# In-Operando XRD of LSM/YSZ Cathodes in Combined $\mathrm{H}_{2} \mathrm{O}+\mathrm{CO}_{2}$ during 1000+h SOFC Tests 

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Experimental Parameters for $\mathbf{1 0 0 0 +}$ hour tests Cell Operation
Temperature: $775^{\circ} \mathrm{C}$

- Electrical: Constant Current approximating 800 mV
- Cathode Gas: Flowing air with no contaminants; or $3 \% \mathrm{H}_{2} \mathrm{O}$; or $3 \% \mathrm{H}_{2} \mathrm{O}+12 \% \mathrm{CO}_{2}$.
- Fuel: Moist $\mathrm{H}_{2}$ - Fuel: Moist $\mathrm{H}_{2}$


## XRD Parameters <br> - Repeated 1 hour scans <br> - 29 Range: $25-85^{\circ}$

- Step Size: $0.02^{\circ}$
- Time/Step: 1.1 second


## Typical Results \& Analysis



CONTAMINANTS CAUSED PERFORMANCE DEGRADATION


TIME-DEPENDENT PEAK SHIFTS EXHIBITED BY LSM Rietveld refinement finds that contaminants cause expansion


AVERAGE PHASE COMPOSITION (wt\%)

|  | 0\% $\mathrm{H}_{2} \mathrm{O}+0 \% \mathrm{CO}_{2}$ | $3 \% \mathrm{H}_{2} \mathrm{O}+0 \% \mathrm{CO}_{2}$ | $3 \% \mathrm{H}_{2} \mathrm{O}+12 \% \mathrm{CO}_{2}$ |
| :---: | :---: | :---: | :---: |
| LSM | 45.7 +/-1.1 | 46.4 +/-1.1 | $45.2+$ - 2.2 |
| YSZ | 43.3 +/-1.2 | $45.0+/-1.0$ | 45.1 +/-2.4 |
| Tet-Ysz | $0.6+/-0.1$ | $1.0+/-0.5$ | $1.1+/-0.8$ |
| $\mathrm{Mn}_{3} \mathrm{O}_{4}$ | $3.0+/-0.2$ | $2.3+/-0.2$ | $2.1+/-0.4$ |
| $\mathrm{MnO}_{2}$ | $3.5+/-0.3$ | $2.0+1-0.2$ | ND |
| MnO | $2.1+/-0.2$ | $1.1+/-0.1$ | $6.0+1-0.9$ |
| $\mathrm{La}_{2} \mathrm{O}_{3}$ | $1.8+/-0.2$ | $1.9+/-0.2$ | $0.3+/-0.1$ |
| $\mathrm{La}_{2} \mathrm{Zr}_{2} \mathrm{O}_{7}$ | <0.1 | <0.1 | $0.1+/-0.1$ |

NO SIGNIFICANT DIFFERENCES IN SEM/EDS


WHAT COULD LEAD TO MEASURED LSM EXPANSION IN CONTAMINANTS? Loss of Sr or O causes expansion in LSM

Rate of Sr loss that correlates to the measured rate of expansion


SUMMARY

- Tetragonal YSZ, Mn oxides, La oxide, and trace amounts of La zirconate pyrochlore form during sintering of LSCF cathodes.
- Power density decreased with increasing cathode air contaminants.
- In air with no added contaminants, the LSM/YSZ cathode exhibited stable performance while in contaminants, it degraded - at nearly the same rate with and without $\mathrm{CO}_{2}$
- In air with no added contaminants, the LSM crystal lattice contracted, while in contaminants, it expanded at similar rates with and without $\mathrm{CO}_{2}$.
- A loss of $\sim 0.00003 \mathrm{Sr} / \mathrm{h}$ from the chemical formula of LSM could cause the expansion experienced during exposure to contaminants in cathode air.
- No significant differences in cathode microstructure or chemistry due to contaminant exposure were observed in SEM/EDS.

