DEPOSITION OF YTTRIA STABILIZED ZIRCONIA THIN FILMS BY RADIO FREQUENCY MAGNETRON SPUTTERING FOR SOLID OXIDE FUEL CELL

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I dedicate this work

To my beloved father and lovely mother,
Tuan Hj Jaffar Bin Atan
Norhayati Chang Binti Abdullah
For the love, kindness, patience and prayer that have brought me to this far.

To my siblings and loves,
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ABSTRACT

Yttria stabilized zirconia (YSZ), the most significant material of electrolytes in solid oxide fuel cell (SOFC) requires careful synthesis and thorough characterizations with improved properties. Presently, YSZ electrolytes are largely manufactured by screen printing or spraying techniques followed with subsequent sintering. Despite their low-cost with high throughput, these techniques cannot produce dense YSZ electrolytes thin film of thickness with nanometer size. In this research, this problem was resolved by depositing dense YSZ electrolyte thin film with good electrical properties through radio frequency (RF) magnetron sputtering (RFMS) technique. YSZ thin films were successfully deposited on alumina (Al_2O_3) substrate through reactive RFMS technique. The formation of fully dense and highly porous films for efficient SOFC fabrication is dependent upon deposition parameters of RFMS such as gas pressure, deposition power and rate, substrate temperature and sputtering time. Pure nanostructured YSZ thin films were prepared in the atmosphere of mixed argon and oxygen gas. To optimize the YSZ film properties, deposition parameters such as RF power, substrate and annealing temperature were varied. Samples were characterized using X-ray diffraction (XRD), field emission scanning electron microscopy (FESEM), energy dispersive X-ray (EDX) spectroscopy, atomic force microscopy (AFM), surface profiler, and four point probe analysis. XRD spectra revealed the growth of Zr-Y-O nanocrystallites along the lattice plane of (220) and (111), where the average crystallite size increased from 24.81 nm to 68.56 nm with the increase of RF power. The FESEM images displayed the homogeneous surface morphology of the deposited YSZ thin film. Meanwhile, the AFM topological analysis showed an increase in grain size from 26.29 nm to 76.41 nm and that surface roughness was reduced from 3.28 nm to 1.67 nm with increasing RF power from 50W to 150W. The resistivity of YSZ films was reduced from 745 Ω.cm to 1.33 Ω.cm with the increase of substrate temperature from 37°C to 500°C. Furthermore, the resistivity of the film was diminished with the decrease of YSZ thin film thickness from 66.08 nm to 8.25 nm. RF power of 100W and substrate temperature of 300°C was shown as the optimum parameter for depositing nanostructured YSZ thin films. The findings have proven that by lowering the thickness of nanostructured YSZ thin film deposited with substrate temperature of 500°C or less, an optimum film can be achieved. Overall, the present findings may contribute towards the development of YSZ thin film based electrolytes beneficial for SOFC.

ABSTRAK

Zirkonia terstabil yttria (YSZ), bahan paling penting sebagai elektrolit di dalam sel bahan bakar oksida pepejal (SOFC) memerlukan sintesis yang cermat dan pencirian untuk sifat yang lebih baik. Pada masa ini, elektrolit YSZ sebahagian besarnya dihasilkan melalui teknik percetakan skrin atau teknik penyemburan diikuti dengan pensinteran. Walaupun berkos rendah dengan truput yang tinggi, teknik ini tidak boleh menghasilkan saput tipis elektrolit YSZ tumpat dengan ketebalan berukuran nanometer. Dalam kajian ini, masalah ini telah diselesaikan dengan mengendapkan saput tipis elektrolit YSZ tumpat dengan sifat elektrik yang baik melalui teknik percikan magnetron frekuensi radio (RFMS). Saput tipis YSZ telah berjaya diendapkan ke atas substrat alumina (Al_2O_3) melalui teknik RFMS reaktif. Pembentukan saput yang berketumpatan penuh dan berkeliangan tinggi untuk pembinaan SOFC yang efisen bergantung kepada parameter pertumbuhan RFMS seperti tekanan gas, kuasa dan kadar endapan, suhu substrat dan masa percikan. Saput tipis YSZ tulen berstruktur nano telah disediakan pada persekitaran atmosfera campuran gas argon dan oksigen. Untuk mengoptimumkan sifat saput YSZ, parameter pertumbuhan seperti kuasa RF, suhu substrat dan suhu sepuhlindap telah diubah. Sampel dicirikan dengan pembelauan sinar-X (XRD), mikroskopi imbasan elektron pancaran medan (FESEM), spektroskopi sinar-X serakan tenaga (EDX), mikroskopi daya atom (AFM), pembukah permukaan, dan analisis prob empat titik. Spektrum XRD mendedahkan pertumbuhan hablur nano Zr-Y-O di sepanjang satah kekisi (220) dan (111), di mana saiz hablur purata meningkat daripada 24.81 nm kepada 68.56 nm dengan peningkatan kuasa RF. Imej FESEM memaparkan morfologi permukaan saput tipis YSZ terendap yang homogen. Manakala analisis topologi AFM menunjukkan peningkatan saiz butiran daripada 26.29 nm kepada 76.41 nm dan pengurangan kekasaran permukaan daripada 3.28 nm kepada 1.67 nm dengan peningkatan kuasa RF daripada 50W kepada 150W. Kerintangan saput YSZ telah berkurang daripada 745 Ω.cm kepada 1.33 Ω.cm dengan peningkatan suhu substrat daripada 37°C kepada 500°C. Selain itu, kerintangan saput berkurang dengan penurunan ketebalan saput tipis YSZ daripada 66.08 nm kepada 8.25 nm. Kuasa RF sebanyak 100W dan suhu substrat 300°C ditunjukkan sebagai parameter optimum untuk pengendapan saput tipis YSZ berstruktur nano. Penemuan ini menunjukkan saput tipis optimum boleh diperolehi dengan pengurangan ketebalan saput tipis YSZ pada suhu substrat di bawah 500°C. Keseluruhan, penemuan ini boleh menyumbang ke arah pembangunan elektrolit berasaskan saput tipis YSZ yang berfaedah untuk SOFC.

