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Influence of sintering temperature on the power density of samarium-doped-ceria carbonate electrolyte composites for low-temperature solid oxide fuel cells

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Abstract

The effects of sintering temperature on the current–voltage (*I–V*) performance of symmetrical cells with an SDCC electrolyte composite were evaluated in this study. The SDCC electrolyte composite was prepared by mixing 80 wt% SDC with 20 wt% Li and Na carbonate (Li/Na ratio of 2/1) via a solid state reaction method. The resultant SDCC electrolyte composite powder was uniaxially pressed into a circular pellet and sintered at 500 °C, 550 °C, 600 °C and 650 °C for 5 h. A symmetrical cell was finally fabricated by painting a conductive silver paste on both sides of the dense SDCC electrolyte composite pellet, and its *I–V* performance was measured using hydrogen and air as the fuel and oxidant, respectively. The phase structure and microstructure of the composite powders were investigated using an X-ray diffractometer, field emission scanning electron microscope, and transmission electron microscope. Ceramic pellets sintered at 550 °C exhibited a maximum power density of 63.3 mW/cm^2 and an open circuit voltage of 1.14 V at an operating temperature of 650 °C. This study contributes to the development of SDCC electrolyte composite substrates for electrolyte-supported low-temperature SOFCs.

Keywords: A. Sol–gel processes; A. Sintering; B. Composites; C. Electrical properties

1. Introduction

Solid oxide fuel cells (SOFCs) are well known for their ability to produce clean energy using a combination of hydrogen and oxygen fuels. However, despite their many advantages, such as high efficiency, fuel flexibility, and absence of noble catalytic metals, SOFCs have high operating temperatures ranging from 800 °C to 1000 °C. Many researchers have observed that the performance of SOFCs improves when the operating temperature is reduced to ranges from 400 °C to 600 °C [1,2]. Low-temperature SOFCs (LT-SOFC) can significantly improve the compatibility, manufacturing cost, and stability of many materials [1]. Hence, extensive research and other development work are necessary to develop new electrolyte materials with better ionic conductivity to reduce the operating temperature of SOFCs. Sm^{3+} or Gd^{3+}-doped-ceria-based materials are more promising electrolyte materials compared with conventional electrolyte yttria-stabilized zirconia (YSZ) for LT-SOFC applications operating below 500 °C [1].

Samarium-doped-ceria (SDC) is considered by many researchers to be the most promising electrolyte material for LT-SOFC applications due to its superior ionic conductivity, excellent stability, and compatibility with other

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components in cells operating at low temperature [1]. However, SDC has several defects, such as its mixed ionic-electronic conducting behavior, which decreases the overall efficiency and mechanical stability of cells [3]. Many researchers have attempted to resolve the problems associated with SDC by introducing a small amount of alkaline salt to the compound to increase its ionic conductivity and suppress electrical conduction [4]. Among the many materials tested thus far, SDC-carbonate (SDCC) electrolyte composites show the best performance at low temperatures [5]. These composites act as co-ionic (O^{2-}/H^{+}) conductors with an ionic conductivity of 0.1 Scm^{-1} below 600 °C. The quality of SDCC electrolyte composite powders strongly depends on their synthesis methods, composition and calcination temperature [1], all of which also determine the physical and chemical properties of the powders, such as their specific surface area, pore size and volume, and porosity; all of these properties greatly influence the electrolytic properties of the compound. The thermal treatment and surface roughness of the surface also influence the ionic conductivity of the composite electrolyte. Thus, to achieve maximum efficiency, both the material and microstructure of the individual components must be optimized [6].

The overall performance of single cells with SDCC composites as electrolytes has been reported elsewhere [7–10]. However, to our knowledge, the effects of sintering temperature on the microstructure and the cell performance characteristics of the SDCC composite have not been systematically investigated. In this paper, we report a detailed investigation of the significant effect of sintering temperature on the microstructure and the electrochemical behavior of the SDCC composite cell. The main objective of this work is to identify an appropriate sintering temperature that can produce a suitable electrolyte substrate for electrolyte-supported LT-SOFC applications. The electrical properties of SDCC composites depend on the volume fraction of the Lithium and sodium (Li/Na) carbonate and on the ceramic sintering temperature [11]. From the literature, Huang et al. [11] reported that, at lower (Li/Na) carbonate contents (20 wt% to 25 wt%), the SDCC electrolyte composite shows an excellent performance of 1085 mW/cm^2 at lower operating temperatures ranging from 400 °C to 600 °C.

