

Button Cell Tests with LSM/YSZ Cathodes in Air with Quantified Cr Concentrations

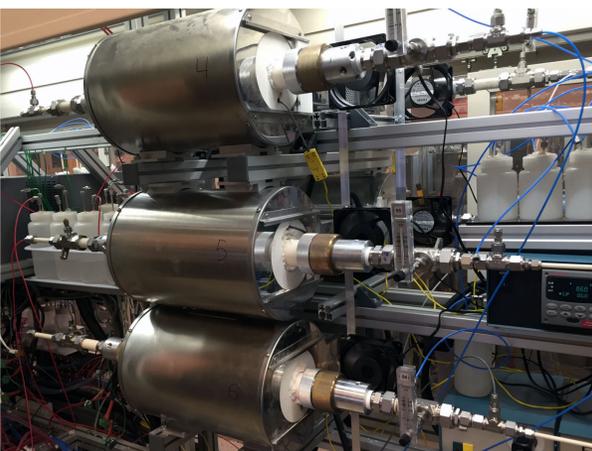


Pacific Northwest
NATIONAL LABORATORY

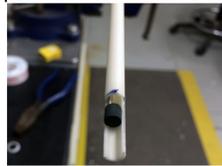
Proudly Operated by Battelle Since 1965

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SIX TEST FIXTURES (THREE ARE SHOWN)



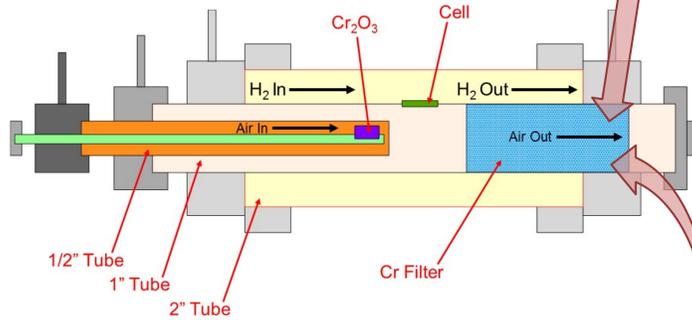
Upstream Chromia Pellet



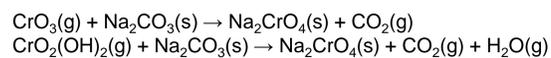
Downstream Filter



Button Cell Cr Test Fixture Schematic (Not to Scale)



Na_2CO_3 was coated on reticulated alumina filters with a spec of 3 ppm Cr or less

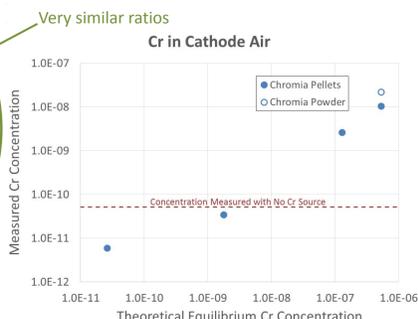


Thermodynamic calculations predict a reduction in the concentration of Cr-species in the air by more than 8 orders of magnitude due to Cr capture

FIXTURE VALIDATION TESTS

Parameters	Cr Mass (μg)	Measured mol% Cr in Air	Theoretical Eqm. mol% Cr	Meas/Theo
Cr ₂ O ₃ Powder at 800C 3% Water	306.17	2.22E-08	5.33E-07	4.17E-02
Cr ₂ O ₃ pellet at 800C 3% Water	143.21	1.06E-08	5.33E-07	1.99E-02
Cr ₂ O ₃ pellet at 600C 3% Water	35.83	2.62E-09	1.28E-07	2.05E-02
Cr ₂ O ₃ pellet at 800C Dry Air	0.46	3.44E-11	1.78E-09	1.93E-02
Cr ₂ O ₃ pellet at 600C Dry Air	0.08	6.01E-12	2.62E-11	2.30E-01
No Cr Dry Air	0.70	5.18E-11	0	N/A

Chromia appeared to have blown out of the container



FILTER CAPACITY TESTS AT HIGH Cr VOLATILITY

Chromia pellet at 800°C in humid air

Time (h)	mol% Cr in Air	Meas/Theo mol% Cr
195	7.08E-09	1.33E-02
312	7.17E-09	1.34E-02
602	1.15E-08	2.16E-02
811	5.41E-09	1.01E-02
1008	8.98E-09	1.68E-02

All are ~1-2% of predicted equilibrium concentration

No saturation after 1000 h

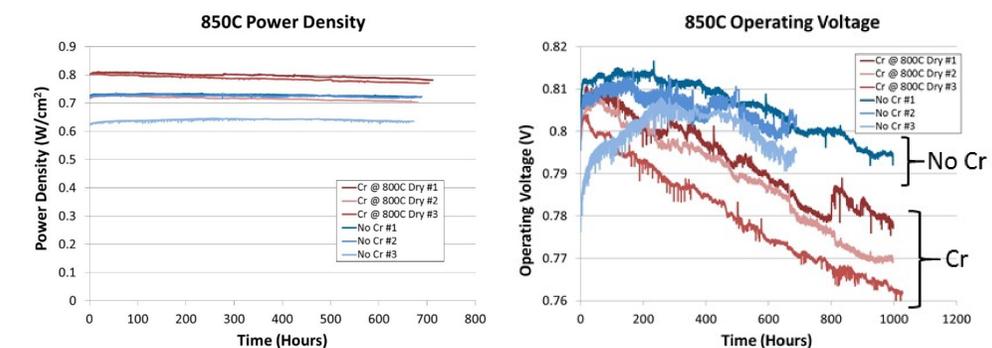
DETECTION LIMIT TESTS AT LOW Cr VOLATILITY

Chromia pellet in dry air

Time (h)	Cr ₂ O ₃ Temp (C)	Meas. Cr Conc. (ppt molar basis)	Meas./Pred. Cr Conc. (%)
1035	N/A	14.8	N/A
1035	600	3.41	13.0
1035	800	64.7	3.64
2015	N/A	47.9	N/A
2015	600	31.4	120
2015	800	87.7	4.94

