# SOFC test fixture development and materials evaluation at PNNL

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# Outline

- Conclusions
- Objectives
- Test Fixture: Materials, Design, and Assembly
- Cell Testing Results and Materials Characterization
- Future Work
- Conclusions
- Acknowledgements

# Conclusions/Accomplishments

- Two stack test vehicles based on 50mm x 50mm cells (40mm x 40mm cathode) have been developed for evaluation/validation of new materials, fabrication processes, design concepts.
- Candidate materials have been validated in a single cell testing for ~2300 h:

1. Refractory sealing glass showed hermeticity, minimal reaction with YSZ and aluminized SS441.
2. (Mn,Co)-spinel coating was effective in blocking Cr at cathode IC.
3. Aluminization was able to minimize (Ba,Sr)-chromate formation.

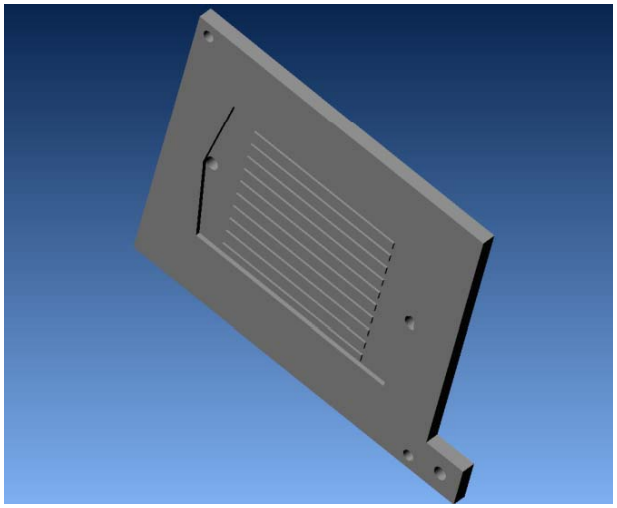
- A novel double-seal concept was developed and demonstrated successfully for 25 deep thermal cycles.
- Developing revised fixture design for multiple cells testing.
- Transferring test capability to NETL.

# Objectives

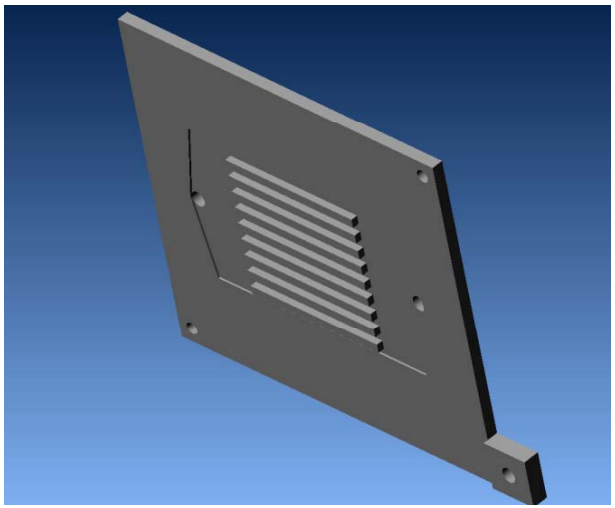
- To develop a SOFC cell/stack test fixture on behalf of Core Technology Program in order to evaluate/validate new materials and fabrication processes under realistic stack conditions.
- To bridge the gap between small-scale tests (e.g., button cells) and SECA industry team stacks.
- To share fixture designs with other SECA participants.

# Test fixture: 1st generation design (co-flow or counter-flow)

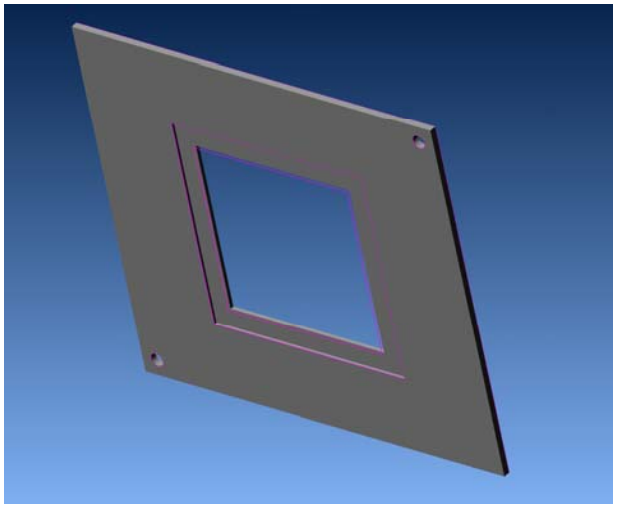
1. Cell (Stark ASC-3) anode-supported YSZ cell 2”x2” with LSM cathode
2. LSM-20 (Praxair) and NiO (Baker) contact paste
3. Refractory glass seal for PEN/WF seal (950$^{o}$C/2h)
4. Perimeter seal in double seal geometry
5. (Mn,Co)-spinel coating for cathode IC plate
6. Aluminization for sealing area
7. SS441 for IC (0.170”) and window frame (0.064”) plates



SS441 anode IC plate

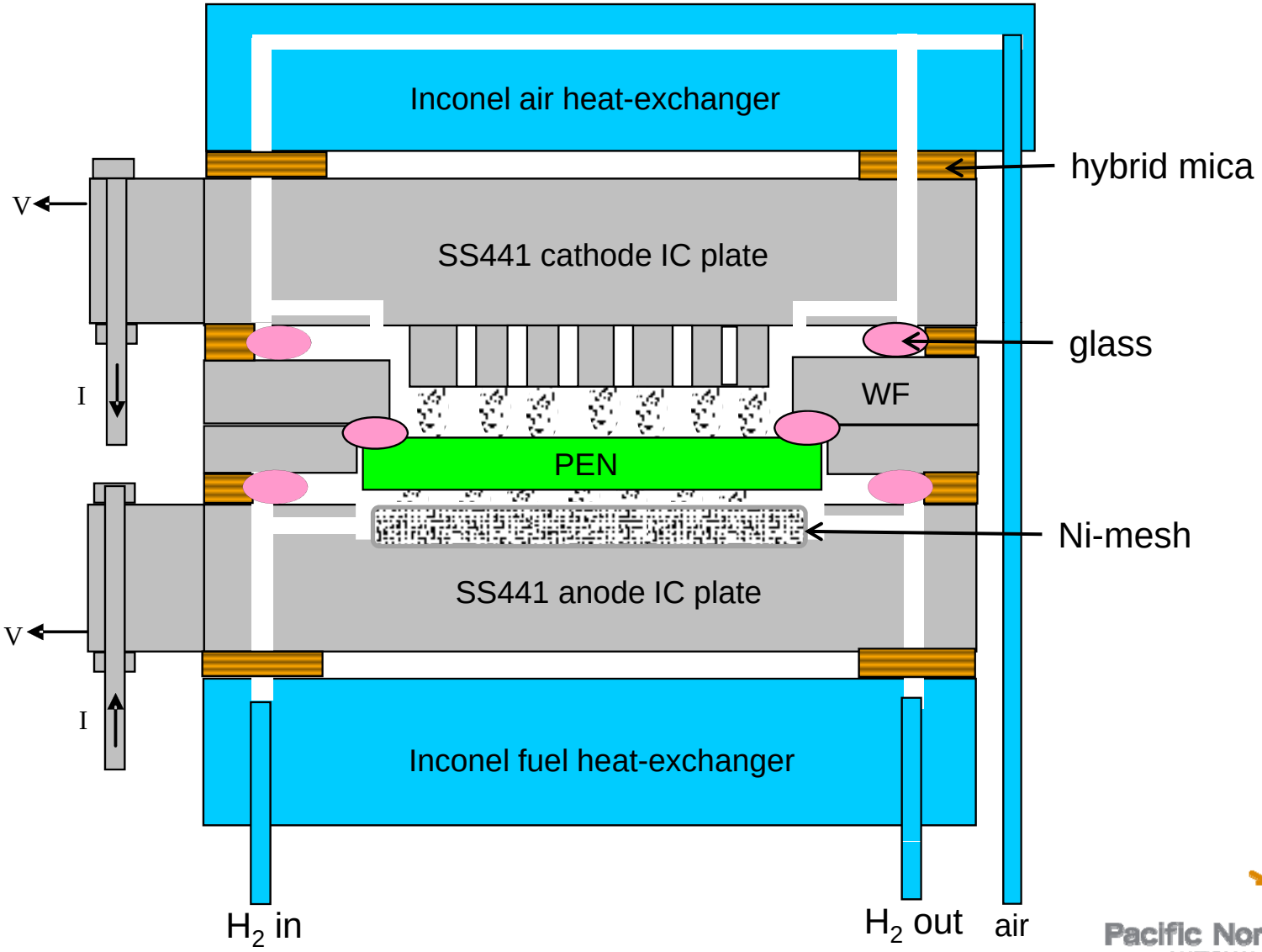


SS441 cathode IC plate



SS441 window frame

# Seal system in cell test



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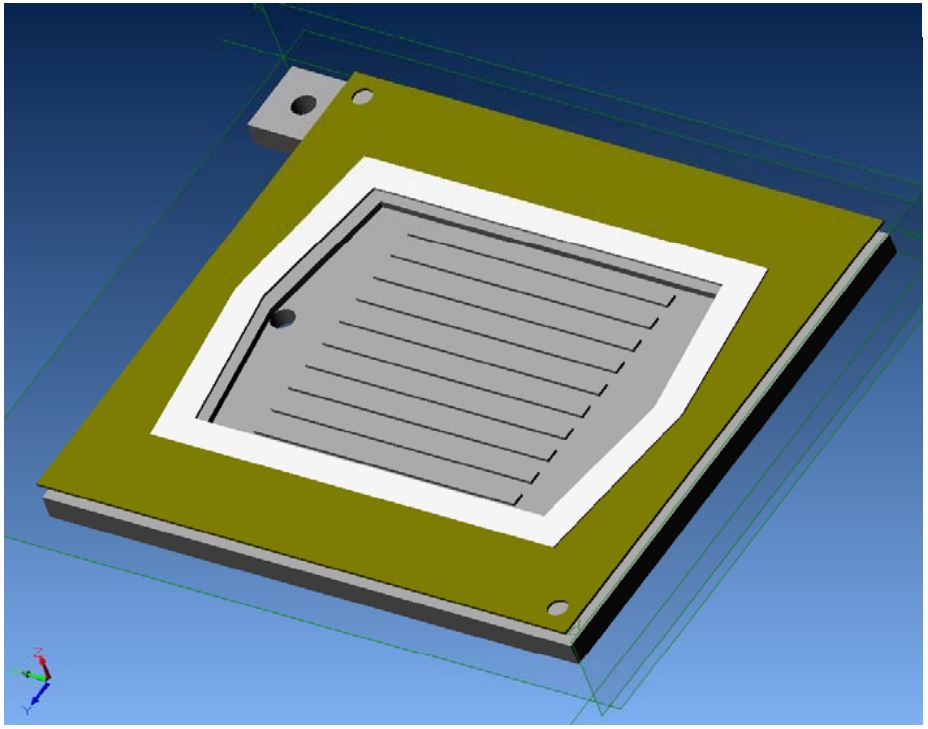
# Double seal (mica + glass) concept

1. Glass will creep/flow under stress and lead to large dimensional change.
2. Hybrid mica was proven stable over 28366 hr @ 800$^{o}$C.
3. The creep/flow of glass may transfer compressive load to fracture PEN/WF seal.



