SECA Stack Test Fixture Development and Implementation

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Presentation Outline

- Conclusions
- Background
- Objectives
- Approach
- Test Fixture Design & Assembly
- Implementation: Test Results
- Future Work
- Conclusions
- Acknowledgements

Conclusions/Accomplishments

- A stack test fixture based on 50mm x 50mm cells (40mm x 40mm cathode) has been developed for evaluation/validation of new materials, fabrication processes, and design concepts.
- Implementation:
  - Performance of refractory glass seals has been validated.
    - Seals exhibited excellent performance during 25 deep thermal cycles
  - Current emphasis is on evaluation/validation of low-cost interconnect alloys and cathode-side interconnect modifications:
    - AISI 441 ferritic stainless steel
    - Ce-MnCo spinel coatings for exposed interconnect surfaces
    - Aluminization of sealing surfaces
- Working to improve performance and reproducibility
  - Minimize "hand-crafted" aspects of assembly; replace with "automated" assembly processes
- Developing revised fixture design
  - Consistent with future available thicknesses of AISI 441 (coil purchased from ATI Allegheny Ludlum by SECA program)
  - Multiple cell capability (3 cells or more)
- Transferring test capability to NETL

Background

- SECA Core Program Testing at Sub-stack Level
  - Materials Characterization
    - XRD, SEM, EDS, TEM, XPS, TGA, DSC, PSA, dilatometry, electrical conductivity, single & dual atmosphere oxidation
  - Multiple Component Tests
    - Button cell testing
    - ASR testing of interconnect/cathode contact/cathode structures
    - Electrical testing and leak testing of seal/interconnect and cell/seal/interconnect structures
- Next Step: Testing under realistic “stack-like” conditions
  - Advantage: Higher degree of relevance to SECA Industry Team cells/stacks
  - Challenges:
    - Multiple components & phenomena, so results more difficult to interpret
    - Increased complexity of assembly, co-fabrication of seals and electrical contact materials

Objectives

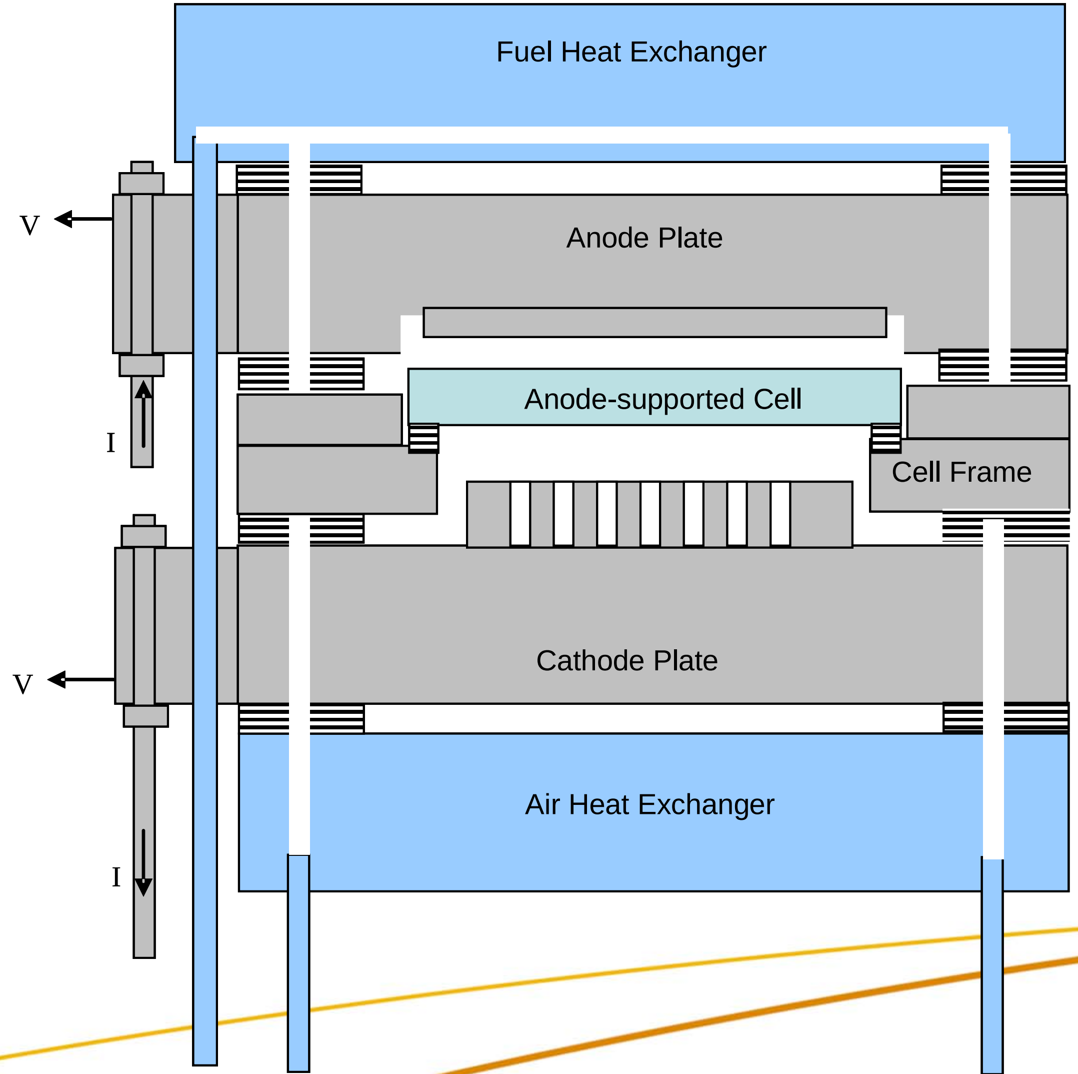
- Develop SOFC stack test fixture for SECA Core Technology Program (CTP)
- Evaluate/validate new materials and fabrication processes under realistic stack conditions
  - Larger cell size (≥50mm x ≥50mm)
  - Complete stack functionality (cell, cell frame, seals, interconnects, electrical contact materials)
- Share fixture design with other SECA participants
  - Easy to assemble and test; Minimal fabrication cost
- Accelerate technology transfer from SECA CTP to SECA Industry Teams
  - Bridge the gap between small-scale CTP tests (e.g., button cells) and SECA industry team stacks

