IMPORVED CHARACTERISTICS OF OPTIMIZED YTTRIA-STABILIZED ZIRCONIA THIN FILM GROWN USING SOL-GEL DIP-COATING TECHNIQUE

NUR FATHIRAH BINTI MOHD RAHIMI

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DEDICATION

To my beloved husband and daughter, for your unconditional love, motivation, support and especially time. We finally go through this together. This is for you both.

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ABSTRACT

Clean and sustainable energy production has become the priority of the 21st century to save Earth's environment from ever-increasing pollution and climate change. Solid Oxide Fuel Cell (SOFC) is one of the emergent technologies that which can produce a vast amount of clean energy using water; thus, considered to be renewable, sustainable, and pollution-free. All previous studies have used high-temperature SOFC electrolyte preparation methods which were expensive and cumbersome. Based on these factors, in this study, good quality yttria-stabilized zirconia (YSZ) electrolyte thin film was synthesizing using soda lime glass and sapphire as a substrate by sol-gel dip-coating method which use low temperature preparation method for YSZ thin film. Several solvents were tested to obtain homogeneous suspension of YSZ such as water, acetone and alcohol and it was found out that alcohol is the suitable one. The samples then were characterized by diverse analytical techniques to determine their potency for the SOFC applications. As-deposited thin films were characterized using X-ray diffractometer (XRD) analyses, atomic force microscopy (AFM), Raman spectroscopy, field emission scanning electron microscopy (FESEM) and Four-point probe techniques. The impact of various substrates, sintering temperatures and layer-count on the structural, morphological and electrical properties of these electrolyte thin films was evaluated. Furthermore, the optimum thin film was chosen to demonstrate its viability as an effective SOFC electrolyte. XRD analysis showed that the YSZ thin films deposited on soda-lime glass and sapphire were highly amorphous and crystalline in nature, respectively. Hence, the sapphire substrate was selected for further deposition of thin films. To improve the overall characteristics of the YSZ films, two types of heat treatments like sintering and normalizing, were performed. The film which had been sintered at 1300 °C revealed the formation of crystallites with lower density than the normalized film. Conversely, the film which had been sintered above 1300 °C showed much better morphology compared to those obtained using the normalized heat treatment. The single-layer YSZ film which had been sintered at 1300 °C exhibited excellent crystallinity, dense morphology with fewer cracks, very low porosity and high electrical conductivity (2.905 S/m). The double-layered YSZ film sintered at 1400 °C displayed the highest conductivity (3.552 S/m) and best crystalline density (5.976 g/cm^3). An increase in the number of layers from three to ten was found to degrade the crystallinity, density and morphology of the grown films. The YSZ electrolyte films deposited with up to three layers were not suitable for practical use in the SOFC. Therefore, the double-layered film which had been sintered at 1400 °C was chosen as the optimum film. The growth of the proposed films was explained using various mechanisms to provide new insight into the preparation of the YSZ electrolyte thin films with tailored properties via the low-cost and simple method at low temperatures. This study focused only on two types of substrates, a few sintering temperatures and one preparation method. However, another synthesis method, substrate and characterization technique such as spin-coating, porous anodic alumina and UV-Visible absorption or emission spectroscopy, respectively, may be useful for better sample optimization. The use of lower temperature for the deposition of the thin films can yield higher electrical conductivity of the electrolyte useful for room temperature operation of SOFC. Based on the results it can be concluded that the interplay of the type of substrate, sintering temperature and the number of coating layers plays a vital role in obtaining high-quality electrolyte thin film, showing that synthesis of the YSZ thin film is essential thus the best sample is established.

