

# Cathode Infiltration

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*Core Technology Program – Electrodes and Contaminant Issues*

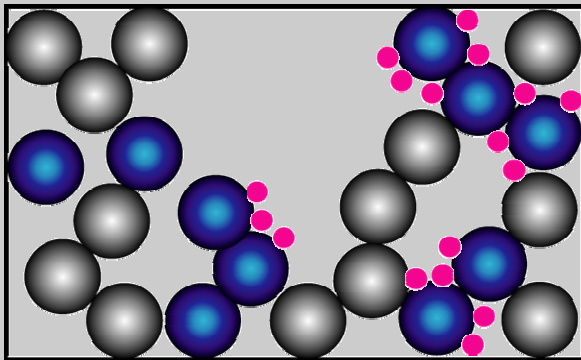
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# Improvement of Air Electrode Low Temperature Performance

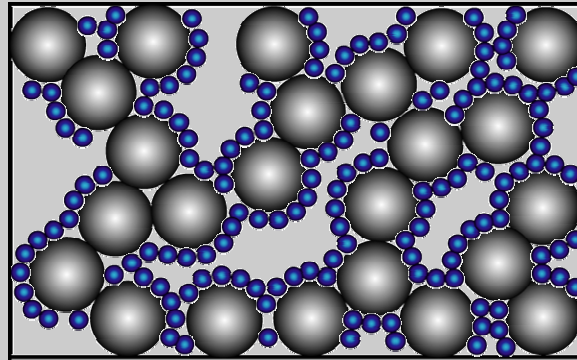
Simple metal nitrates



electrolyte

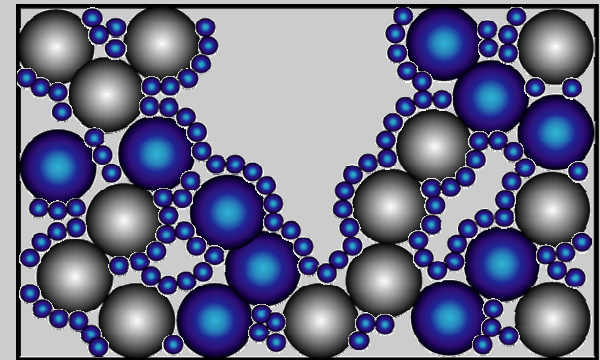
Introduce oxides that would otherwise react at the firing temperature:  $\text{Sm}_{0.6}\text{Sr}_{0.4}\text{CoO}_{3-x}$ ,  $\text{La}_{0.6}\text{Sr}_{0.3}\text{Co}_{0.8}\text{Fe}_{0.2}\text{O}_3$ , etc.

Surfactant dispersed electrode precursors



electrolyte

Porous electrolyte matrix



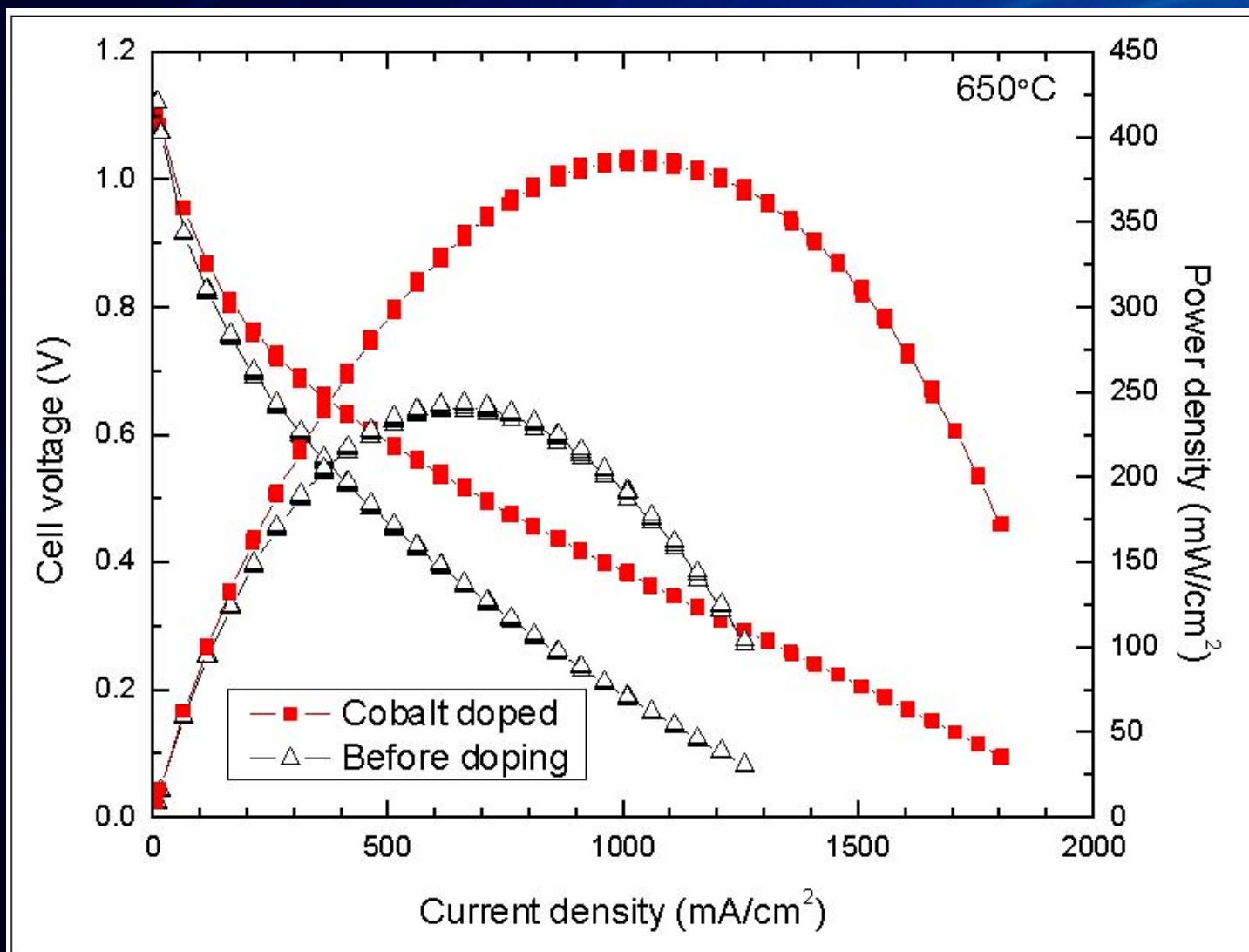
electrolyte

Composite Commercial electrodes (YSZ-LSM)

● electronic conductor  
● ionic conductor

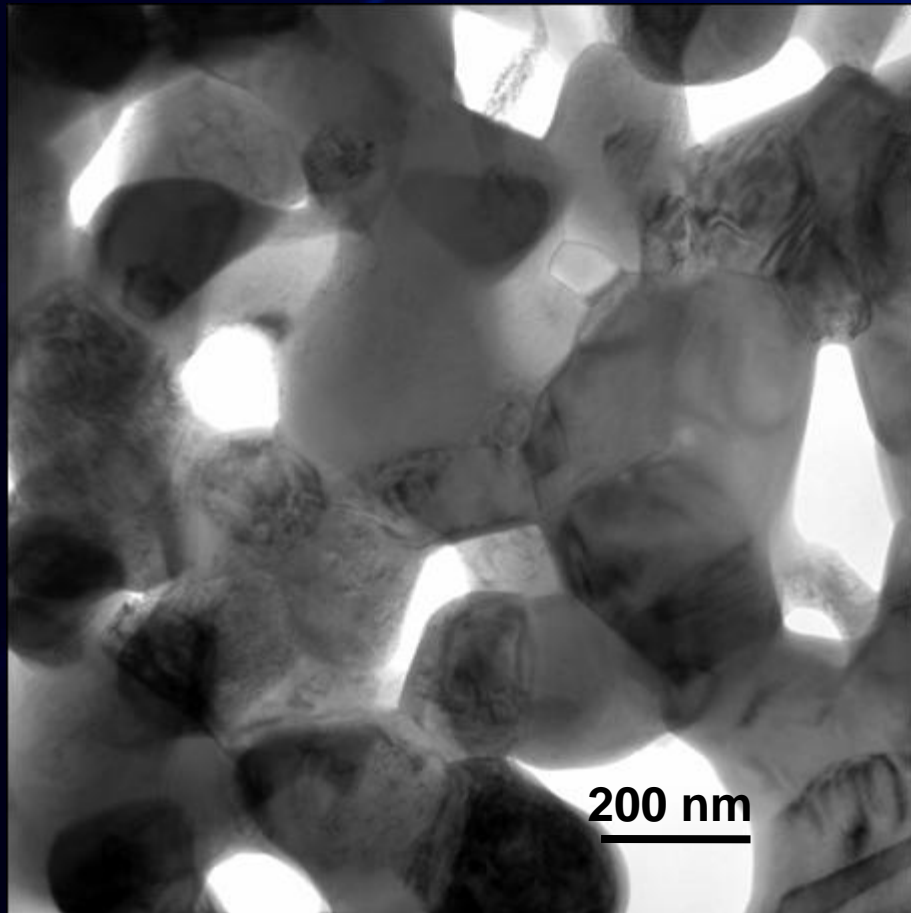
● catalysts

Power density was improved by as much as ~2 times at 650°C by cobalt doping of cathode using a simple infiltration method.

