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 950-1000°C
    - 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/°C; 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)

![Graph showing ohm-m as a function of hours at elevated temperatures]

YSO75 @0.7V & 20%H2O, 2.7%H2/Ar (t<432hrs), 80%H2 (t>432hrs) power outage @ ~700 h
Cell with refractory glass seal demonstrated stable OCV during 25 deep thermal cycles (RT – 750°C)
Cell-to-frame glass seal remained intact (no dye penetration)

Refractory glass selected as baseline seal composition for stack fixture testing
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

- Anode plate: 441 Steel
- Cathode plate: 441 Steel w/ Dual Coatings
- 441 Frame
- Nickel Mesh
- LSM-20 Contact Paste
- Nickel Oxide Contact Paste
- Cell-to-Frame Seal: Refractory Glass
- Perimeter Seals
- Aluminization
- Ce-MC Spinel
- Pacific Northwest National Laboratory
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 800°C 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

- Aluminized SS441 frame / glass seal after 404h @ 800°C
- 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

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

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

<table>
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<th>Percent of Mean Flow</th>
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<th>Air</th>
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<td>99.7</td>
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<td>Cell 1</td>
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- Flow on both anode and cathode sides within +- 0.4% of mean
- Small $\Delta P$ in manifolds (slots)
- Larger $\Delta P$ over cell
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
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Acknowledgements

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