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Characterization of $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ Ceramic Powder Produced Via Solid State Reaction

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ABSTRACT

The study on perovskite type dense ceramic membrane derived from Lanthanum Strontium Cobaltite Ferrous Oxide or $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ (LSCF) ceramic powder has shown potential as economical, clean and efficient means to produce pure oxygen. In this research, LSCF powders were prepared by using solid state reaction method. This method was chosen since it was reported to be effective in producing large amount of powder and involve straightforward procedures. Preliminary characterization was done by using X-Ray Diffraction (XRD) technique to identify the substances in the produced powder. The result was then compared to XRD pattern of commercially procure LSCF powder which was manufactured by using spray drying method. Results shows that the XRD pattern from the LSCF powder produced via solid state method gave similar peaks as the commercially available LSCF powder pattern.

KEYWORDS: Dense Ceramic Membrane; Solid State Reaction; Characterization; X-Ray Diffraction.

INTRODUCTION

Separation of oxygen (O_2) from air for industrial use is a big business producing nearly 100 million tons of O_2 each year. This market will massively expand in the near future because virtually all large-scale clean energy technologies require O_2 as a feed [1]. Current industrial normal practices to produce pure O_2 from air are using cryogenic distillation and pressure swing adsorption (PSA). Cryogenic distillation process is known to be complex, expensive and energy intensive. The latter practice; PSA involves the separation of air at ambient temperature using molecular sieve adsorbents to trap nitrogen in order to produce oxygen with purities of 90% to 95%. The inability to achieve higher oxygen purity reduces the probability of PSA to be use in future clean energy endeavour [2]. It is obvious that both current practices have their flaws.

Based on [3], oxygen separation from air through perovskite dense ceramic membrane $La_{1-x}Sr_xCo_{1-y}Fe_yO_{3-\delta}$had showed potential to become an alternative method to produce pure O_2. Perovskite-type membrane with structure of ABO_3 contains transition metals at B site that show high electrical conductivity. The partial substitution of A site cations by other metal cations with lower valencies resulted in formation of oxygen vacancies and appearance of ionic conduction. This oxygen sorptive property plus the electronic conductive properties mentioned above suggest the possibility of using the defect perovskite-type oxides as an oxygen permeating membrane which can work without any need of electrodes and external electric circuit [3]. Previous studies also [4-8] indicate that perovskite dense membranes can provide an economic, clean and efficient option to produce pure O_2.

In order to achieve high oxygen flux through the perovskite dense membrane, several factors must be taken into consideration. First, the powder synthesized routes chosen; must possess the ability to produce a single phase perovskite structure, maintain correct stoichiometry and exhibit high surface area, in addition to the practicalities surrounding each technique [9]. Second, the selection of membrane type configuration should exhibit the least oxygen permeation resistance and have appreciable structural strength under different air flow rate and pressure [10]. Lastly, it is imperative that the perovskite chemical composition selected gave good chemical stability under reducing environment and at elevated temperature [8].

In [8] reported that perovskite dense membrane of $La_{0.6}Sr_{0.4}Co_{0.2}Fe_{0.8}O_3$ (LSCF) composition showed no phase changes when the LSCF powder was heated at 850°C in helium or argon for for 8 hours or nitrogen for 12 hours. This demonstrates that partial substitution of the A site (La^{3+}) with Sr^{2+} in LSCF can maintain its chemical stability in inert gas at 850°C.

In [11] suggested that the solid state reaction method is easily controlled and effective method to synthesize large amount of perovskite powder as compared to EDTA pyrolysis and modified citrate pyrolysis methods. A review by [12] also state that solid state reaction method is an attractive synthesis process to produce perovskite

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powder since it involved simple procedures and can give fast product. Solid state reaction method consists of physical stoichiometric compounding of the solid raw material in water using conventional grinding apparatus such as planetary ball miller. The resultant slurry will then be dried and sintered accordingly to produce the final black powder. This method involves simple and straightforward procedures with few variables that can affect the quality of the powder. Previous studies also showed that solid state reaction method is able to produce other type of oxygen-transporting membranes [13-15].

