SINGLE STEP CO-EXTRUDED TRIPLE LAYER MICRO-TUBULAR SOLID OXIDE FUEL CELL

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ABSTRACT

Fabrication of micro-tubular solid oxide fuel cells (MT-SOFC) typically involves multiple and repetitive steps of deposition and sintering. This study aimed to develop triple-layer hollow fiber (TLHF) of MT-SOFC in a single-step fabrication, as an effort to simplify the complexity of the conventional technique. Phase inversion-based co-extrusion/co-sintering technique was employed in this study to simultaneously fabricate triple-layer anode/electrolyte/cathode hollow fiber based MT-SOFC with a desired structure. In order to overcome the challenges during the fabrication process due to the different sintering behaviors of each layer, the modification of the cathode layer had been conducted in the first stage of this study by analyzing the compatibility of different cathode materials (lanthanum strontium manganite (LSM); lanthanum strontium cobalt ferrite (LSCF)) with electrolyte material, yttria-stabilized zirconia (YSZ) at varied sintering temperature of 1250~1450 °C. Composite LSM-YSZ cathode hollow fiber had higher chemical compatibility at optimum sintering temperature of 1400 °C, in comparison to LSCF-YSZ cathode. In the second stage of the study, the effect of co-extrusion/co-sintering parameters on TLHF fabricated, namely co-sintering temperature (1300~1450 °C) and cathode extrusion rate (3~6 ml min^{-1}), were investigated. The fabrication of TLHF via this advanced technique was optimized at 1450 °C of co-sintering temperature with 6 ml min^{-1} of cathode extrusion rate. Additionally, the usage of micron size YSZ at cathode site and rapid sintering rate of 10 °C min^{-1} are good approaches to ensure the success of the fabrication process of cathode together with anode and electrolyte; yielding 0.75 Wcm^{-2} of power output at 800 °C. As the co-sintering of triple-layer together might cause the densification of cathode, thus, addition of different graphite loading (2.5~15 wt.%) in cathode suspension was performed in the third stage of the study, in order to further improve the cathode structure. It was discovered that 2.5 wt.% of graphite loading enhanced the cathode structure with the generation of fine microscopic pores which were uniformly distributed throughout the cathode site. The addition of graphite, however, had reduced the size of the $TLHF_{gr}$ (with the presence of graphite). Thus, 8~16 ml min^{-1} of bore fluid flow rate was examined to improve the $TLHF_{gr}$ structure; and 12 ml min^{-1} was found to be the most suitable bore fluid flow rate in fabricating $TLHF_{gr}$. Afterward, the stability analysis of TLFH and $TLHF_{gr}$ for 24 hrs of operation at 800 °C had been conducted in the fourth stage of the study. The result revealed that TLHF displayed a stable performance throughout the operation. Along with that, comparative analysis between MT-SOFC fabricated via singe-step preparation of phase inversion-based co-extrusion/co-sintering technique; and multiple-step preparation of phase inversion-based co-extrusion of anode/electrolyte and cathode brush-painting technique, was examined. As a result, the novel technique of single-step preparation of phase inversion-based co-extrusion/co-sintering was found to be able to offer greater performance of 1.46 W cm^{-2} with 1.08 V OCV at 800 °C; as well as desired TLHF MT-SOFC quality of asymmetric anode (sharp-thin finger-like void and sponge-like void), thin and dense electrolyte, and porous cathode with strong adhesion bond between the layers, compared to the conventional multiple-step preparation technique (0.09 W cm^{-2}, 0.57 V OCV). The utilization of this new technique brought an end to cathode delamination and unsteady cathode thickness problems caused by conventional cathode deposition technique.

