STRUCTURAL CHARACTERIZATION OF YTTRIA STABILIZED ZIRCONIA THIN FILM DEPOSITED BY MAGNETRON SPUTTERING

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DEDICATION

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ABSTRACT

Yttria stabilized zirconia (YSZ) thin film is of great interest as an ion-conductor for the electrolyte-electrode sandwich in solid oxide fuel cell (SOFC) applications. YSZ electrolytes have several advantages, but its applicability has been mainly limited because of the inability to synthesize YSZ films at low temperatures due to its high melting point. One way to overcome such limitation is to establish the YSZ structure by optimizing the percentage of crystallinity and densification of the thin film. The current study was focused on the comparative evaluation of crystallization and densification of dense-thin YSZ film for electrolyte in SOFC. The oxygen flow rate (0-50 sccm), substrate bias voltage (0-120 V), substrate temperature (200-300°C), and deposition time (30-120 min) were evaluated in order to develop dense-thin YSZ film with high crystallinity at low substrate temperatures by radio frequency (RF) magnetron sputtering. The deposition parameter controlled the general morphology and the film thickness, whereas the annealing parameter (300-600°C) affected the crystal orientation in thin films. The current study also determined the effects of the selected deposition parameters on the properties and structures of YSZ thin films. The produced thin films were characterized by glancing angle X-ray diffraction (GAXRD), field emission scanning electron microscopy (FESEM), atomic force microscopy (AFM), Raman spectroscopy, Fourier-transform infrared spectroscopy (FTIR) and transmission electron microscopy (TEM). Based on the results, a dense-thin YSZ film was produced with an average thickness of approximately 200 nm without oxygen flow. The GAXRD pattern of YSZ thin film revealed the existence of a columnar structure (cubic phases) with preferred growth along (200) lattice orientation. YSZ thin films grown at 120 V exhibited good homogeneity and uniformity (100 nm thick and 10-12 nm crystallite size) accompanied by a large microstrain along (111) lattice orientation. The sample obtained at the highest substrate temperature (300°C) revealed the lowest microstrain (0.028%) and the highest crystallinity (43%) with a non-columnar structure. The main effect of the deposition time (60 min) had the strongest effect on the lattice microstrain and the thickness of the YSZ thin film. In conclusion, the combined effects of the substrate temperature (300°C) and annealing factor (400°C) were successful in the development of dense-thin YSZ film with high crystallinity (60%) for potential electrolyte use in SOFCs.

ABSTRAK

Saput tipis Yttria Stabilize Zirconia (YSZ) telah menarik perhatian sebagai konduktor-ion dalam kepingan berapit elektrolit-elektrod untuk kegunaan sel api pepejal oksida. Kelebihan yang ditawarkan elektrolit YSZ selalu terbatas kerana sukar untuk disintesis pada suhu rendah akibat dari suhu takat didih yang tinggi. Bagi mengatasi kekurangan ini, YSZ ini akan disintesis dan dicirikan bagi mendapatkan struktur yang optimim melalui peratus kehomogen dan saput tipis yang padat. Penyelidikan ini fokus kepada penilaian perbandingan kehomogen dan padat saput tipis nanostruktur YSZ sebagai elektrolit. Memanipulasikan parameter percikan magnetron frekuensi radio (RF) termasuk kadar aliran oksigen (0-50 sccm), biasan voltan substrat (0-120 V), suhu substrat (200-300°C) dan masa pengendapan (30-120 minit), membolehkan untuk menghasilkan saput tipis yang padat dan tinggi homogen pada suhu substrat yang rendah. Parameter pertumbuhan mengawal morfologi dan ketebalan saput tipis amnya manakala kesan penyepuhlindapan (300-600°C) mempengaruhi orientasi kristal saput tipis. Kajian terkini mengkaji kesan parameter pertumbuhan terhadap sifat morfologi dan struktur YSZ. Saput tipis yang dihasilkan dicirikan menggunakan peralatan pembelauan sinar X-ray (GAXRD), mikroskop imbasan kesan medan electron (FESEM), mikroskop daya atom (AFM), raman spektroskopi, spektroskopi inframerah transformasi Fourier (FTIR) dan mikroskop elektron penghantaran (TEM). Berdasarkan hasil kajian, sampel padat ketebalan 200 nm diperolehi tanpa aliran oksigen. Paten sudut pembelauan X-ray saput tipis YSZ menunjukkan kewujudan struktur turus (fasa kubik) dengan (200) orientasi kekisi. Saput tipis YSZ yang ditumbuhkan pada 120 V menunjukkan kehomogenan yang baik dan seragam (tebal 100 nm dan saiz kristal dalam lingkungan 10-12 nm) disamping mikroterikan besar sepanjang (111) orietasi kekisi. Sampel pada suhu tertinggi substrat (300°C) menunjukkan mikroterikan paling rendah (0.028%) dan homogen tertinggi (43%) dengan struktur tidak turus. Kesan utama masa pertumbuhan (60 minit) menjadi pengaruh terkuat kepada mikroterikan kekisi dan ketebalan YSZ. Sebagai penutup, kesan penggabungan suhu substrat (300°C) dan faktor penyepuhlindapan (400°C) telah berjaya membina saput tipis YSZ dengan homogen tinggi (60%) dan padat berpotensi sebagai elektrolit dalam SOFC.

