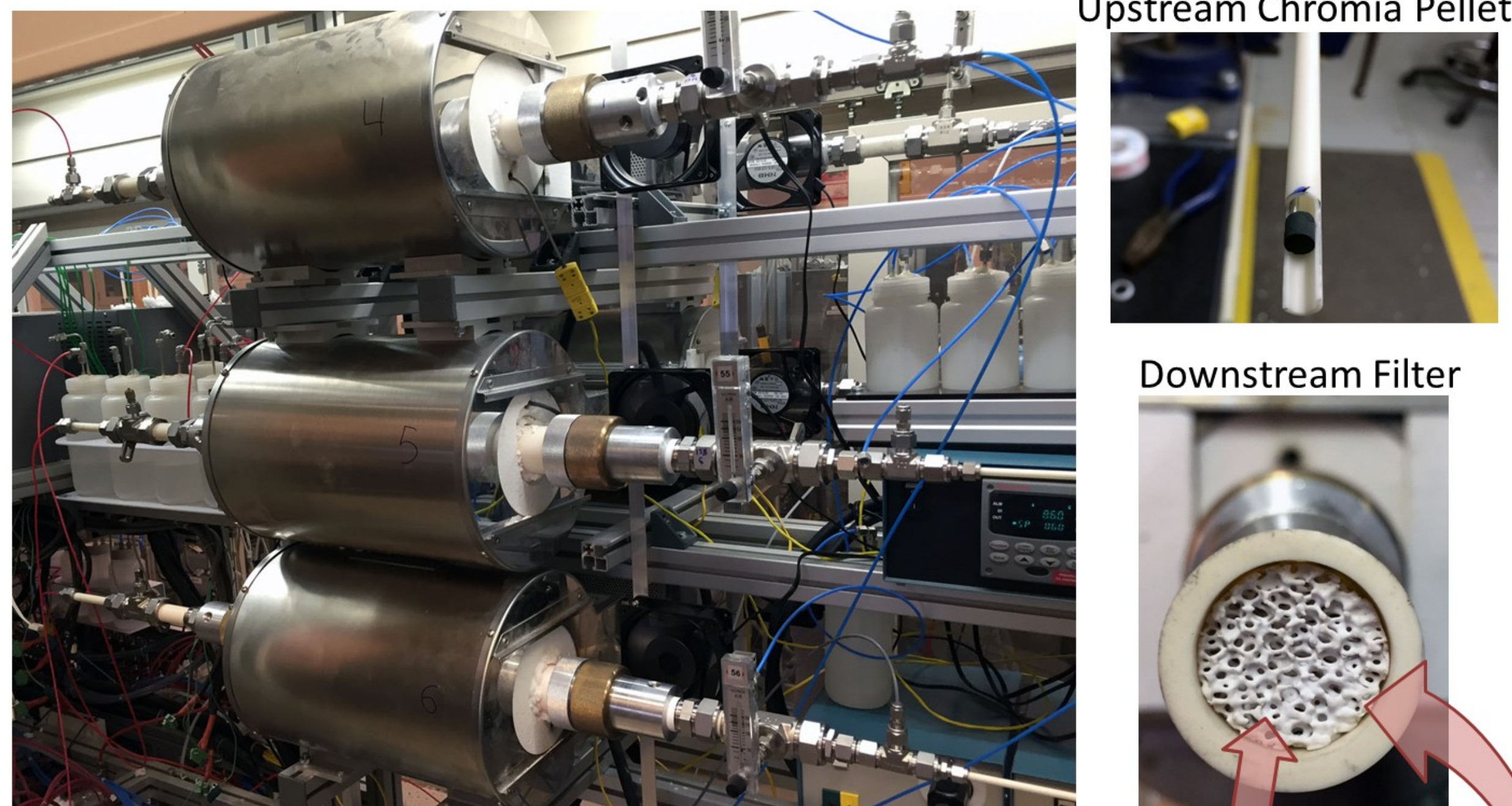


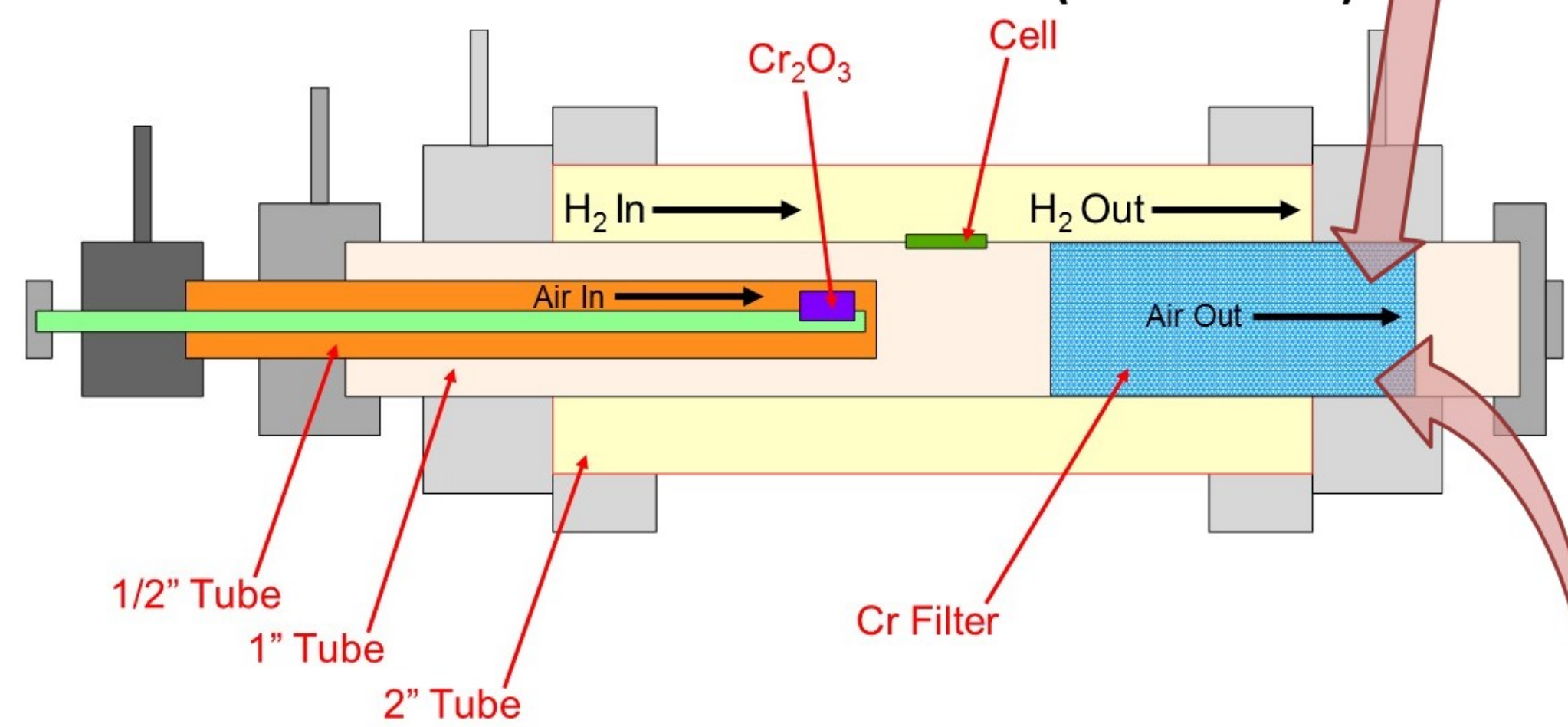
# Effects of Cr Concentrations in Air on LSM/YSZ & LSCF Cathode Degradation

John S. Hardy, Christopher A. Coyle, James J. Neeway, Dan J. Edwards, Nathan L. Canfield, Arun Devaraj, and Jeffrey W. Stevenson

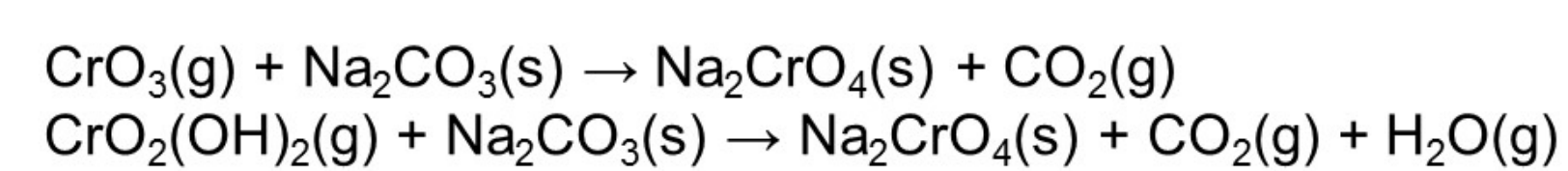
## SIX TEST FIXTURES (THREE ARE SHOWN)



Button Cell Cr Test Fixture Schematic (Not to Scale)