In this research, solid state reaction route was chosen to produce LSCF powder and investigation was done on several process parameters such as size of mill balls and milling time.

METHODOLOGY

Powder Preparation

All raw materials for solid state reaction method were purchased from Merck Sdn. Bhd. Stoichiometric amount of La_2O_3, Fe_2O_3, Co_2O_3, $SrCO_3$ powder were then mixed and milled with water using planetary ball miller at 300rpm for duration of 24 hours. The mixture was then dried in oven for 24 hours at 80°C. Finally, the sample was calcined in a furnace at 900°C for 5 hours. Depending on the characterization results, the processing parameters, size of mill balls used and milling time were manipulated accordingly.

The commercially available LSCF powder (C-LSCF) was procured from Inframat Advanced Materials, United States of America. This particular LSCF was used a standard for results comparison and was synthesized by using spray drying method.

Powder Characterization

Both LSCF powders, produced by solid state reaction and commercially procured underwent X-ray diffraction analysis to determine its composition and phase condition.

RESULTS AND DISCUSSION

All As expected, it can be seen from Figure 1 the XRD measurement of C-LSCF powder gave a single phase perovskite pattern as reported in previous studies [6, 16-17].

Figure 2 shows the XRD pattern for the first sample of LSCF powder produced via solid state reaction and this sample is denoted as (SS1) in Table 1. The milling parameters used in producing were as stated before. It can be seen Figure 2 that an impurities exist at four different points.

By increasing the milling time up to 48 hours, the resulting LSCF powder gave a similar XRD measurement pattern as C-LSCF with sharper peaks as shown in Figure 3.

Similar XRD measurement pattern can also be seen in Figure 4. This happened when additional balls were introduced in the miller while maintain the milling time of 24 hours. This clearly shows that by adding extra balls will give better powder composition.

This research concurs with the study by [18] which proves that by increasing milling time, a single perovskite phase will be produced.

This research also shows that by increasing the milling intensity, a single perovskite phase can be achieved with less milling time. The parameters set up for this research is illustrated in Table 1.

Table 1: Solid state reaction method parameter

Parameter	SS1	SS2	SS3
Num. of 10mm balls	10	10	20
Num. of 20mm balls	0	0	10
Milling time (hours)	24	48	24