Approach

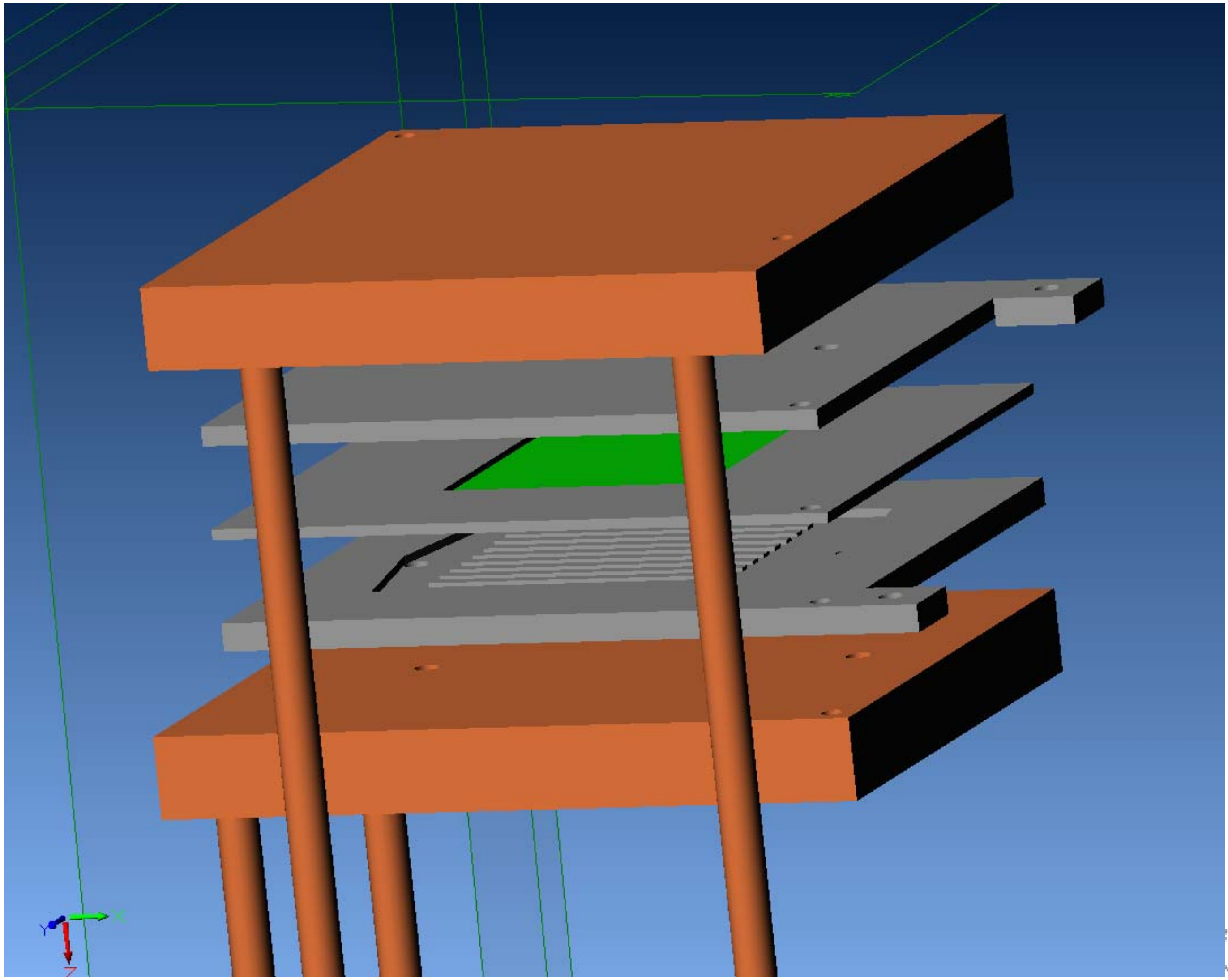
- Design
  - Initial fixture design provided by LBNL and NETL
  - Design modified by PNNL to incorporate cell-in-frame design concept, both cell-to-frame and perimeter seals, alternative flow path geometries. Iterative design approach – i.e., results from previous tests used to identify design limitations
- Current Implementation
  - Parts
    - Cells purchased from H.C. Starck (InDEC)
      - 50 mm x 50 mm ASC3 anode-supported cells; LSM/YSZ cathode; ~$200 each
    - Other components (interconnects, cell frame, seals, contact pastes) fabricated at PNNL
      - AISI 441 stainless steel provided by Allegheny Technologies, Inc.
  - Assembly and testing at PNNL
    - Electrochemical performance evaluation via I-V and EIS analysis
    - Post-test analysis via optical microscopy, XRD, SEM, EDS, TEM, XPS, etc
    - Compare stack results with results from tests on individual materials and sub-stack structures, as well as modeled results

Schematic Illustration of Stack Test Fixture

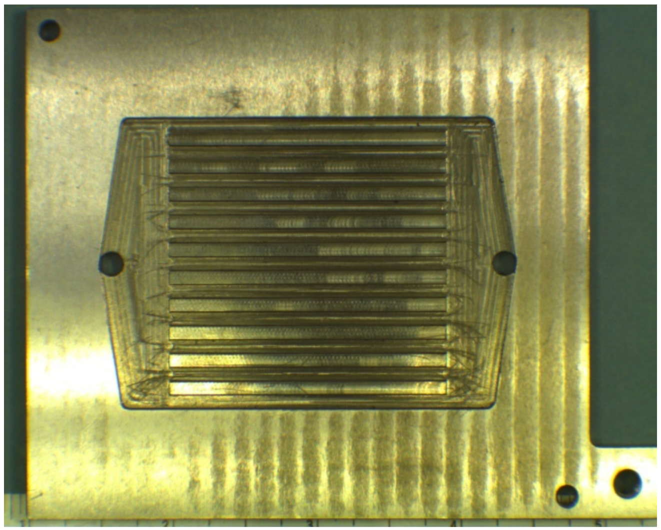
Stack Test Cross-Section
(Not to Scale)



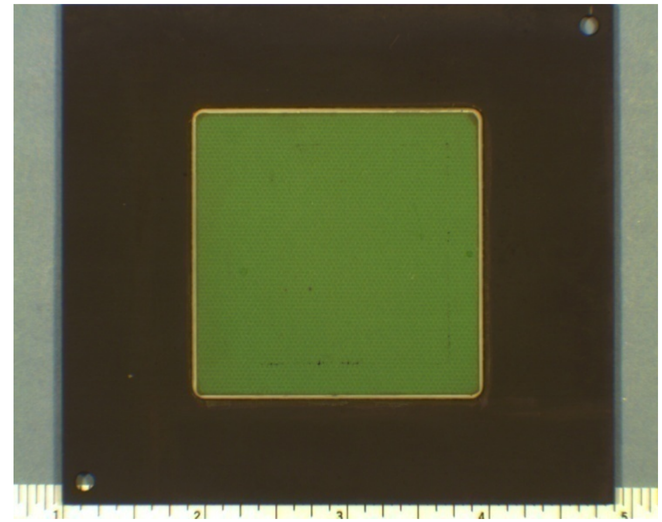
Scale Drawing of Counter-Flow Test Fixture



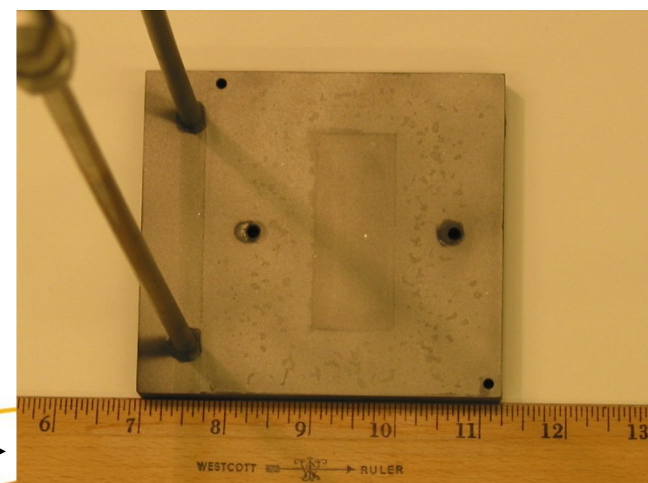
Stack Test Fixture Components



Anode interconnect plate (SS441)



