

Synthesis and characterization of $\text{Ce}_{0.6}\text{Sr}_{0.4}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$ perovskite material: Potential cathode material for low temperature SOFCs

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Abstract: A sol-gel method and a modified chemical vapour deposition technique were used to produce nanostructured $\text{Ce}_{0.6}\text{Sr}_{0.4}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$ materials at temperatures as low as 400 °C. Powders were characterized using Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, thermo gravimetric analysis, powder X-ray diffraction (XRD), scanning electron microscopy (SEM) with energy dispersive X-ray spectroscopy, high resolution transmission electron microscopy (HRTEM), and nitrogen sorption at 77 K. FTIR spectra showed that the sol-gel method resulted in residual carbon groups on the materials after calcination, while the Raman and XRD analysis confirmed that both synthesis methods resulted in cubic perovskite structure. However, the chemical vapour deposition (CVD) method resulted in materials with a smaller crystallite size when compared to those prepared via the sol-gel route. The overall morphology of the powders was irregularly shaped aggregated particles as observed by SEM and HRTEM. In addition, HRTEM analysis showed that the materials were highly crystalline. Textural analysis revealed the powders had some mesoporosity, and the surface areas were 76.69 and 65.90 m²/g for materials synthesized using the CVD and sol-gel methods, respectively. The synthesized perovskite powders were used to fabricate button cells employing samarium doped ceria (SDC) as the electrolyte and NiO/SDC as the anode materials. As cathode materials, the maximum power density observed was 308.4 mW/cm² at 500 °C.

Keywords: nanostructured materials; low temperature SOFCs; perovskites; ceria; rare earths

Solid oxide fuel cells (SOFCs) are electrochemical energy conversion devices that produce electricity through chemical reactions between an oxidant and a fuel gas. Typically, air is supplied to the cathode and then molecular oxygen is reduced at the porous air electrode (cathode) to give oxygen anions^[1,2]. The produced ions migrate through the solid electrolyte, which can be the standard yttria-stabilized zirconia (YSZ) or samarium doped ceria (SDC), to the fuel electrode, and react with the oxidized fuel, H₂ or a hydrocarbon (e.g. CH₄), to give H₂O or CO₂ plus heat and electricity. The advantages of SOFCs over other types of fuel cells are the higher energy efficiency and excellent fuel flexibility^[3-5]. Applications of SOFCs can be found in mobile and also in stationary energy systems. In terms of stationary SOFC systems, prototype and commercial systems have been deployed as heat and power systems for a house, factory or off grid systems for a small settlement^[6-10]. In terms of transportation systems and portable equipment, some SOFC systems have been developed and demonstrated as auxiliary power units for commercial trucks, and as portable generators for home-based and commercial environments^[6,9-13].

In general, SOFC processes are typically run at relatively high temperatures of 800–1000 °C. These higher temperatures are problematic because they result in various forms of degradation, provide immense technological challenges and economic obstacles^[14]. In order to commercialize SOFC technology, operating temperatures need to be further reduced so that more conventional materials can be used. In addition, this will allow more widespread applications in various mobile and portable systems. Unfortunately, SOFC performance decreases as the operating temperature is reduced, and this is especially due to increased ohmic resistance at the cathode. Therefore, low temperature solid oxide fuel cells (LT-SOFCs), operating at 300–500 °C, with improved electrochemical performance have been the subject of extensive investigation for several years and still continue to be an active area of research^[15].

Extensive research on the development of LT-SOFCs has been done by re-designing the cell, reducing the electrolyte thickness, or by developing new materials with high ionic conductivities, high electrolytic activity, low chemical reactivity and superior physical characteristics^[4,8,14]. One promising area of research is the use of

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materials with high mixed ionic-electronic conductivities (MIECs), such as perovskites. The introduction of perovskite materials as cathodes has been found to be a promising strategy for developing LT-SOFCs with enhanced electrical characteristics^[16–18]. Previous researches on perovskites as candidate materials for SOFCs have found that the overall performance of the system depends on not only the operating temperature of the system, but also the particle size and morphology^[17,19]. Some past research efforts have focused on improving the electrochemical performance of LT-SOFC materials by reducing the particle size of the material through various synthetic approaches such as sol-gel methods^[4,20].

