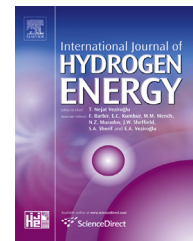




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Engineering the solid oxide fuel cell electrocatalyst infiltration technique for industrial use



Regis P. Dowd Jr.^{a,b,*}, Shiwoo Lee^{a,c}, Yueying Fan^{a,c}, Kirk Gerdes^a

^a United States Department of Energy, National Energy Technology Laboratory, Morgantown, WV 26507, USA

^b Department of Chemical and Petroleum Engineering, The University of Kansas, Lawrence, KS 66045, USA

^c AECOM, Morgantown, WV 26507, USA

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ABSTRACT

In this work, we explore various parameters for infiltrating $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$ (LSCo) into the $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$ (LSCF) – $\text{Ce}_{0.8}\text{Sm}_{0.2}\text{O}_2$ (SDC) cathode of a planar solid oxide fuel cell (SOFC) using an automated solution dispensing technique for commercial deployment of infiltrated SOFCs by industry. Substrate temperature, chelating agent concentration, and surfactant type were explored to develop a 1-step infiltration process for delivering 8–10 weight percent of LSCo electrocatalyst to the cathode active layer. The results confirm increased fuel cell performance and durability by optimizing the infiltrate solution for increased transport into the cathode's microstructure.

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Introduction

Solid oxide fuel cells (SOFCs) have great potential in electrical energy generation due to their high-energy conversion efficiency and low greenhouse gas emissions. The elevated exhaust temperatures of SOFCs make them prime candidates for combined heat and power (CHP) applications. Additionally, a SOFC can be combined with a steam or gas turbine, known as a bottoming cycle, to provide very efficient power generation [1]. For example, Bicer et al. proposed a novel system to produce syngas from underground coal without mining to generate electricity from an integrated gasification combined cycle and SOFC [2]. If the DOE/SECA cost targets are reached, Siefert et al. showed that a SOFC coupled to an anaerobic digester would be an economical way of producing electricity from biogas [3].

SOFC performance, long-term stability, and cost are critical to the successful application of fuel cells in the electric power industry. Infiltration, also known as wet impregnation, is one method that has been used to produce electrodes with high electrocatalytic activity and stability by depositing nano-sized particles into the electrode structure [4–9]. Lee et al. previously showed that cathode infiltration can reduce polarization resistances of baseline cells with no measurable negative effect on stability [10,11]. They confirmed that infiltrated electrocatalysts play an important role in the reduction of oxygen molecules and adsorption of oxygen atoms in the cathode catalyst layer [12].

Currently, the wet impregnation infiltration process for the cathode requires multiple iterations using a low electrocatalyst concentration in order to prevent agglomeration at the cathode's surface while also depositing a sufficient amount of electrocatalyst at the cathode active layer to

* Corresponding author. Address: 1530 W. 15th, Lawrence, KS 66045, USA. Tel.: +1 352 514 5579.

E-mail address: RegisDowdJr@gmail.com (R.P. Dowd).

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positively impact performance and degradation [13–15]. Li et al. established a one step infiltration method by submerging a tubular fuel cell into an electrocatalyst and then heating the solution [13]. Others have demonstrated how SDC and $\text{La}_{0.80}\text{Sr}_{0.20}\text{MnO}_{3-\delta}$ (LSM) infiltration enhance the performance of tubular SOFCs [16–18]. Additionally, Zhou et al. infiltrated planar SOFCs by using a vacuum dip-coating method [19]. In these approaches, the electrocatalyst cannot be tailored for the anode and cathode independently. Moreover, these processes require additional energy input in the form of heat and vacuum, which makes it more challenging for integration into an existing manufacturing process. Chen et al. demonstrated that a single step wet infiltration process is able to deliver sufficient LSM electrocatalyst into a bismuth oxide porous backbone in order to increase performance and durability [20]. It is highly desired to determine the optimal conditions for wet impregnation infiltration in order to shorten the manufacturing process to a single step, thereby greatly reducing the overall time and cost.

The key variables for optimizing the wet infiltration process include surfactant type and concentration, chelating agent type and concentration, substrate temperature, pH level, and electrocatalyst infiltrate concentration. Godinez et al. previously studied the effect of different surfactants and pH levels on the transport of nanoparticles in saturated porous media [21]. Godinez et al. found that transport of nanoparticles in saturated porous media was highly dependent on the type of surfactant added to the solution [21]. Additionally, Zhu et al. studied the effect of various surfactants, chelating agents, and pH on SOFC performance [22]. Starov et al. found that mean pore size in porous media plays a critical role on the effect of surfactant concentration on permeability of the porous media [23]. Depending on the mean pore size, surfactants can have a positive effect of liquid transport through the porous media [23]. Based on these previous studies, we expect that adding a surfactant will achieve a lower surface tension between the cathode backbone and infiltrate solution, thus enabling the infiltrate to more easily penetrate into the cathode.