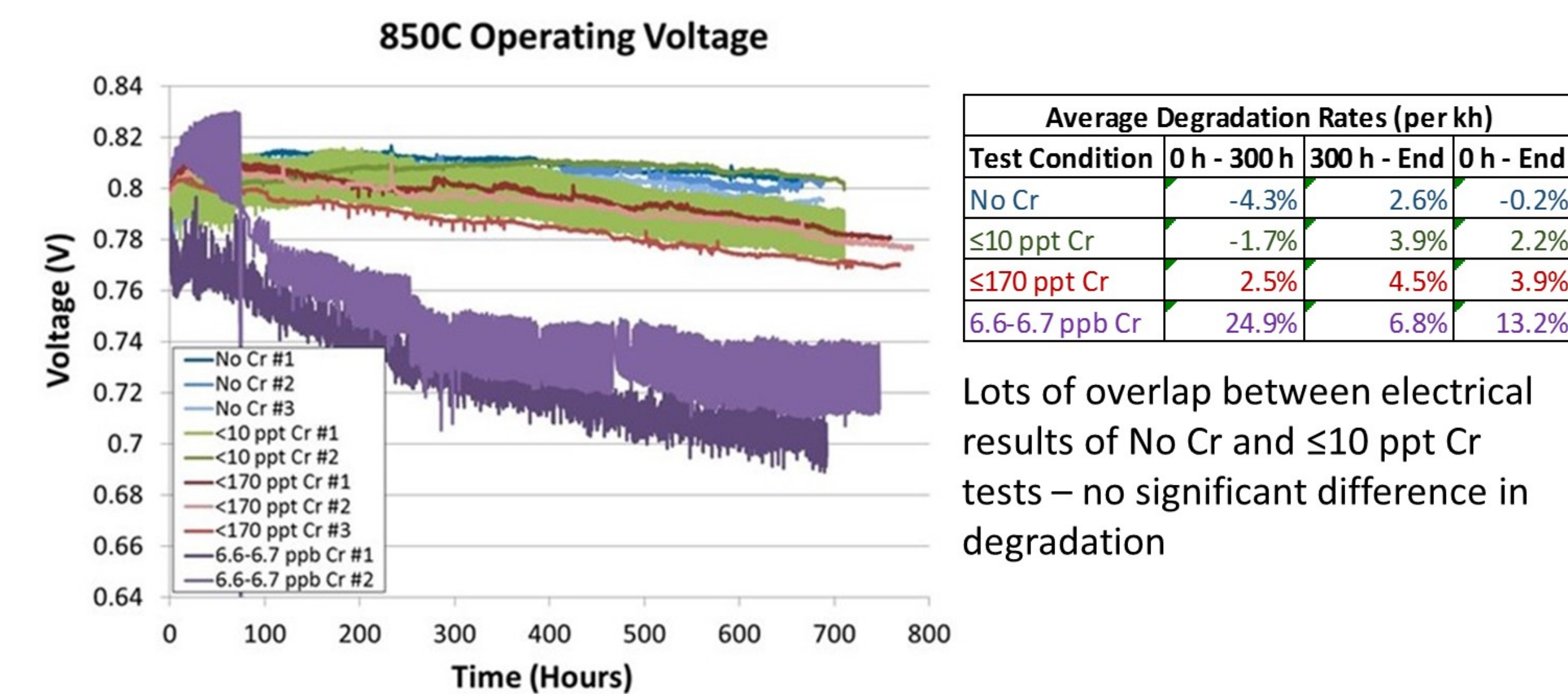


Na<sub>2</sub>CO<sub>3</sub> was coated on reticulated alumina filters with a spec of 3 ppm Cr or less

