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DEVELOPMENT OF OXIDE COMPOSITE MATERIALS FOR CATHODE ELEMENT OF IT-SOFCs

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Investigating the properties of composite oxide for intermediate temperature solid oxide fuel cells (IT-SOFCs) has been done as a new cathode material. Using a solid-state reaction method, the metallic oxide material has been employed to create the composite model system. During the sintering process, a model system of $Sm_{0.5}Sr_{0.35}Ba_{0.15}FeO_{3-\delta}$ (SSBF15) was constructed. Thermal gravimetric analysis (TG) was played to utilize the oxygen content and weight loss of the model. In the meantime, the structure of the composite was characterized using X-ray diffraction (XRD), and the conductivity properties were tested by thermal conductivity. The structural design was made possible by the findings, which revealed that the composite model structure exhibited the crystalline structure with perovskite phase. Weight losses during the construction of the structure were reflected in the decomposition and evaporation of the composite's constituent parts. After the calcination process up to 950 °C, the model system's formation oxygen content was obtained of 2.94 in 800 °C. The electrical conductivity maximum obtained in 12.2 $S\cdot cm^{-1}$ at 430 °C. At low temperatures, the conductive behavior was affected by the metallic element, while at higher temperatures, it was influenced by the ionic structure. As a result, mixed ionic and electric conductors (MIEC) were extensively utilized in the process of generating the conductive properties. The SSBF15 composite has a good chance of being used as an alternative cathode material with a perovskite single phase for future IT-SOFCs applications based on the structure and conductivity results. Additional testing and observation are required to determine the resistance's value when incorporated into the electrolyte and its heat expansion properties

Keywords: solid oxide fuel cells, cobalt-free cathode composite, perovskite structure, oxygen content, electronic conductivity

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1. Introduction

Solid oxide fuel cells (SOFCs) have its own charm because it has high efficiency in energy conversion, renewable fuel and produces low emissions so it is environmentally friendly. Due to their high performance, SOFCs are the electrochemical device that has been continuously developed [1]. The device is being used extensively for commercial implementation, making it simple to use in a wide range of applications. This technology has been developed for both the satisfaction of economics evaluation for residential power systems [2], and the energy efficiency of buildings [3]. In addition, it has been advocated for fuel cell electric vehicles as a power plant, improving SOFCs reformer efficiency by as much as 20 % by reducing energy loss [4]. However, up to now, the SOFCs operates at temperatures ranging from 800 to 1000 °C [5]. Its temperature restricts the materials that can be used in cells and auxiliary components, makes it more difficult to seal, and causes cell performance and interconnecting elements between components to deteriorate. To advance technology toward commercialization, some significant technical issues must be resolved, particularly those pertaining to durability and dependability.

In intermediate temperature solid oxide fuel cells (IT-SOFCs) made of perovskite type oxides, represented by $ABO_{3-\delta}$, mixed ionic and electric conductors (MIEC) are widely promoted [6]. They can be used as electro-catalysts for solid oxide fuel cells (SOFCs), oxygen-permeable membranes, and oxygen absorbers, among other things. In some experiments [7, 8], it was found that the MIEC cathode had high electrochemical activities due to its high oxygen transport and surface exchange properties, high electro-catalytic activities, and good performance. However, the high thermal expansion of the electrolyte makes these cobalt-based MIECs unsuitable for long-term applications, preventing the development of cathode IT-SOFCs [9]. Cobalt's (Co) high price in comparison to other rare metals is another consideration for its successful application [10]. For IT-SOFCs, the investigation is necessary to discover a novel composite structure made of free-cobalt cathode material to overcome the numerous limitations listed above. Therefore, research on the study characterization related to structure, oxygen content and electronic conductivity of composite oxide for cathode elements of IT-SOFCs is relevant.

