Novel and Improved Electrode Structures Through Infiltration

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Core Technology Program – Electrodes: Performance and Degradation

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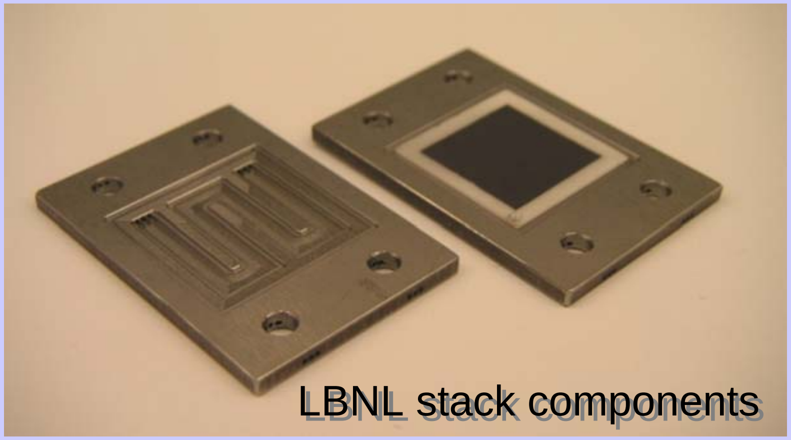
LBNL SECA Core Program

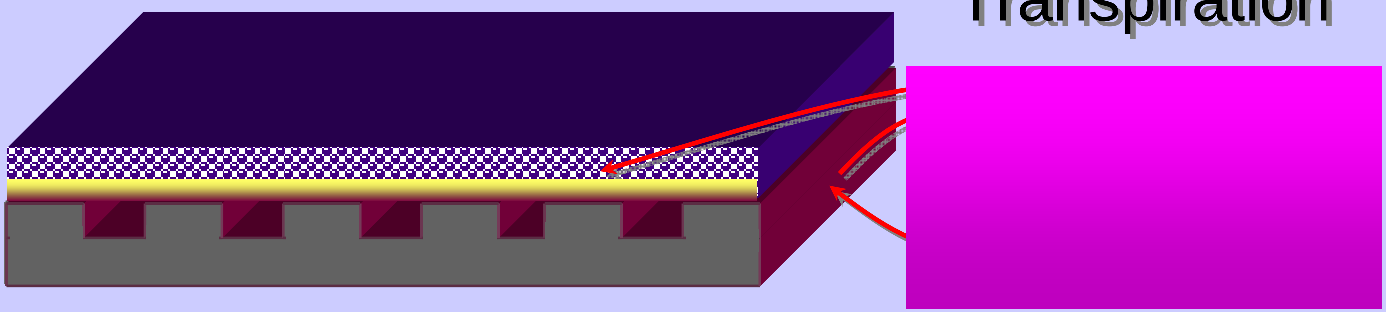
In FY07 the LBNL core effort was focused on the following areas:

1) Infiltration of perovskites and other appropriate catalysts into composite cathodes to form a interconnected network of nanoparticulate coating;
2) Infiltration of ceria and other appropriate materials into Ni-YSZ anodes to improve sulfur tolerance;
3) Determination of baseline performance and long term stability of infiltrated and non-infiltrated electrodes;
4) Design and fabrication of 2-cell stack for national labs and industrial teams as a standard platform for testing electrodes, interconnects, contact paste, and seals in a manner that allows reliable comparison across research teams;
5) Continued optimization of interconnect coating technology and elucidation of the mechanism of chromium migration through protective coatings.

Metal Stability & Interactions

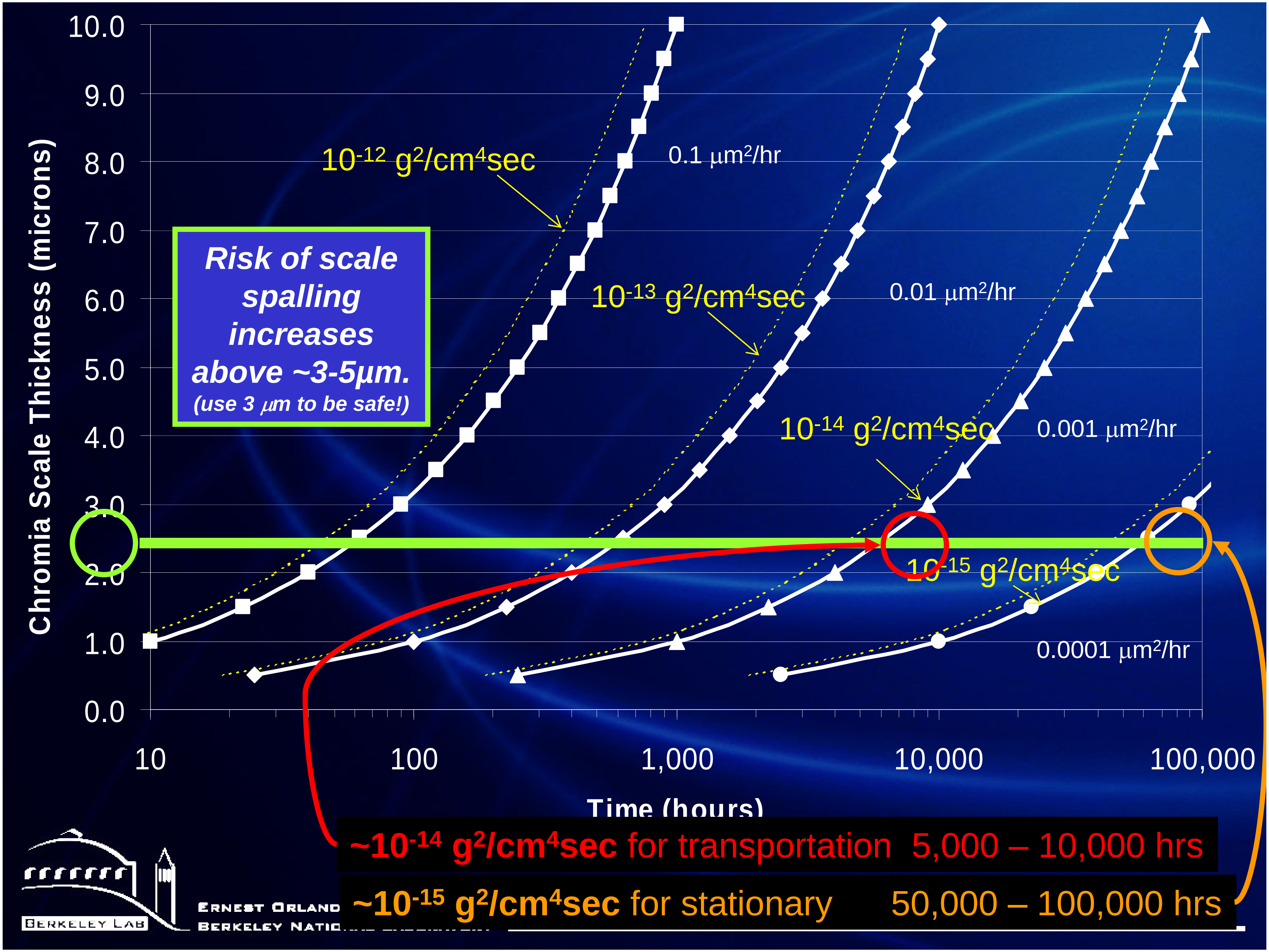


- Oxidation behavior
- Oxide spallation
- Area specific resistance
- Chromium migration