ABSTRAK

Pengeluaran tenaga yang bersih dan mampan menjadi keutamaan pada abad ke-21 ini untuk menyelamatkan alam sekitar daripada pencemaran dan perubahan iklim yang semakin meningkat. Sel Fuel Oksida Pepejal (SOFC) ialah salah satu teknologi baru yang boleh menghasilkan sejumlah besar tenaga yang bersih dengan menggunakan air, dengan itu ia dianggap boleh diperbaharui, mampan dan juga bebas pencemaran. Kesemua kajian yang terdahulu menggunakan suhu tinggi dalam kaedah penyediaan elektrolit SOFC yang sangat mahal dan rumit. Berdasarkan faktor-faktor ini, dalam kajian ini, filem nipis elektrolit yttria zirkonia terstabil (YSZ) berkualiti tinggi telah disintesis menggunakan substrat kaca soda-kapur dan batu nilam sebagai substrat dengan kaedah salutan celup dalam sol-gel yang menggunakan suhu rendah untuk filem nipis YSZ. Beberapa pelarut telah diuji untuk mendapatkan ampaian homogen YSZ seperti air, aseton dan alkohol dan telah dikenalpasti bahawa alkohol adalah yang paling sesuai. Sampel kemudiannya dicirikan oleh pelbagai teknik analisis bagi menentukan potensi mereka untuk penggunaan aplikasi SOFC. Filem nipis telah dicirikan menggunakan analisis pembelauan sinar-X (XRD), mikroskopi daya atom (AFM), spektroskopi Raman, mikroskop imbasan pancaran medan elektron (FESEM) dan penduga empat titik. Kesan kepelbagaian substrat, suhu pensinteran dan kiraan lapisan kepada sifat struktur, morfologi dan elektrik filem nipis ini telah dinilai. Tambahan lagi, filem nipis optimum dipilih untuk menunjukkan kelangsungannya sebagai elektrolit SOFC yang berkesan. Analisis XRD menunjukkan bahawa filem YSZ yang dimendapkan pada substrat kaca dan nilam masing-masing adalah sangat amorfus dan berhablur. Oleh itu, substrat nilam telah dipilih untuk pemendapan filem selanjutnya. Bagi menambah baik ciri-ciri keseluruhan filem YSZ, dua jenis rawatan iaitu pensinteran dan penormalan telah dilakukan. Filem yang disinter pada suhu 1300 °C mendedahkan pembentukan hablur dengan ketumpatan yang lebih rendah daripada filem yang dinormalkan. Sebaliknya, filem yang disinter di atas 1300 °C menunjukkan morfologi yang lebih baik berbanding dengan yang diperoleh menggunakan rawatan haba ternormal. Filem YSZ lapisan tunggal yang disinter pada suhu 1300 °C mempamerkan kehabluran yang sangat baik, morfologi yang tumpat dengan retakan yang sedikit, keliangan yang sangat rendah dan kekonduksian elektrik yang tinggi (2.9047 S/m). Dua lapisan filem YSZ yang disinter pada suhu 1400 °C memaparkan kekonduksian tertinggi (3.5516 S/m) dan ketumpatan hablur terbaik (5.976 g/cm^3). Peningkatan bilangan lapisan daripada tiga ke sepuluh lapisan didapati merendahkan kehabluran, ketumpatan dan morfologi filem. Filem elektrolit YSZ yang dimendapkan sebanyak tiga lapisan ke atas tidak sesuai untuk kegunaan praktikal dalam SOFC. Oleh itu, dua lapisan filem yang disinter pada suhu 1400 °C telah dipilih sebagai sampel yang optimum. Pertumbuhan filem telah dijelaskan dengan kepelbagaian mekanisme untuk memberikan pandangan yang baru tentang penyediaan filem nipis elektrolit YSZ dengan sifat yang sesuai melalui kaedah yang berkos rendah dan mudah pada suhu rendah. Walaupun kajian ini hanya tertumpu kepada dua jenis substrat, beberapa suhu pensinteran dan satu kaedah penyediaan, bagaimanapun, kaedah sintesis, substrat dan teknik pencirian yang lain seperti salutan putaran, alumina anodik berliang dan spektroskopi penyerapan atau pelepasan ultralembayung-cahaya nampak, masing-masing mungkin berguna untuk pengoptimuman sampel yang lebih baik. Penggunaan suhu yang lebih rendah untuk filem nipis boleh menghasilkan elektrolit dengan kekonduksian eletrik yang lebih tinggi untuk digunakan dalam SOFC beroperasi suhu bilik. Berdasarkan keputusan ini, ia dapat disimpulkan bahawa interaksi diantara jenis substrat, suhu pensinteran dan bilangan lapisan salutan memainkan peranan penting dalam mendapatkan filem nipis elektrolit berkualiti tinggi lantas menunjukkan bahawa sintesis filem nipis YSZ adalah penting justeru itu sampel yang terbaik telah dibuktikan.