2. Literature review and problem statement

High-performance cathode materials for intermediate temperature SOFCs (IT-SOFCs) have received a lot of attention. Recently, a few cobalt based-blended directing perovskite oxides, like $La_{0.4}Sr_{0.6}Co_{0.9}Sb_{0.1}O_{3-\delta}$ (LSCSb)-$xCe_{0.8}Sm_{0.2}O_{1.9}$ composite materials have been studied systematically as cathodes for intermediate-temperature solid oxide fuel cells (IT-SOFCs) [11]. The performance of composite cathodes is good in the electrical conductivities, power density and the lowest polarization. However, the presence of cobalt elements in cathode materials raises the thermal expansion coefficient (TEC) of the cathode component and causes a mismatch in TEC value with electrolytes. In other cathode system, co-doped double perovskite-type cobaltite $Pr_{0.9}Y_{0.1}BaCo_{1.8}Ni_{0.2}O_{6-\delta}$ (PYBCN) with 30 wt% addition of samarium doped ceria $Ce_{0.8}Sm_{0.2}O_{1.9}$ (SDC) exhibited a good conductivity and high electro-chemical activity [12]. Moreover, rare-earth-abundant layered perovskite cathodes of $LnBa_{0.5}Sr_{0.5}Co_{1.5}Fe_{0.5}O_{5+\delta}$ (Ln=La and Nd) are exploited for solid oxide fuel cells (SOFCs) at reduced temperatures [13]. The higher output performance of NBSCF cathode is ascribed to its faster surface oxygen exchange. Both LBSCF and NBSCF exhibit high electrical conductivity (>1000 S/cm) at all operation temperatures. Unfortunately, despite the higher cost of cobalt itself, the presence of this elements increases the TEC value influencing a mismatch with electrolytes. While a new composite cathode $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$–$Sm_{0.2}Ce_{0.8}O_{1.9}$ carbonate (BSCF-SDCC) was studied [14], have been proposed and read up as cathode materials for IT-SOFCs due to the excellent properties in electrical conductivities, power density and the lowest polarization. These materials have high intermediate temperature electronic conductivity and excellent catalytic reactivity for oxygen reduction. Due to its excellent capability in a reduced atmosphere, this cobalt-containing cathode is known for its excellent electrochemical performance at lower operating temperatures. However, their performance instability and thermal mismatch with electrolytes, cobalt-based cathodes have a high cost and significant performance degradation over time. Cobalt cations' convenience of reduction and evaporation are the source of these issues [15].

As a potential solution to the challenges associated with IT-SOFC implementation, the free Co-based cathode has been the subject of extensive research to date [16]. As a result, the new cobalt-free cathode model was continuously developed [17]. Due to the strong electronic structure of Fe ions, MIEC model with Fe on the B side on the composite structure of the cathode system is anticipated to be less expensive and more durable under SOFC operation conditions (500 to 800 °C and air atmosphere) [18, 19]. In the A side-based perovskite oxides, the composite model has been constantly evolving to achieve excellent cathode performance up to this point. A lot of attention has been paid to the use of the metal transition to strengthen the A-side structure of perovskite [20, 21]. One of the well-known components that is utilized in the A-side of a composite structure as a BLF system for the cathode element is barium (Ba) [22, 23]. The phenomenal design is connected with the electro-reactant exercises for oxygen reduction, as well as the great functional security. Moreover, the succession of samarium (Sm), Ba and strontium (Sr) components in the A-site shows the extraordinary exhibition of cathode components [24, 25]. As a composite with Fe ions in the B-site, its development is not yet complete. This composite system has been demonstrated in previous studies, but its conductive properties remain low [26]. However, further study of cobalt-free cathode structures with Ba, Sm, and Sr in the A-site and Fe ions in the B-side of a composite structure was rarely studied.

All this allows to assert that it is expedient to conduct a study on development of oxide composite materials for cathode elements of IT-SOFC. To gain a thorough comprehend of the properties of the cathode composite material, characterization using thermal gravimetry, x-ray diffractometer, and electrical conductivity will be carried out.

3. The aim and objectives of the study

The aim of the study is development of oxide composite materials for cathode elements of IT-SOFC. The composition of the composite elements oxide's, the calcination process, could influence the construction of the structure, the oxygen content and conductive properties of the model compositeб which could all be improved as a result of this.

To achieve this aim, the following objectives are accomplished:

– to determine the optimal calcination temperature as well as the formation of lattice oxygen and oxygen content;

– to evaluate the composite model's structure;

– to examine the free-cobalt cathode's conductivity properties.

