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
October 25-27, 2022

DOE Project Manager: Drew O'Connell

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Company Background

Utah, USA R&D/Manufacturing - 2017

- Office, laboratory, and manufacturing facility (24,000 ft^2)
- NASA, DOE, DOD and commercial contracts
- Tape casting, cell and stack production, and testing
- End-to-end power to synfuels pilot plant

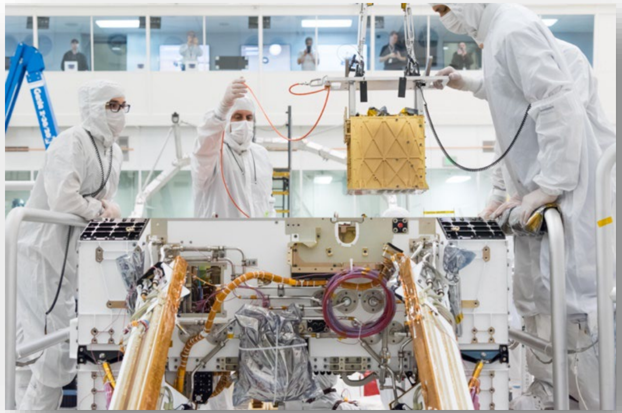


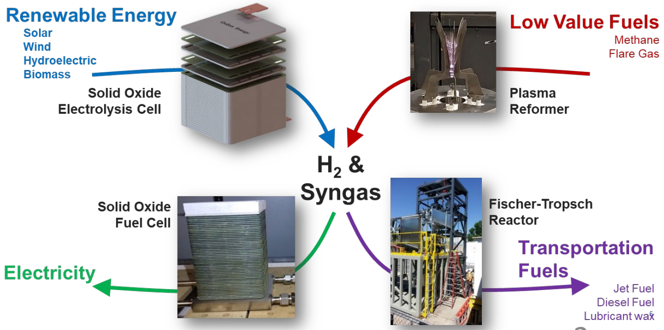
Image credit NASA/JPL-Caltec

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

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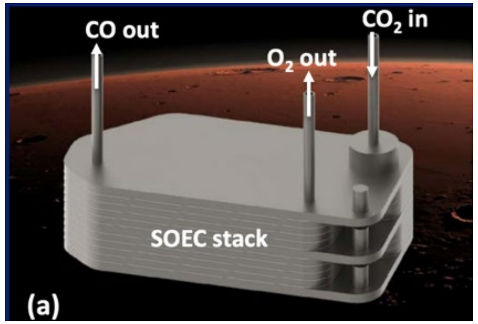
Mars **OX**ygen **I**SRU **E**xperiment aka "The Oxygenator"

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)

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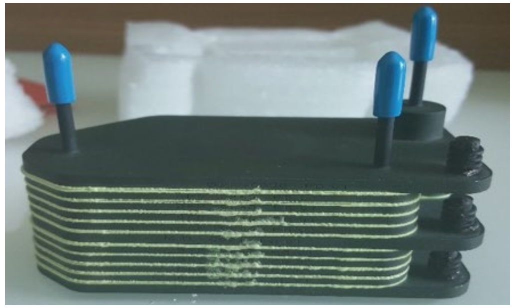
PLANSEE

JPL Jet Propulsion Laboratory

MIT



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Flight Qualification

Baseline Performance

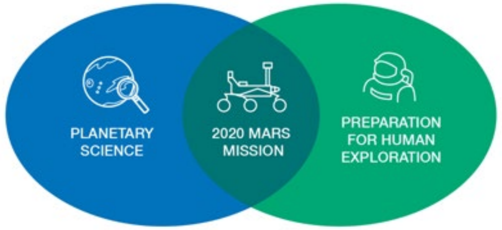
- **21 consecutive stacks** built with *aerospace quality standards and traceability* having a maximum baseline performance of 1.6 ohm-cm^2 dry CO_2 and 99.9%+ O_2 purity

