

**PRODUCTION OF SINGLE CELL ANODE-SUPPORTED SOFC
USING NiO/YSZ-YSZ-LSM/YSZ POWDER MATERIALS**

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**A thesis submitted in fulfillment of the requirement for award the Degree of
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For my parents, wife, son and daughter:

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Sulistyo

ABSTRACT

Solid oxide fuel cell (SOFC) is an energy conversion device that converts gas into electricity directly. The components consist of anode, electrolyte and cathode which have different properties and operate at high temperatures up to 1000 °C. SOFC requires a thin, robust, porous anode, dense electrolyte and porous cathode. Manufacturing of single cell using low compaction value and high diameter to thickness ratio is yet to be developed. The challenge of the higher diameter to thickness ratio is inhomogeneous density distribution which causes cracks during sintering. This research investigates the manufacturing of single cell using low compaction and high diameter to thickness ratio. The performance of single cell was evaluated using hydrogen and oxygen that are supplied to the anode and cathode respectively. Initially the anode was produced using ultrasonic (U/S) process and ball mill (BM) process. The materials for single cell are NiO/YSZ, YSZ, LSM/YSZ as anode, electrolyte and cathode, respectively. The cornstarch is used to create anode porosity while the material binder of the powder is polyvinyl alcohol (PVA) to increase green compact strength. The consolidation of anode was done using a low compaction load of 16 MPa with a diameter of 40 mm. The electrolyte and cathode layer were coated using wet powder spraying (WPS) technique. The sintering for single cell used a three step air sintering. The characterizations of the anode include porosity, permeability, bending strength, microstructure and these were tested to evaluate the performance of the single cell SOFC. The results demonstrated that the thin SOFC single cell was successfully obtained. The single cell SOFC has a thickness of 600 μm where the distribution of thickness for the anode, electrolyte, and cathode were 557.5 μm , 26.1 μm and 16.4 μm , respectively. The cell diameter obtained was 36 mm. The low compaction load and the diameter to thickness ratio were 16 MPa and 60, respectively. Performance of open circuit voltage (OCV) for U/S and BM process is 0.8 V and 0.66 V, respectively. Power density performance for U/S and BM process is 200.41mA/cm² and 151.63mA/cm² respectively. The results indicated that the performance of OCV and power density for U/S were 33.34% and 32.17 % higher than BM process.

ABSTRAK

Sel bahan api oksida pejal (SOFC) adalah suatu alat penukaran tenaga yang menukarkan tenaga gas kepada tenaga elektrik secara langsung. Komponen ini terdiri daripada anod, katod dan elektrolit yang mempunyai ciri-ciri yang berbeza dan mampu beroperasi pada suhu yang tinggi mencecah 1000°C. Keperluan ciri-ciri sel SOFC adalah ianya nipis, tahan lasak, mempunyai anod berliang, elektrolit padat dan katod berliang. Penghasilan sel tunggal menggunakan nilai pemadatan yang rendah dan nisbah diameter kepada ketebalan yang tinggi masih belum dikembangkan sepenuhnya. Cabaran dalam penghasilan nisbah diameter kepada ketebalan yang tinggi adalah taburan kepadatan yang tidak homogen, yang menjadi punca retak semasa proses pensinteran. Kajian ini mengkaji penghasilan sel tunggal menggunakan nilai pemadatan yang rendah serta nisbah diameter kepada ketebalan yang tinggi. Prestasi sel tunggal telah dinilai menggunakan hidrogen dan oksigen yang dibekalkan masing-masing kepada anod dan katod. Pada awalnya, anod telah diproses menggunakan teknik ultrasonik (U/S) dan pengisar bebola (BM). Bahan untuk sel tunggal adalah NiO/YSZ – YSZ – LSM/YSZ, masing-masing sebagai anod, elektrolit dan katod. Tepung jagung digunakan untuk membuat keliangan anod manakala pengikat bahan serbuk adalah polyvinyl alkohol (PVA) untuk meningkatkan kekuatan padat hijau. Penggabungan anod telah dilakukan menggunakan beban pemadatan rendah sebanyak 16 MPa dan diameter 40 mm. Manakala elektrolit dan lapisan katod telah disalut menggunakan teknik semburan serbuk basah (WPS). Pensinteran untuk sel tunggal menggunakan tiga langkah pensinteran dalam pensinteran udara. Ciri-ciri anod yang diuji adalah keliangan, kebolehtelapan, kekuatan lenturan, mikrostruktur dan ujian prestasi bagi SOFC sel tunggal. Keputusan menunjukkan bahawa sel SOFC nipis telah berjaya dihasilkan. Sel tunggal SOFC mempunyai ketebalan 600µm dengan taburan ketebalan untuk anod, elektrolit dan katod adalah masing-masing 557.5µm, 26.1µm, 16.4µm. Diameter sel yang diperolehi adalah 36mm. Nilai beban pemadatan rendah dan nisbah diameter kepada ketebalan adalah masing-masing 16 MPa dan 60. Prestasi litar voltan terbuka (OCV) untuk U/S dan BM adalah 0.8 V dan 0.66 V. Prestasi ketumpatan kuasa pula untuk U/S dan BM adalah 200.41mA/cm² dan 151.63mA/cm². Keputusan menunjukkan bahawa prestasi OCV dan ketumpatan kuasa untuk U/S adalah 33.34% and 32.17 % lebih tinggi daripada BM.