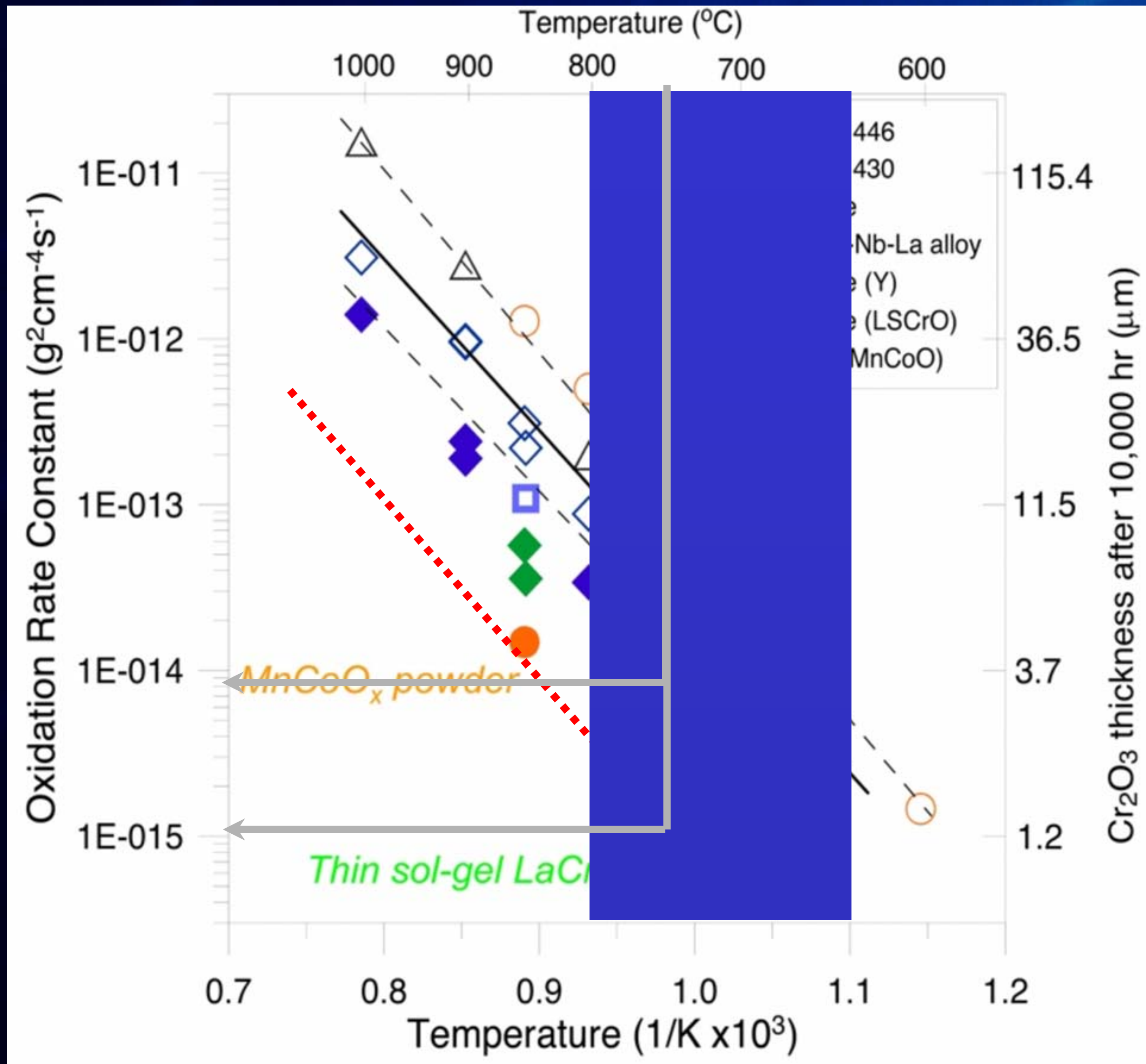
Transpiration

- Vapor chromium transport
- Bulk & grain boundary Cr transport
- Surface migration



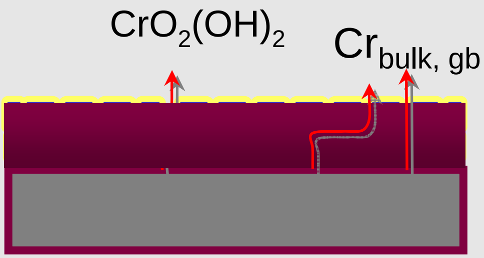
Chromia Scale Thickness (microns)
10.0
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
10
100
1,000
10,000
100,000
Time (hours)
10-12 g2/cm4sec
0.1 μm2/hr
10-13 g2/cm4sec
0.01 μm2/hr
10-14 g2/cm4sec
0.001 μm2/hr
10-15 g2/cm4sec
0.0001 μm2/hr
Risk of scale spalling increases above ~3-5μm.
(use 3 μm to be safe!)
~10-14 g2/cm4sec for transportation 5,000 – 10,000 hrs
~10-15 g2/cm4sec for stationary 50,000 – 100,000 hrs
Berkeley Lab
Ernest Orlando
Berkeley Natio

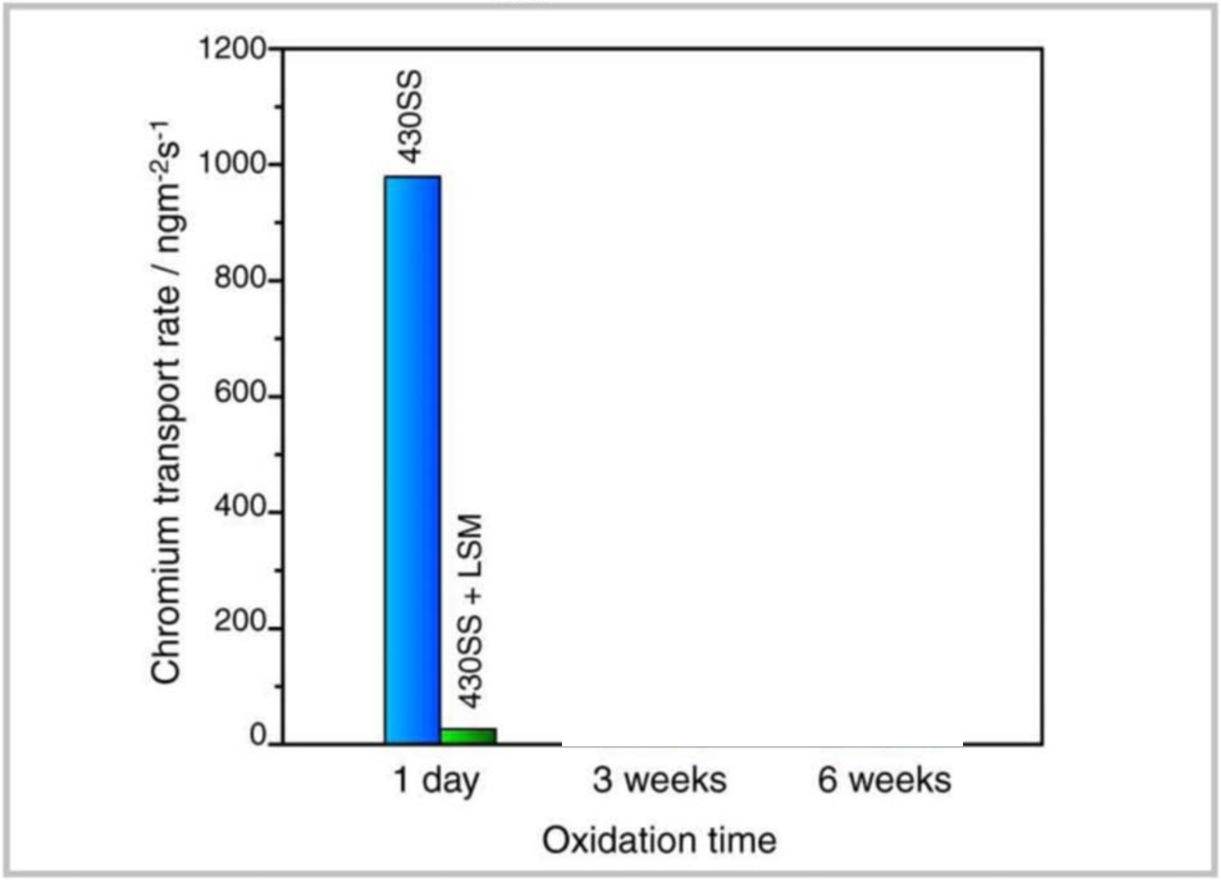
High Temperature Oxidation of Metal Components



Long-term Stability of Coatings for Preventing Cr Loss

- **Oxidation: 1073 K, P_{H2O} = 2.0x10^3 Pa, 3.33x10^{-6} m^{3}s^{-1} (200ml/min)**
- **Cr test: 1073 K, 86.4 ks (24 hrs), P_{H2O} = 1.0x10^4 Pa, 3.33x10^{-6} m^{3}s^{-1} (200ml/min)**





Condition for minimum spallation of (~1%) scales on 430ss after isothermal oxidation and fast cooling to RT

QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.