In this paper, the (Li/Na) carbonate content and SDC of the composite electrolyte are maintained at 20 wt% and 80 wt% respectively. The effect of sintering temperature on the composite electrolyte is reported by *I–V* performance measurement with hydrogen and air as the fuel and oxidant, respectively. Both sintering and operating temperatures are maintained between 500 and 650 °C to avoid deterioration of (Li/Na) carbonate from the SDC phase. The phase and microstructure of the SDCC composite powder and pellets sintered at various temperatures are also investigated in this study.

2. Experimental procedure

SDC was prepared using the sol–gel technique assisted by a citric acid-based method. All of the reagents were purchased from Sigma Aldrich, Malaysia, with 99.8% purity. Stoichiometric amounts of $Sm(NO_3)_3 \cdot 6H_2O$, $Ce(NO_3)_3 \cdot 6H_2O$, and citric acid powder were dissolved in distilled water with magnetic stirring. The molar ratio of the total metal ion:citric acid was 1:1.2. The transparent solution mixture was heated to 80 °C to allow water to evaporate, forming a transparent viscous gel. The gel was heated continuously at a lower temperature to initiate combustion, forming of a pale yellow colored ash. The resulting ash was placed overnight in a pre-heated oven at 120 °C (12 h) to complete the combustion reaction. The dried powder was calcined at 850 °C for 5 h to obtain the desired SDC powder [10].

The (Li/Na) carbonate was prepared by high-speed ball milling (Fritsch Pulverissete 6, Germany) of lithium carbonate (Li_2CO_3) and sodium carbonate (Na_2CO_3) at a molar ratio of 2:1 for 16 h and then drying overnight at 200 °C. The (Li/Na) carbonate and SDC powder were mixed thoroughly by wet ball milling for 16 h in ethanol at a weight ratio of 2:8, and then dried in an oven at 120 °C for 12 h to evaporate the ethanol. The resultant mixture was heated in a furnace at 680 °C for 1 h to produce the SDC-carbonate (SDCC) electrolyte composite powder.

2.1. Characterization

The thermal decomposition behaviors of the (Li/Na) carbonate were investigated by thermogravimetry (TG)/differential scanning calorimetry (DSC) (Model 851, Mettler) at the temperature range from 25 °C to 800 °C with a heating rate of 3 °C/min in air. X-ray diffraction (XRD) patterns were recorded at room temperature using an X-ray diffractometer (Shimadzu XRD-6000) with CuKα ($\lambda = 1.5418$A°) radiation and 2θ ranging from 20° to 80° to identify the crystalline phase of the pure SDC and SDCC composite powders. A field emission scanning electron microscope (FESEM, Zeiss Supra-55VP) was used to examine the microstructure, particle size, shape, and morphology of the pure SDC and SDCC composite powders. The microstructure and particle size of the prepared powders was observed by a transmission electron microscope (TEM, Philips CM12) operating at 80 kV. TEM was used to detail the microstructure of the prepared powders. The sample for TEM analysis was prepared by dispersing the powders in ethanol using ultrasonic bath. A drop of the dispersed suspension was deposited on 150-mesh carbon-coated Cu grid for analysis.

2.2. Electrolyte pellet fabrication and performance

The composite SDCC electrolyte powders were cold-pressed at 80 MPa into cylindrical pellets (25 mm diameter and 1 mm thickness) using a uniaxial die-press. The as-prepared green pellets were then sintered at 500 °C, 550 °C, 600 °C, and 650 °C for 5 h in air. The porosity of the sintered pellets was determined by the Archimedes method with propanol at room temperature [12]. For *I–V* performance measurement, Symmetrical cells were prepared by coating silver conductive paint on both sides of the pellets to form silver/SDCC/silver and heated at 150 °C for 15 min. The effective working area of the pellet was 0.78 cm^2. During the measurements, an alumina tube was employed as the testing holder of the pellet, assisted by a platinum mesh on two sides and sealed by a glass O-ring. The cells were tested at temperatures ranging from 500 °C to 650 °C using hydrogen and air as the fuel and oxidant, respectively. Gas flows of air and hydrogen were controlled to 60 mL/min and 100 mL/min, respectively, under 1 atm pressure. The cell performances were measured using a computerized solid oxide fuel cell testing machine (CHINO, Japan). The *I–V* curves were measured with a Multi-channel Potentiostat/Galvanostat (Autolab AUT302 FRA) with a computer interface and NOVA software.

