La$_{0.6}$Sr$_{0.4}$Co$_{0.2}$Fe$_{0.8}$O$_{3-\delta}$−SDC carbonate composite cathodes for low-temperature solid oxide fuel cells

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HIGHLIGHTS

- Cell performance is among first reported for LSCF−SDC carbonate composite system.
- Correlation between calcination temperatures and cathode properties are reported.
- Low calcination temperature of cathode powder offer cell with excellent power density.
- This study proved LSCF−SDCC has great potential as cathode material for LTSOFCs.

ARTICLE INFO

ABSTRACT

Composite cathodes with La$_{0.6}$Sr$_{0.4}$Co$_{0.2}$Fe$_{0.8}$O$_{3-\delta}$ (LSCF6428) and samarium-doped ceria carbonate (SDCC) were developed and investigated for solid oxide fuel cell (SOFC) applications at low operating temperatures. The LSCF−SDCC (50 wt% LSCF:50 wt% SDCC) composite cathode powders were mixed by the ball-milling technique followed by calcination at 700, 750, 800, and 850 °C for 2 h. To fabricate single SOFCs using LSCF−SDCC as the cathode, SDCC as the electrolyte, and NiO−SDCC as the anode, a single-step pressing and co-firing process was employed. The electrolyte-supported button cells were tested between 475 and 550 °C. The composite cathode exhibited well-matched TEC values with the SDCC electrolyte. Calcination at lower temperatures (700 and 750 °C) produced powders with surface areas of 10 m^2 g^{-1}−11 m^2 g^{-1}. Using LSCF−SDCC powders sintered at 700 and 750 °C to fabricate button cells resulted in power densities of 117.9 and 120.4 mW cm^{-2}, respectively, at an operation temperature of 550 °C with the latter cathode yielding the lowest R_p value.

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1. Introduction

High power output and long-term performance stability are the most important criteria for low-temperature solid oxide fuel cells (LTSOFCs). A low operating temperature ranging from 400 °C to 600 °C is needed to overcome problems associated with high-temperature operation, such as densification of electrodes, large differences among the thermal expansion coefficients (TECs) of cell components, rapid degradation of electrode and electrolyte materials as well as the high cost of interconnection materials. For the last two decades, research on durability of SOFC has been carried out in order to overcome the degradation problems [1]. The current technique for overcoming problems related to the use of high-temperature solid oxide fuel cells (SOFCs) while maintaining high power output is used to lower the operating temperature of SOFCs [2–4]. Consequently, great efforts have been exerted to develop solid electrolytes with high ionic conductivity at low temperatures, especially doped ceria carbonate composite electrolytes [5–8].

Apart from using electrolytes with desirable properties, the electronic and ionic conductivities of cathodes can be enhanced by adding a second phase. Kiros et al. asserted the addition of 20 wt% AgO (high electronic conductivity) to Ca$_{0.9}$La$_{0.1}$MnO$_3$ perovskite cathode (good oxygen ion conductivity) has been able to promote faster electrode dynamics and achieve higher performances of fuel cell [9]. Other perovskite cathode, La$_{0.6}$Sr$_{0.4}$Co$_{0.2}$Fe$_{0.8}$O$_{3-\delta}$ (LSCF6428)

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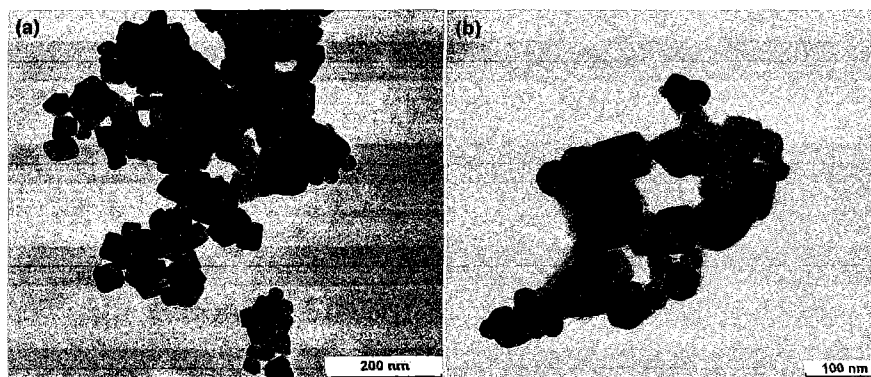
Fig. 1. TEM micrographs of (a) SDCC electrolyte and (b) NiO–SDCC anode powders calcined at 680 °C.