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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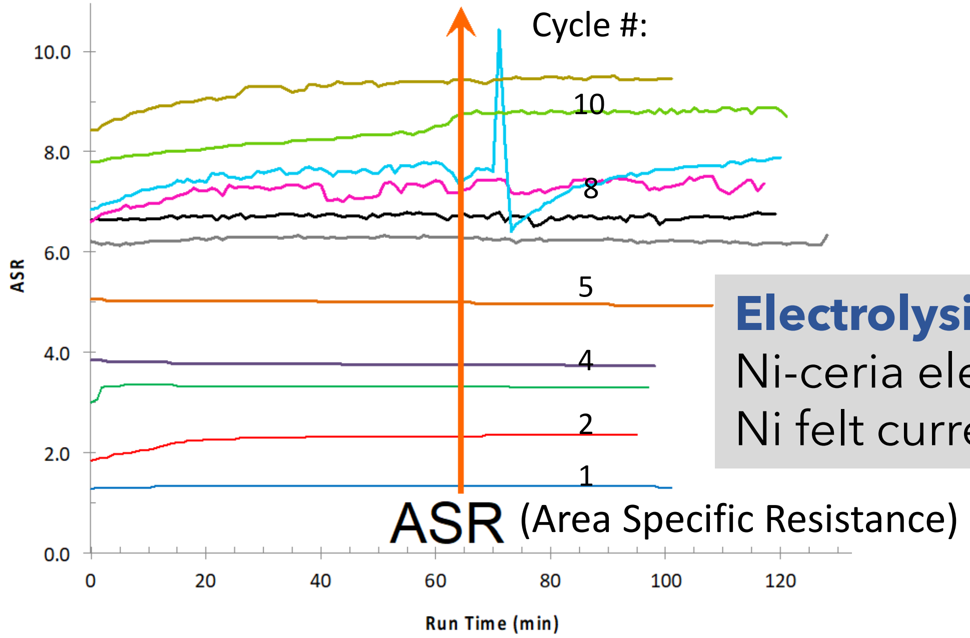
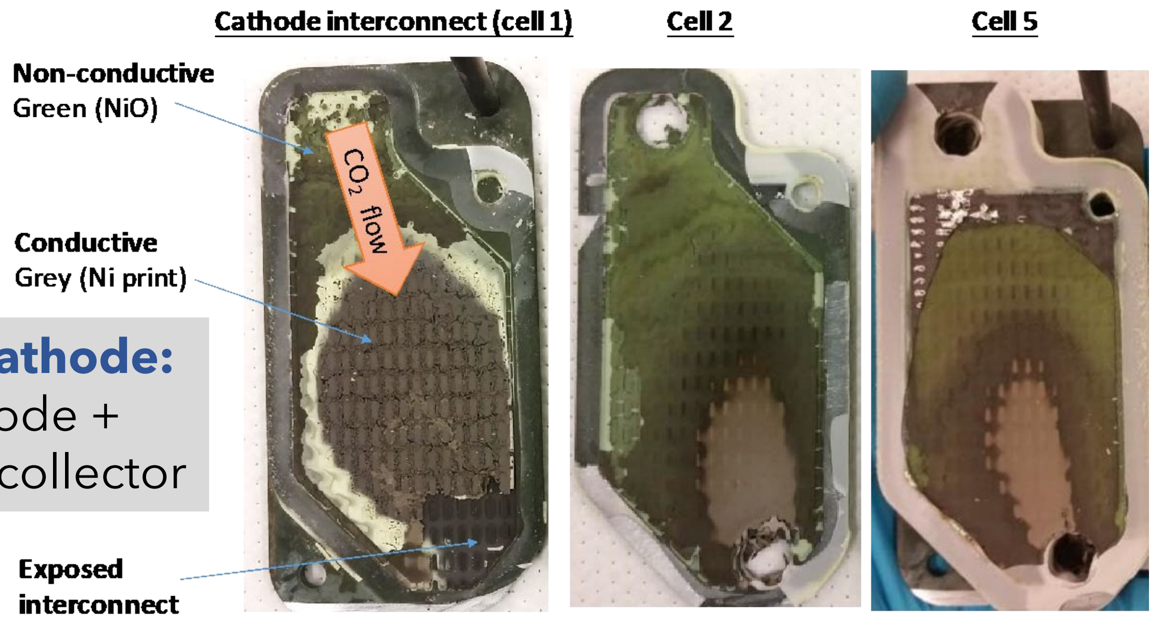
Cryo-Cycling

- 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



- 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

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

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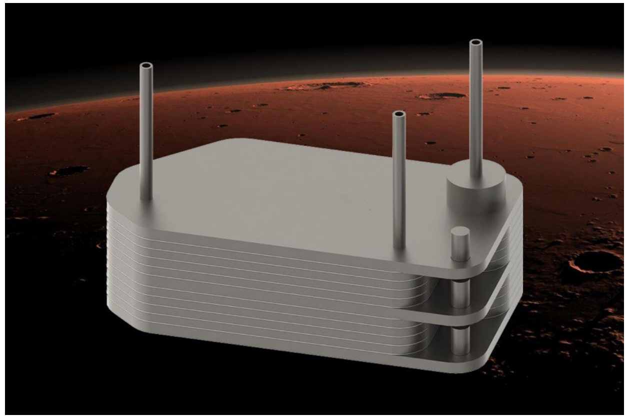
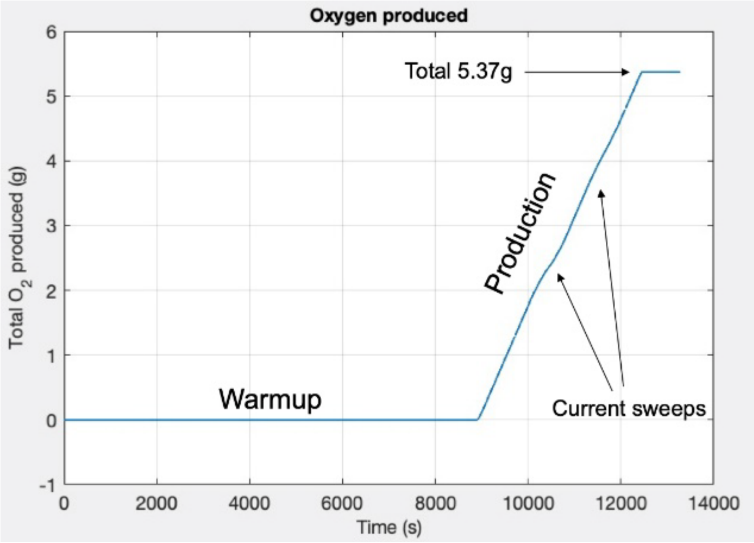
Flight Test Success – First Ever ISRU Demonstration!

