

# Multi-Scale Ordered Cell Structure for Cost Effective Production of Hydrogen by High Temperature Water Splitting (HTWS)

Project ID: PD144

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# Overview

## Timeline

- Project Start Date:** October 1, 2016
- Project End Date:** September 20, 2019
- Percent Completed:** 5%

## Budget

- Total Project Budget:** \$ 1,875,101
  - Total Recipient Share:** \$ 375,130
  - Total Federal Share:** \$ 1,499,971
  - Total DOE Funds Spent\*:** \$ 151,840

\* As of 3/31/17

## Barriers

- F. Capital Cost
- G. System Efficiency and Electricity Cost
- I. Grid Electricity Emissions
- J. Renewable Electricity Generation Integration

## Partners

- PARC:** Electrode Feature Mfg Dev
- GAIA:** Technoeconomic and Lifecycle Analyses

# Relevance to DOE Goals

- **Objectives:** Develop and test advanced HTWS stacks to demonstrate the potential pathway to
  1. Meet the FCTO **cost goals for hydrogen production** <\$2/kg, excluding delivery, compression, storage, and dispensing, and
  2. Ability to operate on intermittent renewable energy sources for **energy storage** and grid ancillary services applications
- **DOE Mission Relevance**
  - Reduce petroleum use
  - Reduce greenhouse gas and air pollution emissions
  - Reduce market barriers to commercialization

# Relevance to Technical Barriers

- **Cost Goal**

- Improve stack performance
- Operate at lower voltage
- Increase lifetime
- Lower capital cost
- Reduce electric input
- Lower levelized H<sub>2</sub> cost

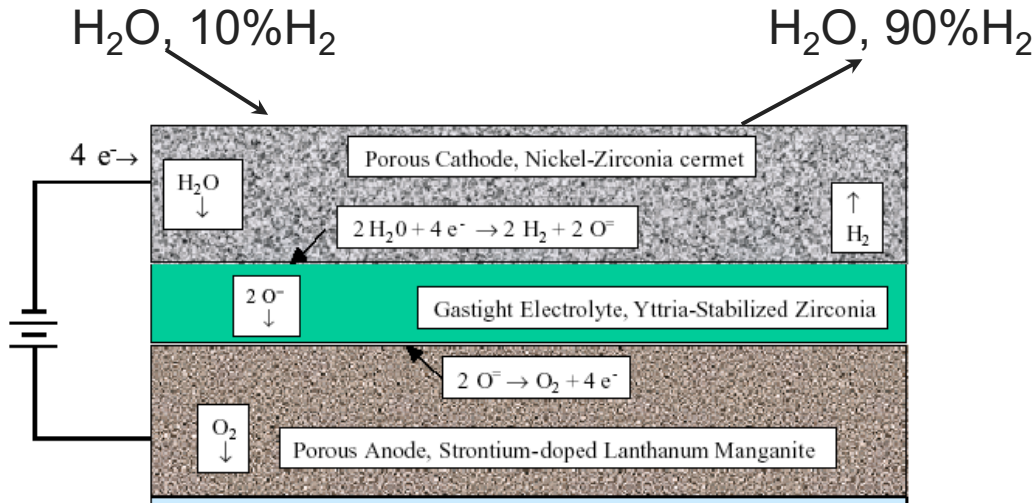
- **Integration with Renewable Energy**

- Reduce gas diffusion resistance
- Mechanical robustness
- Response to transient load
- Faster heat up/cool down
- Thermal cycle capability

- **Pressurized H<sub>2</sub> Delivery**

- Robust seal
- Operation at pressure
- Thermal cycle

# High Temperature Water Splitting



- Conventional Design
  - Electrolyte or Cermet as support
  - Combination of tape casting and screen printing

# Approach - Technical Challenges

## Performance Limitations

- Electrical resistance of cell
- Electrode non-ohmic resistance
- Heat up/shut down, load follow
- Hydrogen purity
- Hydrogen delivery at pressure
  