is reportedly compatible with doped ceria electrolytes such as samarium-doped ceria (SDC). In LSCF6428, improving the three-phase boundary (TPB) enables a more efficient chemical reaction to occur, thereby enhancing the cell performance [2,10,11]. At the moment, fuel cells based on LSCF–carbonate salt composite cathodes and composite electrolytes are said to have excellent and promising optimal performance. Thus, the application of these materials in the development of LTSOFCs has gained considerable attention [8,12,13]. The presence of molten carbonate salt in LSCF composite cathodes enhances the ionic conductivity of the electrode close to the electrolyte interface and extends the TPB to the entire cathode surface, leading to the adequate performance of this SOFC system [14,15].

Cell performance depends on various parameters, particularly the cathode microstructure, gas diffusion activity, and thickness of the cathode substrate. The cathode microstructure can be manipulated by varying the composite cathode composition. The effect of the cathode composition on cell performance has been extensively studied [16–18]. However, research on the relationship between the calcination or sintering temperature and the characteristics of the starting powders of carbonate cathodes is limited. As a result, this relationship needs to be understood because the starting powders significantly affect the performance and durability of the cathode and SOFCs. Therefore, the impact of the calcination temperature on LSCF carbonate composite cathodes in LTSOFCs must be studied in detail.

In the present study, composite cathodes with submicron LSCF6428 powder and nanosized SDC carbonate (SDCC) were prepared by the simple ball-milling technique. The calcination temperature range of 700 °C–850 °C was employed in the fabrication of the composite cathode LSCF–SDCC powders. The properties of the composite cathode powders were studied in relation to the calcination temperature. The influence of the calcination temperature on the performance of button cells was also investigated.

2. Experimental procedure

2.1. Preparation of LSCF composite cathode powders

LSCF–SDCC composite cathode powders were developed by mixing commercial LSCF6428 powder (Sigma–Aldrich) with nanosized SDCC composite powder developed in-house. The particle sizes of the LSCF6428 powder were 0.3 μm–0.6 μm. The SDCC composite electrolyte contained 20 wt% carbonate (Li₂CO₃/Na₂CO₃). The procedure for the fabrication of SDCC electrolyte powder is described in our previous study [19]. The developed SDCC powder has nanosized particles as shown on the transmission

electron microscopy (TEM) image in Fig. 1(a). Initially, 50 wt% SDCC powder was ball milled (Fritsch Pulverisette 6, Germany) with LSCF6428 powder in ethanol. The milling process was conducted at room temperature using a zirconia jar and balls as grinding media. After milling for 2 h at 550 rpm, the powders were dried overnight at 60 °C in an oven. The dried powders were thoroughly ground in an agate mortar and then calcined at 700, 750, 800, and 850 °C for 2 h at a heating rate of 10 °C min⁻¹ in air. Hereafter, the composite cathode powders are called LSCF–SDCC (700), LSCF–SDCC (750), LSCF–SDCC (800), and LSCF–SDCC (850).

2.2. Characterization of the powders

The phase structure and grain size were examined using an X-ray diffractometer (Bruker D8 Advanced) with Cu Kα radiation ($\lambda = 1.5418$ Å) at room temperature. The diffraction patterns were obtained from the 2θ range of 20°–80° at 0.02° scanning intervals. The resolution or spectral bandwidth of XRD was approximately 3%. A Micromeritics surface area analyser (model ASAP 2010, USA) based on the Brunauer–Emmett–Teller (BET) principle using N₂ gas was employed to measure specific surface areas. TECs were measured using cylindrical samples with alumina as the reference material. The cylindrical samples were heated in air at a rate of 5 °C min⁻¹ from room temperature to 800 °C using a dilatometer (Netzsch DIL 402C, Germany). The morphology of the calcined powders was studied under a field emission scanning electron microscopy (FESEM) system (Zeiss Supra 55VP, Germany).
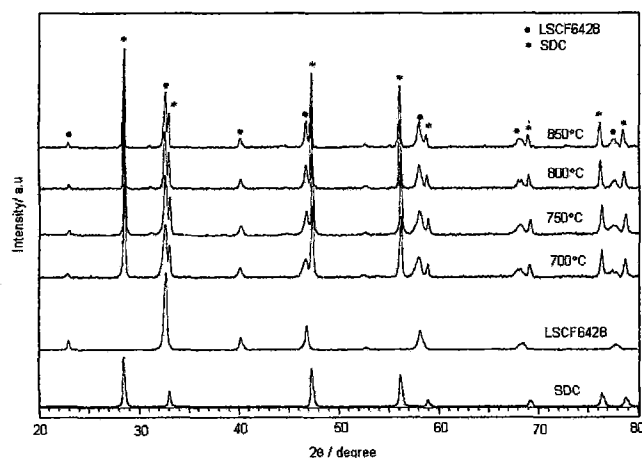