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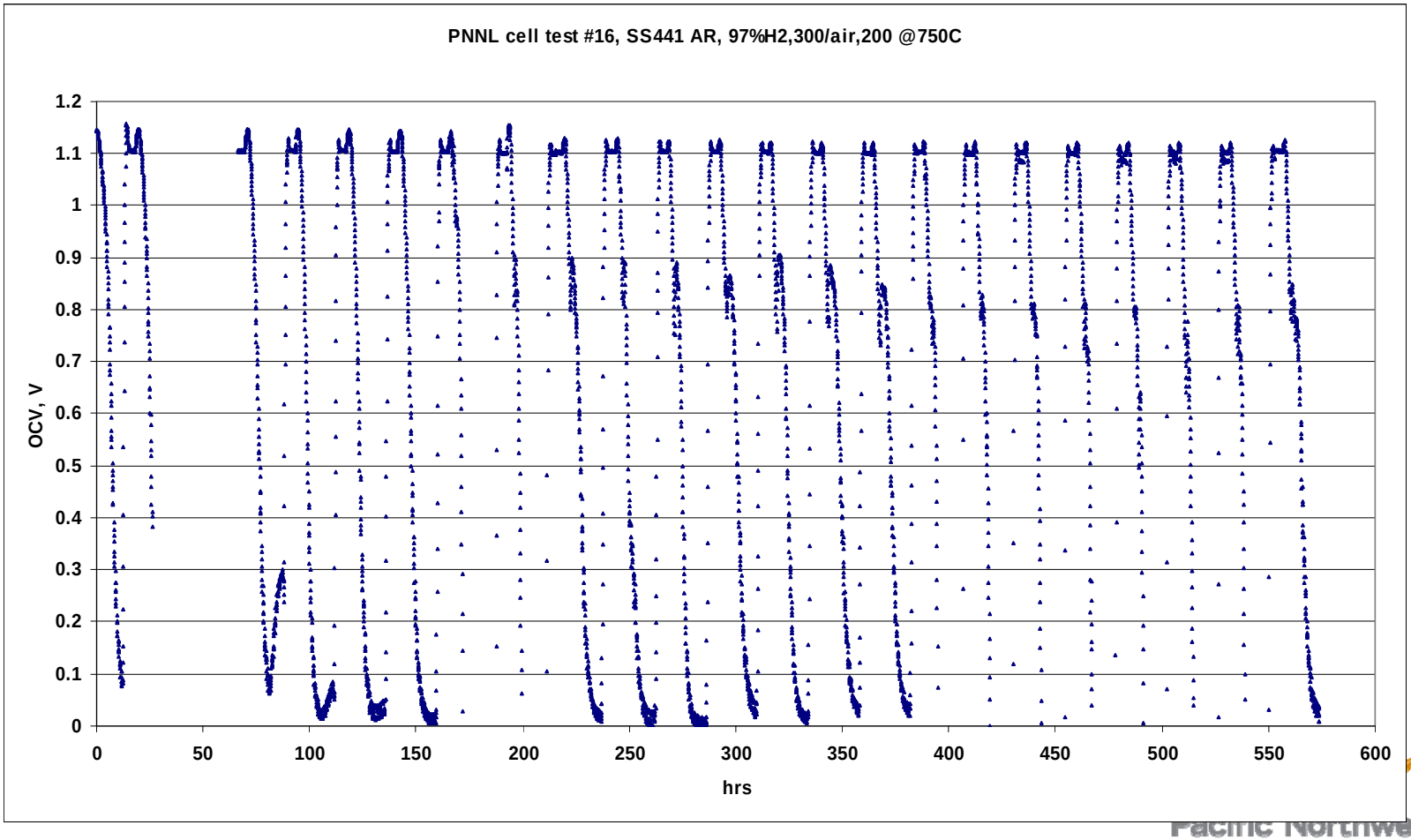
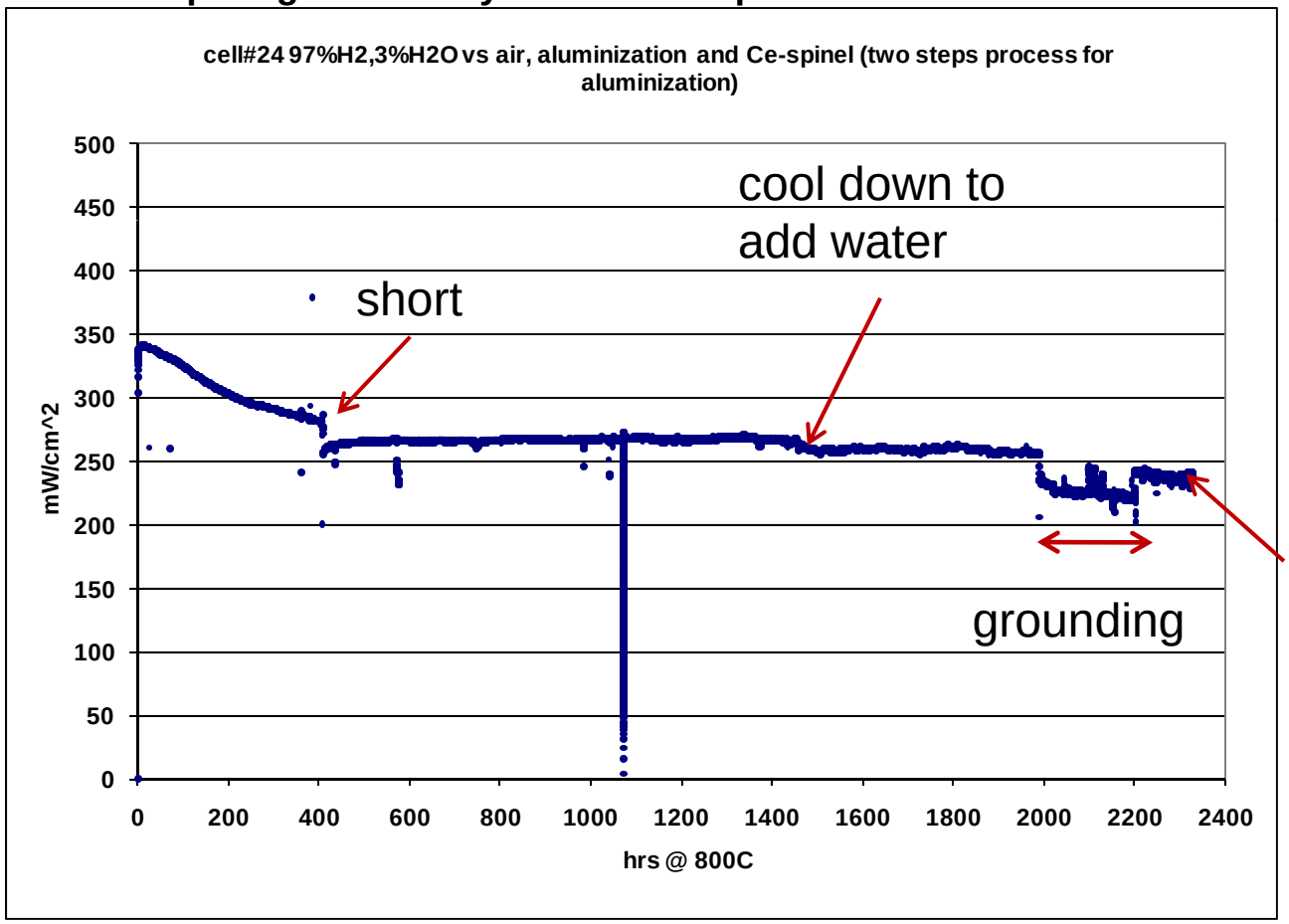
# Cell #16: refractory glass seal showed good thermal cycle stability

25 deep cycles with constant OCV of ~1.10 V @ 800°C with, 97%$H_2$+3%$H_2O$ vs. air
RT to 750°C in 3 hr, 750°C/3h, furnace cool to ~RT in18hr



# Cell test #24: candidate materials validation at constant voltage

- 1st generation V3 design with (Mn,Co)-spinel and aluminization using thick SS441 plates (0.170" and 0.064")
- Standard LSM and NiO/Ni mesh for contact
- Double seal concept of glass and hybrid mica for perimeter seal