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LIST OF ABBREVIATIONS

YSZ	-	Yttia Stabilized Zirconia
UTHM	-	Universiti Tun Hussein Malaysia
DC	-	Direct Current
RF	-	Radio Frequency
Y_2O_3	-	Yttria
GAXRD	-	Glancing Angle X-ray Diffraction
SAED	-	Selected Area Electron Diffraction
SOFC	-	Solid Oxide Fuel Cell
PLD	-	Pulse Laser Deposition
A_{1g}	-	Singly Degenerate (Symmetric)
B_{1g}	-	Singly Degenerate (Anti-Symmetric)
E_g	-	Doubly Degenerate
F_{2g}	-	Triply Degenerate (Symmetric)
NA	-	Not Applicable
FHWM	-	Full width at half maximum intensity
m-YSZ	-	Monoclinic Yttria Stabilized Zirconia
t- YSZ	-	Tetragonal Yttria Stabilized Zirconia
c- YSZ	-	Cubic Yttria Stabilized Zirconia
IR	-	Infra-Red
UV	-	Ultra-Violet
EEDF	-	Electron Energy Distribution Function
TEM	-	Transmission Electron Microscopy
FESEM	-	Field Emission Scanning Electron Microscopy
AFM	-	Atomic Force Microscopy
FTIR	-	Fourier Transform Infrared
EDX	-	Energy Dispersive X-Ray
JCPDS	-	Joint Committee on Powder Diffraction Standards
ICSD	-	International Centre for Diffraction Data
OCV	-	Open Circuit Voltage
FCC	-	Face Centre Cubic

LIST OF SYMBOLS

ε	-	Microstrain
γ	-	Surface energy per unit area
D	-	Crystallite size
d	-	Lattice spacing
V	-	Volt
MHZ	-	Mega hertz
sccm	-	Standard cubic centimetres per minute
W	-	Watt
Å	-	Angstrom
A	-	Area
υ	-	Rayleigh scattering
K	-	Shape factor
GPa	-	Giga pascal
wt%	-	Weight percent
mTorr	-	Millitorr
deg.	-	Degree
a.u.	-	Arbitrary unit
keV	-	Kiloelectron volts
ohm	-	Electrical resistance
mW	-	Milliwatt
MgO	-	Magnesium oxide
O^{2-}	-	Ion oxygen

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

The reversible reaction of the fuel cell was first reported scientifically by Sir William Grove in 1839 [1]. Fifty years later, in 1889, a fuel cell using coal gas as fuel was designed by Charles Langer and Ludwig Mond. The concept of Nernst Diffusion Layer was established by Nernst in 1899, which was later interpreted as Nernst's law for gas-ion transport in electrolytes [2]. The electrolyte, generally known as fast-ion conductors, consists of a highly dense structural material intended to prevent the direct mixing of fuel gas (hydrogen) and oxidising gas (oxygen) [3].