Fig. 2. XRD patterns of SDCC, LSCF6428, and LSCF–SDCC powders calcined at various temperatures.

Table 1

Grain size and surface area of the LSCF–SDCC composite cathodes.

Calcination temperature (°C)	Surface area (m² g⁻¹)	Grain size (nm)	
		LSCF	SDC
700	10.9	22.2	31.3
750	10.1	25.6	35.3
800	7.5	29.5	38.5
850	6.5	31.1	42.3

2.3. Fabrication of cells

Symmetrical cells of LSCF–SDCC/SDCC/LSCF–SDCC with a diameter of 13 mm and a thickness of about 1.4 mm were uniaxially co-pressed. The green half cells were then sintered at 600 °C for 2 h. Electrolyte-supported button cells were produced by a single-step dry pressing and co-firing method. This study used electrolyte-supported button cell as it has been employed in other researches for LTSOFCs [20,21]. The anode material was a mixture of 60 wt% NiO commercial powder (particle sizes less than 50 nm; Sigma–Aldrich) and 40 wt% SDCC. The NiO and SDCC powders were ball milled in propanol medium, dried overnight, and then heated at 680 °C for 1 h (Fig. 1(b)). The composite anode, electrolyte, and cathode powders were fed into a 25 mm-diameter die layer by layer and uniaxially co-pressed at approximately 46 MPa to form a button cell. The thickness of the cathode layer for each button cell was about 0.4 mm. The button cells were sintered in air at 600 °C for 2 h at a heating rate of 2 °C min⁻¹.

2.4. Electrochemical measurements

The electrochemical impedance was measured by a Solatron 1255 Frequency Response Analyser coupled to a 1286 Electrochemical Interface. The impedance data were taken at frequencies ranging from 1 Hz to 100 kHz with an excitation potential of 100 mV. The impedance characteristics were measured in air over the temperature range of 475–550 °C. The sample was placed in a quartz sample holder fitted within a tube furnace. The temperature

of the sample surface was measured using a K-type thermocouple placed directly above the sample surface.

The performance of the electrolyte-supported button cells was evaluated by collecting voltage–current data using an SOFC test machine (Chino Corporation, Japan). Silver paste coated on each side of the electrode and Pt gauze was used as the current collector. The effective working area of the cell was 0.87 cm². The performance of the button cells was tested from 475 °C to 550 °C. Hydrogen was used as fuel and purified air as oxidant were applied at flow rates of 60 and 100 mL min⁻¹, respectively, under 1 atm pressure. For the performance test, three types of button cells with the following cell structures were investigated:

LSCF–SDCC (700)/SDCC/NiO–SDCC   Cell A

LSCF–SDCC (750)/SDCC/NiO–SDCC   Cell B

LSCF–SDCC (800)/SDCC/NiO–SDCC   Cell C

The difference between these button cells was the calcination temperature used in preparing the LSCF–SDCC composite cathode powder. The cell power density was calculated by multiplying the closed circuit voltage with the current density.