OCV=1.0V @2325hr large data fluctuation Inconel tubing leak

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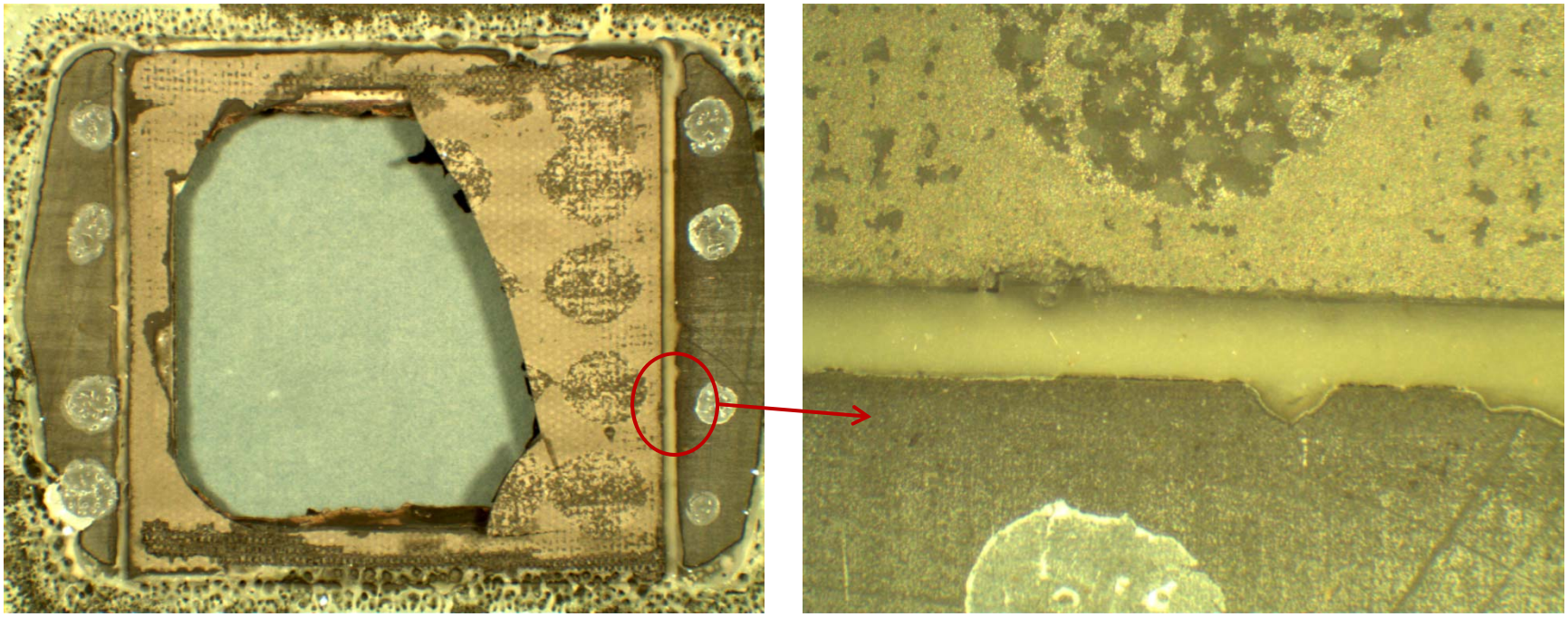
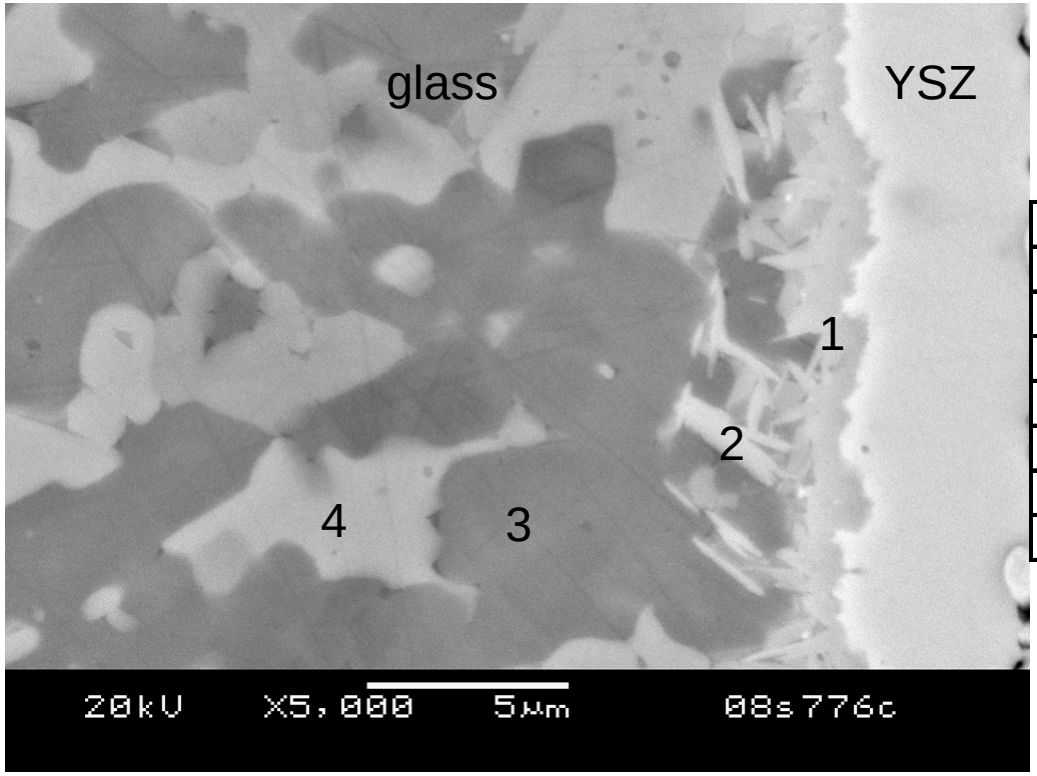
# Post-test analysis of cell #24, optical

PEN plate fractured during dissembling, indicating strong bonding of Ni contact paste. Glass seal remained whitish indicating good seal and minimum reaction with Interconnect due to aluminization.



# Cell #24: sealing glass characterization at glass/YSZ interface

Minimal reaction of refractory glass with YSZ after ~2300hr @800ºC near fuel side
Needle formation along interface ($BaZrO_3$, CTE ~10)




| at% | #1 | #2 | #3 | #4 |
|---|---|---|---|---|
| O | 52.64 | 48.73 | 38.18 | 47.33 |
| Si | 0.00 | 12.06 | 30.49 | 5.19 |
| Sr | 1.46 | 13.00 | 28.43 | 25.38 |
| Zr | 24.09 | 11.86 | 0.00 | 0.00 |
| Ba | 18.44 | 14.35 | 2.90 | 5.56 |
| Y | 0.00 | 0.00 | 0.00 | 16.53 |
| Ni | 3.38 | 0.00 | 0.00 | 0.00 |



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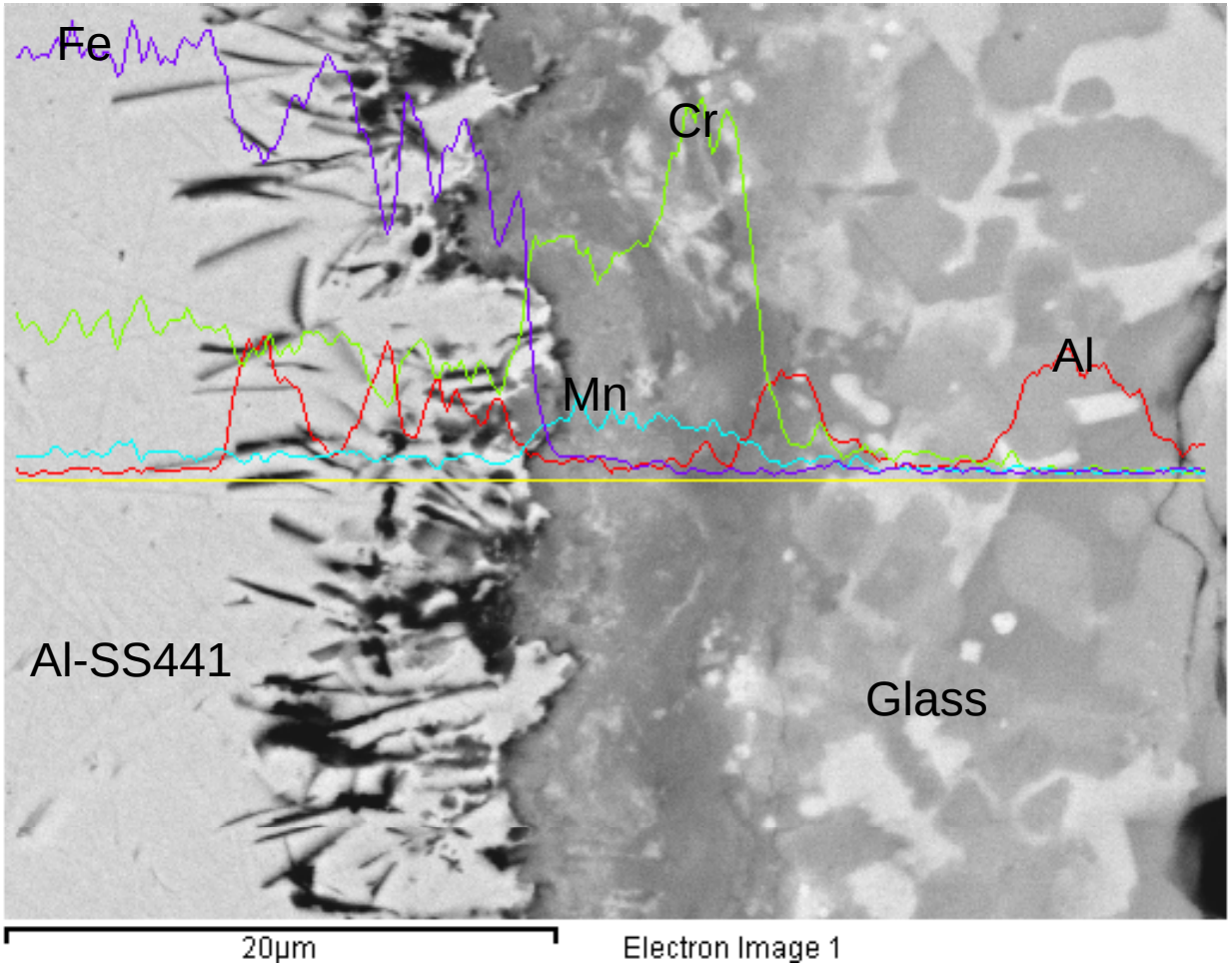
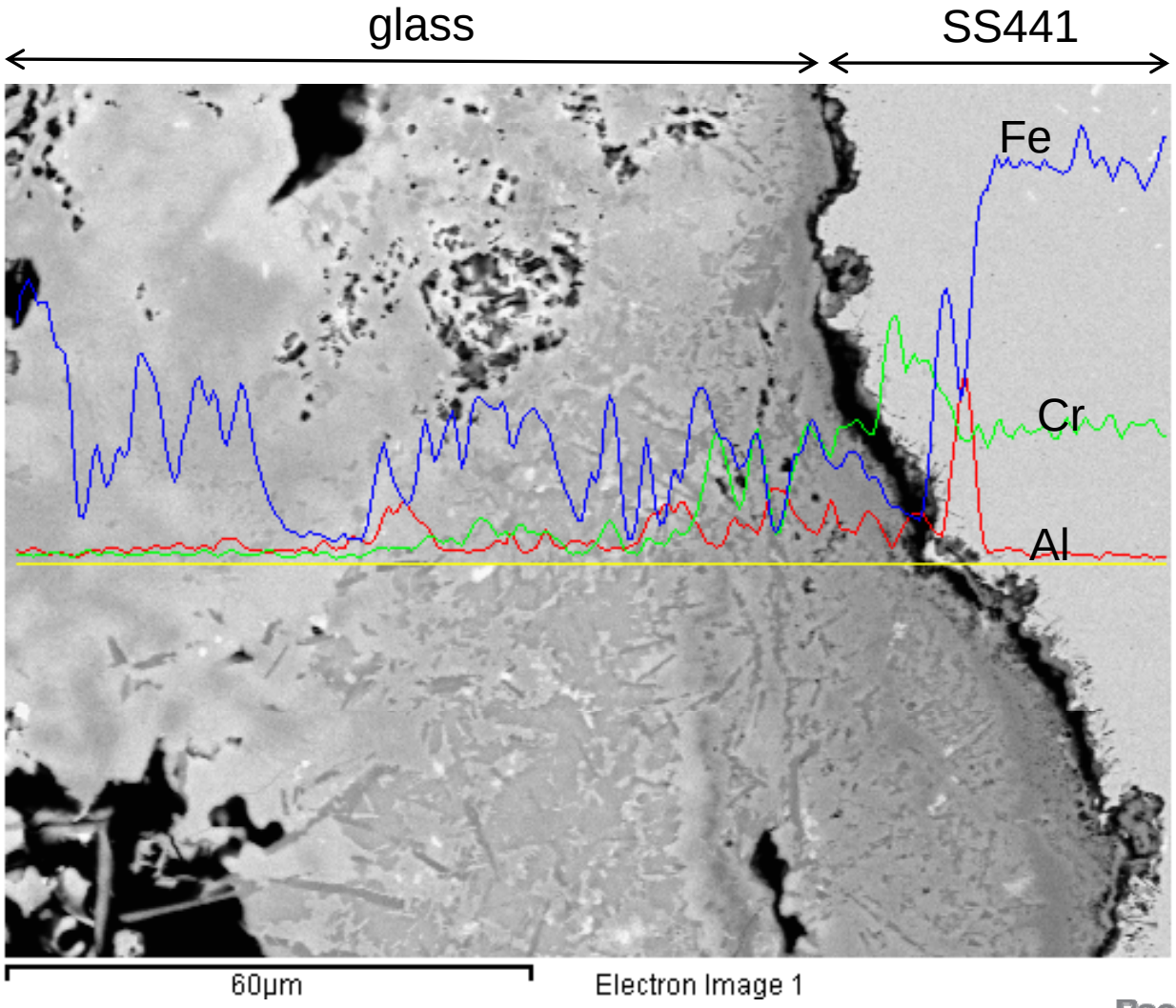
# Cell #24: glass/aluminized SS441 interface of fuel side

