The Effect of Precursor Solution Desiccation or Nano-Ceria Pre-Infiltration on Various La$_0.6$Sr$_{0.4}$Fe$_y$Co$_{1-y}$O$_{3-x}$ Infiltrate Compositions

Theodore E. Burye, Hongjie Tang, and Jason D. Nicholas$^*$

Department of Chemical Engineering and Material Science, Michigan State University, East Lansing, Michigan 48824, USA

Here, electrochemical impedance spectroscopy, X-ray diffraction, and scanning electron microscopy were used to determine the effect of precursor solution desiccation and nano-ceria pre-infiltration on Solid Oxide Fuel Cell cathodes with various infiltrate compositions. The calcium chloride desiccation of citric acid containing La$_0.6$Sr$_{0.4}$FeO$_{3-x}$ (LSF), La$_0.6$Sr$_{0.4}$Fe$_0.8$Co$_{0.2}$O$_{3-x}$ (LSFC), La$_0.6$Sr$_{0.4}$Fe$_0.5$Co$_{0.5}$O$_{3-x}$ (LSF55), and La$_0.6$Sr$_{0.4}$CoO$_{3-x}$ (LSC) infiltrate precursor solutions reduced average infiltrate particle sizes from the ~53 nm obtained with standard processing to ~22 nm. Similarly, infiltration of the aforementioned La$_{0.6}$Sr$_{0.4}$Fe$_{0.0}$Co$_{0.0}$O$_{3-x}$ precursor solutions into porous cathodes containing pre-infiltrated gadolinium doped ceria (GDC) particles reduced average La$_{0.6}$Sr$_{0.4}$Fe$_{0.0}$Co$_{0.0}$O$_{3-x}$ infiltrate particle sizes from the ~53 nm obtained with standard processing (i.e., in the absence of nano-GDC pre-infiltration) to ~27 nm with nano-GDC pre-infiltration. These desiccation and nano-GDC pre-infiltration induced infiltrate particle size reductions resulted in improved cathode performance. For example, in comparison with the 650°C operating temperatures required for standard LSC-GDC cathodes to achieve a polarization resistance of 0.1 Ωcm$^2$, identical cathodes subjected to desiccation or nano-GDC pre-infiltration achieved 0.1 Ωcm$^2$ at 630°C and 570°C, respectively. These promising results suggest that precursor solution desiccation or nano-GDC pre-infiltration may be useful for reducing the sizes of a variety of SOFC infiltrate materials.

© The Author(s) 2016. Published by ECS. This is an open access article distributed under the terms of the Creative Commons Attribution 4.0 License (CC BY, http://creativecommons.org/licenses/by/4.0/), which permits unrestricted reuse of the work in any medium, provided the original work is properly cited. [DOI: 10.1149/2.0431609jes] All rights reserved.

Manuscript submitted May 23, 2016; revised manuscript received June 23, 2016. Published July 2, 2016. This was Paper 1362 from the San Diego, California, Meeting of the Society, May 29–June 2, 2016.

Solid Oxide Fuel Cells (SOFCs) and Solid Oxide Electrolysis Cells (SOECs) (i.e. SOFCs operated in reverse) can be used for high-efficiency electricity generation, energy storage, chemical sensing, and chemical synthesis applications.1 Unfortunately, the poor oxygen surface exchange kinetics within typical SOFC cathodes have limited device performance and restricted commercial SOFCs to operating temperatures greater than 650°C.2–4 To realize reduced SOFC operating temperatures, many researchers have used the infiltration method to fabricate nano-composite cathodes consisting of Mixed Ionic and Electronic Conducting (MIEC) nano-particles atop necked, porous ionic conducting (IC) scaffolds.5–7 These MIEC-IC nano-composite cathodes have typically been produced by 1) dissolving precursor nitrates and organic solution additives in water, 2) infiltrating the resulting solutions into porous IC scaffolds, 3) gelling the infiltrated solutions through the application of heat and/or uncontrollable solvent evaporation in air, and 4) thermally decomposing the resulting nitrate gels to form oxide MIEC nanoparticles. Unfortunately, it has proven difficult to control infiltrate particle sizes using the infiltration method. For instance, Sm$_{0.5}$Sr$_{0.5}$CoO$_{3-δ}$ (SSC) infiltrate particles produced on ceria scaffolds have displayed average infiltrate particle sizes ranging from 40 to 100 nm for studies utilizing nominally identical compositions, heating rates, and firing conditions.8–10 Similar average infiltrate particle size variations have been reported in the literature for other infiltrate compositions such as La$_{0.6}$Sr$_{0.4}$Co$_{0.3}$Fe$_{0.7}$O$_{3-δ}$ (LSCF) and La$_{0.6}$Sr$_{0.4}$Co$_{0.3}$Fe$_{0.7}$O$_{3-δ}$ (LSCF)