Cell sealed in “window frame” (SS441) w/ refractory glass



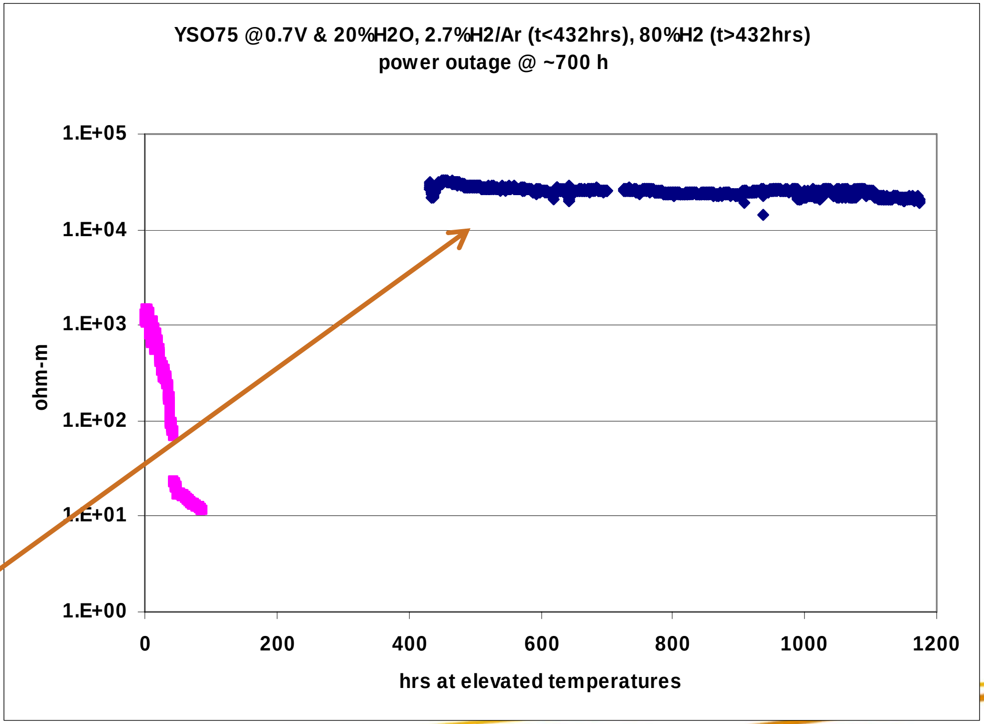
Air heat exchanger (Inconel 600)

Implementation of Stack Fixture Tests

- Selected Test Results
  - Seal validation
    - Refractory glass seals tested under thermal cyclic conditions
  - Evaluation of low-cost steel interconnects with protective coatings
    - Simultaneous testing of spinel-coated and aluminized alloy surfaces

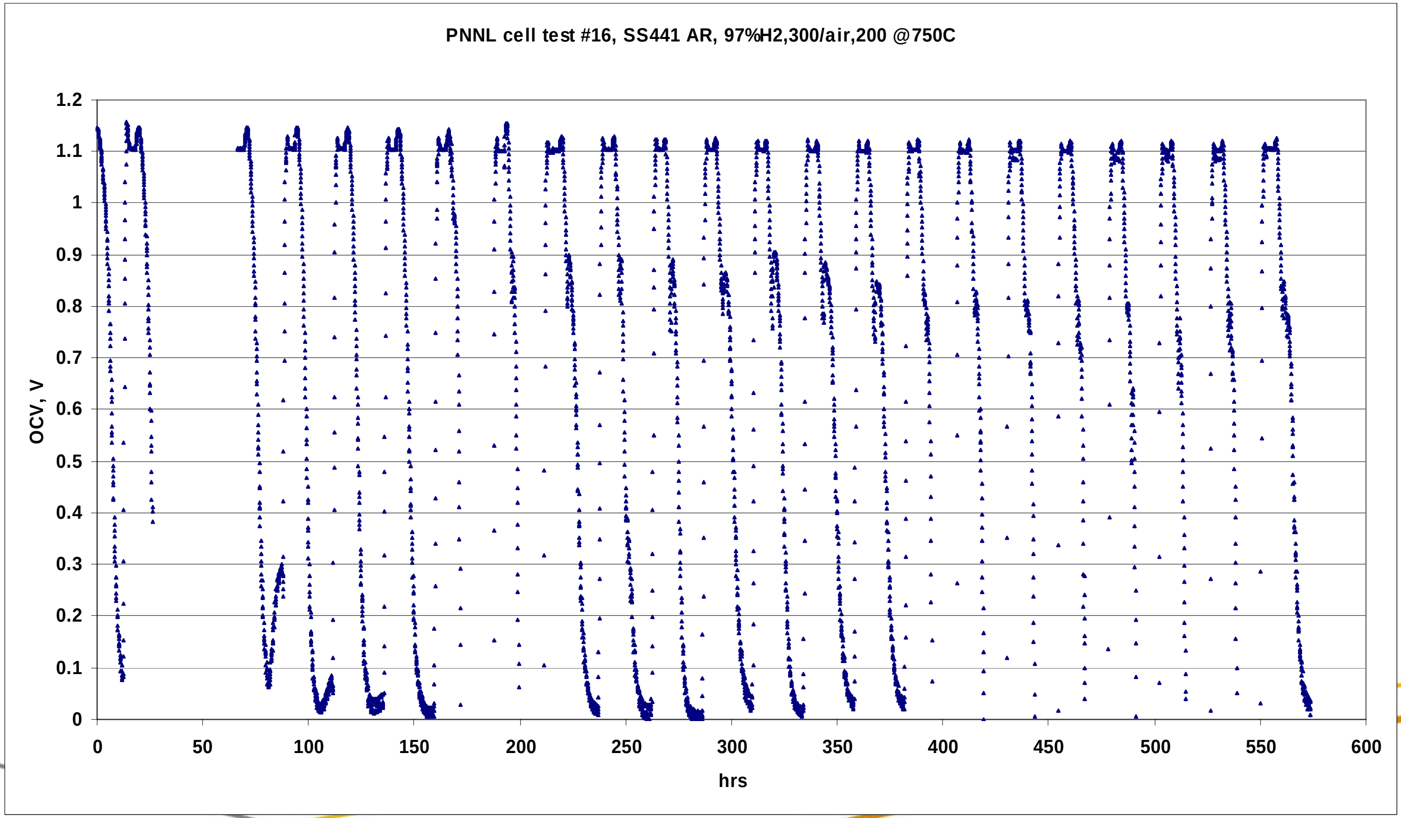
SECA CTP Stack Test Fixture: Refractory Glass Seal Evaluation

- Refractory sealing glasses --- Compositional modifications to G18 glass have resulted in:
  - Excellent wetting/sealing behavior to YSZ and interconnect alloys at 950-1000ºC
    - Potentially improves electrical conductivity and strength of cathode/interconnect contacts
  - Rapid de-vitrification and stabilization of polycrystalline phases
  - Stable, matching CTE (11.5-12.5 ppm/ºC; stable CTE up to 2,000 hours testing)
  - Improved chemical stability at operating temperatures
  - Stable, high electrical resistance in contact with steel interconnect (up to 1,200 hours testing)



