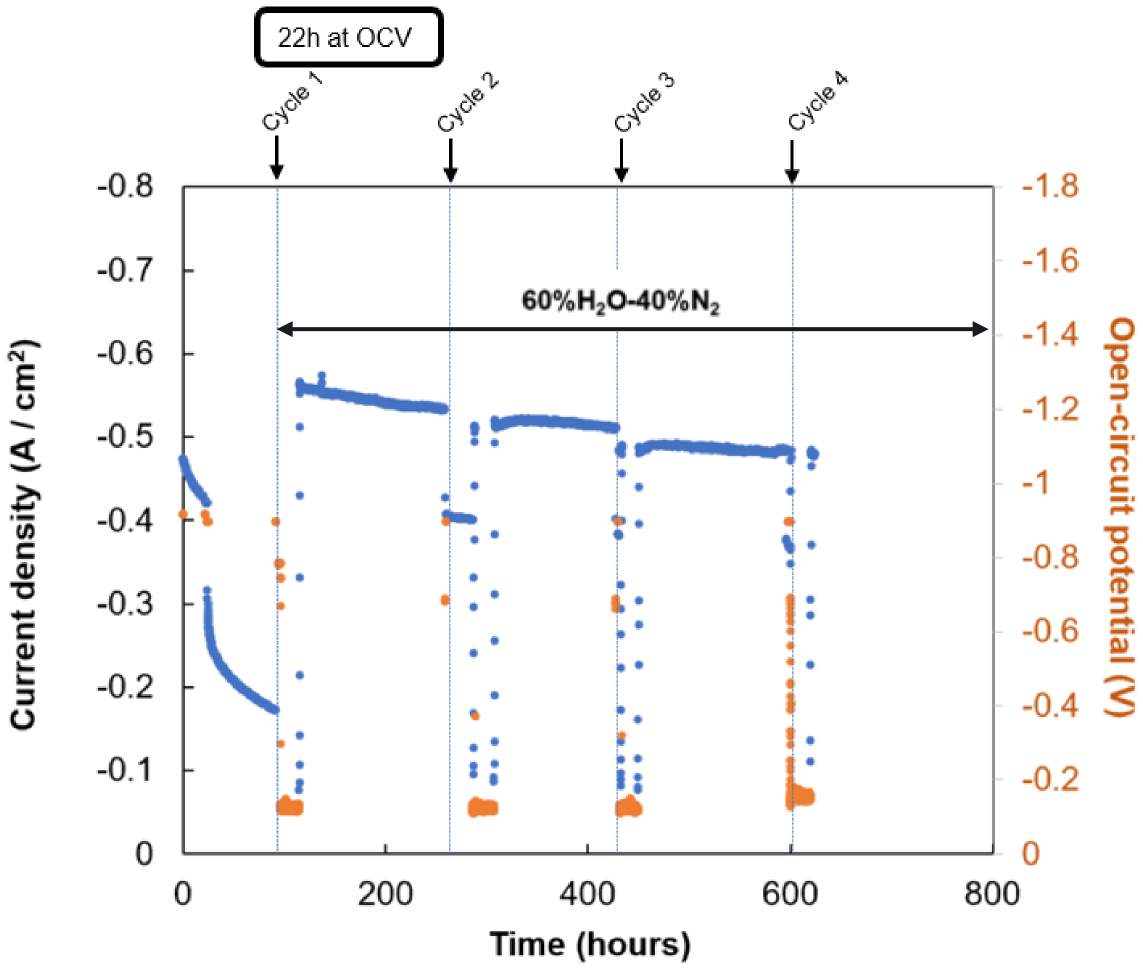
- **Stack: Short (20 min) and long (12 hrs) exposure to CO_2**
- **Application of voltage – full recovery of performance**

NASA SBIR Funded

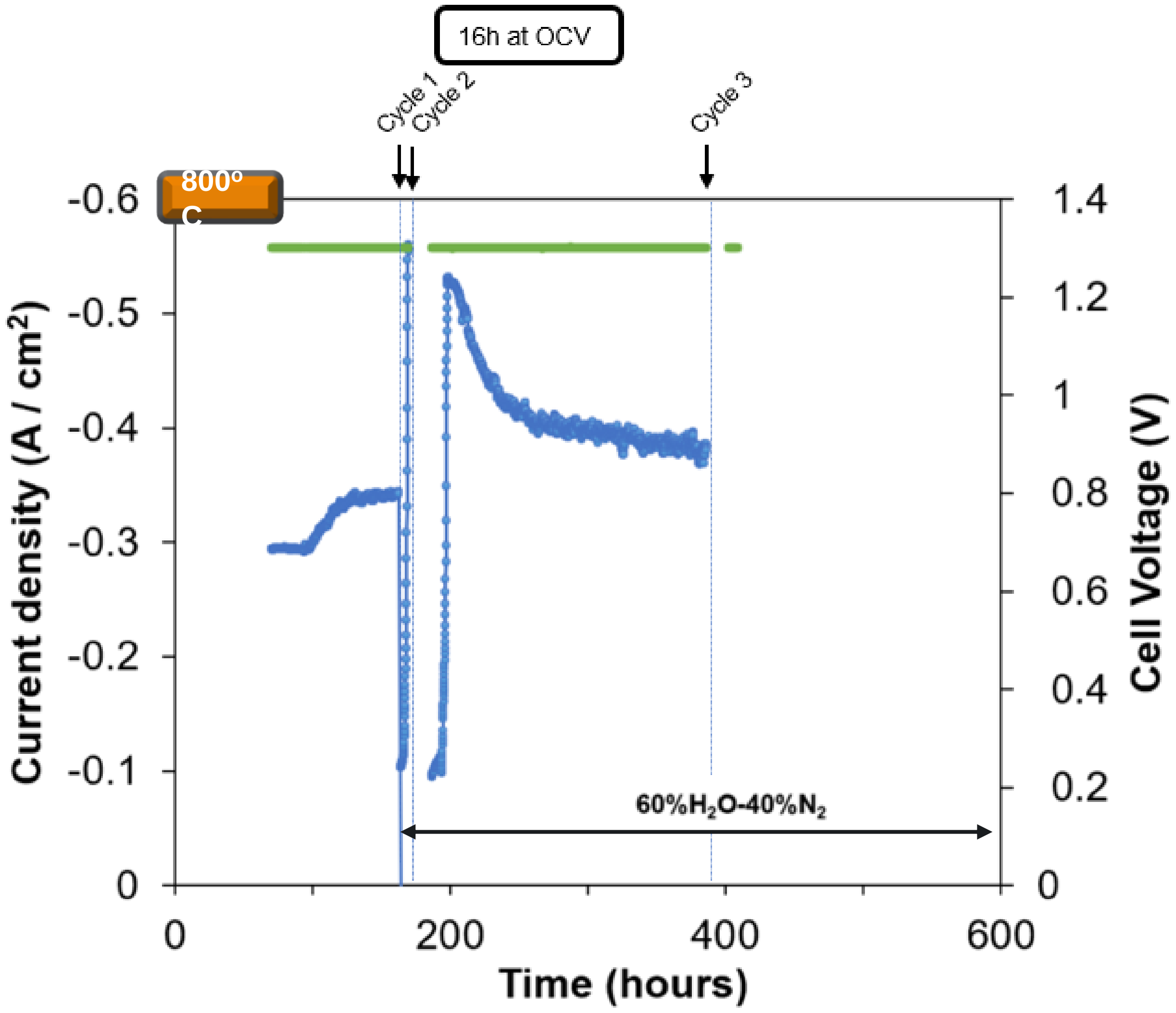
Redox test #1

Redox test #2





Redox Test #3



- 0.35 A/cm^2 at 1.3V initially with 20%H_2;
- Current increases after each redox cycle, then drops; 3 cycles completed: 1h, 16h, 16h