4. Materials and methods

As a potential candidate for use as the cathode element in IT-SOFCs, it is investigated the structure and properties of a cobalt-free cathode based on Sm, Sr, Ba, and Fe composite materials. Using a composite of Sm, Sr, and Ba elements on the A side, the solid-state reaction method was used to alter the perovskite structure, resulting in a composite cathode for the SSBF system. In addition, the characterizations will be carried out to acquire additional information regarding the structure and conductivity properties of the cathode model.

The metallic oxide compound Sm_2O_3, $SrCO_3$, BaO, and Fe_2O_3 serve as the composite's raw materials (SSBF model). The traditional method for producing SSBF cathodes is solid-state reaction. The method was used to prepare and synthesize the cathode material, as described in other publications by our group [9, 27, 29]. A stoichiometric volume of metallic oxide compound was mixed with zirconia balls for 12 hours in a solution containing 96 percent ethanol. The powder mixture was then dried for 24 hours in an oven at 75 °C. The SSBF precursor powders were normally cooled in the furnace after being calcined for four hours at 950 °C in an air atmosphere with a heating rate of 3 °C min^{-1}. After being ground with an agate mortar, the calcination powder was sieved using 200 mesh. The oxygen content of the powders was determined through Thermogravimetric (TG) testing with a heating rate of 10 °C min^{-1}, the temperature was raised to 1000 °C before being lowered to room temperature. After that, other powders were pressed into a 5×7×10 mm square bulk. The bulk of the cathode was sintered for four hours at 1200 °C with a heating rate of 4 °C min^{-1}, and the furnace naturally cooled. The crystal structure of the cathode powder was also determined by XRD, and electrical conductivity of bulk samples was measured between 400 and 800 °C.

5. Research results of development of oxide composite materials for cathode elements of IT-SOFC.

5. 1. Thermal gravimetric analysis (TGA)

The differential thermal analysis (DTA) and thermal gravimetric analysis (TGA) of SSBF15 composite oxide powders are depicted in Fig. 1. The TG value of the SSBF15 composite is represented by the blue curve, while the thermal behavior of the composite during the calcination process is represented by the black curve. The thermal gravimetric method was used to track the material's weight loss from either evaporation or decomposition. The temperature used for the subsequent calcination and sintering step can be determined using this method. According to the curve, the points A, B, and C indicate that the weight of the sample decreased as a result of the evaporation of water and some materials' impurities. While the compound decomposition reaction led to a weight loss at points D.

Fig. 1 depicts the DTA and TG curves of the oxide powder that has been heated from room temperature to 1100 °C. For point B, C and D of DTA, there are four steep valleys on the bend: 291, 426 and 914 degrees Celsius. The decreasing curve was also constructed in the same location as the TG curve.

Fig. 2 demonstrates the heating process's TG analysis of the model system's composite structure for investigating the weight loss.

During the heating process, the oxygen content of the composite material was monitored. Utilizing $O_{3-\delta}$ in the perovskite structure, the oxygen content of the model was determined. The slope of the curve was comparable to that of the TG study depicted in Fig. 2. The point of the P-Q region relatively contained the stable curve. From Q to R, the decreasing curve of 0.06 $O_{3-\delta}$ was observed.

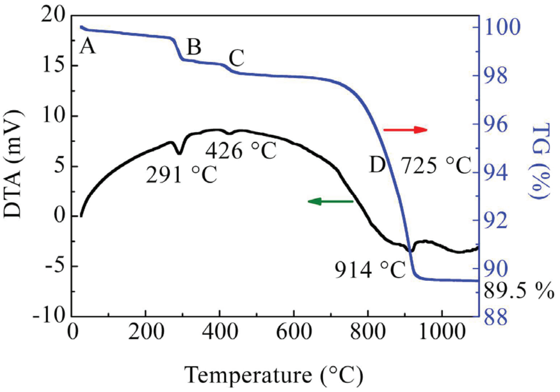

Fig. 1. Thermal gravimetric analysis (TG) and differential thermal analysis (DTA) of precursor SSBF15 powders