- Lifetime

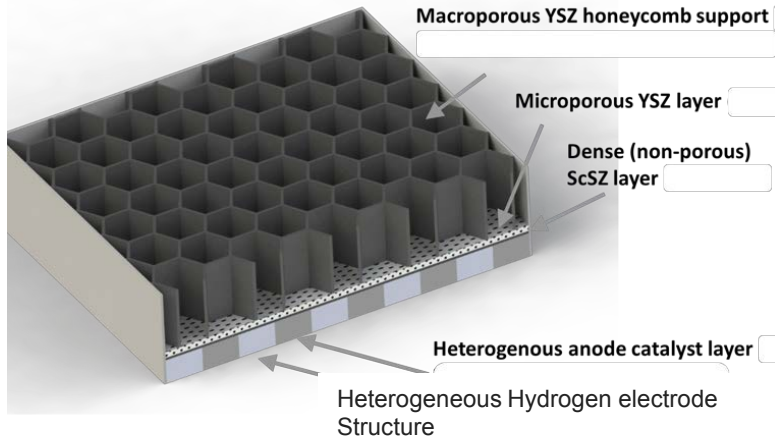
## Limiting Factors

- Layer thicknesses
- Electrode catalytic activity
- Gas diffusion resistance
- Mechanical robustness
- Inadequate seal
- Seal characteristics
  
- Electrode chemical instability  
(Oxygen electrode delam)
- Electrode physical Integrity  
(H<sub>2</sub> electrode coarsening)

# Approach – Technical Concept

- Performance Improvement
  - Cell Design: Multi-scale Ordered Cell Structure
    - **Thin, high conductivity electrolyte** (low electrolyte resistance)
    - Macro-scale features as **mechanical support on air electrode**
      - Eliminate steam diffusion limitation of fuel support
    - Micro-scale **ordered hydrogen electrode**
      - Improve electrode performance by increasing density of reaction sites for electrochemical reduction of water
- Lifetime Improvement
  - Thermochemically **stable electrode compositions**
    - Oxygen electrode – stability of stoichiometry
    - Hydrogen electrode – high steam stability

# Approach – Performance Improvement



Not to scale

- Higher conductivity electrolyte (Scandia doped)
- Mechanical support on oxygen evolution side
- Macroporosity reduces gas diffusion resistance
- Structured hydrogen electrode
- Control of composition and reaction site density
- Thinner electrode eliminates  $\text{H}_2\text{O}/\text{H}_2$  diffusion resistance



# Approach – Technical

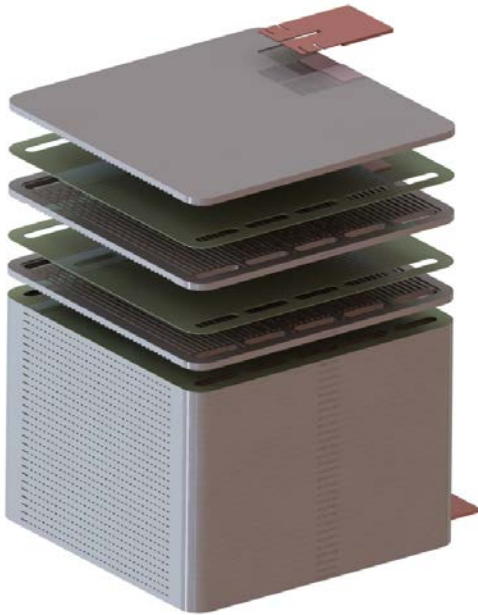
## • Cell fabrication development

- Thin, high conductivity electrolyte      Tape casting, lamination (Ceramatec)
- Ordered electrode structure              3D Printing (PARC)
- Mechanical Support                         3D Printing (PARC)
- Seal    Glass (Ceramatec)

## • Materials Selection

- Stable dopant in oxygen electrode to eliminate delamination and cation mobility
- Glass seal to limit gas cross over for hydrogen purity and pressurized operation
- Inert oxide dispersion to make hydrogen electrode (nickel) coarsening resistant in high steam conditions

# Approach – Stack Fabrication



- Interconnects and cell joining layers from ongoing NASA CO<sub>2</sub> electrolysis project
- Typical ASR ~ 0.8 – 1.0 ohm-cm<sup>2</sup> at 800°C  
(0.6 – 0.7 ohm-cm<sup>2</sup> when corrected for activation polarization and CO<sub>2</sub> conversion)

Notional Stack Design

# Approach – Milestones & Go/No-Go

## Major Technical Tasks

- Baseline materials set to identify largest contributors to cell resistance
- Develop process for novel cell structure that addresses resistance contributors
- Button cell testing of new cell structure
- Production of full-scale cells
- Stack production development with new cells
- Stack testing to prove targets of 1 kg H<sub>2</sub>/day production
- Modeling of GHG emissions vs. SMR
- Levelized cost model for target of <\$2/kg H<sub>2</sub>