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

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

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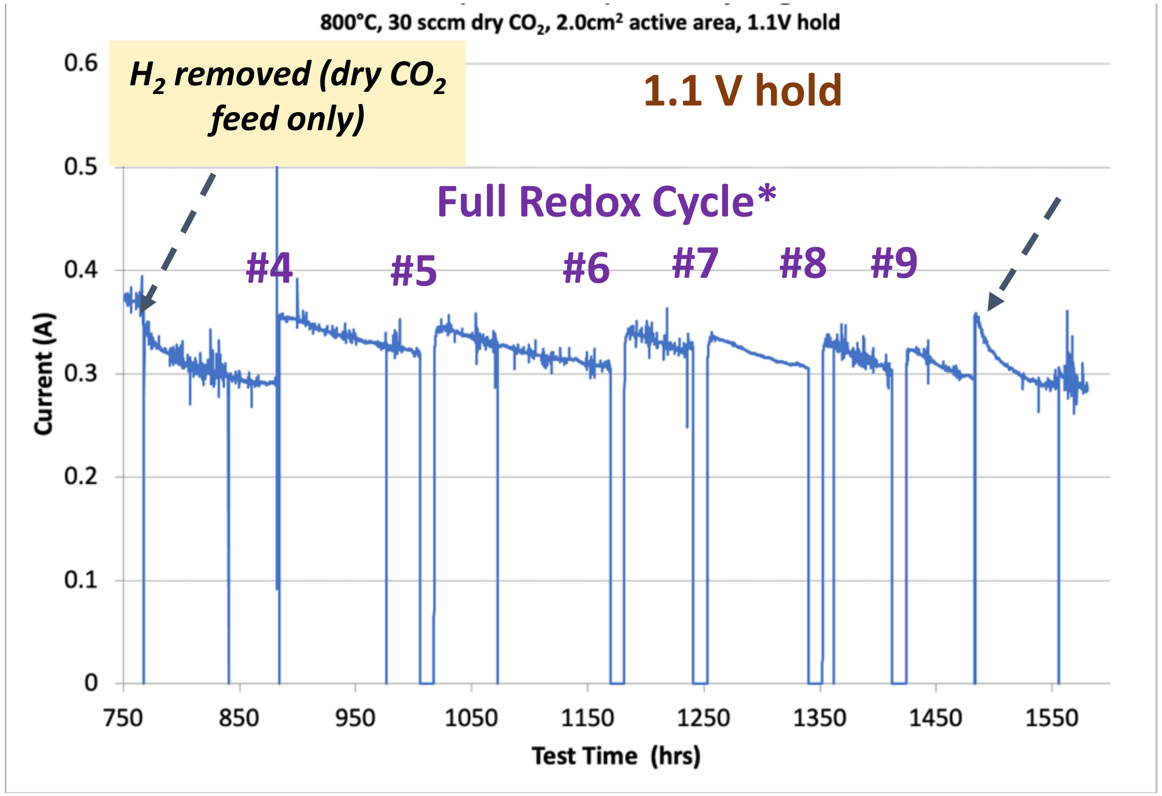
Redox Tolerance for CO_2 Electrolysis (NASA SBIR)