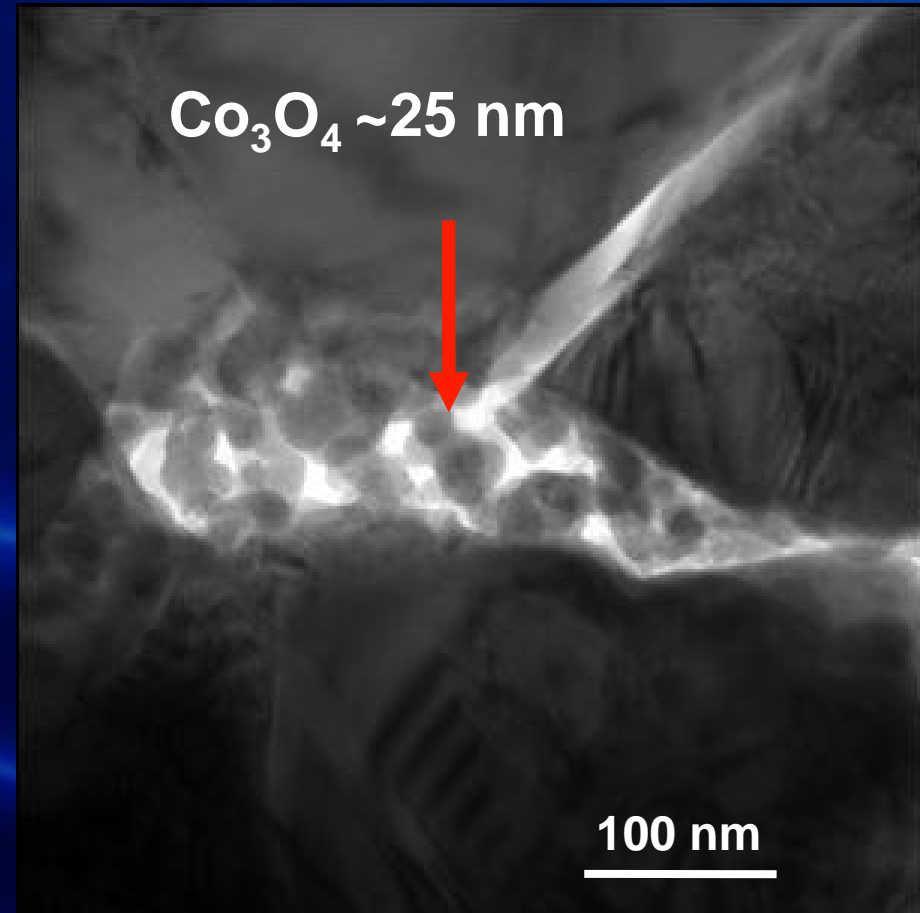


# TEM: $\text{Co}_3\text{O}_4$ Particles in the Pores of LSM-10Sc1YSZ Composite

**Before Doping**

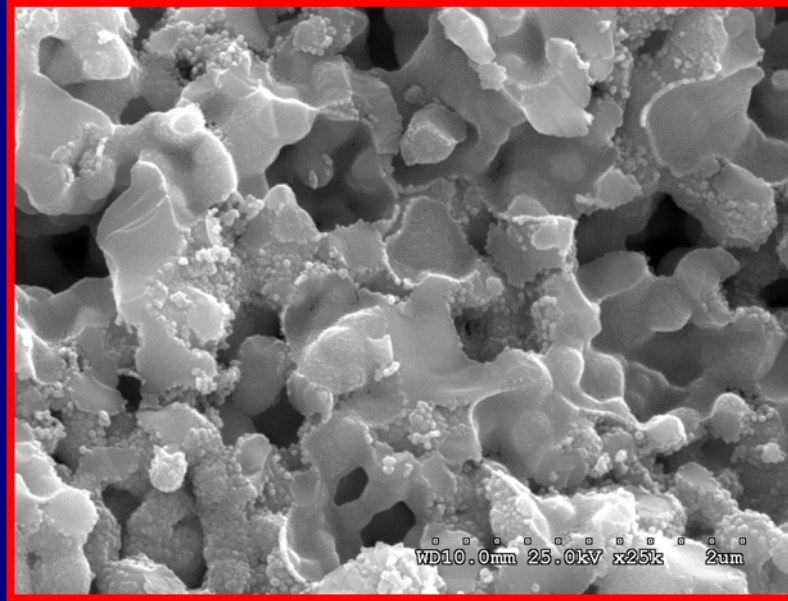
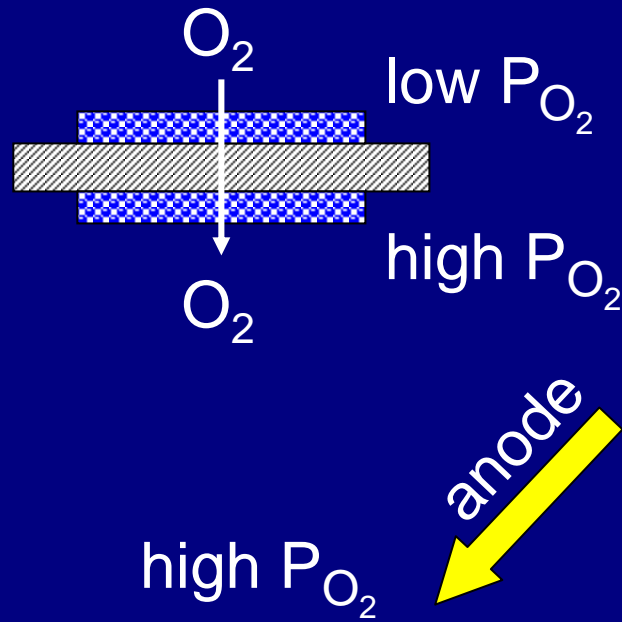


**Cobalt Doped**





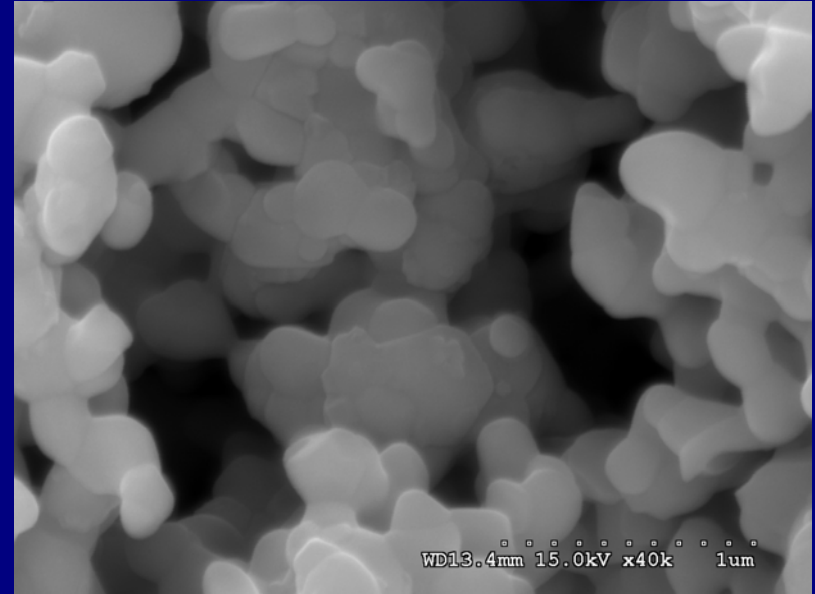
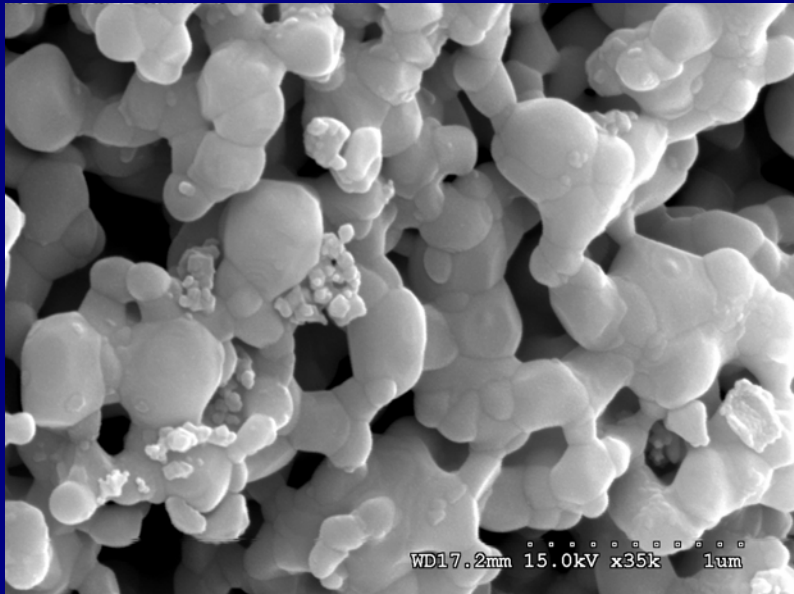
# LSM-YSZ electrode infiltrated with 20mg/cm<sup>2</sup> Co Nitrate



cathode

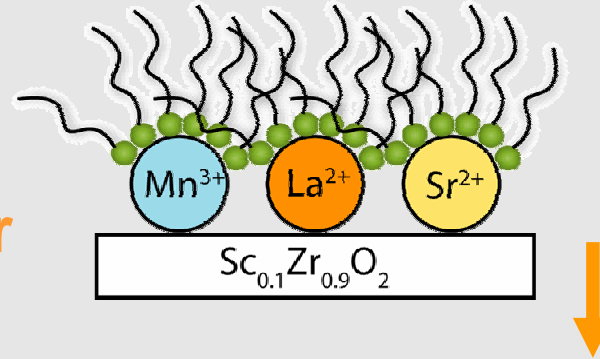
3A/cm<sup>2</sup> at 800°C

low  $P_{O_2}$

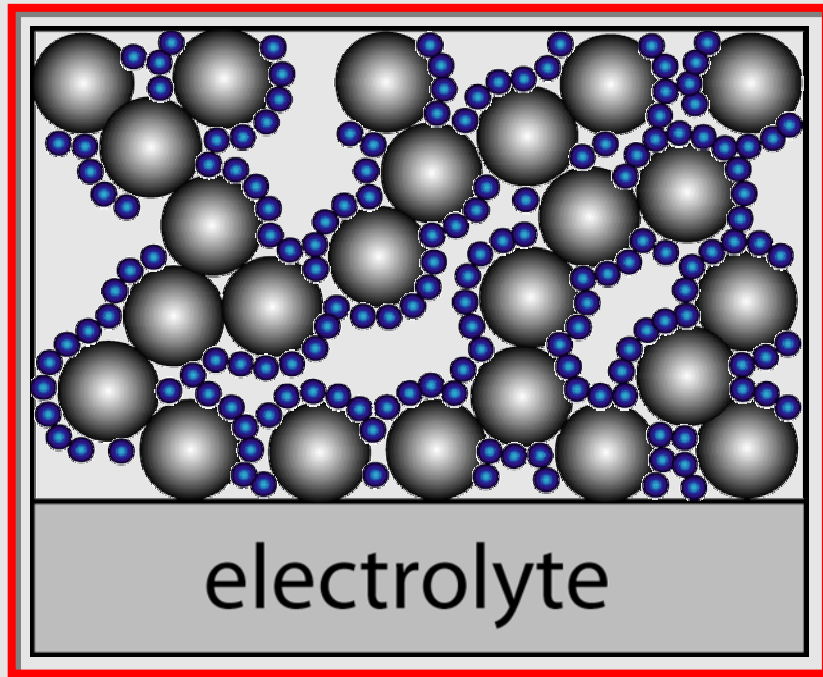


# Infiltration Step

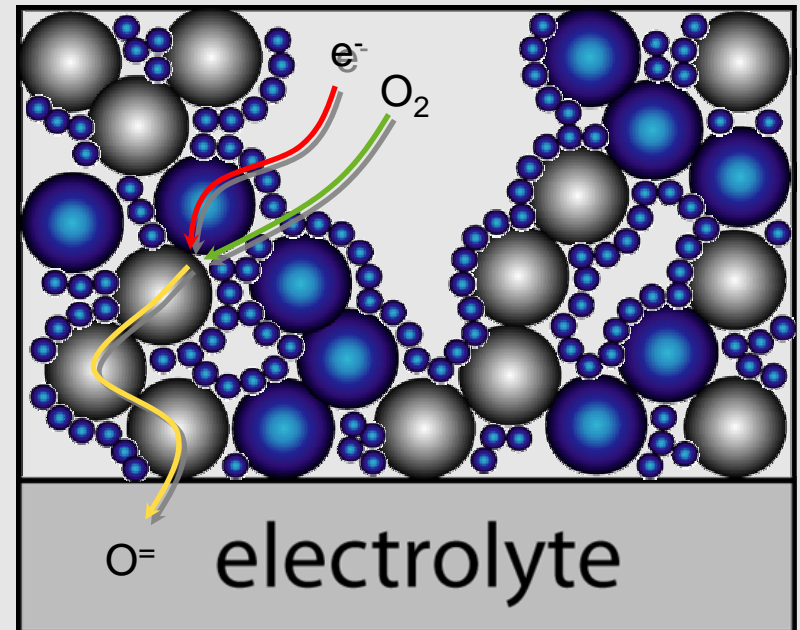
Nitrate-Surfactant  
Concentrated Precursor