The lower the operating temperature the thicker the scale can be

Cr_2O_3 not only grows slower but also can be thicker before failure

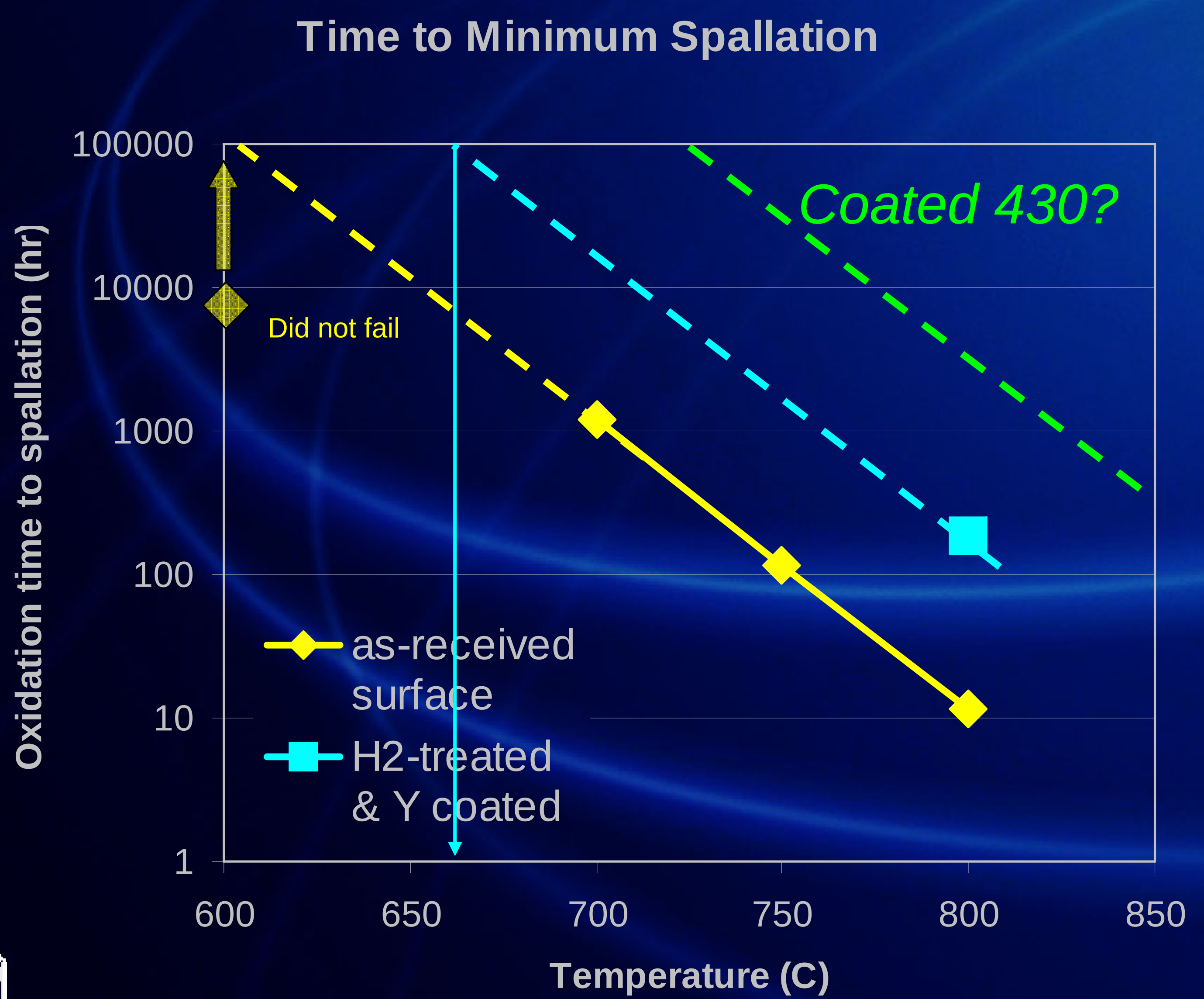
RE slow scale growth and increase adhesion/thickness

Reducing atmosphere treatment also increase adhesion

Sweet spot between 650–750 C

Scale thickness decrease because of higher thermal stresses and/or more defect formation at high oxidation temperatures

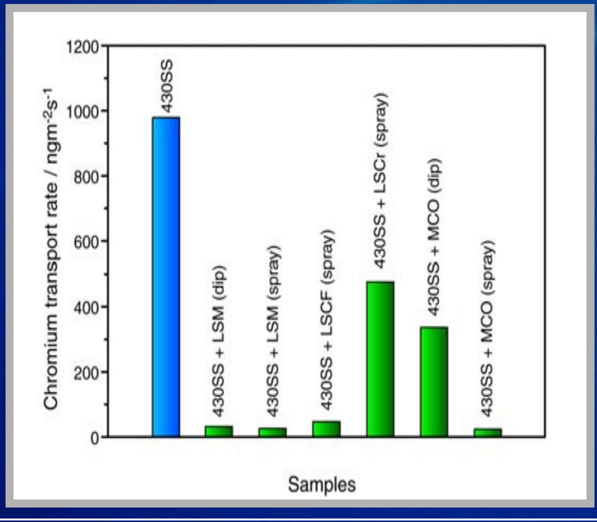
Conditions to reach ~1% Spallation in static air after isothermal oxidation and fast coolng to RT



What have we done to solve the Cr problem?

Cr Evaporation

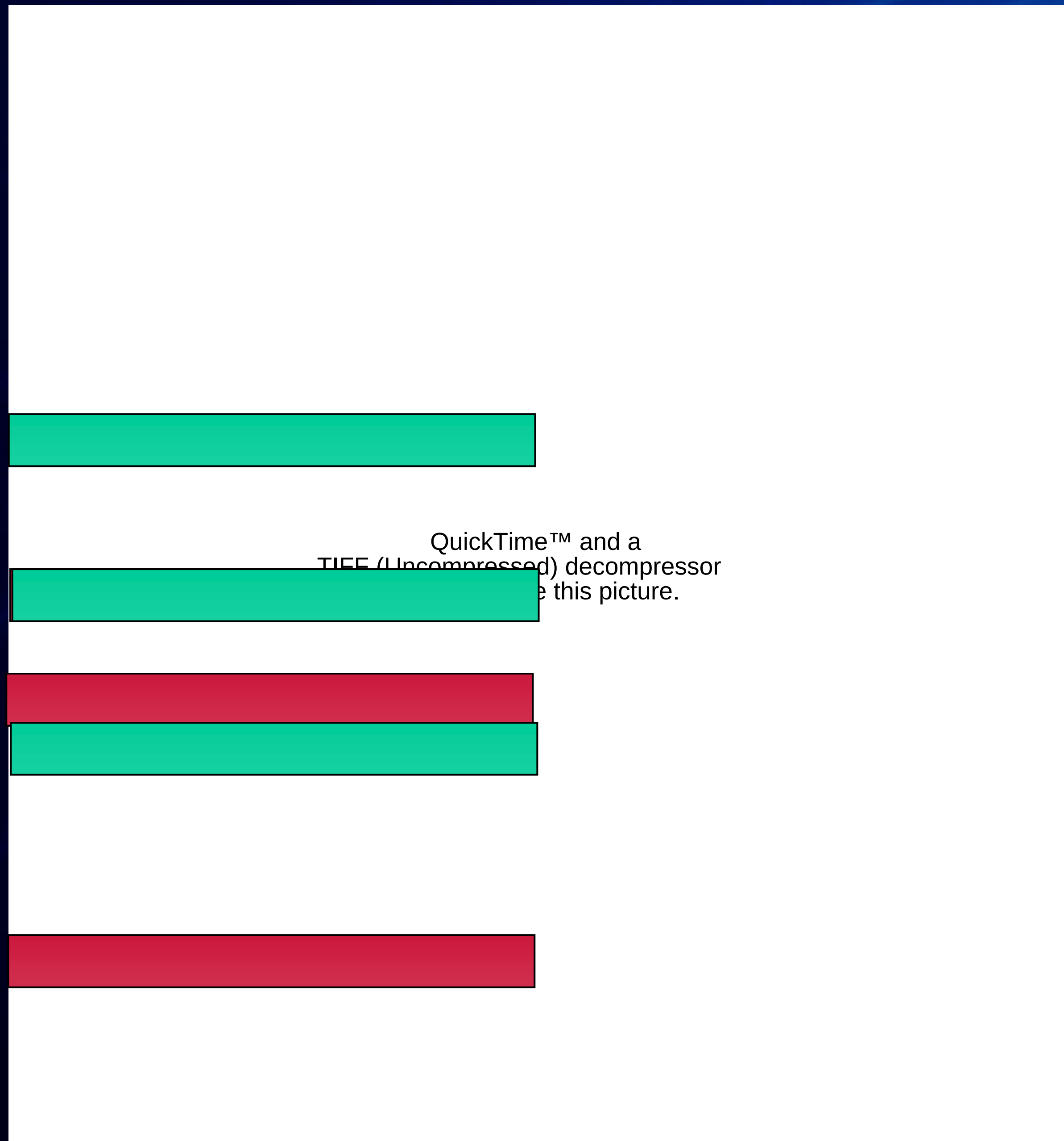
- Coat steel to prevent Cr diffusion to electrodes
- Density of coating seems more important than coating material