Recent work has shown that either the chemical desiccation of polymer containing nitrate precursor solutions11,12 or the infiltration of polymer containing nitrate precursor solutions into scaffolds containing pre-existing Ce$_{0.7}$Sr$_{0.3}$O$_{1.95}$ (GDC) nanoparticles13 is useful to tailor the size and phase-purity of LSCF infiltrate particles. In fact, use of these techniques with LSCF precursor solutions containing the surfactant Triton X-100 have produced nano-composite SOFC cathodes with some of the smallest average infiltrate particle sizes (22 nm13,14 and best low-temperature performance (open-circuit polarization resistances of 0.1 Ωcm$^2$ at 540°C)13 ever reported. Similar, although less dramatic, particle size and performance improvements have also been observed for LSCF precursor solutions utilizing citric acid, instead of Triton X-100, as an organic solution additive.14,15 However, it is still unknown how these new processing techniques affect other MIEC infiltrate compositions. Therefore, the objective of the present work was to evaluate how precursor solution desiccation or nano-ceria pre-infiltration impact the microstructure, phase purity and electrochemical performance of nano-composite cathodes produced by infiltrating GDC scaffolds with La$_{0.6}$Sr$_{0.4}$FeO$_{3-δ}$ (LSF), La$_{0.6}$Sr$_{0.4}$Fe$_0.8$Co$_{0.2}$O$_{3-δ}$ (LSFC), La$_{0.6}$Sr$_{0.4}$Fe$_0.5$Co$_{0.5}$O$_{3-δ}$ (LSF55), La$_{0.6}$Sr$_{0.4}$Fe$_0.2$Co$_{0.8}$O$_{3-δ}$ (LSCF), or La$_{0.6}$Sr$_{0.4}$CoO$_{3-δ}$ (LSC) infiltrate precursor solutions.

Experimental

The first step in the production of the symmetric cathode-electrolyte-cathode cells analyzed here was to produce porous, well-necked GDC cathode scaffolds on either side of dense GDC electrolyte pellets using the methods described in Burye and Nicholas.14 Then, three different infiltration techniques (a standard, precursor solution desiccation, and a nano-GDC pre-infiltration technique) were used to introduce infiltrate nanoparticles into these porous cathode scaffolds. The 0.5 M La$_{0.6}$Sr$_{0.4}$Fe$_{0.0}$Co$_{0.0}$O$_{3-δ}$ (0 ≤ y ≤ 1) nitrate precursor solutions used for all three infiltration techniques were produced by dissolving 99.999% pure La(NO$_3$)$_3$·6.8 H$_2$O, 99.965% pure Sr(NO$_3$)$_2$, 99.999% pure Co(NO$_3$)$_2$·6.9 H$_2$O and 99.999% pure Fe(NO$_3$)$_3$·9.84 H$_2$O (Alfa Aesar; Ward Hill, MA) in 18.2 MΩ water (Merck Millipore; Billerica, MA) containing 0.75 mol of nitrates per mole of pre-dissolved citric acid (EMD Chemicals; Gibbstown, NJ). After nitrate dissolution, NH$_4$OH (Sigma Aldrich; St. Louis, MO) and additional 18.2 MΩ water were added to bring the solution pH to 3 and the solution molarity to 0.5 mol each liter of solution. The 1.5 M GDC nitrate precursor solutions used in the nano-GDC pre-infiltration method were prepared by dissolving 99.999% pure Ce(NO$_3$)$_3$·6.0 H$_2$O and 99.999% pure Gd(NO$_3$)$_3$·4.0 H$_2$O (Alfa Aesar; Ward Hill, MA) in 18.2 MΩ water containing 3 wt% of pre-dissolved Triton X-100 (148 MΩ water containing 300 mg Triton X-100/mass nitrate) and 18.2 MΩ H$_2$O. In all cases, the number of nitrate waters of hydration were determined prior to weighing from the mass loss occurring inside a Q500 thermogravimetric analyzer (TA
For electrodes fabricated with a particular infiltration method, Figure 1 shows that the electrode resistance systematically decreases as the infiltrate Co:Fe ratio increases. This behavior is consistent with previous literature studies showing that at typical SOFC operating temperatures the oxygen surface exchange resistance ($R_s$) of materials within the LSC-LSF solid solution decrease as the Co:Fe ratio increases,17,22–24 and suggests that oxygen surface exchange is a dominant source of resistance in these cathodes at 600°C. Figure 1 shows that at 600°C the performance of the GDC pre-infiltrated cells was better than the performance of the desiccated cells. In addition, Figure 1 shows that at 600°C the performance of the standard cells was worse than either the GDC pre-infiltrated or desiccated cells.