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LIST OF ABBREVIATIONS

SOFC	-	Solid Oxide Fuel Cell
YSZ	-	Yttria Stabilized Zirconia
XRD	-	X-ray Diffractometer
AFM	-	Atomic Force Microscopy
FESEM	-	Field Emission Scanning Electron Microscopy
UV-Visible	-	Ultra Violet Visible
UTM	-	Universiti Teknologi Malaysia
SDGs	-	Sustainable Development Goals
Y_2O_3	-	Yttria Oxide
RAMAN	-	Raman Spectroscopy
NO_x	-	Nitrogen Oxide
CO_2	-	Carbon Dioxide
PEMFC	-	Proton Exchange Membrane Fuel Cell
DMFC	-	Direct Methanol Fuel Cell
MCFC	-	Molten Carbonate Fuel Cell
EPD	-	Electrophoretic Deposition
NiO	-	Nickel Oxide
LSC	-	Lanthanum Strontium Cobaltite
LSM	-	Lanthanum Strontium Manganite
GDC	-	Gadolinium deposited Ceria
Ni-YSZ	-	Nickel-Yttria Stabilized Zirconia
TEC	-	Thermal Expansion Coefficient
ORR	-	Oxygen Reduction Reaction
Al_2O_3	-	Aluminium Oxide
SiO_4	-	Silicate

LIST OF SYMBOLS

MW	-	Megawatt
kW	-	Kilowatt
°C	-	Celsius
W	-	Watt
E, e	-	Electron
f	-	Lattice mismatch
a_s	-	the unstrained lattice constant of the substrate
a_f	-	unstrained lattice constant of the film
M	-	Metal
R	-	An alkyl group
X	-	Valence state of the metal
XOH	-	Hydroxylated compounds
V	-	Volt
g	-	Gram
cm	-	Centimetre
nm	-	Nanometre
m	-	Meter
µm	-	Micrometre
Mpa	-	Megapascal
Psi	-	Pounds per square inch
K	-	Kelvin
ŋ	-	Refractive Index
ρ	-	Density
k	-	Thermal Conductivity
C_P	-	Specific Heat
LCA	-	Life Cycle Assessment

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CHAPTER 1

INTRODUCTION

1.1 Background

A thin film is a low-dimensional substance made by condensing atomic, molecular, or ionic species of matter one at a time. A thin film's thickness is usually less than a few microns. For more than half a century, thin films have been used in electrical appliances, hard coatings industry, optical coatings, and aesthetic components. Thin films are still being made on a regular basis these days, as they are an important component in the creation of novel materials in the twenty-first century, such as nanometre materials. Since the quantity utilized is restricted to only the surface or as a thin film layer, the materials and equipment necessary for making thin films with the least amount of hazardous ingredients are accessible. Thin film processing can also minimise energy usage during production, making it an ecologically friendly material technology for the twenty-first century (Wasa *et al.*, 2004).

Thermal evaporation, chemical breakdown, or irradiation of energetic particles or photons are all methods for depositing thin films on a substrate. Film structure, crystal phase and orientation, thickness, and microstructure are all important variables to consider. All of these properties can be controlled by deposition conditions. Other than these, thin film acquires several unique properties compared to bulk materials, such as the unique properties of materials from the atomic growth process and the size of the effect can be characterized by the thickness, orientation of crystallization and multilayer characteristics (Wasa *et al.*, 2004).