ABSTRAK

Fabrikasi tiub-mikro sel bahan api pepejal teroksida (MT-SOFC) biasanya melibatkan beberapa langkah pemendapan dan pensinteran berbilang dan berulang. Kajian ini bertujuan untuk membangunkan gentian berongga tiga lapisan (TLHF) MT-SOFC dalam satu langkah fabrikasi, sebagai usaha untuk mempermudah kerumitan teknik konvensional. Teknik penyemperitan/pensinteran berasaskan penyongsangan fasa digunakan dalam kajian ini untuk menghasilkan TLHF anod/elektrolit/katod secara serentak yang berasaskan MT-SOFC dengan struktur yang dikehendaki. Untuk mengatasi cabaran sewaktu proses fabrikasi yang disebabkan oleh ketidaksamaan kelakuan pensinteran pada setiap lapisan, pengubahsuaian lapisan katod telah dilakukan pada peringkat pertama kajian ini dengan menganalisis keserasian bahan katod yang berbeza (lanthanum strontium manganit (LSM); lanthanum strontium kobalt ferit (LSCF)) dengan bahan elektrolit, zirkonia stabil yttria (YSZ) pada suhu pensinteran bervariasi 1250~1450 °C. Gentian berongga katod komposit LSM-YSZ mempunyai keserasian secara kimia yang tinggi pada suhu pensinteran optimum, 1400 °C, berbanding LSCF/YSZ. Pada peringkat kedua kajian, pengaruh parameter ko-penyemperitan/ko-pensinteran terhadap TLHF yang difabrikasi, iaitu suhu pensinteran (1300~1450 °C) dan kadar penyemperitan katod (3~6 ml min^{-1}), disiasat. Pembuatan TLHF melalui teknik canggih ini dioptimumkan pada suhu pensinteran 1450 °C bersama dengan kadar ko-penyemperitan katod 6 ml min^{-1}. Selain itu, penggunaan bahan YSZ bersaiz mikron di tapak katod; dan kadar pensinteran cepat iaitu 10 ° C min^{-1} menjadi pendekatan yang baik untuk memastikan kejayaan proses fabrikasi katod bersama-sama dengan anod dan elektrolit; menghasilkan 0.75 Wcm^{-2} kuasa pada 800 °C. Proses ko-pensinteran tiga-lapisan mungkin menyebabkan kepadatan lapisan katod, oleh itu, penambahan muatan grafit yang berbeza (2.5~15 wt.%) pada ampaian katod dilakukan pada peringkat ketiga kajian, untuk memperbaiki lagi struktur katod. Didapati bahawa 2.5 wt.% muatan grafit, meningkatkan struktur katod dengan penghasilan pori-pori mikroskopik halus yang terbentuk secara seragam pada seluruh katod. Penambahan grafit, bagaimanapun, telah mengurangkan saiz $TLHF_{gr}$ (dengan kehadiran grafit). Oleh itu, kadar aliran cecair penggerak iaitu 8~16 ml min^{-1} diperiksa untuk memperbaiki struktur $TLHF_{gr}$; dan kadar aliran cecair penggerak 12 ml min^{-1} didapati sesuai dalam pembuatan $TLHF_{gr}$. Selepas itu, analisis kestabilan TLFH dan $TLHF_{gr}$ selama 24 jam operasi pada suhu 800 °C telah dilakukan pada peringkat keempat kajian. Hasil kajian menunjukkan bahawa TLHF menunjukkan prestasi yang stabil sepanjang operasi. Bersamaan dengan itu, analisis perbandingan antara MT-SOFC yang dibuat melalui satu langkah penyediaan iaitu teknik ko-penyemperitan/ko-pensinteran berasaskan penyongsangan fasa; dan pelbagai langkah penyediaan iaitu teknik ko-penyemperitan anod/elektrolit berasaskan penyongsangan fasa ditambah dengan teknik salutan lapisan katod melalui teknik melukis menggunakan berus, diperiksa. Hasilnya, teknik baharu melalui satu langkah penyediaan terhadap ko-penyemperitan/ko-pensinteran bersama berasaskan fasa penyongsangan menawarkan prestasi MT-SOFC yang cemerlang sebanyak 1.46 W cm^{-2} dengan 1.08 V OCV pada suhu 800 °C; serta kualiti TLHF MT-SOFC yang diinginkan iaitu struktur anod yang tidak simetri (struktur seperti jari nipis tajam dan struktur seperti span), elektrolit nipis dan padat, katod berliang dengan ikatan lekatan kuat yang tercipta di antara lapisan, berbanding dengan teknik penyediaan pelbagai langkah konvensional (0.09 W cm^{-2}, 0.57 V OCV). Penggunaan teknik baharu ini mengakhiri masalah penyingkiran lapisan katod dan masalah ketebalan katod yang tidak stabil disebabkan oleh teknik salutan katod secara konvensional.

