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Oxide Coatings for Metallic SOFC Interconnects

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Outline

Cost Effective Interconnect Coating (IC) Process Development

- Phase I: Aerosol spray deposition (ASD) demonstrated as a commercially-viable process
- Phase II: Process Refinement and Validation

1. **Project Objectives and Conclusions**
2. **Summary of Commercialization Activities**
3. **Summary of cost modeling**
    - Continuous process improvements
4. **Performance evaluation results**
    - Oxidation kinetics
    - Long-term ASR results
    - Mechanical Characterization

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Project Objectives

Track I (Cost Modeling): Develop and production-validate cost models for ASD coating at various production volumes.

- Develop customer-specific cost curves.
- Develop Customer-preferred paths to market
- Identify and address customer specific technical hurdles.
- Identify manufacturing strategies to reduce volume manufacturing costs.

Track II (Performance Validation): Demonstrate ASD-coated ICs performance to reinforce value proposition.

- Identify test methods to simulate 40,000 hours service.
- Develop model for IC degradation.
- Based on models, identify cost and performance optimized coatings.
- Evaluate performance of ASD coated components.

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Conclusions

❖ **Refined cost and manufacturing models to encompass volumes from prototyping through full volume production.**

- Market forecast and demand curves defined for three OEM profiles at various stages of commercialization.
- Three-stage technology roadmap developed.

❖ **Identified manufacturing strategies to reduce volume manufacturing costs.**

- Materials processing scale-up to 25 kg batch sizes and beyond
- Plant designs for up to 12M/year coating

❖ **Defined key process limits for ASD coated ICs.**

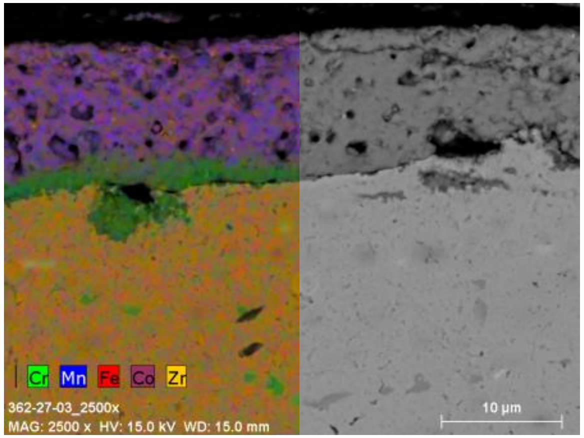
- Lifetime stability tests in progress (> 11,000 hrs operation at ≥ 800 °C in single atmosphere configurations).
- 1800 h testing in dual atmosphere conditions

❖ **Identified key failure mechanisms and acceleration factors.**

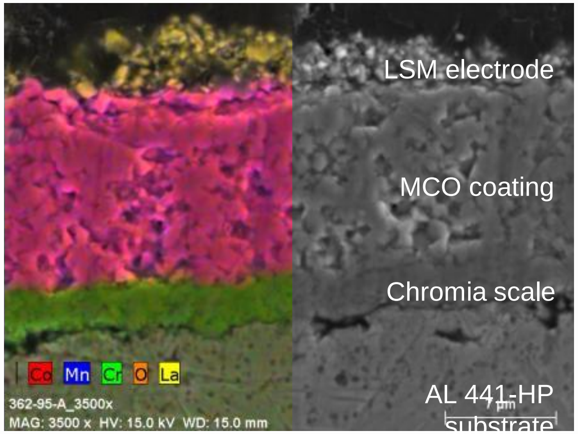
- Predictive lifetime models successfully applied to long-term stability tests.

Overlay Protective Coatings

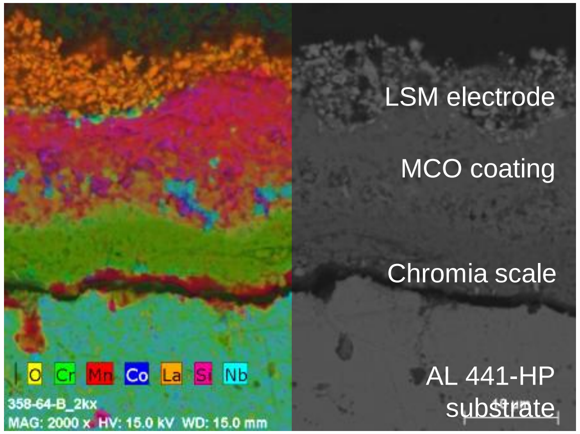
After Deposition



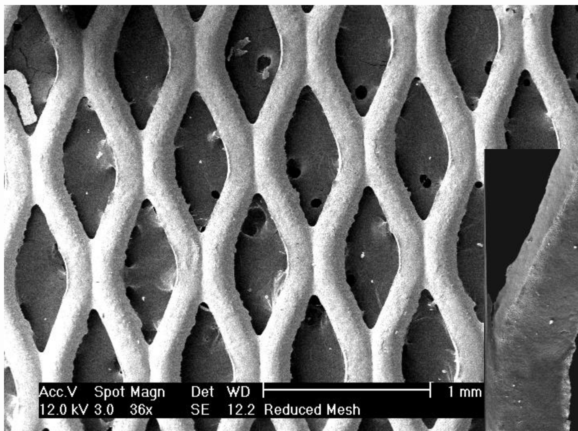
800 hrs 800 °C

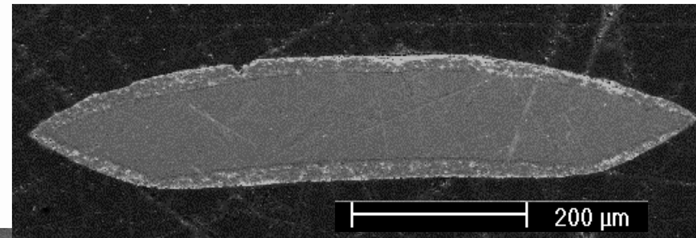


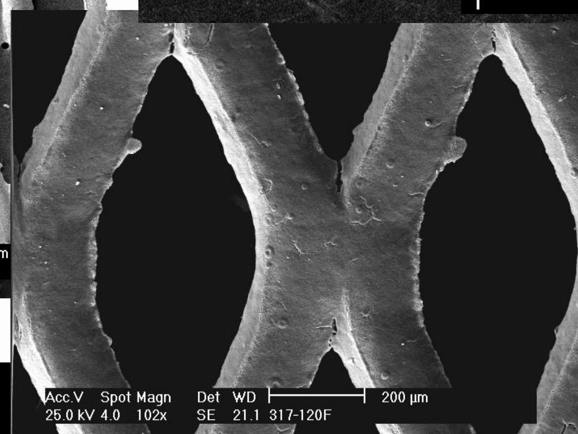
> 7000 hrs 800 °C/900 °C

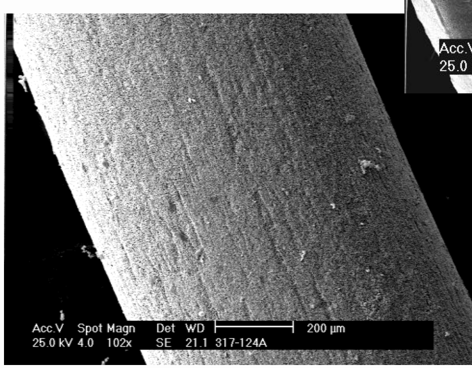


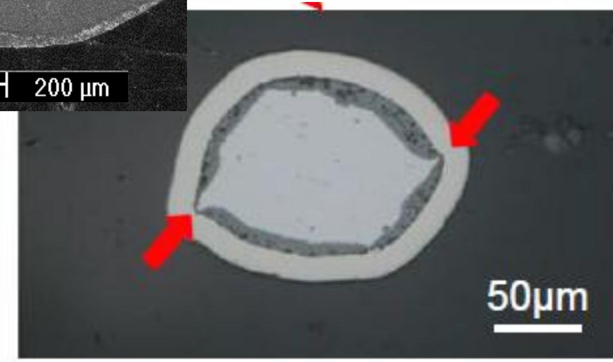
Overlay Coated Meshes

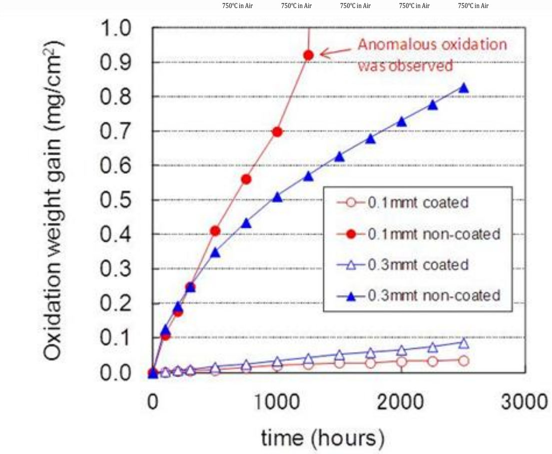












Flexibility of ASD process enables wide range of components to be coated.