Thin film technology can be used in energy applications like fuel cells and solar cells. It is needed in improving the usefulness of the cells, reducing working temperature and expanding their life span. Fuel cells produce electrical energy from chemical energy. They generate energy through the oxidation of natural gas, hydrogen or other fuels. They can operate continuously as long as they can access to the supply such as hydrogen and oxygen. Fuel cell applications may be divided into three categories: portable power production, fixed power generation, and transportation power. There are many different types of fuel cells, but they always include an anode, a cathode, and an electrolyte that permits positively charged hydrogen ions to travel between them. Solid Oxide Fuel Cells (SOFCs) is a major area of interest within the field of fuel cells. SOFCs use a solid ceramic oxide as an electrolyte. SOFCs operate at a high operating temperature of around 800 °C to 1000 °C. Current research tends to focus on lowering the operating temperature so that SOFCs can also be applied to suitable portable devices such as for transportation or mobile devices. Usually, power generation for residential use is around 1-10kW, while for commercial use, it is around 2-250MW (Choudhury *et al.*, 2013). However, by decreasing the operating temperature, the electrolyte ohmic resistance losses will increase due to the thermally activated behaviour of ionic transport (Litzelman *et al.*, 2008). Introducing thin electrolyte layers into SOFCs can decrease the internal cell resistance and thus drastically enhance the power density (Menzler *et al.*, 2013).

This SOFC research has been using thin film for a long time since this technology is the most suitable for SOFC application. William Groove introduced the fuel cell idea for the first time in 1893. Grove was studying the electrolysis of water when he noticed that when the current was turned off, a small current continued to flow through the circuit in the opposite direction as a result of a reaction between the electrolysis products, hydrogen and oxygen, which was catalysed by the platinum electrodes. Grove also made the critically important insight that there must be a "notable surface of action" between the gas, the electrode, and the electrolyte phases in a cell. Grove recognised the idea of connecting several of these in series to create a gaseous voltaic battery. Maximizing the "triple-phase boundary," or the area of contact between the electrode, electrolyte, and gaseous reagent, continues to be at the forefront of fuel cell research and development (Ormerod, 2003)

The choice of YSZ as the material of electrolyte based on its high ionic conductivity material, stable in both oxidizing and reducing environments and good high temperature mechanical properties (Jacobson, 2010).

Ultimately, we are targeting to contribute by giving ideas in relation to solid oxide fuel cells, to achieve high-efficiency cells to make it applicable in people's daily lives. By achieving this, energy would be saved at a time when the climate is changing and global energy demand is continuously increasing. Thus, we have undertaken the task by increasing the output performance of SOFC electrolyte thin films.

On 25 September 2015, United Nations Member States met at the United Nations in New York and adopted the 2030 Agenda for Sustainable Development. These 17 Sustainable Development Goals (SDGs) set out an agenda for sustainable development, peace and prosperity for all people and the planet, for now and for a future that embraces growth in the economy and social inclusion while protecting the environment. Some issues supported in the SDGs were water accessibility, energy security, urbanization, transportation and climate crisis. Out of 17 SDGs, this research had targeted 4 goals and would be able to directly contribute to 2 goals. They were Goal 7: Affordable and Clean Energy and Goal 13: Climate Action. Another 2 goals which we have indirectly focused on were Goal 3: Good Health and Well-being and Goal 11: Sustainable Cities and Communities (Kroll *et al.*, 2019).

1.2 Problem Statement

During the last two decades, with the energy crisis and renewable energy development, SOFC materials have emerged as a starting material on the horizon of material science to overcome the problem of environmental pollution resulting from fossil fuel burning and excessive use of non-renewable energy such as from fossil fuel, crude oil and natural gas. Extensive research has been performed to find the optimum YSZ material for making the SOFC electrolyte (da Silva and de Souza, 2017; Abdalla *et al.*, 2018; Zakaria *et al.*, 2020). Various researchers have produced SOFC thin films mostly with high-temperature processing (more than 100 °C), but only a few reports exist on low-temperature processing (room temperature) (Ramesh *et al.*, 2016; Son *et al.*, 2018). However, the quality of the grown film depends on the optimum thickness, structural and morphological properties. To determine the effect of optimum growth parameters on these properties' thorough and careful characterization and preparation of the samples are required.

Several studies have been performed to determine the effect of sintering temperature and sol-gel processing parameters using different methods. However, it has been found that growth temperature is the most crucial and significantly affects the layer properties and the layer thickness. Based on these facts, it has been found that most of the techniques had used high temperature and had required a lot of other processing parameters which makes it more expensive and complicated compared to the low-temperature processing method, where samples prepared by sol-gel method can be used to achieve better properties (Lee *et al.*, 2016; Ma *et al.*, 2016; Venkataramana *et al.*, 2017). One of the advantages of the sol-gel method compared to other methods is that densification can easily be achieved at low temperatures. As an inexpensive process, chemical composition of the product can be controlled. A small amount of base material can be applied to the solution and can take part in affecting the final result (Lee *et al.*, 2016). However, the regularity of the thin film structure, size of the particle and morphology of the particle distribution has not yet been clarified. In this view, the present study attempted to overcome the limitation of past studies to try to improve the properties by making a high-quality film for SOFC electrolyte application.