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LIST OF ABBREVIATIONS

ASR	Area-specific resistance
DLHF	Dual-layer hollow fiber
$DLHF_{gr}$	Dual-layer hollow fiber with graphite addition
EDS	Energy dispersive X-ray spectroscopy
HF	Hollow fiber
$La_{0.8} Sr_{0.13} Mn O_3$	Lanthanum strontium manganese oxide
$La_2 Zr_2 O_{7,}$	Lanthanum zirconium oxide
LSCF	Lanthanum strontium cobalt ferrite
LSM	Lanthanum strontium manganite
MT	Micro-tubular
NiO	Nickel Oxide
NMP	N-methyl-2-pyrrolidone
PESf	Polyethersulfone
SEM	Scanning electron microscopy
SLHF	Single-layer hollow fiber
SOFC	Solid oxide fuel cell
Sr Fe O	Strontium iron oxide
$Sr Zr O_3$	Strontium zirconate
TEC	Thermal expansion coefficient
TLHF	Triple-layer hollow fiber
$TLHF_{gr}$	Triple-layer hollow fiber with graphite addition
TPB	Triple phase boundary
XRD	X-ray diffractometer (XRD)
YSZ	Yttria-stabilized zirconia

LIST OF SYMBOLS

%	Percentage
°C	Degree Celsius
µm	Micrometer
A	Area of hollow fiber
cm	Centimeter
cp	Centipoise
D_i	Inner diameter
D_O	Outer diameter
hr	Hour
J	Joule
L	Length of hollow fiber
m	Meter
mm	Millimeter
N	Load
P	Gas permeability
Pa	Pascal
P_a	Atmospheric pressure
P_i	Final pressure
P_o	Initial pressure
R	Constant
W	Watt
Wt. %	Weight percentage

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CHAPTER 1

INTRODUCTION

1.1 Research Background

World is moving toward renewable resources as their main source of energy. The dependency on fossil fuel as our energy resources is depleted from day to day because of awaken globally that against the pollution bring by the combustion of fossil fuels, coal, natural gas and oil. The effect of that pollution not only affects the human being but it actually gives the bad impact to the overall ecosystem. Moreover, it was reported that the increasing in carbon dioxide (CO_2) content in our atmosphere being the main cause of global warming [1]. Therefore, the topic of shifting energy resources is a controversial one among experts. It has opened up the possibility for researchers to explore and investigate more about alternative energy sources that can solve or reduce the problem that arises. Thereupon, fuel cell technology can be seen as one of the excellent alternatives to this issue.

Commonly, there are several types of fuel cell as a function of its electrolyte classification as shown as Table 1.1. This classification is usually based on its electrolyte type and its operating temperature. Alkaline fuel cell (AFC), phosphoric acid fuel cell (PAFC) and molten carbonated fuel cell (MCFC) are classified under liquid type electrolyte; while proton exchange membrane fuel cell (PEMF), direct methanol fuel cell (DMFC) and solid oxide fuel cell (SOFC) are using solid type electrolyte made from polymer or ceramic membranes. Furthermore, MCFC and SOFC required high operating temperature compared to others type of fuel cell that usually run at low operating temperature.

Table 1.1 Type of Fuel Cells (U.S Department of Energy) [2]

Name	Electrolyte	Anode gas	Cathode gas	Temperature	State
AFC Alkaline Fuel Cell	Potash	Hydrogen	Oxygen	Below 80 °C	Commercial
PEMFC Proton Exchange Membrane Fuel Cell	Polymer Membrane	Hydrogen	Oxygen or Atmospheric Oxygen	to 120 °C	Being developed
DMFC Direct Methanol Fuel Cell	Polymer Membrane	Methanol	Atmospheric Oxygen	90 – 120 °C	Being developed
PAFC Phosphoric Acid Fuel Cell	Phosphorus	Hydrogen	Atmospheric Oxygen	200 °C	Commercial
MCFC Molten Carbonated Fuel Cell	Alkali-carbonates	Hydrogen Methane	Atmospheric Oxygen	650 °C	Being developed
SOFC Solid Oxide Fuel Cell	Ceramic Oxide	Hydrogen Methane	Atmospheric Oxygen	500 – 1000 °C	Being developed

SOFC that made from ceramic membranes and operates at high temperature had exhibited various advantages over other types of fuel cell. By using solid electrolyte, this type of fuel cell could be easy to maintain as it can avoid corrosion and the catalyst wetting problem. Besides, high operating temperature allows the fuels that fed into SOFC system to internal reforms and oxidized without any involvement of expensive external reformer. As well as its internal reforming, this type of fuel cell leads to the many advantages available on its system such as high energy conversion efficiency, low emissions and excellent in fuel flexibility.