3. Results and discussion

Fig. 1 shows the DSC curves of the (Li/Na) carbonate prepared by milling mixing of Li_2CO_3 and Na_2CO_3 measured as a function of temperature ranging from 25 °C to 800 °C. The two endothermic peaks observed at 89 °C and 503 °C correspond to small weight losses due to the evaporation of moisture and melting of (Li/Na) carbonate, respectively. A typical melting peak observed above 500 °C indicates the formation of binary eutectic salts of (Li/Na) carbonate. The DSC curves are similar to those reported by Huang et al. [11] for (Li/Na) carbonate powders. No melting peak was observed beyond 503 °C, indicating that the mixed (Li/Na) carbonate are capable of forming eutectic salts using the simple and inexpensive method of dry ball milling.

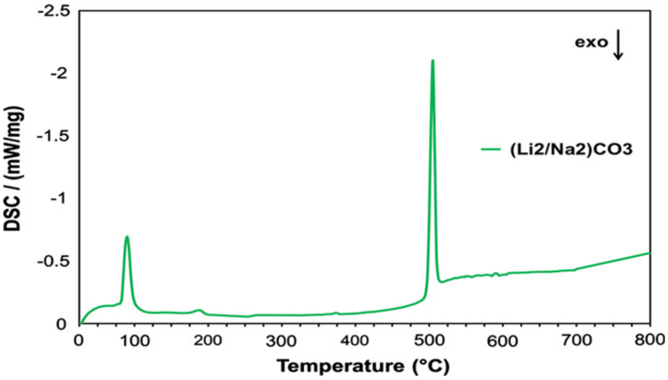

Fig. 1. DSC curve of the (Li/Na) carbonate prepared by milling mixing of Li_2CO_3 and Na_2CO_3.

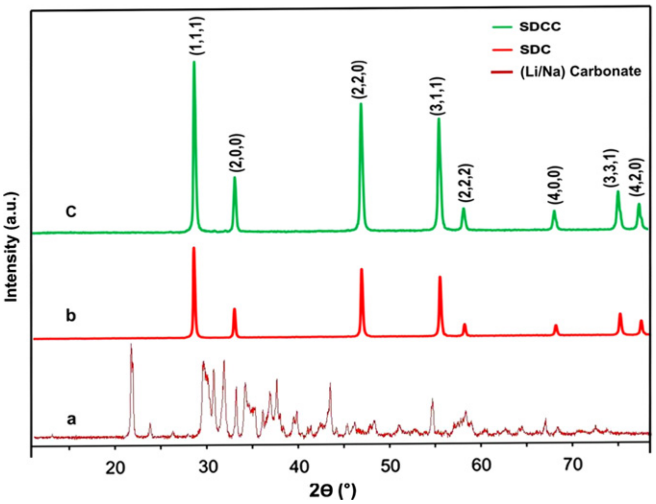

Fig. 2. XRD pattern of the (a) (Li/Na) carbonate powder, (b) pure SDC particle and (c) SDCC composite powder.

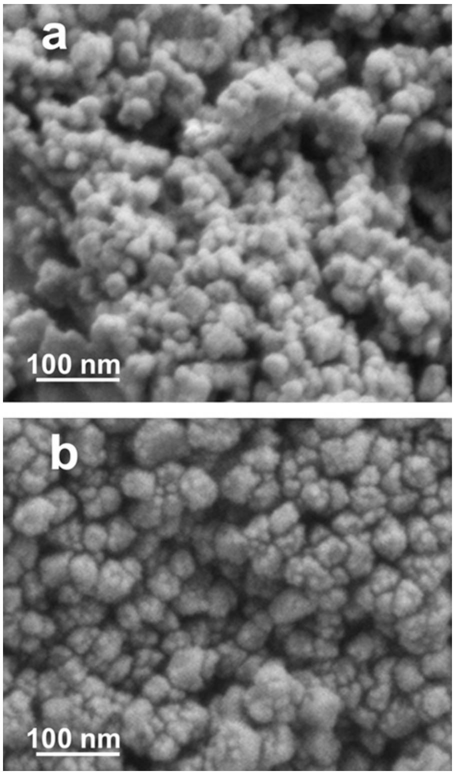

Fig. 3. FESEM images of the (a) pure SDC particle and the (b) SDCC composite.

