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Effect of Milling Process and Calcination Temperature on the Properties of BSCF-SDC Composite Cathode

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Abstract. The ionic conductivity, super conductivity, ferroelectricity, and magnetic resistance of barium strontium cobalt ferrite (BSCF) make it a good solid cathode material. This study aims to investigate the behaviour of nanocomposite cathode BSCF-samarium-doped ceria (SDC) on influence of milling process and calcination temperature. There is two type of milling process involve mixing the BSCF-SDC composite powders, namely, wet milling and dry milling process. The composite cathode powders were mixed through wet milling by high-energy ball milling at 550 rpm for 2 hours. For dry milling, the powders were milled at 150 rpm for 30 minutes. The powders then underwent calcination at 900°C, 950°C, 1050°C and 1150°C for 2 hours. The composite cathodes were examined on the basis of phase and microstructure through field emission scanning electron microscopy (FESEM) and x-ray diffraction (XRD), respectively. In conclusion, selection of suitable milling process and calcination temperature is important in eliminating secondary phases in BSCF-SDC composite cathodes and in enhancing their properties.

Introduction

Electrochemical mechanism that immediately transmute a chemical energy into electrical energy [1] in silent and environmental action [2] are called fuel cells. Fuel cell consist of three main parts which is electrolyte, cathode and anode. Its functioning by fed fuel continuously in the anode part and fed a suitable oxidant that usually air into the cathode part. Another part of fuel cell is electrolyte, purpose of this part is to avoid the fuel supply directly contact with an oxidant during electrical connecting to the anode and cathode. Besides that, in the electrochemical reaction electrolyte can let on the reductant ions or oxidant to passage the other side to take part in the reaction [1]. There are many type of fuel cell, one of it is solid oxide fuel cells (SOFC). The use of SOFC is as a promising power generation technology, because it can exchange the chemical energy in fuels form directly into electric power with higher in efficiency and low emission [3]. SOFCs are widely used because of their environmentally conversion technique with a minor levels of noise pollution and potentiality being operate with many different type of fuels, and it is high efficiency [4].

The electrolyte for SOFC materials should exhibit some stabilization state when at higher temperature with under oxidizing and reducing atmosphere also high in chemical compatibility, oxygen ionic conductivity and high mechanical strength to support anode or cathode and has low electronic mobility [5]. Ceria doped with alkaline or rare earth oxides is a promising candidate material for SOFC operating in low temperature based on its considerably high ionic conductivity at low temperatures [6]. Barium strontium cobalt ferrite (BSCF) materials on perovskite structure are attractive and effective cathode materials for SOFCs because of their high conductivity with incomparable oxygen transport, and catalytic activity [7].

Oxygen vacancies that form by the substitution of rare earth cations into ceria lattice has make the ionic conductivity of ceria increasing remarkably [6]. The achievement of new cathodes by

developed a mixed conducting phase (BSCF) with an ionic conducting phase, samarium-doped ceria, considerably improves the electrochemical properties. The SDC content greatly affects the electrochemistry properties of combination electrode. In addition, after scattered SDC particles on the BSCF matrix, the connectivity of both sides are not shows a good result, and the function on electrochemistry cannot be well performed [8].

From all numerous capable compositions, $\text{Ba}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.8}\text{Fe}_{0.2}\text{O}_{3-\delta}$ (BSCF) shows a good aspirant for the application due to its high-level oxygen permeation and oxygen-ion diffusion [9]. However, BSCF will at the higher stabilization state when operated at high temperatures and at high state of oxygen partial pressures and at temperature below 790°C, it will be transformed to a vacancy-ordered brownmillerite with low oxygen conductivity. The BSCF is stabilized and the brownmillerite phase form was prevented by substituting the A-site of the perovskite with the large-size cation Ba^{2+} . By this additional action the increasing in the permeation rate can be observed [10].

The structural instability of cubic BSCF phase at low temperatures is a concern. Therefore, research and investigations on the phase stability of Barium Strontium Cobalt Ferro and the corresponding properties are expected.

Materials and Method

Commercial BSCF and SDC (Kceracell, Korea) powders were mixed through two milling processes, namely, wet milling and dry milling. The composite cathode powders consisted of 50 wt% BSCF and 50 wt% SDC powders. Mixing was performed by ball milling; for wet milling, ethanol was used as a mixing medium. This method was performed with a speed of 550 rpm within 2 h at room temperature. The mixed powder was dried in the dry oven at 80 °C for 12 hours after milling. Dry milling was performed at 150 rpm within 30 min. The dried mixed powder was placed in an agate mortar and crushed. Then, the powder underwent calcination with an electric furnace at 900°C, 950°C, 1050°C and 1150°C for 2 hours. The calcined composite cathode powder was ground again using the agate mortar to produce fine powders. Table 1 summarizes the sample identification.