Surfactants are versatile materials used in diverse industries (i.e. chemical, pharmaceutical, cleaning, food, etc.). Surfactants lower the surface tension between two materials, such as the interface between two liquids or a liquid and a solid. One way to measure the effect of a surfactant on a liquid's surface tension is by using a goniometer. A goniometer is an instrument that can measure a liquid's surface tension, as well as the contact angle of a liquid in contact with a solid. The contact angle is typically calculated by first capturing a digital image of the droplet's side view, such that the liquid–solid surface is in the same plane as the camera's viewing angle. Then, image analysis software can be used to precisely measure the angle formed at the outer edge of the liquid droplet between the liquid–air boundary and solid–air boundary. In this study, a goniometer will be used to measure the effect of various types of surfactants and surfactant concentrations on infiltrate surface tension and contact angle with the cathode backbone material.

A suitable surfactant concentration must be chosen in order to sufficiently reduce the infiltrate's surface tension, while also keeping the amount of surfactant added to the

infiltrate to a minimum. One method of characterizing the amount of surfactant added to a liquid is by quantifying the critical micelle concentration (CMC). CMC is the surfactant concentration above which all additional surfactant goes into making micelles instead of individual surfactant molecules in solution. The CMC is important because typically a solution's surface tension lowers sharply when approaching CMC, but changes much less above the CMC. We will use the CMC literature values for the chosen surfactants to quantify the amount of surfactant added to the infiltrate solution.

Chelating agents are compounds that can form multiple bonds to a single metal ion. Citric acid is a chelating agent that has been shown to homogeneously disperse metal ions through a process called sol–gel citrate method, which makes it easier to form the perovskite oxide structures. The sol–gel method results in good stoichiometric control and particle size distributions [24]. Ghiasi et al. showed that elevated citric acid concentrations above a 1:1 M ratio (moles of citric acid: moles of metal cations) had a negative effect on the formation of LaMnO_3 perovskite crystalline phase [25]. Nguyen et al. also found that using citric acid in a 1:1 M ratio assisted with making highly dispersed LaCoO_3 perovskite [26]. Taguchi et al. found that lower citric acid molar concentrations (0.5:1 M ratio) resulted in good surface crystallinity and showed the highest catalytic activity for CO oxidation [27]. Previous research supports the viewpoint that the chelating agent concentration should be no more than a 1:1 M ratio in order to promote good crystallinity and phase formation.

Our goal was to develop a one-step infiltration method to deposit 8–10 weight percent of an electrocatalyst into the $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$ (LSCF) – $\text{Ce}_{0.8}\text{Sm}_{0.2}\text{O}_2$ (SDC) cathode of a planar SOFC using an automated solution dispensing technique. The electrocatalyst used in this work was $\text{La}_{0.6}\text{Sr}_{0.4}\text{Co}_{0.3-\delta}$ (LSCo). We determined the effect of various compositional changes on the fuel cell performance and durability. The results of this experiment will guide the commercial deployment of infiltrated SOFCs by industry.

Experimental

LSCo electrocatalyst solutions were prepared at different molar ratios ranging from 0.5M to 1.0M using aqueous solutions of $\text{La}(\text{NO}_3)_3 \cdot 6\text{H}_2\text{O}$ (Alfa Aesar, 99.9%), $\text{Sr}(\text{NO}_3)_2$ (Alfa Aesar, 99.0%), $\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$ (GFS Chemicals), and citric acid (Alfa Aesar, 99.5%). LSCo was observed to precipitate at 1.2M when held at room temperature. Citric acid was added as a chelating agent to complex with the metal ions and to assist with forming the correct LSCo phase upon calcination. The citric acid concentration was based on the concentration of metal ions in solution. When the moles of citric acid equaled the moles of metal ions, the mixture was designated 1:1 for identification purposes. Other citric acid concentrations were tested, including 1:0.33 (moles of metal ions: moles of citric acid) and 1:0.67. LSCo solutions were also prepared using the following surfactants: Triton™ X-100 (Sigma–Aldrich), sodium dodecylbenzenesulfonate (SDBS) (Sigma–Aldrich), and sodium dodecyl sulfate (SDS) (Sigma–Aldrich). The LSCo surfactant solutions were sonicated for 1 h to ensure adequate mixing and uniformity. The surfactants were added according

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