Surfactant dispersed Electrode Precursors



Porous electrolyte matrix



Composite Commercial  
electrodes (YSZ-LSM)

● electronic conductor    ● ionic conductor

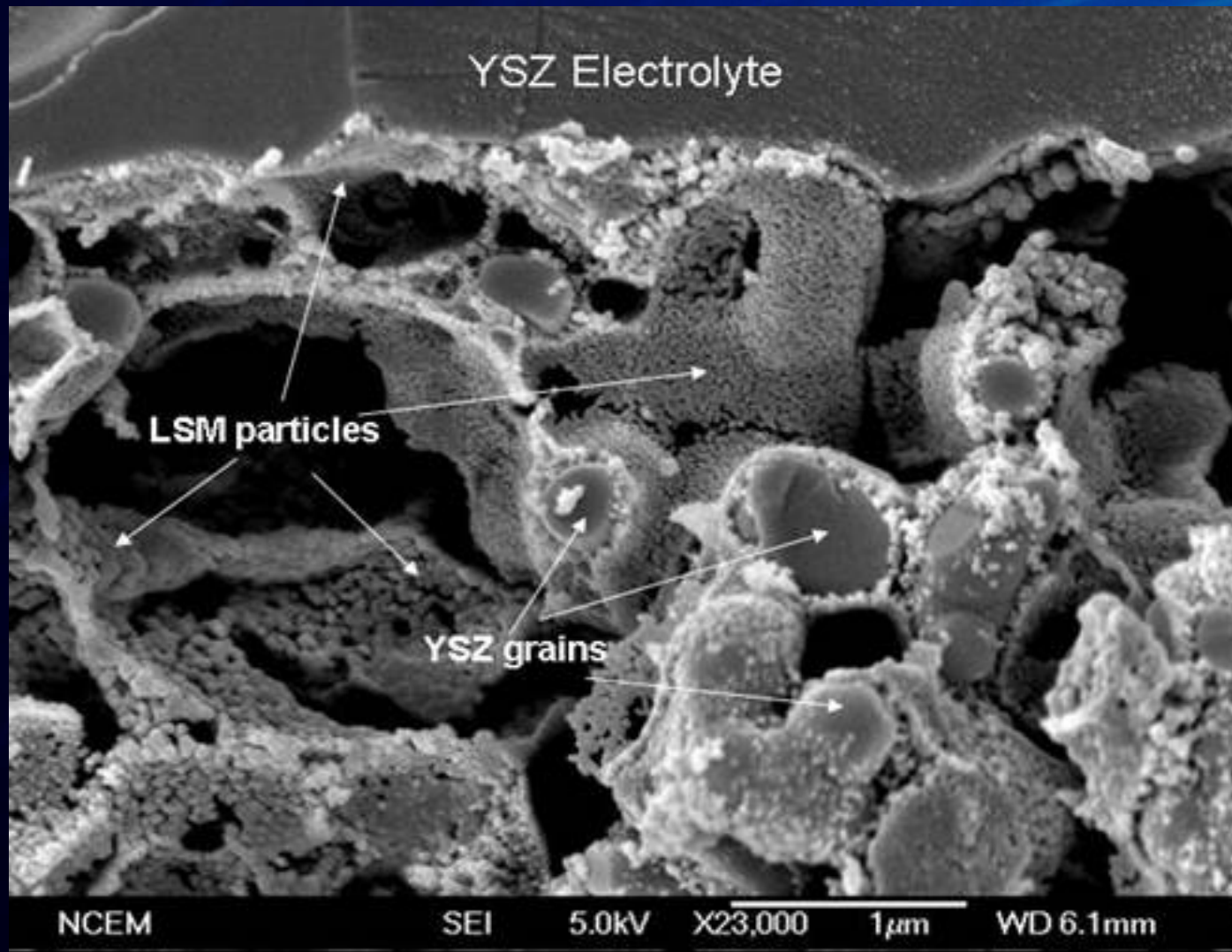
# Nano-particulate SOFCs

- Technology involves vacuum impregnation of concentrated perovskite electrode (or electrolyte) precursor solutions that can generate the entire electrode in a single infiltration step.
- Can be used to fabricate unique electrode microstructures:
  - Nanoparticulate network infiltrated into **porous electrolyte** (allows stack fabrication in reducing atmosphere followed by introduction of perovskite structure) - nanoparticulate network must be continuous for electron path.
  - Nanoparticulate network deposited onto **existing electrode** (to improve low temperature performance) - existing LSM network used for current collection, expands reaction surface area.
- LBNL has used this approach to improve the performance of conventional electrodes and to fabricate unique structures
- LBNL is refining the technology to control the depth of penetration into porous structure and size of resulting nanoparticles