Characterization Method

The composite cathode powder samples were characterized on the basis of their phase and crystalline structures by using X-ray Diffraction (XRD) (Bruker D8 Advance, Germany). XRD is widely used for the identification of unknown crystalline materials, minerals, and inorganic compounds. The test was conducted at room temperature and metered by Cu K α radiation, that is, $\lambda = 0.15418 \text{ \AA}$. This diffraction pattern was scanned in the range of 2θ , starting from 20° to 90°, with a step scanning of 0.02°. The analysis data were then defined using Eva Diffac Plus software.

Morphology grain and microstructure analysis was examined through Field Emission Scanning Electron Microscopy (FESEM) (JSM 6700F-Jeol, Japan). In this study, samples were obtained from ceramic materials and coated with platinum to ensure the conductive phase in conducting electron radiation. The desired image was scanned using FESEM. Then, by using ImageJ software (Version 4.18) measured the average grain size of the samples.

Table 1. Identification of BSCF–SDC sample with variant of milling speed and temperature of calcination process.

Sample	Milling Speed [rpm]	Calcination Temperature [°C]
WET-B	550	-
DRY-B	150	-
WET-B 900	550	900
DRY-B 900	150	900
WET-B 950	550	950
WET-B 950	150	950
DRY-B 1050	550	1050
WET-B 1050	150	1050
DRY-B 1150	550	1150
DRY-B 1150	150	1150

Experimental Result

Figure 1 shows the XRD diffractogram of BSCF–SDC composite samples that undergo wet milling and calcination with four different temperatures. The temperatures used for this study are 900°C, 950°C, 1050°C and 1150°C. The XRD results for BSCF–SDC composite powder samples after being calcined at four temperatures show that the BSCF spectrum generates at JCPDS No: 55-0563, which is the same spectrum pattern as that of the pure commercial BSCF powder. Moreover, the pure commercial SDC powder shows excellent purity at JCPDS No: 75-0157. The milled samples also generate secondary peaks that can be observed on the basis of the XRD result. Iron carbonate, Fe (CO₃) (JCPDS No: 83-1764), and barium carbonate, BaCO₃ (JCPDS No: 71-2394), are formed at the early stage of BSCF–SDC composite powder even before calcination. This result is due to the heat generated during milling process that make an impact forces by conversion of kinetic energy which breaks the particles.

The milled powder then being calcined at 900° and 950° shows that the BSCF–SDC sample indicates secondary peaks of barium carbonate, BaCO₃ (JCPDS No: 71-2394), and strontium iron oxide, Sr₂FeO₄ (JCPDS No: 42-0480), in both milled powders. Furthermore, the composite powder calcined at 1050 °C and 1100 °C forms secondary peaks that are rarely found, namely, strontium iron, Sr₄Fe₆O₁₂ (JCPDS No: 82-3765), and strontium iron cobalt oxide, Sr₄Fe₂ (Fe_{2.4}Co_{1.6}) O_{12.96} (JCPDS No: 88-0130). The secondary phases may form because of the heating state generated during the high-energy ball milling [11]. Secondary-phase peaks are clearly formed, and many peaks are generated when the composite powder is calcined at high temperature. These finding phases are called surface carbonates that form by the reaction of alkaline earth oxide (Barium [Ba] and Strontium [Sr]) and CO₂ during the calcination [11]. The formation of secondary phases should be eliminated because this phenomenon will affect the electrochemical performance of the sample.