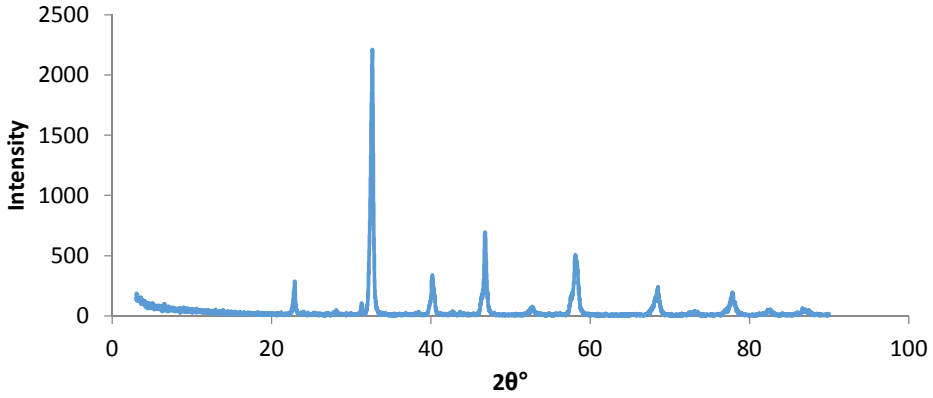

Figure 1: XRD pattern of C-LSCF

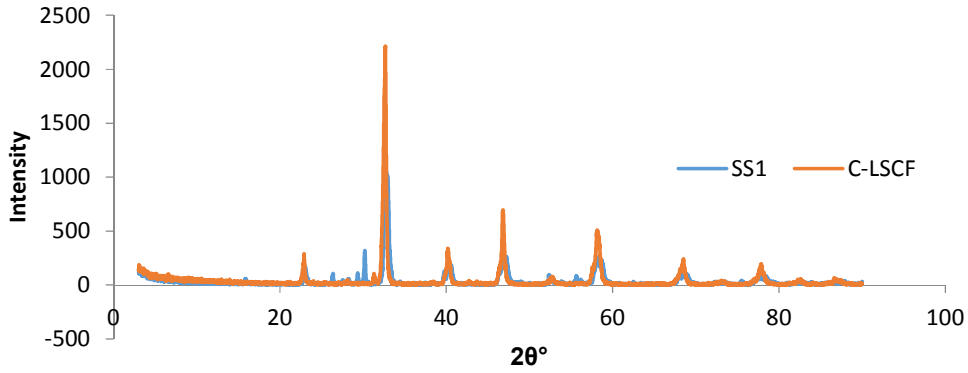

Figure 2: Comparison XRD pattern of C-LSCF and SS1

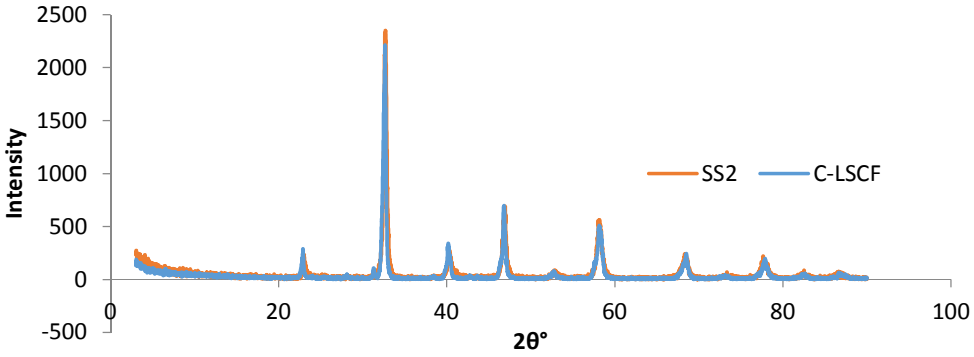

Figure 3: Comparison XRD pattern of C-LSCF and SS2

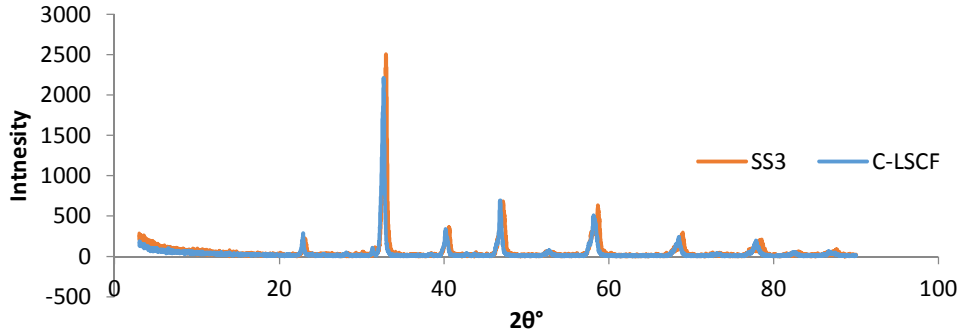

Figure 4: Comparison XRD pattern of C-LSCF and SS3

CONCLUSION

Increasing milling time or milling intensity can improved the LSCF powder composition produced via solid powder. Based on the findings through XRD measurement, it can be concluded that the powder produced via solid state reaction is on par with the commercialized LSCF powder produced via spray drying method.

In order to better understand the properties of LSCF powder produced via solid state reaction, future powder characterizations should be conducted such as surface morphology observation through scanning electron microscopic (SEM) and surface area calculation using Brunauer, Emmett, Teller (BET) theory.

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