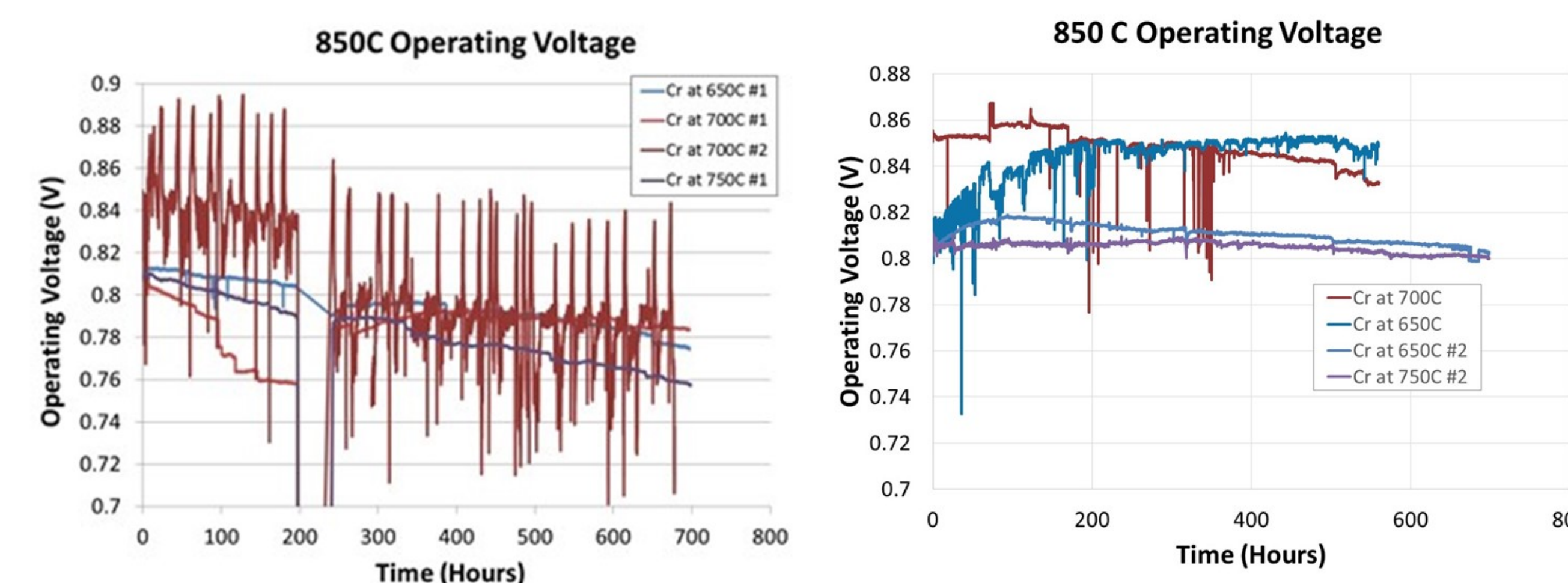


Thermodynamic calculations predict a reduction in the concentration of Cr-species in the air by more than 8 orders of magnitude due to Cr capture