The requirement for low temperature during thin films deposition is needed to obtain high electrical conductivity of the electrolyte so that SOFC can be operated at room temperature since there has been increasing concern that SOFCs are being disadvantaged when operating at high temperature due to its start-up time, materials demand and application being limited to only large stationary sources (Timurkutluk *et al.*, 2016).

It is important to establish the correlation among the low-temperature processing, environmental friendliness and the cost-effectiveness of this sol-gel grown thin film. Therefore, it was important for the SOFC application that any material must meet the environmental sustainability criteria, especially since the SOFC material was for the renewable energy sector. As per this rationale, the present research work attempted to establish this yet-to-be established correlation.

1.3 Research Objectives

This research had three objectives, which were as follows:

(a) To synthesis YSZ thin films in soda lime glass and sapphire substrates using sol-gel dip-coating technique;

(b) To determine the effects of growth parameters such as substrates, coating layer numbers and sintering temperatures on the modified structural, morphological and electrical characteristics of YSZ thin films;

(c) To optimize the growth parameters of YSZ thin films via structural, morphological and electrical characterizations useful for SOFC applications.

1.4 Scope of Study

The study used the method of sol-gel by dip-coating of YSZ electrolyte thin films. Growth parameters for the experiment included layers of coating (1 to 10 layers) and sintering temperature (300 °C to 1300 °C). The time of coating (60 seconds) and speed of coating (150 mm/min) remained constant.

The solid electrolyte material had been chosen due to the general criteria, including strong ionic conductivity, low electronic conductivity, oxidizing and reducing environment durability, and excellent mechanical properties (Jacobson, 2010; Biswas and Sadanala, 2013). Zirconia-based oxide ion fulfilled the criteria mentioned above. Addition of rare earth, alkaline earth and lanthanide oxides could stabilize monoclinic zirconia to cubic fluorite phase at room temperature. The ionic conductivity also increases the oxygen partial pressure range during the SOFC operation. One of the oxides which needed to be used as a dopant for zirconia was yttria (Y_2O_3) (Figueiredo and Marques, 2013). Yttria-Stabilized Zirconia (YSZ) has a wide temperature range starting from room temperature until 2300 °C to achieve stability. In this study, special attention was given to the characteristic of the electrolyte thin film. Dense and low-porosity thin film is very important for ionic transportation when electrons transfer from anode to cathode. The stabilization of the cubic phase was needed to achieve very high ionic conductivity. The solid solution of cubic form induces the presence of oxygen vacancies according to the substitution of Zr^{4+} and Y^{3+} in the cationic network (Hidalgo *et al.*, 2011; Figueiredo and Marques, 2013).

Sapphire substrate was chosen in this research due to its nearest lattice mismatched between the substrate and thin film. YSZ lattice constant is 5.12 while sapphire is a: 4.785, c: 12.991. Additionally, to produce high crystallinity YSZ film, a high sintering temperature is needed. Sapphire substrate melting point can be up to 2040 °C. Glass substrate was used to test and compare the results. However, glass substrate can only withstand up to 700 °C which is not sufficient for YSZ thin film to achieve crystallinity. The dip-coating method is the simplest method to produce a thin film. Apart from that, it is easier to control the thickness of the film and other growth parameters. Time (60 seconds) and speed (150 mm/min) of coating remained unchanged. For growth parameters, the layer of coating (1 to 10 layers) and sintering temperature (1000 °C to 1500 °C) (Zhou *et al.*, 2014; Jang *et al.*, 2016) were discussed. The sintering temperature and coating layers is a very important parameters in determining a suitable temperature to produce high-quality YSZ thin film.