SECA CTP Stack Test Fixture: Refractory Glass Seal Evaluation

Cell with refractory glass seal demonstrated stable OCV during 25 deep thermal cycles (RT – 750ºC)



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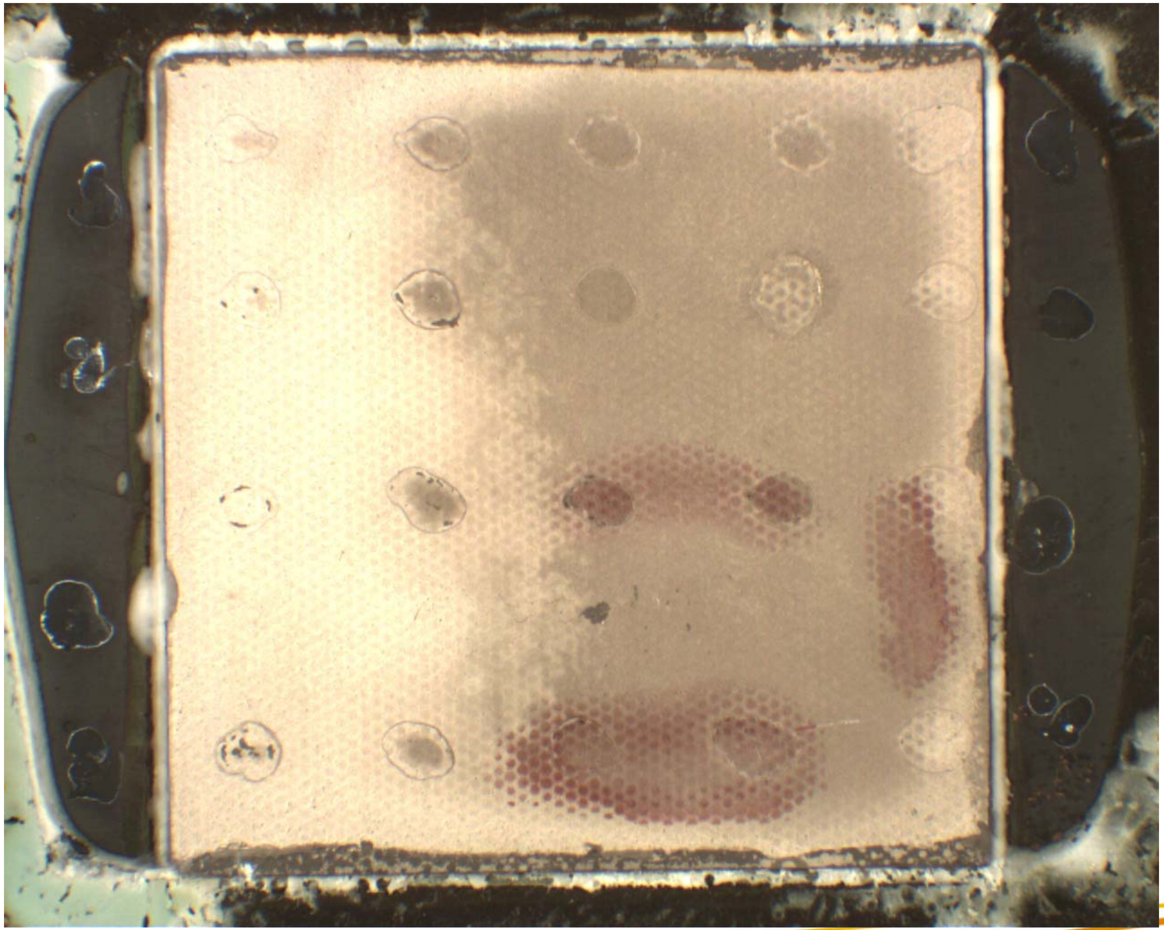
SECA CTP Stack Test Fixture: Refractory Glass Seal Evaluation - Post-test Analysis

- **Cell-to-frame glass seal remained intact (no dye penetration)**
- **Refractory glass selected as baseline seal composition for stack fixture testing**



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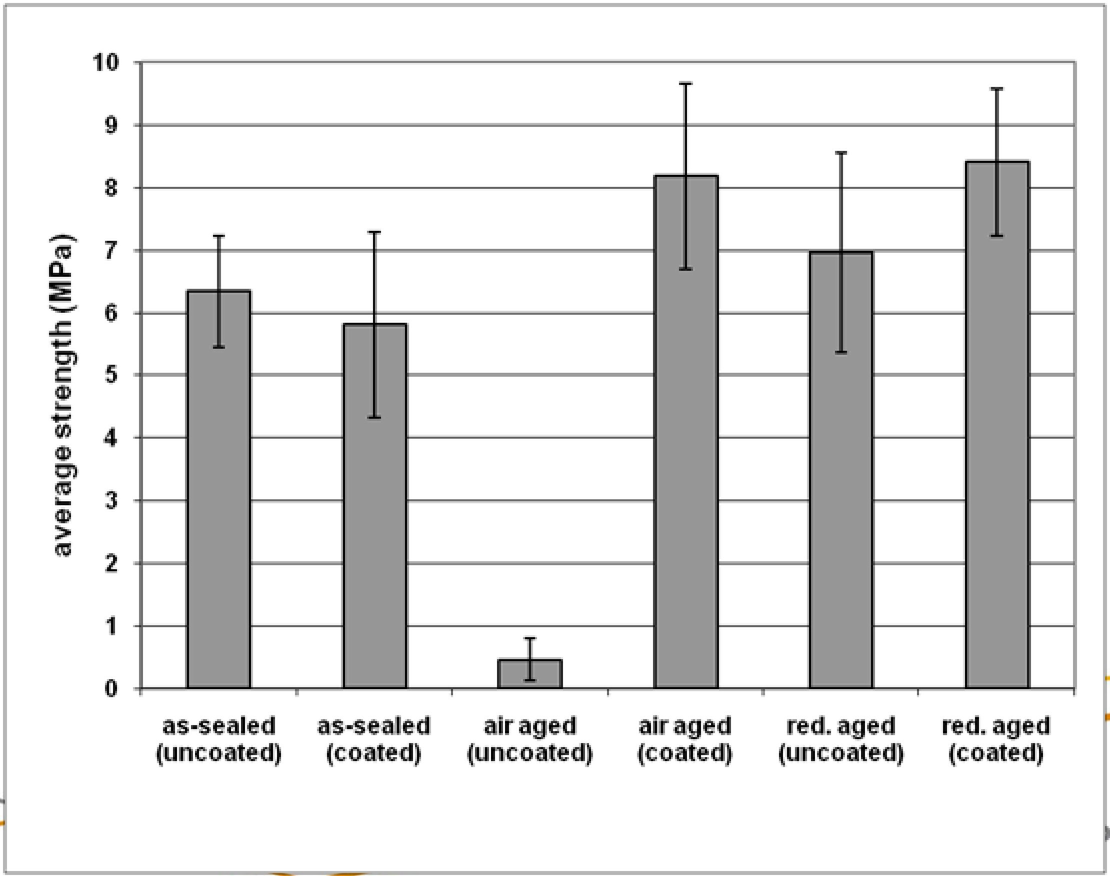
Evaluation of Low-Cost Steel Interconnects with Protective Surface Coatings