Ni-based electrode

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

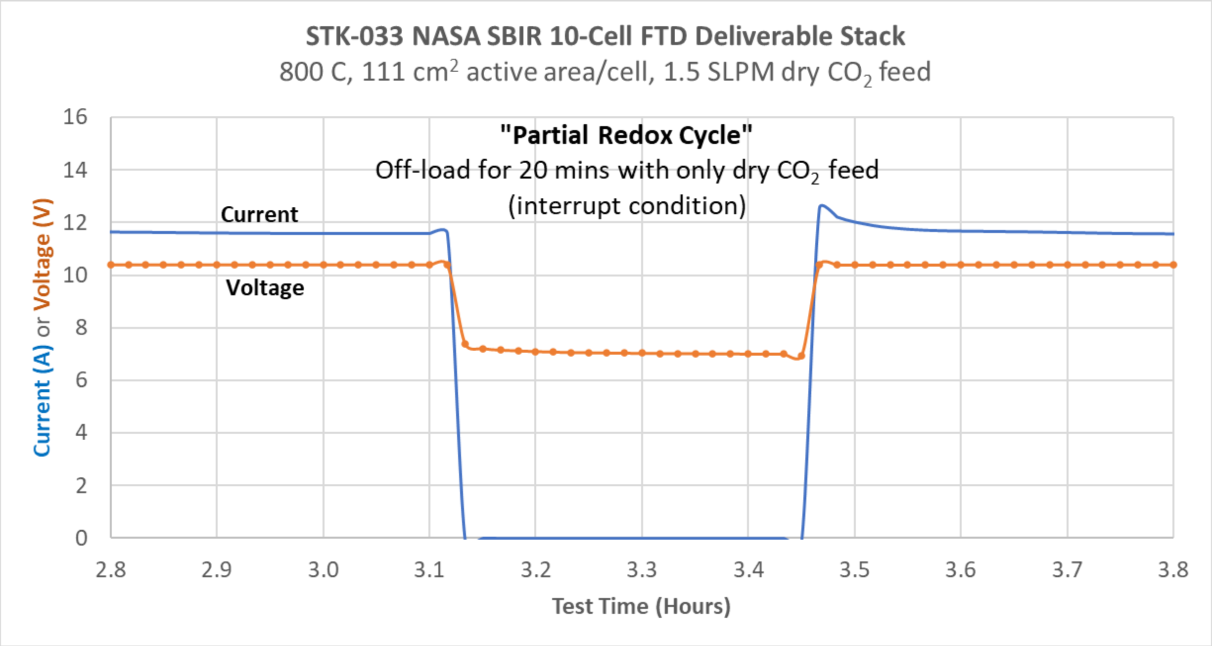


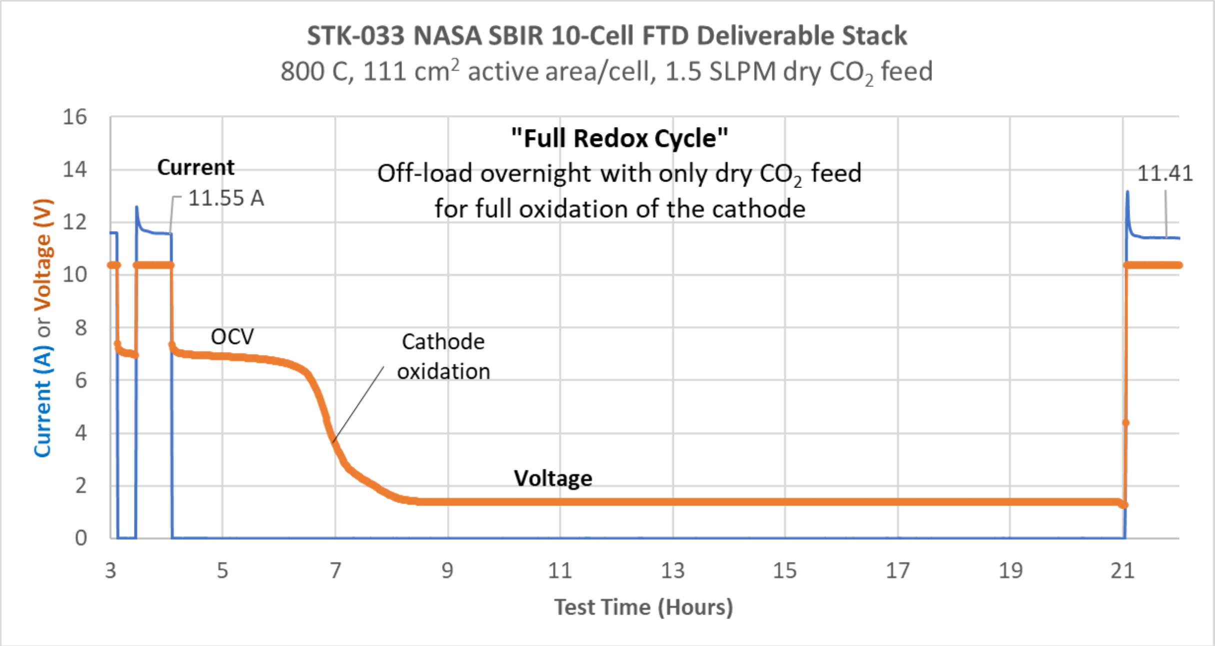
Down-selected Composition
Ni-Ceria based (N85)