Basically, SOFC composes of three main components which are anode, electrolyte and cathode. Each of the component has their own roles in the SOFC system. In term of its configuration, SOFC presents either in planar or tubular geometry design. Kendall is the first pioneer that introduces the micro-tubular solid oxide fuel cell (MT-SOFC), in the early 1990s [3]. MT-SOFC are much smaller in the size of millimeter scale compared to the ten centimeter scale of typical tubular SOFC. When compare this both geometry designs, tubular design having an advantage in better thermal-cycling due to of easiness on sealing gas. Besides, this small size SOFC having ability on quicker start-up, tolerance to thermal cycling, giving higher volumetric output density and portability compared to planar and conventional tubular SOFC [4].

Conventionally, the fabrication of a complete fuel cell of micro-tubular design which consists of anode, electrolyte, and cathode components will take time and costly because of the multiple processes involved [5]. The common techniques that have been employed to fabricate the micro-tubular fiber are including the plastic mass ram extrusion and dry-jet wet extrusion. These both techniques are only used to fabricate a single-layer of support, and thus, still require a multi-step in order to develop a complete fuel cell. By using dry-jet wet or ram extrusions, a support layer, for example anode tube is first prepared and pre-sintered to provide mechanical strength to the fuel cell. Then deposition of the electrolyte layer takes place and sintered prior to the final coating of cathode layer. Each step involves at least one high temperature heat treatment, making the cell fabrication time-consuming and costly. In terms of membrane fabricated, the adhesion bond between the layers may difficult to be achieved with unstable control over cell quality.

To date, the production of this MT-SOFC will involve multiple deposition and sintering steps. Co-extrusion technique that firstly introduced for fabricating polymeric dual-layer membrane about 30 years ago [6] give an idea for the fabrication of ceramic membrane. This technique offers an attractive prevalence which is saving the production cost and time, reducing a defect rate on the fabricated membrane and promote a great adhesion between layers. After all, until 2017, the progress of MT SOFC hollow fiber's fabrication via phase inversion-based co-extrusion is more focused on fabricating dual layer hollow fiber [7, 8, 9, 10]. After all, very limited work has been done to fabricate triple-layer hollow fiber (TLHF) composed of anode, electrolyte and cathode using the co-extrusion technique. Therefore, the introduction of phase inversion-based co-extrusion/co-sintering fabrication technique could be the best solution to the deficiency of the conventional fabrication method, in order to fabricate anode/electrolyte/cathode MT-SOFC at the same time for high temperature application

1.2 Problem Statement

Phase inversion-based co-extrusion has been introduced to enhance the conventional method of fabricating triple-layer MT-SOFC. Commonly, the anode/electrolyte dual-layer hollow fiber fabricated will be sintered at high temperatures before being deposited by cathode layer as a final layer. The deposited cathode layer will be subjected to another sintering process to solidify the final layer of hollow fiber. There are several techniques that are usually applied to deposit a cathode layer onto an anode/electrolyte hollow fiber, including brush painting [11,12] and dip-coating [13,14]. In most cases, multiple layers of cathode are deposited on the electrolyte surface in order to achieve the desired thickness of the cathode layer. Each cathode layer will involve one sintering step at high temperature. Thus, the process of completing an anode/electrolyte/cathode MT SOFC requires additional effort and time. In fact, this is not a simple or short process to fabricate a single complete cell of MT-SOFC.

