

LSM/YSZ Button Cell Tests in Cathode Air with Measured Cr Concentrations

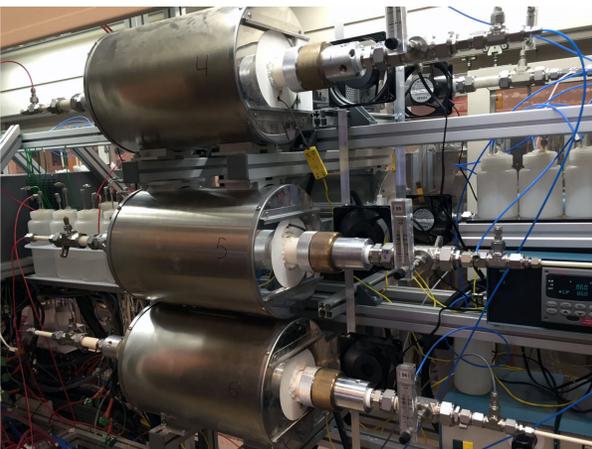


Pacific Northwest
NATIONAL LABORATORY

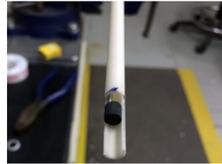
Proudly Operated by Battelle Since 1965

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SIX TEST FIXTURES (THREE ARE SHOWN)



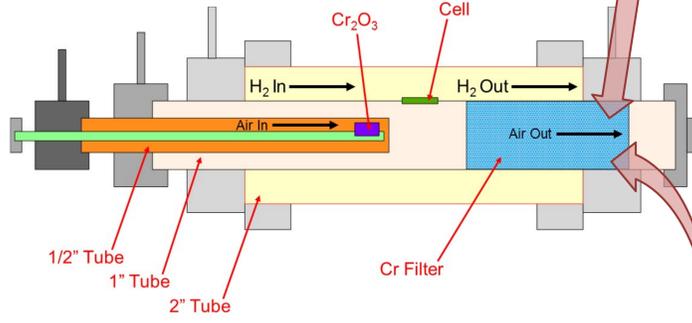
Upstream Chromia Pellet



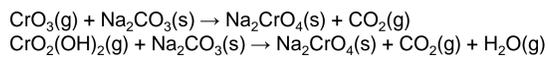
Downstream Filter



Button Cell Cr Test Fixture Schematic (Not to Scale)