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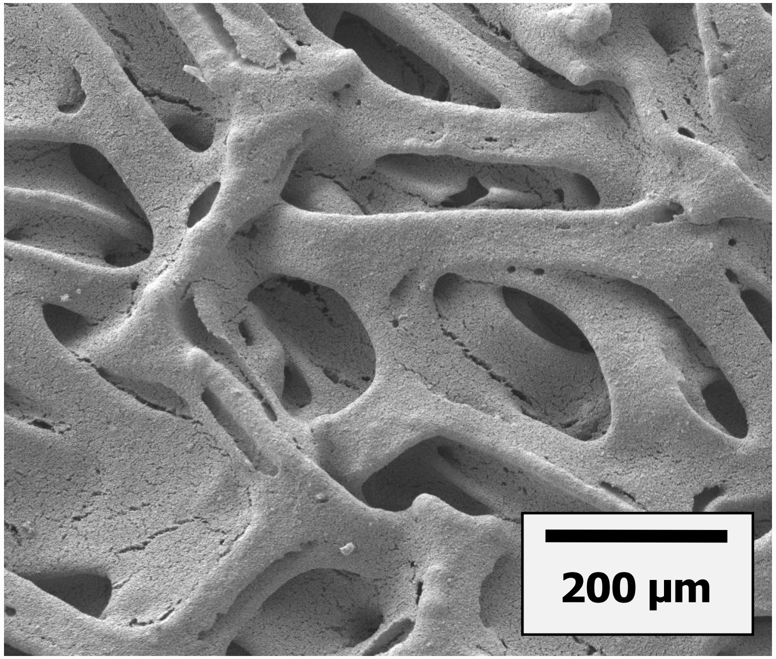
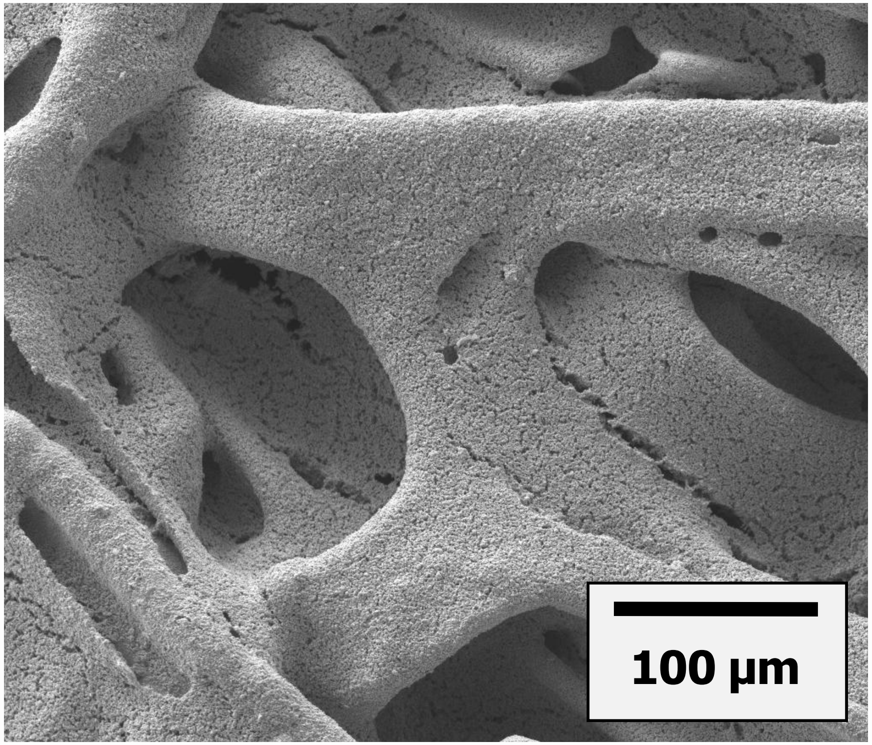
Catalyst Coating on Metal Foams

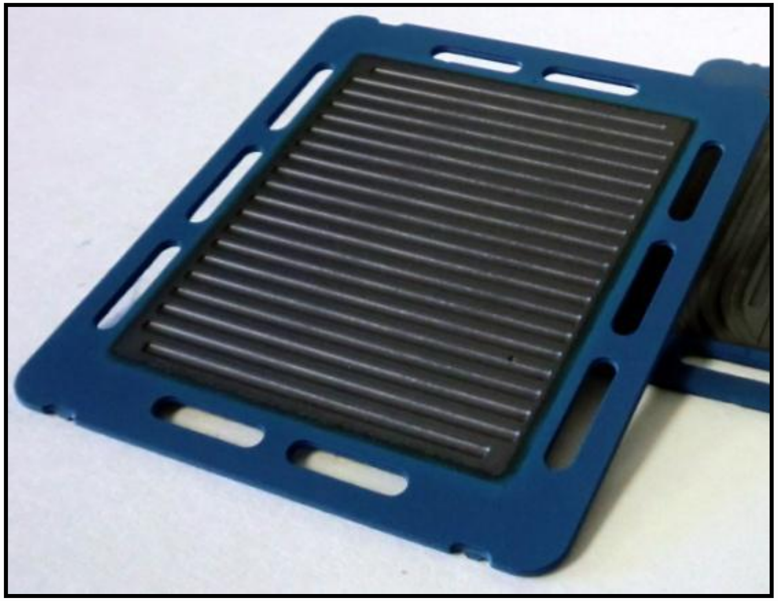




Non-Active Area IC Coatings

- ❑ NexTech has developed two complementary coating approaches for non-active, seal protection coating.
- ❑ Masking allows for multiple coatings to be applied to each side of component.

Insulating Overlay Coating



Aluminide Diffusion Coating

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Potential for BoP applications

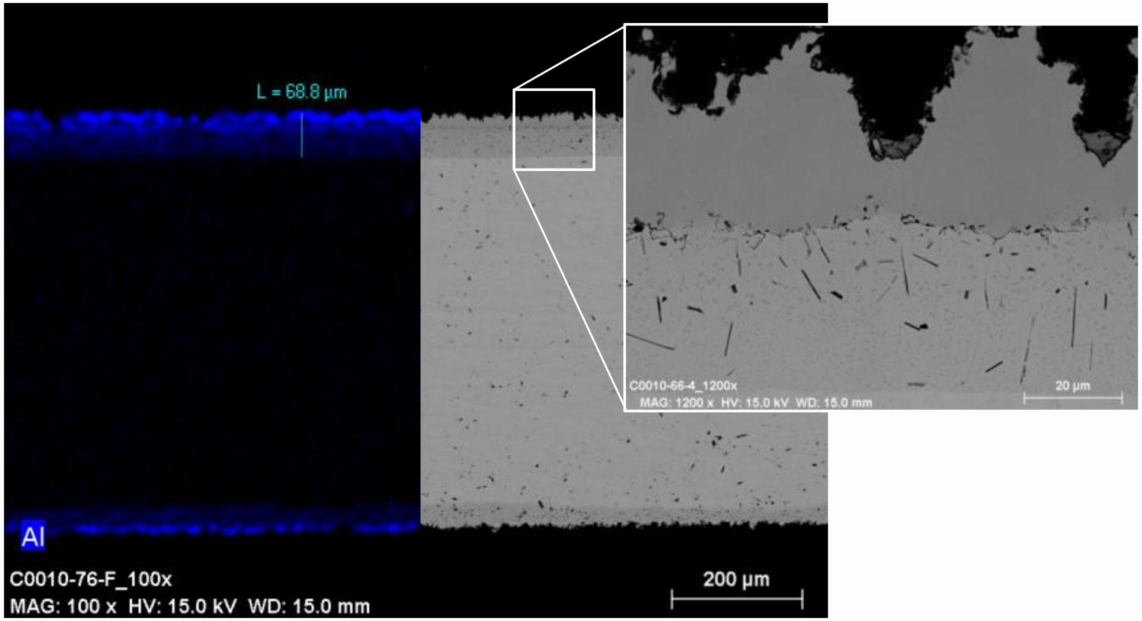
❑ In addition to IC sealing area aluminization process is of interest for high-temperature BoP corrosion protection applications

Application	Coating	Value Proposition	Requirements
SOFC metallic interconnects		Enabling dual-coating technology for total interconnect solution	• Process compatible with MCO heat treatments. • Compatibility with MCO coating • Reduced interactions with seal materials.
High temperature corrosion protection		Cost reduction versus existing aluminization processes	• High temperature corrosion protection •Manufacturability (forming, welding, brazing components)

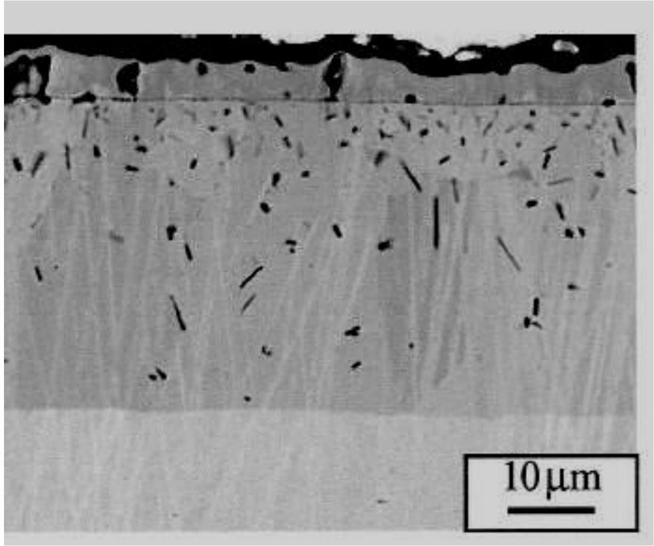
Substrate compatibility: Stainless Steels

- ❑ Demonstrated process compatibility with both austenitic and ferritic stainless steels

Cross-section SEM and Al compositional EDS map for NexTech aluminide coating on Grade 304 stainless steel



Cross-section SEM of aluminide coating produced by CVD on Grade 304



B. A. Pint et al., Evaluation of Iron-Aluminide CVD Coatings for High Temperature Corrosion Protection, Materials at High Temperature 18(3) (2001) 1.