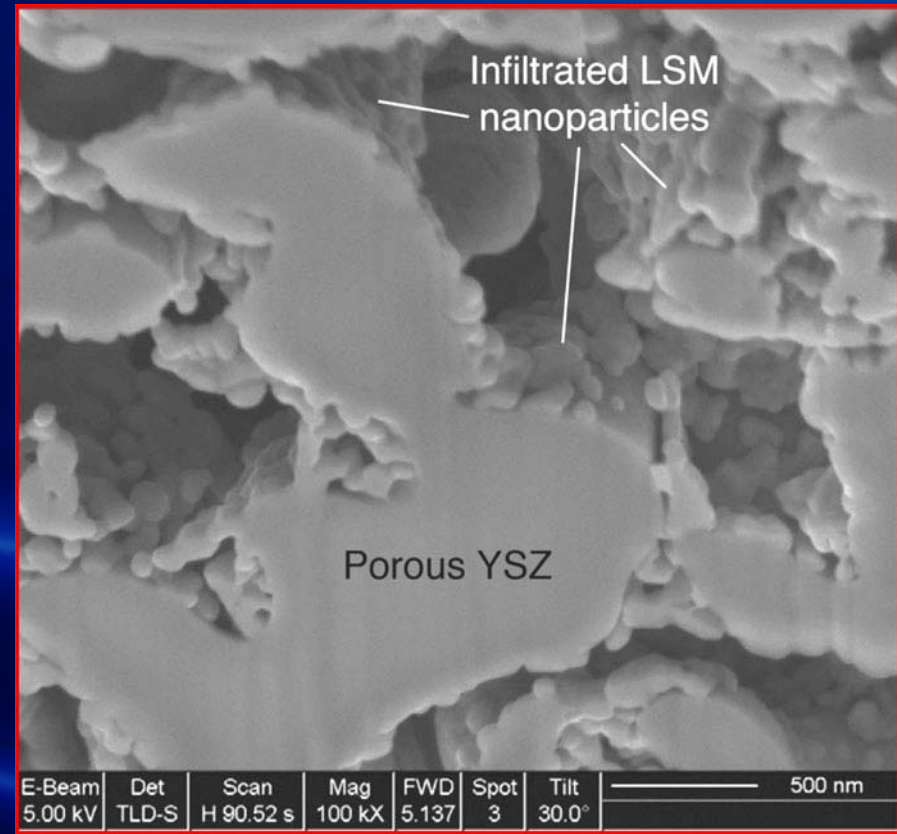


# Nanoparticulate SOFC Electrodes



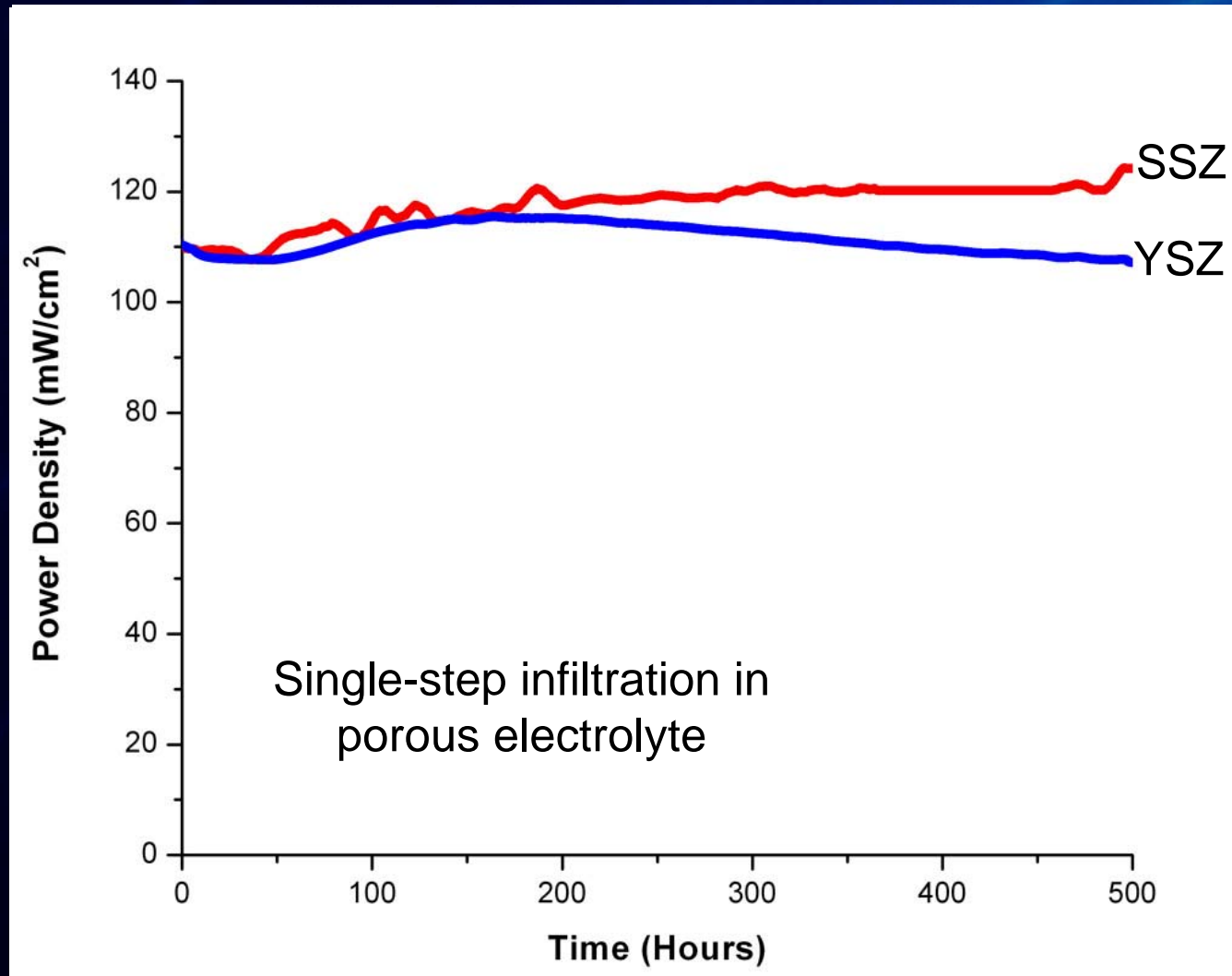


# Focused-ion-beam (FIB) trench showing cross-section of single-step LSM infiltrated thin-film SOFC

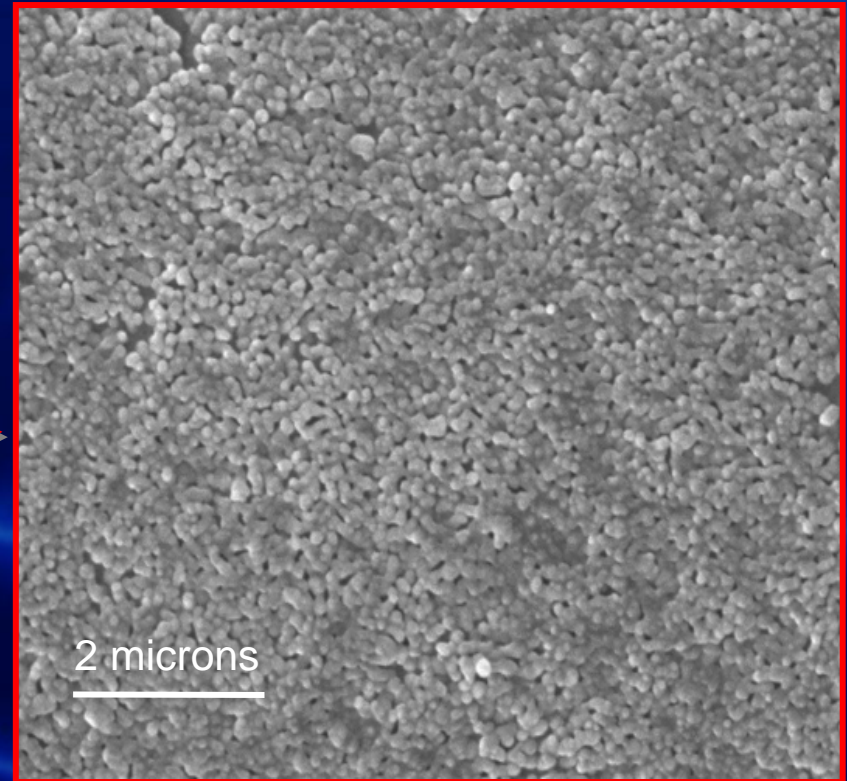
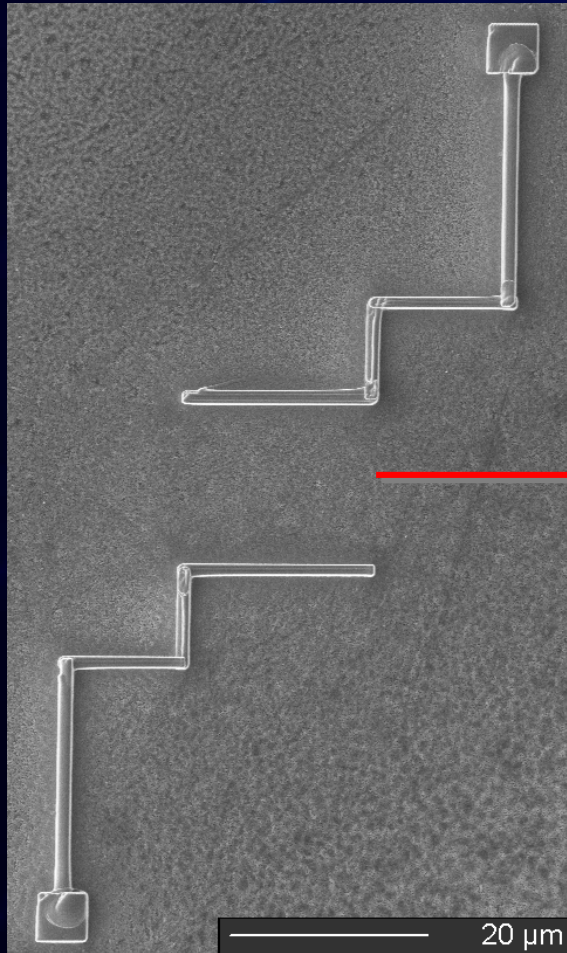