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LIST OF ABREVATIONS

AFM	-	Atomic force microscope
YSZ	-	Yttria Stabilized Zirconia
RF	-	Radio frequency
SOFC	-	Solid oxide fuel cell
PLD	-	Pulsed laser ablation
PVD	-	Physical vapor deposition
EB-PVD	-	Electron beam physical vapor evaporation
CVD	-	Chemical vapor deposition
ALD	-	Atomic layer deposition
DCMS	-	Direct current magnetron sputtering
XRD	-	X-Ray diffraction
FESEM	-	Field emission scanning electron microscope
AFC	-	Alkaline fuel cell
PAFC	-	Phosphoric acid fuel cell
MCFC	-	Molten carbonate fuel cell
PEMFC	-	Polymer electrolyte membrane fuel cell
HVC	-	High vacuum coater
ICSD	-	Inorganic crystal structure database
FWHM	-	Full width at half maxima
EDX	-	Energy dispersive x-ray

LIST OF SYMBOLS

~	-	approximately
<	-	Less than
>	-	More than
Al	-	Aluminium
Al_2O_3	-	Aluminum Oxide
Ar	-	Argon
Bi	-	Bismuth
Co	-	Cobalt
CO_2	-	Carbon dioxide
Cr	-	Chromium
Cs	-	Caesium
D	-	Crystallite size
e^-	-	Electron
eV	-	Electron volt
H^+	-	Hydrogen ion
H_2	-	Hydrogen
H_2O	-	Water
H_2SO_4	-	Sulphuric Acid
H_3PO_4	-	Phosphoric acid
HCl	-	Hydroxyl Chloride Acid
He	-	Helium
HF	-	Fluoric Acid
HNO_3	-	Nitric Acid
I	-	Current
K	-	Potassium

KOH	-	Potassium hydroxide
L	-	Grain size
Li	-	Lithium
Mg	-	Magnesium
n_0	-	Refractive index
Na	-	Sodium
Ni	-	Nickel
O_2	-	Oxygen
OH^-	-	Hydroxide
Sc	-	Scandinavia
sscm	-	standard cubic centimeters per minute
T	-	Temperature
V	-	Voltage
Y	-	Yttrium
Zn	-	Zinc
Zr	-	Zirconia
ZrO_2	-	Pure zirconia
β	-	The full width at half maxima
δ	-	Dislocation density
θ	-	The Bragg’s diffraction angle
λ	-	Wavelength

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CHAPTER 1

INTRODUCTION

1.1 Background of the Study

Solid oxide fuel cell (SOFC) innovation offers numerous focal points over conventional vitality change frameworks, including low discharge and high effectiveness. This has made SOFC turned out to be progressively appealing to the utility, car, and guard ventures. SOFC requires high temperature (700-1000°C) to work as it is an all strong state vitality transformation gadget. Power is delivered by electrochemically joining the fuel and oxidant gasses over an ionically leading oxide film.