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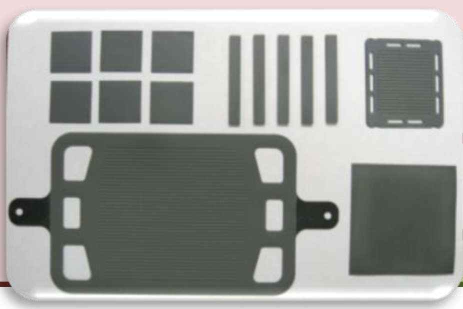
Manufacturing Strategies

Prototyping Project



Contract Coating Services

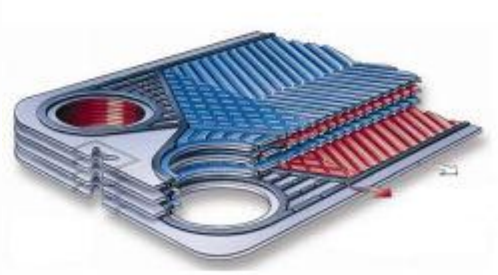
Manufacturing Implementation

Materials Manufacturing, Process Optimization and Materials Supply:

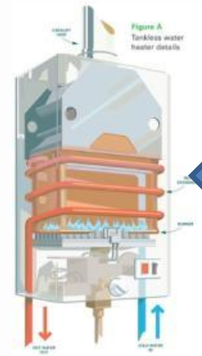
- NexTech is scaling production of materials to tonnage scale
- Process development to reduce materials cost, enhance usability of value-added products
- Materials provided by NexTech include licenses to applicable intellectual property.

Markets for Protective Coatings

High Temperature Heat Exchange



Appliances

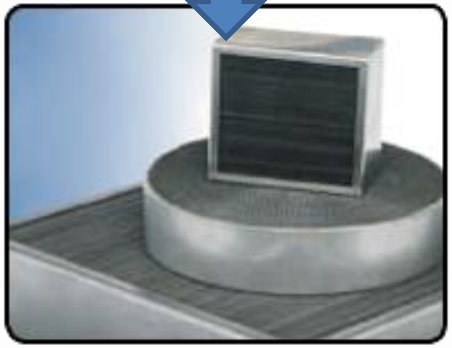


Solid Oxide Fuel Cells

Chemical/Refining

Automotive

Catalyst Reactors



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MCO Coating Development

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Cost Analysis for ASD process

- Significant refinement of the ASD coating cost model developed in Phase I has been performed.
  - Improve accuracy
  - Increase flexibility to accommodate a wide-range of production volumes
  - Model designed for both pilot-production through high volume manufacturing (HVM)

Model Attribute	Model property
Volume Production Range	1,000-10M parts/year
Single/doubled sided coated components	Both single and doubled sided components
Multiple coatings (masking)	Four different coating areas and three different coatings can be incorporated
Reduction heat treatment furnace	Batch furnaces Continuous: Belt and Pusher furnaces
Heat treatment selection	Optional oxidation firing

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Identification of Process Equipment

- NexTech has met with a wide variety of vendors in order to understand and account for the appropriate equipment needed for scale up of the ASD coating process
- Budgetary quotations for the equipment along with equipment utility usage rates fed-back into the cost model

Integrated Spray/Dry System

Controlled Atmosphere Electric Pusher Kiln

Automated QA/QC Inspection Equipment

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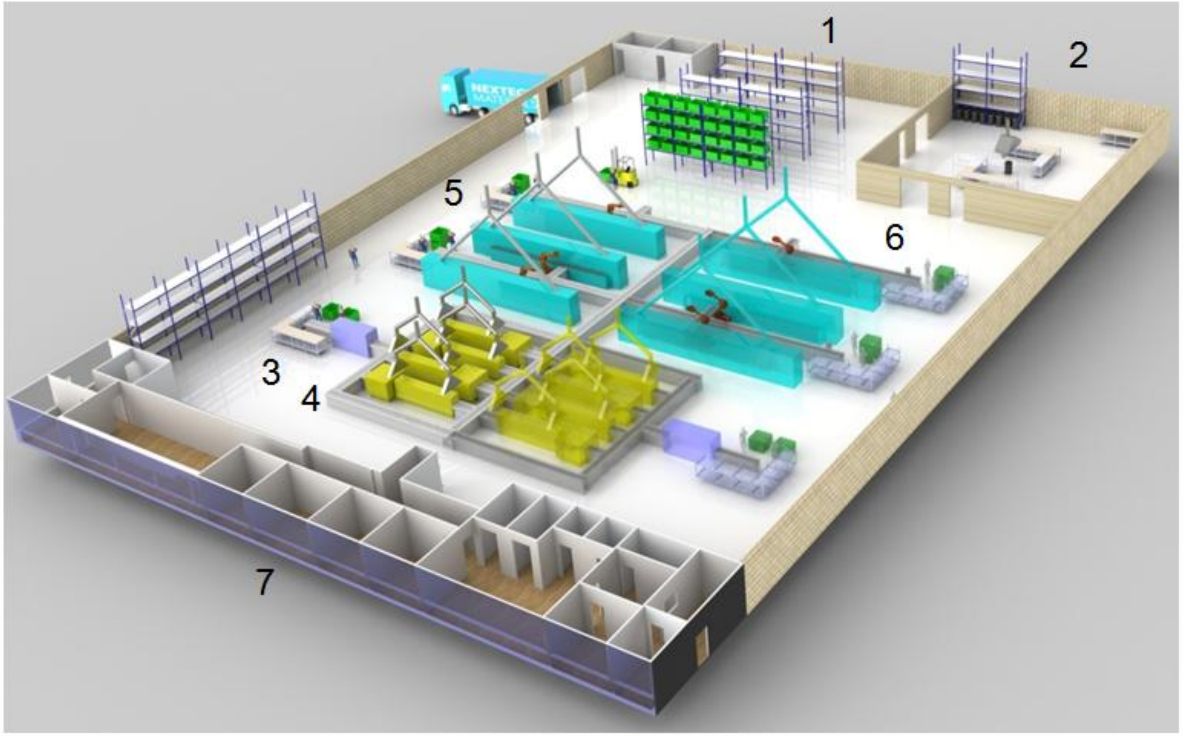
High Volume Manufacturing



Plant is 45,000 sq. ft

1. Storage racks for receiving and storage of interconnects.
2. Suspension Preparation Area
3. Inline cleaning station
4. Spray-coating operation
5. Continuous, controlled atmosphere furnaces
6. Mirrored process line demonstrating space for expansion (multiple lines for additional capacity)
7. Support Offices/Conference Room

Capacity planning and optimization

Operation Step	Scale-up Approaches
Receive and Inspection Substrates	• Automated visual inspection of parts
Clean and Stage Substrates	• Simplification of cleaning approach • Elimination of ultrasonic cleaning
Slurry Premix	• Materials Production Scale-up from 25 to 250 kg • Extending Suspension Lifetime
Spray and Dry	• High throughput spray equipment • Conveyor system for part delivery • In-situ drying of parts within spray system
Fire	• Continuous (pusher or belt furnace) furnace firing • Firing temperature profile optimization • Sintering Atmosphere
Final Inspection and QC	• Automated visual inspection of parts • QC sampling methodology

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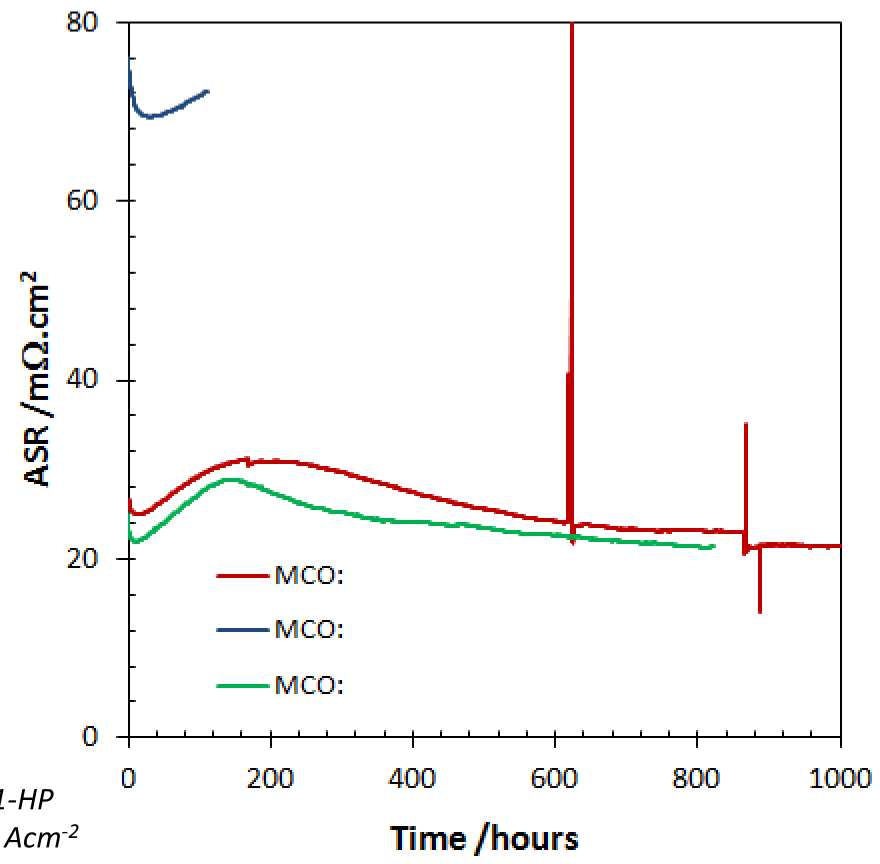
Simplification of part cleaning process