The phase of the composite powders was identified by XRD. The XRD patterns of SDC-carbonate composite powders prepared using the solid-state reaction method, pure SDC powder and (Li/Na) carbonate powder, are presented in Fig. 2. The prepared composite electrolyte only exhibited a cubic fluorite structure with a space group of Fm3m (JCPDS File no. 34-03 94), representing the SDC phase. Thus, only the SDC phase was observed in the XRD patterns, with no new peaks appearing for the prepared composites containing amorphous (Li/Na) carbonate. Moreover, incorporation of the (Li/Na) carbonate did not alter the phase structure of the ceramic and did not form a chemical reaction or a new compound between the SDC and the (Li/Na) carbonate phase, similar to other reported studies [13–15]. By comparing the XRD pattern for SDCC composite electrolyte powder with that of pure (Li/Na) carbonate powder, no extra peaks can be seen. Therefore, the (Li/Na) carbonate particles exist mainly

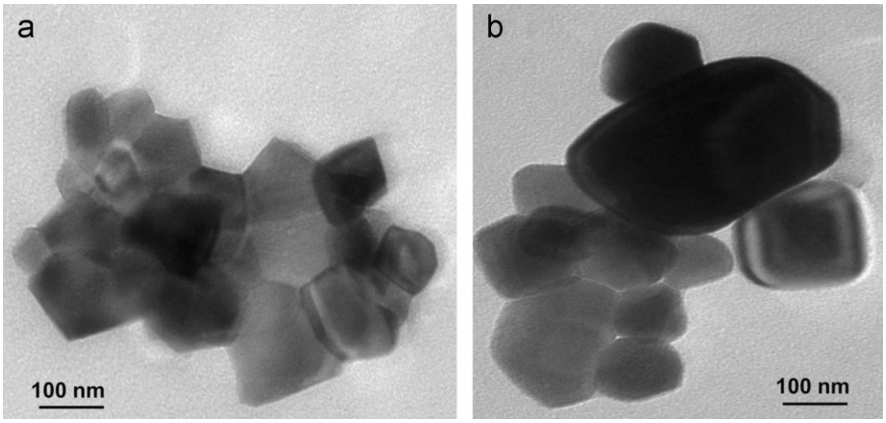

Fig. 4. TEM images of the (a) pure SDC particle and the (b) SDCC composite.

surrounding the SDC particles and not on the SDC lattice. The findings thus far indicate that a very chemically stable SDCC electrolyte composite powder for LT-SOFC applications can be formed.

FESEM and TEM images of the pure SDC and the SDCC electrolyte composite powders are shown in Figs. 3 and 4a and b respectively. These figures illustrate the rough dimensions of the particle grains and particle surface morphology. TEM was used to detail the microstructure of the prepared powders. The added (Li/Na) carbonate did not affect the shape or size of the hexagonally structured SDC particles (Figs. 3 and 4a). The SDC particles had similar particle shapes and sizes even after incorporation of the (Li/Na) carbonate (Figs. 3 and 4b). Moreover, the FESEM and TEM images showed the (Li/Na) carbonate-coated pure SDC resulting from the melting of the (Li/Na) carbonate at approximately 500 °C and the uniform wetting of the SDC grains, which prevented the agglomeration of the SDC particles [9,14]. Hence, the amorphous nature of the SDCC composite morphology may be caused by the (Li/Na) carbonate-coated SDC particles [7,14]. The densification and surface morphology of the electrolyte pellet can be studied by FESEM analysis.

Fig. 5 and 6a to d show FESEM images of all SDCC electrolyte composite pellets sintered at different temperatures at low, medium, and high magnifications. Fig. 5a shows the porous pellets sintered at 500 °C; poor melting of the (Li/Na) carbonate may be observed. The SDC and (Li/Na) carbonate do not form a continuous phase in the FESEM image. Fig. 5b shows relatively dense pellets with no pores and stable microstructure. Pores were hard to distinguish in the sample sintered at 550 °C since SDC and (Li/Na) carbonate phases were evenly distributed without large (Li/Na) carbonate crystals. Increases in the sintering temperature to 600 °C and 650 °C show that the (Li/Na) carbonate are not uniformly distributed on the surface of the SDC with the formation of larger (Li/Na) carbonate crystals; instead, they exhibit a cloudy appearance, as shown in Fig. 5c and d. The FESEM images reveal that the number of micropores decreases with increasing sintering temperature and the complete covering of the SDC surface by the (Li/Na) carbonate. When the sintering temperature was below the melting point of the (Li/Na) carbonate, many pores appeared on the surface of the SDCC electrolyte pellet sintered at 500 °C, also lowering the OCV (Fig. 7). When the sintering temperature was above the melting point of the (Li/Na) carbonate, the (Li/Na) carbonate formed a continuous phase and dispersed uniformly on the SDC particles. Wetting of the molten (Li/Na) carbonate filled the pores, resulting in a gas-tight electrolyte, as confirmed by the high OCV for pellet sintered at 550 °C. The pellets sintered at 600 °C and 650 °C showed a slight decrease in OCV due to microstructural changes, particularly in the (Li/Na) carbonate phase. At sintering temperatures above 650 °C, degradation occurs due to deterioration of the (Li/Na) carbonate in the electrolyte composite. To avoid loss of cell performance, the cell must be operated beyond the transition temperature of the composite electrolyte, which is between 500 °C and 650 °C [5]. The cell performance can be improved by optimizing single-cell fabrication. Reducing the electrolyte thickness and developing highly catalytic electrodes can help improve the performance of LT-SOFC operating at low temperatures [10].