3. Results and discussion

3.1. Chemical compatibility and structure analysis

Fig. 2 shows the XRD patterns of the LSCF–SDCC composite powders calcined at different temperatures. The LSCF–SDCC powders exhibited two main structure phases which belong to perovskite LSCF6428 and cubic fluorite SDC. The molten carbonate in SDCC electrolyte powder exists as an amorphous phase and does not influence the phase of the pure SDC. Therefore, only peaks related to SDC and LSCF6428 were observed from XRD for the composite cathode [8,21]. The XRD results confirmed that chemical reactions did not occur between LSCF and SDCC as no additional peak or secondary compound was identified. Similar findings have
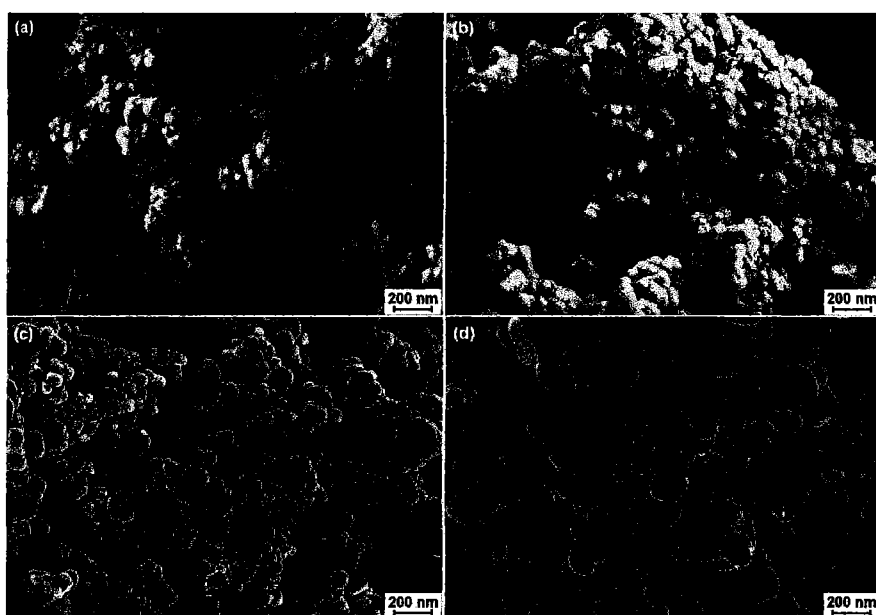

Fig. 3. FESEM micrographs of LSCF–SDCC composite cathode powders calcined at (a) 700, (b) 750, (c) 800, and (d) 850 °C.

been observed in previous studies [12,22–24]. The grain sizes of the particles were calculated using Scherrer's equation:

$$D = \frac{0.9\lambda}{\beta_{1/2}\cos\theta} \quad (1)$$

where D is the grain size (nm), λ is the radiation wavelength (0.15406 nm in this case, Cu target), θ is the diffraction angle, and $\beta_{1/2}$ is the corrected line width at half-peak intensity.

The effect of the calcination temperature on the grain size and surface area of the powders is given in Table 1. Increased calcination temperature resulted in enlarged LSCF and SDCC crystallites. The BET specific surface area decreased from 10.9 m^2 g^{-1}–6.1 m^2 g^{-1} when the calcination temperature was increased from 700 °C to 850 °C. A similar relationship between the grain size and surface area was observed by Ghouse et al. [25]. The obtained surface areas were comparable with those of other composite cathodes in a previous research [26]. The increase in grain size was due to particle growth during the calcination, which contributed to the reduction in the surface area [27]. However, the increment in the grain size of the powders was able to be controlled at nano range through the application of the high energy ball-milling process. It has been proven by several studied that high energy ball-milling can promote the breaking down of particles to a much smaller size [28,29]. The LSCF–SDCC composite cathode demonstrates good chemical compatibility as well as the appropriate grain size and surface area. Hence, this cathode can produce porous composite cathodes at low calcination temperatures [2,23,30,31] for intermediate to low temperature SOFCs.

3.2. Thermal expansion and microstructural analysis

The technical TEC values for the LSCF–SDCC composite cathodes were measured at a low temperature range (100 °C–550 °C). Calcination temperature did not significantly influence the TECs of the composite cathodes. The LSCF–SDCC composite cathodes which were calcined at 700 °C–850 °C had TEC values between 3.0 × 10^{-6} and 3.6 × 10^{-6}. A difference of less than 10% was found between the TECs of LSCF–SDCC and SDCC electrolyte (3.36 × 10^{-6}). Mechanical compatibility prevents delamination during processing or applications at elevated temperatures [32]. Hence, the LSCF–SDCC composite cathode is expected to match SDCC electrolyte for the fabrication of SOFCs. Fig. 3 shows the morphology of the LSCF–SDCC composite cathode powders calcined at different temperatures. The calcination temperature significantly influenced the particle sizes of the composite cathode powders. High calcination temperatures allowed the growth of powder particles. These observed changes in particle size were consistent with the surface area data, that is, decreased surface area due to increased powder particle size [27,33]. Therefore, this study shows that low calcination temperatures can lead to the production of smaller particle sizes with higher specific surface areas.