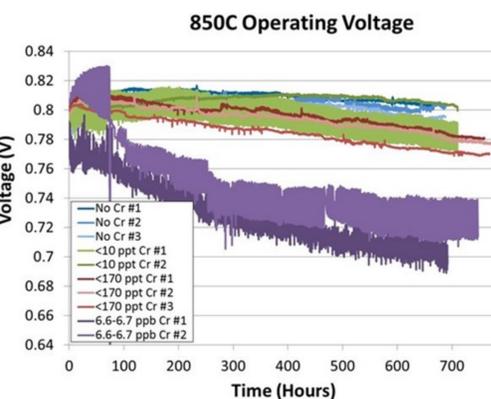


Na₂CO₃ was coated on reticulated alumina filters with a spec of 3 ppm Cr or less



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

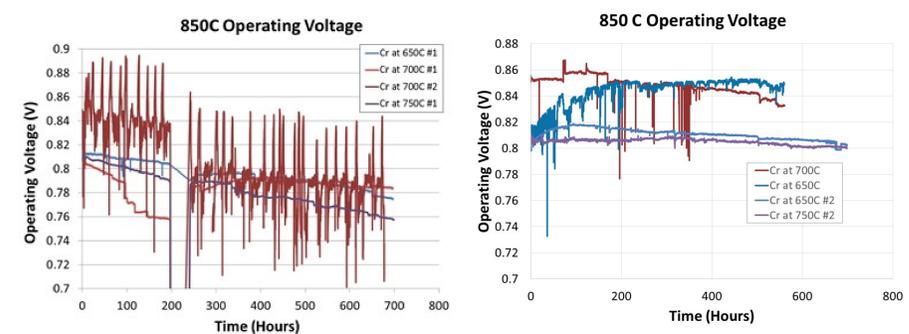
BUTTON CELL TEST RESULTS



Test Condition	Average Degradation Rates (per kh)		
	0 h - 300 h	300 h - End	0 h - End
No Cr	-4.3%	2.6%	-0.2%
≤10 ppt Cr	-1.7%	3.9%	2.2%
≤170 ppt Cr	2.5%	4.5%	3.9%
6.6-6.7 ppb Cr	24.9%	6.8%	13.2%

Lots of overlap between electrical results of No Cr and ≤10 ppt Cr tests – no significant difference in degradation

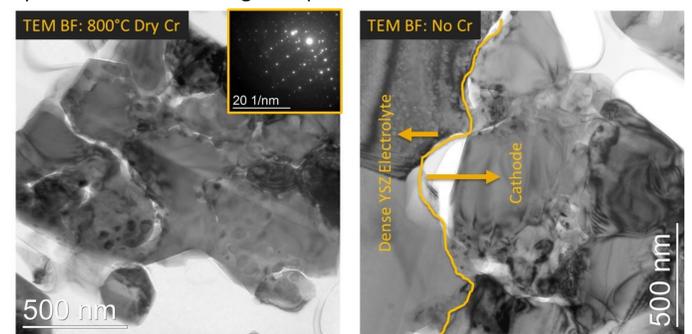
ADDITIONAL BUTTON CELL TESTS AT INTERMEDIATE Cr LEVELS



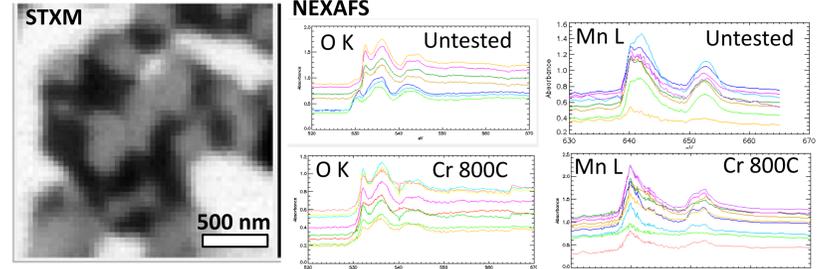
Cr ₂ O ₃ Temp (C)	Cr in Air (ppt)	Avg Degr Rate (%/kh)
650	≤43	-0.8
700	≤224	3.9
750	≤164	4.9