kW class stack: Redox Cycle in Steam Electrolysis

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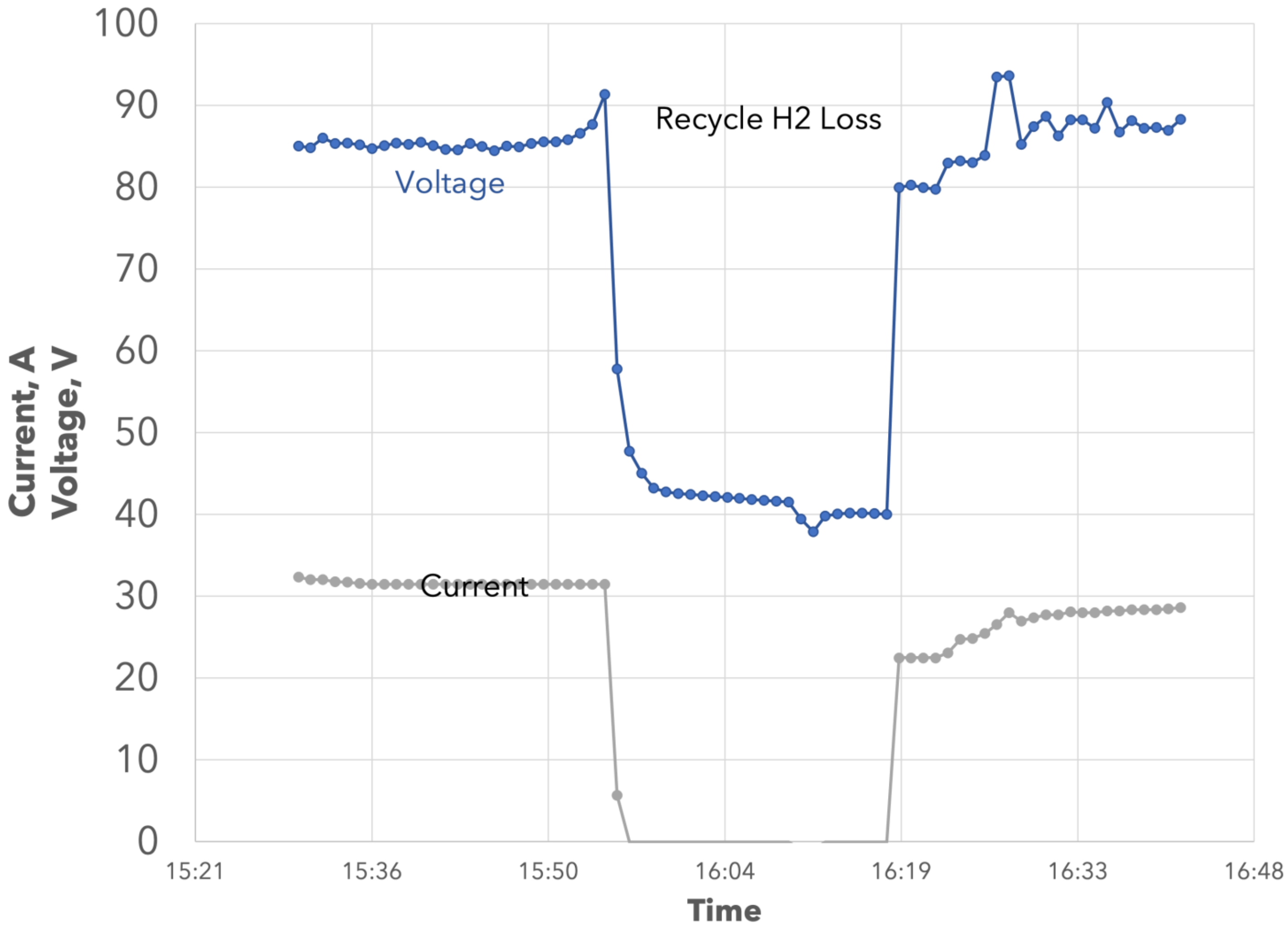

Stack test at Colorado School of Mines:

- **Stack in Lunar vacuum**
- **Power supply problem**
    - Recycle H_2 stopped
    - Current to zero
    - Voltage dropped
    - Restored power supply
    - Stack performance recovery

NASA Funded (Tipping Point Project)

Electrode Improvement - DE-FE0032105

Focus: Address known/suspected degradation mechanisms

Integrated Approach to Addressing SOEC Degradation

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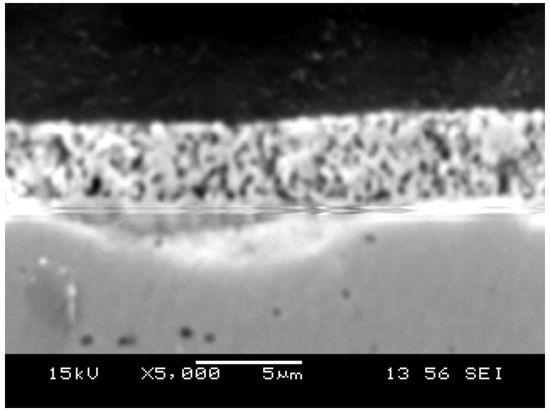
No.	Degradation mechanism	Effect	SOEC component	Activity	Project Support
1	Cr transport from interconnect	Poisons active electrochemical sites	O_2 electrode	Poisoning Effect (PNNL) Spinel Coating	**DOE/NETL** NASA Phase II-E
2	Perovskite composition instability over time	Catalytically inactive and electrically resistive grains/ Non-catalytic secondary phases	O_2 electrode / current collector	Composition modification	**DOE/NETL** NASA Phase II E
3	SiO_2 migration from seal	Contaminates electrodes	O_2 electrode Fuel electrode	Poisoning Effect (PNNL)	**DOE/ NETL** NASA Phase II E
4	Cation diffusion	Formation of more resistive phases	Electrolyte CeO_2 barrier	Process modification	**DOE/ NETL** NASA Phase II E

Oxygen Electrode Interface Improvement

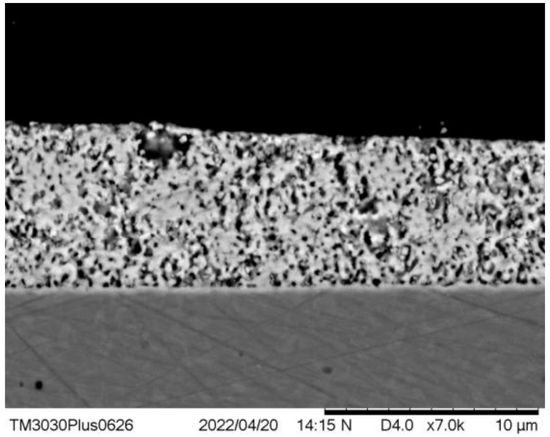
SDC Barrier layer

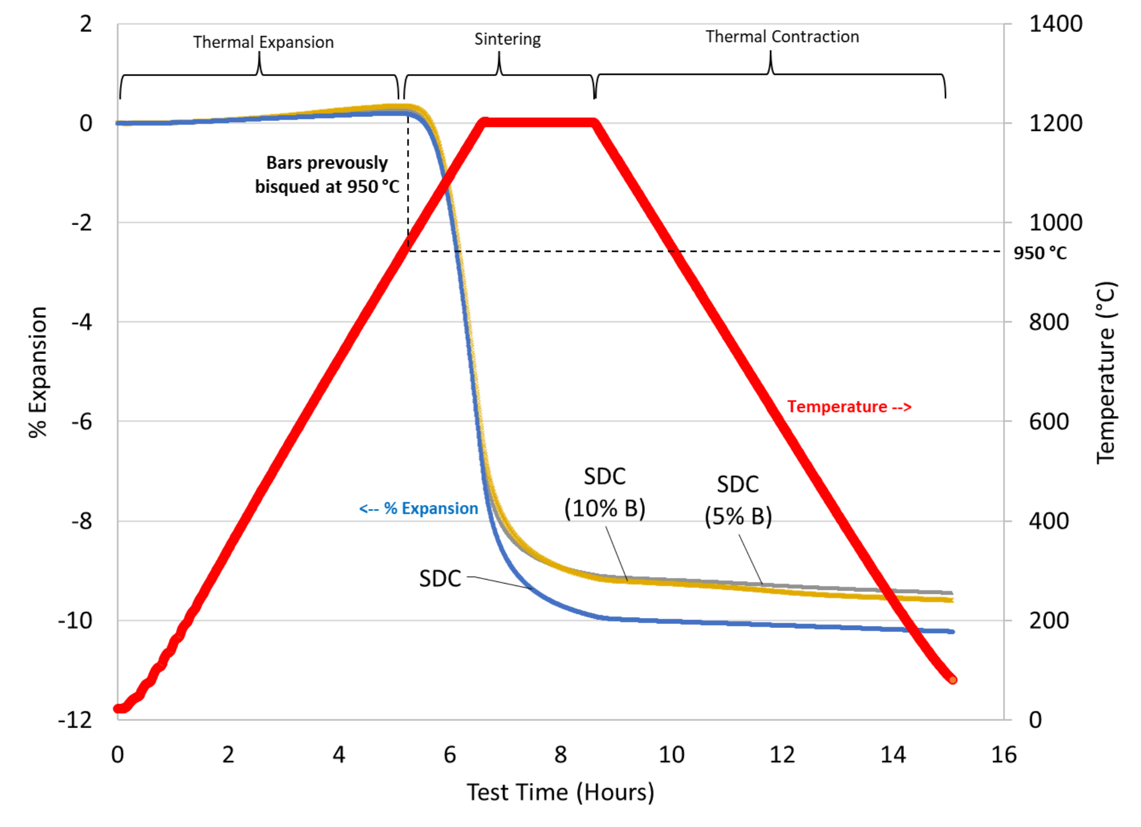
- Improve sinterability to lower sintering temperature
- Eliminate interface reaction between ceria and zirconia

