MORPHOLOGY, STRUCTURAL AND ELECTRICAL PROPERTIES OF NICKEL-OXIDE-YTTRIA-STABILIZED-ZIRCONIA THIN FILM FOR SOLID OXIDE FUEL CELL ELECTRODE APPLICATION

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DEDICATION

I dedicate this work,

To my beloved and lovely father and mother,
Md Amin Bin Alwi
Fatimah Binti Awang

And all my siblings and friends,
Love and spirit from all of you gives me inspired to complete my study

Special thanks to my supervisors and co-supervisor
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ABSTRACT

Achieving high quality electrode materials for efficient solid oxide fuel cell (SOFC) operation is challenging. SOFC requires porous electrode material. One of the common anode materials used is nickel oxide-yttria stabilized zirconia (NiO-YSZ). NiO-YSZ thin films were successfully deposited on glass substrates using sol-gel dip coating technique. The growth parameters considered were different coating layers (1, 2, 3 and 4 layers), different annealing temperature (300 °C, 400 °C, 500 °C and 600 °C) and different solution temperature (30 °C, 40 °C, 50 °C and 60 °C). The suspension was made from the mixture of NiO-YSZ powder and acetylacetone. All the prepared samples were characterized using X-ray diffraction (XRD), atomic force microscope (AFM), field emission scanning electron microscope (FESEM), energy dispersive X-ray (EDX) spectroscope, four point probe measurement and attenuated total reflectance-Fourier transformed infrared (ATR-FTIR) spectrometer. The thickness of the thin films was measured using surface profiler. The XRD results showed that intensity increased at the Layer-3 as number of coating layers increased. From the FESEM and AFM analysis, the presence of porosity was clearly shown as the number of coating layers and annealing temperature increased. The presence of Ni, O, Y, Zr and Si elements were revealed using EDX spectroscope. The ATR-FTIR results show the presence of bonds at 780 cm^{-1}, 940 cm^{-1} and 1230 cm^{-1} which correspond to Si-O, ZrO_2 and Zr-O-Si bonding at solution temperature 30 °C. Overall, the present findings conclude that annealing, coating layers and solution temperature play vital role in producing beneficial quality NiO-YSZ thin films for solid oxide fuel cell (SOFC) anode.

ABSTRAK

Mendapatkan bahan elektrod berkualiti tinggi untuk operasi sel bahan bakar oksida pepejal (SOFC) yang cekap adalah mencabar. Sel SOFC memerlukan bahan elektrod yang berliang. Salah satu bahan anod yang biasa digunakan ialah nikel oksida-yttria terstabil zirkonia (NiO-YSZ). Saput tipis NiO-YSZ telah berjaya diendapkan di atas substrat kaca dengan menggunakan teknik salutan sol-gel. Parameter pertumbuhan yang dipertimbangkan ialah lapisan salutan yang berbeza (1, 2, 3 dan 4 lapisan), suhu penyepuhlidapan yang berbeza (300 °C, 400 °C, 500 °C dan 600 °C) dan suhu larutan yang berbeza (30 °C, 40 °C, 50 °C dan 60 °C). Ampaian dibuat daripada campuran serbuk NiO-YSZ dan asetilaseton. Semua sampel yang tersedia telah dicirikan dengan menggunakan pembelauan sinar-X (XRD), mikroskop daya atom (AFM), mikroskop imbasan elektron pancaran medan (FESEM), serakan tenaga sinar-X (EDX), pengukuran prob empat titik dan spektrometer pengecilan kepantulan inframerah transformasi Fourier (ATR-FTIR). Ketebalan saput tipis diukur menggunakan pemprofil permukaan. Keputusan XRD menunjukkan keamatan meningkat pada lapisan ke 3 apabila bilangan lapisan salutan meningkat. Daripada analisis FESEM dan AFM, kewujudan keliangan jelas ditunjukkan apabila bilangan lapisan salutan dan suhu sepuhlindapan meningkat. Kewujudan unsur Ni, O, Y, Zr dan Si telah didedahkan menggunakan spektroskop EDX. Keputusan ATR-FTIR menunjukkan kewujudan ikatan pada 780 cm^{-1}, 940 cm^{-1} dan 1230 cm^{-1} yang sepadan dengan ikatan Si-O, ZrO_2 dan Zr-O-Si pada suhu larutan 30 °C. Secara amnya, penemuan ini menyimpulkan bahawa sepuhlindapan, lapisan salutan dan suhu larutan memainkan peranan penting dalam menghasilkan saput tipis NiO-YSZ yang bermanfaat untuk anod sel bahan bakar oksida pepejal (SOFC).