The parameters of synthesis (e.g. temperature) play a significant role in determining the microstructure of SOFC materials. Lower synthesis temperature may help in the overall cost reduction when processing precursors to produce SOFC materials, and little is known about the performance of these materials in SOFCs operating at temperatures of 300–500 °C. The present work is focused on the synthesis and characterization of the perovskite cathode nanomaterials and preliminary testing for LT-SOFCs. Two synthesis methods are compared; these are a modified sol-gel methodology, and a dry mix metal organic chemical vapour deposition (MOCVD) method, both operating at relatively low temperatures. One of the main problems with SOFCs is the high polarization at the cathode during the reduction of oxygen^[21]. Improvement can be achieved by reducing the particle size to the nanoscale, hence, increasing the active area of the material^[17]. The use of nanostructured materials seems as the most promising strategy at these temperatures with the high surface area compensating for the increase in resistance at lower temperatures^[17,22]. Perovskites such as $\text{Pr}_{0.6}\text{Sr}_{0.4}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_3$ (PSFC) have shown promising performance for SOFCs operating at temperatures around 700 °C^[17], and recently a $\text{Y}_{0.8}\text{Ca}_{0.2}\text{BaCoFeO}_{5+\delta}$ perovskite has good operational characteristics between 550 and 650 °C^[23]. Further reduction of temperatures seems possible. Therefore, this study was focused on the synthesis and electrochemical characterization of $\text{Ce}_{0.6}\text{Sr}_{0.4}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$ (CSFC) nanoparticles cathodes for SOFCs operating at temperatures of 300–500 °C under asymmetrical cell using NiO-SDC as the anode and SDC as the electrolyte. Furthermore, the high costs of the current SOFC fabrication processes can be reduced by replacing expensive praseodymium (Pr) with the more abundant and commercially available cerium (Ce) in the A-site of the ABO_3 structure^[14,19].

1 Experimental

1.1 Materials

Pluronic F-127, cerium (III) acetylacetonate hydrate

(99.9 wt.%), strontium (II) acetylacetonate hydrate (98.5 wt.%), iron (III) acetylacetonate (99.99 wt.%), and cobalt (III) acetylacetonate hydrate (99.9 wt.%) were purchased from Sigma Aldrich (South Africa, Durban) and were used without further purification. Deionized water was employed during the whole synthesis (sol-gel method). Crude ethanol was used for the preparation of polymeric solutions (sol-gel). The powders of sodium carbonate (NaCO_3) and nickel carbonate (NiCO_3) used for electrolyte preparation and as the anode precursor were purchased from Sigma Aldrich (South Africa, Durban) and were used as received.

1.2 Synthesis

1.2.1 Surfactant preparation

The solvent mixture was prepared by adding 155 mL of ethanol and 45 mL of deionized water in a 200 mL Erlenmeyer flask and mixed thoroughly by shaking and inverting the flask several times. The solution was then transferred to four 50 mL volumetric flasks (four aliquots). Pluronic F-127 (6.00 g) was added as the surfactant to the four different 50 mL solvent system solutions. The solutions were then sonicated for 5 min and left covered with foil in a fume hood until they were ready for further use.

1.2.2 Synthesis of $\text{Ce}_{0.6}\text{Sr}_{0.4}\text{Fe}_{0.8}\text{Co}_{0.2}\text{O}_{3-\delta}$ perovskites and samarium doped ceria electrolyte

In the MOCVD method, stoichiometric amounts of the commercial metal acetylacetonates (acac), 0.275 g cerium acetylacetonate, 0.114 g strontium acetylacetonate, 0.282 g iron acetylacetonate, and 0.0721 g cobalt acetylacetonate were weighed and transferred into a mortar, and then ground with a pestle for homogenization of the mixture for about 2 min. For the sol-gel method, the metal acacs were weighed stoichiometrically as above. The metal acacs were then transferred into a 100 mL beaker and dissolved with 10 mL of Pluronic F-127 pre-made solution. This mixture was stirred for 15 h before placing the beakers in a convection oven at 100 °C for 2 h until the samples were dry. Each of the different samples, designated CSFCs, synthesized by MOCVD and sol-gel were then packed into a homemade stainless steel reactor assembled from standard SwagelokTM parts for heat treatment. The sealed reactor was placed into a horizontally aligned tube furnace, and then the reactor was connected to vacuum pump with appropriate valves for sealing the steel reactor. After evacuation to approximately 10^{-3} mbar, the valve was closed and the furnace was set to heat slowly to 400 °C with a ramp rate of 1 °C per minute, and held at 400 °C for 8 h after the cooling samples were recovered and taken for characterization.

The electrolyte material was made by adopting and modifying methods reported by Chen et al.^[14]. The electrolyte material ($\text{Ce}_{0.8}\text{Sm}_{0.2}\text{O}_2/\text{Na}_2\text{CO}_3$) was prepared us-

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