3.3. Performance of cells

The performance of the electrolyte-supported button cells was investigated from 475 °C to 550 °C. Hydrogen was used as fuel and air served as oxidant. Fig. 4(a)–(c) shows the plots of the cell voltage and resultant power densities as a function of the single-cell current density using LSCF–SDCC composite cathode powders calcined at 700, 750, and 800 °C. The open circuit voltages (OCVs) of all button cells were lower than the theoretical values [34] within the operating temperature. The OCVs ranging from 0.79 V to 0.93 V were comparable with the values in several publications [5,17,31,35]. The low OCVs were due to the application of

ceria-based electrolytes, which perform electronic conduction in the SDC phase. Similar observations have been reported [17,35,36]. A composite electrolyte with a low density may cause gas crossover, which reduces cell performance. The existence of the carbonate phase in a composite electrolyte also lowers OCVs when volume changes occur during the transition from the molten to the solid phase of carbonate [36].

The output power and current densities for each cell increases with an increased operating temperature. The current densities at 0.6 V for cells A, B, and C were 198.0, 201.5, and 119.3 mA cm^{-2}, respectively, at 550 °C. The power density at 0.6 V, 120.4 mW cm^{-2} at 550 °C, was achieved by cell B, which was followed by cell A
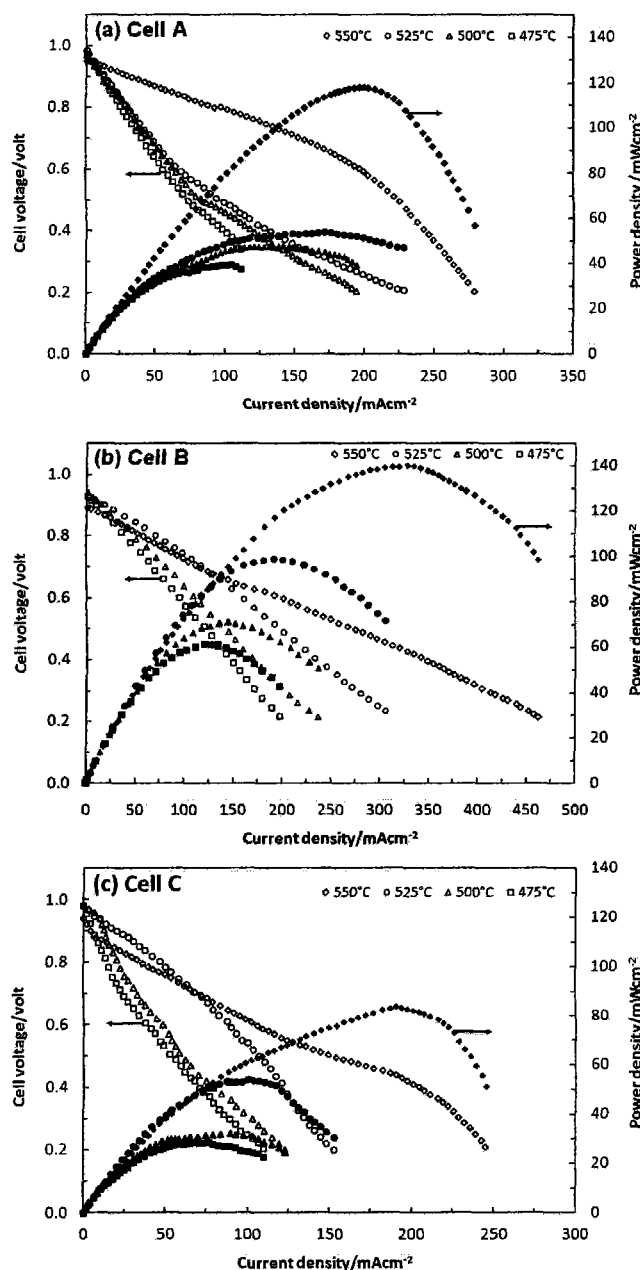