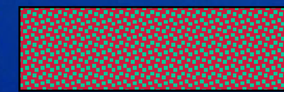
Cr Deposition

- Pairwise MOx-Cr interactions suggest Cr tolerant catalysts
- Enhance Cr tolerance of commercially available electrodes by infiltration

QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.



Rxn Couples

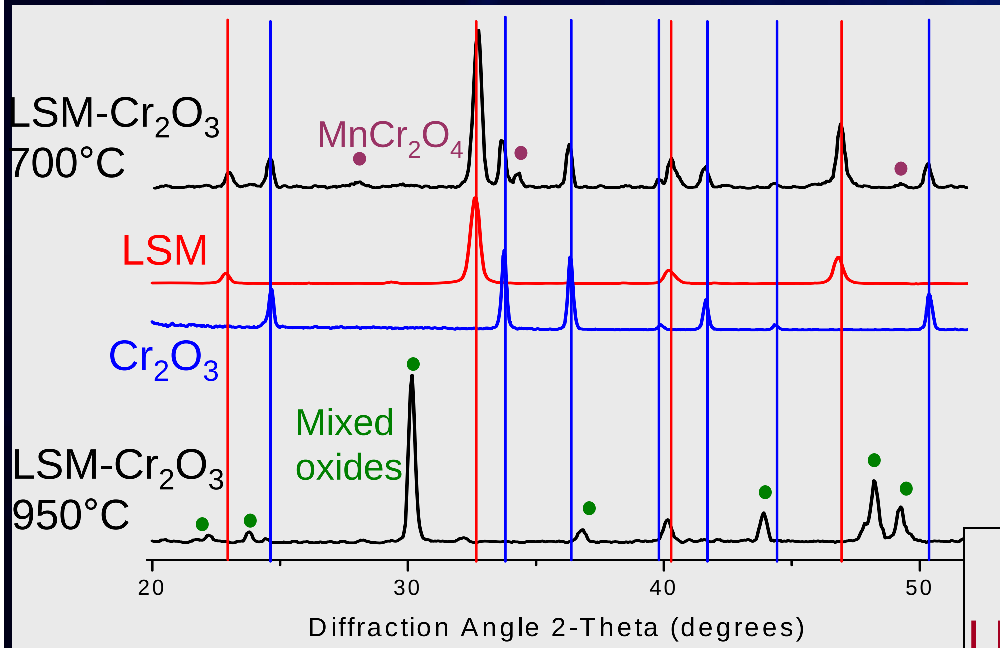


LSM + Cr_2O_3

LNF + Cr_2O_3

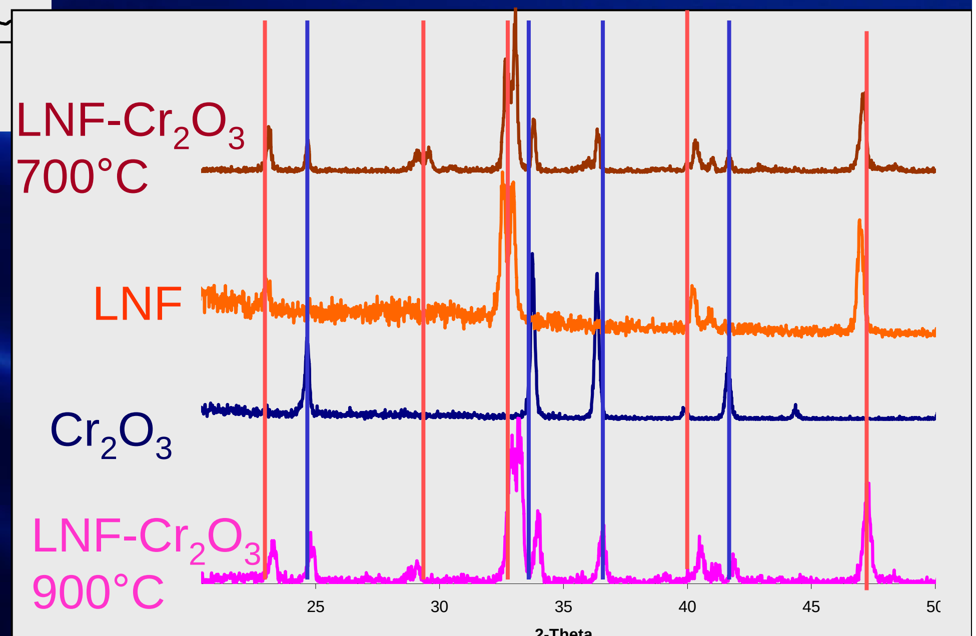
LNF Does Not React With Cr_2O_3

Pellets of LNF-Cr_2O_3 and LSM-Cr_2O_3 powder mixtures reacted for 150h at 700°C and 900-950°C



LSM-Cr_2O_3
700°C minor reaction
950°C complete reaction

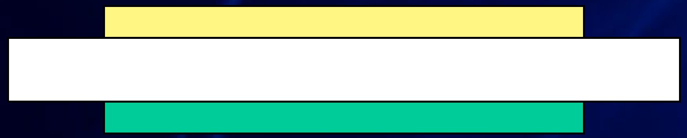
LNF-Cr_2O_3
700°C NO reaction
900°C NO reaction



LBNL Infiltration Core Program

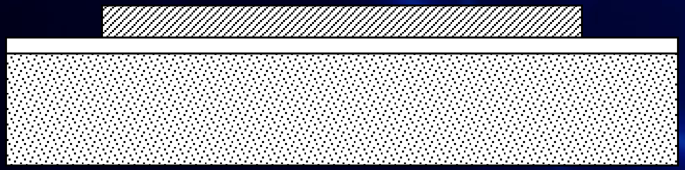
– Improve existing structures (and novel electrode design)
  - Improve cathode performance at low temperature
  - Improve tolerance to Cr
  - Sulfur tolerant anodes
– Novel Electrode Design
  - Infiltration technology allows flexibility in SOFC design and processing
  - Enables mSOFCs