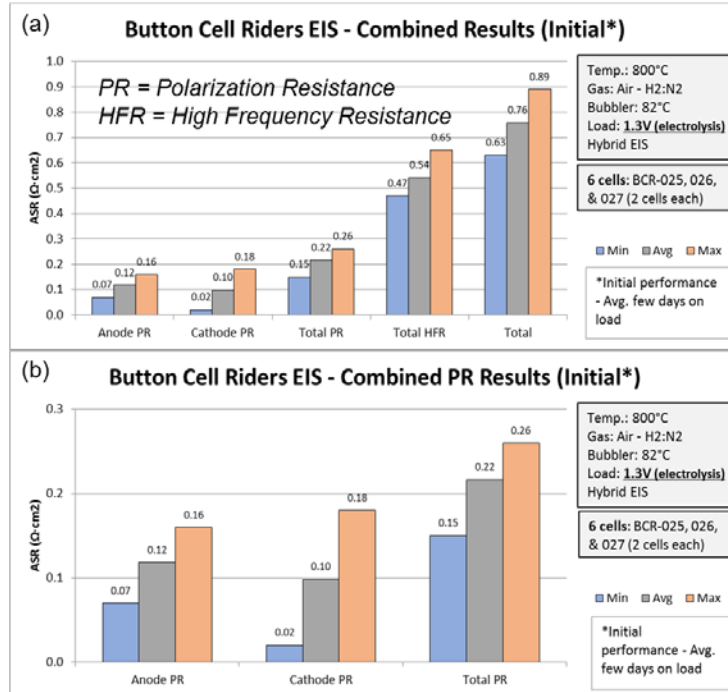
# Approach – Milestones & Go/No-Go

Milestone/Go-NoGo Description	Anticipated Completion Date	% Complete
M1.0.1: Finalized Cell Design, Fabrication Process	12/17	100
M2.2.1: Cathode Ink Formulated	3/17	90
M2.4.1: Anode Ink Formulated	3/17	90
M3.2.1: Micro-patterned Cathode Films Fabrication	6/17	10
M3.3.1: Support Structure Fabricated	9/17	
<b>Go/NG: Button Cell Structure Tested <math>\leq 0.4 \text{ ohm-cm}^2</math></b>	<b>9/17</b>	
M5.0.1: Button Cell ASR measured	9/17	
M6.2.1: Patterning Developed for Support	3/18	
M6.4.1: Cell Fabrication	6/18	
M7.0.1: Seal Demonstrated	9/17	
M8.0.1: Verification Short Stacks	6/18	
<b>G/NG: Short Stack ASR of <math>0.4 \text{ ohm-cm}^2</math></b>	<b>9/18</b>	
M9.0.2: Stack capable of 1 kg H <sub>2</sub> /day	9/19	
M10.0.1, 2, 3: GHG Emissions of HTWS vs SMR Comparison	9/17,9/18,9/19	
M10.2.1, 2, 3: Levelized production cost $< \$2/\text{kg H}_2$	9/17,9/18,9/19	

2 quarters of effort completed

# ACCOMPLISHMENTS – Baseline Testing

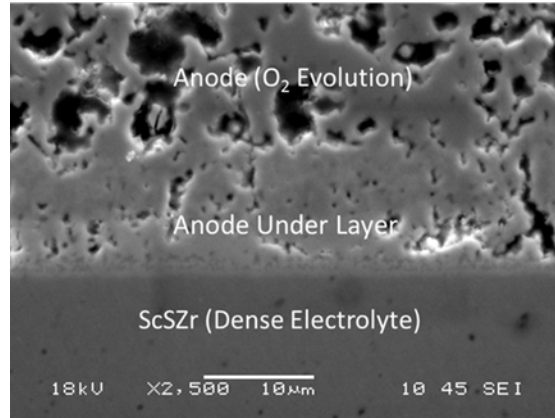
- Button cell testing of baseline components
  - Target to reduce Electrode Polarization resistance to 0.1-0.2 ohm-cm<sup>2</sup> range
  - Baseline results include thick electrolyte
  - ASR is the sum of HFR (ohmic) and PR (electrode)



Baseline electrodes average polarization resistance  
~ 0.2 - 0.25 ohm-cm<sup>2</sup>

Further reduction possible with micro-featuring

# Oxygen Electrode Symmetric Cell



- Prior typical current density 0.3 to 0.4 A/cm<sup>2</sup>
- High Current Density (1 A/cm<sup>2</sup>) operation shows no delamination after 300 hours