No (Ba,Sr)-chromate formation at fuel side; however, alumina appeared to be leached into glass matrix.



# Cell #24: glass/aluminized SS441 interface of air side



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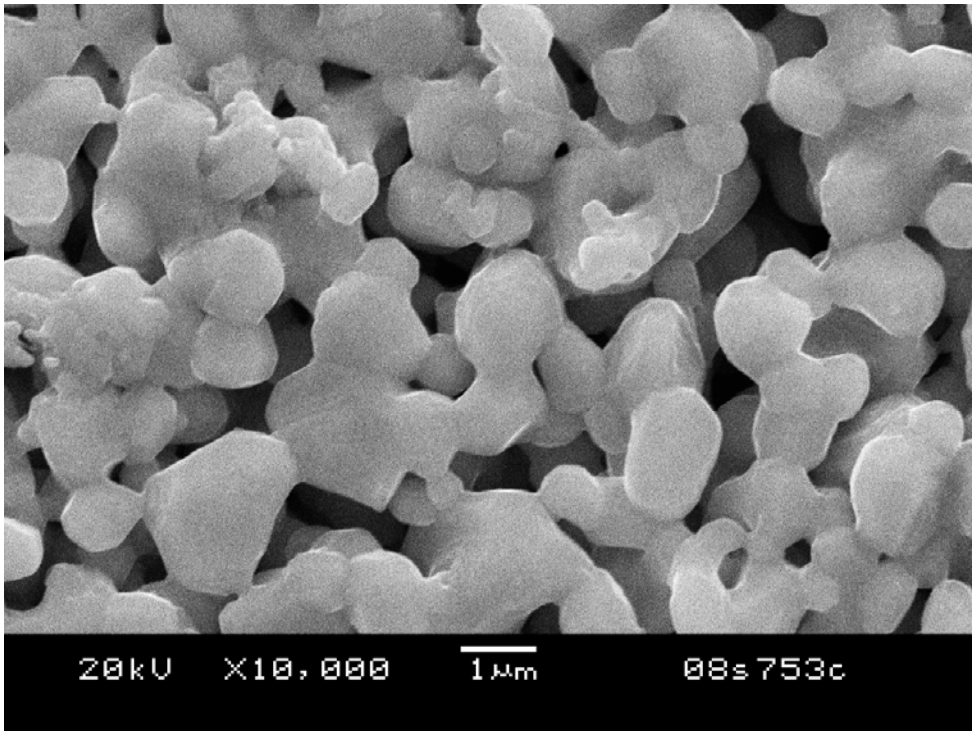
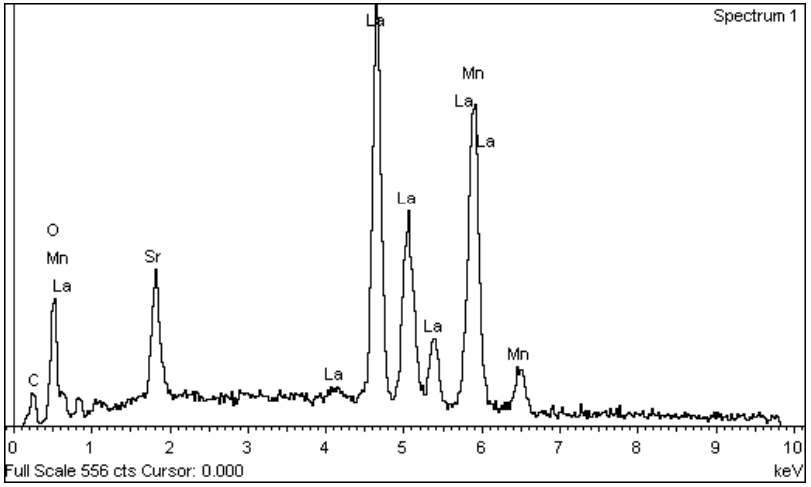
# Post-test analysis of cell #24, cathode top surface




| Element | Weight% | Weight% Sigma |
|---|---|---|
| O K | 8.57 | 0.53 |
| Mn K | 27.69 | 0.6 |
| Sr L | 8.85 | 0.61 |
| La L | 54.89 | 0.75 |

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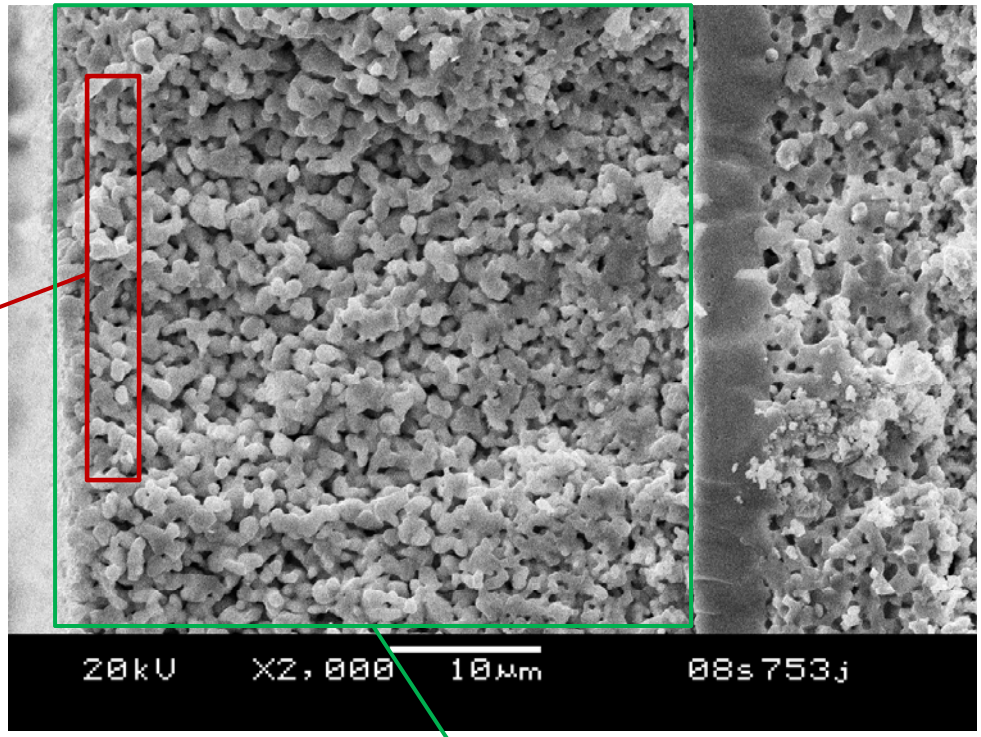
# Cell #24: characterization of Cr near YSZ electrolyte

| Element | Weight% | Weight% Sigma |
|---|---|---|
| O K | 8.73 | 0.52 |
| Mn K | 27.88 | 0.58 |
| Sr L | 9.07 | 0.58 |
| Zr L | 0.0 | 0.0 |
| La L | 54.32 | 0.72 |




| Element | Weight% | Weight% Sigma |
|---|---|---|
| O K | 9.52 | 0.64 |
| Mn K | 23.83 | 0.61 |
| Sr L | 7.17 | 0.64 |
| Zr L | 12.98 | 0.85 |
| La L | 46.51 | 0.87 |