Figure 2 shows infiltrate polarization resistance vs. inverse temperature Arrhenius plots obtained from impedance curves such as those in Figure 1. Figure 2 shows that the trends of 1) GDC pre-infiltrated cells performing better than the desiccated cells, which, in turn, perform better than the standard cells, and 2) increased performance with increasing Co:Fe infiltrate composition also holds at all temperatures between 400 and 700°C. Here, nano-GDC pre-infiltrated CAD LSC-GDC electrodes displayed the best performance, achieving 0.1 Åm² (the resistance commonly defined as that needed for SOFC operation$^{25}$) at 570°C. Although this performance is not as good as the 0.1 Åm² at 540°C displayed by the nano-GDC pre-infiltrated Trion X-100 derived LSCF-GDC electrodes reported in Burye and Nicholas,15 (due to the larger size of LSCF infiltrate particles produced using citric acid instead of Trion X-100) it is still among the best performing infiltrated SOFC cathodes ever reported.$^{26,27}$

Figure 3 shows representative micrographs of LSF, LSFC, LSCF55, LSCF, and LSC infiltrated cells produced using either the standard, desiccated, or GDC pre-infiltration processing method. While only La$_{0.6}$Sr$_{0.4}$Fe$_y$Co$_{1-y}$O$_{3-δ}$ infiltrate particles are shown in the standard and desiccated Figure 3 micrographs, both La$_{0.6}$Sr$_{0.4}$Fe$_y$Co$_{1-y}$O$_{3-δ}$, infiltrate (at a 12.0 vol% loading level) and the nano-GDC pre-infiltrate (at a 7.4 vol% loading level) are present in the nano-GDC Figure 3 micrographs. Because the 25 nm average size of the nano-GDC pre-infiltrate particles$^{15}$ is close to the average infiltrate particle sizes shown in the nano-GDC Figure 3 micrographs, the average infiltrate particle sizes obtained from all Figure 3 micrographs were treated as those of the La$_{0.6}$Sr$_{0.4}$Fe$_y$Co$_{1-y}$O$_{3-δ}$ infiltrate alone. As discussed in more detail in Burye and Nicholas,14,15 although additional experiments are needed, it is possible that a combination of evaporation induced self-assembly, infiltrate particle coarsening, or other mechanisms are responsible for particle size variations observed here.

Figure 4, which summarizes infiltrate particle sizes obtained from micrographs such as those in Figure 3, shows that within a standard deviation of the observed infiltrate particle sizes, the size of the infiltrate particle sizes obtained with each of the three processing techniques was independent of the La$_{0.6}$Sr$_{0.4}$Fe$_y$Co$_{1-y}$O$_{3-δ}$ Co:Fe ratio. Figure 4 also shows that across all infiltrate compositions, the infiltrate produced via nano-GDC pre-infiltration was significantly smaller (P < 0.001) than the infiltrate produced via desiccation, which, in turn, was significantly smaller (P < 0.02) than the infiltrate produced with standard processing. Smaller MIEC infiltrate particle sizes are desirable because they can lower cathode resistance by providing additional surface area for the incorporation of oxygen into the electrode. (Note, CaCl$_2$ desiccation of GDC pre-infiltrated LSCF-GDC samples (not

---

**Results and Discussion**

Table I provides a summary of the processing conditions and resulting microstructural characteristics for the symmetric cells analyzed here.