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscope
YSZ	-	Yttria Stabilized Zirconia
NiO	-	Nickel Oxide
NiO-YSZ	-	Nickel Oxide-Yttria Stabilized Zirconia
SOFC	-	Solid oxide fuel cell
PVD	-	Physical vapor deposition
CVD	-	Chemical vapor deposition
LPCVD	-	Low pressure chemical vapor deposition
PECVD	-	Plasma enhanced chemical vapor deposition
MOCVD	-	Metal organic chemical vapor deposition
LECVD	-	Laser enhanced chemical vapor deposition
ATR-FTIR	-	Attenuated total reflected-fourier transform infrared
XRD	-	X-Ray diffraction
FESEM	-	Field emission scanning electron microscope
AFC	-	Alkaline fuel cell
NASA	-	National Aeronautics and Space Administration
PAFC	-	Phosphoric acid fuel cell
MCFC	-	Molten carbonate fuel cell
PEMFC	-	Polymer electrolyte membrane fuel cell
FWHM	-	Full width at half maxima
EDX	-	Energy dispersive x-ray
kW	-	kilowatt
ITSOFC	-	Intermediate temperature solid oxide fuel cell
SIS	-	Segmented-in-series
MEA	-	Membrane electrolyte assembly
CTE	-	Coefficient of thermal expansion
TPB	-	Triple phase boundary
EPD	-	Electrophoretic deposition
CNT	-	Classical Theory of Nucleation

LIST OF SYMBOLS

~	-	Approximately
$<$	-	Less than
SiO_2	-	Silica Oxide
CO_2	-	Carbon dioxide
d	-	Crystallite size
e^-	-	Electron
eV	-	Electron volt
H^+	-	Hydrogen ion
H_2	-	Hydrogen
H_2O	-	Water
I	-	Current
Ni	-	Nickel
O	-	Oxygen
OH^-	-	Hydroxide
T	-	Temperature
V	-	Voltage
Y	-	Yttrium
Zr	-	Zirconia
ZrO_2	-	Zirconia Oxide
β	-	The full width at half maxima
θ	-	The Bragg's diffraction angle
λ	-	Wavelength
°C	-	Celcius
μm	-	Micrometer
%	-	Percent
O	-	Oxide
r*	-	Critical radius
ΔG	-	Total Gibbs free energy
ε	-	Microstrain
γ_{sv}	-	Surface energies per unit area between substrate-vapour

γ_{ns}	-	Surface energies per unit area between nucleus-substrate
γ_{vn}	-	Surface energies per unit area between vapour-nucleus
σ	-	Conductivity
(% T)	-	Transmittance
ρ	-	Resistivity

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CHAPTER 1

INTRODUCTION

1.0 Introduction

This research is regarding the investigation of the synthesis of NiO-YSZ as an anode using sol-gel dip coating for solid oxide fuel cells. This section consists of the background of the study, problem statement, objectives of study, scope of the study and significant of study for this research.

1.1 Background of the Study

Fuel cell is a device that uses hydrogen and oxygen to create electricity. There are several types of fuel cells such as, Alkaline Fuel Cell (AFC) (Zhao *et al.*, 2015), Molten Carbonate Fuel Cell (MCFC) (Zhang *et al.*, 2015), Phosphoric Acid Fuel Cell (PAFC) (Ansari, Tucker and Angell, 2013), Polymeric Electrolyte Membrane Fuel Cell (PEMFC) (Najafi *et al.*, 2015) and Solid Oxide Fuel Cell (SOFC) (Zhongfu *et al.*, 2015). Compared to other types of fuel cells, SOFC have taking great attention due to high electrical efficiency, high energy density and excellent fuel flexibility (Cho, Lee and Cha, 2014). Over the years, solid oxide fuel cells (SOFCs) operate at high temperature in the range of 800 °C to 1000 °C. However, high temperature leads to drawbacks such as increase the cost of SOFC, durability affects and life-time of the system. Reducing temperature below 800 °C helps reduce fabrication costs of the system thus wider range of material can be used (Joh *et al.*, 2017). Moreover, it would affect faster start-up time along the mechanical and chemical stability for portable applications. In intermediate temperature range between 500 °C to 750 °C of SOFCs, nanoscaled thin film was given a huge attention. The decrease in physical dimension down to nanometer scale is often linked with a dramatic change of the physical and electrochemical properties of materials (Zalga *et al.*, 2011).

According to (Noh *et al.,* 2009) , effective way to suppressing the coarsening of Ni in thin film can be resolved by post thermal treatment at high temperature around 1000 °C. However, within this range of annealing temperature might destroy the nanostructure of thin film components such as electrolyte and electrode. Thus, deposition of thin film required low temperature to obtain nanoscale. Despite that, investigation on the structure of nanocomposite thin film and factor of agglomeration are needed.