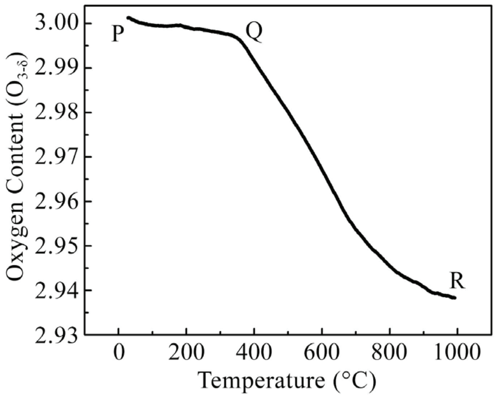

Fig. 2. The oxygen content of cathode powders made of SSBF15 composite

5. 2. Structure characterization

The XRD pattern of the composite SSBF15 structure, which was calcined for four hours at 950 °C, was used as a reference for the perovskite structure of lanthanum ferrite ($LaFeO_3$) with 75-0541 shown in Fig. 3.

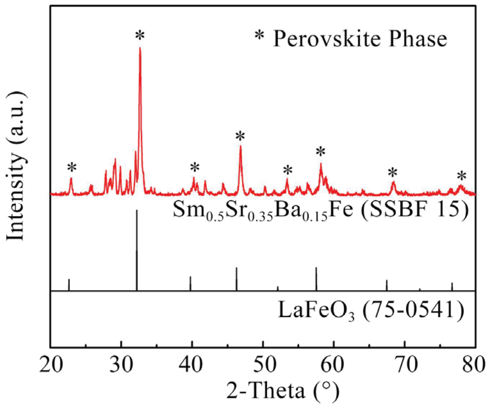

Fig. 3. SSBF15 cathode XRD diffraction pattern

The composite model system that was used to construct the single phase of the SSBF structure. The order shown in Fig. 3 provided detailed evidence of the peak's sharpness and narrowness (B). There are nine major peaks in the pattern, each with a scale of 60 degrees in 2-theta. The peak positions were 23.0°, 32.7°, 40.7°, 46.9°, 52.5°, 58.0°, 67.7°, 72.8°, and 77.4°. The perovskite structure references are comparable to those peaks. While, another phase relates to the second phase which is fathomed from impurity material.

5. 3. Electrical conductivity properties

Fig. 4 display the cathode composite products' electrical conductivity curve. The tests were conducted at temperatures ranging from 400 °C to 800 °C under atmospheric pressure and an oxygen concentration of 0.21 atm (21 % O_2).

At 400 °C, the conductivity value of 11.8 $S\cdot cm^{-1}$ displayed the parabolic curve. At 430 °C in point P, the conductivity reaches a peak of 12.2 $S\cdot cm^{-1}$ and simultaneously decreases to 9.3 S cm^{-1} as the temperature rises up to 800 °C.