- Current part cleaning is not amenable to HVM
- Evaluated simplified cleaning procedure
- Cost reduction achieved through:
  - Labor reduction
  - No flammable solvents
  - Less expensive equipment

Symmetric ASR of MCO coated AL 441-HP



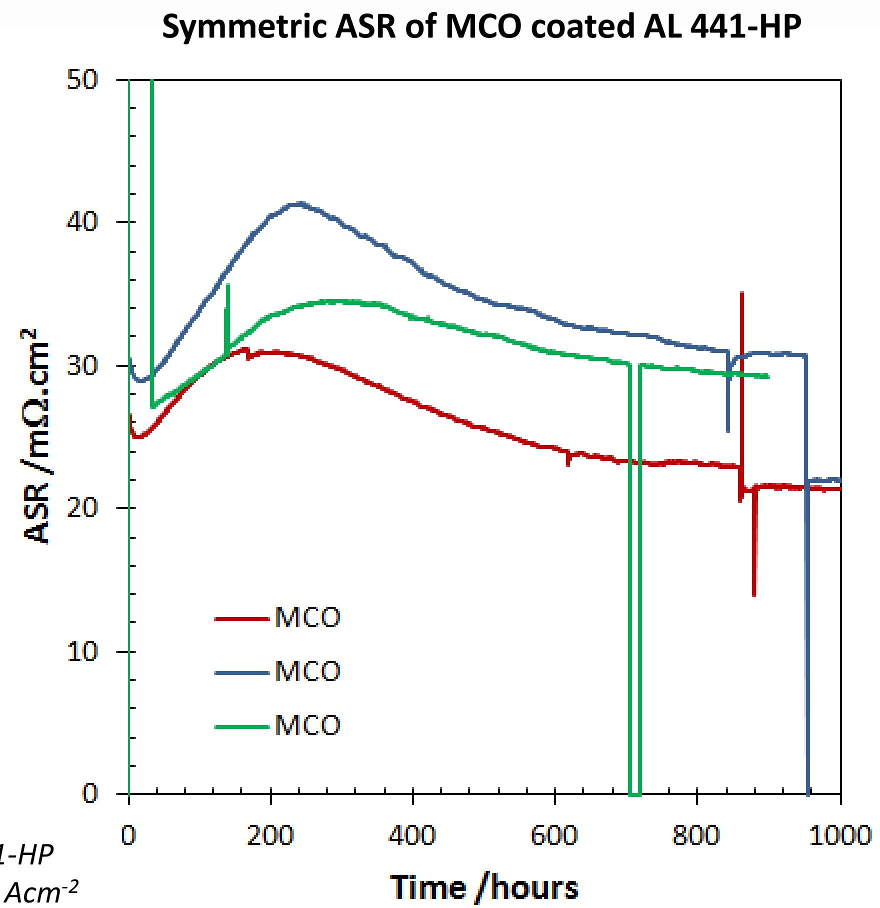
10 µm MCO coated AL 441-HP
Humidified air, 800 °C, 0.5 Acm^{-2}

Suspension Manufacturability

❑ Vehicle used in current suspension will be difficult to scale to high volume:

- Short shelf life
- Poor supplier quality

Suspension Property	V1	V2	V3
Availability	Poor	Good	Good
Manufacturability	Poor	Good	TBD
Suspension shelf life	Poor	Good	TBD
Coating Quality	Good	Poor	Good
ASR performance	Good	Good	TBD



10 μm MCO coated AL 441-HP
Humidified air, 800 °C, 0.5 Acm^{-2}

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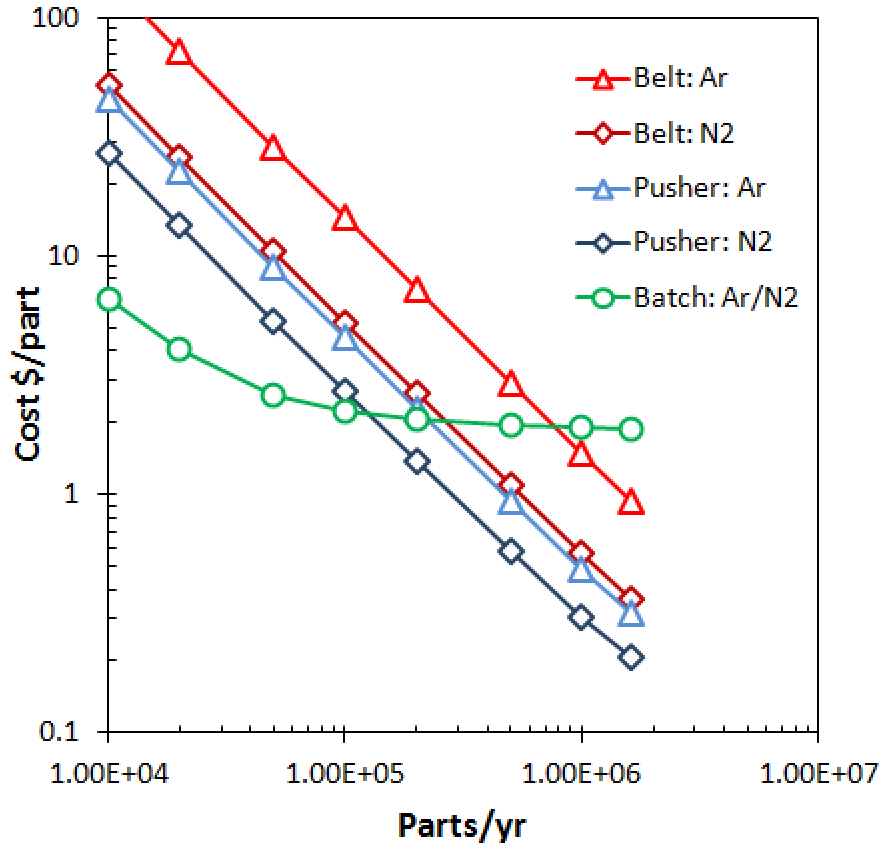
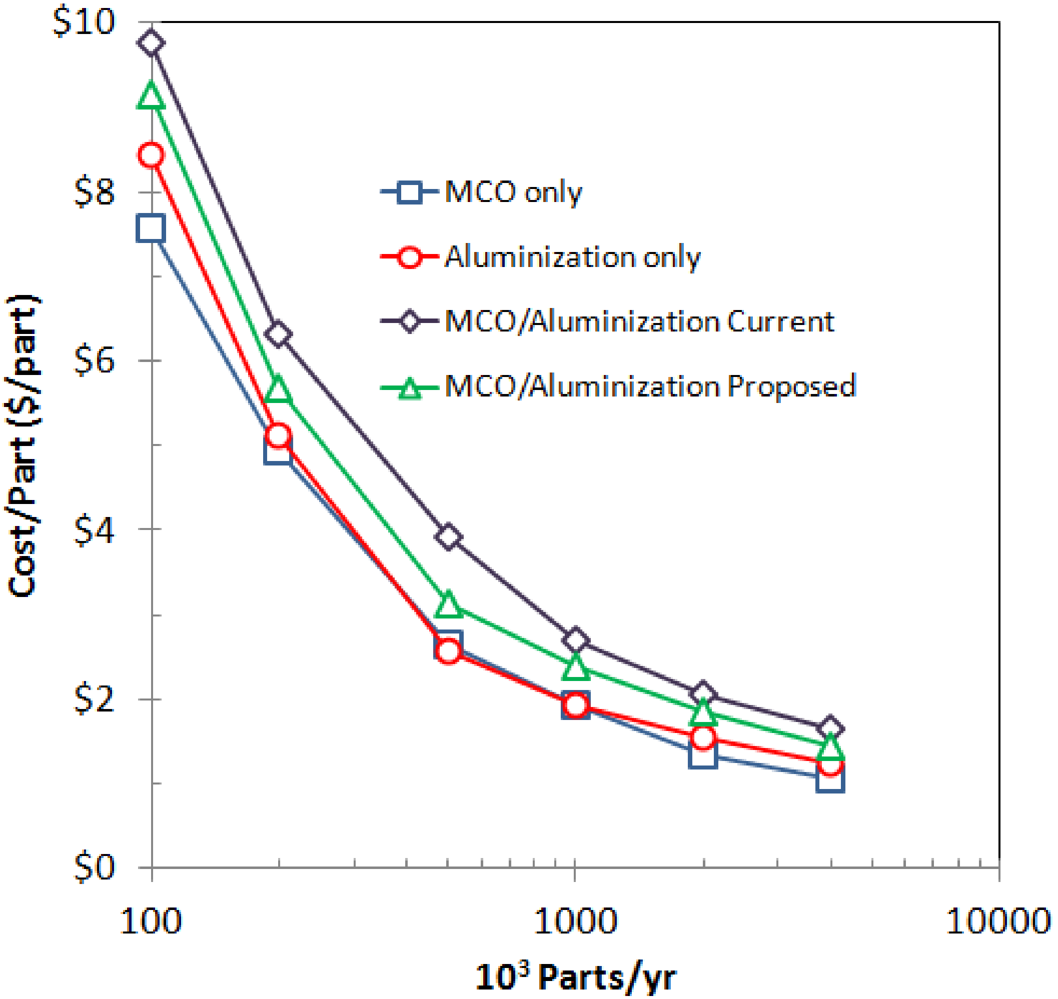
Furnace selection