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PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

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LIST OF SIMBOLS AND ABBREVIATIONS

AES	Air Electrode Supported
AFC	Alkaline Fuel Cell
AFL	Anode Functional Layer
APS	Atmospheric Plasma Spraying
ASR	Area Specific Resistance
BCNO	$\text{BaCe}_{0.9}\text{Nd}_{0.1}\text{O}_{3-\delta}$
BCY	$\text{BaCe}_{0.8}\text{Y}_{0.2}\text{O}_{3-\delta}$
BCZY	$\text{Ba}_{0.98}\text{Ce}_{0.6}\text{Zr}_{0.2}\text{Y}_{0.2}\text{O}_{3-\delta}$
BM	Ball mill
BZCY7	$\text{Ba}(\text{Zr}_{0.1}\text{Ce}_{0.7})\text{Y}_{0.2}\text{O}_{3-\delta}$
CaO	Calcium Oxide
CaSZ	Calcium Stabilized Zirconia
CCS	CO ₂ Capture and Storage
CSP	Concentrated Solar Power
CFCL	Ceramic Fuel Cells Limited, Australian SOFC company
CFL	Cathode Functional Layer
CHP	Combine Heat and Power
CO ₂	Carbon dioxide
CTE	Coefficient of Thermal Expansion
CVD	Chemical Vapor Deposition
DC	Direct Current
ECN	Energy research Centre of Nederland
EVD	Electrochemical Vapour Deposition
EDS	Energy Dispersive X-Ray Spectroscopy

Er ₂ O ₃	erbium oxide
FWHM	Full Width of the peak at Half of the Maximum intensity
GDC	Gadolina Doped Ceria
GT	Gigatons
HTSOFC	High Temperature Solid Oxide Fuel Cell
IEA	International Energy Agency
ICE	Internal Combustion Engine
ITSOFC	Intermediate Temperature Solid Oxide Fuel Cell
LaCoO ₃	Lanthanum cobalt oxide
L/A	Length to the cross-sectional ratio
LSCF	Lanthanum Strontium Cobalt Ferrum Oxide
LSCF/SDCC	Lanthanum Strontium Cobalt Ferrum Oxide- Samarium Doped Ceria Carbonate
LSM/YSZ	Lanthanum Strontium Manganite-Yttria Stabilized Zirconia
LTSOFC	Low Temperature Solid Oxide Fuel Cell
MA	Mechanical Alloying
MCFC	Molten Carbonate Fuel Cell
MEA	Membrane Electrolyte Assembly
MHI	Mitsubishi Heavy Industry
MnO ₂	mangan oxide
MPa	Mega pascal
MW	Megawatts
NASA	National Aeronautics and Space Administration
NO _x	Nitrogen mono oxide
NiO/SDCC	Nickel Oxide-Samarium Doped Ceria Carbonate
NiO/YSZ	Nickel Oxide – Yttria Stabilized Zirconia
NiO/YSZ8	Nickel Oxide-Yttria Stabilized Zirconia 8 mole %
NiO/BCIO ₃	Nickel Oxide-Barium Cerium Indium Oxide
NO _x	Nitrogen mono oxide
OCV	Open Circuit Voltage
PAFC	Phosphoric Acid Fuel Cell
PEM	Polymeric Electrolyte Membrane
PEMFC	Polymeric Electrolyte Membrane Fuel Cell
PSA	Particle Size Analyzer

