

GROWTH AND STRUCTURAL CHARACTERIZATION OF
LANTHANUM-STRONTIUM-COBALT THIN FILM SYNTHESIZED BY
SOL-GEL DIP COATING FOR SOLID OXIDE FUEL CELL

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UNIVERSITI TEKNOLOGI MALAYSIA

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DEDICATION

IN THE NAME OF ALLAH, THE ALMIGHTY

To my lovely father and mother,

Deraman Bin Abdullah and Fatimah Salwah Binti Daud who gave me endless love, trust, constant encouragement over the years, and for their prayers.

To my spouse,

Mohammad Farizuddin Bin Salebi for being very understanding and supportive in keeping me going, enduring the ups and downs during the completion of this thesis.

To my family, for their patience, support, love and prayers

Thank you for all your love and support...

This thesis is dedicated to them.

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ABSTRACT

The porosity of a solid oxide fuel cell (SOFC) cathode made of lanthanum-strontium-cobalt (LSC) has not been addressed sufficiently. The LSC thin films were deposited on glass substrates by sol-gel dip coating method with different number of layers of film (1 – 4 layers), different annealing temperatures (400 – 600 °C) at 100 °C temperature intervals, and different solution temperatures (30 – 60 °C) at 10 °C temperature intervals. The dip coating solution was prepared using 40 ml of ethanol, 1 g LSC powder and 0.07 g polyvinyl alcohol (PVA). The structural properties of SOFC cathode were characterized using X-ray diffraction and Fourier transform infrared spectrophotometer. The morphology of SOFC cathode was determined using atomic force microscope and field emission scanning electron microscope. The elemental compositions of LSC films were confirmed using energy dispersive X-ray spectroscopy. The X-ray diffraction results showed that crystalline temperature of LSC was 500 °C and crystallite size decreased as annealing temperature increased. From the atomic force microscope and field emission scanning electron microscope analysis, the LSC film became smoother as number of layers and annealing temperature increased. Fourier transformed infrared spectrophotometer results showed presence of metal-oxygen bonds at 447 cm^{-1} and 916 cm^{-1} which correspond to Co-O, Sr-O, La-O bonding at solution temperature 30 °C. Hence, it can be concluded that annealing and solution temperature plays vital role in producing high quality LSC thin films.

ABSTRAK

Keliangan katod sel fuel oksida pepejal (SOFC) yang diperbuat daripada lanthanum-srontium-kobalt (LSC) belum dapat ditangani dengan secukupnya. Saput tipis LSC telah diaplikasikan pada substrat kaca dengan kaedah salutan celup sol gel dengan bilangan lapisan saput yang berlainan (1 – 4 lapisan), suhu sepuh lindap yang berlainan (400 – 600 °C) pada selang suhu 100 °C dan suhu larutan yang berbeza (30– 60 °C) pada selang suhu 10 °C. Larutan salutan celup telah disediakan menggunakan 40 ml etanol, 1 g serbuk LSC dan 0.07 g polivinil alkohol (PVA). Sifat struktur katod SOFC dicirikan dengan menggunakan pembelauan sinar-X dan spektrofotometer inframerah transformasi Fourier. Morfologi katod SOFC ditentukan dengan menggunakan mikroskop daya atom dan mikroskop elektron pengimbasan pancaran medan. Komposisi unsur saput LSC telah disahkan dengan penggunaan spektroskopi sinar-X penyebaran tenaga. Keputusan pembelauan sinar-X menunjukkan bahawa suhu hablur LSC ialah 500 °C dan saiz hablur menurun apabila suhu sepuh lindap meningkat. Daripada analisis mikroskop daya atom dan mikroskop elektron pengimbasan pancaran medan, saput LSC menjadi lebih licin apabila bilangan lapisan dan suhu sepuh lindap meningkat. Keputusan spektrofotometer inframerah transformasi Fourier menunjukkan kehadiran ikatan logam-oksigen pada 447 cm^{-1} dan 916 cm^{-1} yang mewalili ikatan Co-O, Sr-O, La-O pada suhu larutan 30 °C. Oleh itu, dapat disimpulkan bahawa suhu sepuh lindap dan suhu larutan memainkan peranan penting dalam menghasilkan saput tipis LSC berkualiti tinggi.