- Reduction firing furnace identified as key capital investment for ASD process
  - Low volume: Batch Process
  - High volume: Continuous Process
- Determination of volume required for transition important to ensure effective use of equipment and resources



Current Cost Projections

- Component Active Area 625 cm^2
- Spray Deposition Processing
- Process Evaluation over Range of Manufacturing Scales



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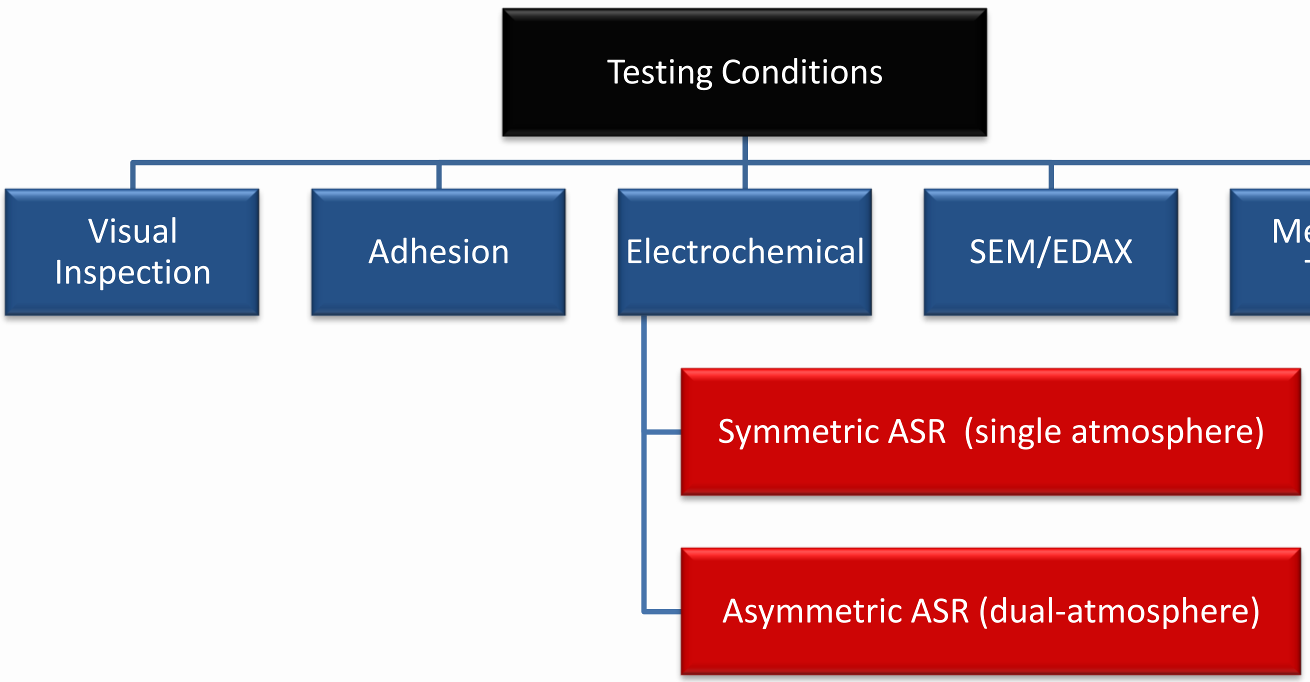
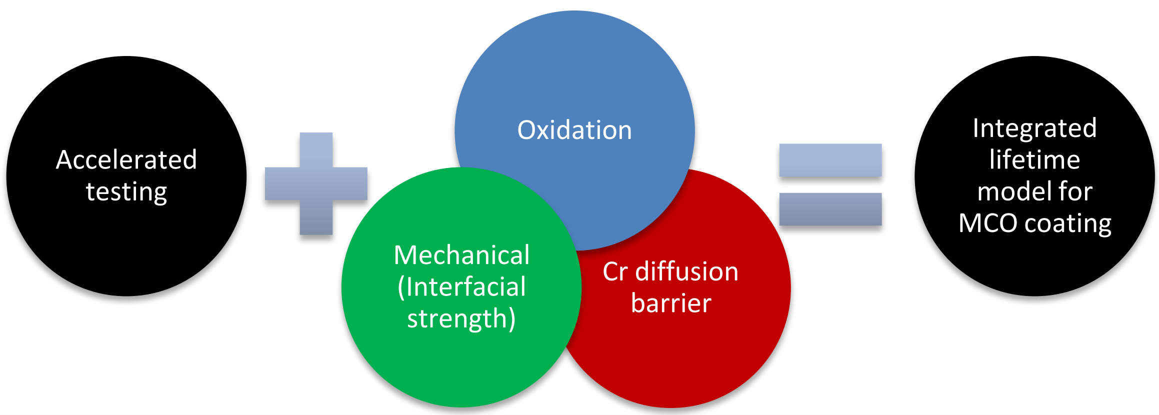
MCO coating Performance Validation

- The performance of the MCO coating is evaluated through a range of testing methods



Interconnect Coating Failure Mechanisms

- ❑ Initial coating performance influenced by a range of factors (substrate, coating).
- ❑ Pareto analysis of coating failure mechanisms conducted
- ❑ Oxidation driven failure mechanisms identified as most likely limiter of component lifetime.



- ❑ Use accelerated oxidation kinetics (determined from oxidation experiments) to estimate coating lifetime based on long-term electrical stability data.

10 μm MCO coating on AL 441-HP substrate 900 °C, 200 hours, Air

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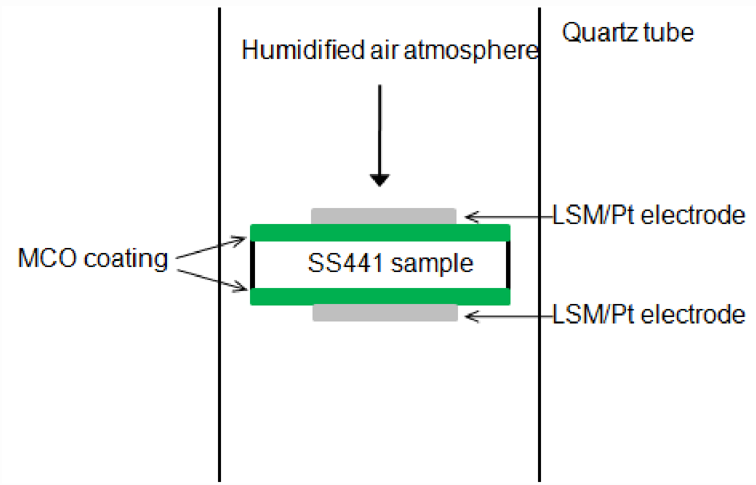
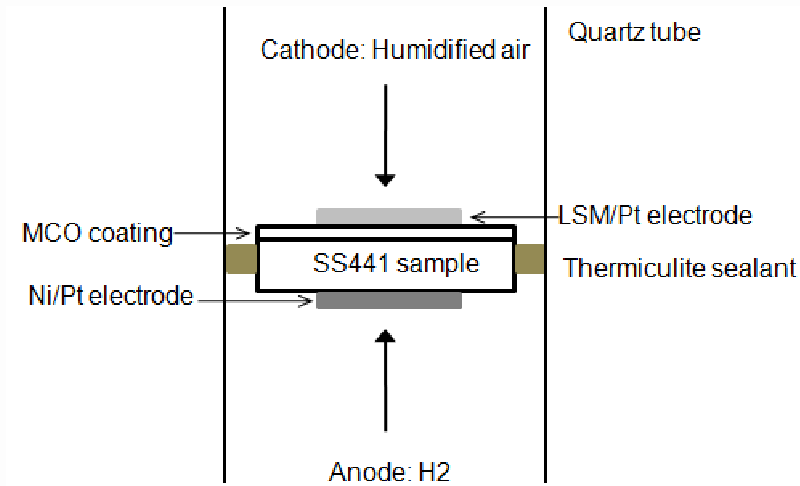
MCO coating performance testing

❑ Electrical performance of MCO coating evaluated by four-point area-specific resistance (ASR) testing in both symmetric (single atmosphere) and asymmetric (dual-atmosphere) configurations

Symmetric ASR test configuration



Asymmetric ASR test configuration



❑ Electrical performance of MCO coating evaluated by four-point area-specific resistance (ASR) testing in both symmetric (single atmosphere) and asymmetric (dual-atmosphere) configurations