Standard SDC



>150 °C Reduction



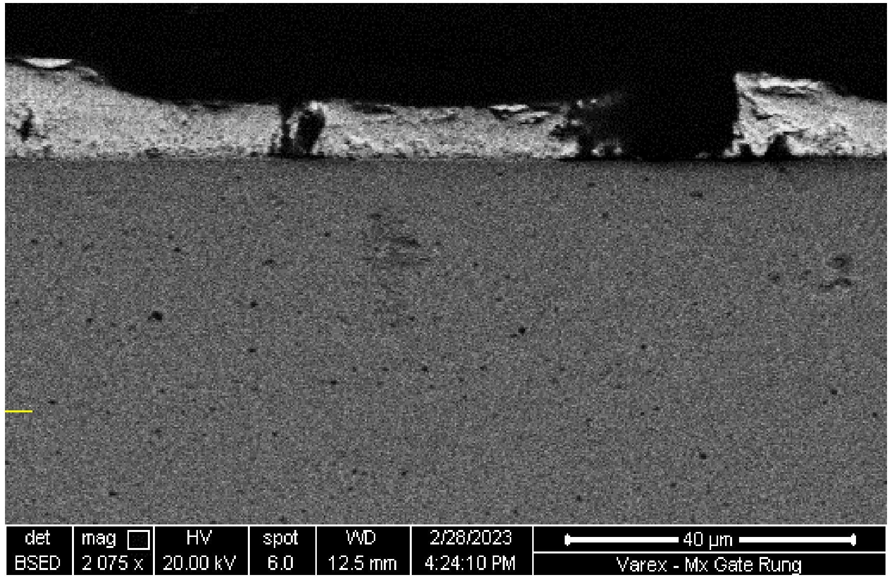


This Project + NASA SBIR

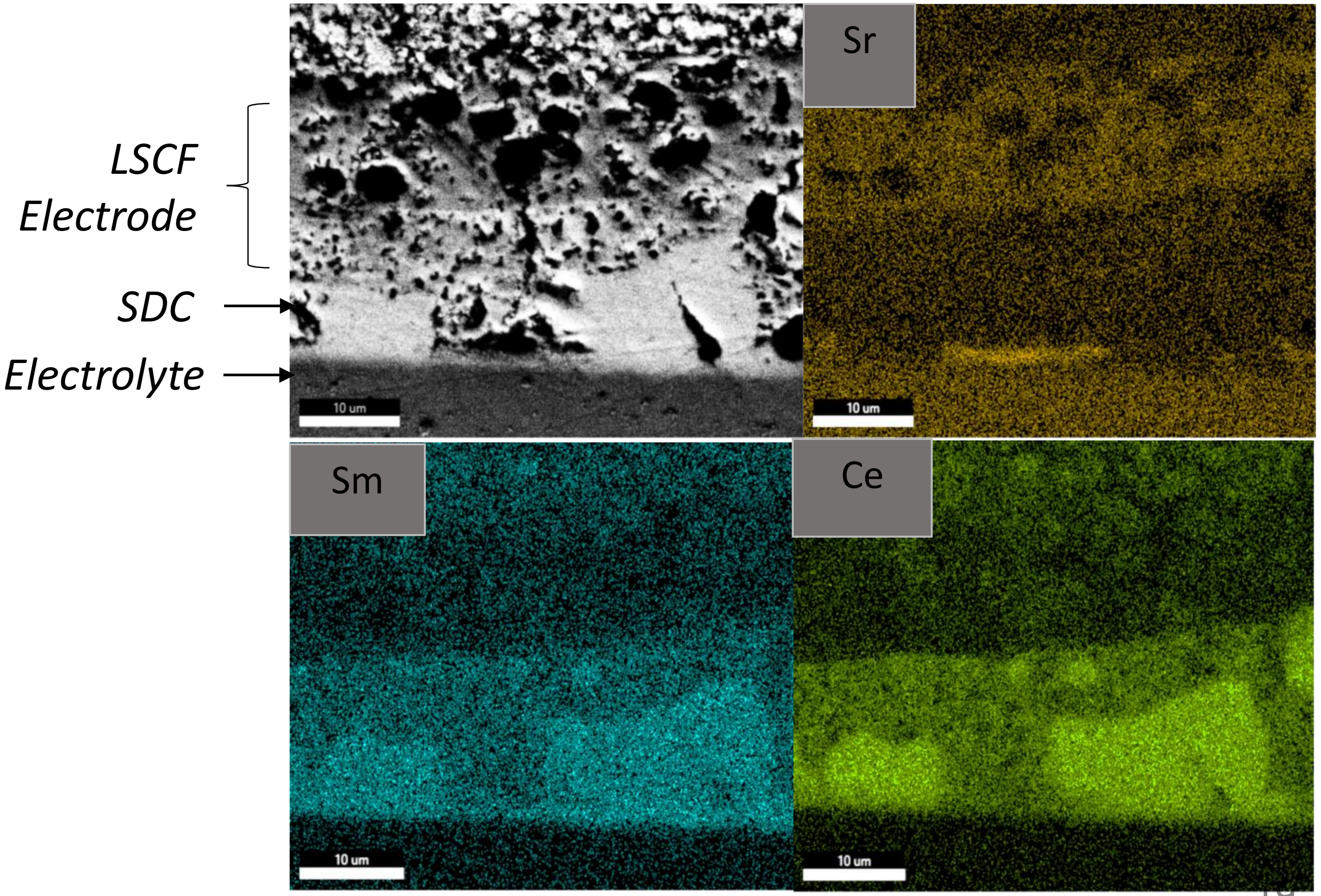
Oxygen Electrode Interface Improvement

SDC Barrier layer

- Sr migration occurs at discontinuous and porous regions in the barrier layer
- “Ideal” sintering temperature balances densification, interface reaction, & manufacturability (co-sintering with electrodes)



Discontinuous >150 °C Reduction barrier layer.



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Oxygen Electrode / Current Collector

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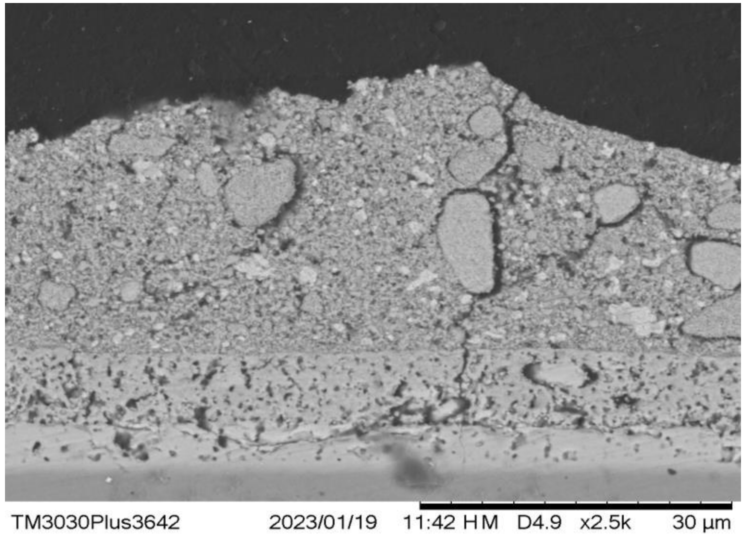
Eliminate Sr in current collector layer by replacing LSCF with Sr-free perovskite mixed with Ag-alloy

- Ohmic resistance close to expected based on electrolyte thickness
- Polarization resistance indicate LSCF layer is not required for water splitting

LCAP10 current collector shows good bonding to electrode layer



Electrode	Current collector met Vol% in cermet	ASR (Ω·cm^2)		
		Total	Ohmic	Polarization
Sr-free	10%	0.79	0.66	0.13
Sr-free	30%	0.66	0.59	0.08
Sr-free	50%	0.62	0.50	0.12
LSCF	50%	0.71	0.52	0.19