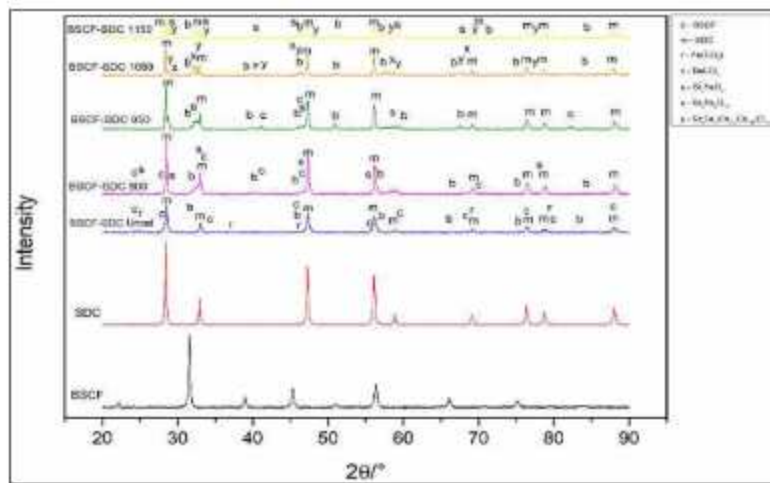


Fig. 1. XRD diffractogram of BSCF-SDC composite powders that undergo wet milling.

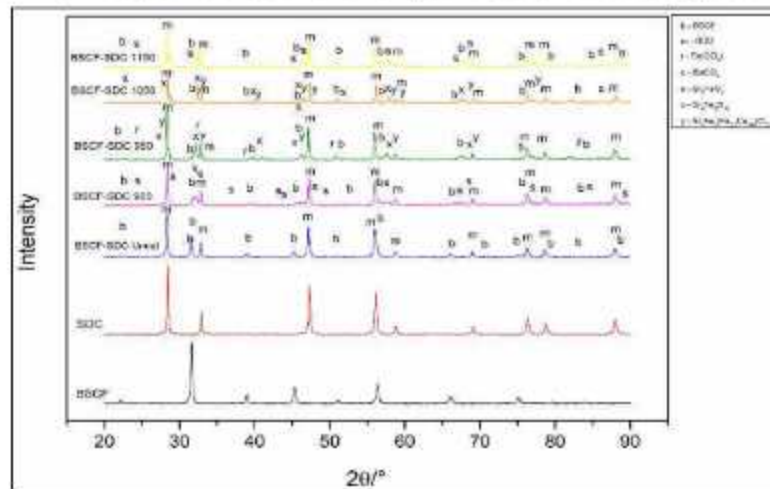


Fig. 2. XRD diffractogram of BSCF-SDC composite powders that undergo dry milling.

Fig. 2 displays the XRD results of BSCF-SDC composite samples that undergo dry milling and are calcined at 900°C, 950°C, 1050°C and 1150°C. Obviously, the BSCF (JCPDS No: 55-0563) and SDC (JCPDS No: 75-0157) spectrum generates at the same spectrum pattern as the pure commercial BSCF and SDC powder. The spectrum pattern is the same as that generated at the wet milling process. The milled samples generate a secondary peak at the early stage before calcination when undergoing wet milling, but no secondary peak is observed at the dry milling sample of uncalcined BSCF-SDC.

At 900 °C, strontium iron oxide, Sr_2FeO_4 (JCPDS No: 42-0480), is formed and generates more secondary peaks for samples calcined at 950 °C and 1050 °C than those at other temperatures. From this formation, the peaks correspond to iron carbonate, $\text{Fe}(\text{CO}_3)$ (JCPDS No: 83-1764), strontium iron oxide, Sr_2FeO_4 (JCPDS No: 42-0480), and strontium iron cobalt oxide, $\text{Sr}_4\text{Fe}_2(\text{Fe}_{2.4}\text{Co}_{1.6})\text{O}_{12.96}$ (JCPDS No: 88-0130). The peaks observed for the composite powder calcined at 1150 °C correspond to strontium iron oxide, Sr_2FeO_4 (JCPDS No: 42-0480). These finding phases are same with the previous result of wet milling sample which is has surface carbonates phases that form during the calcination process [11].