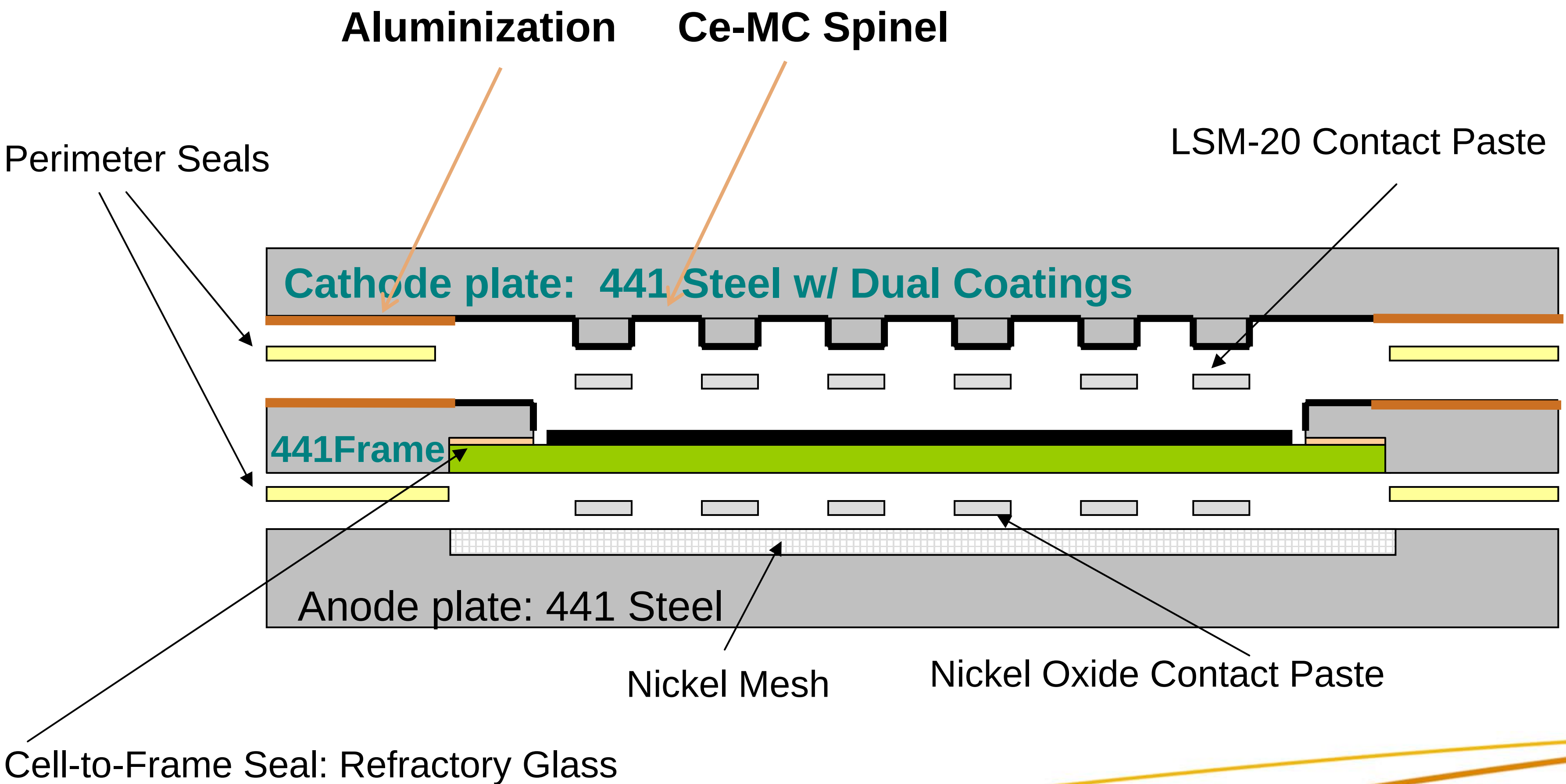
- Interconnect Components:
  - AISI 441 stainless steel
    - Pros: Inexpensive, matching CTE, easy to fabricate, conductive Cr-based scale, no SiO_2 subscale layer
    - Cons: Oxidation leads to increased electrical resistance, poor scale adherence, Cr volatility, reaction with glass seals
  - Ce-modified MnCo Spinel Coating
    - Reduces oxidation rate, improves scale adherence, mitigates Cr volatility
  - Aluminization
    - Mitigates reaction with glass seals

Evaluation of Low-Cost Steel Interconnects with Protective Surface Coatings

- Aluminization of alloy surface
  - Mitigates chromate formation, which degrades strength of seal