Yttria stabilized zirconia (YSZ) is the most usually utilized material for SOFC electrolytes (Shaula *et al.*, 2009) as it is a sufficient ionic conductor, has a low electronic conductivity, and is moderately shabby to handle contrasted with other electrolyte materials. The ionic conductivity in the stabilized zirconia framework is because of the versatility of O^{2-}opportunities made when substituting Zr^{4+} by Y^{3+} in the cationic system. Be that as it may, the ionic conductivity of cutting edge YSZ based electrolytes would just be adequate at raised temperatures (800-1000°C) which then brought about expanded reactivity of cell center parts, radically constrained lifetimes and requires the utilization of costly interconnect materials (Sønderby *et al*., 2015). Along these lines, diminishment of the operation temperature to an intermediate temperature run (200-300°C) is a key goal in SOFC research to deal with the specified issue. It is promising to accomplish by reducing operation temperatures, the decrease of electrolyte thickness will diminish the general resistance (Bernay *et al.*, 2003; De Souza, 1997; Feng *et al*., 2015).

The utilization of SOFC and other solid state ionic gadgets in energy applications is restricted by their prerequisite for hoisted working temperatures, regularly above 800°C. Thin-film layers permit low-temperature operation by diminishing the ohmic resistance of the electrolytes. However, scaling up remains a huge test since large area membrane under ~100 nm thick is helpless to mechanical failure (Bernay *et al*., 2003). This research will look for the execution of YSZ nanostructure electrolyte for SOFC application that is equipped for working temperature below 400 °C to create electric power for the compact application.

Such microstructures are unfavorable as they may result in internal leakage in the cell leading to decreased open cell potential. Therefore, an undesirable extra manufacturing step in the form of post-deposition annealing is often required to eliminate pores in such films and solve the issue (Lin *et al.*, 2014). Reports on cell tests performed on SOFCs containing magnetron sputtered electrolytes with a columnar structure have shown an electrochemical performance comparable to or inferior to cells containing tape cast electrolytes. This is not a significant improvement as it would have expected from the reduced electrolyte thickness (Aijaz *et al.*, 2014). Leaks in the electrolyte due to the columnar morphology are responsible for the poor performance. This has been proved by Nedelec *et al*.(2000) because, in contrast, their study has shown deposited electrolytes without visible continuous columnar structure has achieved significant improved electrochemical performance at low temperatures *(*Smeacetto *et al.*, 2010) .

The latter can be achieved by synthesizing YSZ thin films employing a variety of chemical and physical methods (Hidalgo e*t al*., 2011) such as pulsed laser ablation (PLD), chemical vapor deposition (CVD) (Feng, Chen *et al.*, 2015), atomic layer deposition (ALD) (Bernay, Ringuedé *et al.*, 2003), spin coating (K. Chen *et al*., 2006) and magnetron sputtering (Rezugina *et al*., 2010; Shaula *et al*., 2009; Sønderby *et al*., 2015). However, depositing YSZ thin films by physical vapor deposition (PVD) techniques for example; magnetron sputtering at relatively low synthesis temperatures will cause the formation of denser and columnar microstructures. RF magnetron sputtering is the suitable technique for the generation of the thin film to form highly dense and packed thin film with high porosity of films.

Radio frequency magnetron sputtering is a PVD-based technique for thin film deposition. Inherent to this technique is a relatively large fraction of ionization of the sputtered material with energies up to several 10s of eV that have been shown to provide added means for controlling the microstructural evolution during film growth (Depla *et al.*, 2016). This has shown to favor the growth of dense films at relatively low growth temperatures and to suppress columnar microstructure giving rise to globular film morphology (Wang *et al.*, 2013) which are desired in the production of thin film electrolytes for SOFC. Films were also synthesized by pulsed direct current magnetron sputtering (DCMS), for reference. As previous studies have shown that the substrate bias is a crucial parameter in order to obtain dense films suitable for SOFC application (Rezugina *et al.*, 2010), the bias voltage has been varied between depositions. Figure 1.1 shows the example of SOFC prototype which is used by industries for transportation application.

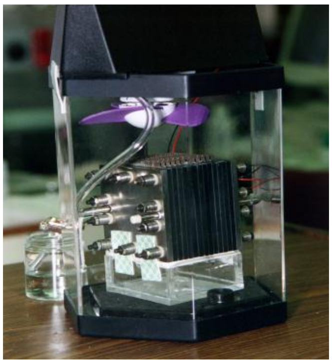

Figure 1.1 Example of prototype Solid Oxide Fuel Cell (Salam *et al*., 2008)

1.2 Problem Statement

Presently, SOFCs with high operating temperature in the range of 500°C-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 as well as mechanical and chemical compatibility. To overcome such drawbacks efforts are made to lower the operating temperatures of SOFCs below 600^{o}C which in turn gave the birth of so called intermediate temperature SOFCs (IT-SOFCs). Thus, creation of SOFCs with lower operating temperatures and high energy conversion efficiency will be prospective for transportation and portable power generation. Furthermore, such IT-SOFCs will lower the cost metallic interconnects, reduce the thermal stress, and shorten the start-up times. To achieve such goal good quality nanostructured YSZ electrolytes with lower ohmic losses that originate from the thermally activated ionic transport need to be synthesized using accurate yet cost-effective and easy method.

