SOFC Development at PNNL: Cathode Task

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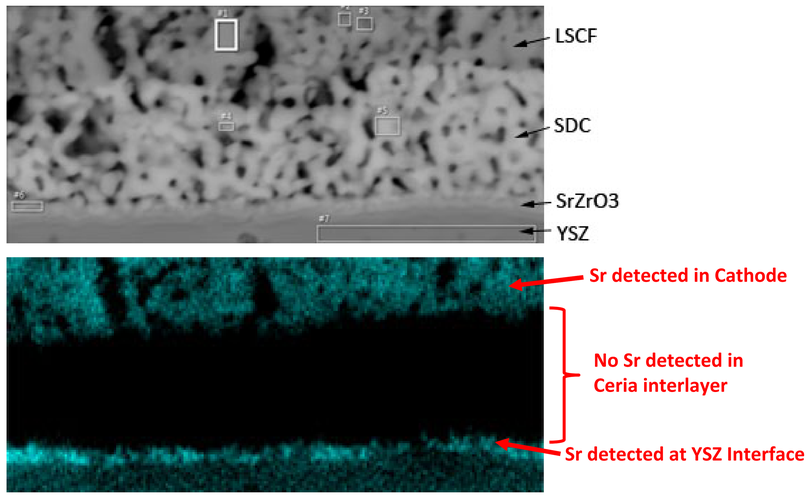
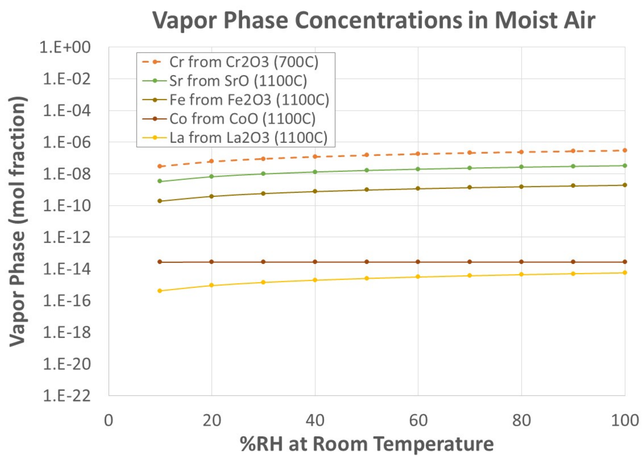
MOTIVATION – TYPICAL SEM-EDS Sr MAPS OF SINTERED LSCF CATHODES



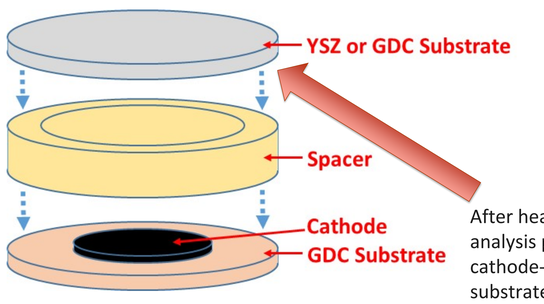
During sintering, Sr from LSCF cathode diffuses to YSZ interface and reacts to form strontium zirconate. A Sr concentration gradient characteristic of solid phase diffusion that would be expected across the ceria interlayer is absent. Could vapor phase transport play a role?

THERMODYNAMIC CALCULATIONS SUPPORT Sr VAPOR TRANSPORT



Sr volatility at 1100°C is within an order of magnitude of the level of Cr that poisons SOFC cathodes at 700°C and is the highest amongst LSCF components.

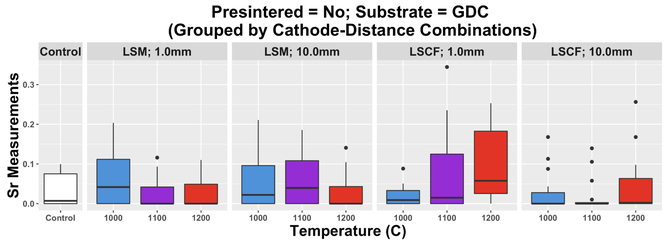
EXPERIMENTAL METHODOLOGY

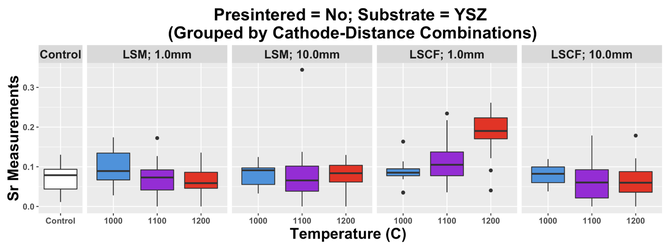


After heating, SEM-EDS or XPS analysis performed on cathode-facing surface of top substrate

ROUND 1 EXPERIMENTS (SEM-EDS ANALYSIS)

Variables	Settings
Cathode State	Sintered; Unsintered
Cathode Composition	LSM/YSZ; LSCF
Substrate (Sr Source) Composition	GDC
Substrate (Sr Sink) Composition	GDC; YSZ
Spacer	1 mm; 10 mm
Test Temperature	1000°C; 1100°C; 1200°C
Time at Temperature	0.5 h; 2 h