- **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

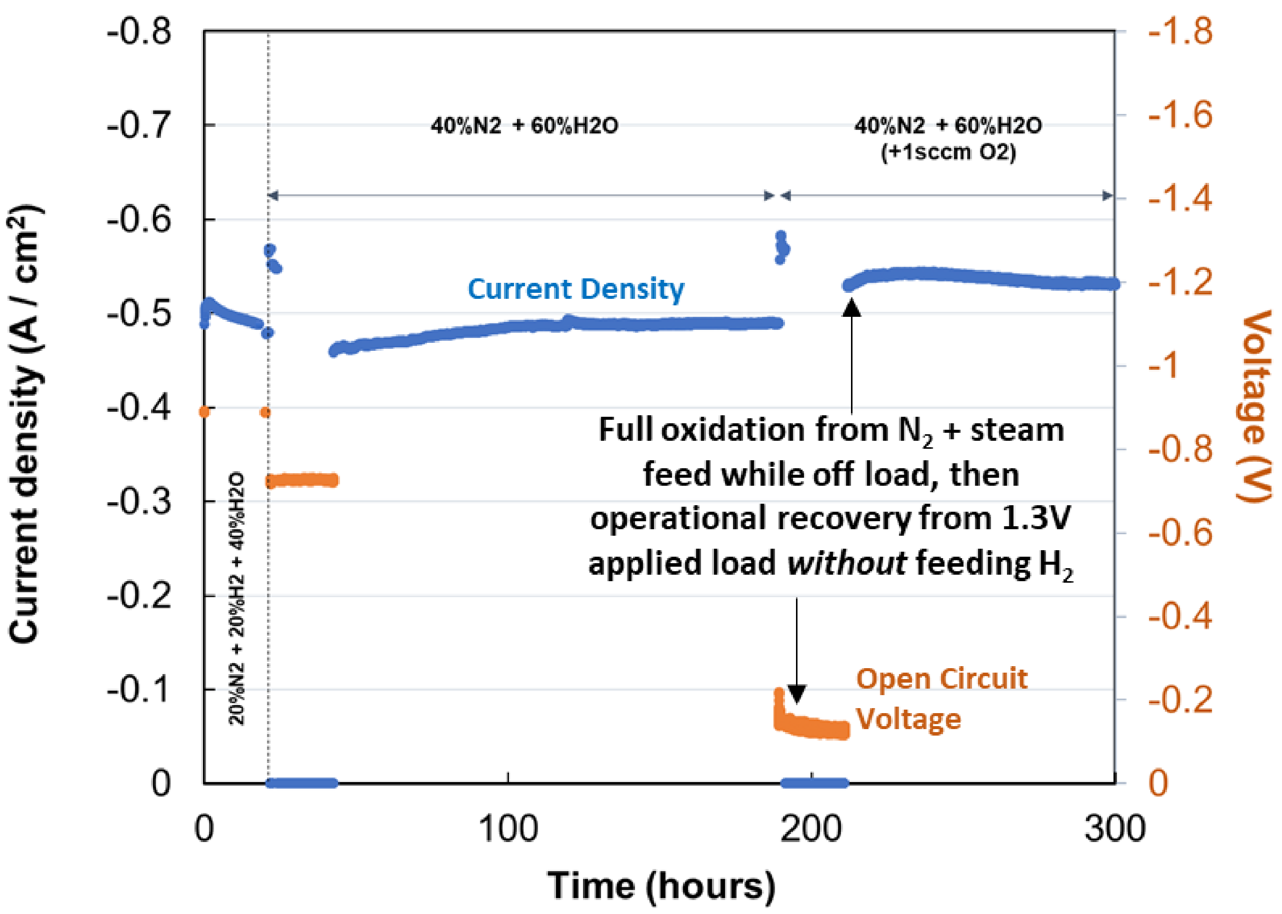




- **Stack: Short (20 min) and long (12 hrs) exposure to CO_2**
- **Application of voltage – full recovery of performance**

NASA SBIR Funded

Redox Cycle in Steam Electrolysis



Cell test at PNNL using Redox Tolerant fuel electrode:

- **Steam oxidation overnight Application of 1.3 V**
- **No external H_2 feed**
- **Full recovery of performance**

This Project

kW class stack: Redox Cycle in Steam Electrolysis

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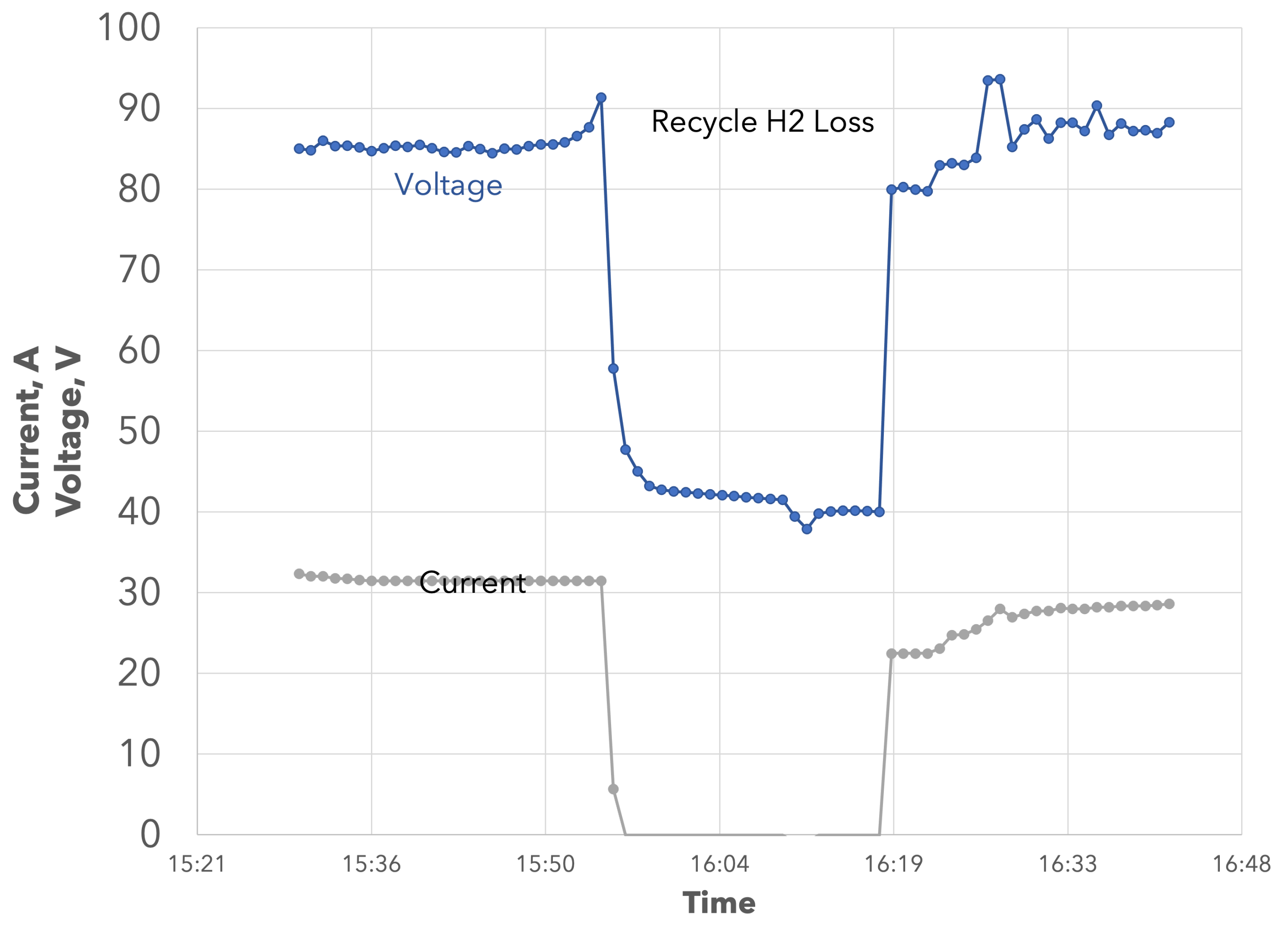