Infiltration Structures & Challenges



Electrolyte supported: porous electrodes - straightforward

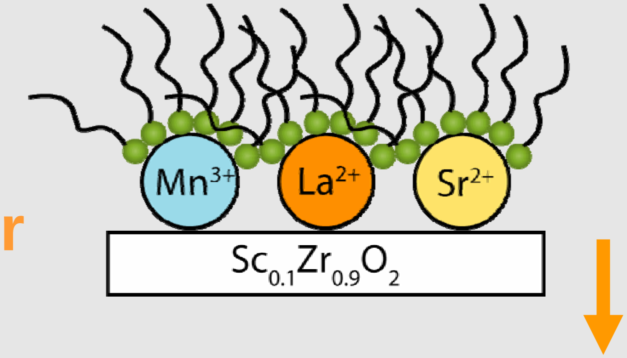
Anode supported: cathode is straightforward, anode may be too dense in unreduced state



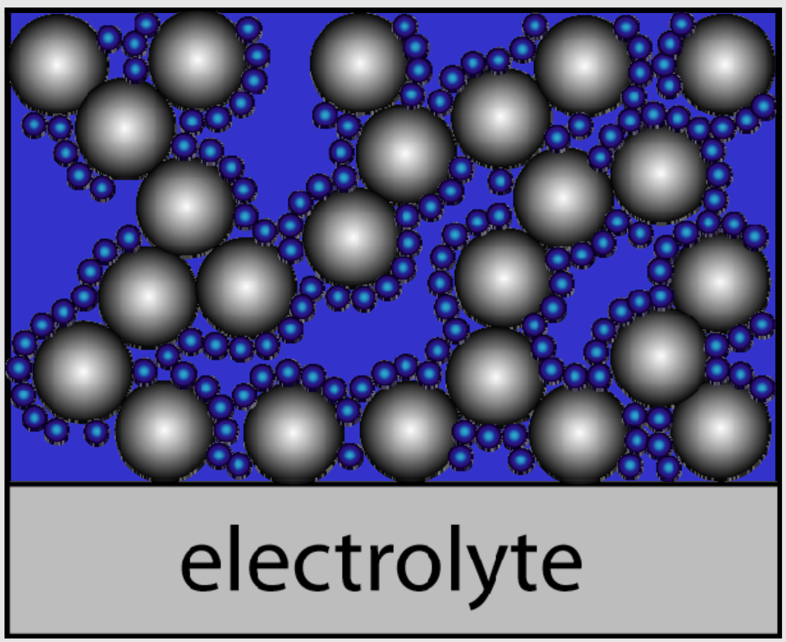
Metal supported: engineered for infiltration - entire electrode structure is infiltrated

Infiltration Step

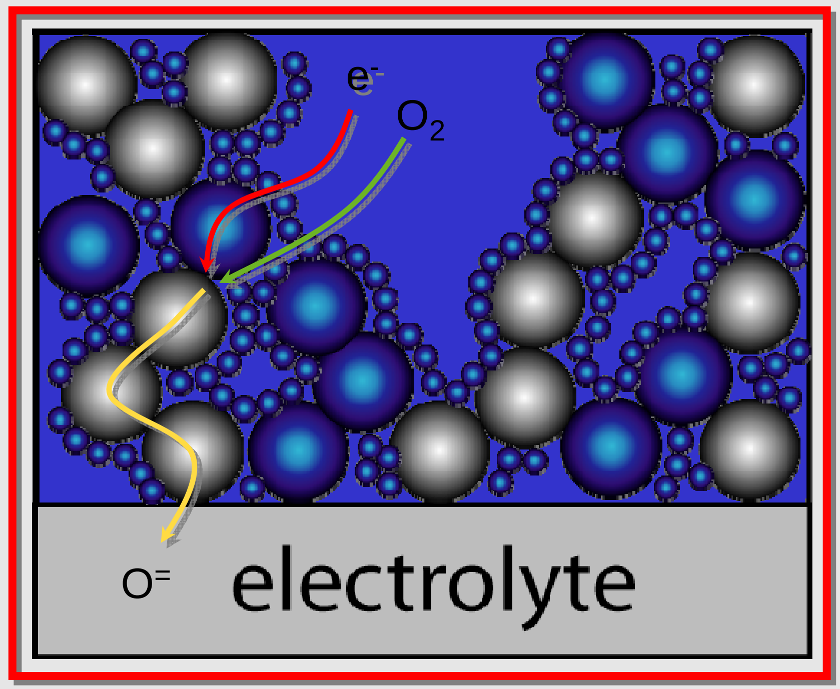
Nitrate-Surfactant Concentrated Precursor



Surfactant dispersed Electrode Precursors



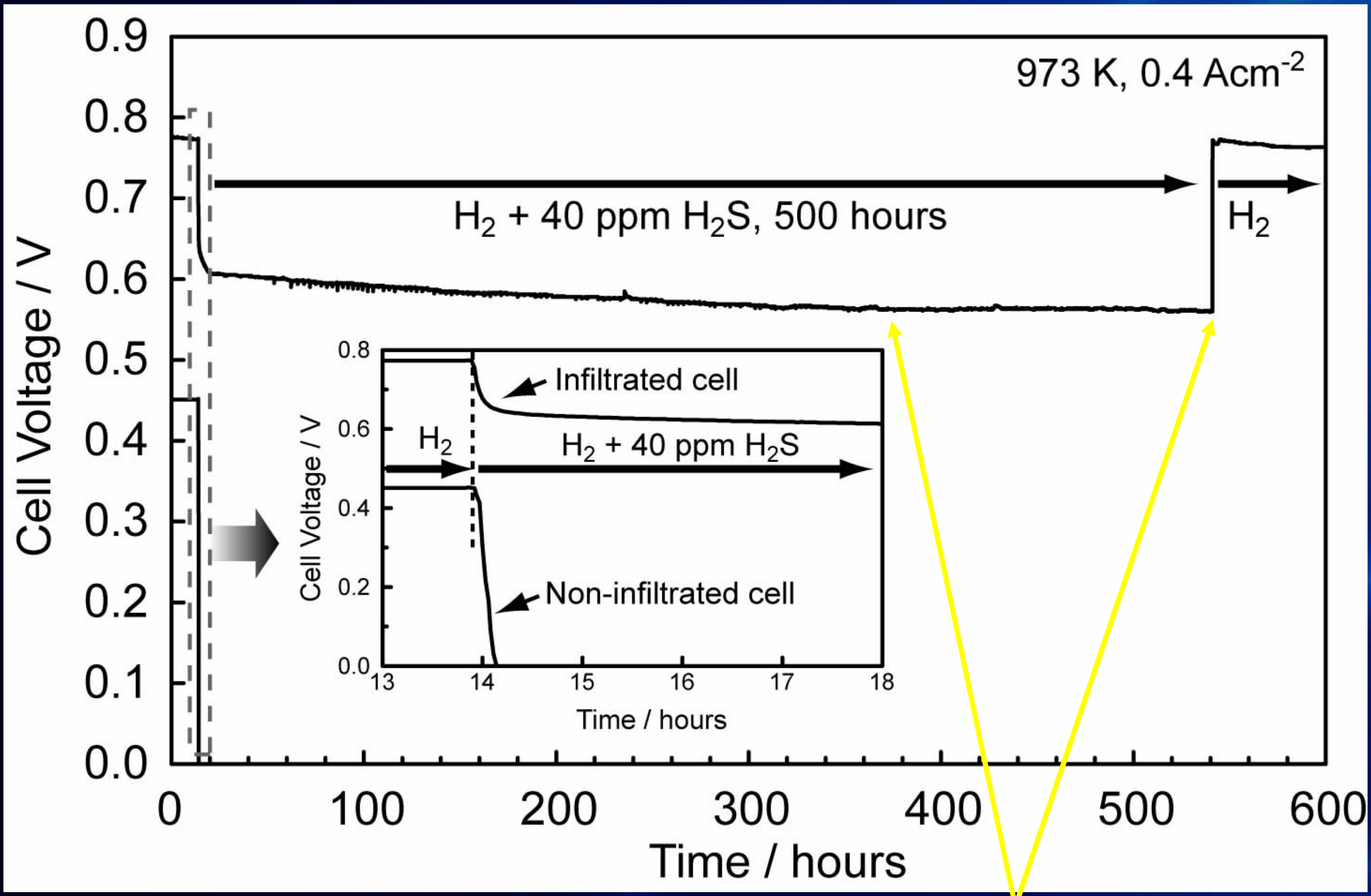
Porous electrolyte matrix



Composite Commercial electrodes (YSZ-LSM)