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LIST OF ABBREVIATIONS

SOFC	-	Solid Oxide Fuel Cell
IT-SOFC	-	Intermediate Temperature-Solid Oxide Fuel Cell
LSC	-	Lanthanum Strontium Cobalt
AFM	-	Atomic Force microscopy
FTIR	-	Fourier Transform Infrared
XRD	-	X-ray Diffraction
EDX	-	Energy Dispersive X-Ray
FESEM	-	Field Emission Scanning Electron Microscope
PVD	-	Physical Vapour Deposition
CVD	-	Chemical Vapour Deposition
RMS	-	Root Mean Square
RA	-	Average of Roughness
W-H	-	Williamson-Hall

LIST OF SYMBOLS

ϵ	-	Microstrain
d	-	Spacing between atomic planes
D	-	Mean grain size
λ	-	Wavelength
θ	-	Angle
k	-	Rate constant
E_a	-	Activation Energy
R	-	Gas constant
n	-	Integer
\AA	-	Lattice parameter
β_S	-	Strain broadening
β_D	-	Size broadening

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Solid oxide fuel cell (SOFC) is a device that produce power of electrochemical transformation from oxidizing a fuel. For the electrochemically changing, over substance vitality into electrical vitality and warmth without the requirement of direct combustion as a moderate stride, the fuel cell are very suitable gadgets and giving higher transformation efficiencies than routine vitality frameworks (Sun, Hui and Roller, 2009). Fuel cells are also gadget that produces power by a mixture response. The responses that create power occur at the terminals. They are various kinds of fuel cells, two electrodes, one positive and one negative, which are the cathode and anode (Shao, Zhou and Zhu, 2012). Some of the fuel cell has an electrolyte, which conveys electrically charged particles from one anode to the next, and a catalyst, which speeds the responses at the cathodes. Power device described by their electrolyte material, the SOFC has a strong oxide or ceramic electrolyte.

Fuel cells include high efficiency, long-term stability, fuel flexibility, low emissions and relatively low cost. High temperature energy components, for thin film SOFC, work between 600 °C and 1000 °C and can use hydrogen, regular gas or hydrocarbons. They work at high temperature between to guarantee satisfactory ionic conduction in the electrolyte. Lowering the temperature of a SOFC to 550-800 °C can keep the debasement of the cell parts, diminish the manufacture cost and enhance the adaptability in cell configuration, however would expand the ohmic resistance (Bansal and Zhong, 2006). Therefore, for over 10 years, broad research has been pursued to prepare ceramic oxides with blended ionic and electronic conductivity (perovskite oxides) and to concentrate the plausibility of utilizing them as cathode materials for low temperature solid oxide energy components (Hieu, Park and Tae, 2012).

The Lanthanum Strontium Cobalt (LSC) was used as a material for the film in SOFC. Based on the other oxide material, the mixed ionic-electronic conducting Lanthanum Strontium Cobalt Ferrite (LSCF) or Lanthanum strontium cobaltite (LSC) perovskites are well known class of SOFC cathode film materials. The LSC have been demonstrated as high-performance materials for intermediate temperature SOFC (Han *et al.*, 2012). LSC can upgrade oxygen reduction kinetics compared to the conventional other cathode material LSM by providing an additional pathway through the bulk of the perovskite structure (Banel *et al.*, 2013). There are many methods for the LSC can be performed such as sputtering, thermal evaporation and pulsed laser deposition (PLD). Previously, many researches focused on synthesizing LSC films via Chemical Vapour Deposition (CVD) method. The chemical technique can be used such as spray pyrolysis, CVD and sol-gel method (Baque *et al.*, 2006). Among all of the methods, further studies were used sol-gel method such as dip coating technique to synthesis LSC thin film because dip coating technique process was used wet chemical material which is major method of sol-gel (Bi and Traversa, 2014).

Experimental studies are bringing out at temperatures and oxygen partial pressures of suitable for IT-SOFC (Egger *et al.*, 2012). Besides, LSC have low catalytic compared than other materials for cathode film. The polarization resistance also low and have advantage of its in case to make the single chamber conditions was stable (Rembelski *et al.*, 2012). The one of the rate-determining steps for the oxygen reduction was exchanging the oxygen at the perovskite or air interface. When the thicknesses of the porous layers are expanding, it gives that increasing perovskite area and makes the larger number of sites for oxygen exchange. Therefore, the improvement of the cathode activity can be accomplished by using nonporous thin films with small grain sizes (Evans *et al.*, 2015).

Meanwhile, the homogeneous and heterogeneous can be observed in many view of natural and unnatural substances. Local measurement of particle orientation and size distribution would have recognized the nucleation growth in a material system. The development of homogenous and heterogeneous can control by nucleation process and grain growth (Baniassadi *et al.*, 2011). It is clear that by using grain growth, the nucleation can be developed at certain level based on their morphology

performance. Based on the random nucleation points, the growth occurs as in crystalline grain growth when the initial microstructure generation basic cells are created. As conclusion, LSC were investigated based of their, chemical stability, polarization resistance and oxygen performance in film and nucleation growth mechanism compare to other material that used for cathode of SOFC.