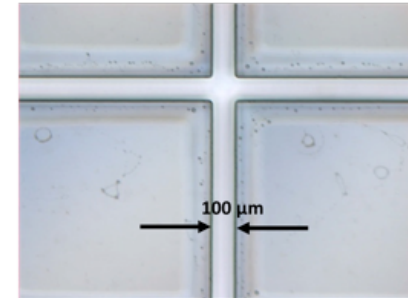
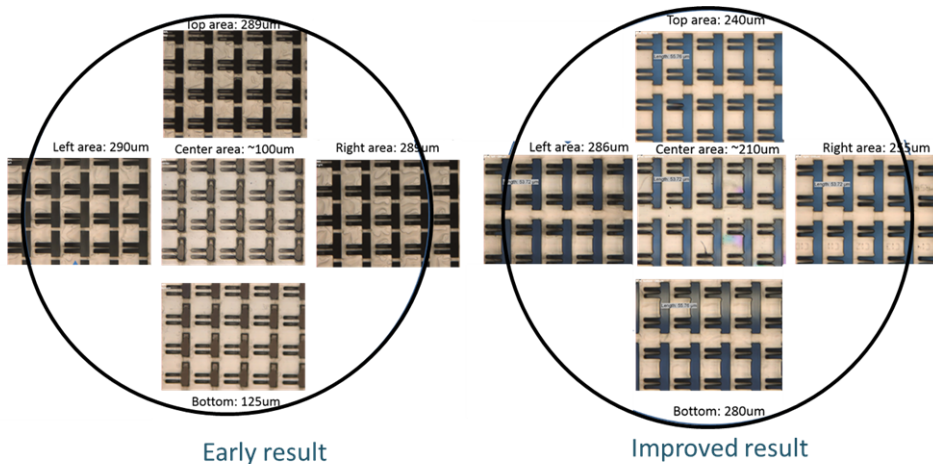
# Accomplishments

## Tape Casting Trials; Print Ink Formulation

- Thin tape casting trials (Ceramatec)
  - Tape formulation evaluation for compatibility
    - With featured electrode
    - Support structure
    - Sintering multi-layer structure
- Electrode/Support Structure Inks (PARC)
  - Ceramic powder (Ceramatec)
  - Ink viscosity, solvent boiling point, rheology control
  - Formulation developed

# Accomplishment – Patterning Ink Formulation for Oxygen Electrode Support

- Advanced Printing Techniques for Cell Design
  - Use of existing tooling for process development

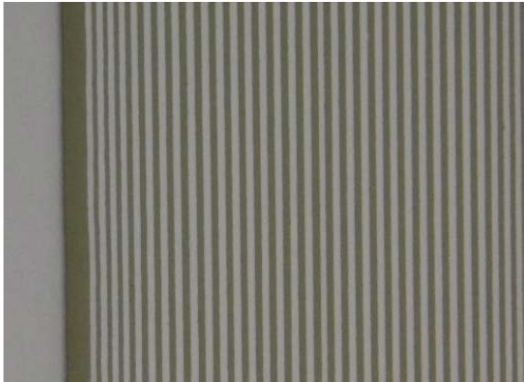


Wet layer

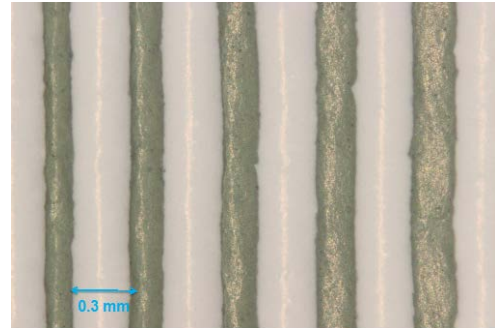
- Thickness variation reduced from  $\pm 90 \mu\text{m}$  to  $\pm 40 \mu\text{m}$  by controlling baking process
- New mask design in progress



# Accomplishment – Micropatterning Hydrogen Electrode



- As printed hydrogen electrode
- Awaiting sintering trials



Magnified Image

Control of line thickness, pitch and composition possible

# COLLABORATIONS

- **Prime: Ceramatec**
  - Ionic Ceramic device focused industry
  - Division of CoorsTek
- **Sub: PARC**
  - Development and commercialization of Printing technology
  - Division of Xerox Corporation
- **Sub: GAIA**
  - Technoeconomic and life cycle analysis models
  - Economically disadvantaged women owned small business (EDWOSB)