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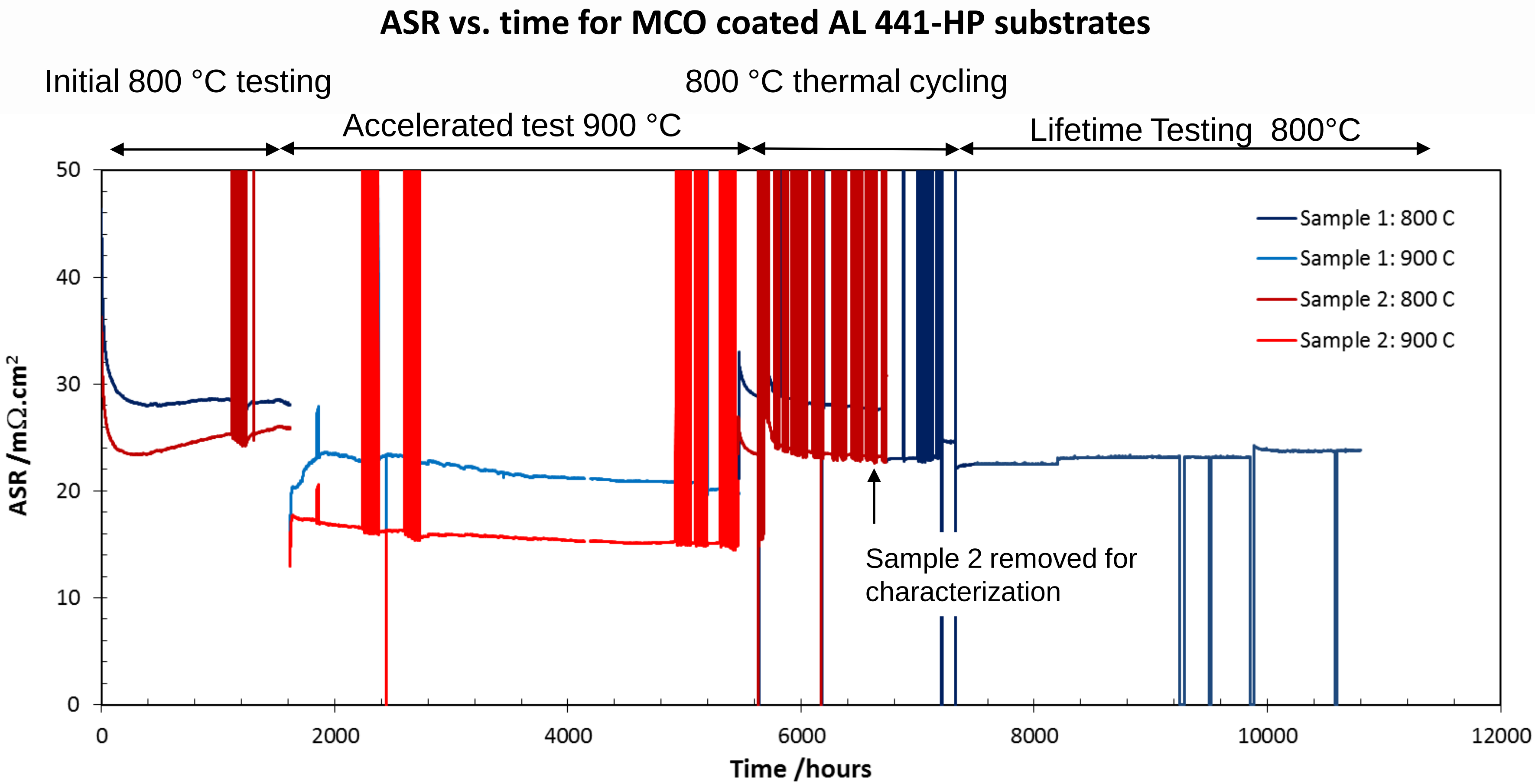
Long-term ASR performance validation



***Test Conditions**: Symmetrically MCO coated AL 441-HP, Humidified air, 800 °C/900 °C to 50 °C, 0.5 A.cm^{-2}*

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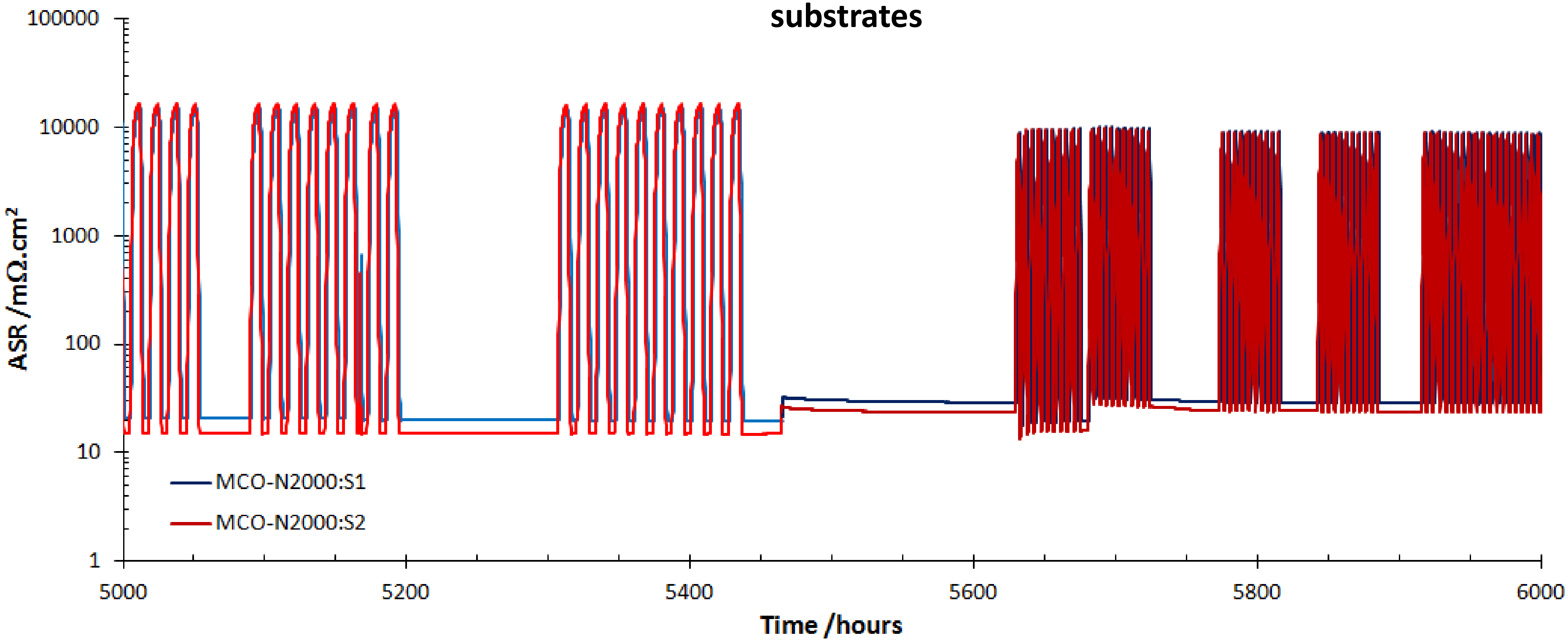
Thermal Cycling performance

- ❑ Thermal cycling is incorporated into stability testing to evaluate the resistance of the coating to thermally driven spallation.

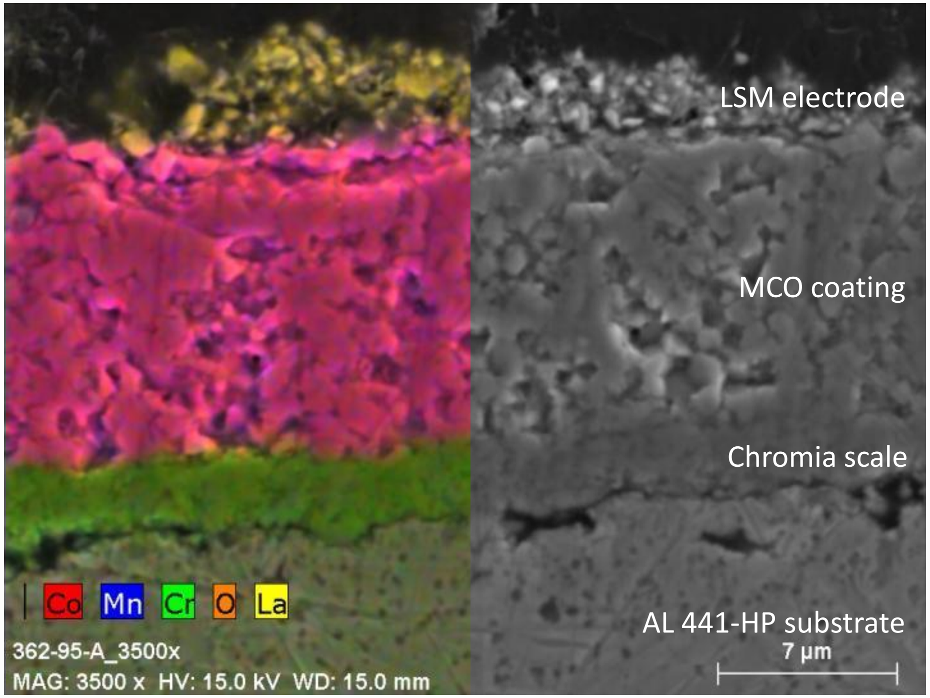
Enlargement of thermal cycling after 5000 hours on test: ASR vs. time for MCO coated AL 441-HP substrates