Infiltrated cathode after 500 hours continuous operation at 650 °C

# Performance & Stability of Nanoparticulate Electrodes at 650 °C

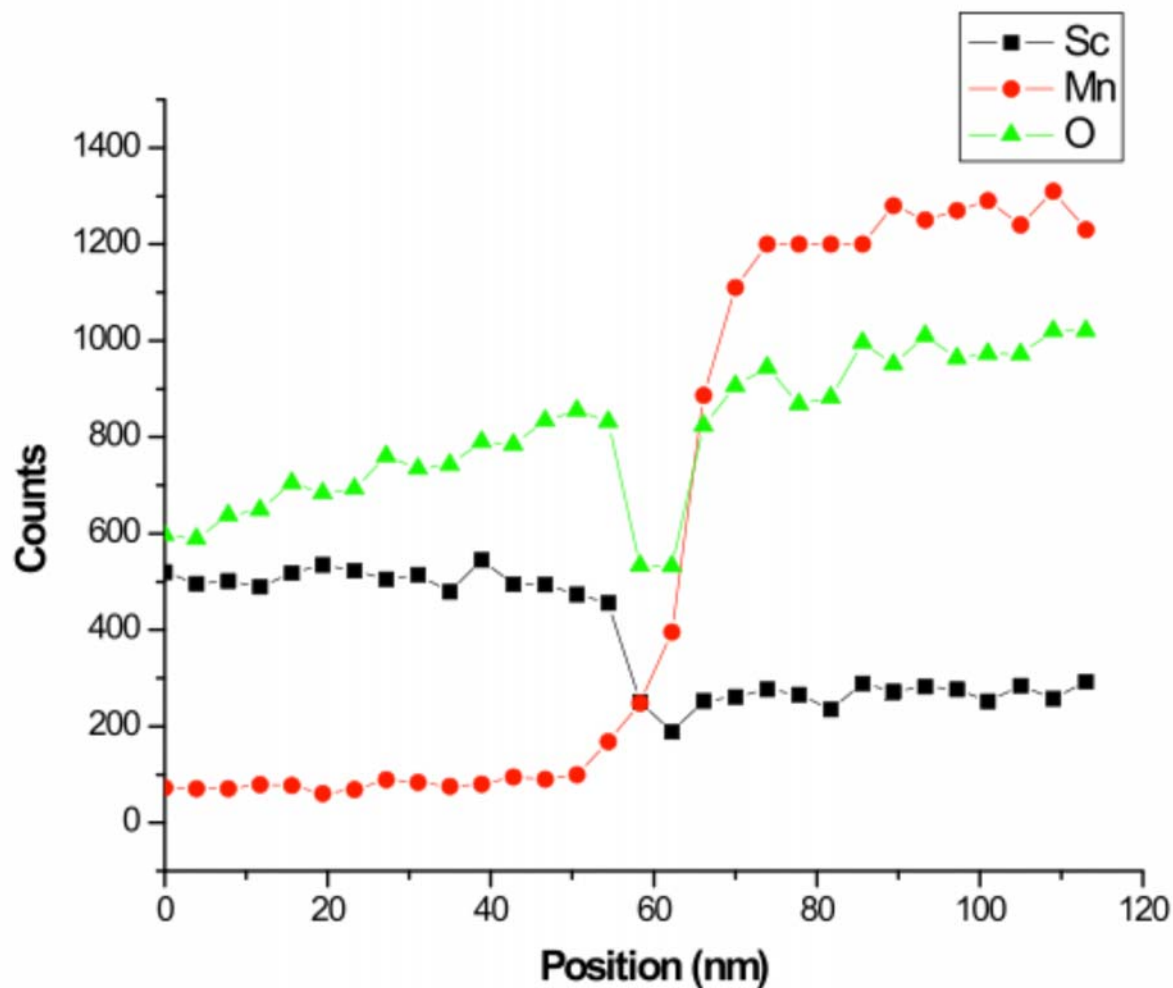


# Supported 2-D Monolayer of Particulate Catalyst LSM Network with Electronic Probes





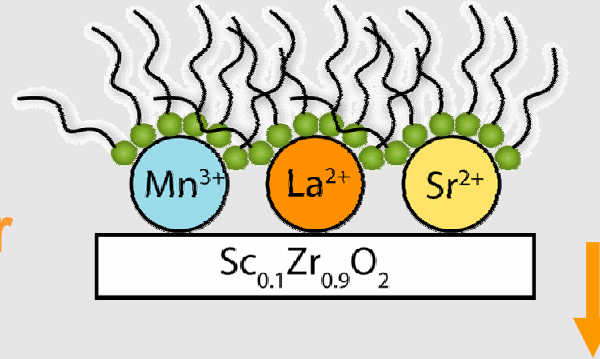
# EDX Line Scan SSZ to LSM



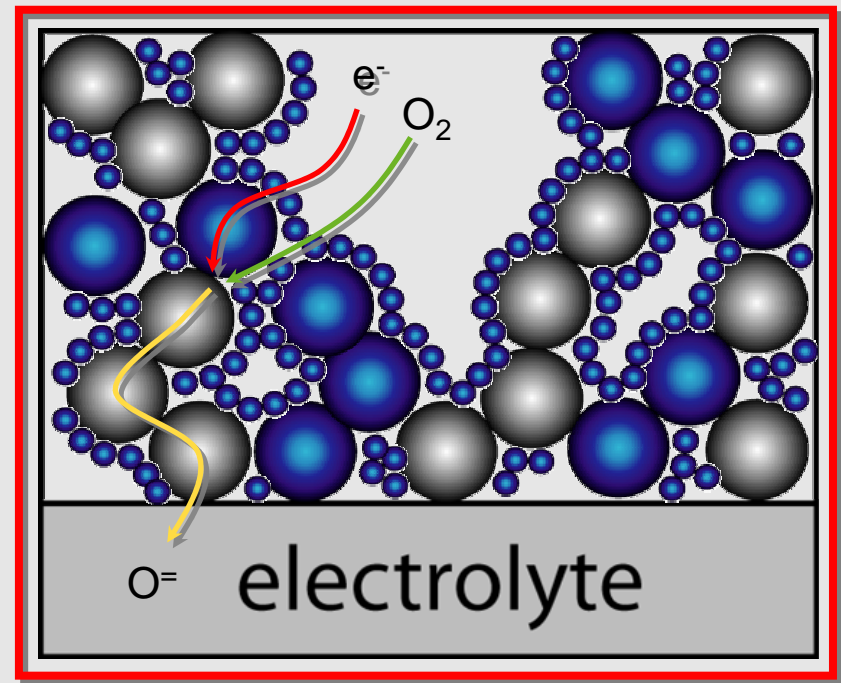
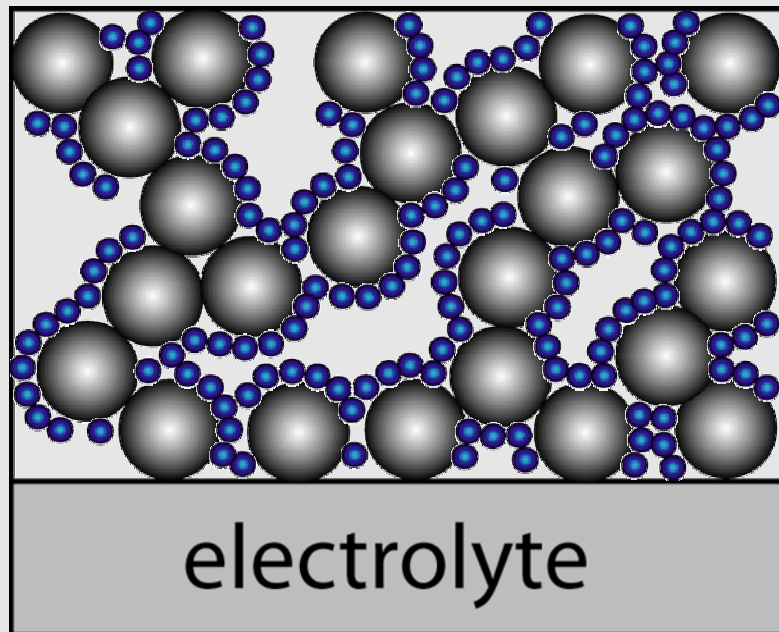


# Infiltration Step

Nitrate-Surfactant  
Concentrated Precursor



Surfactant dispersed Electrode Precursors



Porous electrolyte matrix

Composite Commercial  
electrodes (YSZ-LSM)

● electronic conductor    ● ionic conductor

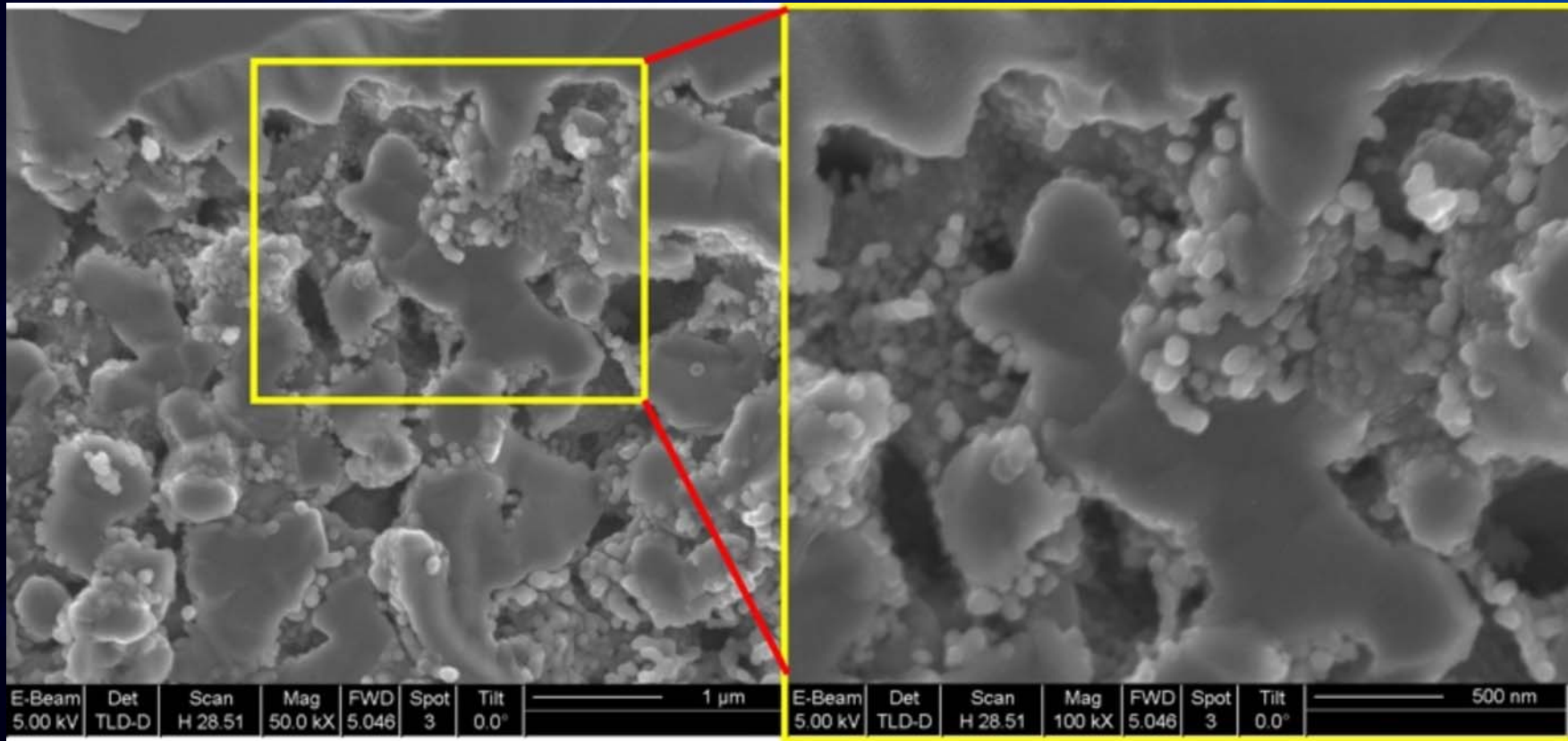
# Commercial Symmetric Electrolyte Supported LSCF Cell from INDEC LSCF-YDC/TZ3Y/YDC-LSCF



# Infiltration of LSM Nanoparticles into LSM-YSZ Composite

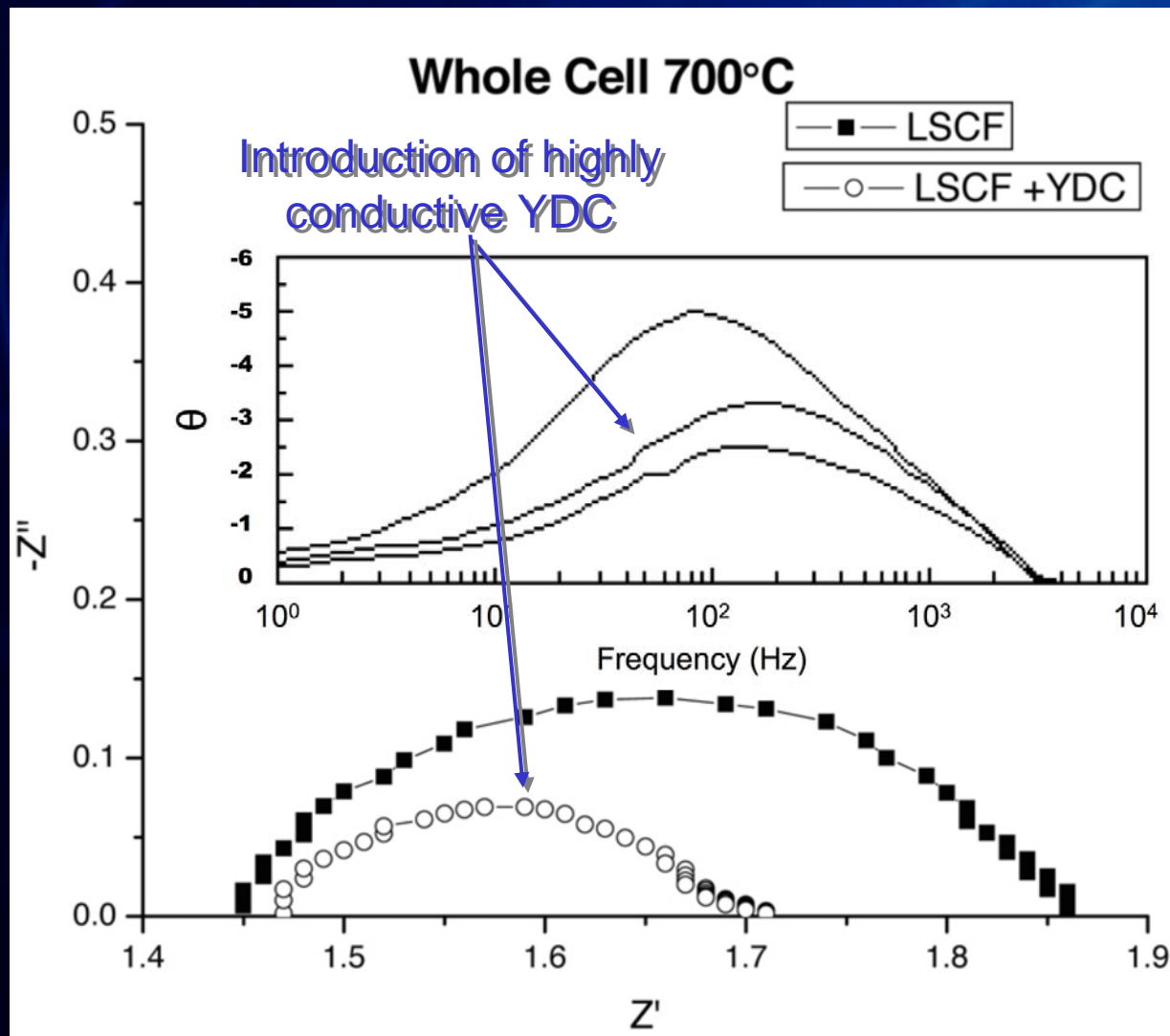
50 kX

100 kX



SEM Image of LSM-YSZ composite sintered at 1200°C for 4 hours, then infiltrated with LSM-surfactant precursor and fired at 900°C for 1 hour