Figure 1 shows representative 600°C impedance curves for La$_{0.6}$Sr$_{0.4}$Fe$_y$Co$_{1-y}$O$_{3-δ}$ GDC symmetric cells with standard processing, precipitate precursor solution desiccation, and nano-GDC pre-

---

**Instruments; New Castle, DE** upon nitrate heating under nitrogen to 850°C.

For cells infiltrated in the standard manner (i.e. no desiccation or nano-ceria pre-infiltration), La$_{0.6}$Sr$_{0.4}$Fe$_y$Co$_{1-y}$O$_{3-δ}$ nitrate precursor solutions were pipetted into GDC cathode scaffolds, allowed to soak into the scaffold for five minutes, gelled in flowing air at 80°C for 10 minutes, fired at 100°C/min to 700°C, held at 700°C for 1 hour, and then cooled at >25°C/min to room temperature. This process was repeated six times to produce cells with 12.0 vol% of infiltrate (defined as vol.

For cells infiltrated and then desiccated, La$_{0.6}$Sr$_{0.4}$Fe$_y$Co$_{1-y}$O$_{3-δ}$ nitrate precursor solutions were pipetted into porous GDC cathode scaffolds, allowed to soak into the scaffold for five minutes, and gelled in flowing air at 80°C for 10 minutes, before being chemically desiccated with CaCl$_2$ for 8–10 hours. The infiltrated cathodes were then heated at 10°C/min to 700°C, held at 700°C for 1 hour, and then cooled at >25°C/min to room temperature. This process was repeated six times to produce cells with 12.0 vol% of infiltrate.

For nano-GDC pre-infiltrated cells, GDC nitrate solutions were first infiltrated into porous GDC scaffolds, allowed to soak for 5 minutes, gelled in flowing air at 80°C for 10 minutes, heated at 10°C/min to 700°C, held at 700°C for 1 hour, and then cooled at >25°C/min to room temperature. This step was repeated two times to produce cathodes with 7.4 vol% nano-GDC oxide particles. Then, La$_{0.6}$Sr$_{0.4}$Fe$_y$Co$_{1-y}$O$_{3-δ}$ precursor solutions were infiltrated, gelled and fired in the same manner as the samples processed with the standard technique. This step was repeated up to six times to achieve the desired La$_{0.6}$Sr$_{0.4}$Fe$_y$Co$_{1-y}$O$_{3-δ}$ loading level of 12.0 vol% of infiltrate.

Finally, all cells were prepared for electrochemical testing by applying lanthanum strontium manganite (LSM) and gold current collectors using previously described methods.13 All cells were then characterized via Electrochemical Impedance Spectroscopy (EIS) using previously described methods.13 Specifically, cathode polarization resistance ($R_p$) values were determined by dividing the distance between x-axis intercepts on an EIS Nyquist plot by two (to account for the fact that each symmetric cell had two cathodes) and multiplying by the electrode’s geometric area (0.5 cm$^2$). Based on the definition of resistance, ohmic resistivity ($\rho_{ohmic}$) values for each cell were determined by dividing the high-frequency Nyquist plot x-axis intercept by the electrolyte thickness and multiplying by the electrode’s geometric area (0.5 cm$^2$).

To help understand the observed performance trends, $R_p$ predictions were made using a previously-described10,12 Simple Infiltrated Microstructure Polarization Loss Estimation (SIMPLE) model that accounts for electrical losses associated with oxygen exchange into the MIEC infiltrate and oxygen conduction through the GDC scaffold. Specifically, SIMPLE Model $R_p$ predictions were made by inputting 1) La$_{0.6}$Sr$_{0.4}$Fe$_y$Co$_{1-y}$O$_{3-δ}$ densities from the Joint Committee on Powder Diffraction Standards (JCPDS) database,16 2) the MIEC infiltrate loading levels, cathode thicknesses, geometric cathode areas, cathode porosities, MIEC nano-particle diameters, and GDC scaffold column loading levels, cathode thicknesses, geometric cathode areas, cathode porosities, MIEC nano-particle diameters, and GDC scaffold column loading levels.
Table I. LSCF-GDC Nano-Composite Cathode Characteristics.