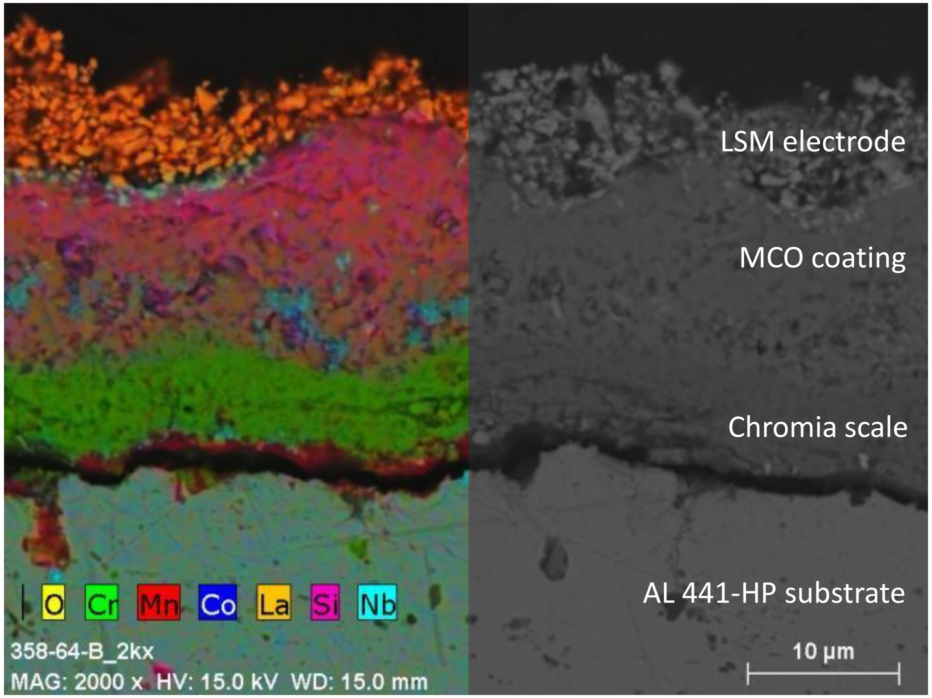
Test Conditions: *Symmetrically MCO coated AL 441-HP, Humidified air, 800 °C/900 °C to 50 °C, 0.5 A.cm^{-2}*

Microstructural Evolution of MCO coating

800 hrs 800 °C



> 7000 hrs 800 °C/900 °C



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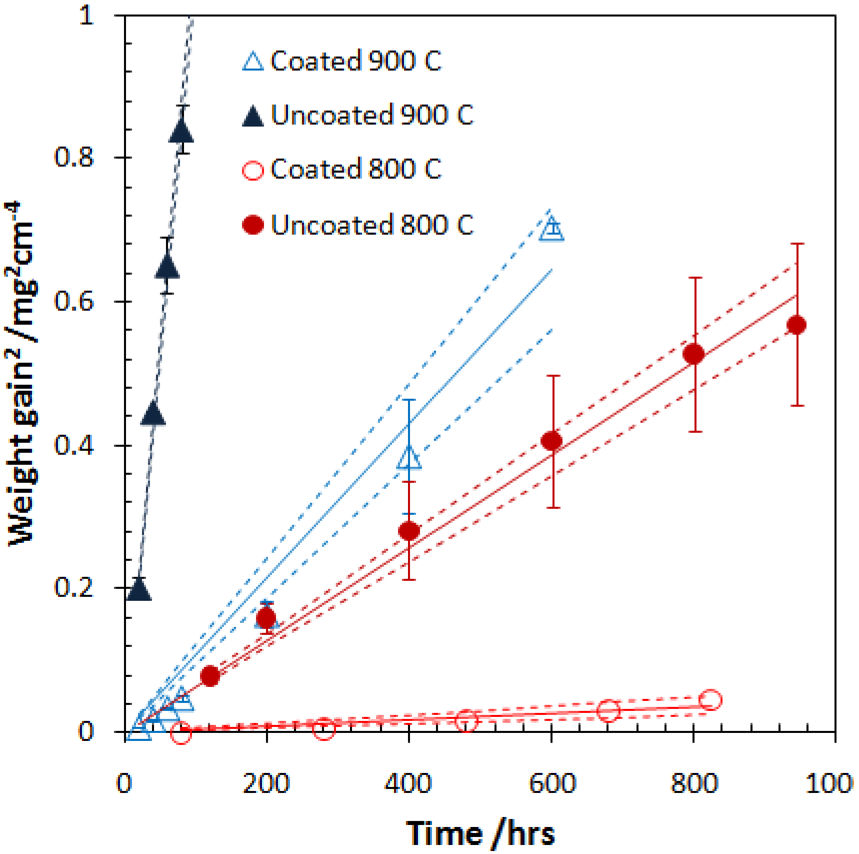
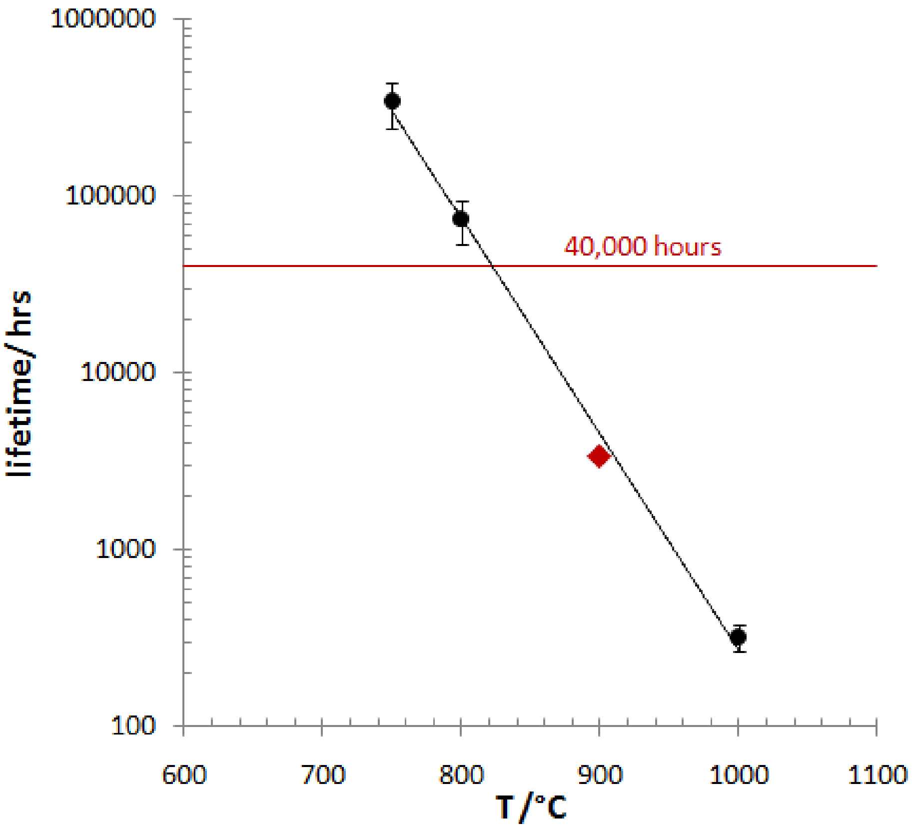
Coated Interconnect Lifetime Predictions

Oxidation (weight-change)^2 vs. time for coated and uncoated AL 441-HP at 800 and 900 °C.



Lifetime predictions



10 µm MCO coating – AL 441-HP substrate, Symmetric ASR testing: 800/900°C, Humidified Air, 0.5 Acm^{-2} current density

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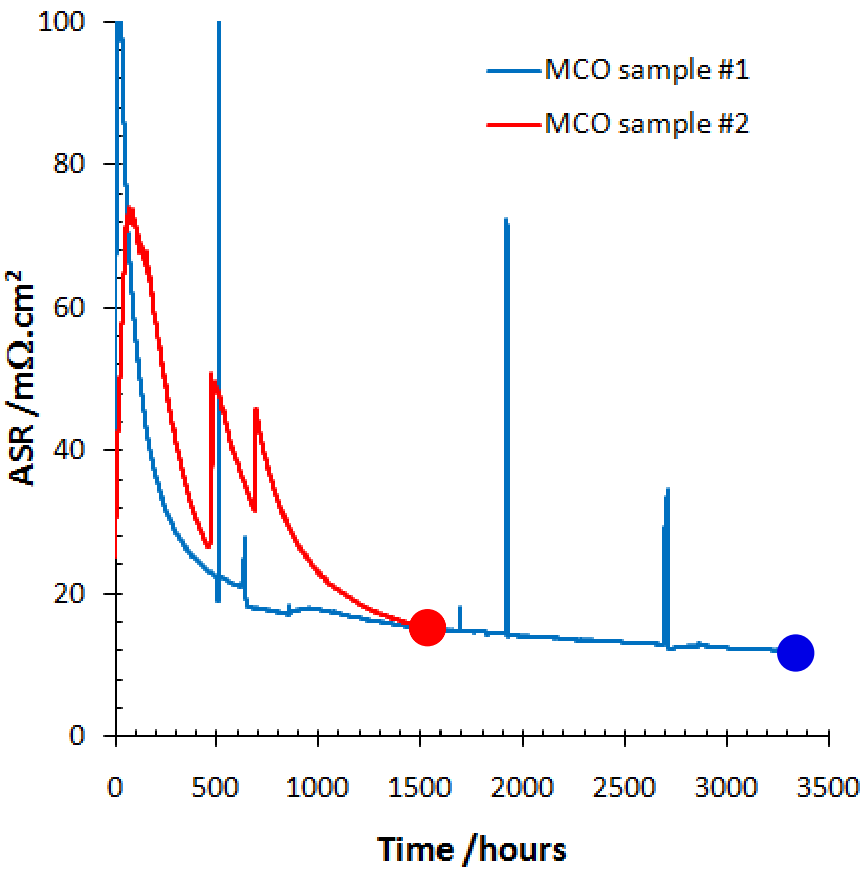
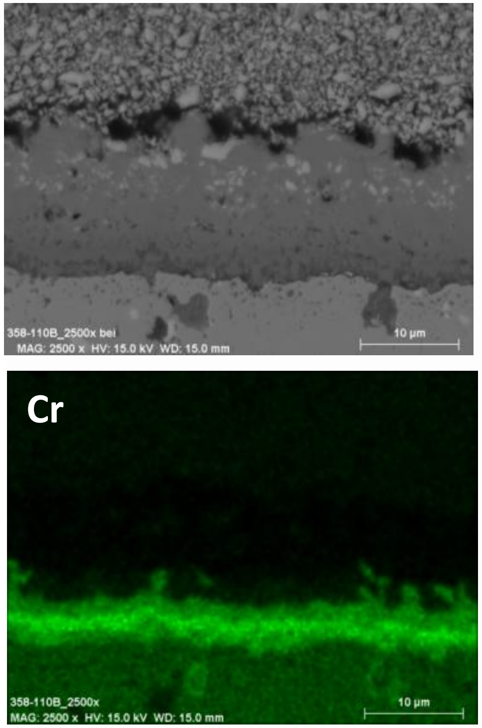
Asymmetric ASR performance testing