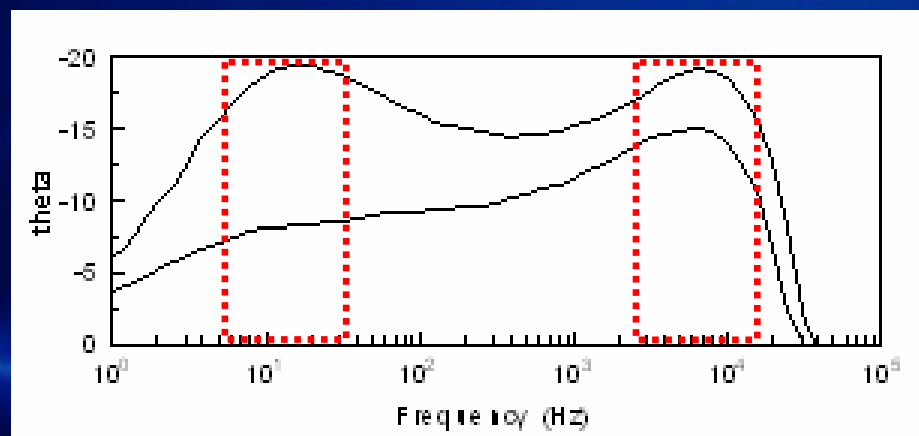
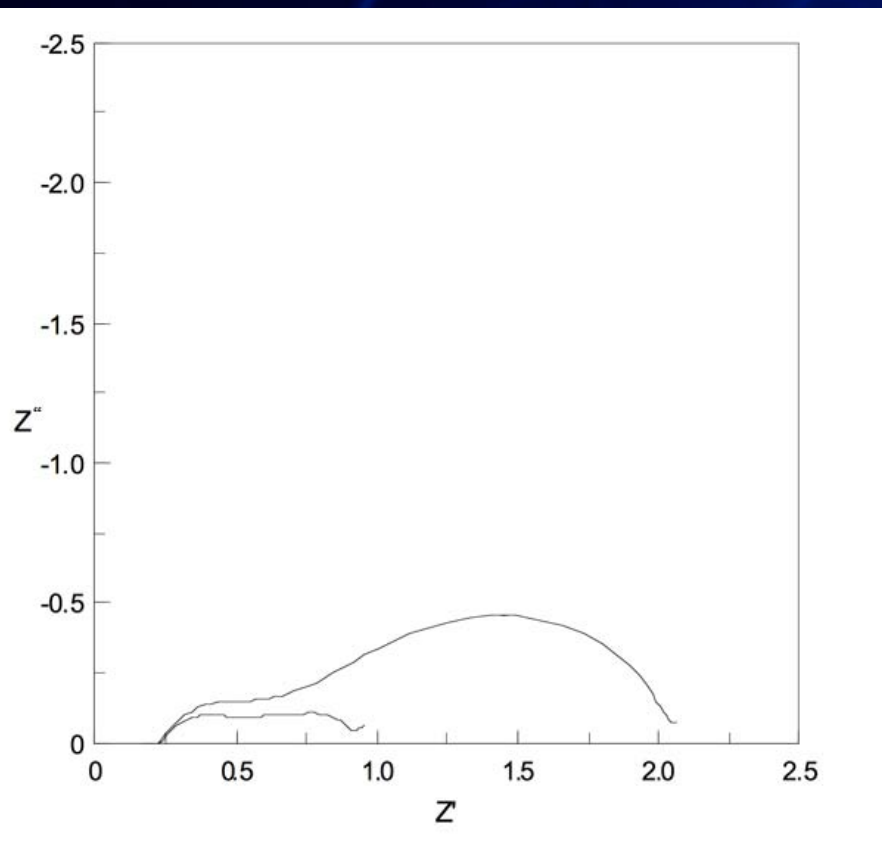
# Commercially Produced Cell - InDec, (LSCF-YDC/TZ3Y/YDC-LSCF) W/ and W/out YDC Connected Nanoparticulate Infiltration





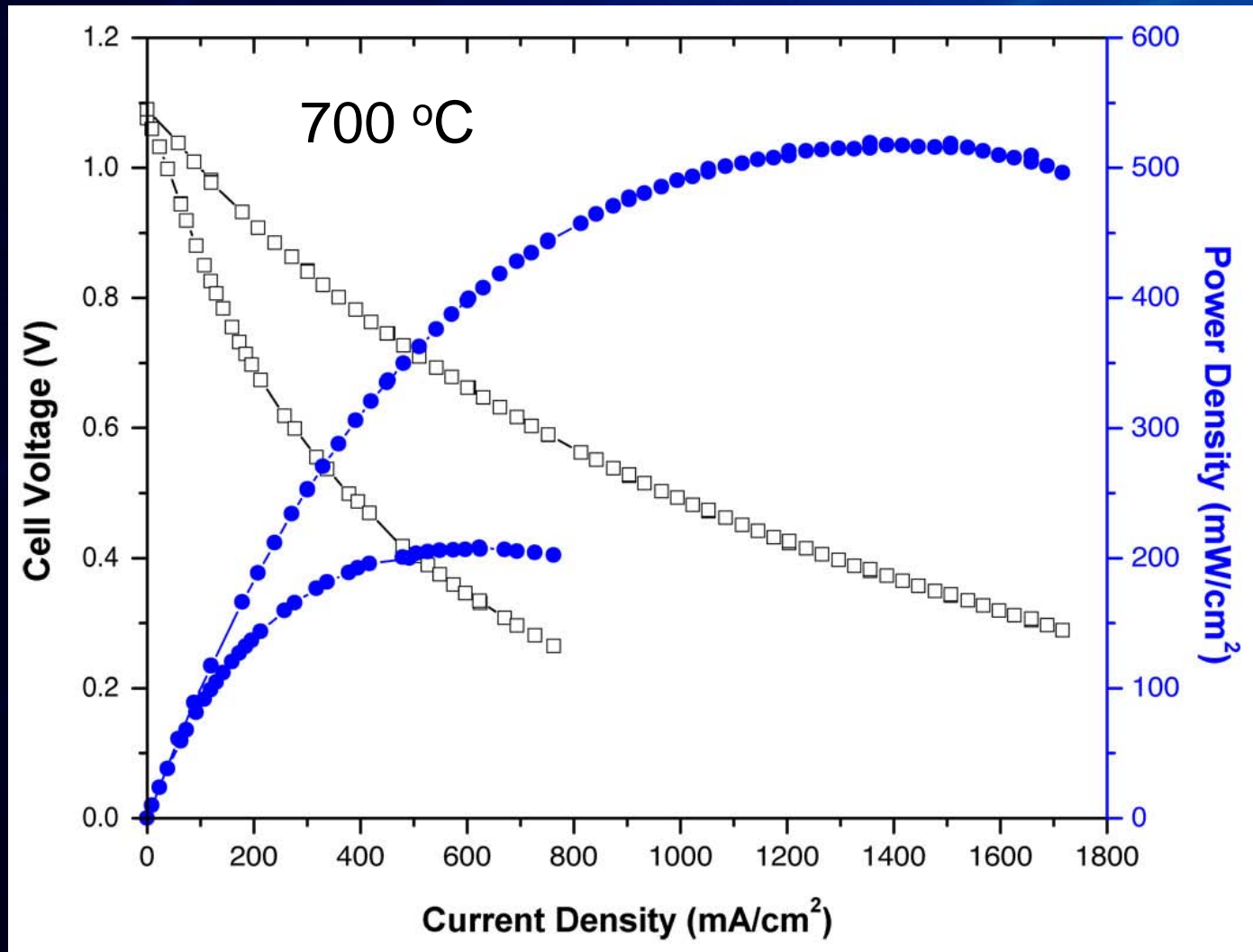
# Anode supported Ni-YSZ/YSZ/LSM-YSZ cell infiltrated with YDC