The performances of the symmetrical SDCC electrolyte composite cells sintered at 500 °C, 550 °C, 600 °C, and 650 °C were investigated at 500–650 °C. *I–V* and *I–P* characteristics for the symmetrical SDCC electrolyte composite cells are shown in Fig. 7. The maximum OCV of the composite electrolyte cells with pellets sintered at 500 °C, 550 °C, 600 °C, and 650 °C reached 1.06 V, 1.14 V, 1.1 V, and 1.1 V, respectively at an operating temperature of 650 °C. Interestingly, the maximum OCV of the pellet sintered at 550 °C was higher than those of the pellets sintered at higher temperatures and was very close to the theoretical OCV at the testing temperature of 650 °C. The power density was very low for the pellet sintered at 500 °C, 600 °C, and 650 °C, and the OCV decreased linearly with increasing sintering temperature. The maximum power density of the SDCC electrolyte composite cell with the pellet sintered at 550 °C reached 63.5 mW/cm^2. Fig. 8

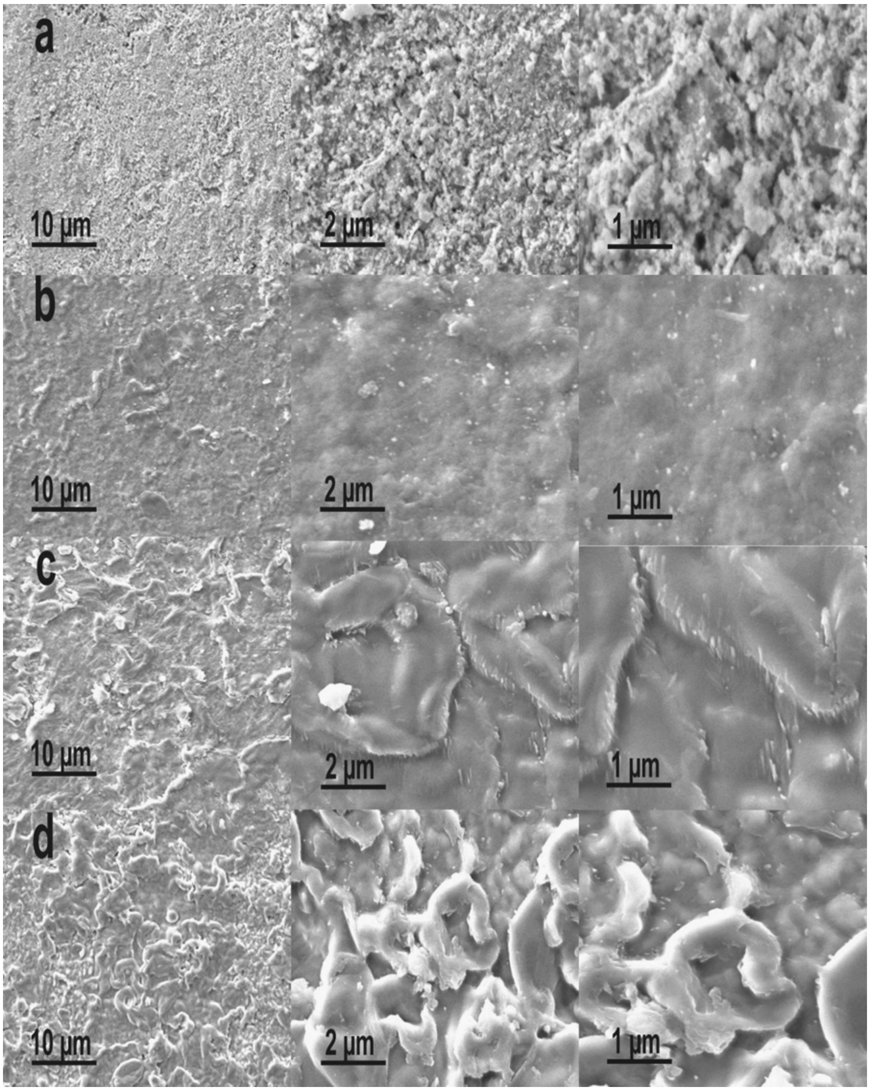

Fig. 5. Low to high magnification of FESEM images of the SDCC electrolyte composite pellets sintered at various temperature (a) 500 °C, (b) 550 °C, (c) 600 °C and (d) 650 °C.

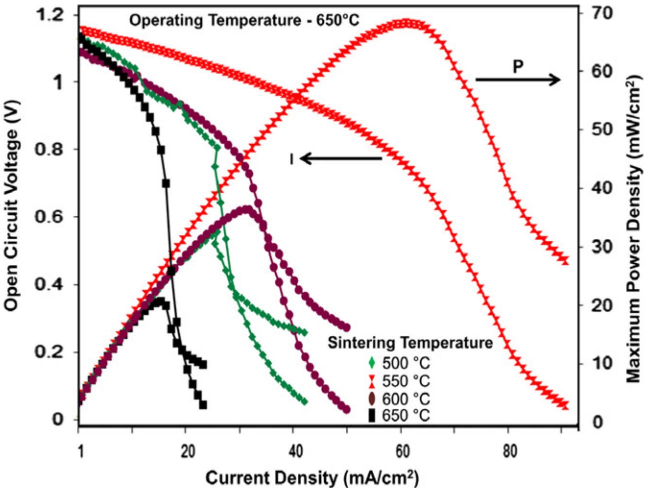

Fig. 6. *I–V* and *I–P* curves at operating temperature of 650 °C for SDCC electrolyte composite pellets sintered at various temperatures.

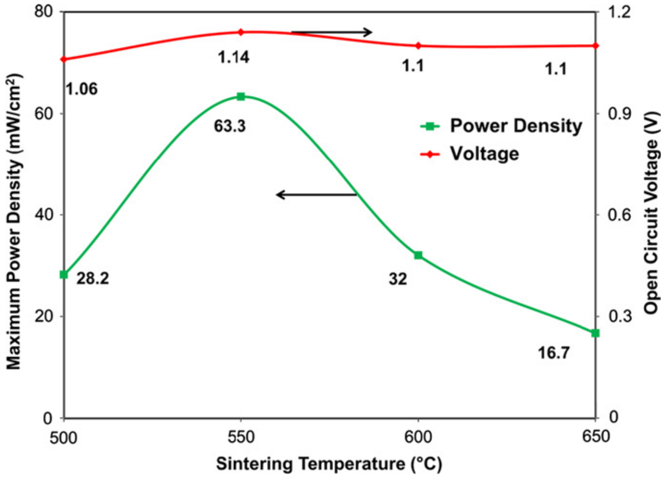

Fig. 7. Maximum power density and OCV of SDCC electrolyte composite pellets at operating temperature of 650 °C as a function of sintering temperature.

shows the maximum power density and open circuit voltage (OCV) of the SDCC electrolyte composite cells at an operating temperature of 650 °C. The OCV of all of the sintered cells in this study are very close to the theoretical values and are higher than earlier reported data [7,9,10,11]. All of the composite electrolyte pellets in this study were manufactured in similar ways but exhibited different electrical behaviors due to the variations in sintering temperature, as evidenced by differences in their surface morphology.