ROUND 1 ANALYSIS SUMMARY

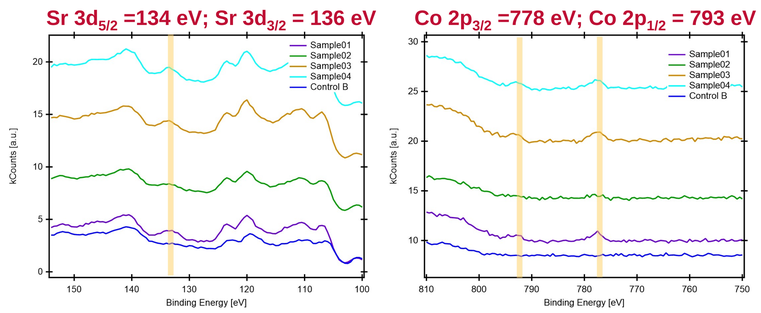
- Significant Sr vapor phase transport was measured when:
  - The cathode was not sintered prior to testing
  - The cathode composition was LSCF
  - The distance of the substrate from the cathode was shorter (1 mm)
  - The temperature was higher (1100 or 1200ºC)
- No Sr vapor phase transport occurred with:
  - Pre-sintered cathodes
  - The cathode composition was LSM/YSZ
  - Longer (10 mm) separation between substrate and cathode
  - Lower temperature (1000ºC)

ROUND 2 EXPERIMENTS (XPS ANALYSIS)

Variables	Settings
Cathode State	Unsintered
Cathode Composition	LSCF
Substrate (Sr Source) Composition	GDC; YSZ
Substrate (Sr Sink) Composition	GDC; YSZ
Spacer	1 mm; YSZ or Alumina
Test Temperature	1100°C; 1200°C
Time at Temperature	2 h

$Sr\ 3d_{5/2}$ =134 eV; $Sr\ 3d_{3/2}$ = 136 eV

$Co\ 2p_{3/2}$ =778 eV; $Co\ 2p_{1/2}$ = 793 eV



All test conditions were tested in duplicate.

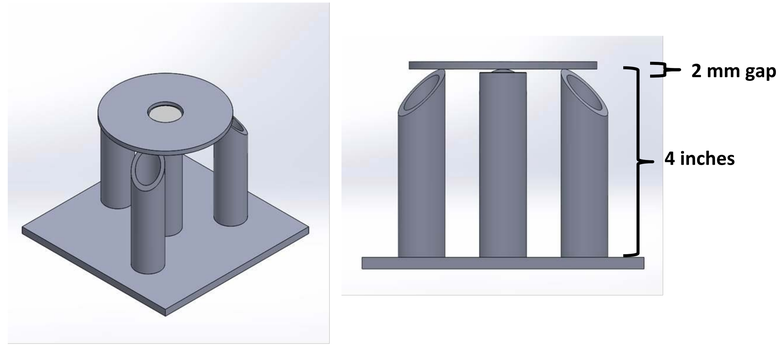
Sr was detected on 18 of 18 samples.

Co was detected on 17 of 18 samples.

ROUND 2 ANALYSIS SUMMARY

- Used conditions from initial experiments that showed Sr transport
- XPS is a more sensitive measurement than SEM-EDS
- Sr and Co transported across spacer gap (1 mm)
- Could transport be via surface diffusion rather than via the vapor phase?

ROUND 3 EXPERIMENTS (VERY LONG SURFACE PATHWAY)



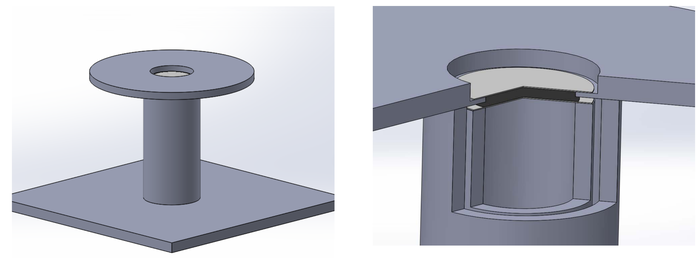
Transport testing was performed using the alumina test setup shown here. A very long surface pathway and small contact points are designed to ensure any transport is happening via the vapor phase.

Variables	Settings
Cathode State	Unsintered
Cathode Composition	LSCF
Substrate (Sr Source) Composition	GDC
Substrate (Sr Sink) Composition	YSZ
Spacer	None, 2mm air gap
Test Temperature	1000°C; 1050°C; 1100°C
Time at Temperature	2 h

ROUND 3 ANALYSIS SUMMARY

- Long surface path length to isolate vapor transport
- XPS analysis
- No Sr or Co transport detected
- Open air gap may allow vapor to dissipate before transport

ROUND 4 EXPERIMENTS (ENCLOSED AIR GAP)



Experiments are underway using the modified alumina test setup shown here. An outer tube now supports the top substrate holder while also enclosing the space where Sr and Co vapor will be present. This will allow the vapor phase species to remain concentrated. The surface pathway is still very long, with a small gap separating the inner and outer support tubes. This ensures any detected transport will have occurred through the vapor phase.

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