Nowadays, a variety of industrial resources such as coal, oil and natural gas have been wasted with the energy loss ratio of approximately 65% for traditional central power plants and 10% for fuel cells. Despite these advantages, successful attempts were made to assess the potential of electrochemical devices such as solid oxide fuel cells (SOFCs) [3,4]. Recently, considerable progress has been made in the commercial use of low-temperature SOFC thin-film, covering many existing and emerging applications. Much of the previous research on low-temperature SOFCs (LT-SOFCs) have described the role of the solid-oxide crystal structure in facilitating ion movements from one site to another at operating temperatures down to 300-500 °C [5–7].

In general, thin-films with micro- and nano-film electrolyte nanostructures have become increasingly important in the research industry; they have become attractive alternatives to conventional methods, such as powder sieving, compaction, sol-gel, chemical deposition, hydrothermal, photoelectrochemical, liquid phase and extrusion. These corresponding methods are usually impossible to synthesise at low temperatures when compared to direct current (DC) or radiofrequency (RF) magnetron sputtering. The thin-films offer a number of significant advantages and attractions over bulk materials: (i) Thin-film structures are almost always compact, especially when the structure is produced in a non-columnar microstructure that occupies pinhole-free films [8]. (ii) Thin-film is subjected to stress/strain effect, grain growth, microstructure evolution and phase transformation, which influence the bonding between the film and substrate. This factor also leads to the formation of phases, surface microstructures and film thicknesses that are important for electrolyte applications [9]. (iii) The thin-film of a small thickness (100 nm) may have the void of blocking the grain boundary perpendicular to the current flow, thus reducing the contribution of electron conductivity [10]. (iv) Thin-film systems may be adopted by manipulating factors such as ionic defects, concentration, mobility, structural orientation, microstructure, grain boundary and heterostructure interfaces. Therefore, the creation of well-designed nanostructures with two or three dimensions is difficult to achieve in bulk materials [11,12]. (v) Thin-film nanotechnology is safe with a relatively low environmental impact because of its ability to deposit high melting point oxides at low annealing/substrate temperatures and is compact in size [13].

The performance of Yttria Stabilised Zirconia (YSZ) based thin-film electrolyte materials are described in terms of crystallinity (purity) and density (compact or non-columnar). The crystallinity indicates the oxygen vacancy structure or the crystal rate of the desired structure, whereas the density indicates the degree of structure enrichment [14–16]. Furthermore, these pieces of literature have established different theories that describe the initial stages of the YSZ growth mechanism such as the challenge of depositing dense YSZ thin-film and the high melting temperature (2730 °C) for cubic YSZ growth.

To date, the effects of magnetron sputtering deposition have not yet been empirically studied on the growth of YSZ nano-thin film; therefore, the correlation between grain growth and stress strain evolution as an electrolyte has not been reported. The order of the oriented crystal structure clearly affects the mixing of microstructure and conductivity. Besides that, thermodynamics is one of the aspects of consideration. This research aims to determine whether nanocrystalline YSZ structure and nanofilm thickness can be produced using the RF magnetron deposition technique. The Gibbs free energy of formation (G_f), per cent crystallinity, phase identification, morphology and thickness of YSZ were analysed in this study. The findings of this research will help to develop high-performance electrolytes by understanding the relationship among the following factors: structural evolution pattern, percentage of crystallinity, morphology and physicochemical properties.

1.2 Problem Statement

YSZ is a ceramic material that does not have excellent conductivity at low temperatures. Nevertheless, they need a high-temperature operation (900-1000 °C) to maintain their ionic conduction and electrochemical performance. During this operation, electrodes, electrolytes, and interconnectors covering the structure of SOFCs are subjected to physical and chemical modifications that lead to deterioration. Therefore, in order to overcome this issue, SOFCs with high-resistance materials can be easily developed to be operated at low temperatures especially in the presence of thin-film technology. Indeed, YSZ thin-film, which simultaneously has a high value of crystallinity and ionic conductivity, would lead to a thin and dense-thin layer [17–19]. However, this is contrary to the findings of equiaxed and columnar nanostructures from previous [20,21] studies.