Fig. 8 shows the OCV and power density of the symmetrical SDCC electrolyte composite cell sintered at 550 °C at different operating temperatures. The recorded OCV value decreased from 1.14 V at 650 °C to 0.53 V at 500 °C. The OCV is well known to decrease at higher temperatures due to increases in the electronic conductivity of the SDC electrolyte under anodic fuel environments [3,4,11]. However, the recorded OCV data from this study show a different result. The OCV increased with increasing operating temperature. The maximum OCV (1.14 V) was obtained at a higher operating temperature of 650 °C. Variations in the OCV with increasing operating temperature may be explained by the uniform melting of (Li/Na) carbonate, which results in a larger amount of gas-tight electrolytes and decreases in the electronic conductivity at higher temperature. The increasing deviation of the experimental OCV from the theoretical OCV of the SDCC electrolyte composite cell at the lower operating temperature of 500 °C may be due to poor melting of the (Li/Na) carbonate, resulting in the formation of pores on the surface of the pellets. The (Li/Na) carbonate in the electrolyte composite become porous at operating temperatures below the melting point; hence, they cannot melt and densify to prevent gas crossover. In general, a high OCV indicates lower electrolyte porosity [15]. This finding indicates that the introduction of (Li/Na) carbonate to the SDC phase prevents the anodic reducing atmosphere from directly coming into contact with the SDC electrolyte.