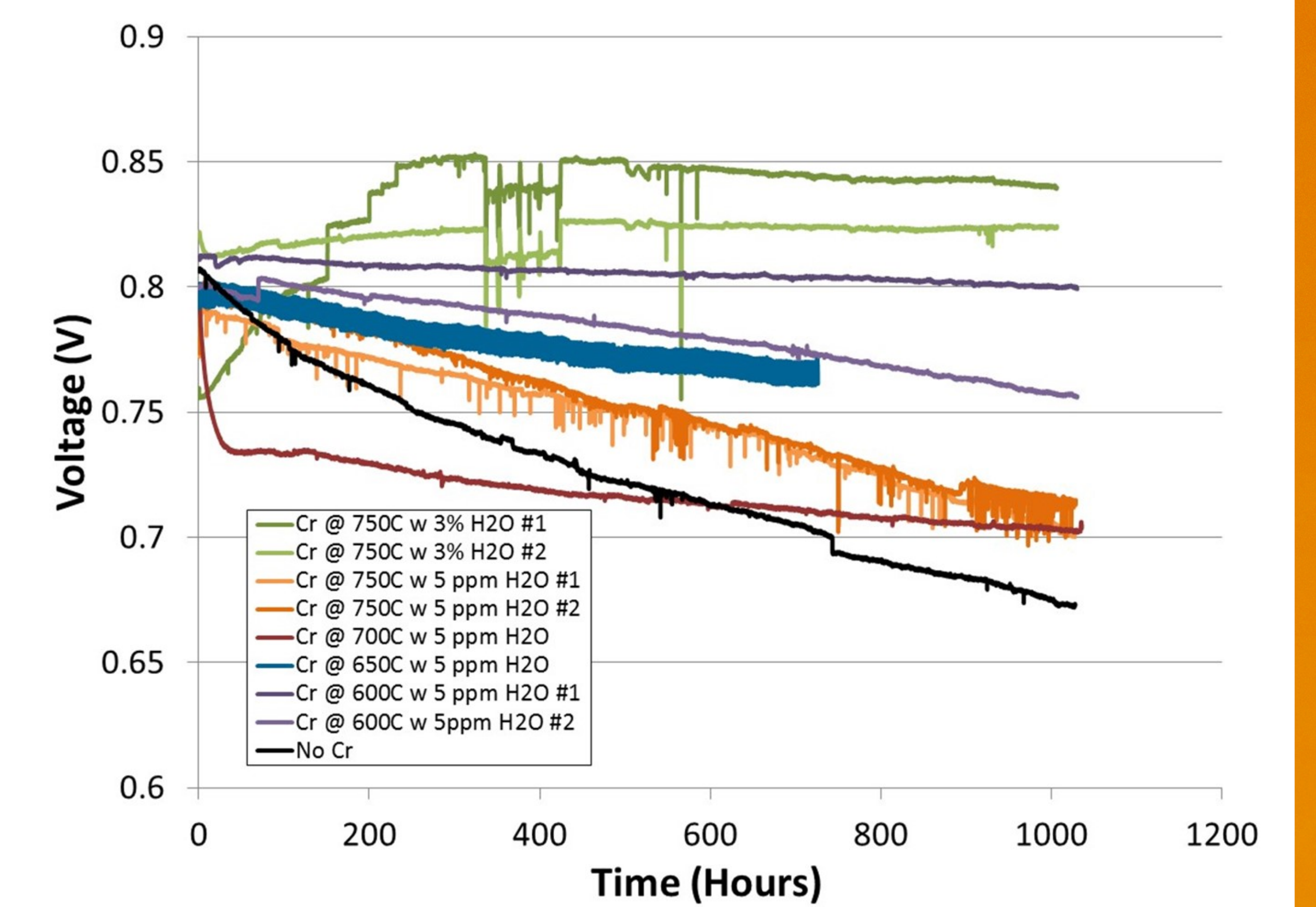
## LSM-YSZ BUTTON CELL TEST RESULTS



## ADDITIONAL LSM-YSZ CELL TESTS AT INTERMEDIATE Cr LEVELS



## LSCF BUTTON CELL TEST RESULTS



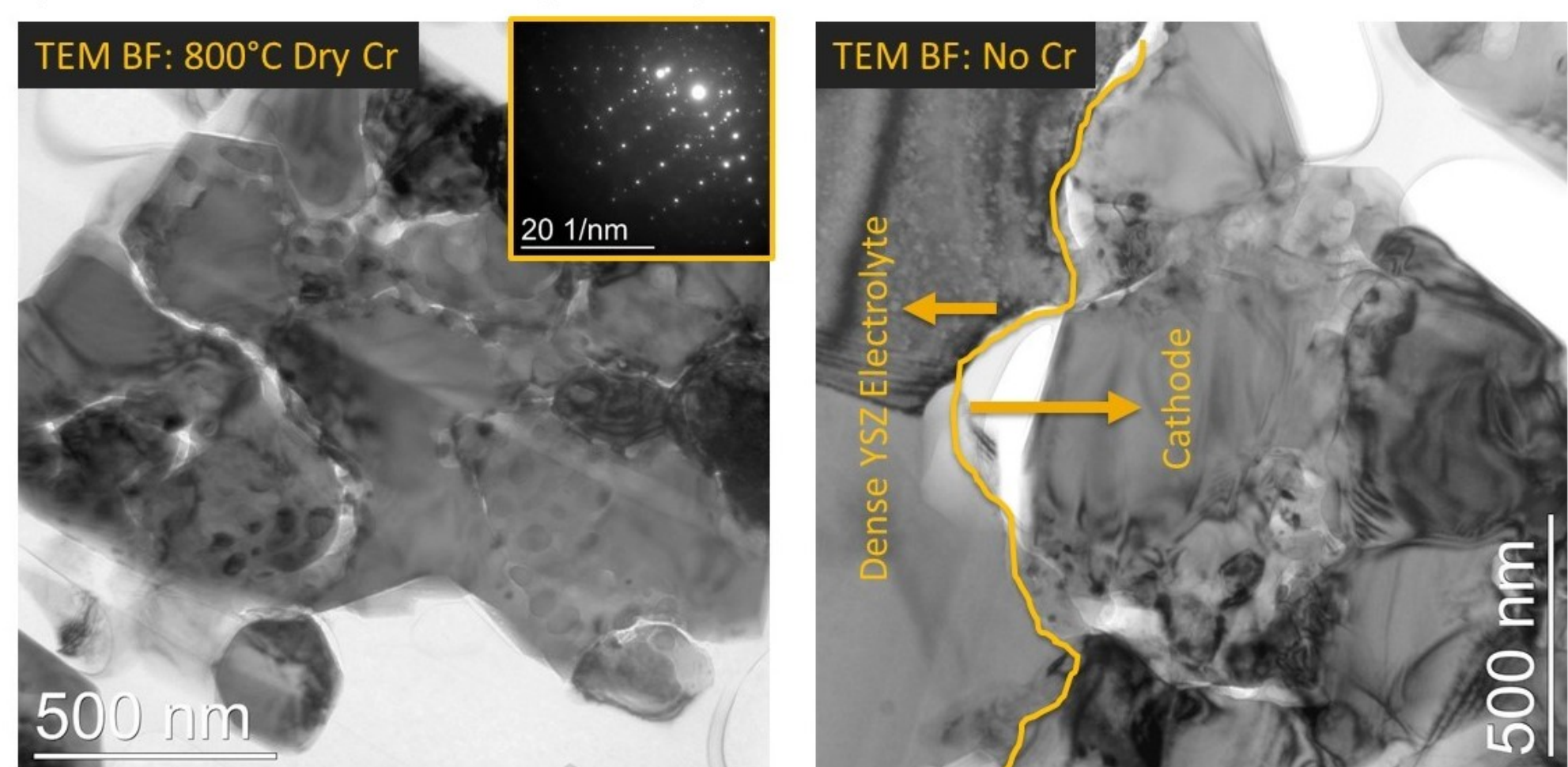
Average Degradation Rates (per kh)

Test Condition	Last 250 h
No Cr	11.2%
≤4 ppt Cr	2.9%
≤39 ppt Cr	3.8%
≤47 ppt Cr	3.7%
≤93 ppt Cr	10.4%
1-2 ppb Cr	1.3%