To the extent of our knowledge, there is only few studies conducted on TLHF based MT-SOFC fabricated by phase inversion based co-extrusion/co-sintering technique. This technique had been employed to fabricate symmetric CGO electrolyte layer, symmetric anode functional layer and asymmetric CGO-Ni anode layer [15]. While, in year 2018, a complete set of CGO intermediate temperature-SOFC also had been produced using the same technique [16]. The power output of 0.48 W cm^{-2} had been recorded in this study at temperature of 525 °C. The low power output resulted from immature electrode structure and insufficient electrolyte thickness, which caused some gas leakage. Over-densified lanthanum strontium cobalt ferrite LSCF cathode due to high sintering temperature of 1450 °C had impaired the MT-SOFC performance result. Yet, no much result has been discussed and instead, the attempt applied was limited in the intermediate temperature application SOFC. In a word, this simplified and time saving technique of phase inversion based co-extrusion/co-sintering technique will be used and systematically investigated in the proposed study to produce a novel cathode/electrolyte/anode TLHF in a single step especially for high temperature SOFC.

However, there is a challenge during the fabrication of this TLHF due to the different sintering behavior between the triple-layer that can lead to the defect. The structure of the SOFC must be porous on the electrode while dense on the electrolyte. An anode that consists of nickel oxide (NiO) need to be sintered at high temperature, normally at 1400 °C in order to enhance the interfacial bonding between NiO and yttria-stabilized zirconia (YSZ) as well as maintain its high electronic conductivity [17]. While, 1350 °C of sintering temperature is needed for YSZ to achieve 95% of theoretical density [18], which very essential for electrolyte gas-tightness property Since, this novel technique will involve the sintering of anode, electrolyte and cathode at the same time. Hence, high temperature of co-sinter may be beneficial for both anode and electrolyte, while cathode might be become less porous due to the high temperature of co-sinter.

Seeing that lanthanum strontium manganite (LSM) and lanthanum strontium cobalt ferrite (LSCF) are basic cathode materials that widely used in YSZ electrolyte-based SOFC [19, 20, 21, 22, 23, 24] and having its own advantages. Thus, the selection of cathode material needs to be conducted first to ensure the compatibility and stability of those materials (LSM; LSCF) with electrolyte material, specifically under high co-sintering temperature application. The suitable cathode material will be used for the next development of TLHF MT-SOFC. Furthermore, as this study employs a novel technique of phase inversion based co-extrusion/co-sintering, further investigation is needed in term of its co-extrusion/co-sintering parameters, so that each layer of MT-SOFCs can have their desired structure. Besides, in order to counter the problem regarding dense cathode layer resulted from high co-sintering temperature, the modification of the cathode material chosen by adding the pore former is essential in order to get the porous cathode layer. Thereupon, by establishing suitable fabrication parameters (i.e. co-sintering temperature, cathode extrusion rate and pore former loading) would allow the fabrication of defect free MT-SOFC HF precursor with the desired microstructure.

To ensure the reliability of TLHF MT-SOFC developed from this novel technique, a durability test is important in order to examine its stability under long term operation of high temperature SOFC system. In addition, a benchmarking analysis would be the last stage involved in this study. This analysis is necessary to determine the effectiveness of the fabrication technique used in this study in developing a high quality of MT-SOFC. Hence, in this stage, the cell (TLHF) that prepared via the novel technique would be compared with the cell (DLHF) prepared via phase inversion plus brush-painting technique. The comparative analysis will involve the characterization test such as, morphology, gas-tightness, mechanical strength, porosity, as well as their current-voltage performance results.

Therefore, with all the approaches applied, the introduction of phase inversion-based co-extrusion/co-sintering technique could be an alternative simplified or fast way to fabricate a complete cell of MT-SOFC via one-step preparation. It is expected the TLHF fabricated will be composed of asymmetric anode, dense and gas-tight electrolyte as well as porous cathode. Besides, this TLHF MT-SOFC could exhibit adequate mechanical strength, appropriate cathode porosity, high durability performance, and comparable to those that fabricated via conventional way. It believes that this novel outcome would be beneficial to the researchers in this area in the application of high temperature MT-SOFC.

1.3 Objectives of Research

Main goal of this research is to develop anode/electrolyte/cathode micro-tubular solid oxide fuel cell (MT-SOFC) via single-step preparation of phase inversion-based co-extrusion/co-sintering technique for high temperature application.

The objectives of this research are:

I. To study the compatibility on the phase transformation of cathode hollow fiber made from lanthanum strontium manganite (LSM) and lanthanum strontium cobalt ferrite (LSCF) as the main cathode material with the electrolyte material as a function of high sintering temperature.