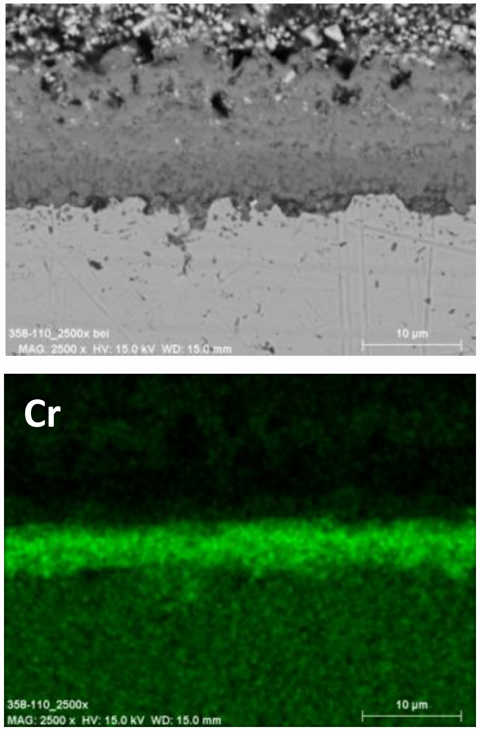
ASR vs. time for MCO coated AL 441-HP substrates



1600 hours (#2)



3400 hours (#1)



AL 441-HP: 10 μm MCO coating on cathode / No coating on anode.
800°C , H2/Humidified Air, 0.5 A.cm^{-2}

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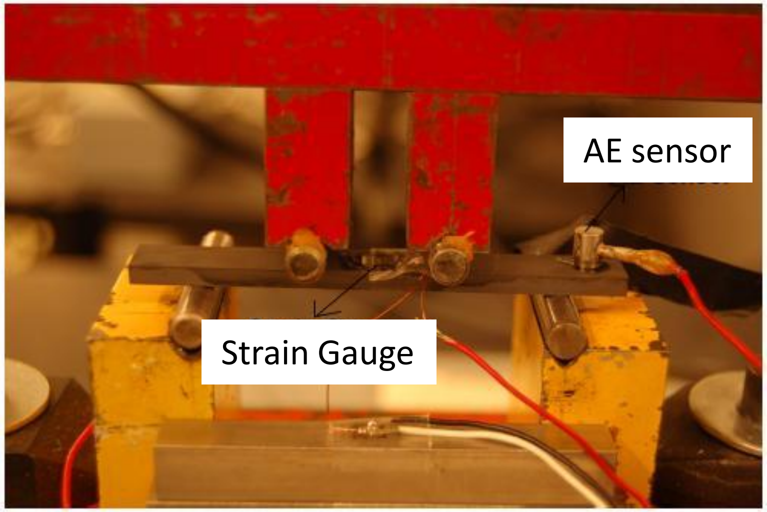
DEPARTMENT OF MECHANICAL AND AEROSPACE ENGINEERING

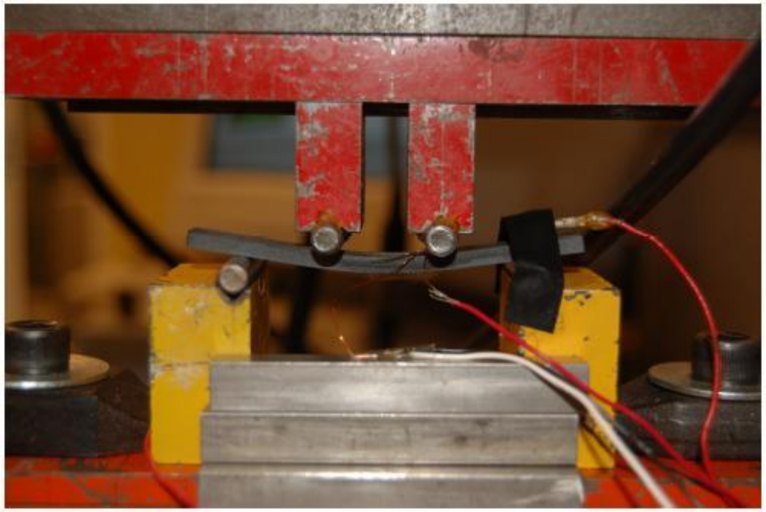
Mechanical Testing

- ❑ Collaborating with Dr. Mark Walter’s group at OSU through NSF, GOALI program.
- ❑ Investigating interfacial and shear strength of our coating on a range of substrates through synchronized four-point bend and acoustic emission testing

Four-point bend set-up



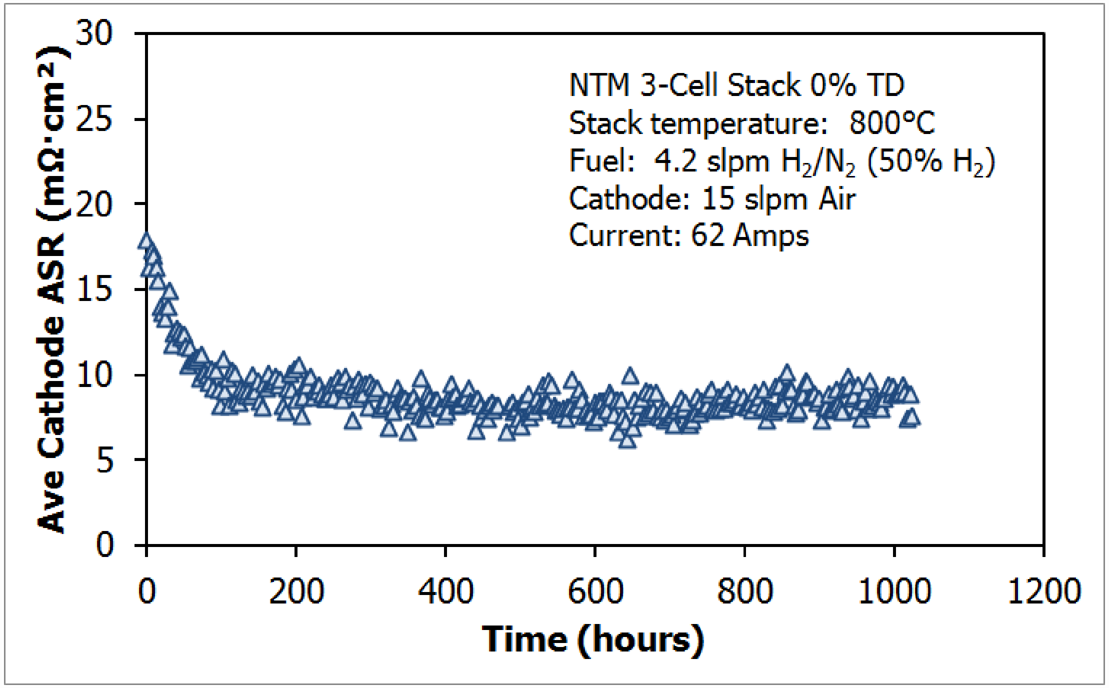
Coated IC bar undergoing test



- ❑ Objective is to understand how interfacial strength changes with time to develop predictive lifetime model for IC coating – enable design of optimized coating/substrate solutions.

Performance Validation: 1 kW stack test

- Integrated coating technology into SOFC stacks from three-to-five cell short stacks up to 1 kW stacks.



- Post Mortem characterization of coated IC components by SEM/EDAX after stack tests is in progress.

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Coating Performance Validation: Conclusions

- ❑ Baseline Interconnect Coating performance identified
- ❑ Successfully demonstrated excellent long-term performance of MCO coating in life-time stability testing:
  - > 11,000 hours on test (at both 800 °C and 900 °C)
  - > 200 thermal cycles
  - Post-test SEM/EDAX characterization indicates minimal degradation of the coating
- ❑ Accelerated testing at 800/900 °C related to oxidation kinetics to simulate > 40,000 hours service
- ❑ Potential failure modes for MCO coating identified and lifetime model for coating developed
- ❑ Coated interconnects successfully incorporated into SOFC stacks
  - Stack stability improvement demonstrated for coated vs. uncoated interconnects

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Conclusions

- **Refined cost and manufacturing models to encompass volumes from prototyping through full volume production.**
  - Market forecast and demand curves defined for three OEM profiles at various stages of commercialization.
  - Three-stage technology roadmap developed.

- **Identified manufacturing strategies to reduce volume manufacturing costs.**
  - Materials processing scale-up to 25 kg batch sizes and beyond
  - Plant designs for >600,000 m^2/year coating

- **Defined key process limits for ASD coated ICs.**
  - Lifetime stability tests in progress (> 11,000 hrs operation at ≥ 800 °C in single atmosphere configurations).
  - 1800 h testing in dual atmosphere conditions

- **Identified key failure mechanisms and acceleration factors.**
  - Predictive lifetime models successfully applied to long-term stability tests.

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Acknowledgements

Clients, Colleagues and Collaborators

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Briggs White-NETL

Jeffry Stevenson-PNNL