# REMAINING CHALLENGES/BARRIERS

- Fabrication of new cell design
  - Lamination/Printing Compatibility
  - Sintering of multi-layer cell structure
- Button cell Area Specific Resistance  $<0.4 \text{ ohm-cm}^2$
- Stack assembly
  - Joining materials compatibility with new cell structure
  - Cell flatness for stacking
  - Stack ASR  $0.4 \text{ ohm-cm}^2$
  - Seal integrity for pressurized operation
- 1 kg/day production
- Manufacturing cost verification to support  $<\$2/\text{kg H}_2$

# PROPOSED AND FUTURE WORK

- Remainder of FY17:
  - Produce structured button cells for testing
    - Demonstrate  $<0.4$  ohm-cm<sup>2</sup> performance
  - Begin scale up to full cell size
  - Demonstrate glass seal capability
- FY18 Tasks
  - Short stack testing
    - Demonstrate 0.4 ohm-cm<sup>2</sup> performance
- FY19 Task
  - 1 kg H<sub>2</sub>/day stack test
- LCA & TEA with increasing fidelity

# Technology Transfer Activities

- Internal planning for manufacturing development in progress
- Integration and optimization of combining Ceramatec and PARC processes

# SUMMARY

- Key objective is to develop HTWS stack technology to meet
  - Performance and cost goals
- Tasks are designed to improve stack performance
  - Use of multi-scale ordered structure to address performance limiting factors
- Fabrication development well underway
  - Materials selection completed
  - Tape cast process development underway
  - Printing ink development complete
  - Printing process development underway
- Button cell testing planned for end of year 1
  - Task on schedule

**Thank you**

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# TECHNICAL BACK-UP SLIDES

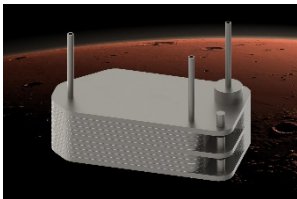
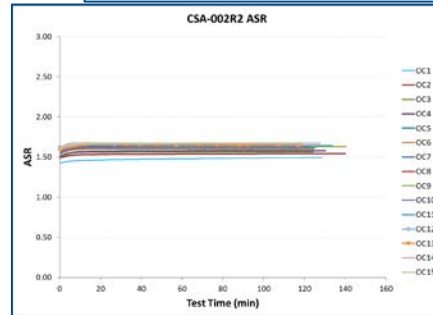
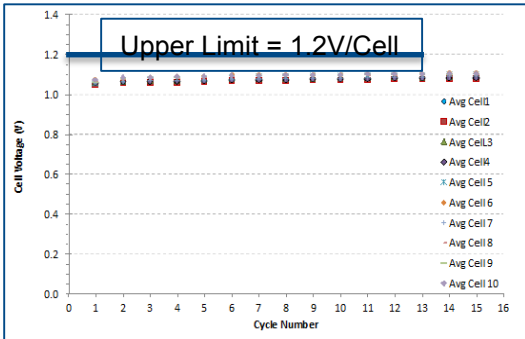
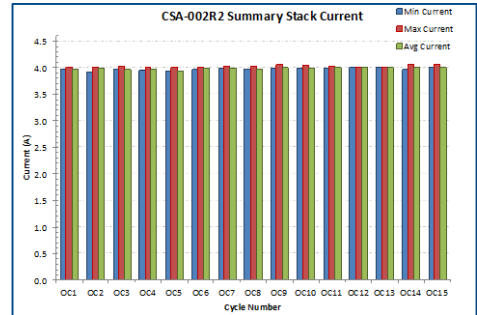


# Materials set developed for NASA Mars Project will be used

## MOXIE: Operational Cycle Testing



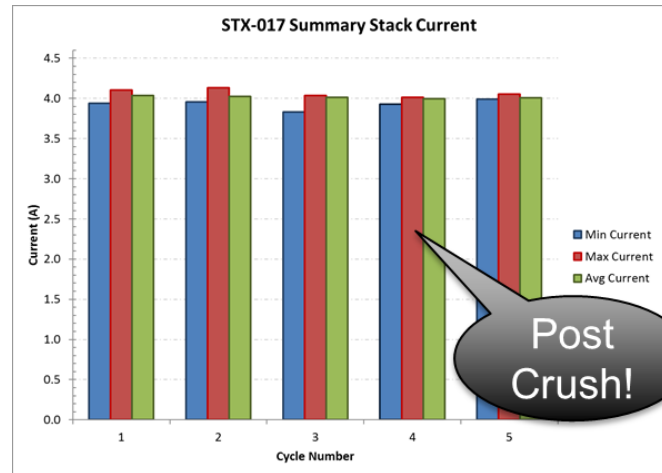
- Cycle life testing on **dry CO<sub>2</sub>**
  - 120 minute operational cycles
- No significant cycle to cycle degradation
- Excellent Cell-to-Cell Voltage Grouping