II. To investigate the effect of co-extrusion/co-sintering parameters (i.e. cathode extrusion rate and co-sintering temperature) on the physical and chemical properties of triple-layer hollow fiber (TLHF) fabricated via phase inversion-based co-extrusion/co-sintering technique.

III. To examine the influence of the different loading addition of graphite pore-former in the cathode dope suspension and bore fluid flow rate adjustment to the structure and physical properties of the TLHF fabricated via phase inversion-based co-extrusion/co-sintering technique.

IV. To evaluate triple-layer hollow fiber (TLHF) MT-SOFC current-voltage performance for long term operation; and conducting benchmarking study between the novel co-extruded anode/electrolyte/cathode TLHF based cell and the commonly prepared anode/electrolyte dual-layer hollow fiber (DLHF) with brush-painted cathode based cell

1.4 Scope of Research

In this research, there are several scopes have been identified in order to achieve all of the objectives stated above. The scopes of this research are

I. Study of the different type of cathode material:
   a. Preparing cathode spinning suspensions made from LSM-YSZ and LSCF-YSZ.
   b. Examining the effect of the different of cathode materials (LSM/YSZ; LSCF/YSZ) on the phase transformation of single-layer cathode hollow fibers.
   c. Analyzing the effect of sintering temperature on single-layer cathode hollow fiber made from different cathode material. The sintering temperature is varied in between (1250 °C, 1300 °C, 1350 °C, 1400 °C and 1450 °C). The effect will be investigated in term of its morphology, porosity distribution and crystal structure.

II. Investigation of the co-extrusion/co-sintering parameters on the properties of anode/electrolyte/cathode triple-layer hollow fiber (TLHF) MT-SOFC:
   a. Preparing anode, electrolyte and cathode spinning suspensions by using the selected cathode material from previous finding in objective I.
   b. Shaping the spinning the suspensions prepared into TLHF using phase inversion-based co-extrusion technique by varying cathode layer extrusion rate within 3 ml min^{-1}, 4 ml min^{-1}, 5 ml min^{-1} and 6 ml min^{-1}.
   c. Consolidating the hollow fiber precursor via co-sintering process in high temperature tube furnace. The sintering temperature will be varied from 1300 °C to 1450 °C.
   d. Characterizing the physical and chemical properties of TLHF in term of its morphology by using Scanning Electron Microscope (SEM), Energy dispersive X-ray spectroscopy (EDS), X-ray Diffraction (XRD) test for crystal structure analysis, mechanical strength determination via 3-point bending test, and gas tightness properties using N_2 permeation test.

e. Developing the MT-SOFC reactor and conducting a performance test by potentiostat/galvanostat at temperature range of 700 °C to 800 °C. The open-circuit voltage, power density and impedance data will be recorded.

III. Enhancing the cathode structure of triple-layer hollow fiber (TLHF) MT-SOFC by:

a. Adding the different graphite pore-former loading of 2.5 %, 5 %, 7.5 %, 10 %, 12.5 % and 15 % to the cathode suspension.
b. Fabricating TLHF with the addition of graphite ($TLHF_{gr}$) via phase inversion-based co-extrusion/co-sintering technique by following the optimum co-sintering temperature and ideal cathode extrusion rate found out from objective II
c. Adjusting the various bore fluid flow rate of 8 ml min^{-1}, 10 ml min^{-1}, 12 ml min^{-1}, 14 ml min^{-1}, and 16 ml min^{-1} during the fabrication process.
d. Analyzing the viscosity of cathode suspensions prepared and characterizing the $TLHF_{gr}$ in term of its morphology, cathode porosity, mechanical strength and gas-tightness property.
e. Performing the current-voltage performance at different range of operating temperature between 700 °C to 850 °C.

IV. Performing stability test and benchmarking study of triple-layer hollow fiber (TLHF) MT-SOFC developed by:

a. Conducting the performance test for TLHF MT-SOFC with and without the graphite addition for 24 h of operating time.
b. Fabricating dual-layer hollow fiber (DLHF) of anode/electrolyte via phase inversion technique and depositing the cathode with and without the presence of graphite, via brush-painting technique. The graphite loading used in this part will based on the result of objective III.
c. Characterizing the dual-layer hollow fiber in term of its morphology mechanical strength, and cathode porosity.
d. Comparing the characteristics and current-voltage performance of dual-layer hollow fiber and TLHF MT-SOFC with and without graphite.