However, reducing the temperature will loss the resistivity of the electrolyte and over potential at anode are exaggerated thus decreases the cell performance. To overcome such problems, reducing the thickness of the component and optimize the electrode and interfaces of electron and ions transfer need to be improved. Recently, the fabrication of the components SOFCs has increase in the application of thin film technology. This is due to the advantages of thin film where reducing of ohmic loss and easily controlled in interfaces (Charpentier *et al.*, 2000). According to Mahato, at lower temperature the cell performance can be improve with using very thin electrolyte (few micrometers) and for the electrode and electrolytes nanoscale materials in required (Mahato *et al.*, 2015). Anode plays important role in the oxidation of fuel to generate electrons. The oxidation acts to remove the reaction products for the whole anode substrate which provide suitable electrical conductivity to avoid ohmic losses and prevent concentration polarization as well. It is found that the microstructural parameters such as particle size and phase distribution influence the gas permeability and electrical conductivity (Sanson, Pinasco and Roncari, 2008).

Nowadays, development of SOFCs was focus to low temperature which below 600 °C in order to achieve the commercialization. High conductivity more than 0.1 S cm^{-1} can be effective below 600 °C (Yuan *et al.*, 2018). This research studies the performance of NiO-YSZ thin film anode for SOFC application that equipped for temperature below 600 °C to create electric power for the compact application. Grain size, connectivity and permeability are the main microstructural characteristics for the anode performance in SOFC. Therefore, the microstructural and electrochemical relation performance of an electrode has been explored (Jung, Kim and Tuller, 2015). Electrodes nanostructured in terms of grain size have been applied intensively. This is due to the influenced of particle size and surface area of the catalyst effect the rate of charge transfer in electrode. Thus, polarization resistance at the electrode at low temperature increased the rate of electrode reaction by the nanostructured electrodes (Noh *et al.,* 2015 and Evans *et al.,* 2015).

The growth processes of such thin film electrodes play a decisive role in their functionality, wherein the formation of nano-sized islands in the initial deposition phase of the film must be controlled to achieve the best performance (Kajiokawa and Noda, 2005). An in-depth understanding on the mechanism of such degradation requires the knowledge of nucleation and growth processes of the thin film electrodes. It was also argued that the entire NiO grain surface associated to individual islands could not spread for complete occupation, a phenomenon called wetting layer mismatch (Vasylyev *et al.*, 2014). Growth of ZrO_2 film along the entire surface of NiO grain follows the planar mechanism. In this mechanism, several small nuclei are involved instead of one big nucleus wherein the zirconia grain serves as the nucleus for the condensation of layer by layer. Therefore, the growth mechanism of these nuclei to occupy the entire NiO grain surface can be assumed as islands rather than lateral spreading. In short, some of the nucleated crystals get separated and grown on each NiO grain. Despite many efforts such growth mechanism of multi-layered NiO-YSZ thin films is far from being clarified.

Conventional or non-conventional methods can be done on manufacturing the SOFC cell. Tape casting, slip casting, compaction or pressing, dip coating, screen printing and spraying are listed for the conventional methods. Whereas, the processes involve chemical, physical and electrochemical vapour depositions are include in the non-conventional methods (Meng *et al.*, 2013).

However, PVD and CVD techniques require expensive equipment and limited by relatively slower deposition rate than other methods. Meanwhile, EPD is a cost effective colloidal method (Lee *et al.*, 2007). Although many methods have been used to develop thin film, the cost-effective for commercial application of SOFCs is desirable to fabricate. Sol gel dip coating gives advantages in eliminating major problems such simple synthesis, long-term stability, solution, and low temperature processibility compared to other techniques (Settanni *et al.*, 2016). Dip coating exhibits remaining contacts quality between thin film layers which leads to narrow the delamination failure of the thin film layer after sintering process (Jang *et al.*, 2016).

1.2 Problem Statement

Presently, SOFCs with high operating temperature in the range of 800 °C to 1000 °C can incorporate internal fuel reformation by allowing multiple fuel options including natural gas. However, the high operating temperature of SOFC is disadvantageous in terms of long start-up time and high fabrication cost (Solovyev *et al.,* 2017). To overcome such drawbacks efforts are made to lower the operating temperatures of SOFCs below 600 °C. Reduction of temperature would decrease in the thermal stresses due to thermal expansion mismatch of the different materials used in a SOFC, negligible electrode sintering and chemical stability of the cell components (He *et al.,* 2017). Nevertheless, electrodes are expected to limit the performance of the cell (Fleig *et al.,* 2004). This emphasizes the importance of the electrode microstructure and suggests either the use of electrodes with nanometer-sized grain. Thus, electrodes need to be nanostructured for SOFC applications.