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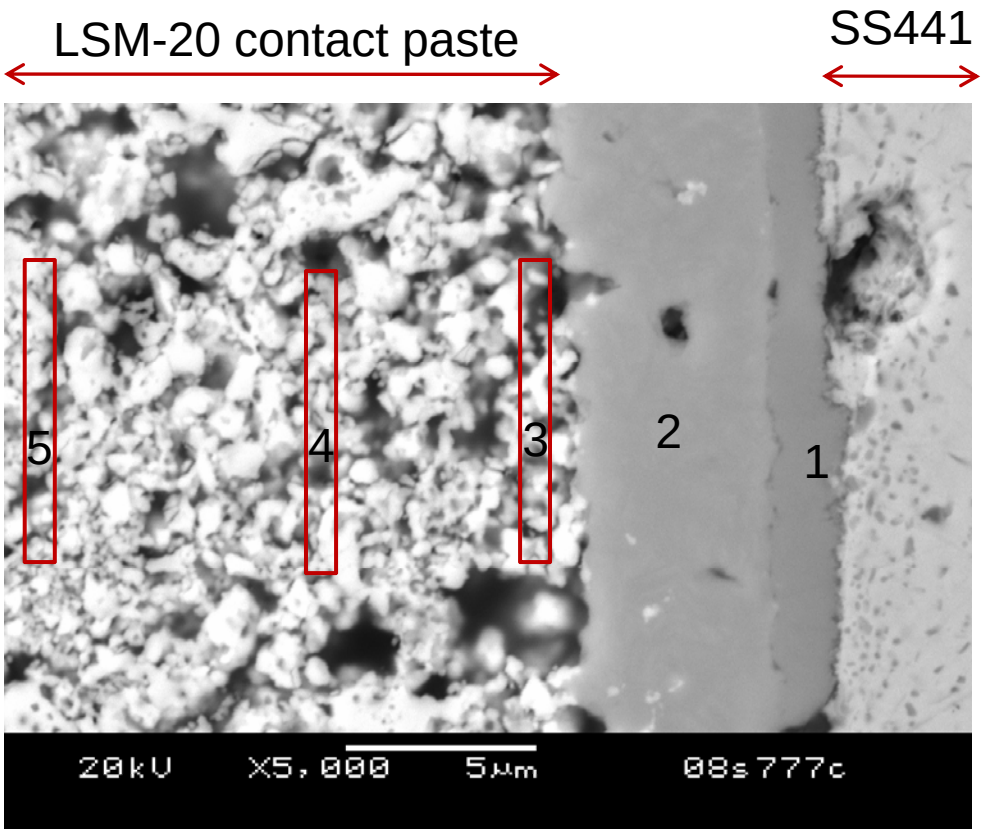
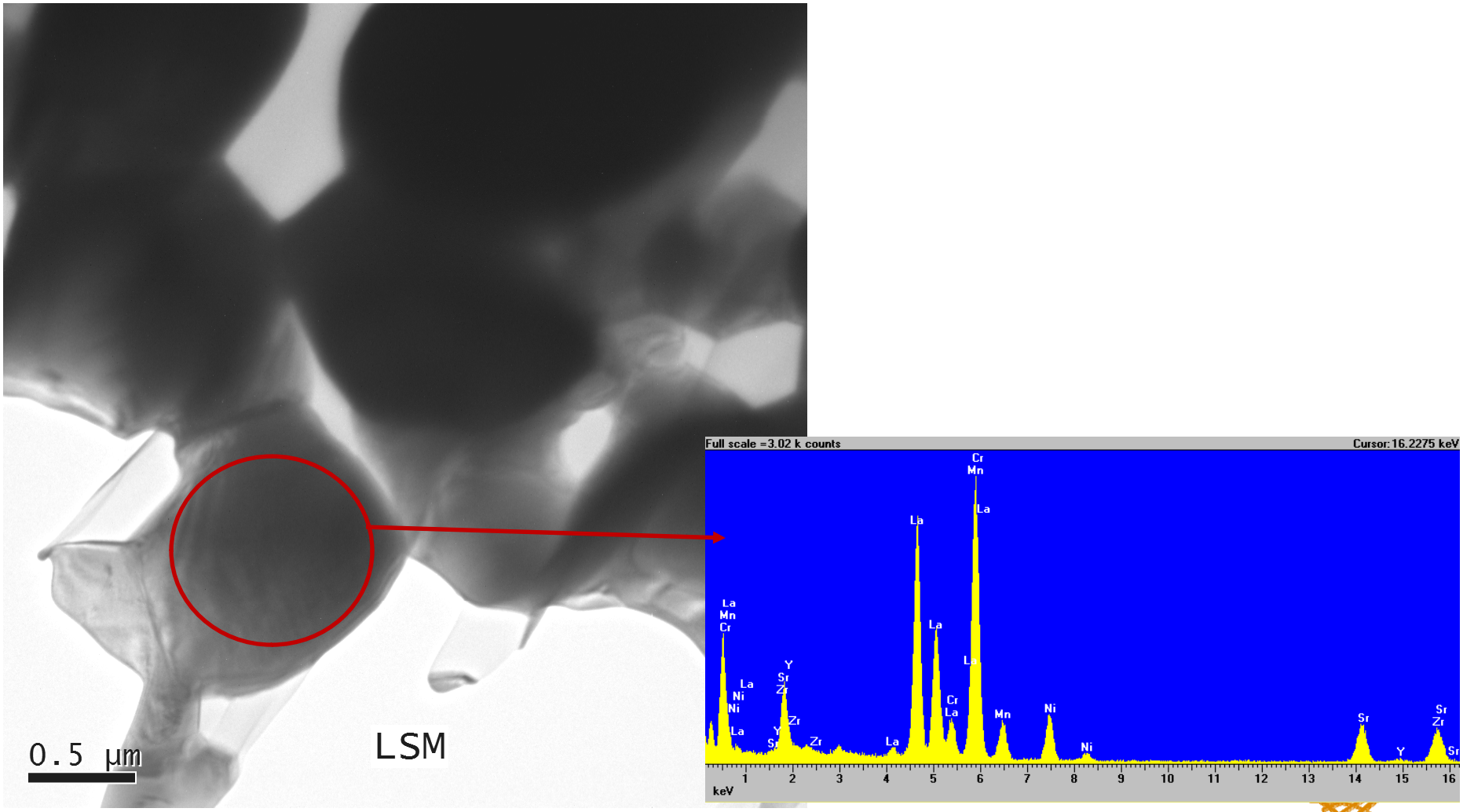
# Cell #24: characterization of Cr near (Mn,Co)-spinel coated SS441




| at% | #1 | #2 | #3 | #4 | #5 |
|---|---|---|---|---|---|
| O | 37.96 | 33.65 | 30.72 | 34.11 | 32.31 |
| Si | 0.68 | 0.00 | 0.00 | 0.00 | 0.00 |
| Ti | 0.63 | 0.50 | 0.00 | 0.00 | 0.00 |
| Cr | 56.77 | 0.87 | 0.86 | 0.00 | 0.00 |
| Mn | 1.02 | 39.35 | 30.89 | 32.77 | 33.73 |
| Fe | 2.94 | 0.00 | 0.00 | 0.00 | 0.00 |
| Co | 0.00 | 25.62 | 5.06 | 0.00 | 0.00 |
| Sr | 0.00 | 0.00 | 6.46 | 6.05 | 6.65 |
| La | 0.00 | 0.00 | 26.02 | 27.07 | 27.32 |

# Cell #24: LSM grain characterization



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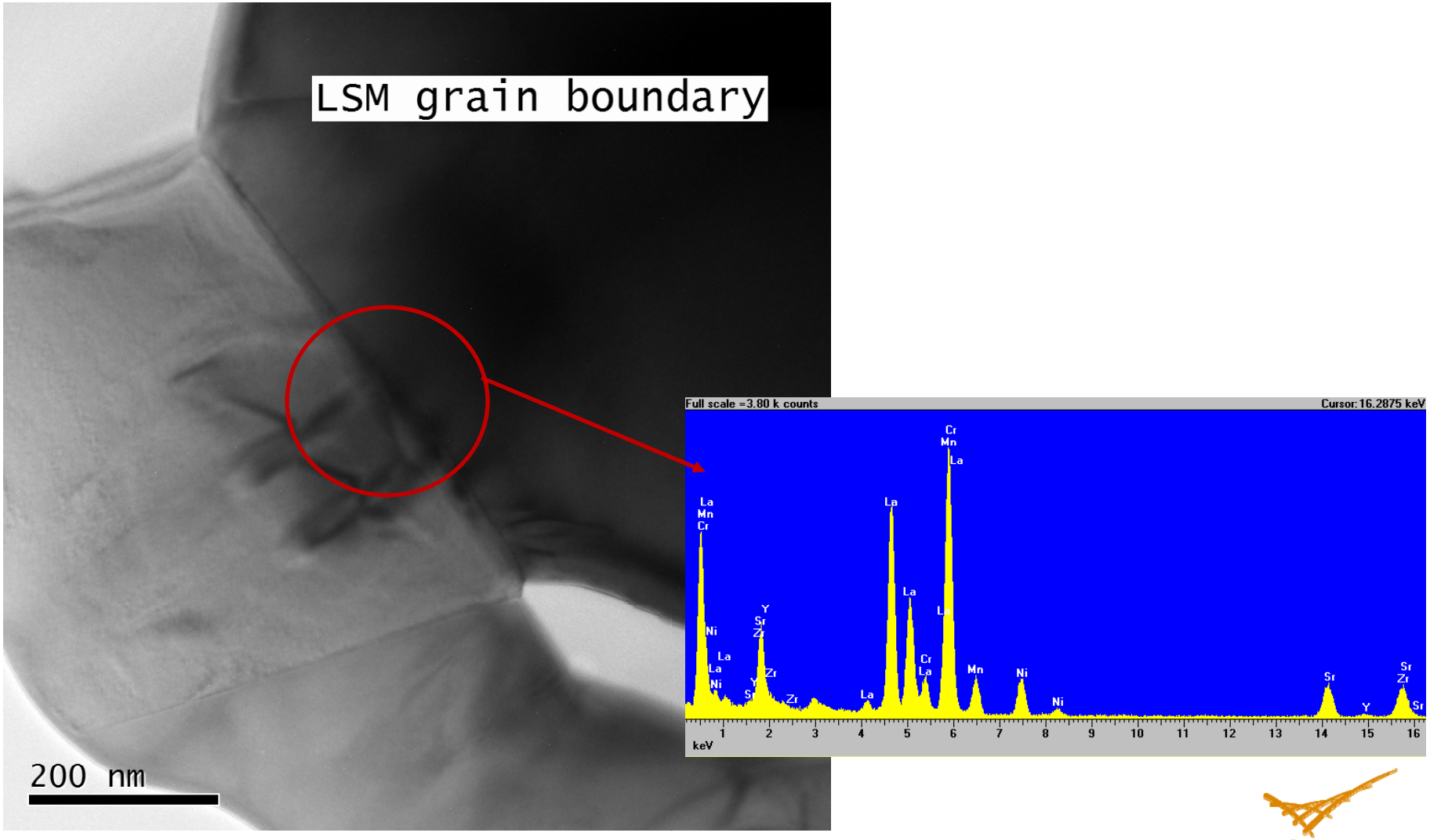
# Cell #24: LSM grain boundary characterization