## Cell Impedance at 700 °C



# Anode supported Ni-YSZ/YSZ/LSM-YSZ cell infiltrated with YDC

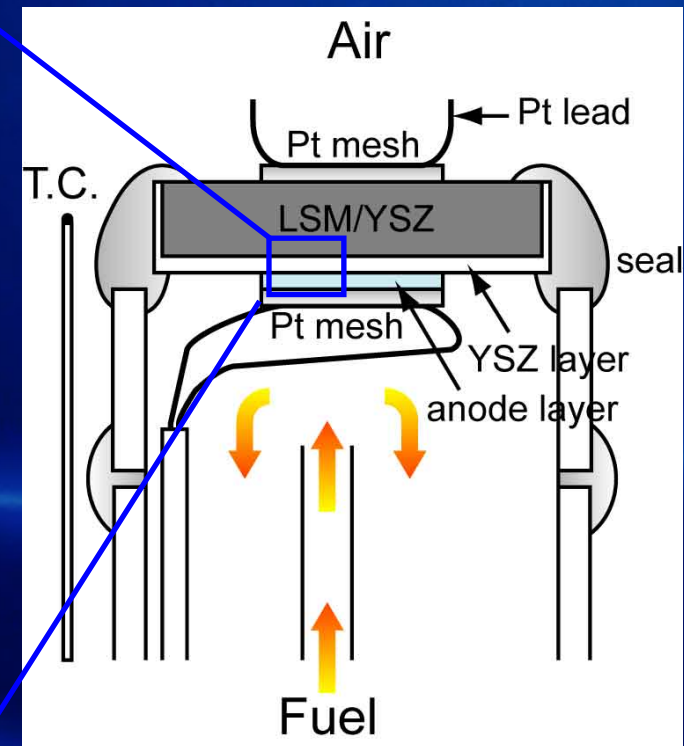
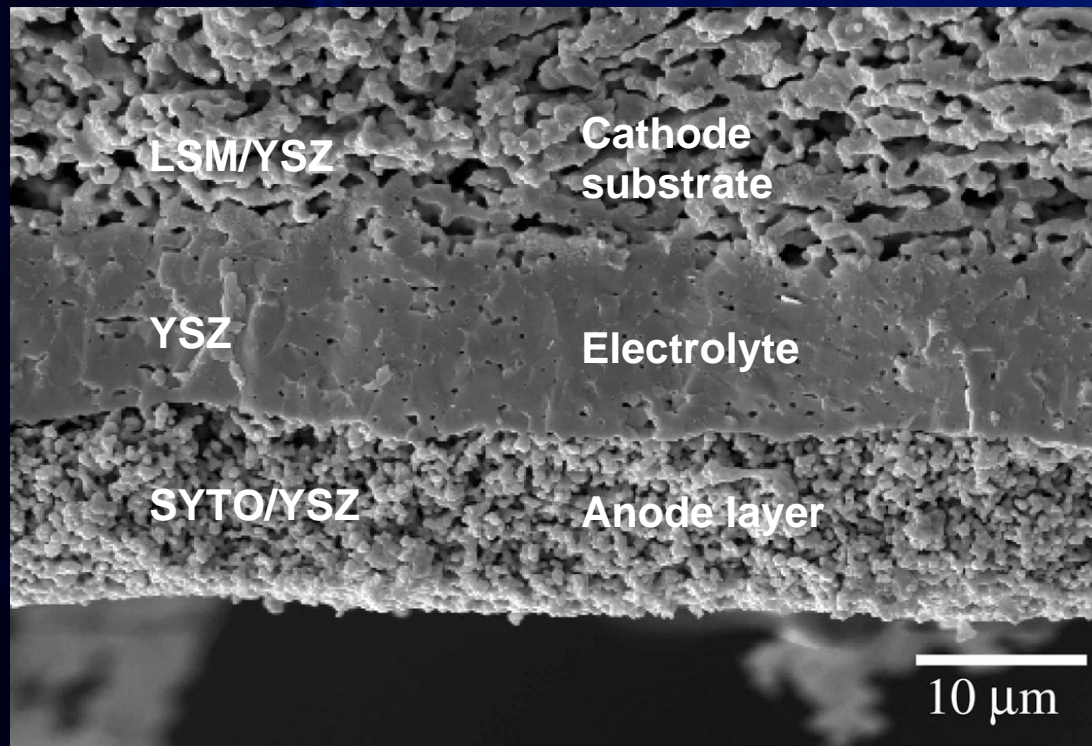
## Cell Performance at 700 °C



## • Sulfur-tolerant Anode with Single-Step infiltration

*Sulfur-tolerant, cathode supported cell with an SYTO/YSZ anode was developed and the performance improved with infiltration of  $\text{CeO}_2$  and Ru.*

Cross section of cathode supported cell



See Ray Gorte's work on SYTO/ $\text{CeO}_2$ /Ru

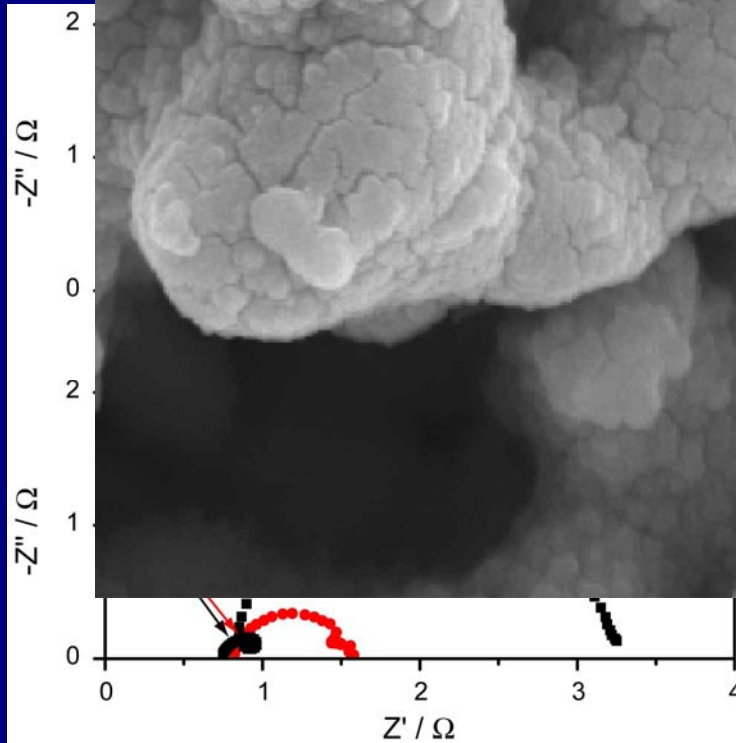
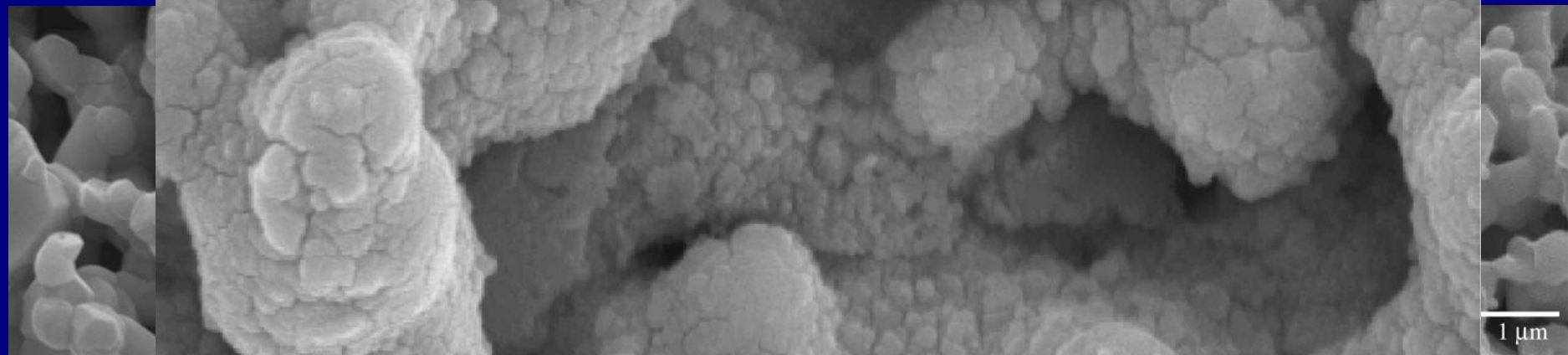
# *The effect of single-step infiltration on sulfur-tolerant anode*

● SYTO/YSZ + dispersed catalytic materials (on 500 $\mu$ m YSZ disk)