Fig. 4. Current–voltage (open symbols) and power density (solid symbols) characteristics of the electrolyte-supported button cells: (a) cell A, (b) cell B, and (c) cell C. The cathode component of each cell was fabricated using LSCF–SDCC powders calcined at 700, 750, and 800 °C, respectively.
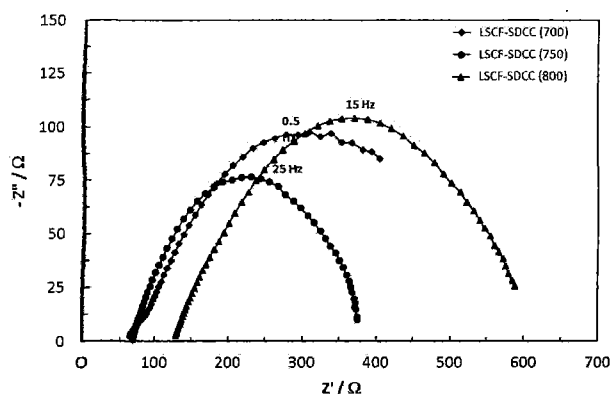
Fig. 5. Impedance spectra of symmetrical cells with different LSCF–SDCC composite cathodes measured in air at 550 °C.

(117.9 mW cm^{-2}) and cell C (71.8 mW cm^{-2}). These results were higher than or comparable with some previous reports on single cells with LSCF composite cathode tests at equivalent temperatures, for example, 35 mW cm^{-2} at 550 °C and 55 mW cm^{-2} at 600 °C [17], 27 mW cm^{-2} at 550 °C and 75 mW cm^{-2} at 650 °C [31], 92 mW cm^{-2} at 550 °C [5], 45 mW cm^{-2} at 550 °C [16], 238 mW cm^{-2} at 535 °C [37], 700 mW cm^{-2} at 600 °C [38]. The higher power densities acquired from cells A and B can be attributed to the finer particle sizes of the LSCF–SDCC composite cathode powders (Fig. 3). The higher surface areas of the powders (Table 1) increased the TPB within the electrode for a higher catalytic reaction, and the TPB increase enhanced the cell performance. Analogous trends have been reported in previous studies [4,39,40].

Fig. 5 illustrates the impedance data measured at 550 °C for the different LSCF–SDCC/SDCC/LSCF–SDCC cells. The intercepts of the impedance arcs with the real axis at high frequencies correspond to the ohmic resistance, R_0, of the electrolyte and contact wires. The symmetrical cell with the LSCF–SDCC (800) cathode gave a higher R_0 compared to the other composite cathodes at 550 °C. The increment of R_0 could be caused by the poor electrode/electrolyte interface. In addition, all the spectra feature one dominating semicircle at low frequency, which is attributed to cathode polarization resistance, R_p. This phenomenon indicates that diffusion and exchange of oxygen species is the main rate-limiting mechanism [2,41]. The symmetrical cell with the LSCF–SDCC (750) cathode exhibited the lowest R_p (319.09 Ω), followed by LSCF–SDCC (700) and LSCF–SDCC (800) with values of 466.00 Ω and 483.81 Ω respectively. The impedance results are consistent with the performance analysis, where the composite cathode LSCF–SDCC (750) with the lowest R_p generated the highest power density at 0.6 V and 550 °C of a button cell (Cell B).

In the present work, conductivity analysis was done for the LSCF–SDCC composite cathode, pure LSCF and SDCC electrolyte. At 550 °C, the SDCC electrolyte gave a good value of ionic conductivity which is 0.131 S cm^{-1} as required for SOFC application [24]. As for cathodes, the LSCF–SDCC (750) composite has been identified to provide higher conductivity, 9.53 × 10^{-4} S cm^{-1} compared with the LSCF6428 (8.0 × 10^{-4} S cm^{-1}) at 550 °C. This result support that the electrochemical characteristics for the LSCF–SDCC55 (750) composite is appropriate and meets the criteria as LT-SOFC cathode material.

The performance of low-temperature cells does not only depend on the calcination or sintering temperatures of the cathode. Other factors such as the thickness of the cathode and electrolyte as well as the porosity of both components must also be optimized to achieve outstanding cell performance. Studies on the long-term stability of SOFCs with this composite cathode are considered for further investigation in the future.

4. Conclusions

LSCF–SDCC cathodes with excellent chemical compatibility, appropriate grain sizes, and higher surface areas were obtained by calcining composite powders between 700 and 850 °C. The influence of the calcination temperature on the cathode resistances and the performance of button cells were also investigated. At 550 °C, power densities of 120.4 and 117.9 mW cm^{-2} (550 °C operating temperature) were obtained from a button cell using LSCF–SDCC cathode powders calcined at 750 and 700 °C, respectively, even when other factors were not optimized. The results signify the potential of LSCF–SDCC as a promising cathode with ceria carbonate composite electrolyte for LTSOFCs.

Acknowledgements

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