Most previous studies [22–25] have considered the YSZ film to have: (1) dense equiaxed grains; (2) dense structure without voids/holes with cubic phases; (3) non-columnar structures without dual phase of YSZ thin-film showed better performance of gas tightness and electrochemical reactions compared to cubic phases with columnar structure. On the other hand, few studies [26,27] indicated that the YSZ film, consisting of columnar grains, can still reveal high conductivity and consistency of cell performance.

Furthermore, the crystal orientation and texture coefficient observed on the YSZ thin-film structure may add significant differences to the overall electrolyte properties and cause a moderate loss in crystallinity. Sometimes, the developed strong (220) preferential orientation is considered to have strong texture (stable microstructure) compared to (111) and (200) orientations [9]. On the other hand, it has been shown that the higher intensity along (111) lattice orientation could lead to the formation of dense, less-defective, and restrained columnar grain as studied by Sønderby et al.[28].

Generally, in YSZ thin-film, the crystallinity of the nano-thin film layer is slightly higher than that of the micro thin-film. However, the development of ultra-thin film electrolytes would result in non-oriented or defective crystal structure due to the irregular packing of atoms and the incomplete coalescence of grain growth, leading to a significant degree of crystallinity. Therefore, the quality of YSZ thin-film depends on the thickness of the entire thin-film and is also directly proportional to microstructure, morphology and texture coefficient [29]. In addition to several analyses, such as per cent crystallinity at (111) orientation, the crystallinity area of the YSZ structure was carried out in an attempt to study and correlate the growth parameter with the YSZ growth evolution. The results from glancing angle X-ray diffraction (GAXRD) showed four intense peaks for different growth parameters, which were desirable from a practical standpoint in the majority of cases. To date, few investigations had been conducted elucidating the relationship between the parameters mentioned above (i.e. oxygen flow rate, substrate bias voltage and substrate temperature) and the structural and morphological properties of YSZ; unfortunately, most of their studies have neglected interaction effects between these (111), (200), (220) and (311) peak correlations.

Hence, this study contributes to our knowledge by addressing three important issues that play a key role in determining the crystallinity of YSZ thin-film. First, the fragile thin-film must properly adhere to the substrate as reported by Jiang and Hertz [26]. Second, the crystal orientation, which had an adverse effect on YSZ grain growth, studied by Hill et al. [24] and Sochugov et al. [30]. Third, the growth rate with optimum thickness resulting from different interdiffusion adhesion, grain growth and texture evolution [31]. Despite the importance of YSZ as ionic transport in a low-temperature solid oxide system, there is still a lack of systematic understanding of how YSZ thin-film grows at low temperatures through the use of

RF magnetron sputtering that contributes to phase transformation, microstructure transformation and structural formation of YSZ.

Moreover, many researchers used the liquid vapour method for the preparation of deposition, such as pulse laser deposition (PLD) and atomic layer deposition (ALD), to produce YSZ nano-thin film as an electrolyte. There is an interesting finding of the successful deposition of RF magnetron sputtering due to its ability to deposit high melting point oxides at relatively low substrate temperatures. So far, the deposition rate using RF magnetron sputtering is very low, and therefore difficult to produce a completed dense-thin film. In this study, RF magnetron sputtering is hypothesised to produce an innovative modification because the vacuum-assisted deposition process technique for depositing a thin-film material atom-by-atom or molecule-by-molecule onto a solid structure can be conducted in a chamber. As a result, the outcome of this research should lead to an understanding of the growth of YSZ phases and the morphological structure of thin-film for electrolyte characterisation.

1.3 **Objectives**

1. To prepare YSZ thin-film using various growth parameters such as oxygen flow rate, substrate temperature, substrate bias voltage, deposition time and annealing temperature.
2. To analyse the structural and morphological parameters of the prepared YSZ thin-film as a function of the growth parameter.
3. To determine the effects of the growth parameter on improving the crystallinity and density of YSZ thin-film for optimisation.

1.4 Scope of the Study

Based on the background of this study, the scope of this study includes:

1. RF magnetron sputtering under various growth parameters, such as oxygen flow rate, substrate bias voltage, substrate temperature and deposition time, was used for YSZ thin-film growth. The post-treatment process (annealing temperature) was carried out between 380 and 600 °C.