This Project

Oxygen Electrode / Current Collector

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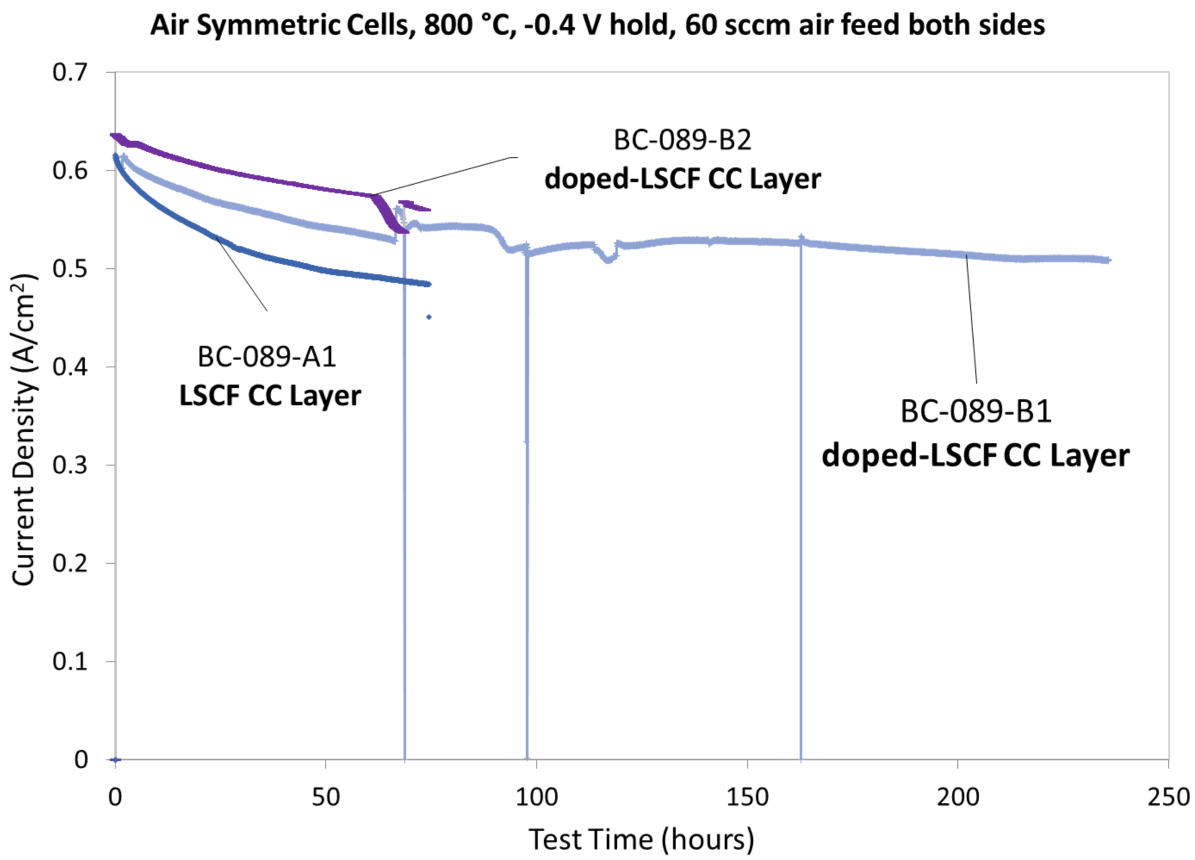
Sr-stabilization in oxygen electrode – multiple approaches

- A-site deficiency in the LSCF perovskite ($A_{1-x}BO_3$)
- Additional dopant in LSCF
- Using LSCF-composite current collector
- LSCF surface treatment

Doped LSCF CC layer cell performance stabilizes after initial degradation, compared with LSCF CC



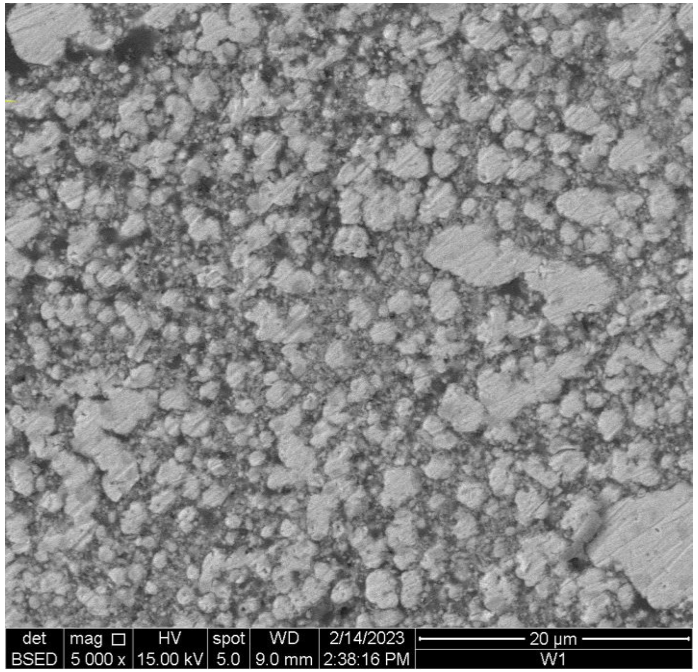
NASA SBIR + Private microgrid

Fuel Electrode / Current Collector

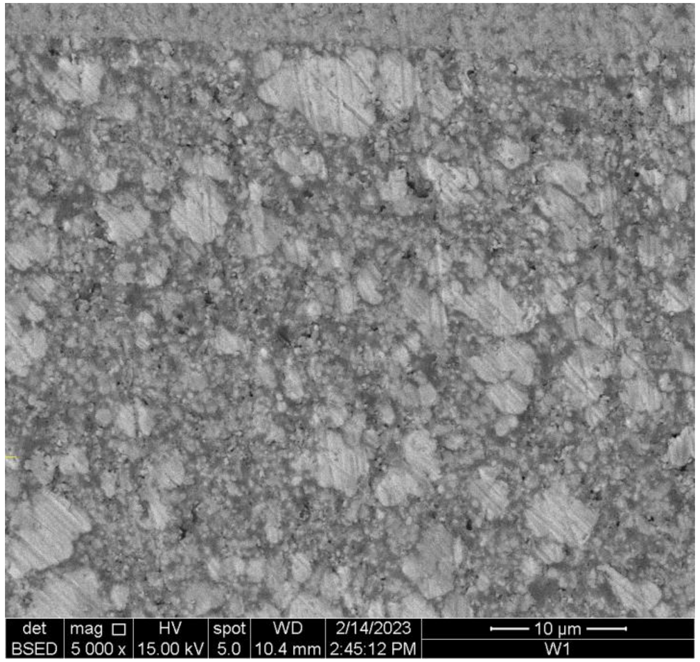
Reduce current collector cost by reducing Ag-alloy concentration (15%, 30%, 50%)

- Reduce cost – but maintain performance and redox tolerance!
- Button cell performance is comparable with lower Ag-alloy concentration – processing modifications will reduce Ag-agglomeration and improve interconnected metallic network