1.2 Problem Statement

The high efficiencies of SOFC and the usage of high energy density fuels give rise to higher energy power sources. By investigate the growth of nucleation on the films, will help the performance of the films. The cathode of SOFC must be a porous layer where the oxygen reduction reaction (ORR) takes place and transported through the porous layer of cathode as current collector to reach the reaction site. The peak must crystallites for the good structure of cathode and also for the stability of the perovskite structure meanwhile for the morphology, need get a rough surface to develop gas permeability and ionic and electronic conductivities. The crystal structure of cathode materials deviates from the ideal cubic perovskite structure, and was commonly orthorhombic or rhombohedral. Dip coating technique shows some of advantages such can control the chemical composition to get multi-component oxide film under simple innovative equipment, low temperature and also based on Aydemir and Karakaya, 2015, the method will improve cell durability and reduce the system cost.

Working with high temperature will brought many problems as state by Gwon in 2014 where will cause the decreasing the cell life time, interfacial diffusions, sealing problems. To build its aggressiveness and make SOFC innovation financially attainable, bringing down the operation temperature to the intermediate range of around 300-600 °C (Shao, Zhou and Zhu, 2012) (Richter *et al.*, 2009). A decrease in temperatures would enable less expensive materials (ferritic hardened steels for interconnect) and enhance reaction to speedy start-up reshaping warm cycling from encompassing to working temperatures (Ding *et al.*, 2014).

Besides, reducing the electrolyte thickness, it will give the results ohmic resistance was reducing and different of the cell performance. With that, fabrication of films of these electrolytes can boost the SOFC performance (Bi and Traversa, 2014). According to the Bu in 2013, the grain boundary and grain resistance are mainly affected to the electrolyte conductivity and caused the exchanging of grain size. The influence on the thickness of LSC electrode as a cathode as state by Evans in 2015 also give the different result of LSC thin film. The cathode layer has porous morphology that enhances oxygen diffusion depending on their thickness (Gwon *et al.*, 2014). The effectiveness about solution temperature also were gave the attention in this performance. By increasing the solution temperature, the performance of the film such as particle distribution was also effected the performance on the film. Table 1.1 shows the summary of findings from previous researchers.

Table 1.1 Summary of findings from previous researchers

Authors, Year	Finding/Result
Benel <i>et al.</i> , 2013	Used Pulsed laser deposition. These methods are not cost-effective and not easy to scale up.
Benel <i>et al.</i> , 2013	The influence of the thickness of LSC electrodes prepared by spin coating on thin-film SOFC and found that the thickness of LSC functional layer was controlled within the range of 150-500 nm by adjusting the dispersion concentration and spin coating parameters. Below of 150 nm no further improvement of the LSC electrodes was observed.
Evens <i>et al.</i> , 2013	LSC thin film cathodes have a larger grain size at 550 °C.
da Côte <i>et al.</i> , 2013	Smooth surface over to a high specific surface area and large number of reaction sites
Muthukrishnan <i>et al.</i> , 2016	Crystallinity and the existence of most stable hexagonal wurtzite structure using ZnO thin films by sol-gel dip coating.
Solovyev <i>et al.</i> , 2017	At temperature below 600 °C, the deposited films were amorphous and they remained amorphous until annealing at 800 and 1000 °C, the film becomes crystalline. The films also dense at low temperature.

1.3 Objectives

The objectives of the research are:

- (a) To evaluate the growth mechanism of studied of Lanthanum Strontium Cobalt thin films.
- (b) To characterize the structure and morphology of Lanthanum Strontium Cobalt thin films prepared using dip coating method.
- (c) To determine the influence of growth parameters such as different layers (one to four), annealing temperature (300-600 °C) and solution temperature (30-60 °C) on the structure and morphology of proposed Lanthanum Strontium Cobalt thin films.

1.4 Scope of the Study

The preparations of LSC thin film will be conducted by dip coating method. Dip coating method will be used in the deposition of LSC thin film with constant withdrawal speed at 60 rpm for different coating layer onto the glass substrate, different annealing temperature and different temperature of the stirring suspension/solution of LSC. The parameters temperature of annealing and the temperature of suspension was constant for the first parameter, meanwhile the layer of thickness are changing. Second parameter was continued with constant thickness of layer and solution temperature of suspension LSC with different annealing temperature. The annealing temperature that used for second parameter was based on the optimum result from the first parameter.

For the last parameter, the optimum results of layers' thickness and annealing temperature were used to get the entire sample the optimal growth with using the different solution temperature of LSC. The properties of the structural were characterized by using X-ray Diffraction (XRD), Fourier Transform Infrared Spectral (FTIR) and Energy Dispersive X-Ray Spectroscopy (EDX). For morphological

characterization were using Field Emission Scanning Electron Microscope (FESEM) and Atomic Force microscopy (AFM). This project is principally experimental.