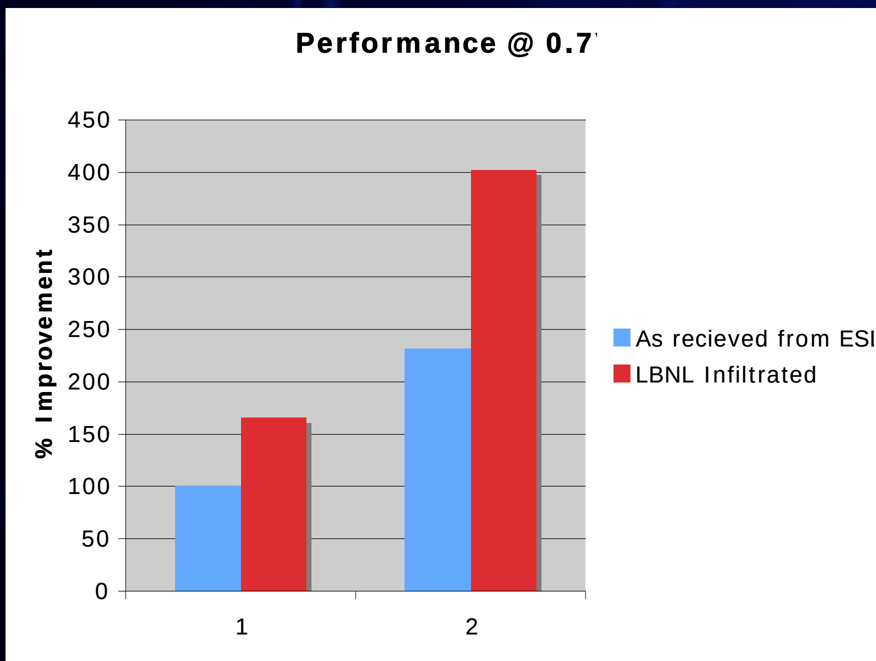
electronic conductor ionic conductor

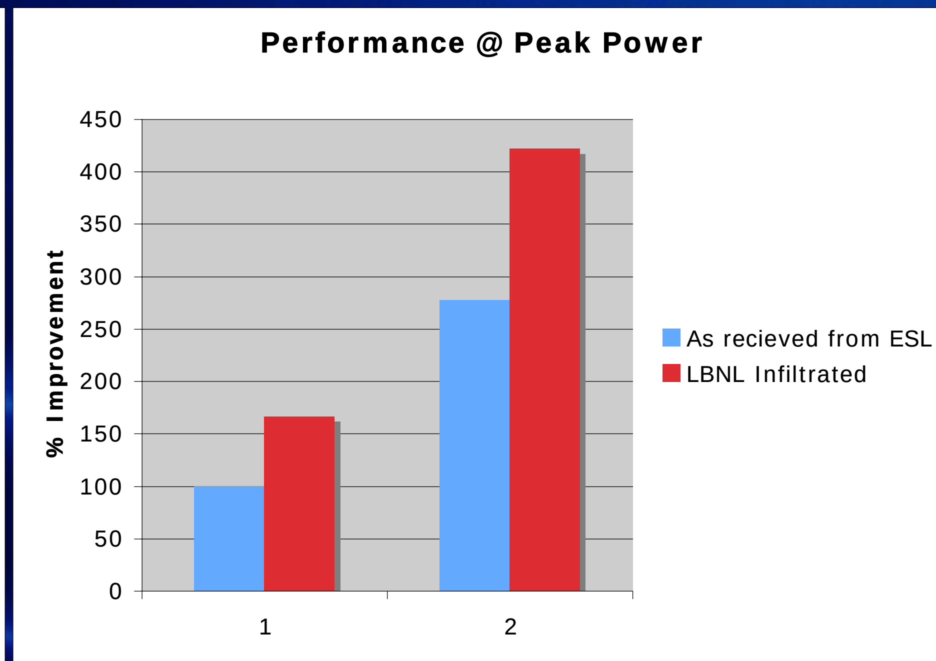
Sulfur Tolerant Ni-YSZ



0% degradation over 180 hrs

LBNL Collaboration with Electro Sciences Lab to Improve Performance of ESL SOFC product





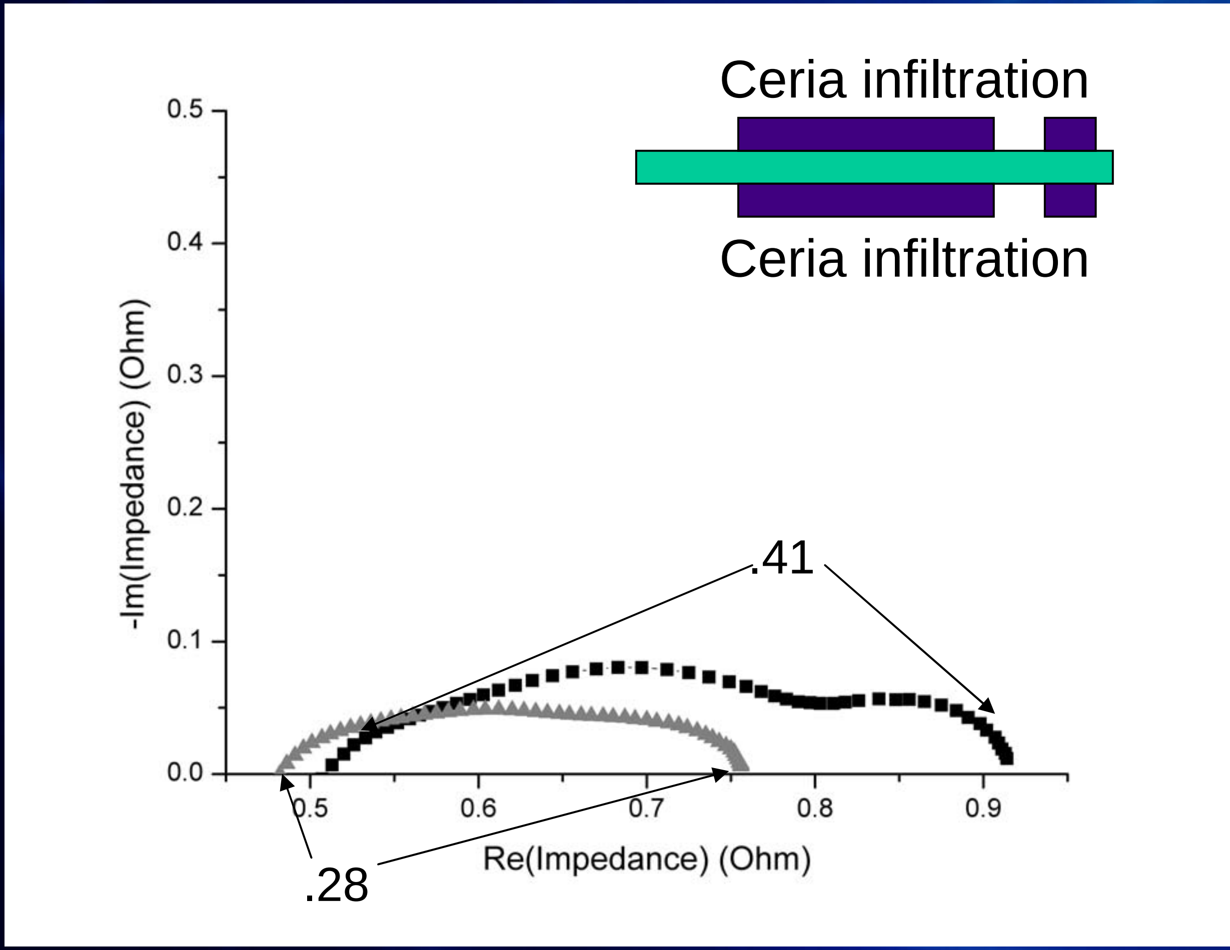
Working on standard cell for <u>**700 C**</u> operation - available to industrial teams, Universities, and National Labs - US supplier

Commercial Symmetric Electrolyte Supported LSCF Cell from INDEC
LSCF-YDC/TZ3Y/YDC-LSCF

H.C. Starck
HCST
Engineered Material Solutions

InDEC B.V.