2. Fine structural details such as percentage of crystallinity, texture coefficient, micro strain, crystallite size, molecule vibration and chemical bonding of YSZ thin-film were determined using GAXRD, Raman spectroscopy and FTIR. This was followed by the determination of the morphological properties of YSZ thin-film such as thickness, grain size, surface roughness, element distribution and lattice structure using FESEM, AFM and TEM.

3. Optimising the resulting YSZ thin-film by comparing different properties using the percentage of crystallinity and dense structure.

1.5 Significance of Research

The role of phases and structure of YSZ as an electrolyte has been extensively studied. Furthermore, YSZ materials are particularly useful in an environment requiring a high level of solid oxide (O^{2-}) conductor that is strongly affected by the temperature. This could be explained by the ionic conduction that is easier at higher temperatures as ions vibrate more vigorously and the defect concentrations are higher. Nevertheless, the ability of SOFC to operate at low temperatures has increased the interest among researchers to extend its application to operating temperatures down to 300-500 °C. Thin-film nanostructures have been developed in an attempt to optimise the ion conductivity compared to the bulk and membrane structure. Moreover, the results from this research will contribute to the enhanced deposition of YSZ thin-film by identifying the suitable growth parameter and fabrication parameters for improved electrolyte-electrode performance and will also contribute to the development of SOFC that can operate at low temperatures.

1.6 Thesis Organisation

The research outline of this study consists of five chapters. Chapter 1 presents the introduction of the study, the problem statement, the specific objectives, scopes, the significance of the study and the research outline. Chapter 2 summarises the available literature on SOFC, electrolyte, YSZ properties and thin-film growth deposition. Chapter 3 summarises the experiments and the characterisation of the thin-film produced in this study. Chapter 4 presents the results and discussions of the experiments performed. Chapter 5 summarises the conclusions of the work presented in this thesis and the suggestions for future work.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. **N.A. Rusli.,** R. Muhammad., S. K. Ghoshal., H. Nur., & N. Nayan. (2020). Annealing temperature induced improved crystallinity of YSZ thin film. *Materials Research Express,* 7(5). https://doi.org/10.1088/2053-1591/ab9039 **(Q2, IF: 1.41)**.

2. **Ahmad Rusli, Nurhamizah**., Muhammad, Rosnita., Ghoshal, Sib., Nur, Hadi., Nafarizal, Nayan., & Jaafar, Siti. (2019). Bias voltage dependent structure and morphology evolution of magnetron sputtered YSZ thin film: A basic insight. *Materials Research Express*, 6(10). https://doi.org/10.1088/2053-1591/ab3907 2018 **(Q2, IF: 1.449)**.

3. **Nurhamizah, A.R**., Ibrahim, Zuhairi., Rosnita, Muhammad., Yussof, Wahab & Sakrani, Samsudi. (2017). Effect of annealing temperature on platinum/YSZ thin film fabricated using RF and DC Magnetron Sputtering, *Solid State Phenomena*, 268, 229-233. 10.4028/www.scientific.net/SSP.268.229 **(Q3, IF: 0.17)**.

Indexed Conference Proceedings

1. **N.A. Rusli**., Z. Ibrahim., R. Muhammad., Y. Wahab., & S. Sakrani. (2018) Review: Development of platinum electrode for low temperature solid oxide fuel cells. *eProceedings Chemistry*, 3, 5-8, eISSN 2550-145.

Non-Indexed Conference Proceedings

1. **N. A. Rusli**., R. Muhammad., S. K. Ghoshal., & H. Nur. (2017). Effect of oxygen ratio on YSZ thin film deposited using RF magnetron sputtering. NanoMITe Annual Symposium (NMAS 2017) on 14 - 15 November 2017 in UPM Serdang, Malaysia.

2. **N. A. Rusli**., R. Muhammad., S. K. Ghoshal., H. Nur., & N. Nayan. (2019). Annealing Temperature Induced Improved Crystallinity of YSZ Thin Film. 7th International Conference and Workshop on Basic and Applied Sciences (ICOWOBAS 2019) on 19 July 2019 in KSL Resort Johor Bahru, Malaysia.