The densification of the composite electrolyte can be further confirmed by FESEM observation. Fig. 9 shows the cross-sectional FESEM images of the composite electrolyte pellets sintered at 500 °C, 550 °C, 600 °C, and 650 °C. Fig. 9(a) to (d) show the porosity of the composite electrolyte pellet and the good phase interface between the (Li/Na) carbonate and SDC phase. Fig. 9(a) shows the (Li/Na) carbonate does not melt and cover the surface of the SDC particles. Thus, the SDC particles are agglomerated, forming micropores, which are visible in the cross

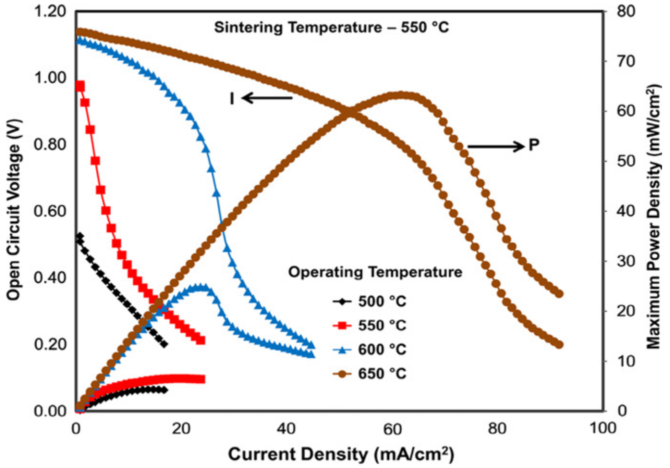

Fig. 8. *I–V* and *I–P* curves for SDCC electrolyte composite pellets sintered at 550 °C at various operating temperatures.

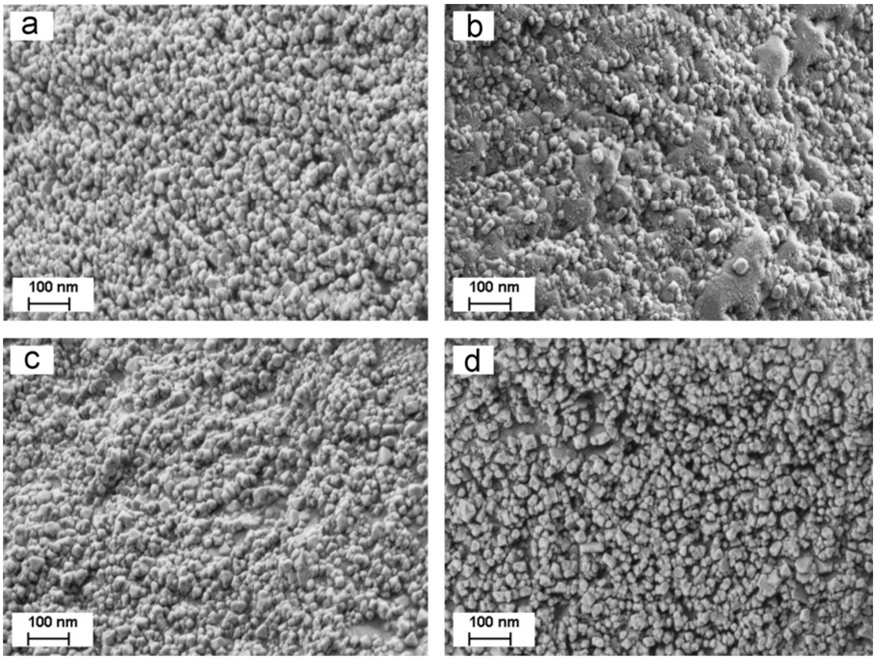

Fig. 9. Cross-sessional images of the SDCC electrolyte composite pellets sintered at various temperatures (a) 500 °C, (b) 550 °C, (c) 600 °C and (d) 650 °C.