HC Starck LSCF/LSCF Cell



=>45% improvement in cell resistance

Electrolyte supported cell: electrode Impedance before and after infiltration 700 ^{o}C

Core Technology Program
Technology Transfer

- Infiltration workshop
- Transfer technology to companies to U.S. companies and labs
- Guidance to manufacturers of cell stack components (ESL) to enhance U.S. competitiveness

Infiltration Workshop: February 16th, 2007

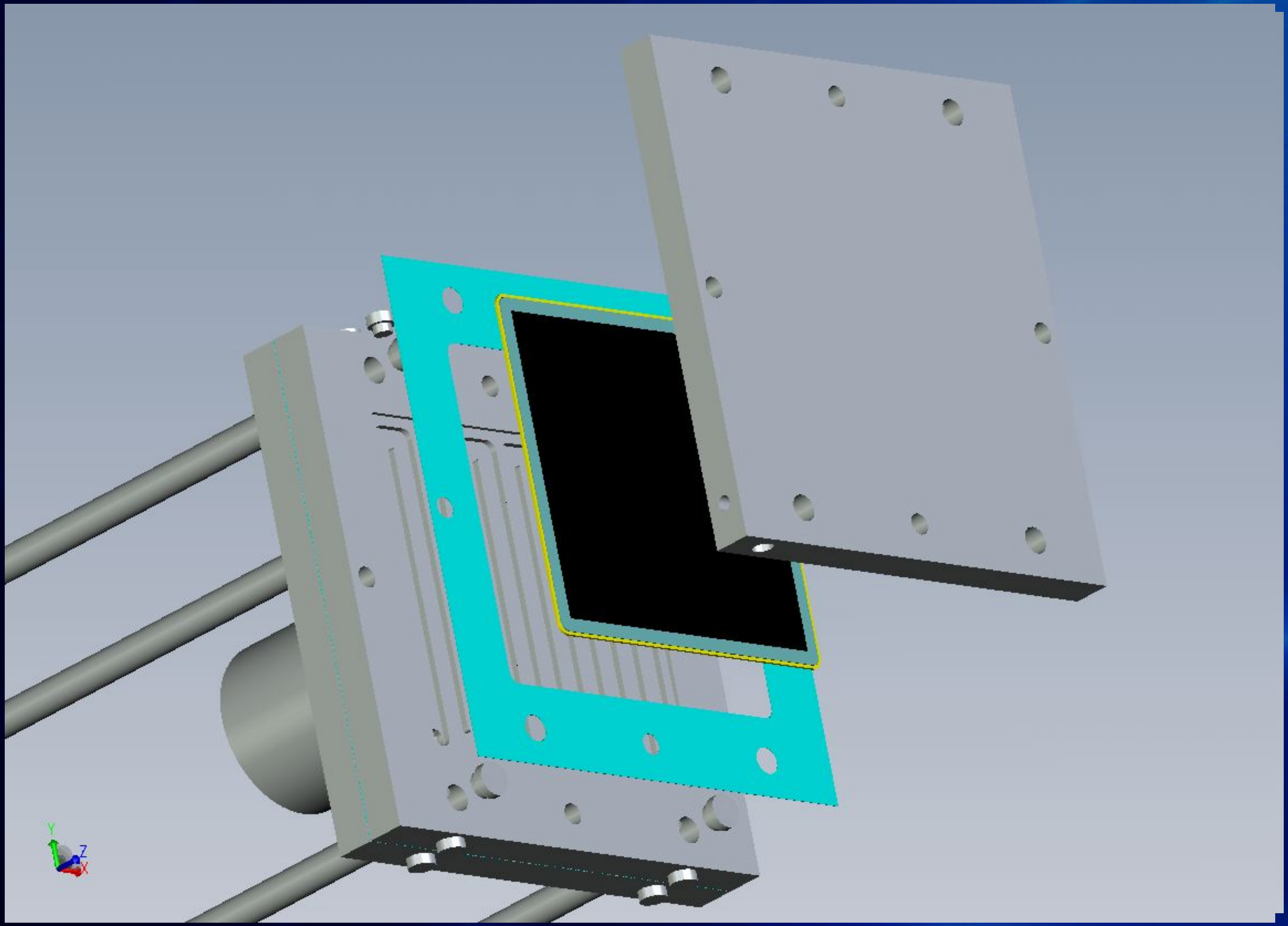
- Argonne National Laboratory
- Pacific Northwest National Lab
- Georgia Tech
- Instructional DVD from Workshop available

QuickTime™ and a
DVD/DVCPRO - NTSC decompressor
are needed to see this picture.

LBNL 2-cell Standard Stack Core Effort

Based on 2.5 cm x 2.5 cm SOFC plates for 2” bore furnace
Original design by Hideto Kurokawa

Scaled-up Standard Stack:
LBNL lead with Lane Wilson & Wayne Surdoval



5 cm x 5 cm SOFC plate design to fit into 3” bore furnace ($1500)

Quotation 4418

McAllister Technical Services
West 280 Prairie Avenue
Coeur d'Alene, ID 83815
Ph: 208-772-9527
Fax: 208-772-3384
Email: solutions@mcallister.com
URL: www.mcallister.com

Date: 02-Feb-07

This estimate is good for 60 days from the date shown above. Prices quoted are for quantities shown.

To

Steven J. Visco
LBL Materials Sciences Division
1 Cyclotron Road
Berkeley, CA 94720
Ph: 510.486.5821
Fax: 510.486.4881
sjvisco@lbl.gov

Terms Offered: Net-30

Delivery: 12 Wks. ARO
(based on current workload)

FOB: Factory, Coeur d'Alene, Idaho

Item	Quantity	Description	Model #	Unit Price	Amount U.S.D.
1	1	**SOFC Cell Assembly,** CNC Program		750.00	$750.00
2	1	**Design**	**SOFC 5X5**	760.00	$760.00
[redacted]					
5	5	**Additional Center Plates** (if ordered at same time)		$135.00	$675.00

Thank you for the opportunity to quote on this project!