SYTO with

SYTO +

SYTO +



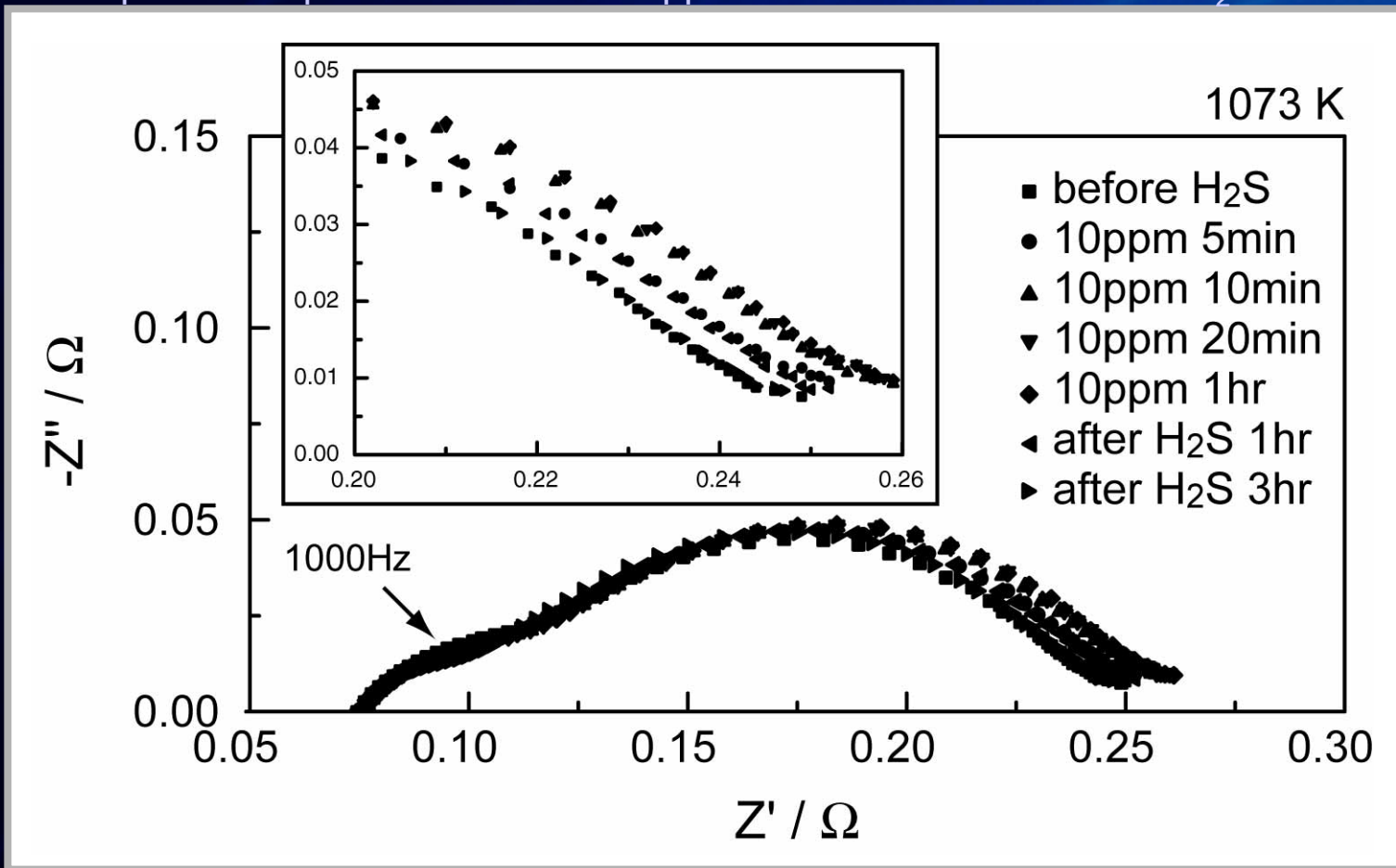
73 K

4



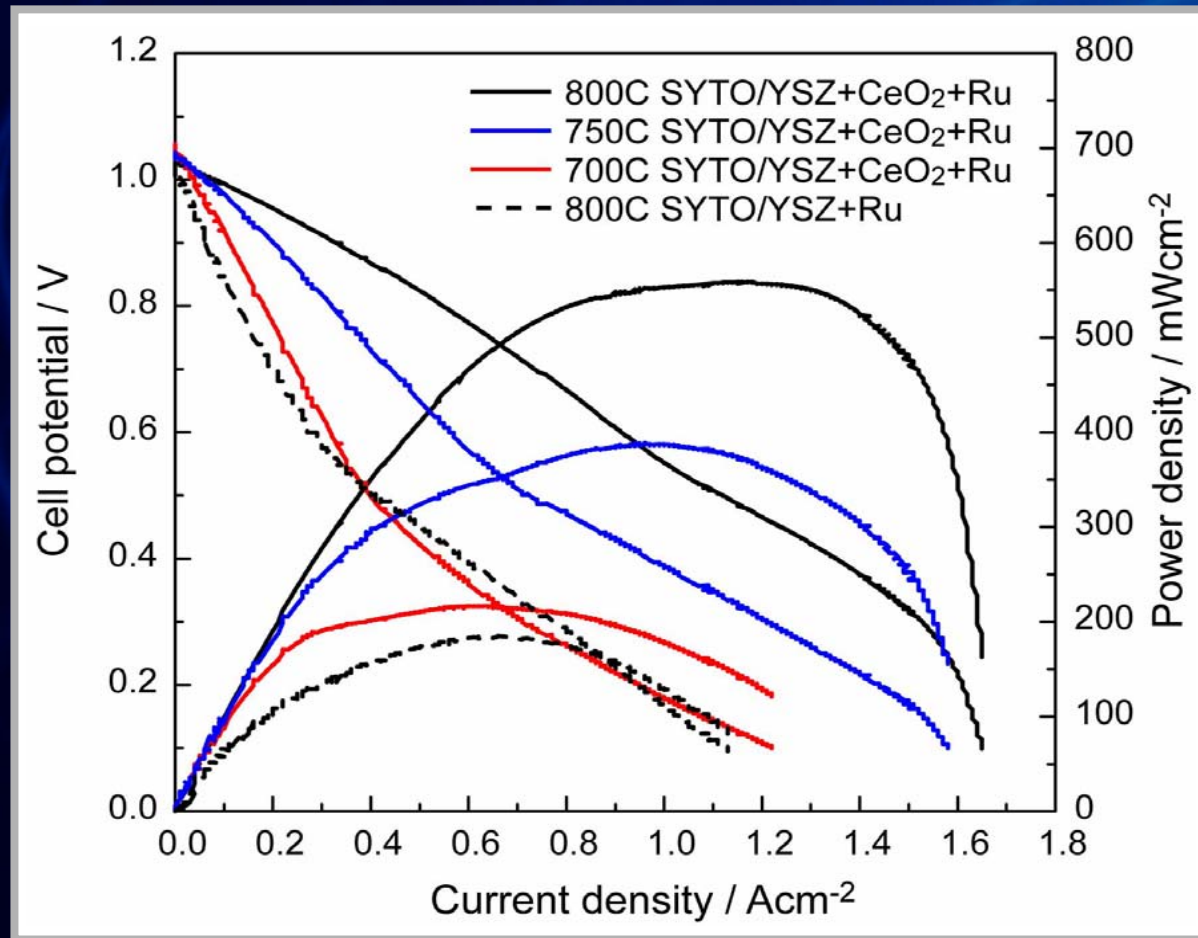
# Sulfur tolerance of SYTO/YSZ+CeO<sub>2</sub>+Ru anode

AC Impedance spectra of cathode support cell with SYTO/YSZ+CeO<sub>2</sub>+Ru anode



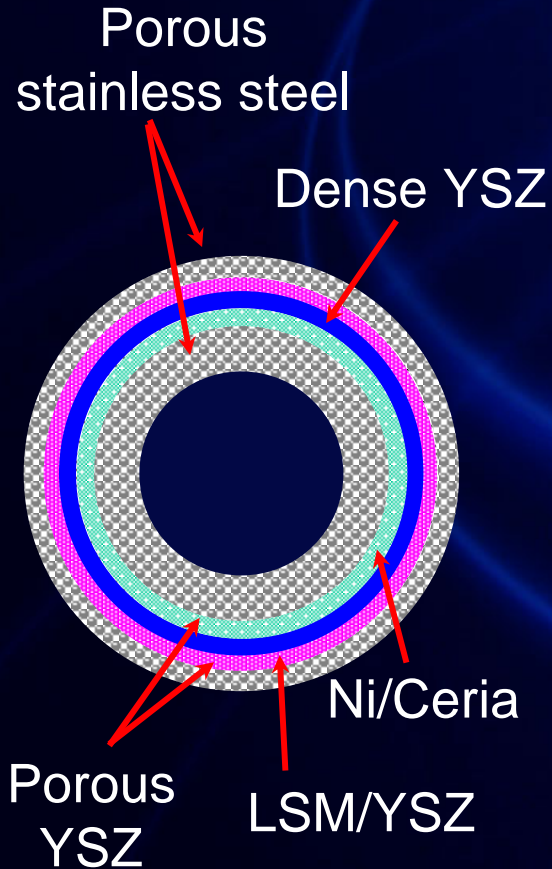
SYTO/YSZ+CeO<sub>2</sub>+Ru anode was not affected by H<sub>2</sub>S very much and completely recovered in H<sub>2</sub>/H<sub>2</sub>O.

# Performance of Single-Step Infiltrated, Cathode-Supported, Sulfur Tolerant Cell at 700 to 800 °C

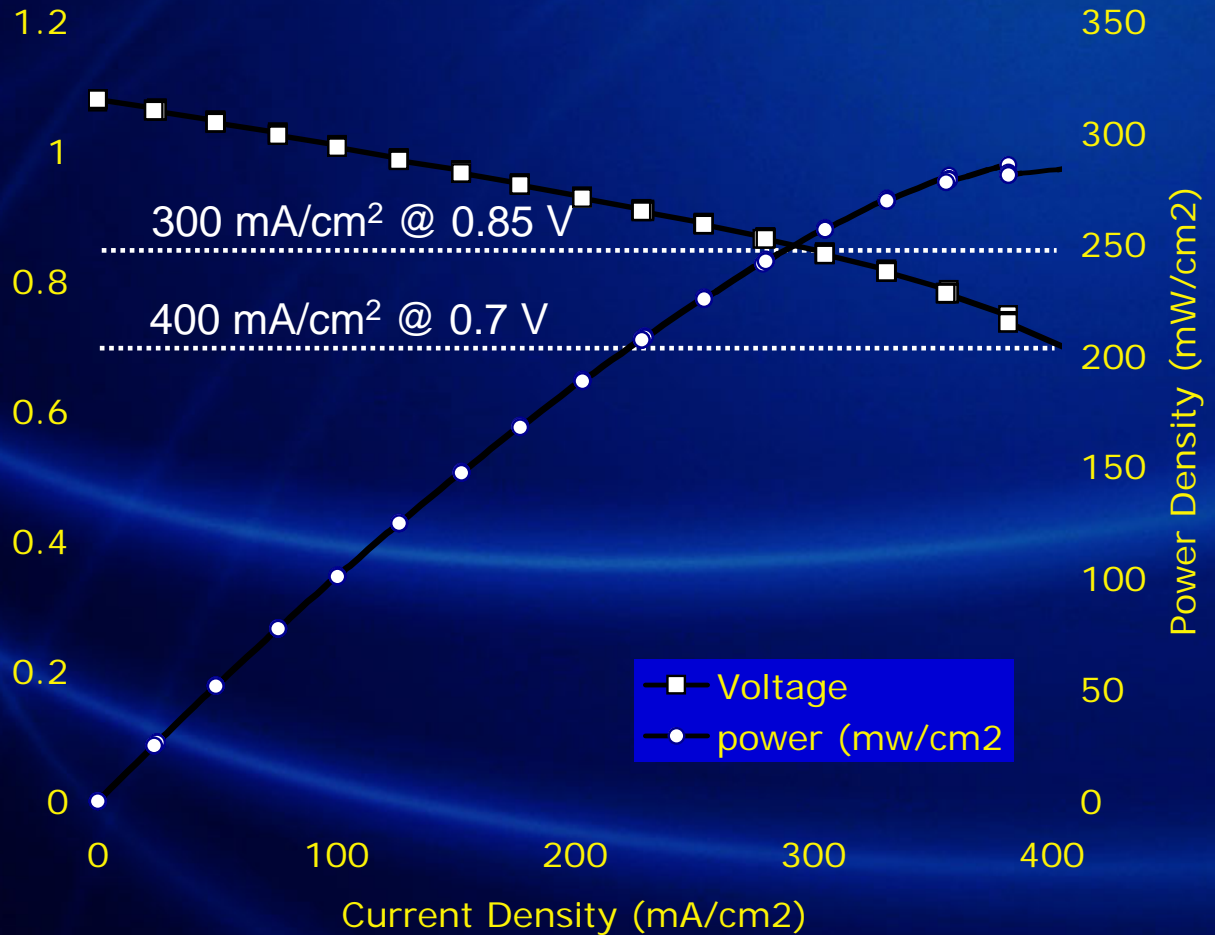


The cell with CeO<sub>2</sub>/Ru infiltration showed **200 mW/cm<sup>2</sup>** at 700 °C and **550 mW/cm<sup>2</sup>** at 800 °C

# Metal Supported Tubular SOFC w/Infiltrated Electrodes



$\text{H}_2 + 3\% \text{H}_2\text{O} / \text{Air}$  at 700 °C



# Team:

## Investigators:

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Lutgard De Jonghe

P.I. Program Lead  
Co-PI

## Scientists:

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Velimir Radmilovic

High temperature corrosion  
NCEM FIB/SEM/TEM

## Post Doc:

Hideto Kurokawa  
Ken Lux

Cr transport phenomena  
Air electrode stability

## Senior Technical Staff:

Craig Jacobson  
Mike Tucker  
Grace Lau  
Inna Belogolovsky

Processing and characterization  
Metal supported SOFC development  
Processing and analysis  
Processing and testing

## Graduate Students:

Tal Sholklapper  
Liming Yang  
Xuan Chen

Nano-particulate catalysts  
Novel anode catalysts  
Infiltration of cathode catalysts





# Acknowledgements

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