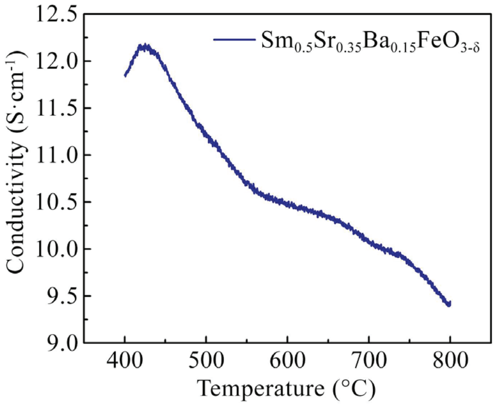

Fig. 4. The SOFC15 cathode composite materials of electrical conductivity

6. Discussion of the results of study characterization of composite oxide for cathode element of IT-SOFC

Thermal gravimetric analysis was used to thoroughly examine the weight loss of the composite material compound. The curve's downward slope during the first TG step, as depicted in Fig. 1 took place from room temperature (A), 291 °C (B) and 426 °C (C), and the weight loss of SSBF15 powders was the cause. The powders' water content could be evaporated in the first step thanks to the device's heat energy. In addition, the quality of the evaporated water was demonstrated by the loss of weight in the temperature range that contributed to the samarium powder element [29]. The endothermic peak at the same temperature caused by substance dehydration was also demonstrated. Samarium content impurity was thought to be the primary cause of the weight loss in the second step at 426 °C (C). The rate of weight loss accelerates as strontium carbonate decomposes into SSBF15 oxide compounds. Some of the system's components reacted to form the new structure, which led to the beginning of the decreasing curve at the third step in point D, which was 725 °C to 914 °C. The solid-state reaction mechanism was forming the perovskite structure during this step. The calcination temperature could be used sufficiently up to 950 °C, according to TG analysis. As shown in Fig. 3 by XRD characterization, the endothermic reaction will also demonstrate the formation of the perovskite matrix. In addition, the system's heat energy influences the oxygen content decrease shown in Fig. 2. As oxygen levels drop to 2.94 of $O_{3-\delta}$, the reduction reaction for the perovskite structure in the SSBF15 cathode may be aided by heat energy. The IT-SOFC system's cathode system allowed for the transfer of mobility oxygen because, unless the temperature rises, the volume of oxygen in the air decreases.

The SSBF15 cathode's XRD pattern was shown in detail to provide a deeper comprehension of the resulting crystalline structure. Fig. 3 has nine main peaks in an XRD pattern that have varying intensities at various theta positions in relation to the crystalline structure (A). On the composite model framework, the perovskite phase was clearly visible in crystalline form. The phase's arrangement is also comparable to that of the nearly cubic $LaFeO_3$ structure shown in Fig. 3 [10, 20], which suggests that the SSBF15 cathode's perovskite-phase crystalline structure was constructed there. On the A-side of the model system, which was set up to accommodate cathode materials in the perovskite phase, the Sm, Ba, and Sr components were utilized. Using the solid-state reaction method, the stoichiometric of composite metal oxide for cathode materials could be developed as a crystalline structure with a perovskite phase based on the XRD pattern above.

Because of the structure's oxygen vacancy and electronic hole, the conductivity performance of SSBF15 composited was a combination of electronic and ionic conductivity. More precise amount of composition between Ba element to Sr systems could be played to generate the oxygen vacancy in the A-side of composite system. Further, the passage of thermally activated electrons through the Fe^{4+} O Fe^{3+} is linked to the rise in conductivity at lower temperatures. So, the conductivity value in low temperature obtained at 12.2 $S\cdot cm^{-1}$. This value is higher than the previous study, which is equal to 10.2 $S\cdot cm^{-1}$[26]. However, the thermal reduction of Fe^{4+} and Fe^{3+}, as well as the formation of additional oxygen vacancies, contribute to the decrease in conductivity above 430 °C. At high temperatures, the electrostatic attractive force between anions (O_2) and thermally reduced cations may deteriorate, leading to this effect. In addition, as the temperature rises, the angle of the curves increases as a result of the absence of oxygen in the cathode materials. For reducing the weight of the composite materials, the thermal conductivity behavior was in line with the trend in oxygen content in the TG analysis. To fully investigate the SSBF15 composite, however, additional characterization, including surface morphology and thermal expansion testing, is required. In addition, future research will be conducted to improve the SSBF's ability to integrate into the electrolyte and cathode layers of the single cell system.

7. Conclusions

1. The decomposition elements process associated with the solid-state reaction for the construction of the new structure in the free-cobalt system was primarily responsible for the weight loss on the model SSBF15 composite in TG analysis. The calcination temperature could be used sufficiently up to 950 °C, according to TG analysis.

2. The composite model system was the foundation for the perovskite structure, as the XRD results demonstrate. Using the solid-state reaction method, the stoichiometric structure of composite metal oxide for cathode materials could be obtained as a crystalline structure with perovskite phase.

3. The electronic conductivity properties of the composite systems were enhanced by more precise amount of composition between Ba element to Sr systems in A-side. The ionic structure acted as an elevated temperature while the metallic

element led to the generation of conductive behavior at low temperatures. Based on these studies, the SSBF15 composite material may be a viable alternative cathode for IT-SOFC.

Conflict of interest

The authors declare that they have no conflict of interest in relation to this research, whether financial, personal, authorship or otherwise, that could affect the research and its results presented in this paper.

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Data availability

Data cannot be made available for reasons disclosed in the data availability statement.

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