| Composition  | Fabrication Method | Nano-GDC Loading Level (vol%) | MIEC Infiltrate Loading Level | Number of MIEC Infiltrations | Number of Nano-GDC Infiltrations | Nitrate Solution Molarity (MIEC/GDC) | Cathode Thickness (μm) Side 1 | Cathode Thickness (μm) Side 2 | Volume of Solution per Infiltration (μL) | Scaffold RMS Roughness (μm) Side 1 | Scaffold RMS Roughness (μm) Side 2 | Cathode Geometric Area (cm²) | Cathode Porosity (%) | Scaffold Column Width (nm) | MIEC Infiltrate Diameter (nm) | Total Infiltrate Surface Area (cm²) Side 1 | Total GDC Surface Area (cm²) Side 1 | Surface Area/GDC Surface Area (%) Side 1 | Surface Area/GDC Surface Area (%) Side 2 |
|--------------|---------------------|-------------------------------|-------------------------------|-----------------------------|-------------------------------|-----------------------------------|-------------------------------|-------------------------------|-----------------------------------------|-------------------------------|-------------------------------|-------------------------------|-------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|-----------------------------------|
| LSF Standard | NA                  | 12.0                          | 6                             | 0                           | 0.5/NA                        | 37.8                              | 2.88                          | 3.99                          | 0.5                                      | 32                            | 120                           | 55                          | 247.8                         | 157                          | 158                           | 226.4                         | 143                          | 158                           |
| LSF CaCl₂    | NA                  | 12.0                          | 6                             | 0                           | 0.5/1.5                       | 37.0                              | 2.76                          | 3.90                          | 0.5                                      | 32                            | 120                           | 44                          | 302.3                         | 153                          | 198                           | 276.3                         | 140                          | 197                           |
| LSF Nano-GDC | 7.4                 | 12.0                          | 6                             | 0                           | 0.5/1.5                       | 36.4                              | 2.74                          | 3.96                          | 0.5                                      | 32                            | 120                           | 44                          | 411.8                         | 147                          | 301                           | 411.3                         | 137                          | 300                           |
| LSFC Standard| NA                  | 12.0                          | 6                             | 0                           | 0.5/NA                        | 34.0                              | 2.58                          | 4.00                          | 0.5                                      | 32                            | 120                           | 45                          | 298.5                         | 155                          | 193                           | 271.5                         | 141                          | 193                           |
| LSFC CaCl₂   | NA                  | 12.0                          | 6                             | 0                           | 0.5/NA                        | 33.9                              | 2.70                          | 4.00                          | 0.5                                      | 32                            | 120                           | 28                          | 478.6                         | 154                          | 311                           | 419.2                         | 135                          | 311                           |
| LSFC Nano-GDC| 7.4                 | 12.0                          | 6                             | 2                           | 0.5/1.5                       | 37.2                              | 2.73                          | 3.92                          | 0.5                                      | 41                            | 120                           | 28                          | 478.6                         | 154                          | 311                           | 419.2                         | 135                          | 311                           |
| LSCF55 Standard| NA                  | 12.0                          | 6                             | 0                           | 0.5/NA                        | 36.7                              | 2.90                          | 4.09                          | 0.5                                      | 32                            | 120                           | 52                          | 254.0                         | 152                          | 167                           | 234.4                         | 141                          | 166                           |
| LSCF55 CaCl₂ | NA                  | 12.0                          | 6                             | 0                           | 0.5/NA                        | 37.2                              | 2.90                          | 4.17                          | 0.5                                      | 32                            | 120                           | 42                          | 318.9                         | 154                          | 207                           | 298.3                         | 144                          | 207                           |
| LSCF55 Nano-GDC| 7.4               | 12.0                          | 6                             | 2                           | 0.5/1.5                       | 36.6                              | 2.80                          | 4.07                          | 0.5                                      | 41                            | 120                           | 28                          | 470.7                         | 152                          | 310                           | 432.0                         | 139                          | 311                           |
| LSCF Standard| NA                  | 12.0                          | 6                             | 0                           | 0.5/NA                        | 37.1                              | 2.72                          | 4.05                          | 0.5                                      | 32                            | 120                           | 50                          | 267.3                         | 154                          | 174                           | 234.5                         | 135                          | 174                           |
| LSCF CaCl₂   | NA                  | 12.0                          | 6                             | 0                           | 0.5/NA                        | 36.5                              | 2.89                          | 4.12                          | 0.5                                      | 32                            | 120                           | 42                          | 313.1                         | 151                          | 207                           | 292.4                         | 141                          | 207                           |
| LSCF Nano-GDC| 7.4                 | 12.0                          | 6                             | 2                           | 0.5/1.5                       | 37.2                              | 2.94                          | 4.14                          | 0.5                                      | 41                            | 120                           | 27                          | 496.1                         | 154                          | 322                           | 451.5                         | 140                          | 323                           |
| LSC Standard | NA                  | 12.0                          | 6                             | 0                           | 0.5/NA                        | 37.9                              | 2.70                          | 4.35                          | 0.5                                      | 32                            | 120                           | 51                          | 267.2                         | 157                          | 170                           | 237.6                         | 140                          | 170                           |
| LSC CaCl₂    | NA                  | 12.0                          | 6                             | 0                           | 0.5/NA                        | 38.3                              | 2.65                          | 4.38                          | 0.5                                      | 32                            | 120                           | 39                          | 353.2                         | 159                          | 222                           | 312.2                         | 140                          | 223                           |
| LSC Nano-GDC | 7.4                 | 12.0                          | 6                             | 2                           | 0.5/1.5                       | 36.6                              | 2.85                          | 4.30                          | 0.5                                      | 41                            | 120                           | 24                          | 549.3                         | 152                          | 361                           | 513.5                         | 142                          | 362                           |
measured ohmic resistivities and the ionic resistivity of GDC into the ionic resistivity of GDC. The good agreement between the experimentally measured and SIMPLE model predictions made using the average particle sizes from Figure 4 are shown in Figure 2. The good Figure 2 agreement between the experimentally measured and SIMPLE model predicted Rp values (except at temperatures of ~700°C where resistive processes, such as the gas concentration polarization known to occur in these cathodes at this temperature, not accounted for in the SIMPLE model result in differences) suggest that processing induced changes in the La0.06Sr0.4FeCo1-yO3-x infiltrate particle size alone were responsible for the observed Rp differences. It is important to realize that infiltrate particle size was the only SIMPLE model input variable that was altered when predicting Rp values for a given La0.06Sr0.4FeCo1-yO3-x infiltrate composition, and the La0.06Sr0.4FeCo1-yO3-x infiltrate Rs value was the only other variable altered with a change in La0.06Sr0.4FeCo1-yO3-x infiltrate composition.