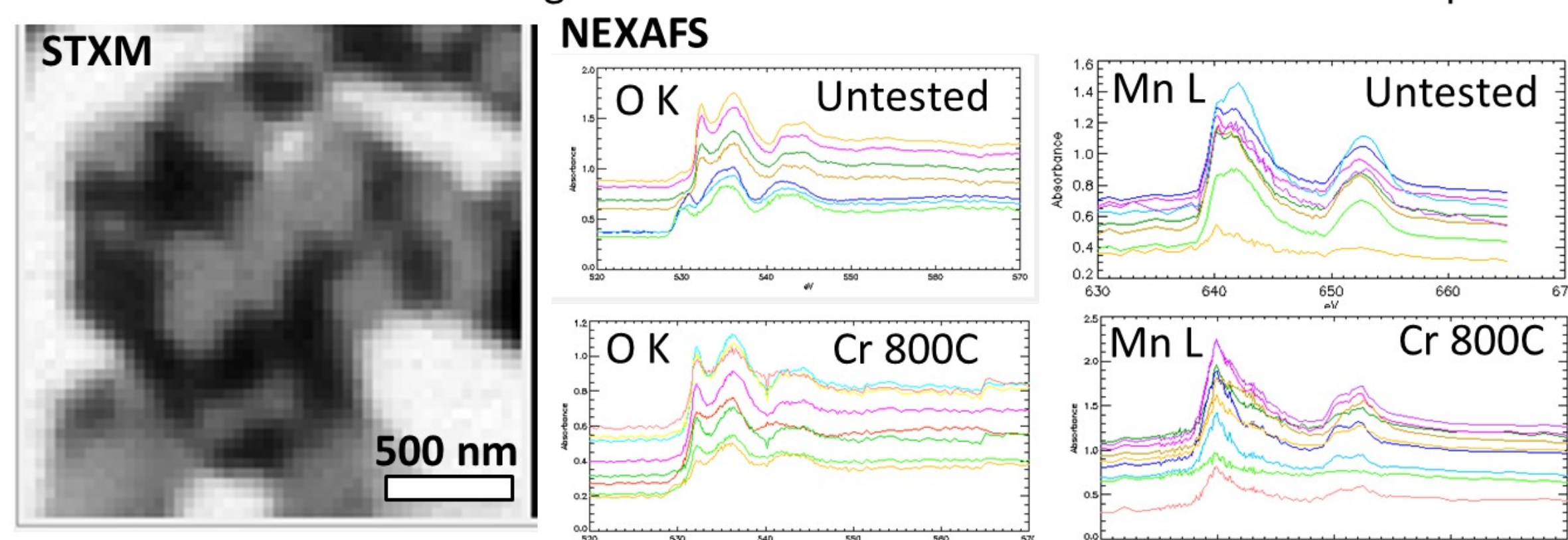
For LSCF cathodes, there was no direct relationship between Cr-content and degradation rate like there had been for LSM-YSZ cathodes. Instead, the highest degradation rate occurred with no Cr exposure, while the lowest degradation rate occurred under the highest Cr vapor concentration.

## SEEKING MICROSCOPIC EVIDENCE OF Cr AFTER HIGH Cr TESTS

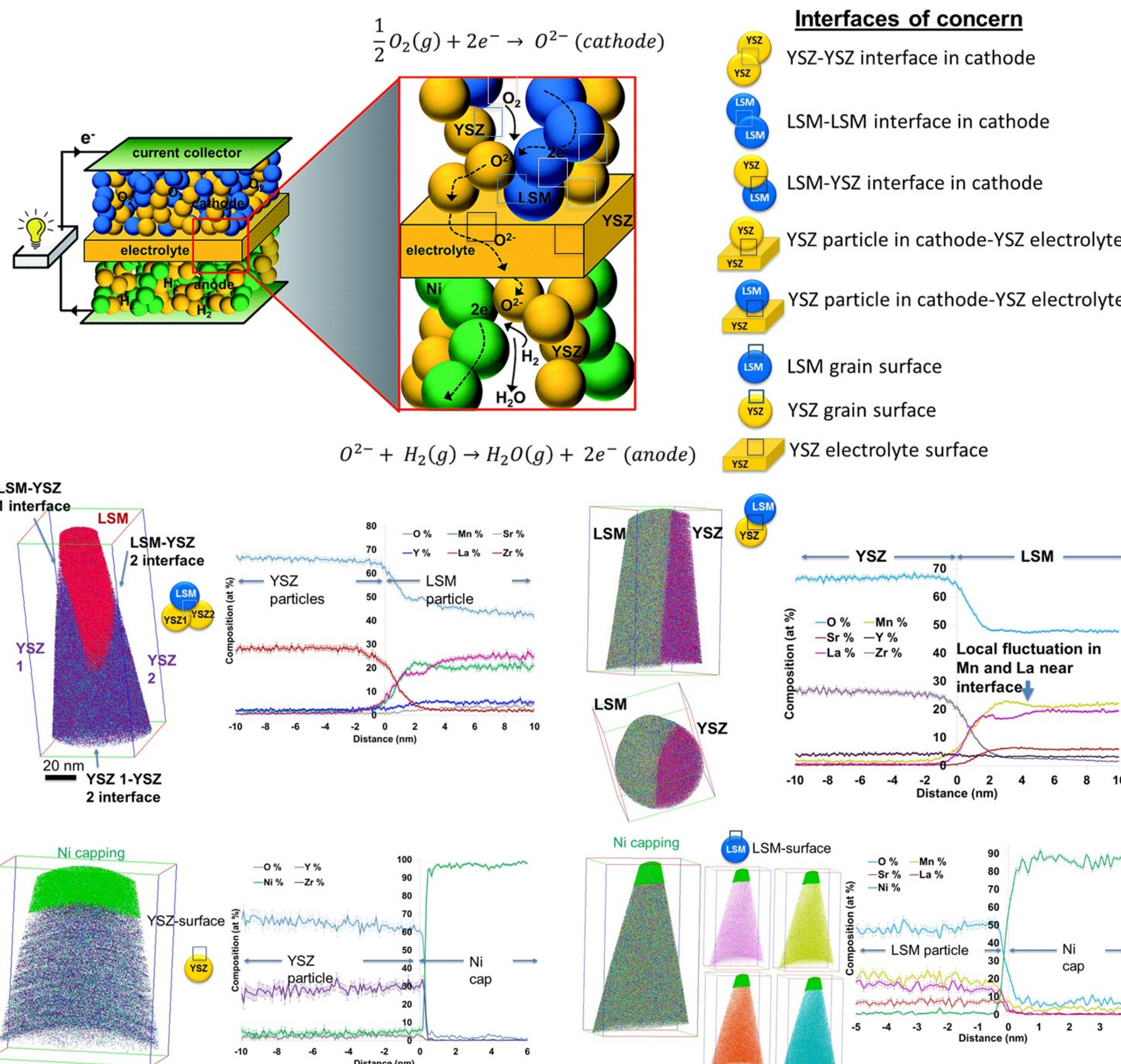
- SEM-EDS and SEM-WDS were not able to detect Cr.
- TEM did not resolve Cr, but did suggest more attack at interfaces between grains and potentially small precipitate formation during Cr exposure:



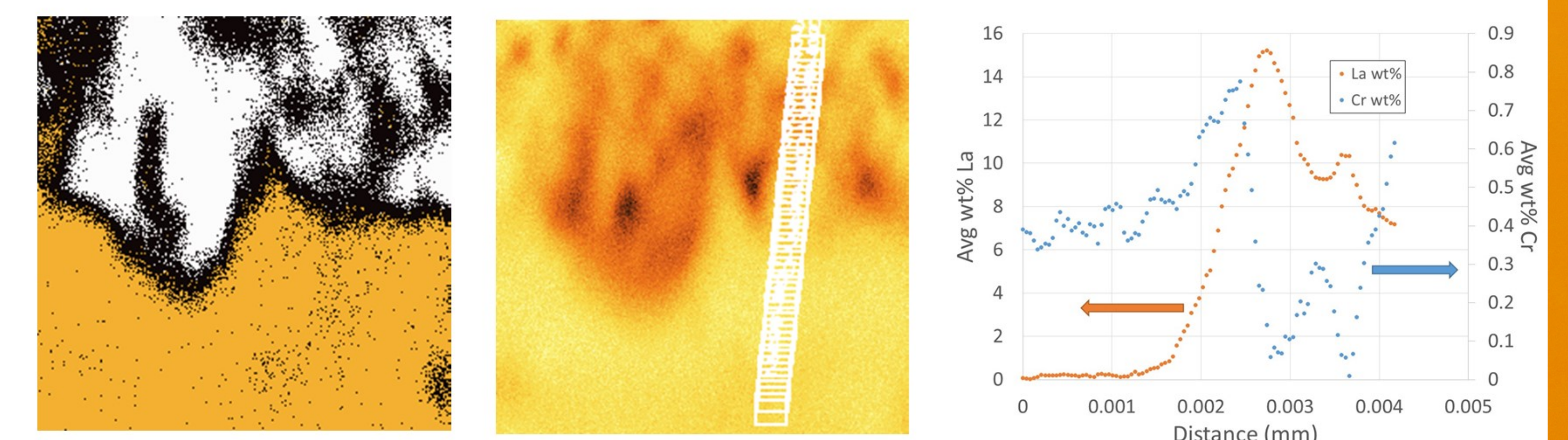
- STXM and NEXAFS at Advanced Light Source, LBNL used to probe the change in oxidation state of Mn and O before and after testing with Cr in air found increased Mn<sup>2+</sup> after Cr exposure:



## ATOM PROBE TOMOGRAPHY CAMPAIGN DID NOT LOCATE Cr



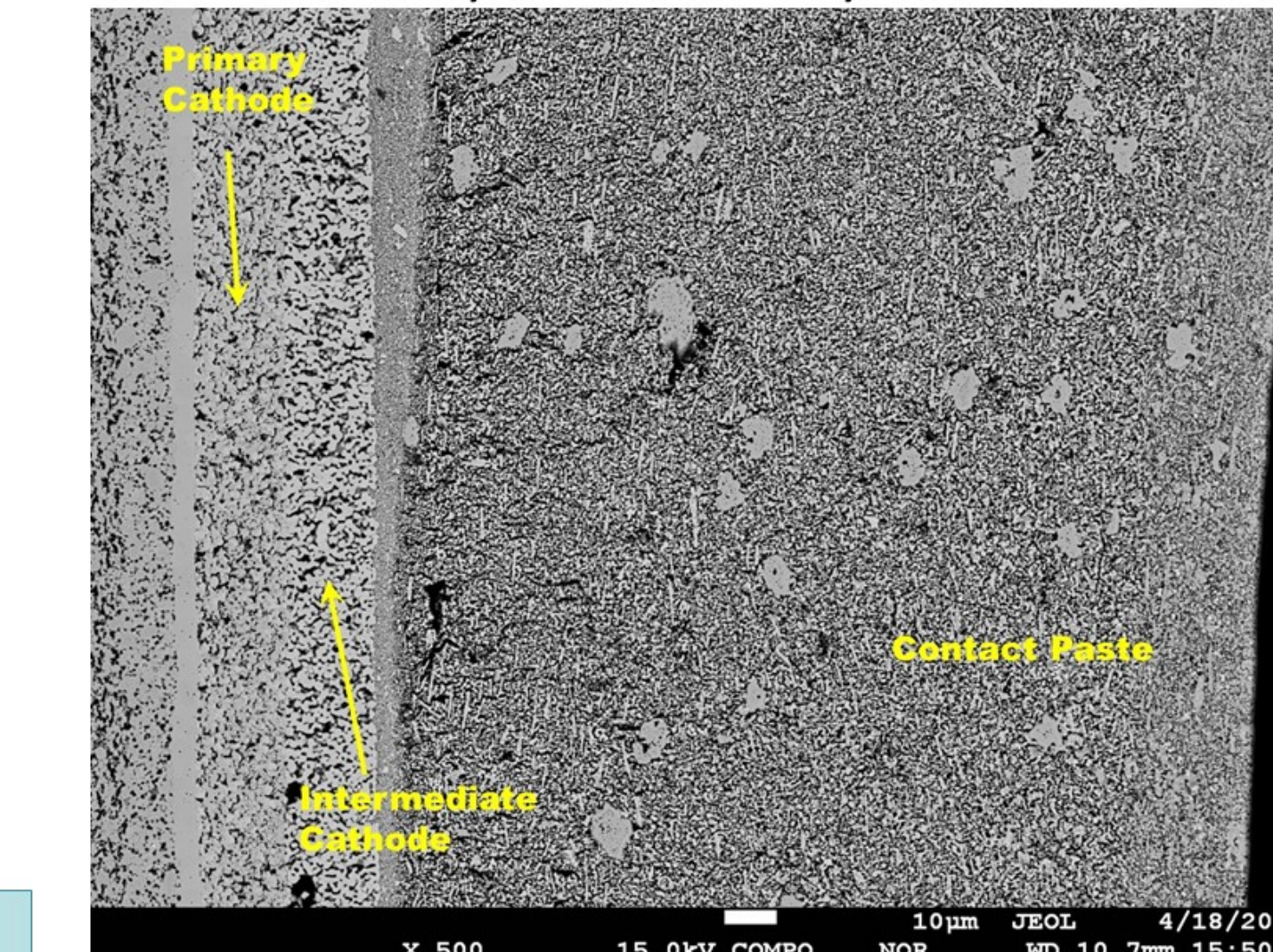
## MICROPROBE ANALYSIS CONFIRMS PRESENCE OF Cr



Cr appears to be associated with the LSM phase. However, there was some concern that the Cr signal may arise from overlapping Cr and La peaks as opposed to actual Cr.

## EVALUATED MICROPROBE'S RESOLUTION OF Cr FROM La

Additional analysis was performed on a cell exposed to excessive levels of Cr in unrelated tests. Prior SEM-WDS analysis had already detected Cr in this cell.



10 line scans were performed in each labeled portion of the cell with Cr and La being analyzed simultaneously. The average at% values were:

	Cr	La	Cr/La
Primary Cathode	0.298	9.885	0.0301
Intermediate Cathode	0.029	31.137	0.0009
Contact Paste	0.372	17.291	0.0215

The results from the intermediate cathode show that the background Cr level caused by peak overlaps with La would be no greater than 0.03 at% even with La as high as 31 at%.

## SUMMARY

- LSM-YSZ cathodes exhibited a direct relationship between Cr in air and degradation
- Threshold concentration below which LSM-YSZ cathode stability is unaffected is somewhere below 165 ppt. Precise determination is hindered by baseline Cr.
- LSCF cathodes did not show direct correlation between Cr in air and degradation.
  - Highest degradation with no Cr in air; Lowest degradation with most Cr in air.
- 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 Mn<sup>2+</sup>
  - APT tests did not discover Cr
  - Microprobe mapping finally found Cr associated with the LSM phase.

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