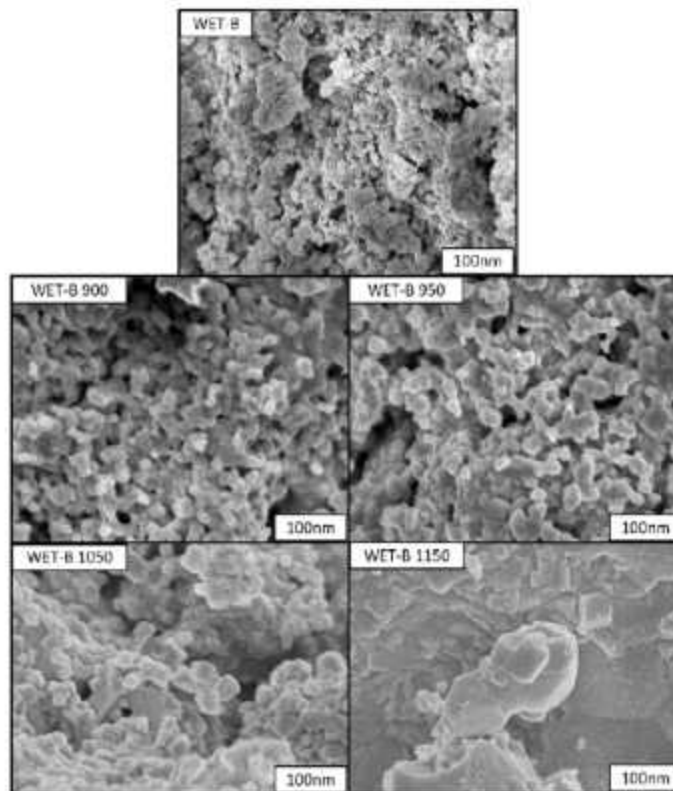


Fig. 3. FESEM micrographs of BSCF-SDC powders under wet milling at different calcination temperatures.

Fig. 3 and 4 show the surface morphology of the BSCF-SDC samples mixed by **two milling processes, namely, wet milling and dry milling.** The samples were calcined at 900°C, 950°C, 1050°C and 1150°C. The result show that the particle increases and agglomerates as the calcination temperature increases. The size of particle is influenced by the increasing in temperature [12]. The particle size growth and its larger than the particle size at the early stage of mixing. This phenomenon occurs because has an increasing in size of particle during calcination and has reduction in surface area. The calcination strengthens the particle bond, but the small particle size simplifies the agglomeration. Figures 3 and 4 show the sample before and after calcination. Obviously, the particles show different sizes and conditions.

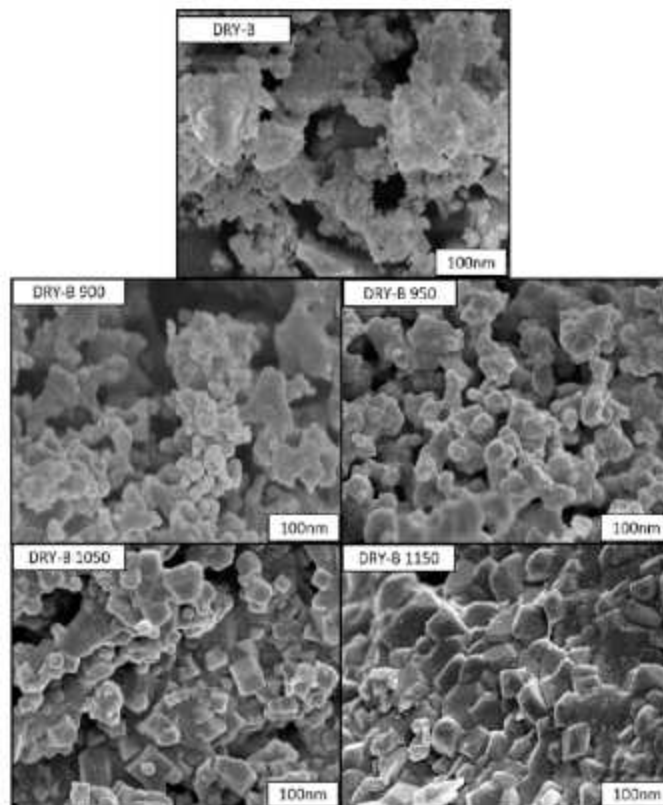


Fig. 4. FESEM micrographs of BSCF-SDC powders under dry milling at different calcination temperatures.

Conclusion

In this research, the effects of milling process and calcination temperature were analyzed based on the properties and phase transformation of BSCF-SDC cathode powders. Different XRD results are recorded for samples from wet milling and dry milling. Secondary peaks are observed when powders undergo wet milling at a high speed of 550 rpm. This result is influenced by the heat generation during the mixing process. By contrast, no secondary peak is observed after mixing for samples that undergo dry milling at a speed of 150 rpm. Therefore, milling at low speed is suitable to avoid secondary phases at the early stage of mixing. The morphology also shows the increment in particle size and the reduction in the particle surface area by increasing in calcination temperature. Notably, milling process and calcination temperature are important parameters, the selection of it needs to be monitored and choose appropriately because its influences the phase stability and particle size of BSCF-SDC cathode powders.

Acknowledgments

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