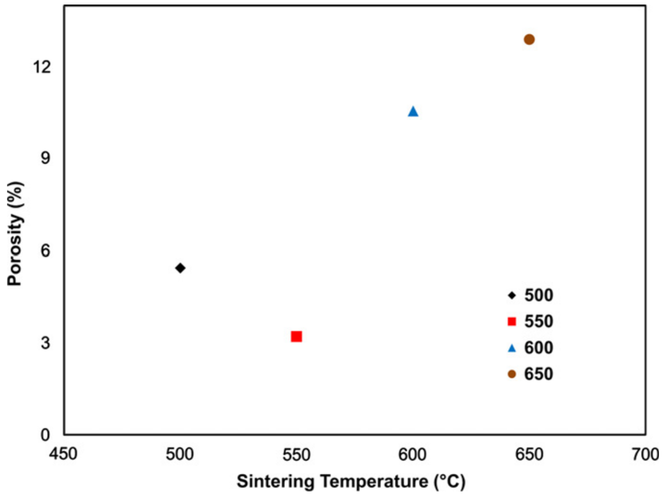

Fig. 10. Effect of sintering temperature on porosity of SDCC electrolyte composite pellets sintered at various temperatures (a) 500 °C, (b) 550 °C, (c) 600 °C and (d) 650 °C.

section of the composite electrolyte. Furthermore, at a low sintering temperature of 500 °C, the melting point of the (Li/Na) carbonate is attained, decreasing the wettability of the surroundings of the SDC solid particles to the (Li/Na) carbonate. Fig. 9(b) shows the continuous (Li/Na) carbonate and SDC phase, as well as the loosely agglomerated particles. When the temperature is increased beyond 500 °C, the solid (Li/Na) carbonate melts and forms a continuous network surrounding the SDC particles. This network forms a gas-tight layer that prevents gases from crossing over. When the sintering temperature is increased to 600 °C and 650 °C, the (Li/Na) carbonate melts and becomes deposited as a background layer without forming a continuous phase interface with SDC particles, as shown in Fig. 9(c) and (d). Moreover, the porosity and average SDC grain size increases as the SDC particles become clearer, as confirmed by the porosity measurement of the SDCC composite electrolyte pellet sintered at various temperatures. Increasing the sintering temperature increases the porosity of the sintered pellets except for the pellet sintered at 550 °C (Fig. 10). A fully dense SDCC composite electrolyte with 3% porosity was observed for the pellet sintered at 550 °C.

The porosity of the sintered composite electrolyte pellets is improved because of (1) the weak agglomeration of SDC particles due to wetting by the (Li/Na) carbonate (Figs. 9(a) and 2) the formation of a continuous phase interface between the SDC and the (Li/Na) carbonate melt phase (Fig. 9b). At 600 °C and 650 °C sintering temperatures, the porosity reaches 11% and 13%, respectively. As shown in Fig. 9(c) and (d), the porosity increases because of the absence of (Li/Na) carbonate around the solid SDC particles. However, in the composite electrolyte pellets sintered at 500 °C and 550 °C, the (Li/Na) carbonate and SDC phase are continuous, indicating that the composite electrolyte is fully dense. Thus, increasing the sintering temperature further does not help increase the density of the ceramic material. The preferred sintering temperature for the SDCC electrolyte composite pellet is therefore 550 °C.

4. Conclusions

The current study presented the development of an SDCC electrolyte composite for use in LT-SOFC applications. XRD analyses showed the stability of the composite powder. No new compound or chemical reaction was found between the SDC phase and the (Li/Na) carbonate phase. The influence of the sintering temperature on the microstructure and *I–V* characteristics of the SDCC electrolyte was investigated in the temperature range from 500 °C to 650 °C. The SDCC electrolyte composite pellet has different surface morphologies at different sintering temperatures. Densification of the pellets sintered at 550 °C was responsible for the high OCV and maximum power density obtained. Lowering the sintering temperature below the melting point of the (Li/Na) carbonate resulted in the poor performance of the SDCC electrolyte composite due to the development of pores on the surface of the electrolyte pellet. The results of this study prove that the sintering temperature greatly influences both the microstructure and the electrical performance of the SDCC composite pellet. The best sintering temperature for the SDCC electrolyte composite pellets was 550 °C. Sintered ceramic pellets at 550 °C exhibited a maximum power density of 63.5 $\mathrm{mW/cm^2}$ and an OCV of 1.14 V at an operating temperature 650 °C. Further studies will be conducted to examine the surface roughness and ionic conductivity of the composite electrolytes at operating temperatures below 650 °C in air.

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