Recent research revealed that by reducing the thickness of YSZ thin film electrolyte it is possible to reduce the ohmic losses that arise from internal resistance of the electrolyte. Nanostructured thin film of YSZ can enhance the electronic conductivity of the thin film electrolyte and thereby the performance of SOFC. Currently, YSZ electrolytes are largely manufactured by screen printing or spraying techniques followed via subsequent sintering (Chen *et al.*, 2004). Despite their low-cost with high throughput, these techniques cannot produce dense YSZ electrolytes thin film of thickness few μm. This problem can be resolved by depositing dense YSZ electrolyte thin film with good electrical properties via RF magnetron sputtering technique. Moreover, by adjusting the growth conditions of the YSZ thin film in sputtering method, enhanced cell performance can be attained. Optimization of the deposition parameters of the RF sputtering method and subsequent systematic characterizations of the YSZ film are prerequisite for attaining efficient IT-SOFC. In this view, present study deposited YSZ nanostructured thin films using RF sputtering method to achieve good quality dense electrolyte useful for SOFC. Such 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 aim of the study was to investigate the optimum RF parameter in deposition of YSZ thin film. This aim was achieved through the following research objectives:

i. To deposit Yttria Stabilized Zirconia (YSZ) nanostructured thin films using radio frequency magnetron sputtering method.
ii. To determine the optimum growth parameters of YSZ nanostructured thin films in terms of radio frequency power, substrate temperature and annealing temperature.
iii. To evaluate the structural, morphological and electrical properties of deposited YSZ nanostructured thin films.

1.4 Scope of the Study

This research focused on the deposition of YSZ nanostructure onto an Al_2O_3 glass substrate using 99.99% pure YSZ ceramic as a target. The deposition process for the thin film by using RF magnetron sputtering method. Deposition of YSZ nanostructure with three different parameters; deposition RF power sputtering, substrate temperature deposition and post-annealing temperature have been conducted. For the deposition power parameter, optimization was focused on increasing temperature with three condition ranges from 50watt to 150watt. Meanwhile, for the deposition substrate temperature, the optimization were focused on 6 different temperature within 28°C to 500°C. For the flow rate of gas pressure, the optimization was focus on Argon gas flow with 5mTorr per hour. The post-annealing temperature were observed at 600°C and 700°C. The annealing temperature used were higher than deposition temperature because of no changes observed if the annealing temperature same as deposition temperature.

The characterization properties of YSZ nanostructure was measured by XRD, AFM, FESEM, surface profile and 4 point probe. The XRD was conducted to determine the orientation of single crystal or grain, to define the crystal structure and to measure the size and shape of small crystalline regions. AFM was used to explore the surface topology on YSZ nanostructure, and surface profiler measurement is done to measure the thickness of the thin film. The surface morphology and cross-section of YSZ nanostructure was observed using FESEM. The thickness of every sample was measured to study the resistivity in 4 point probe measurement. Electrical properties of YSZ nanostructure were assessed by the I-V measurement type 4-point probe machine which resulted the conductivity and resistivity data of the deposited films.

1.5 Significance of the Study

This study may help other researchers to study the effect of YSZ nanostructure by using RF magnetron sputtering method on electrical, structural and topology characteristics when deposited on aluminium oxide (Al_2O_3) substrate for SOFC application. In addition, this study is fundamentally important to explain the optimum growth parameter for deposition of YSZ thin film using RF Magnetron sputtering in terms of RF power, substrate temperature and annealing temperature. For real application in SOFC devices, a major goal is to produce SOFC that can operate with working temperature below 400°C. An interesting alternative strategy has recently been developed in which reducing the thickness of YSZ nanostructure as electrolyte to enhance the conductivity of the thin film. Hence, this research will help to improve understanding, gain new information and identify the changes of resistivity, conductivity, crystallographic texture, surface morphology, thickness and the porosity of YSZ nanostructure.

1.6 Outline

The general background of the study and brief introduction of YSZ nanostructure and solid oxide fuel cell are discussed in Chapter 1. This is followed by problem statement, objectives, and scope of the study. In Chapter 2, the literature review of the solid oxide fuel cell, YSZ nanostructure as an electrolyte of SOFC and theory of RF magnetron sputtering are presented. Chapter 3 focused on deposition method and characterization technique of YSZ nanostructure by RF magnetron sputtering method. Results and analysis on the structural properties, morphology, thickness and conductivity of YSZ nanostructure are reported in chapter 4. Chapter 5 conclusion of the research and provides a suggestion for future work in this study area.

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