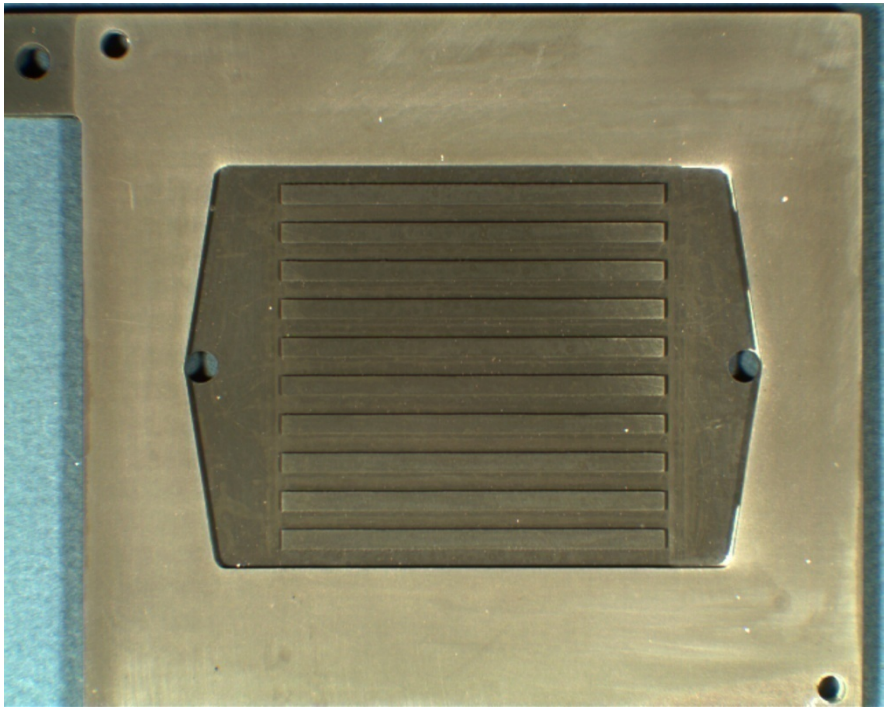


Evaluation of Low-Cost Steel Interconnects with Protective Surface Coatings



Evaluation of Low-Cost Steel Interconnects with Protective Surface Coatings

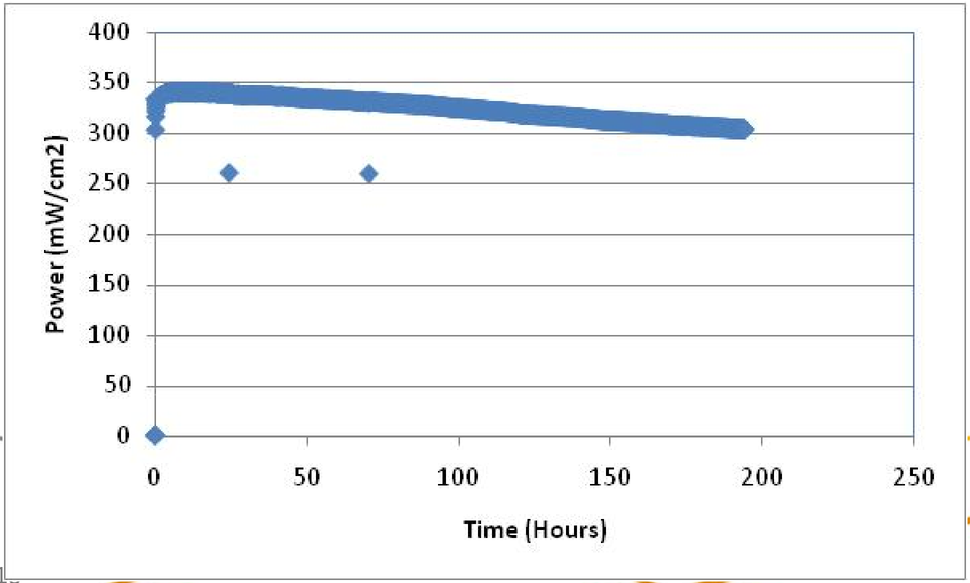
- Dual Coatings
  - Ce-MnCo spinel application to surfaces exposed to air
  - Aluminization of sealing surfaces

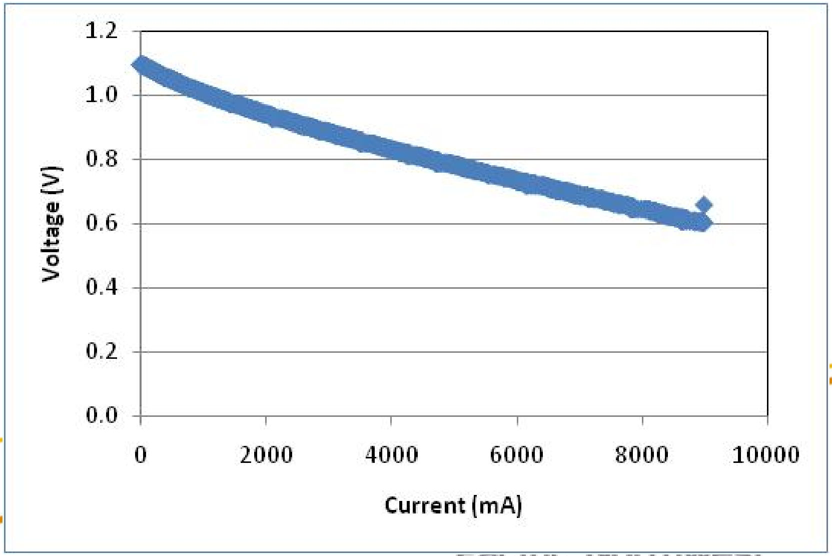


- Aluminization
  - Commercial processes also available from vendors, but challenging when dealing with small quantities of parts
  - New aluminization process under development at PNNL

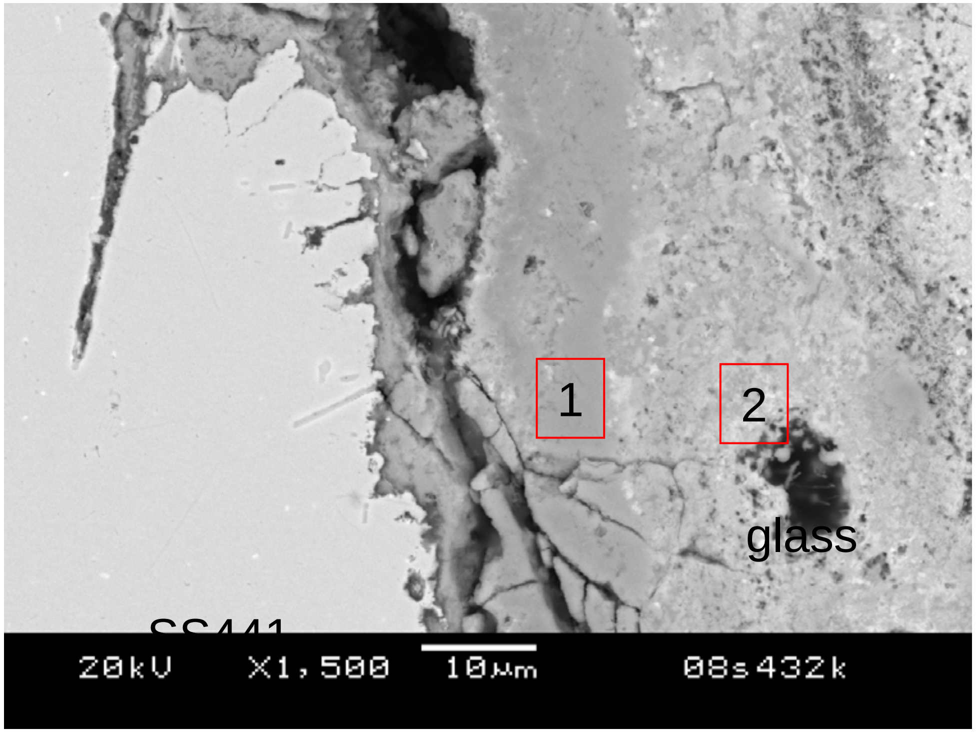
Evaluation of Low-Cost Steel Interconnects with Protective Surface Coatings - Results

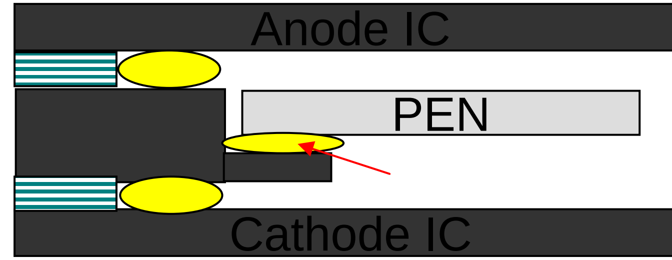
- First test
  - Good OCV, but poor electrical performance
  - Test was held at 800ºC for 400 hours under OCV conditions
  - Post-test structural/chemical analysis performed by SEM/EDS
- Second test
  - Off to good start, but aborted by building power failure
- Third test
  - In progress
  - Plan to run for 1000 hours, then perform post-test analysis