Figure 5 compares the ohmic resistivity of each symmetric cell to the ionic resistivity of GDC. The good agreement between the measured ohmic resistivities and the ionic resistivity of GDC indicates that the electrolyte layer was the dominant source of ohmic resistance in the symmetric cells, that electronic conduction within the electrodes was not a significant source of resistance, and that the processing-induced Rp differences shown in Figures 1 and 2 did not result from infiltrate electronic conductivity differences.

Figure 6 shows X-ray diffractograms for La0.06Sr0.4FeCo1-yO3-x infiltrated cells with standard and desiccated processing. Unfortunately, attempts to obtain nano-GDC pre-infiltrated La0.06Sr0.4FeCo1-yO3-x XRD spectra (not shown) were unsuccessful due to overlap of the main 33° and 47° La0.06Sr0.4FeCo1-yO3-x peaks by those of GDC and the fact that the strong XRD signal from the GDC overwhelmed the signal from the La0.06Sr0.4FeCo1-yO3-x. As shown in Figure 6, phase pure La0.06Sr0.4FeCo1-yO3-x was obtained for all compositions after firing at 700°C, suggesting that the processing-induced Rp differences shown in Figures 1 and 2 did not result from infiltrate phase purity differences.
Figure 3. Scanning electron micrographs for infiltrated La$_{0.6}$Sr$_{0.4}$Co$_y$Fe$_{1-y}$O$_{3-x}$ nano-particles cathodes produced by firing standard (a-e), desiccated (f-j), and nano-GDC pre-infiltrated (k-o) citric acid-containing precursor solutions at 700°C. The scale bar in each image is 50 nm long.