1.5 Significant of the Study

The most important in dip coating methods was the ability to tune the microstructure of the deposited film. This study will be useful for industries since LSC cathode for SOFC application continue to play important role such as improve long term stability, high power density and high energy conversion efficiency. Furthermore, by the lowering the temperature, will low the cost in SOFC and will produce electrical energy with minimal environment impact. By using this information, it will help industrial to develop a better quality of LSC thin film. Dip coating method was one of the alternative methods that usually use in small scale for laboratory. Dip coating method for fabrication of LSC thin film is simple, non-toxic, non-vacuum system and hence suitable for large area coating. On the other hand, this method more advantageous compared to other deposition methods due to its ability to prepare high quality thin films in large scale with excellent control of stoichiometry, apparatus and raw materials (Ghodsi and Absalan, 2010).

1.6 Outline of Thesis

A general background of study and brief introduction of LSC thin film nanostructure and SOFC were discussed in Chapter 1. Then it was followed by problem statement, objective and scope of study. In Chapter 2, the elaboration of thin film, solid oxide fuel cell, LSC structure, sol-gel technique, perovskite and theory of dip coating method was presented. Chapter 3 is focused on the fabrication method and characterization technique of LSC thin film by dip coating method. The Chapter 4 was present the result and analysis on the structural properties, morphology and growth mechanism of LSC thin film. The conclusion of study was summarizing in Chapter 5.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter explains about the hypothesis identified with our exploration, including the definition of SOFC in general, the type of SOFC, the technique using dip-coating method, some basic knowledge of thin film, perovskite and the LSC mechanisms and properties.

2.2 Solid Oxide Fuel Cell

2.2.1 Background

In the 21st century, SOFC known as green energy, because it has very high energy conversion efficiency and very low emissions of air pollution, has high fuel reforming performance (Wen *et al.*, 2002) (Cebollero *et al.*, 2017). According Zhou *et al.*, in 2018 state, SOFC also known as an electricity generation instrument that change over chemical energy directly into electrical energy because of the higher efficiency and less environmental impact than traditional power generation methods. The routine of SOFC work was at high temperature in between of 800-1000 °C and the very effective, environment friendly transformation advancements, so the Chang *et al.*, in 2017 was assume a key part in future vitality supply and capacity. It can produce power and warmth utilizing flexible fills including hydrogen, methane and light hydrocarbons (Wei *et al.*, 2015). Recently, significant effort has committed to the improvement of Intermediate Temperature SOFC (IT-SOFC) (Takeshita *et al.*, 2014) (Zhan *et al.*, 2015). Operating in middle or low temperature (500-700 °C) not just diminishes the corruption of fuel cell stack parts and drags out the lifetime of fuel cell systems, it also enlarges materials choice. The poor activity of traditional cathode materials also

happened that was discovered by Shao and Haile, 2004 and the modest stainless steel materials can be utilized as interconnect at middle or low temperatures based on Vargas *et al.*, 2012. Bringing down the temperature causes less requesting on the seals (Lee *et al.*, 2002) and the equalization of-plant parts, rearranges thermal management, helps in quicker startup (Samat *et al.*, 2016) and chill off, and results in less debasement of cell and stack segments.

The polar resistance (R_p) will loss of voltage or overcapacity when decreasing the SOFC operating temperature. Polarity cathodes contribute to the decline in SOFC performance and it was loss the most common voltages versus ohm polarization and polarization activation. But, these advantages of its more benefits in SOFC which is, activity in the development of SOFC capable of operating in the temperature range of 650-800 °C (Narottam, Bansal and Zhong, 2006) (Weber and Ivers-Tiffée, 2004) (Xia and Liu, 2002) had increased dramatically in the last few years. Besides, annealing above 600 °C would minimize the cracking of membrane of the sample (Angoua *et al.*, 2011) because the limited temperature for the maximum processing temperatures of microscale SOFC was at 600 °C and continue with the increasing of the grain growth and the ratio of crystalline of the material. Moreover, the low polarization resistance for maximum of voltage and power output still can be achieved with the stability of the thin film was maintaining (Angoua *et al.*, 2011). Figure 2.1 shows other advantages of lowering the temperature of cathode.

The operating temperature of a SOFC was confined by thermally actuated transport process particle and electrochemical responses, for example, the oxide particle conductivity of the strong electrolyte and distinctive reaction step in the electrodes, individually, at the cathode or electrolyte interfaces. The improvement of the cell and stacks for a middle of temperature SOFC working financially in range of 500 °C and 700 °C were required, while for new materials, the temperatures were below 600 °C meanwhile cell ideas, creation advancements and appropriate stack originator must be produced according to Weber and Ivers-Tiffée, 2004.

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- 1) Shahirah Deraman, R. Muhammad & W. N. W. Syamsuri, AFM & XRD Analysis of Lanthanum Strontium Cobalt Thin Film Fabricated Using Sol Gel Dip Coating Method, 3rd International Conf on the Application of Science & Mathematics (SCIEMATHIC2017), UTHM Pagoh, 24-25 Oct 2017.