The properties of each component were to be characterized using X-ray Diffractometer (XRD) and field emission scanning electron microscope (FESEM) for the phase and structure of samples. Field Emission Scanning Electron Microscope (FESEM), Raman Spectroscopy (RAMAN) and atomic force microscope (AFM) were to be used for morphological characterization. The 4-point probe was to be used for conductivity and resistivity of electrolyte thin film as an application in SOFC.

Structural and electrical properties are important in producing YSZ thin film. From structural properties we can identify crystallization of thin film, phase structure and crystallite size. Morphologies of thin film would tell whether the thin film is dense as well as whether it had no-crack properties and pores. All these properties would help to get high conductivity and less resistivity in terms of electrical properties.

1.5 Significance of Study

The findings of this study may help other researchers when investigating the effects of Yttria-Stabilized Zirconia thin film using the sol-gel method in term of electrical, structural and morphological characteristics when deposited on the sapphire wafer and soda lime glass for the solid oxide fuel cell application. This study could also contribute to the improvement and development of low pollution and clean technology. The technique used in this study which is sol-gel dip-coating method is one of the simplest and low-cost method to fabricate thin film thus can contribute in cost-effectiveness of the sample. The use of thin film in this study also supports the increasing interest in nanotechnology. In the present, the thin film is useful for small and portable devices. So, this research could help researchers to improve the performance of SOFC electrolyte thin films. This study will also assist to gather new understanding and important to discover variations in resistivity, conductivity, crystal structure, morphologies, thickness, and porosity of a YSZ electrolyte useful for SOFC applications.

1.6 Outline of Thesis

This thesis explains the preparation and synthesis of growth YSZ thin film on soda lime glass and sapphire substrate, characterization and analysis of various properties of the thin film. The simple and less-cost wet deposition, which was the sol-gel dip-coating method, was used to prepare the thin film. The sintering heat treatment process was applied to produce growth thin film. The optimization of the YSZ thin film finally were determined based on the analysis given.

Chapter 1 presents an introduction and the background of the current study. The problem statement, objectives, scope, limitations of the study and significance of the study have been discussed in this section.

Chapter 2 explains relevant literature regarding SOFC applications including type of SOFC, components of SOFC, thin film technologies with growth mechanism explanation and technique used and lastly the mechanism of characterization used to analyse thin film.

Chapter 3 describes the experimental procedure to prepare and to synthesize the YSZ thin film. In addition, information regarding the characterization of the thin film based on its structural and electrical properties have been emphasized.

Chapter 4 highlights all the results obtained from the comparison test of the different substrates and choosing a suitable one. The growth parameter in terms of sintering temperature and different coating layers were chosen. The optimum result from the parameter was determined by characterization analysis using XRD, AFM, and Raman for structural properties, FESEM was used to study morphology and a four-point probe was used to investigate the electrical properties.

Chapter 5 concludes the entire thesis that has been carried out to fulfil the objectives. The most optimized YSZ thin film electrolyte that could be used in SOFC application was prepared. By using the less-cost method, the production cost of the SOFC could be controlled, which is very useful as SOFC needs to be widely used and well-known in the future for better sustainable energy in line with protecting our environment with increased use of green energy.

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LIST OF PUBLICATIONS

Indexed Journal

(a) N.F.M. Rahimi, Sathiabama T. Thirugnana, S. K. Goshal. (2020). Yttria Stabilized Zirconia Thin Film as Solid Oxide Fuel Cell Electrolyte: Temperature Dependent Structures and Morphology. Journal of Environmental Treatment Techniques. 8(2), 604-609.

Indexed Conference Proceedings

(b) N.F.M. Rahimi, Y. Wahab, R. Muhammad. (2017). Morphological of Yttrium Stabilized Zirconia (YSZ) Thin Film of Electrolyte in Solid Oxide Fuel Cell Application. In 2016 Regional Conference on Solid State and Technology RCSSST 2016. Solid State Phenomena, 268, (pp: 343-346). Doi: 10.4028/www.scientific.net/SSP/268.343

(c) N.F.M. Rahimi, Sathiabama T. Thirugnana, S.K. Goshal, R. Muhammad. (2021). Varied Layer Thickness Improves Structural Properties of YSZ Thin Film. 1-6. Doi: 10.1177/23977914211015854