In the previous work, the SOFCs were fabricated with a traditional slip casting technique. Despite their complex, unstable and time cost, these techniques cannot produce porous NiO-YSZ thin film of thickness few µm (Chen *et al.,* 2009). This problem can be resolved by depositing NiO-YSZ thin film via sol-gel dip coating technique. This technique needs no expensive equipment and the working procedure is simple and time effective (Bai, Liu and Wang, 2009). Nonetheless, the effect of solution temperature stirring were not yet been reported. Furthermore, the limitations of materials and deposition (growth) time (Vasylyev *et al.,* 2016) were primarily ascribed. Therefore, nucleation by a Volmer–Weber mechanism may potentially be used in fuel cells due to hollow channels in the microstructure (Ramirez *et al.*, 2008). In this view, present study deposited NiO-YSZ nanostructured thin films using dip coating method to achieve good quality of anode useful for SOFCs. Therefore, NiO-YSZ thin films were thoroughly characterized to evaluate their morphology, structural and electrical properties under optimum deposition condition.

1.3 Objectives of the Study

The primary aims of this study are to investigate the optimum parameters in deposition of NiO-YSZ thin film. This aim was achieved through the following research objectives:

1. To determine the optimum growth parameter of NiO-YSZ thin film based on different layers (Layer-1, Layer-2, Layer-3 and Layer-4), different annealing temperature (300-600 °C) and solution temperature (30-60 °C).

2. To characterize the morphological, structural and growth mechanism of NiO-YSZ thin film effective for SOFC electrode application via AFM, FESEM, XRD, EDX and ATR-FTIR.

3. To determine the electrical of NiO-YSZ thin film using Four point probe.

1.4 Scope of the Study

This research focused on the synthesis of NiO-YSZ thin film using sol-gel dip coating method. For the solution, NiO-YSZ powder with the composition (NiO 60 wt. % and YSZ 40 wt. %) will be mix with the acetylacetone. The withdrawal speed of the deposition of NiO-YSZ thin film is constant to 60 mm/min. Growth parameters such as different layers, annealing temperature and solution temperature have been conducted in deposition of nio-YSZ thin film. For the different layers parameter, optimization was focused on 1 to 4 layers with constant 600 °C annealing temperature and solution temperature 30 °C. Meanwhile, for the annealing temperature parameter, the optimization was focused on annealing temperature range of 300 °C to 600 °C with the fix of 3 layers and 30 °C solution temperature. Lastly, for the solution temperature, the optimization was focus on temperature range 30 °C to 60 °C at constant annealing temperature 500 °C.

The characterization of NiO-YSZ thin film will be analyze by XRD, AFM, FESEM, EDX and four point probe equipments. The XRD is conducted to determine the orientation of single crystal, to find the crystal structure and to measure the size and shape of small crystalline regions. The surface morphology on NiO-YSZ thin film will be explored via AFM. The EDX measurement will be used to measure the compositional and image mapping of the thin film. FESEM will observe the surface morphology and cross-section of NiO-YSZ thin film. The 4-point probe machine which result the conductivity and resistivity data of the deposited films were assessed for the electrical properties of NiO-YSZ.

1.5 Significance of the Study

This study may help other researchers to synthesis of NiO-YSZ thin film using sol-gel dip coating method on structural, morphological and electrical characteristics when deposited on glass substrate for SOFC application. Furthermore, this research fundamentally important to explain the optimum growth parameter for deposition of NiO-YSZ thin film in terms of different layer, annealing temperature and solution stirring. For the application SOFC devices, to produce anode at the lower temperature below 600 °C is the main focus. Development on thin film NiO-YSZ will be the interesting strategy to produce anode SOFC. This research will help to improve understanding, gain new information and identify the crystallographic texture, surface morphology, compositional and the changes of resistivity and conductivity of NiO-YSZ thin film.

1.6 Outline of the thesis

This thesis is constructed into five chapters. The background of the study and brief introduction about NiO-YSZ and solid oxide fuel cell are discussed in Chapter 1. Then, problems related to research, objectives and scope of study are discussed. The literature review of the solid oxide fuel cell, NiO-YSZ anode material and theory of dip coating method were combining in the Chapter 2. The processes related to material selection and characterization is also briefly explained. Chapter 3 is the experimental technique of NiO-YSZ thin film grown by sol-gel dip coating method followed by characterization technique. The structural properties, morphology, electrical properties of NiO-YSZ thin film of the results and analysis are reported in chapter 4. Lastly, Chapter 5 discussing the conclusion of the research and provides a suggestion for future work in this study area.

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Appendix N LIST OF PUBLICATION

Amin, N. H., Muhammad, R., & Shamsuri, W. N. W. (2017). Morphological and Electrical Characterization of Nickel Oxide-Ytrria Stabilize Zirconia Thin Film Prepared Using Sol Gel Dip Coating Method. *Journal of Science and Technology*, 9(3).