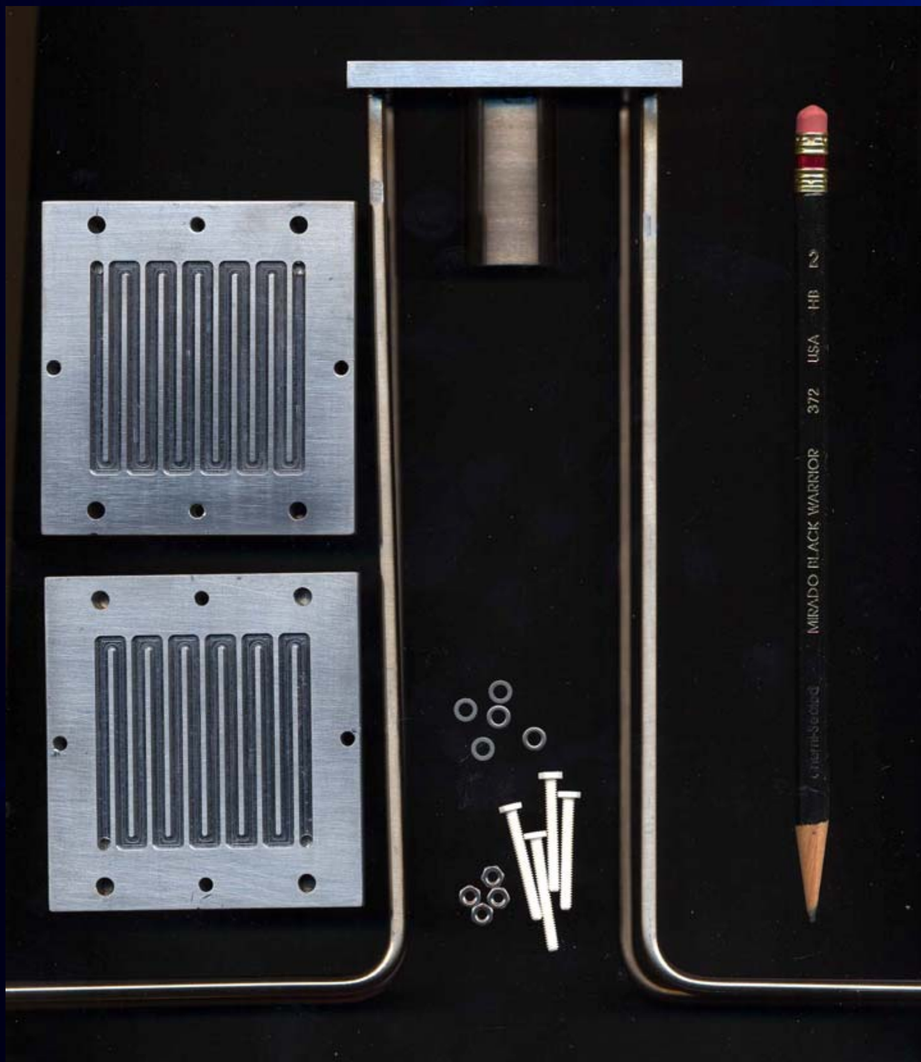
Sales Tax: N/A
Shipping: at cost

Estimate authorized by

Name:

Title: Robert McAllister, President

McAllister Build of 2-cell 5 x 5 cm SOFC Plate Stack

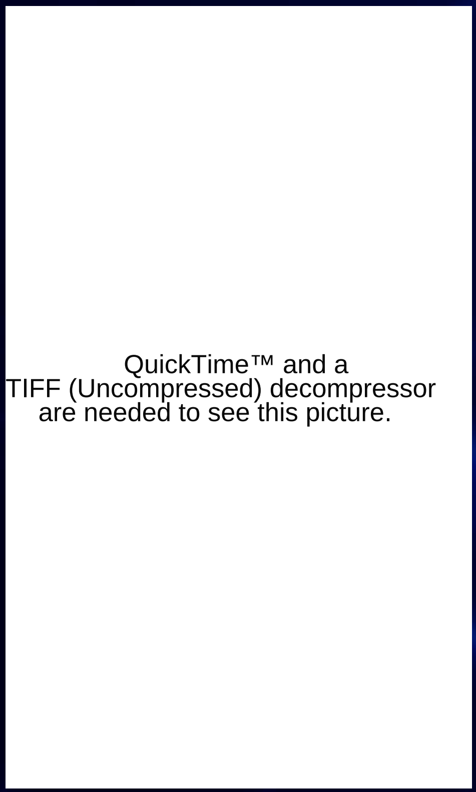


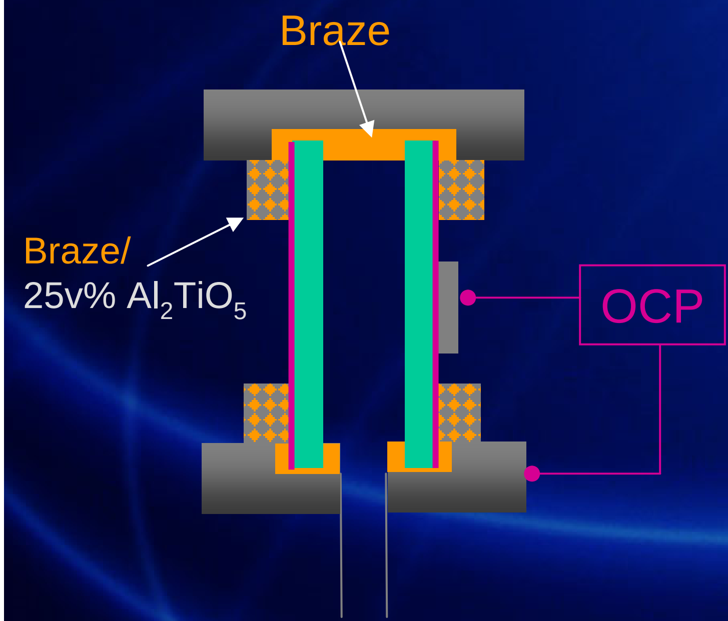
- Standardized test platform
- Allows testing of electrodes, seals, contact pastes, in a uniform manner
- Allows comparison of results between labs, universities, and industry
- Fits in inexpensive furnaces
- Is not intended as a precursor to commercial device
- ~ $800/ea. after initial build

LBNL Work on mSOFCs

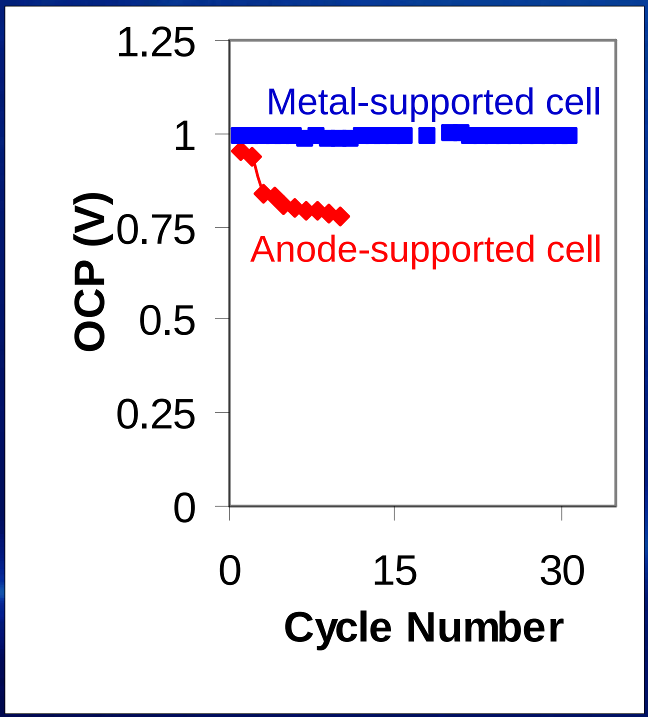
- Build structure from low cost materials
- Obtain performance similar to anode supported cells
- Show long term stability (rapid progress)
- Work with cell manufacturers (licensing & sponsored research)

Rapid Thermal Cycling – Braze-Sealed Cell





150-735°C, ~500°C/min



Anode supported tubular cell cannot tolerate rapid thermal cycling

Cell failed, joint did not

Metal-supported cell/brazed joint is robust to thermal cycling

Unexpected shutdowns, redox cycles

650-700°C Performance

Moist hydrogen fuel, air

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TIFF (Uncompressed) decompressor
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650°C

QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.

QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.

700°C

QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.

Infiltrated Electrodes Support High Power Density

Moist hydrogen fuel, pure oxygen (removes gas transport limitation)

Temperature	Max Power (mW/cm2)	Power at 0.7V (mW/cm2)
650°C	982	726
700°C	>1300	993
750°C	>1300	>1300

Work with manufacturer to ensure manufacturability as continue cell development

Cell Manufacturer Inc. → Cell & Stack → System Integrators

- Military
- APU
- Residential
- CHP
- Portable
- RV/Marine
- Aerospace
- Etc.

High Volume Porous Metal Media

Coal: kW to MW?

QuickTime™ and a
TIFF (Uncompressed) decompressor
are needed to see this picture.

Transitioning Technology to Private Sector

- LBNL is in discussions with cell/stack manufacturers for licensing infiltration and mSOFC technology for both planar and tubular configurations
- Wide range of IP being negotiated for SOFC, an coating for filtration (including spin-off applications for for coal gasification)
- Commercial interest in infiltration and mSOFC technology is rising quickly

Future Work

- Continued focus on infiltration technology as a means of improving cathode (and anode) performance at reduced cell temperatures
- Emphasis on baseline degradation studies on commercial cells as a metric of infiltration performance over time
- Continuing activities in technology transfer
- New stuff

Acknowledgements

This work was supported by the U.S. Department of Energy under Contract No. DE-AC02-05CH11231.

Thanks to Lane Wilson and Wayne Surdoval for their input to the LBNL program

Good luck to Lane at BES