- Measured Cr concentrations with chromia pellets at 600°C in dry air were less than with no chromia present.
- Measured Cr concentrations with chromia pellets at 800°C in dry air were above no chromia, but elevated above the typical 1-2% of predicted equilibrium concentration likely due to baseline Cr contributions.
- Working with an alumina foam manufacturer to get filter substrates made with 6N pure alumina to decrease baseline Cr from the 3 ppm Cr in current substrates

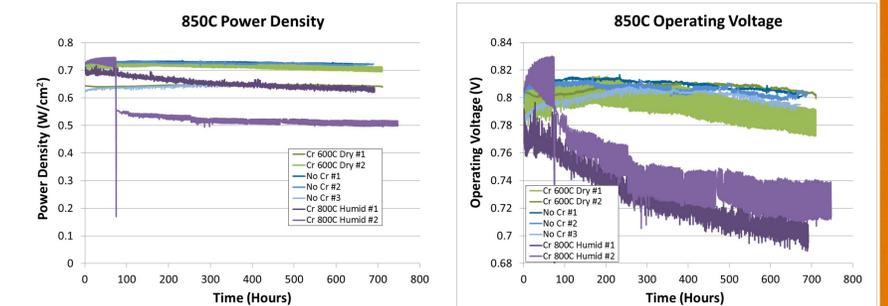
ELECTROCHEMICAL BUTTON CELL TESTS



Cr ₂ O ₃	Meas. Cr Conc. (ppt molar basis)	Pred. Eqm. Cr Conc. (ppt molar basis)	Meas./Pred. Cr Conc. (%)
800°C Pellet in Dry Air	170	1780	9.58
800°C Pellet in Dry Air	41.4	1780	2.33
800°C Pellet in Dry Air	116	1780	6.54
None in Dry Air	7.87	0	N/A
None in Dry Air	38.4	0	N/A
None in Dry Air	171	0	N/A

≤170 ppt Cr in Cathode Air caused 4%/kh degradation

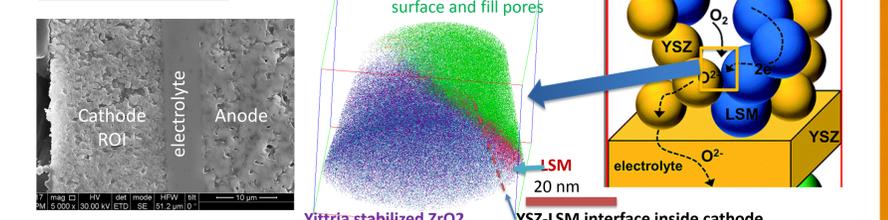
ELECTROCHEMICAL TESTS AT ADDITIONAL Cr LEVELS



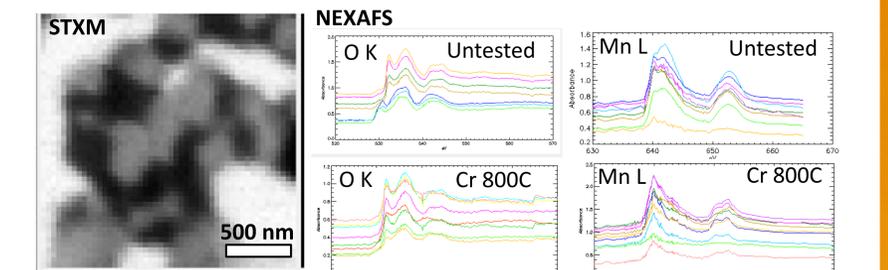
Cr ₂ O ₃	Meas. Cr Conc. (ppt molar basis)	Pred. Eqm. Cr Conc. (ppt molar basis)	Meas./Pred. Cr Conc. (%)
800°C Pellet in Humid Air	6640	573000	1.16
800°C Pellet in Humid Air	6700	573000	1.17
600°C Pellet in Dry Air	2.26	26.2	8.62
600°C Pellet in Dry Air	10.1	26.2	38.7

- 6.6 – 6.7 ppb Cr in Cathode Air caused 14%/kh degradation
- ≤10 ppt Cr in Cathode Air did not cause significant degradation

MICROSCOPY



- TEM (for structure) and Atom probe tomography (a method to map the composition in 3D at ~0.3nm spatial resolution) are used to map the Cr distribution in tested SOFC cathode.



- STXM and NEXAFS at Advanced Light Source, LBNL used to probe the change in oxidation state of Mn and O before and after testing with Cr in air.

SUMMARY

- Cr test fixture can capture Cr from chromia pellet at 800°C in humid air for 1000 hours without saturating the filters.
- Cr captured from chromia pellet at 800°C in dry air for 1000 hours is near background Cr levels.
- Cr captured from chromia pellet at 600°C in dry air for 1000 hours is below background Cr levels.
- Background Cr is believed to come from alumina filter substrates (3 ppm Cr spec)
- Concentration of Cr in air flowing through test fixtures is 1-2% of equilibrium.
- ≤10 ppt Cr in cathode air did not cause significant degradation
- ≤170 ppt Cr in cathode air caused 4%/kh degradation
- 6.6 – 6.7 ppb Cr in cathode air caused 14%/kh degradation

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