Stack test at Colorado School of Mines:

- **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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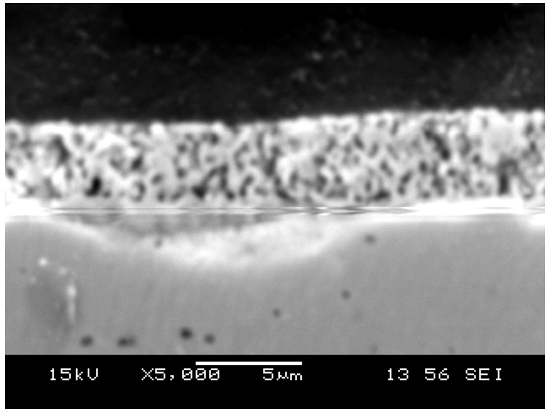
No.	Degradation mechanism	Effect	SOEC component	Activity	Project Support
1	Cr transport from interconnect	Poisons active electrochemically sites	O_2 electrode	Poisoning Effect (PNNL) Spinel Coating	**DOE/NETL** NASA Phase II-E
2	Perovskite composition instability over time	Catalytically inactive and 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

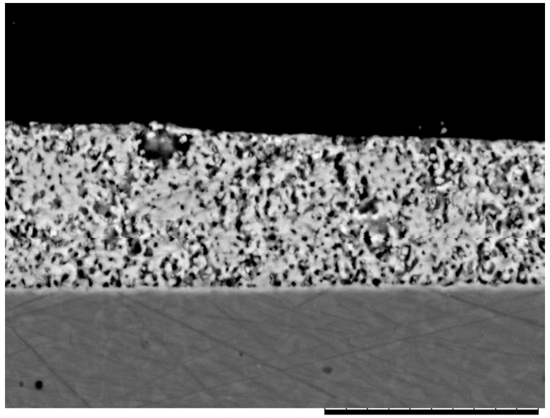
SDC Barrier layer

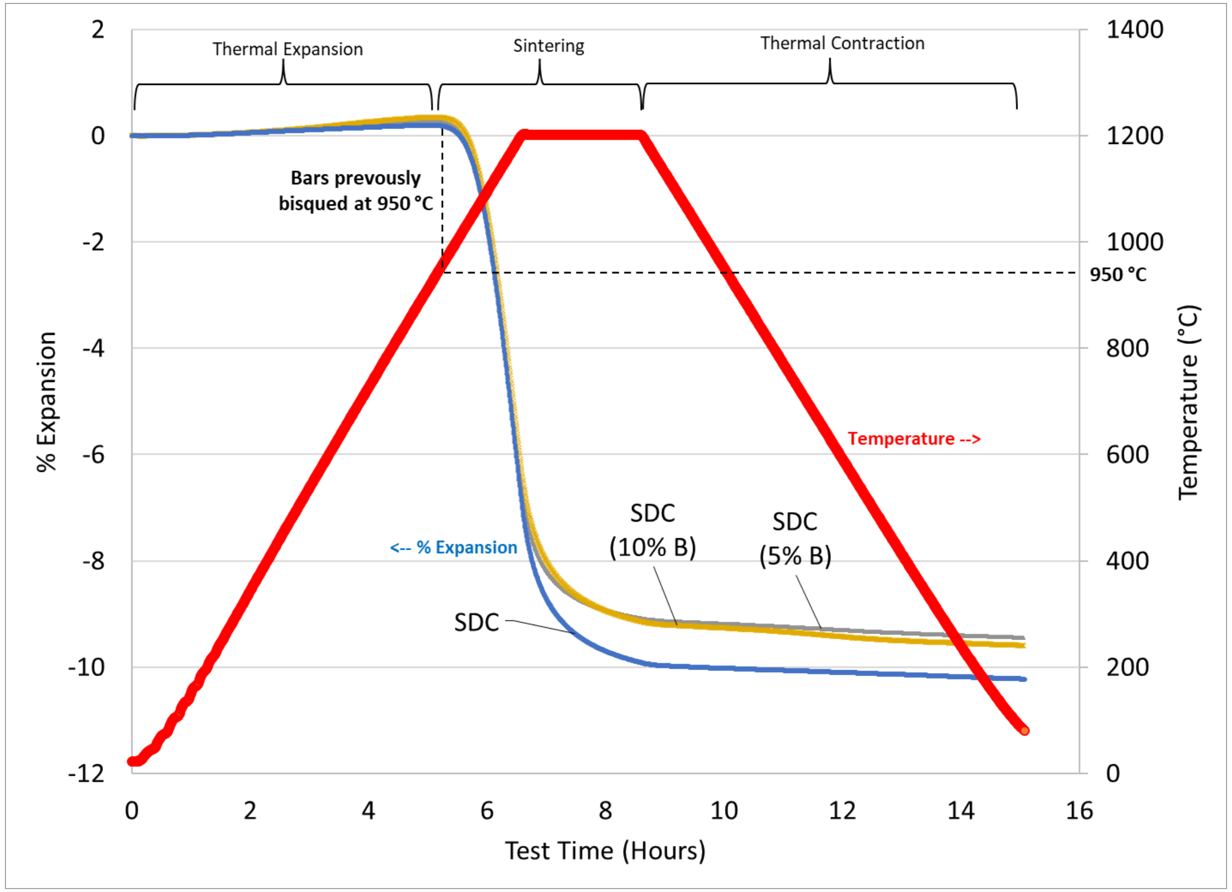
- Improve sinterability to lower sintering temperature
- Eliminate interface reaction between ceria and zirconia
- Additional modification underway