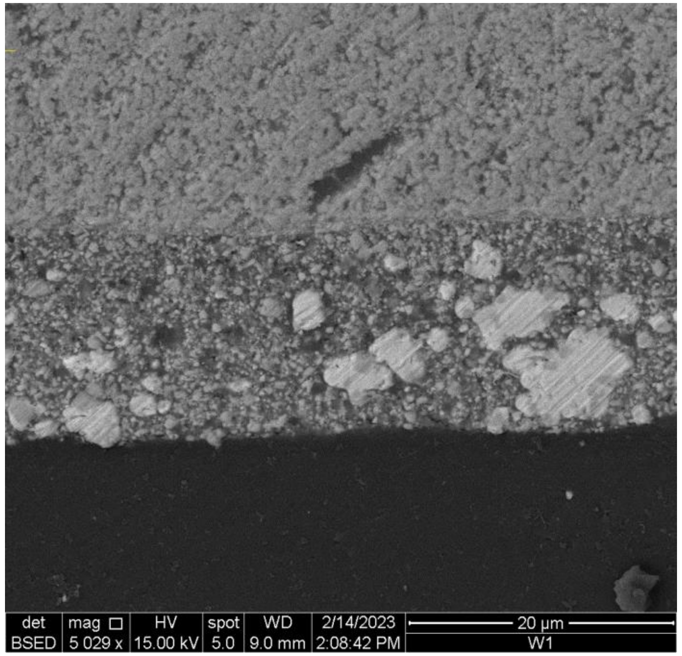
50% Ag-alloy



30% Ag-alloy



15% Ag-alloy



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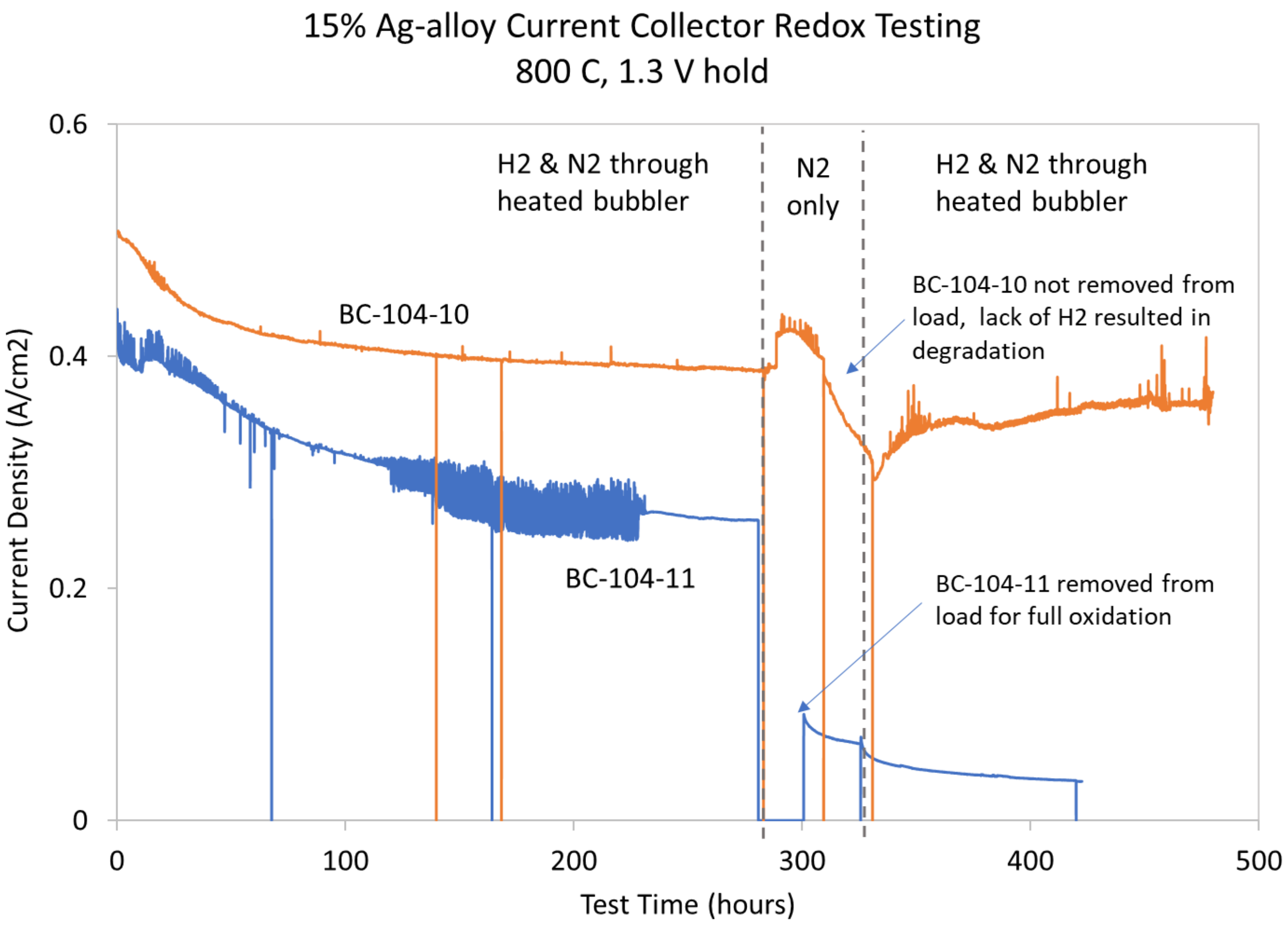
Fuel Electrode / Current Collector

15% Ag-alloy current collector is not redox tolerant

- Steam redox cycle tests show lower Ag-alloy current collector is not redox tolerant
- Removing H_2 from the feed (N_2 and Steam only) resulted in high degradation rate
- Full redox cycle resulted in large performance drop. Cell did not recover.
- Q: better distribution or more Ag??



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Electrode Catalyst

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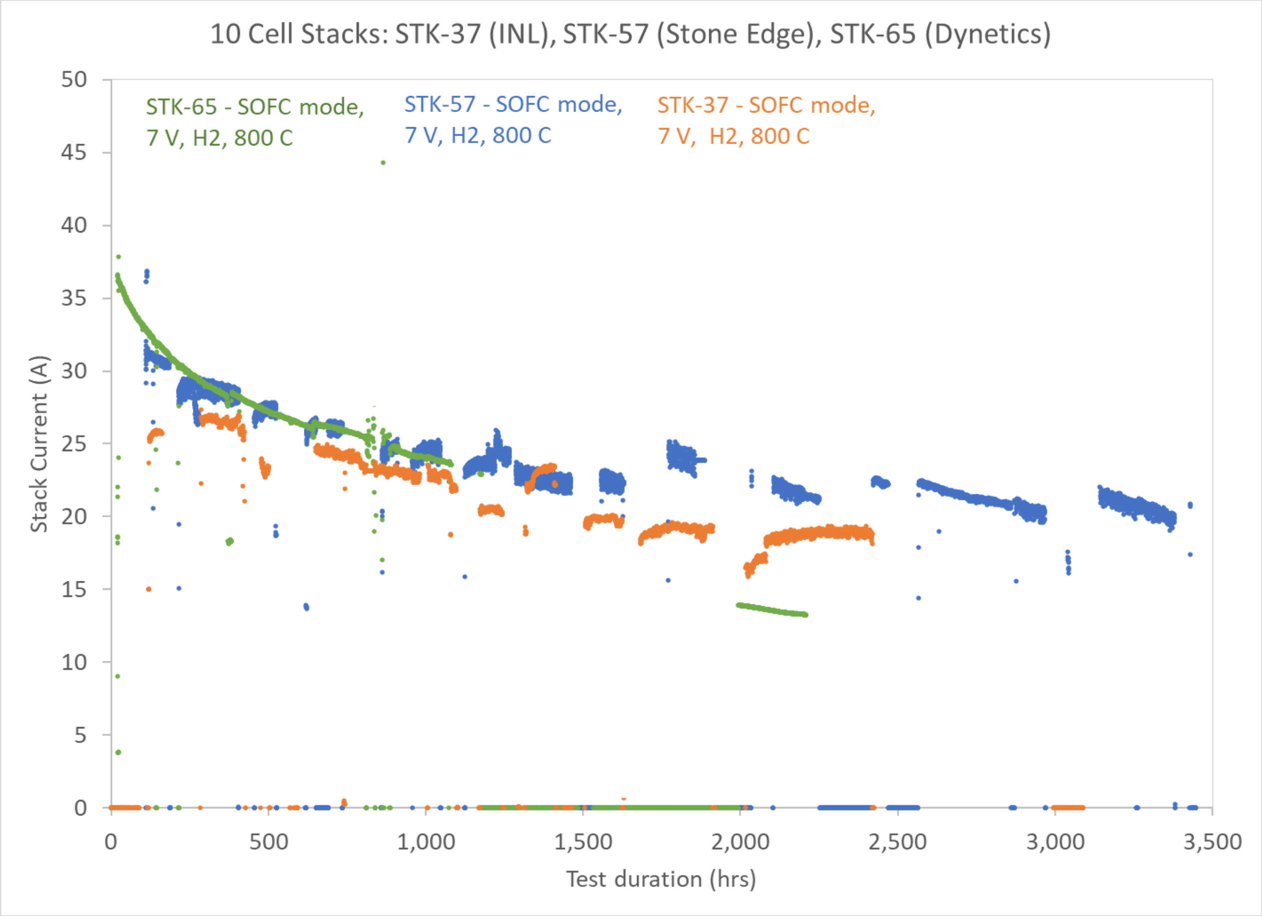
Historical 10-cell stack tests show incremental improvements in SOFC operation