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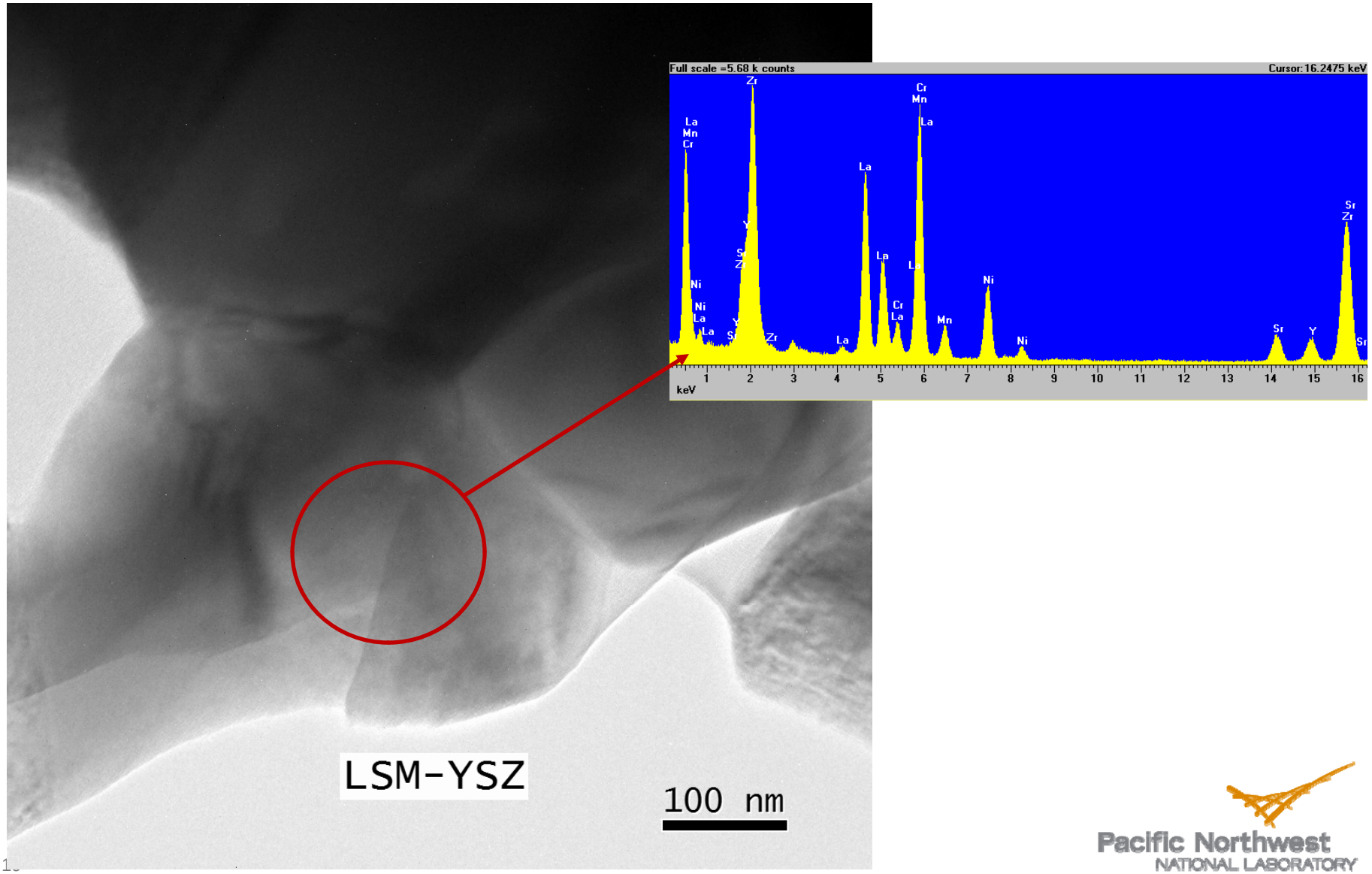
# Cell #24: LSM/YSZ grain boundary

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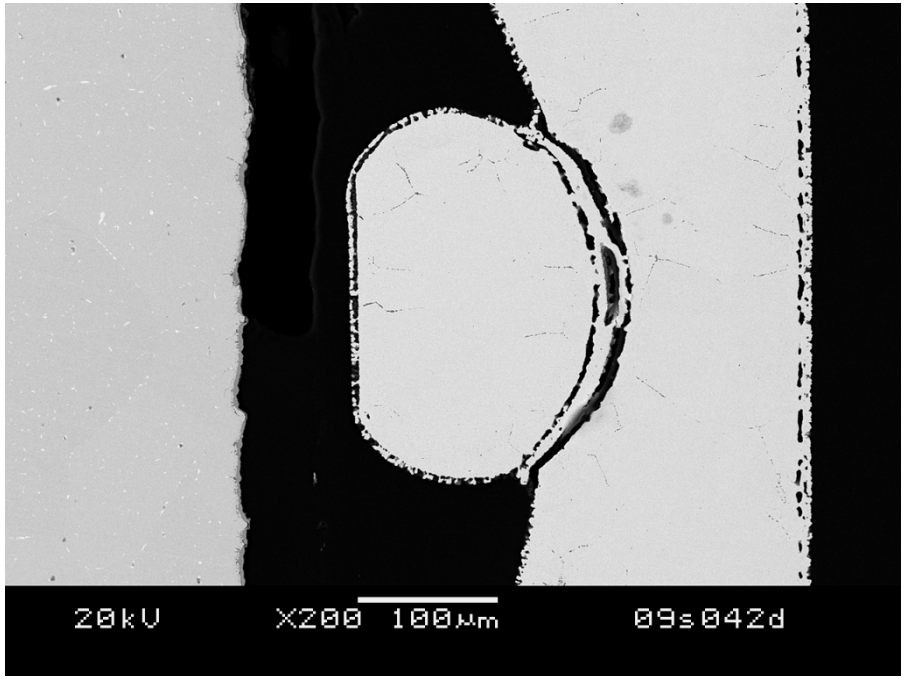
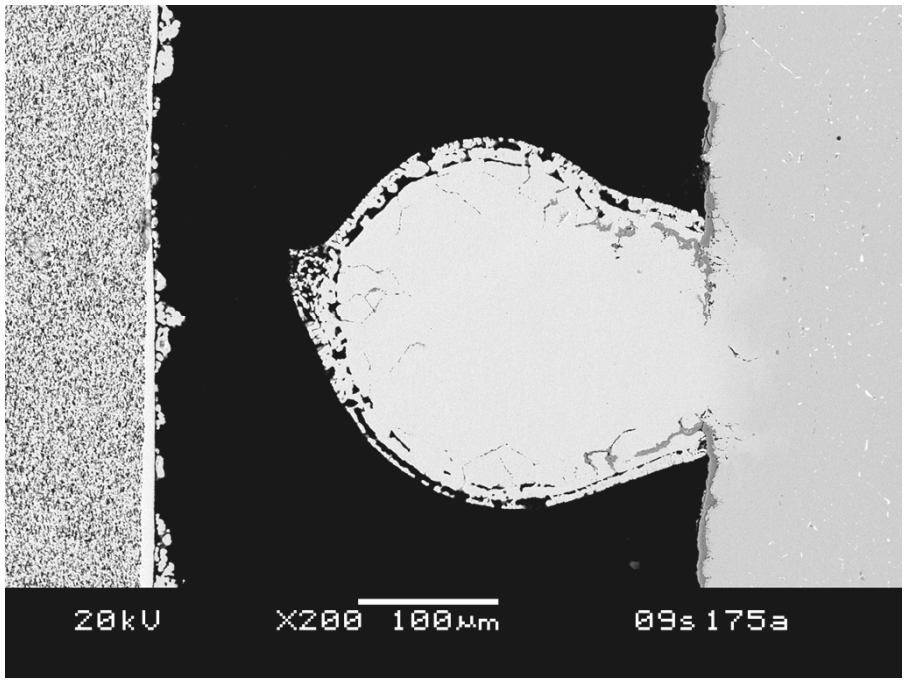
# Cell #24: Ni characterization