Standard SDC



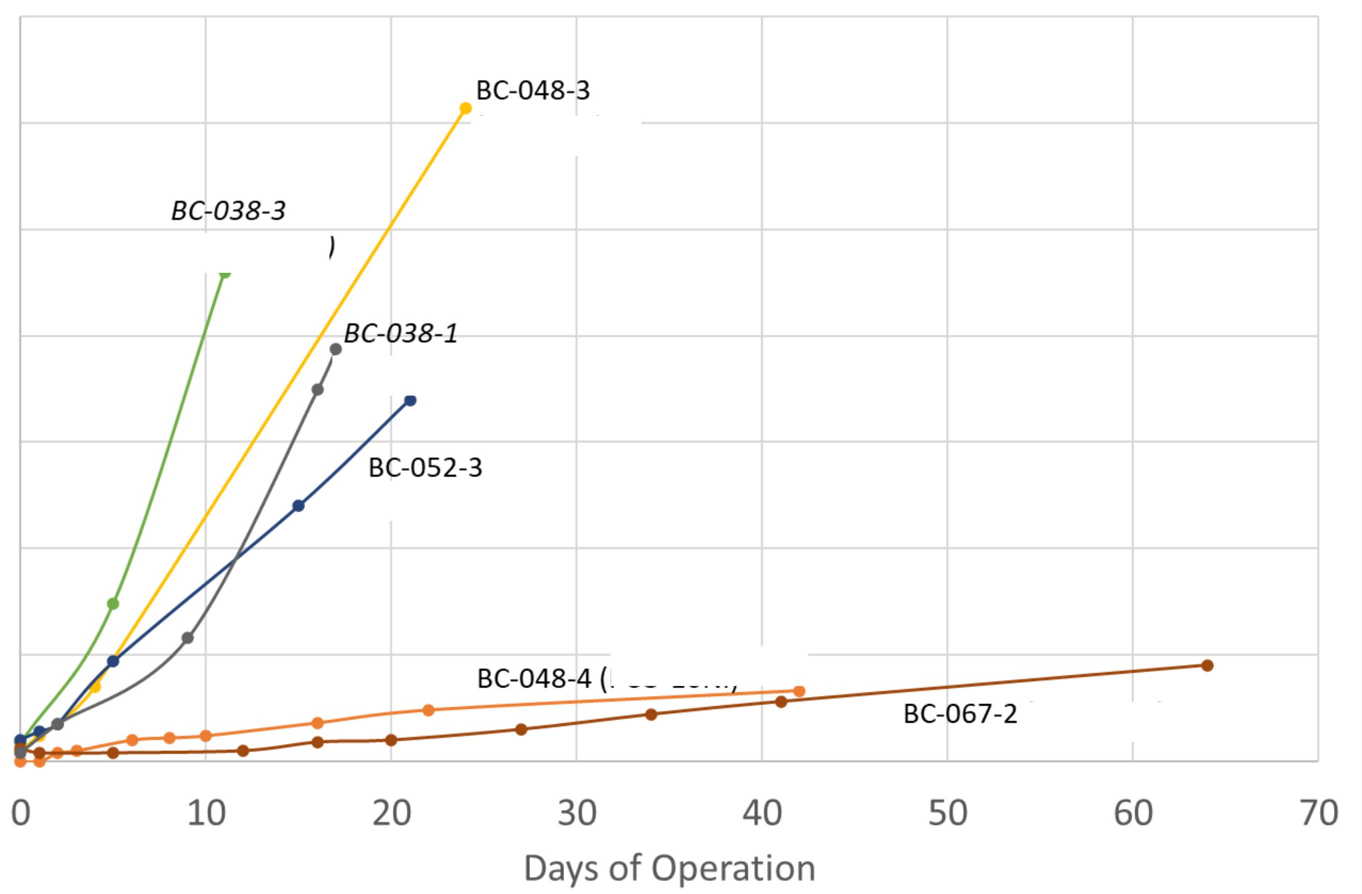
>150 °C Reduction





This Project + NASA SBIR

Fuel Electrode Modification



Fuel Electrode Polarization

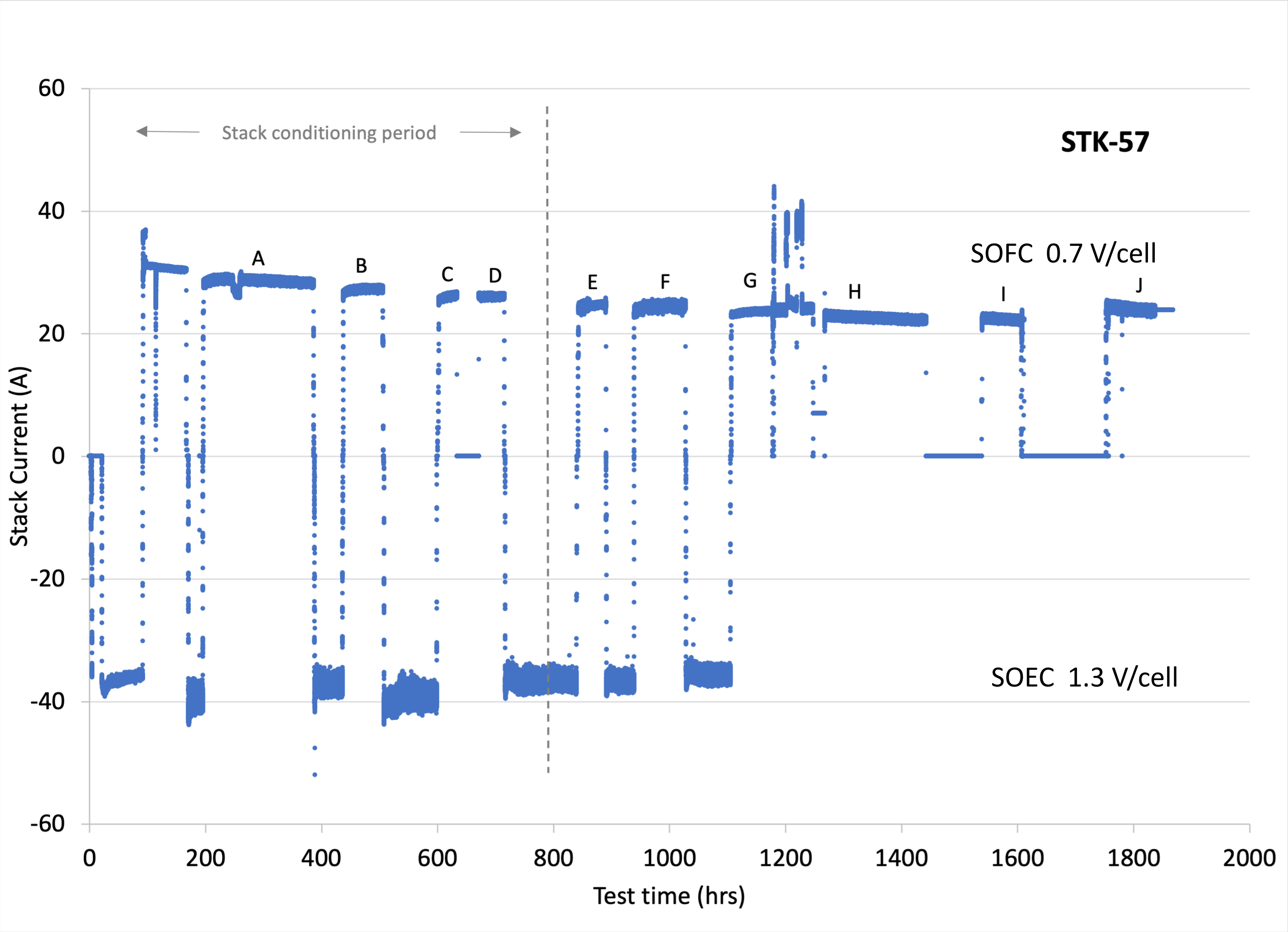
INL 30 kW DOE Project

- **Post-sintering surface treatment by infiltration**
  - Fuel electrode polarization varies with type of treatment

Work done at Yildiz Group (MIT)

Oxygen electrode surface modification by infiltration (MIT, OxEon - Non-Federal project)

Stack Test



Interim Validation: 10 cell stack

Incorporating Barrier Layer/ Electrode Modification

Non-Federal Support

- **Low degradation after ~ 800 hrs**
- **<1% / 1000 hr**

Button Cell Operation at Elevated Pressure

- PNNL
  - Test fixture construction is complete.
  - SOP was sent out for review.
  - Fixture validation to begin after SOP is approved.

- Elevated pressure BC tests will start with PNNL house-made cells after SOP approval and system verification.
- OxEon Cell tests to follow

Oxygen Production (Seal Validation)

- **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

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

NASA Support

Effect of Impurities on Oxygen Electrode Performance

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The effect of Cr impurities on the oxygen electrode in SOEC mode will be investigated.

- OxEon delivered cells to PNNL for testing
  - Two compositions of air electrode

- PNNL starting tests in presence of Cr
  - 1 cell of each set without Cr
  - 1 cell of each set with Cr

Cr barrier layer

- **Process development for improving spinel density**
  - Ongoing

Summary

- **Multiple projects to provide complementary modification**
- **Redox tolerance validated for steam electrolysis**
  - Oxidized Ni electrode recovery without the need for hydrogen in inlet
- **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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NASA NextSTEP and Tipping Point
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