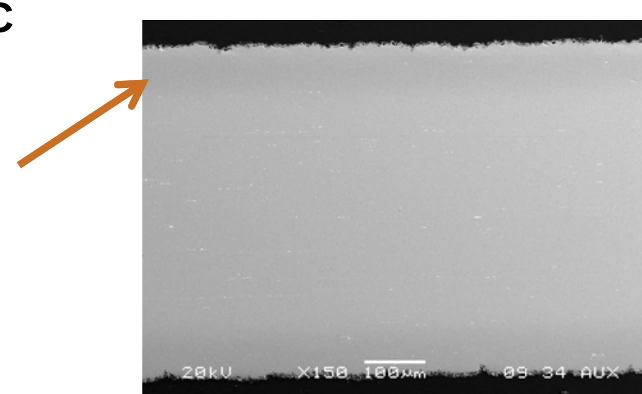
Aluminized 441/Glass Seal Interface



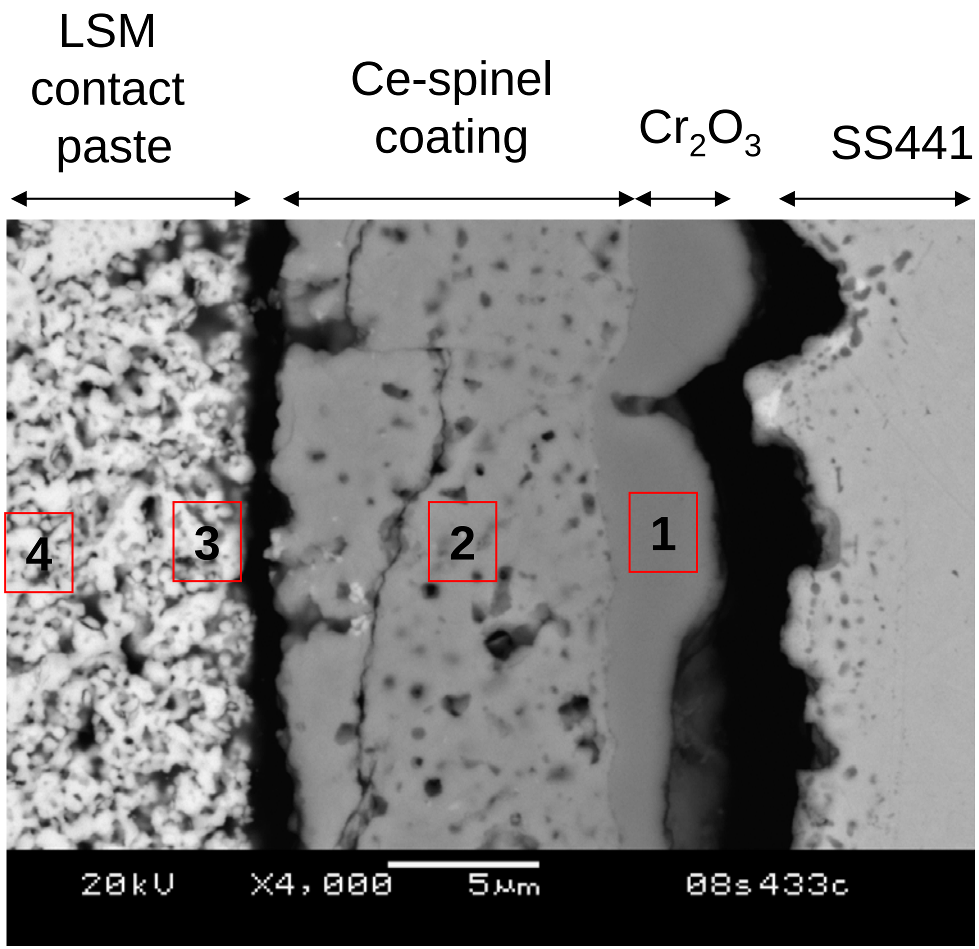


point	1	2
O	35.0	39.2
Al	1.1	5.0
Si	1.6	11.2
Ti	0.5	
Cr	41.8	16.1
Mn	1.1	
Fe	17.4	15.2
Cu	0.9	
Sr	0.6	8.6
Mg		0.7
Y		1.1
Ba		2.9

- Aluminized SS441 frame / glass seal after 404h @ 800ºC
- Cr transport from the alloy observed, possibly due to non-uniform aluminized interface
- Uniform coatings have been obtained in previous work with oxidizing treatment only
- May be consequence of double heat treatment required for dual coating of parts
- Optimization of dual coating process in progress



Spinel-coated 441/LSM Contact Paste Interface



point	1	2	3	4
O	37.7	30.1	31.2	32.0
Ti	1.5	0.4		
Mn		37.7	31.6	33.8
Co		31.8	5.1	
Cr	60.8			
Sr			5.8	6.7
La			26.3	27.5

- Ce-modified spinel-coated section of SS441 interconnect after 404h @ 800ºC
- Results similar to substack testing (ASR tests)
- Effective blocking of Cr by spinel coating