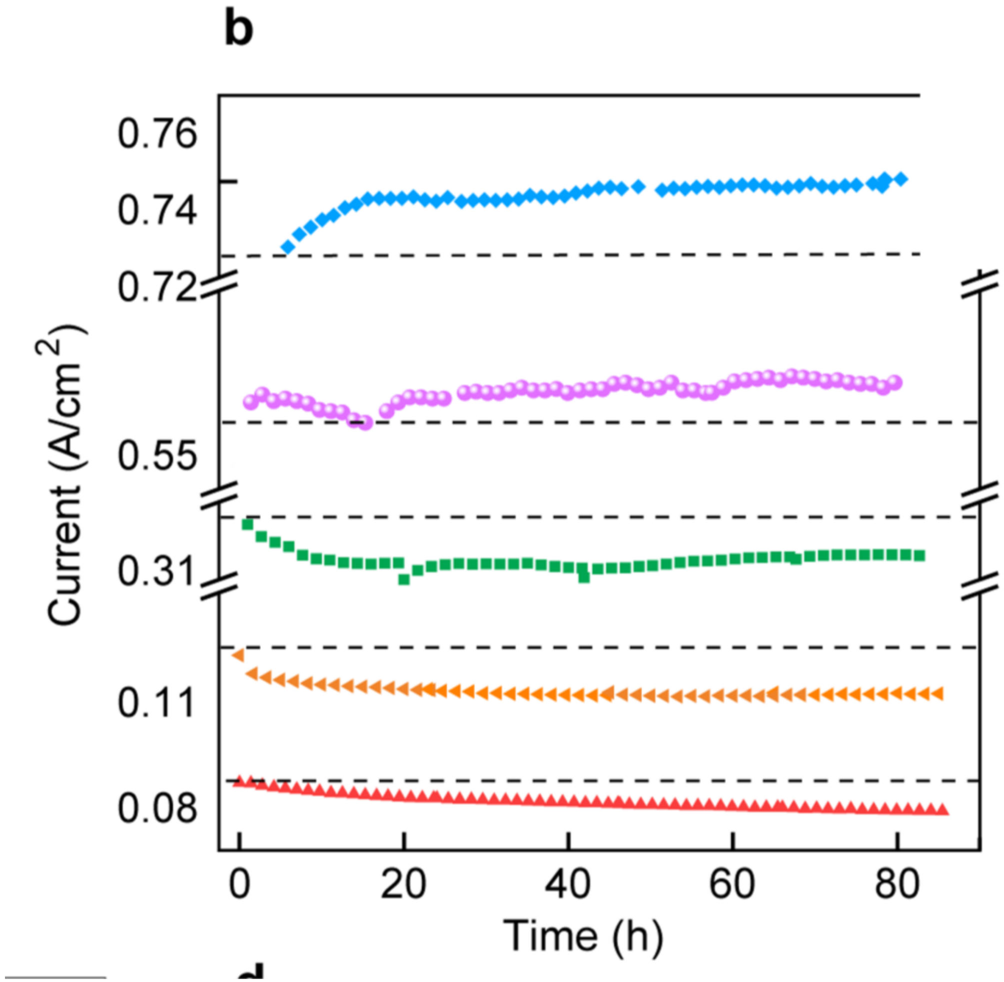
Stack	STK-37	STK-57	STK-65
Program	INL 30 kW	Stone Edge Farm	NASA SBIR II-E
Fuel Electrode	Ni-SDC	Ni-SDC	Redox tolerant
Fuel Electrode Infiltrant	Yes	Modified	modified
Air Electrode barrier layer	SDC	Mod-SDC	Mod-SDC
Air Electrode I	Sr-free+SDC	Sr-free+SDC	Sr-free+SDC
Air Electrode II	LSCF	LSCF+SDC	LSCF+SDC
Air Electrode Infiltrant	Yes	Surf treat + infilt	Surf treat + infilt

This Project + NASA SBIR + INL 30 kW + Stone Edge Farm

Materials Validation and Performance Mapping

Button cells will be tested in steam electrolysis range of temperature, steam utilization, and cell voltages to assess performance and stability

- Cathodic overpotential modifies the surface of LSCF-SDC electrode and improves the chemical and electrochemical stability
    - Main objective: Suppression of Sr segregation



This Project + Stone Edge Farm (MIT)

Effect of Impurities on Fuel Electrode Performance

Effect of **Mn** impurities on the **fuel electrode** in SOEC mode

PNNL – button cell testing with stainless steel shows no Mn in EDS analysis

Tested for 168 hour at 800°C at 0.7V

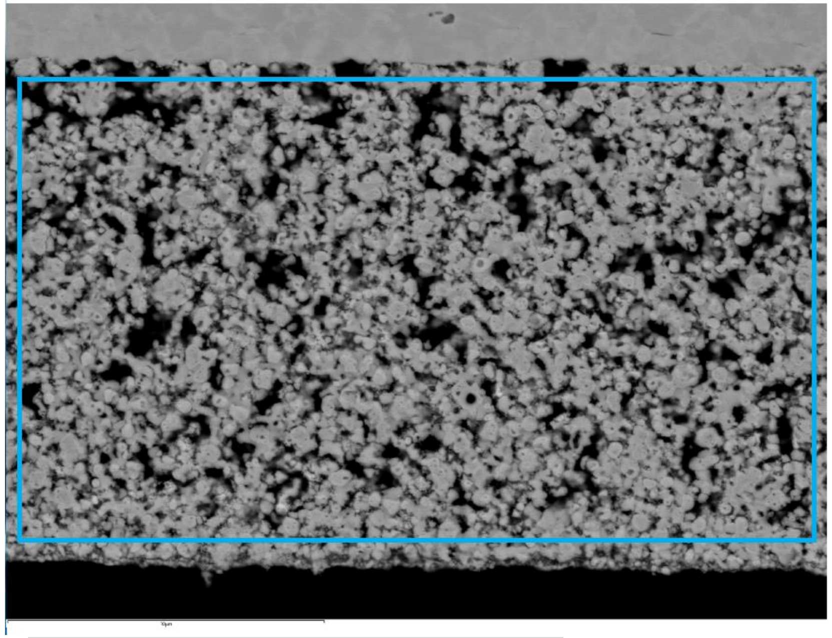
Cell was degrading continuously

No Mn detected in Ni by SEM/EDS

BC-080-? (cell #4)

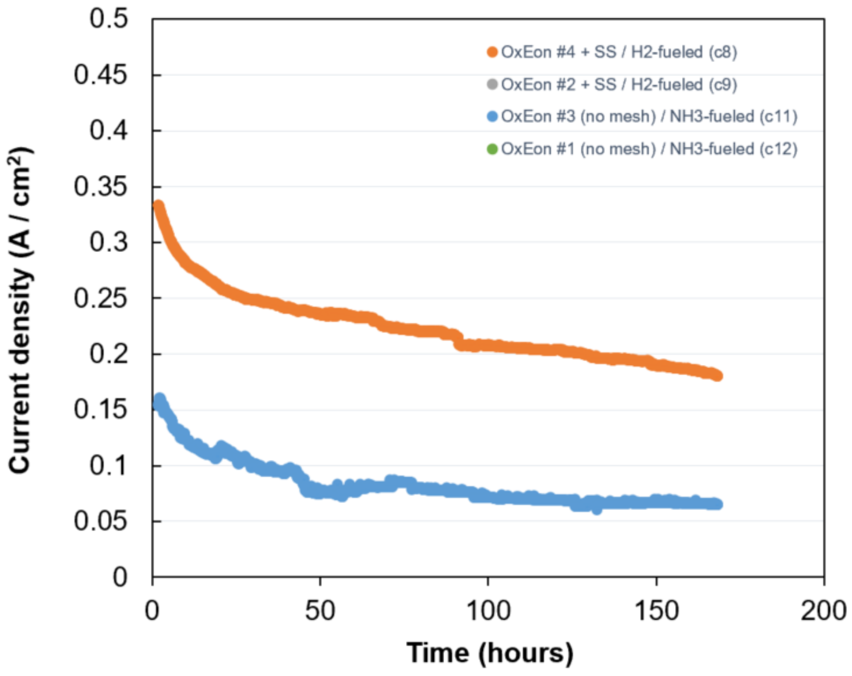
Tested in 100% H_2 for 168 hour at 800°C at 0.7V: **H_2 electrode with SS**

- Electrolyte to H electrode interface – BSE image



No Mn signal

H electrode	Atomic %
O	34.99
Ni	31.32
Zr	16.71
Ce	7.83
Mg	2.92
Al	2.51
Y	1.45
Cu	1.39
Sm	0.64
Sc	0.17
Ca	0.08
Pr	0.00
Total	100.00