Figure 4. LSF, LSFC, LSCF55, LSCF, and LSC infiltrate particle sizes produced by firing a) standard, b) desiccated and c) nano-GDC pre-infiltrated citric acid-containing precursor solutions at 700°C. The error bars indicate the standard deviation of the infiltrate diameters calculated from the SEM images.

Figure 5. The ohmic resistivity ($\rho$) vs. inverse temperature behavior of all the La$_{0.6}$Sr$_{0.4}$Co$_y$Fe$_{1-y}$O$_{3-x}$-GDC cells reported here. The inclined solid line is the resistivity of pure GDC from Nicholas et al.\textsuperscript{9}

Conclusions

Taken together, the results presented in this paper indicate that 1) precursor gel desiccation or nano-GDC pre-infiltration reduces the size of citric acid derived LSF, LSFC, LSCF55, LSCF, and LSC infiltrate particles, and 2) that these particle size reductions alone are
responsible for the observed electrode performance improvements. Further, the infiltrate particle sizes produced via a specific fabrication approach are similar for all \( \text{La}_{0.6}\text{Sr}_{0.4}\text{Fe}_{y}\text{Co}_{1-y}\text{O}_{3-x} \) Fe:Co ratios. Specifically, standard, precursor gel desiccation, and nano-GDC pre-infiltration produced phase pure, infiltrate particles with average sizes of \( \sim 53, 42, \) and 27 nm, respectively. Out of the compositions tested here, the LSC-GDC cathodes performed the best (achieving a 0.1 \( \Omega \text{cm}^2 \) polarization resistance at 630 \( /\Omega \)) here, the LSC-GDC cathodes performed the best (achieving a 0.1 \( \Omega \text{cm}^2 \) polarization resistance at 630 \( /\Omega \)).

Acknowledgments

This work was supported by National Science Foundation (NSF) CAREER Award No. CBET-1254453. Profilometry work was conducted at the Michigan State Keck Microfabrication Facility. Microscopy work was conducted at the Michigan State Composites Center, which is supported by the NSF Major Instrumentation Program and Michigan State University.

List of Symbols

| AC | Alternating Current |
| CAD | Citric Acid Derived |
| DC | Direct Current |
| EIS | Electrochemical Impedance Spectroscopy |
| GDC | \( \text{Gd}_{0.1}\text{Ce}_{0.9}\text{O}_{1.95} \) |
| IC | Ionic Conductor |
| \( L_C \) | Characteristic Thickness |
| LSC | \( \text{La}_{0.6}\text{Sr}_{0.4}\text{CoO}_{3-x} \) |
| LSCF | \( \text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.8}\text{Fe}_{0.2}\text{O}_{3-x} \) |
| LSCF55 | \( \text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.5}\text{Fe}_{0.5}\text{O}_{3-x} \) |
| LSFC | \( \text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-x} \) |
| LSF | \( \text{La}_{0.6}\text{Sr}_{0.4}\text{FeO}_{3-x} \) |
| LSM | \( \text{La}_{0.6}\text{Sr}_{0.4}\text{Fe}_{0.2}\text{MnO}_{4-x} \) |
| MIEC | Mixed Ionic and Electronic Conductor |
| \( R_p \) | Cathode Polarization Resistance |
| \( R_e \) | Oxygen Surface Exchange Resistance |
| SEM | Scanning Electron Microscopy |
| SIMPLE | Simple Infiltrated Microstructure Polarization Loss Estimation |
| SOEC | Solid Oxide Electrolysis Cell |
| SOFC | Solid Oxide Fuel Cell |
| SSC | \( \text{Sm}_{0.8}\text{Gd}_{0.2}\text{CoO}_{3-x} \) |
| TGA | Thermo-Gravimetric Analysis |
| XRD | X-ray Diffraction |