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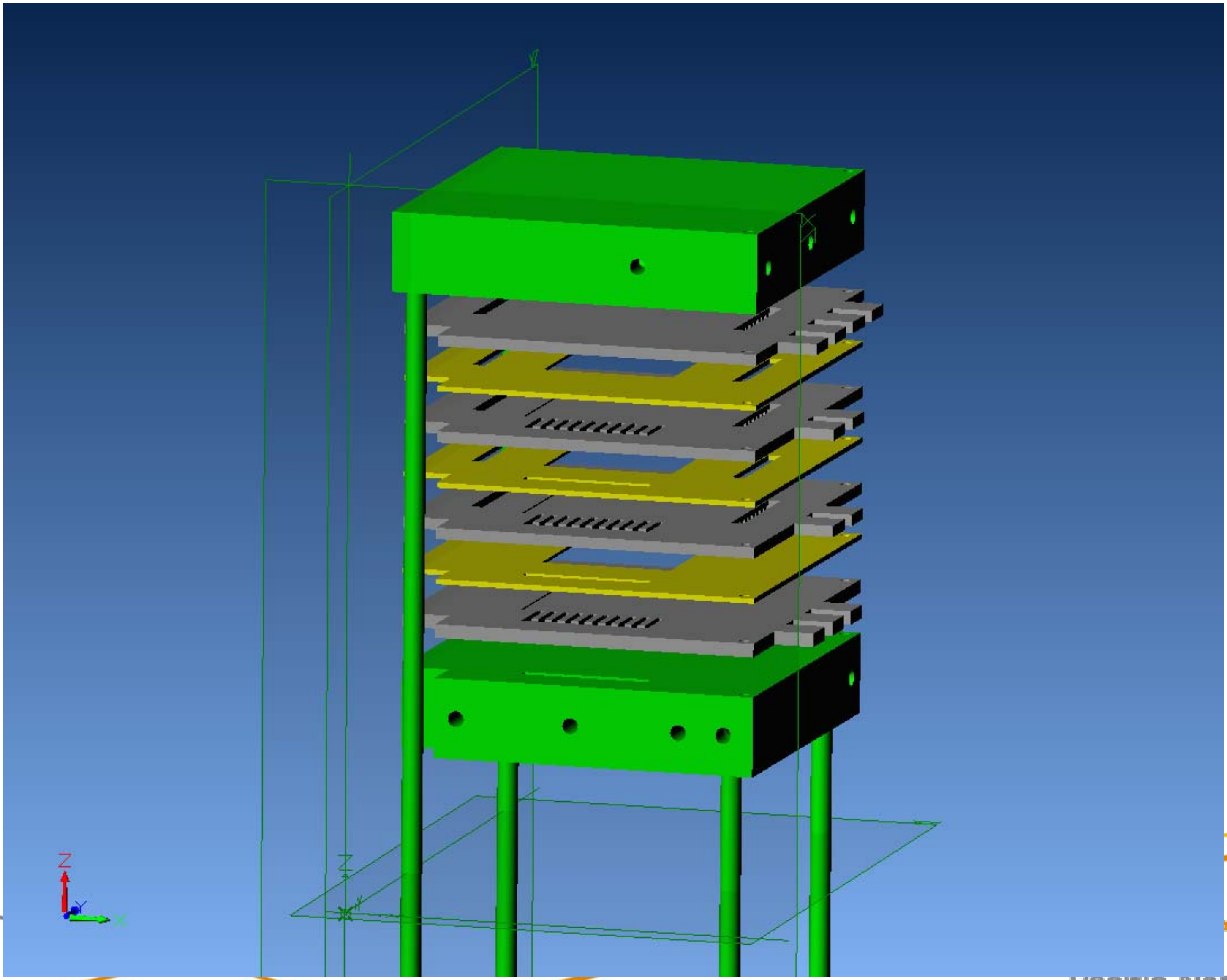
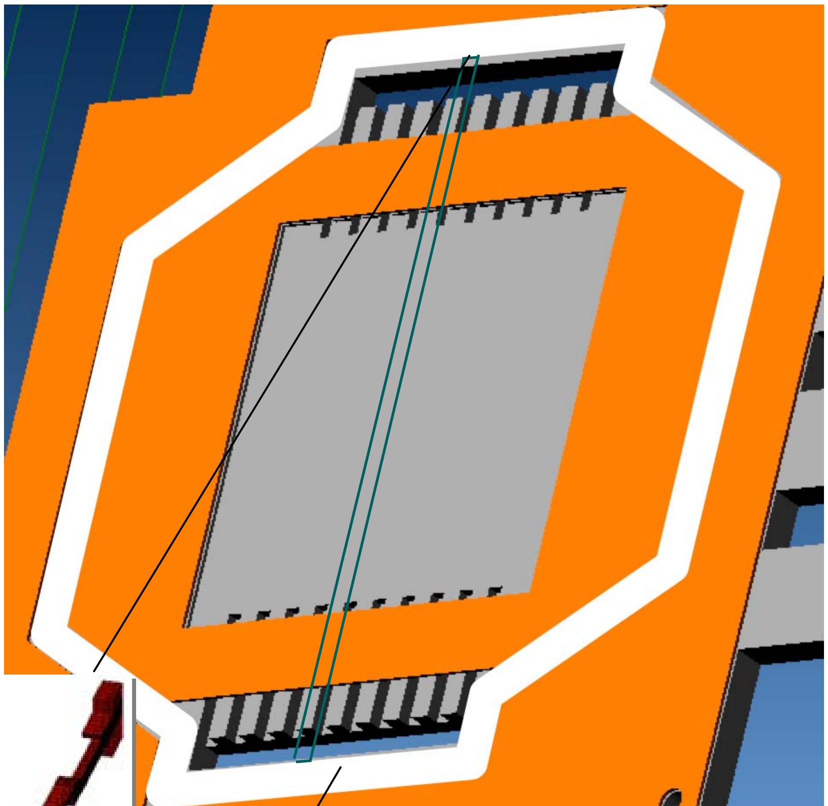
Changes in alloy availability

- Current design is based on 0.175” and 0.060” thick 441 sheet
  - 0.175” no longer available
- SECA program is acquiring large stock of 441 for use by SECA participants
  - 0.133”, 0.040", 0.020", and 0.010" sheet
- Developing new fixture design consistent with available sheet thicknesses
  - Lower mass design – closer replica of real stacks under development
  - Simpler component shapes (less machining, lower fabrication cost)
  - Multi-cell stack capability (3-cell stack tests planned)

Scale Drawing of Revised Design: 3 Cell Stack

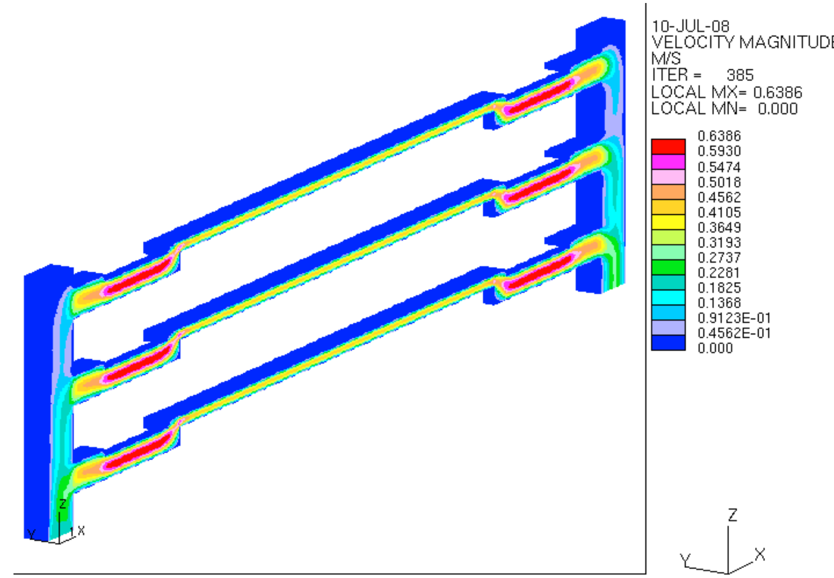


Modeling of Flow Distribution in 3-cell Stack



Small ΔP in manifolds (slots)

Larger ΔP over cell



- Similar oxidant and fuel flows through three cells
- Flow distribution summary:

Percent of Mean Flow		
	Fuel	Air
Cell 3	99.7	99.72
Cell 2	99.93	99.92
Cell 1	100.37	100.36

- Flow on both anode and cathode sides within +- 0.4% of mean

Future Work

- **Design**
  - Transition to revised, reduced-mass stack fixture
    - Single cell tests
    - Multi-cell tests
  - Continue to improve performance and reproducibility
    - Issues related to simultaneous fabrication of stack seals & contact materials
    - Minimize "hand-crafted" aspects; replace with "automated" processes
  - Transfer stack test capability to NETL
- **Implementation**
  - Complete initial evaluation of interconnect materials system: 441 steel with Ce-modified MnCo spinel and alumina coatings
  - Continue to evaluate/validate new materials/processes/design concepts. Candidates include:
    - Contact materials
      - Novel processing approaches (atmospheric cycling)
      - Novel materials (sintering aids, reaction sintering approaches)
      - Examine role of high temperature sealing on contact structure and load distribution (validation of modeling results)
    - New cathode materials
    - New interconnect alloys/coatings
    - Commercial cells

Conclusions/Accomplishments

- A stack test fixture based on 50mm x 50mm cells (40mm x 40mm cathode) has been developed for evaluation/validation of new materials, fabrication processes, and design concepts.
- Implementation:
  - Performance of refractory glass seals has been validated.
    - Seals exhibited excellent performance during 25 deep thermal cycles
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