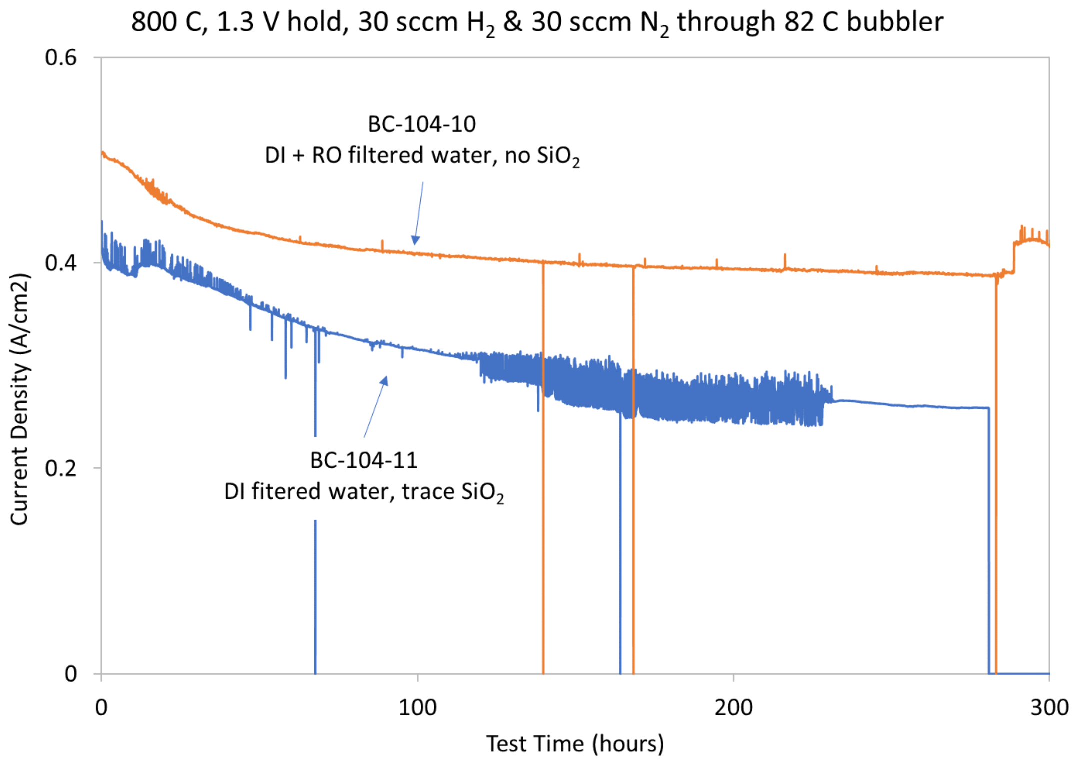
This Project + NASA SBIR

Effect of Impurities on Fuel Electrode Performance

Effect of **Si** impurities on the **fuel electrode** in SOEC mode

- Button cell on steam electrolysis with RO filtered water has measured better stability (BC-104-10).
  - DI water from supplier contained 0.3 mg/L SiO_2
  - No SiO_2 detected after Reverse Osmosis filtration

- *Improved stability in SOEC when steam is produced from RO-treated water.*
- *Distilled water contains trace SiO_2*



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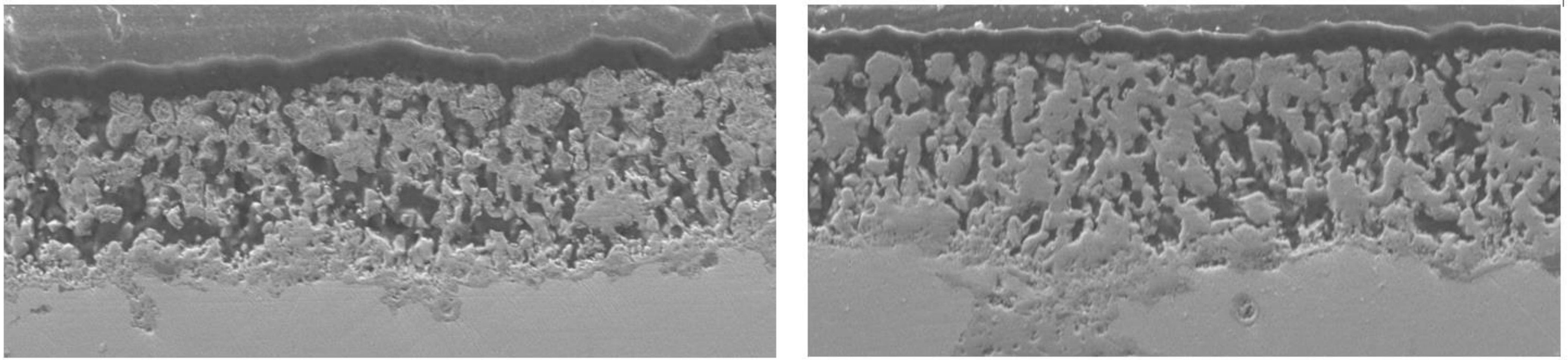
Effect of Impurities on Oxygen Electrode Performance

Improve Cr-barrier interconnect coating

- Optimized coating dispersant, powder loading, and viscosity to increase barrier layer density
- High-density coating regions near the CFY substrate with porosity further up the coating thickness.
- Next steps – characterization of electrodes placed in contact with coated interconnect coupons at 800 C



Two different dispersants selected

Oxygen Production (Seal Validation)

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- **High Purity O_2 on Mars**
  - External to stack Mars ambient ~ 7 millibar
- **Oxygen production at pressure (steam electrolysis test at CSM in vacuum chamber)**
  - Stack in vacuum
  - H_2 production at 1 bar
  - O_2 production up to 3.6 bar via electrochemical compression
- **This Project**
  - Short stack testing in pressurized test stand
  - Test stand modifications are underway

This Project

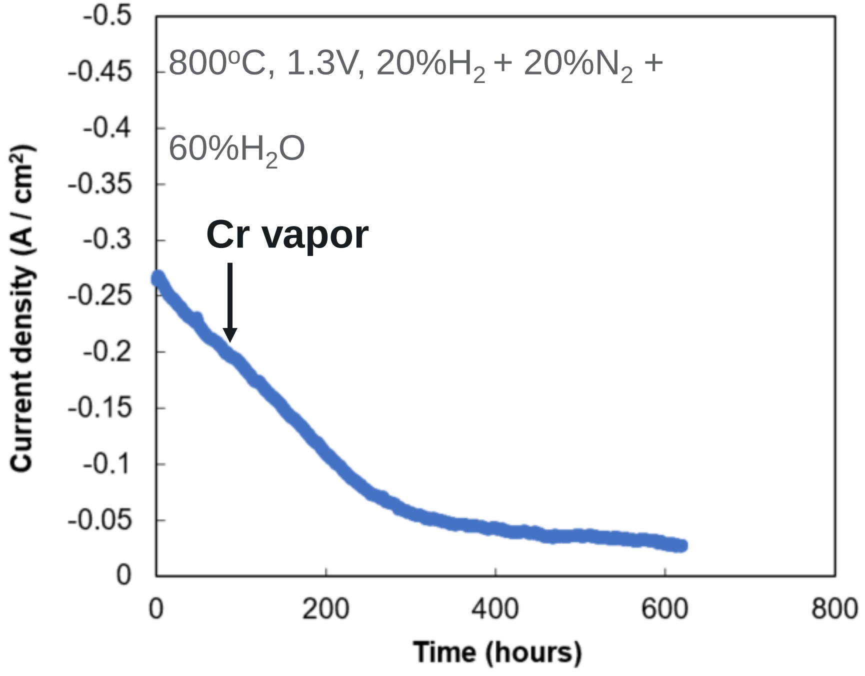
MOXIE scale stack (left) and demonstration system scale stack (right)