1. Cell #24 tested at 800°C for 2325 hr @0.7V with 97%$H_2$,3%$H_2O$ vs. air
2. Previous SS441 oxidation characterization focused on non-welded section




non-welded section




welded section

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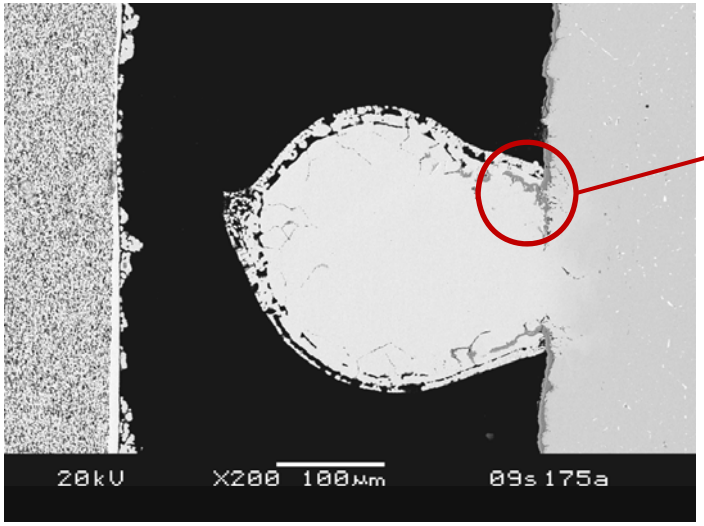
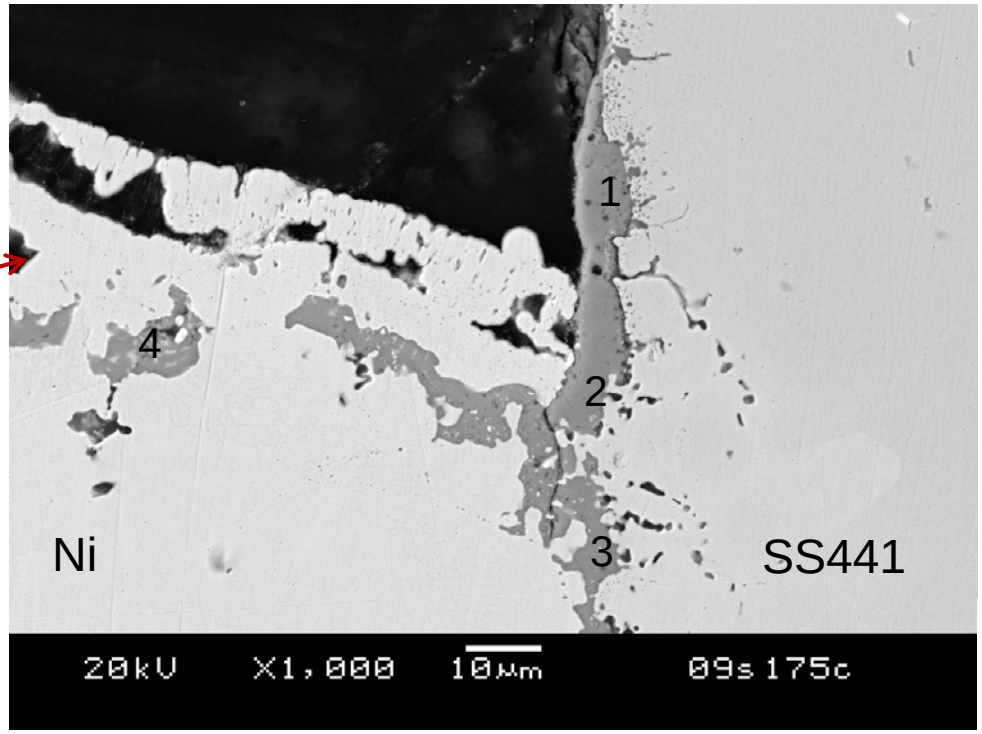
# Cell #24: Ni characterization




| at% | #1 | #2 | #3 | #4 |
|---|---|---|---|---|
| O K | 36.9 | 35.6 | 38.2 | 46.0 |
| Ti K | 1.5 | 1.8 | 2.7 | 0.9 |
| Cr K | 61.0 | 61.2 | 57.8 | 18.7 |
| Fe K | 0.5 | 0.6 | 0.0 | 0.0 |
| Ni K | 0.0 | 0.9 | 0.7 | 2.6 |
| Nb L | 0.0 | 0.0 | 0.8 | 17.3 |
| Mn K | 0.0 | 0.0 | 0.0 | 14.5 |



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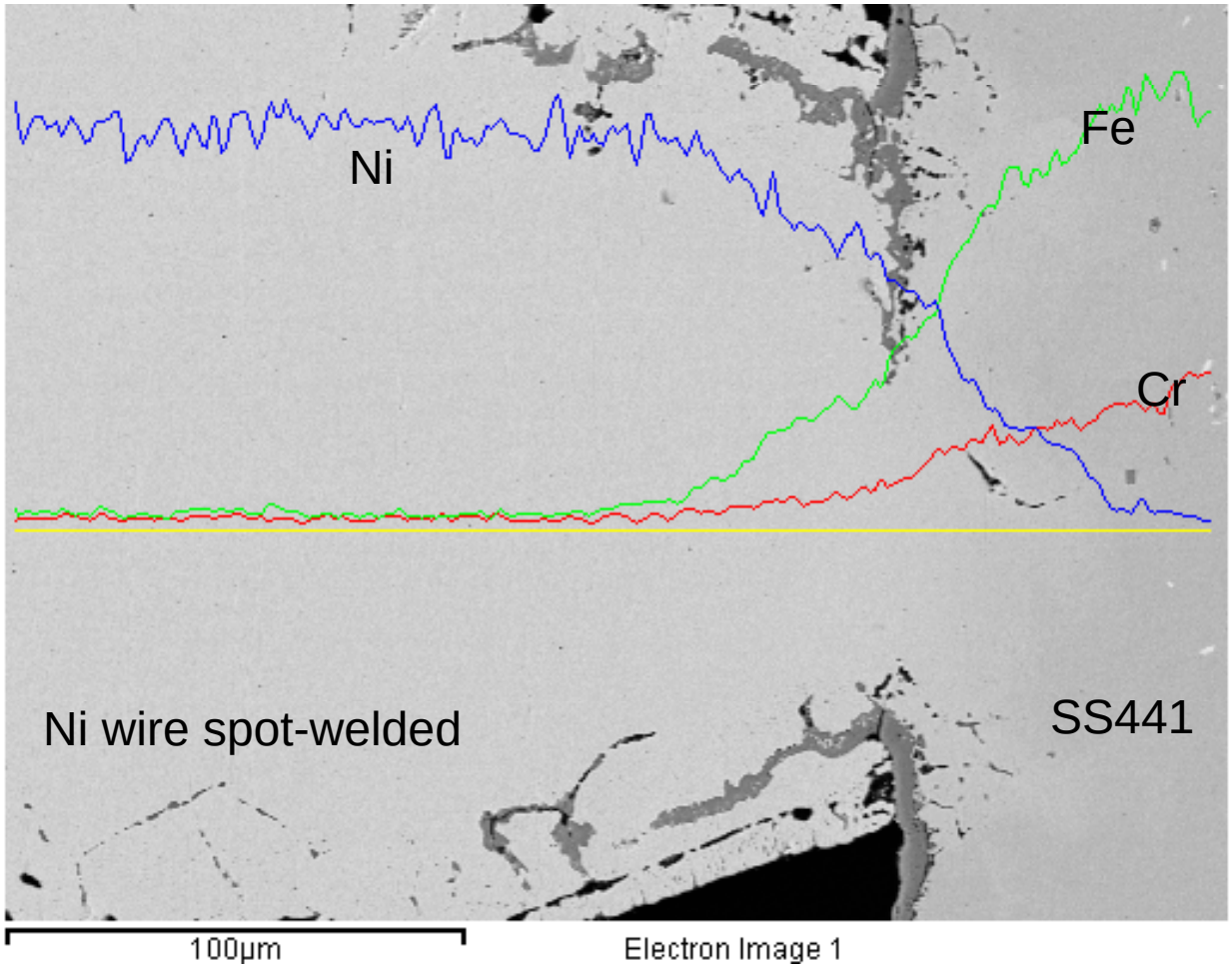
# Cell #24: Ni/SS441 interface characterization

800°C/~2325h



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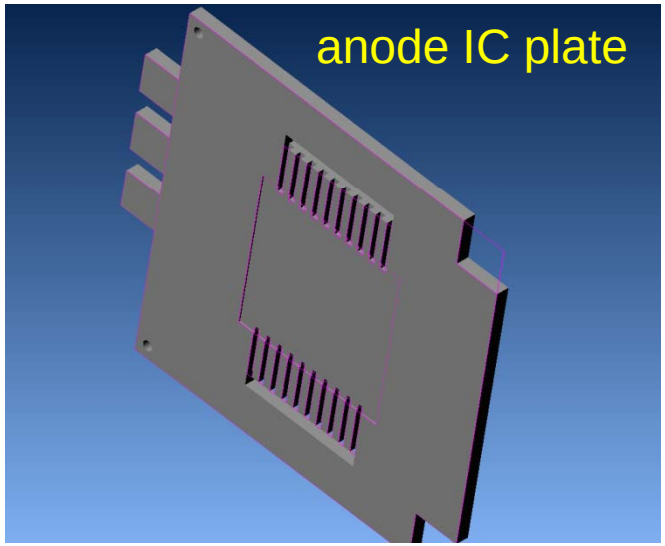
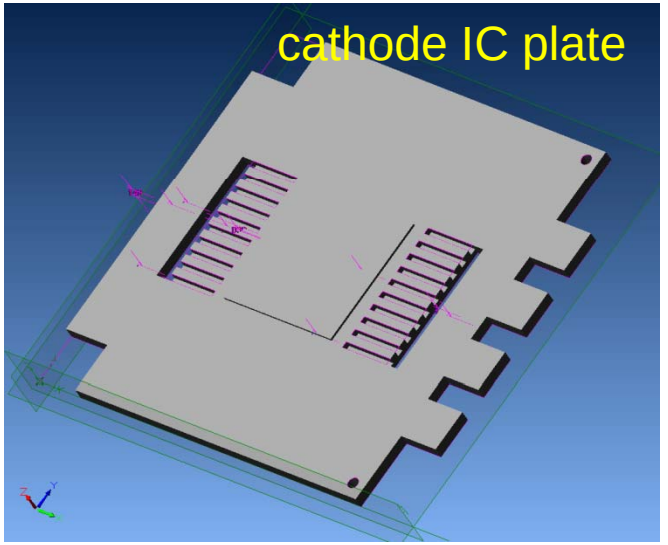
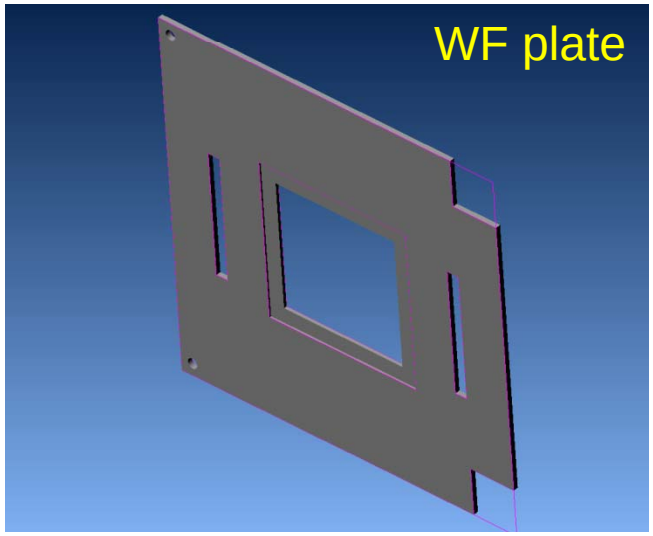
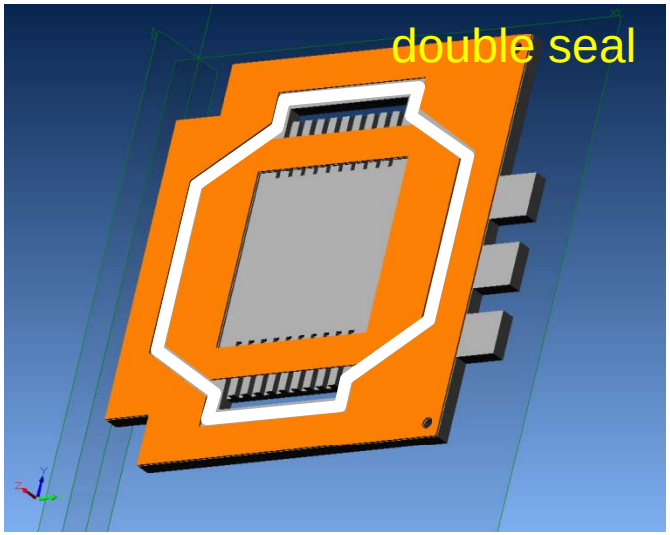
# Test fixture: 2nd generation design