SEEKING MICROSCOPIC EVIDENCE OF Cr AFTER HIGH Cr TESTS

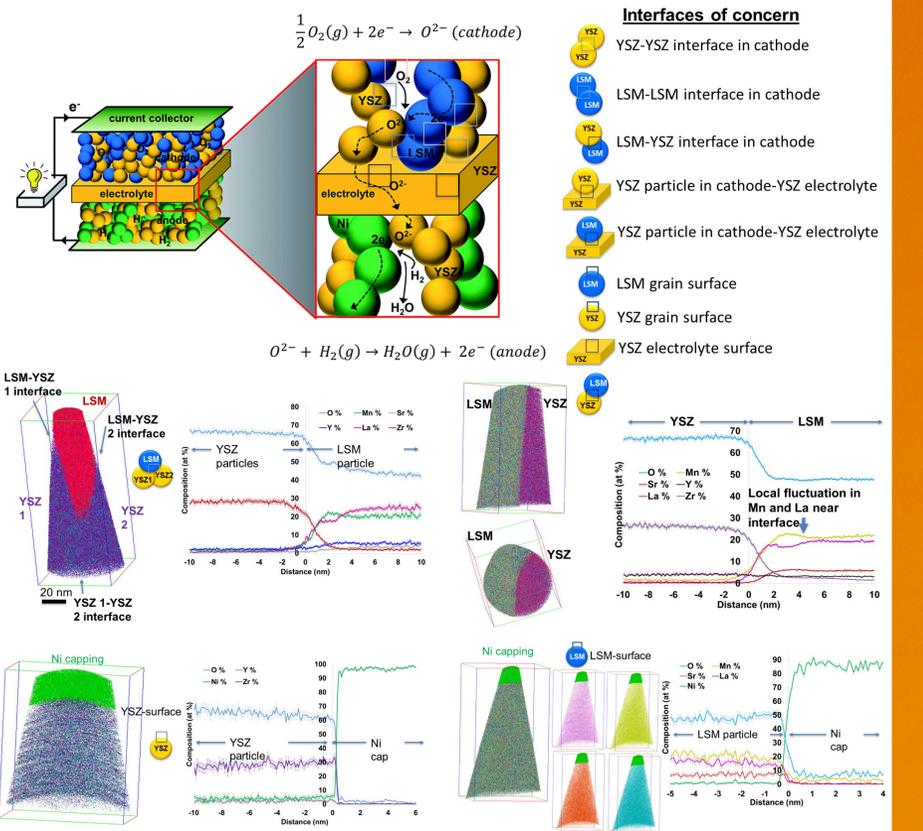
- SEM-EDS and SEM-WDS were not able to detect Cr.
- TEM did not resolve Cr, but did suggest more attack at interfaces between grains and potentially small precipitate formation during Cr exposure:



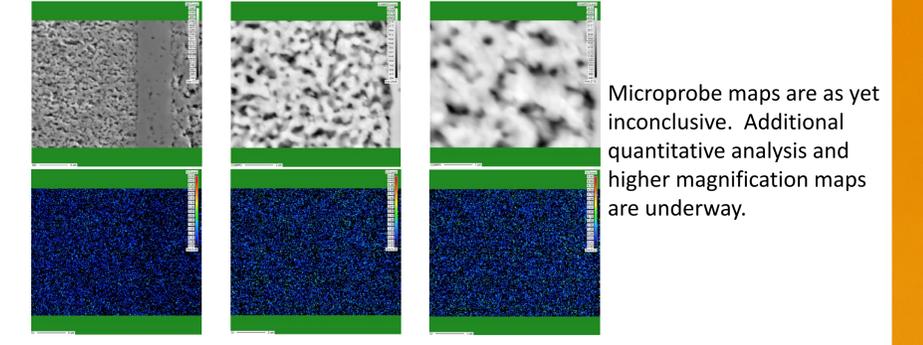
- STXM and NEXAFS at Advanced Light Source, LBNL used to probe the change in oxidation state of Mn and O before and after testing with Cr in air found increased Mn²⁺ after Cr exposure:



ATOM PROBE TOMOGRAPHY CAMPAIGN TO LOCATE Cr



Cr MAPPING BY ELECTRON PROBE MICROANALYZER



SUMMARY

- ≤43 ppt Cr in cathode air did not cause significant degradation
- ≤164 ppt Cr in cathode air caused ~4.9%/kh degradation
- 6.6 – 6.7 ppb Cr in cathode air caused ~13%/kh degradation
- Threshold concentration below which LSM/YSZ cathode stability is unaffected is somewhere below 165 ppt. Precise determination is hindered by baseline Cr.
- After tests with an 800°C chromia pellet in humid air, attempts were made to find Cr:
 - SEM-EDS and SEM-WDS did not detect Cr
 - TEM did not detect Cr, but suggested increased grain boundary attack
 - STXM-NEXAFS spectra had no Cr signal, but showed Mn reduces to Mn²⁺
 - APT tests are still underway but have not yet discovered Cr
 - Microprobe mapping was recently initiated and is, as yet, inconclusive.