# MOXIE: Performance and Stability



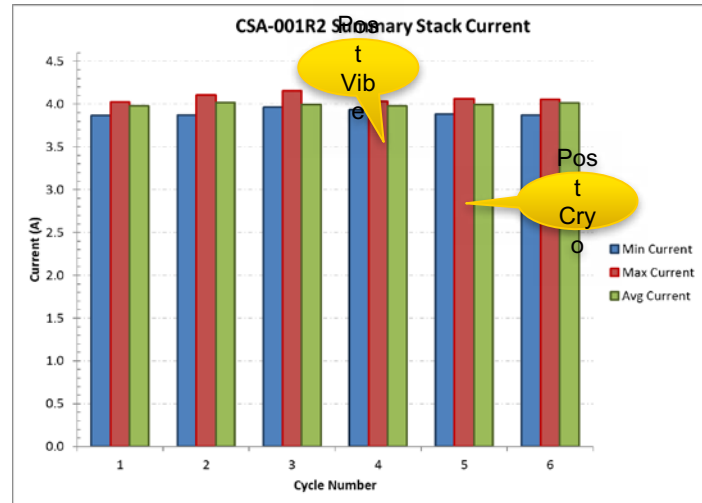
- Mechanical Requirements
  - **Structural Testing: Must withstand compressive forces required for entry, descent, landing packaging design**
    - Multiple stacks tested to 10kN compressive force and then operationally cycled with no leakage or degradation
    - No leakage at 25kN on multiple stacks, only failure at 62.7 kN



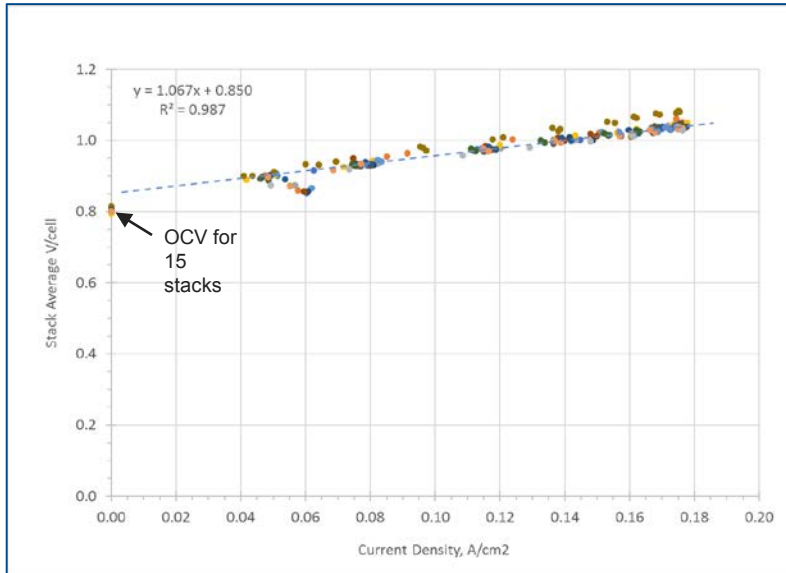
# MOXIE: Performance and Stability



- **Mechanical Requirements**
- **Shock and Vibe**
  - Maintained Performance
  - Maintained O<sub>2</sub> purity > 99.9%
- **Cryo Testing: Must withstand thermal cycles down to -65**
  - 3 cycles from ambient to -55°C
  - 40 cycles to -40°C
  - 1 cycle to -65°C



# MOXIE: Reproducibility



**20+ consecutive stacks had identical performance on Dry CO<sub>2</sub> electrolysis**

# MOXIE: Summary



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## Baseline Performance

- 15 consecutive stacks built with *aerospace quality standards and traceability* having a maximum baseline performance of 1.6 ohm-cm<sup>2</sup> dry CO<sub>2</sub> and 99.9%+ O<sub>2</sub> purity

## Cycling Performance

- 3 stacks with 21 cycles of varying cycle-to-cycle flow rates and final cycle averages of 10.11 g O<sub>2</sub>/hr production and 99.8% purity – Targets exceeded

## Structural Stability Testing

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

## Shock/Vibe Testing

- Stacks vibrated at JPL and post vibe tested at Ceramatec
- **No leak or significant performance change post vibe!**
- **No leak after shock testing, retest for performance underway**

## 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**