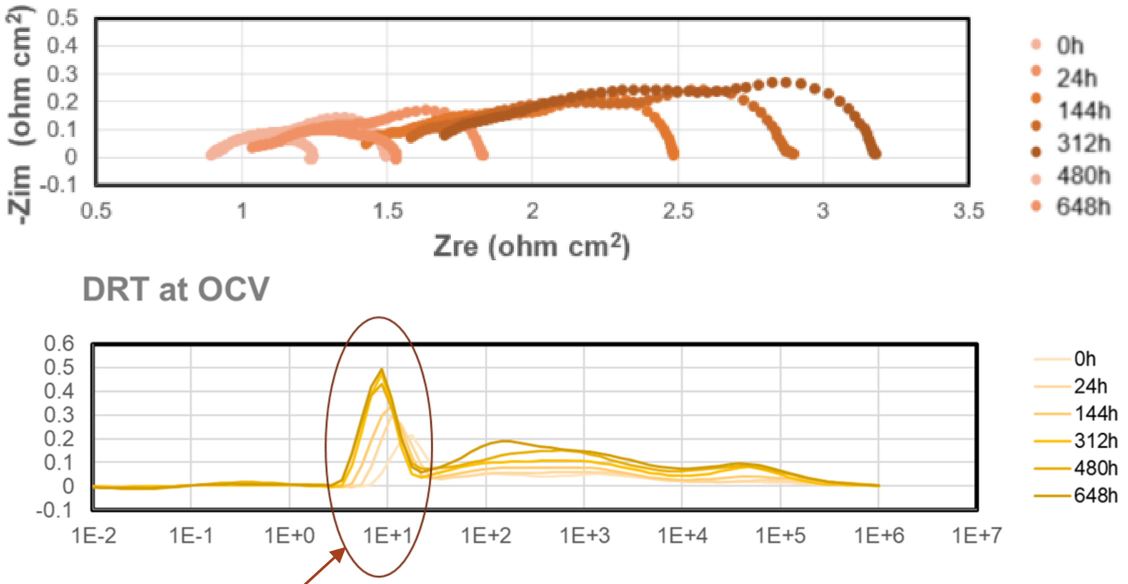
NASA Support

Cr Poisoning Tests

- Cr poisoning tests were performed 3 times, each time using 2 cells
  - Dry air, 800ºC, ~ 1 ppb Cr
  - Wet air, 800ºC , ~ 1 ppm Cr

Cr in getter post-test

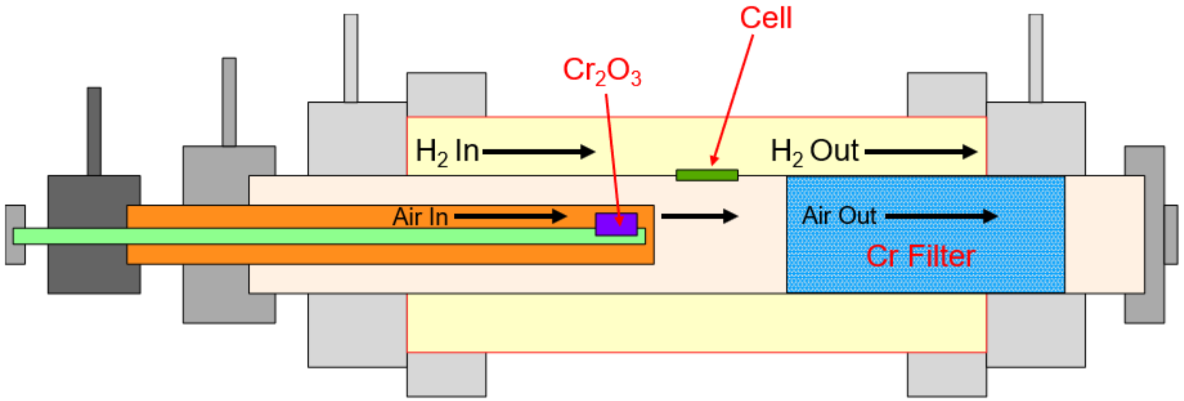


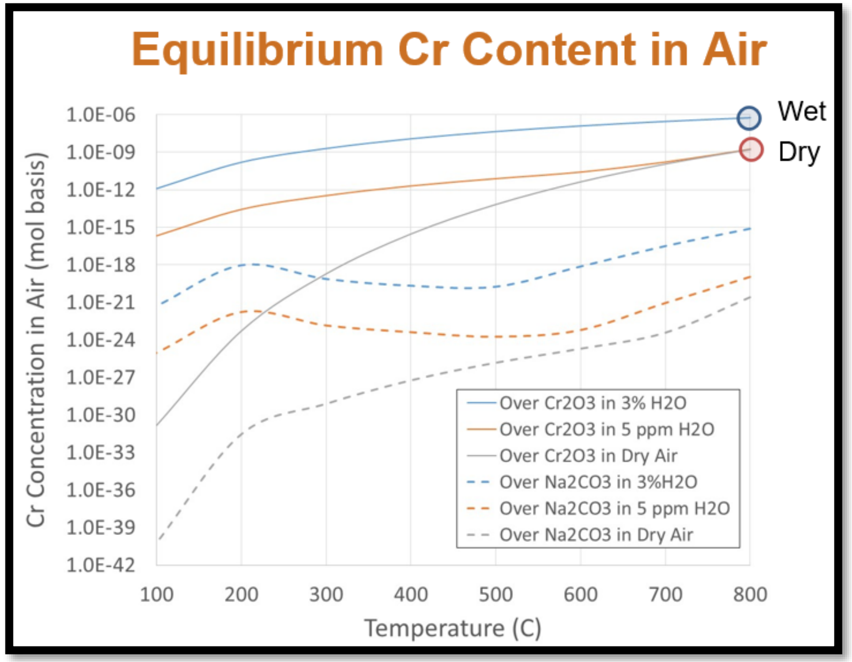


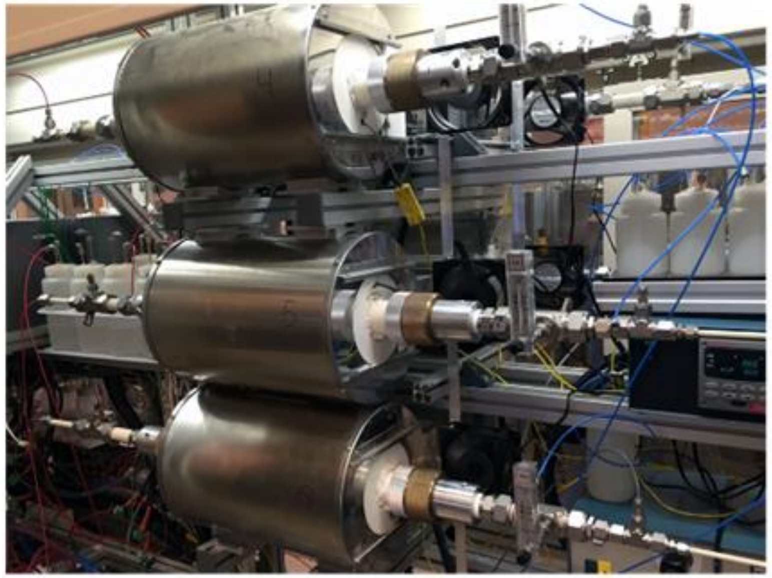
Process increases during testing: Cr poisoning
Not growing in time in clean air

Yet no Cr detected in oxygen electrode by SEM/EDS

Test Description

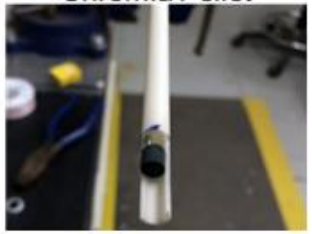


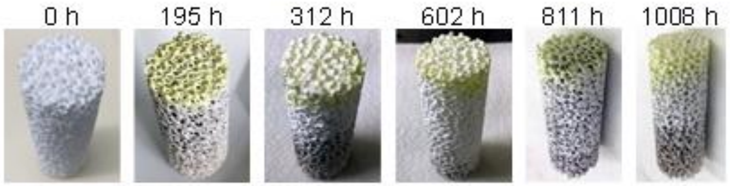




Downstream Filter

Chromia Pellet





Next Task: Pressurized SOEC Operation

Pressurized cell test rig

- High-pressure rig was built in 2022 but had to relocate to a different building
- Faced building operation and infrastructure issues and delays preventing H_2 usage; issues have been resolved
- Initiated pressure regulators testing and troubleshooting the system
- Aiming to start cell testing next week

Summary

- **Multiple projects to provide complementary scope/results**
- **Redox tolerance validated for steam electrolysis**
  - Oxidized Ni electrode recovery without the need for hydrogen in inlet
  - Modifications for improvements will be validated
- **Electrode materials modification – validation in progress**
  - Composition to improve thermochemical stability
  - Surface modification for improving catalytic property
- **Investigation of poisoning effect – ongoing**
- **Pressurized tests: steam electrolysis**
  - button cells – to begin shortly
  - Stack – in Year 2

Thank you

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