References

1. J. D. Nicholas, ECS Interface, 22, 49 (2013).
2. T. Tasi and S. A. Barnett, Solid State Ionics, 93, 207 (1997).
3. S. DeSouza, J. S. Visco, and L. C. DeJonghe, Journal of the Electrochemical Society, 144, L35 (1997).
4. Redox Power Systems LLC, http://www.redoxpowersystems.com/fiq (2015).
5. J. M. Vohs and R. J. Gorte, Advanced Materials, 21, 943 (2009).
6. S. P. Jiang, Materials Science and Engineering A, 418, 199 (2006).
7. D. Ding, X. Li, S. Y. Lai, K. Gerdes, and M. L. Liu, Energy Environ. Sci., 7, 552 (2014).
8. J. D. Nicholas and S. A. Barnett, Journal of the Electrochemical Society, 157, B536 (2010).
9. J. D. Nicholas, L. Wang, A. V. Call, and S. A. Barnett, Phys. Chem. Chem. Phys., 14, 15379 (2012).
10. H. Zhang, F. Zhao, F. L. Chen, and C. R. Xia, Solid State Ionics, 192, 591 (2011).
11. M. Shah, P. W. Voorhees, and S. A. Barnett, Solid State Ionics, 187, 64 (2011).
12. M. Shah, J. D. Nicholas, and S. A. Barnett, Electrochemistry Communications, 11, 2 (2009).
13. T. E. Burye and J. D. Nicholas, Journal of Power Sources, 276, 54 (2015).
14. T. E. Burye and J. D. Nicholas, Journal of Power Sources, 301, 287 (2016).
15. T. E. Burye and J. D. Nicholas, Journal of Power Sources, 300, 402 (2015).
16. International Center for Diffraction Data Joint Committee on Powder Diffraction Standards, Powder Diffraction File 4, in, Newtown Square, PA (2012).
17. F. Zhao, Z. Wang, M. Liu, L. Zhang, C. Xia, and F. Chen, Journal of the Electrochemical Society, 154, B931 (2007).
18. H. Xiong, B. K. Lai, A. C. Johnson, and S. Ramanathan, Journal of Power Sources, 193, 589 (2009).
19. B. Dawson and R. G. Trapp, Basic and Clinical Biostatistics, McGraw-Hill, New York (2004).
20. A. J. Samson, P. Hjalmarsson, M. Segaard, J. Hjelm, and N. Bonanos, Journal of Power Sources, 216, 124 (2012).
21. A. Samson, M. Segaard, R. Kruibe, and N. Bonanos, Journal of the Electrochemical Society, 158, B650 (2011).
22. N. J. Stimick, A. Biebrer-Hutter, T. M. Ryll, J. A. Kilner, A. Atkinson, and J. L. M. Rupp, Solid State Ionics, 206, 7 (2012).
23. M. Kubick, T. M. Huber, A. Welz, A. Penn, G. M. Rupp, J. Bernardi, M. Stoger-Pollach, H. Hutter, and J. Fleig, Solid State Ionics, 256, 38 (2014).
24. A. Eger, E. Bucher, M. Yang, and W. Sitte, Solid State Ionics, 225, 55 (2012).
25. B. C. H. Steele and A. Heimel, Nature, 414, 345 (2001).
26. F. Zhao, Z. Wang, M. Liu, L. Zhang, C. Xia, and F. Chen, Journal of Power Sources, 185, 13 (2008).
27. Z. L. Zhan, D. Han, T. Z. Wu, X. F. Ye, S. R. Wang, T. L. Wen, S. Cho, and S. A. Barnett, RSC Advances, 2, 4075 (2012).
28. J. E. ten Elshof and J. Boeijsma, Powder Diffraction, 11, 28 (1996).
29. G. Brauer and H. Gradinger, Zeitschrift für Anorganische und Allgemeine Chemie, 163, 207 (1904).
30. J. E. ten Elshof and J. Boeijsma, Powder Diffraction, 11, 28 (1996).
31. Y. H. Li, K. Gerdes, T. Horita, and X. B. Lai, Journal of the Electrochemical Society, 160, F343 (2013).
32. R. Sonntag, S. Neov, V. Kozhukharov, D. Neov, and J. E. ten Elshof, Physica B: Condensed Matter, 241–243, 393 (1997).