1.5 Research Contribution

The outcome of this study is expected to give a better understanding of the fundamental concept for the single-step fabrication of TLHF MT-SOFC. It is acknowledged that the phase inversion technique has been used for various types of research nowadays, especially in the development of MT-SOFC field, however little attention has been given to the production of TLHF per spin. The phase inversion technique applied in this study involves the co-extrusion and co-sintering process of anode, electrolyte and cathode at the same time. This novel technique is extremely useful in shortening the fabrication time and reducing production cost. In meeting the effect of high temperature application, the approaches taken in this study could serve as a reference for further exploration of other co-extrusion/co-sintering parameters, in order to enhance the quality of the developed TLHF MT-SOFC. Therefore, this study could be beneficial to the researchers in this area regarding the knowledge on the single-step preparation of phase inversion-based co-extrusion/co-sintering fabrication technique of TLHF as a complete MT-SOFC.

1.6 Thesis Organization

This thesis is organized into eight chapters which provide the description on the fabrication of anode/electrolyte/cathode, TLHF MT-SOFC via single-step preparation of phase inversion-based co-extrusion/co-sintering technique.

Chapter 1 provides a brief overview of the research background in the field of fuel cell, specifically on the micro-tubular solid oxide fuel cells, MT-SOFC. An overview of the thesis outline is presented in this chapter, including the objectives and scopes of the study. **Chapter 2** discusses on a comprehensive literature review on the basic fundamental of SOFC and advancement in its fabrication techniques, involving the conventional and recently introduced co-extrusion/co-sintering technique based on phase inversion concept. Additionally, it provides a review of the common technique of cathode deposition. The materials, instruments, and methodologies used throughout this study are discussed in **Chapter 3.** Detailed illustrations of the entire research framework and working procedures are presented as well.

Result and discussion are discussed in Chapter 4 until Chapter 7. **Chapter 4** focuses on the compatibility study between two different commonly used cathode materials, with an electrolyte material for application at high temperature of sintering. **Chapter 5** presents an in-depth discussion on the fabrication TLHF of anode/electrolyte/cathode via the novel technique of phase inversion-based co-extrusion/co-sintering. It also deliberated the effect of co-extrusion and co-sintering parameters towards the properties of the TLHF fabricated. The parameters involve are co-sintering temperatures and cathode extrusion rate. The chemical and physical characteristics are well explained in this chapter. Further analysis will be conducted using the ideal of co-sintering temperature and cathode extrusion rate choose in this chapter.

In **Chapter 6,** the enhancement of cathode structure is conducted with the addition of graphite pore former. Details analysis of the effect of adding various amounts of graphite to the cathode suspension is presented in this chapter. Furthermore, in order to help optimize the enhancement procedure, the effect of bore fluid flow rate is described on TLHF consisting of graphite loading ($TLHF_{gr}$). A series of characterization and performance analysis of $TLHF_{gr}$ are included in this chapter. **Chapter 7** examines the stability analysis of electrochemical performance of TLHF and $TLHF_{gr}$ for long term operation. In addition, this chapter also reveals the effectiveness of this novel technique by conducting the comparative study between MT-SOFC that fabricated via the multiple-step preparation of phase inversion and brush-painting technique, and single-step preparation of phase inversion-based co-extrusion/co-sintering technique. Finally, **Chapter 8** summarizes the findings of this study and offers recommendations for future research.

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LIST OF PUBLICATIONS

Journal with Impact Factor

1. Ab Rahman, M.; Othman, M.H.D.; Fansuri, H.; Harun, Z.; Omar, A.F.; Shabri, H.A.; Ravi, J.; Rahman, M.A.; Jaafar, J.; Ismail, A.F. Development of high-performance anode/electrolyte/cathode micro-tubular solid oxide fuel cell via phase inversion-based co-extrusion/co-sintering technique. *J. Power Sources* 2020, 467, 228345, doi:10.1016/j.jpowsour.2020.228345. **(Q1, IF: 8.247)**

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Book Chapter

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