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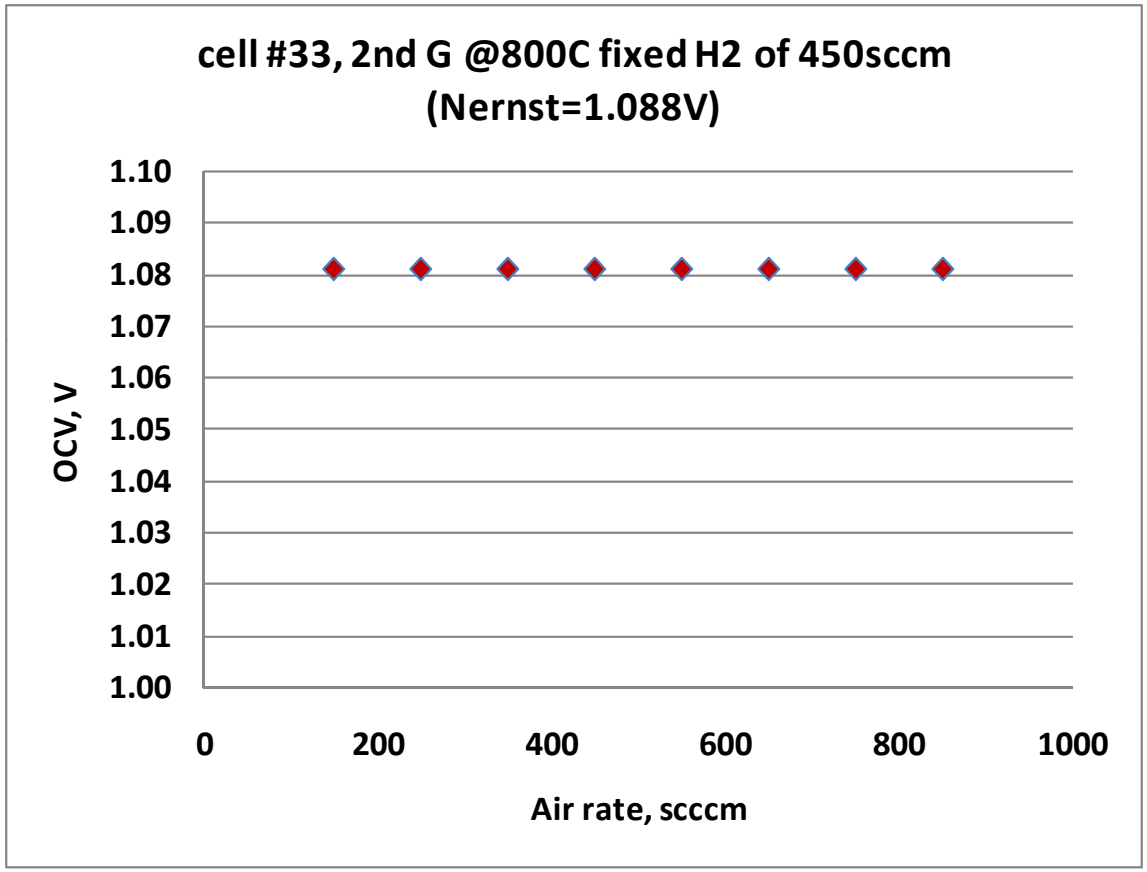
# Cell #33: 2nd G design seal test




No cross-bubbling is consistent with insensitivity of OCV with varying air flow rate, indicating hermetic PEN/WF glass seal.



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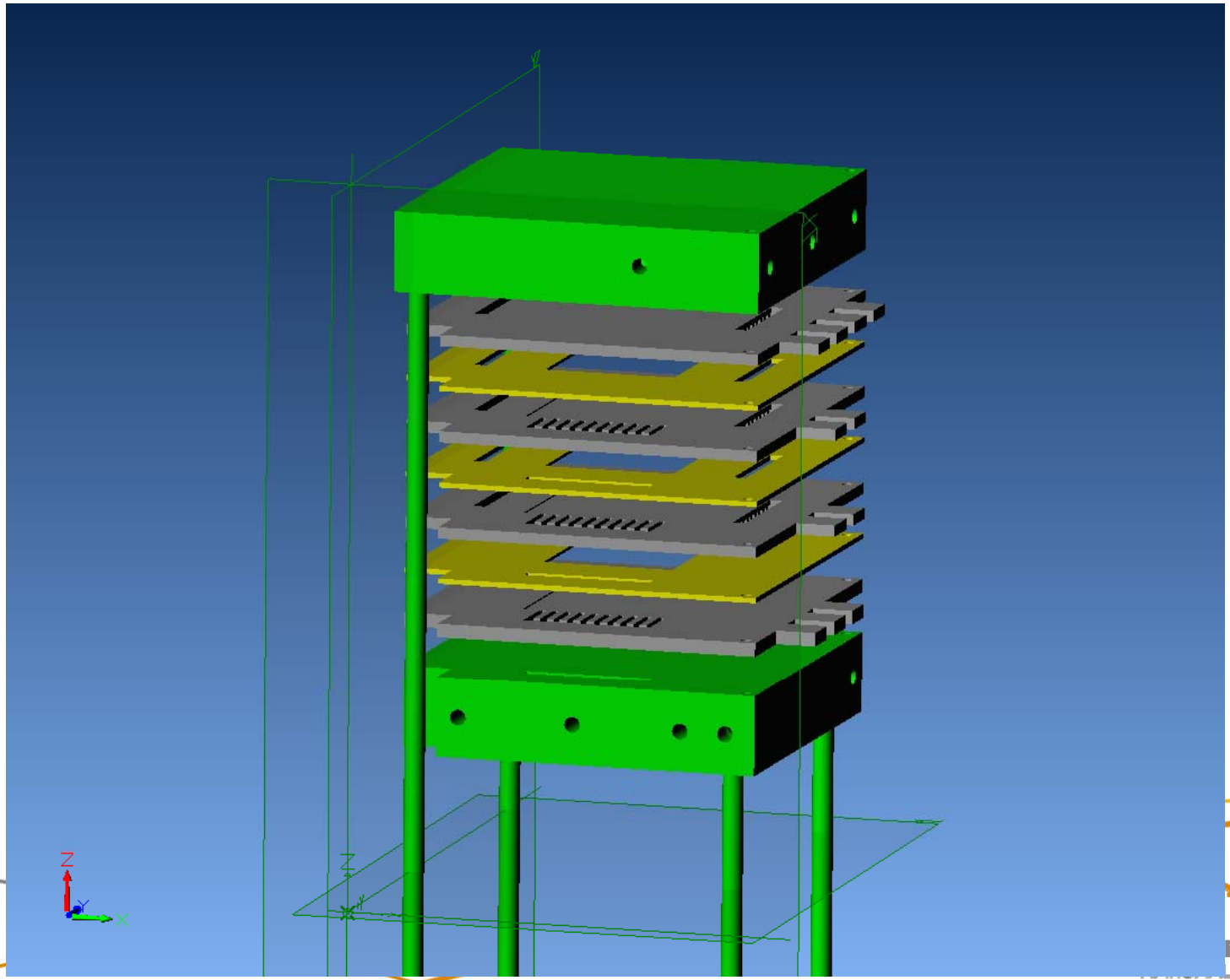
# 2nd G design for 3-cells stack test



# Conclusions/Accomplishments

- Two stack test vehicles based on 50mm x 50mm cells (40mm x 40mm cathode) have been developed for evaluation/validation of new materials, fabrication processes, design concepts.
- Candidate materials have been validated in a single cell testing for ~2300 h:

1. Refractory sealing glass showed hermeticity, minimal reaction with YSZ and aluminized SS441.
2. (Mn,Co)-spinel coating was effective in blocking Cr at cathode IC.
3. Aluminization was able to minimize (Ba,Sr)-chromate formation.

- A novel double-seal concept was developed and demonstrated successfully for 25 deep thermal cycles.
- Developing revised fixture design for multiple cells testing.
- Transferring test capability to NETL.

# Future work

- Candidate materials evaluation in a single cell test using 2nd G design with new SS441 thin stock materials and high moisture content fuel.
- Implement cathode reinforcement in single cell testing.
- Modify current 2nd G designs for short 3-cells stack evaluations.
- Collaboration with ORNL in evaluation of compliant sealing glass with current design.
- Transfer test fixture to NETL.
- Validating other novel materials and processing (e.g., infiltrated cathode) with current test fixture.

# Acknowledgements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- The authors wish to thank Wayne Surdoval, Briggs White, and Travis Schultz at the National Energy Technology Laboratory (NETL) for helpful discussions regarding the initiation and implementation of this study.
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- Additional PNNL contributors: Gary Maupin, Jared Templeton, Kerry Meinhardt, Jim Coleman, Shelley Carlson, Nat Saenz.