PV	Photovoltaic
PVA	Polyvinyl Alcohol
PVD	Physical Vapor Deposition
RADAR	Radio Detection And Ranging systems
SEM	Scanning Electron Microscopy
SDC	Samarium Doped Ceria
ScSZ	Scandia Stabilized Zirconia
SDCC	Samarium Doped Ceria Carbonate
SOFC	Solid Oxide Fuel Cell
SONAR	Sound Navigation and Ranging
SO _x	Sulfur mono oxide
SWPG	Siemens Westinghouse Power Generation
TEM	Transmission Electron Microscopy
TGA	Thermal Gravimetric Analyzer
TPB	Triple Phase Boundary
TPBL	Triple Phase Boundary Length
UK	United Kingdom
U/S	Ultrasonic
Vol	Volume
VO ₂	Vanadium Oxide
wt	Weight
WPS	Wet Powder Spraying
WTI	West Texas Intermediate
XRD	X-Ray Diffraction
Y ₂ O ₃	Yttrium Oxide
YSZ	Yttria Stabilized Zirconia
YSZ8	Yttria Stabilized Zirconia 8 mole %
ZrO ₂	Zirconium dioxide
μm	Micro meter, unit of length
μCT	Micro Computer Topography
A	The area of the layer
C _s	Crystallite size
C _o	Cosntant
D	Diffusion coefficient

E	The electrical potential difference
E°	The standard-state reversible voltage
F	Faraday's constant
G	Gibbs free energy
Δg°	The standard-state free energy change for reaction
Δp	The pressure drop over the membrane
k	Scherrer's constant
m	meter, unit of length
n	Number of moles of electron transfer
P	Maximum load
S/cm	Siemens/ centimeter, unit of electric conductivity
T	Temperature, K
t	Thickness of specimen
V	Bulk Volume
V	Voltage, unit of electric voltage
V_s	Volume of solid
w	Width of specimen
Greek letters	
λ	The wavelength of $\text{CuK}\alpha$ radiation (1.5406 Å)
β	The fullwidth of the peak at half of the maximum intensity (FWHM)
κ	Permeability
θ	The diffraction angle of the corresponding reflection
η	The viscosity of the gas
Φ	Porosity
σ_B	Bending stress

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CHAPTER 1

INTRODUCTION

1.1 Research background

The energy crisis and greenhouse effect has attracted the development for alternative energy. There were several types of renewable energy studied include the geothermal, wave, wind and nuclear energies. Various researchers have worked in developing devices that converts these energies into electricity. One of the potential energy devices is fuel cells.

Fuel cells can be considered as environmental friendly with relatively high efficiency [1]. There exist several types of fuel cells, e.g. Alkaline Fuel Cell (AFC) [2], Molten Carbonate Fuel Cell (MCFC) [3], Phosphoric Acid Fuel Cell (PAFC) [4], Polymeric Electrolyte Membrane Fuel Cell (PEMFC) [5] and Solid Oxide Fuel Cell (SOFC) [6, 7]. The first three use aqueous electrolyte while the rest use dense or solid electrolyte. There are two main disadvantages when using aqueous electrolyte, i.e. unstable performance and high production cost. The usage of the aqueous electrolyte also decreases the performance because of the evaporation occurs in the electrolyte. The material on the electrode sides are both made of expensive material such as platinum, hence make it not cost effective.

The differences between the PEMFC and SOFC are in its operating temperature and material used in the process. The PEMFC operates at low temperature ranging from 80 – 150 °C, whereas the SOFC operated at temperature ranging between 500° – 1000°C. The core of the material of the PEMFC uses polymeric based and both electrodes at the anode and cathode use pure material, such

as platinum. The weakness of the PEMFC is the presence of water flooding in the electrodes which affect the performance. The membrane is also not stable at high temperatures. On the other hand, for the SOFC, ceramic material is used in the electrodes and electrolyte. The advantages of the ceramic material is resistant to operate at high temperature and robust. Hence, the issue of water flooding on the side of the electrode of SOFC does not occur.

SOFC operating temperatures are influenced by its material. In general, there are two types of material used for SOFC, namely the Low Temperature SOFC (LT-SOFC) and High Temperature SOFC (HT-SOFC). The Low-Temperature SOFC (LT-SOFC) operates at a temperature range $500\text{ }^{\circ}\text{C}$ - $650\text{ }^{\circ}\text{C}$, whereas HT-SOFC operates within $800\text{ }^{\circ}\text{C}$ – $1000\text{ }^{\circ}\text{C}$. An example of LT-SOFC in terms of anode–electrolyte–cathode configuration is Nickel Oxide-Samarium Doped Ceria Carbonate (NiO/SDCC)-SDCC-Lanthanum Strontium Cobalt Ferrite-SDCC (LSCF/SDCC) [8, 9]. An example of HT-SOFC in terms of anode–electrolyte–cathode configuration is Nickel Oxide-Yttria Stabilized Zirconia (NiO/YSZ)-YSZ-Lanthanum Strontium Manganite-YSZ (LSM/YSZ)[10, 11].

The performance of LT-SOFC and HT-SOFC are affected by electrical properties of material especially the electrical conductivity. It is reported that at temperature of $550\text{ }^{\circ}\text{C}$, SDCC has ionic conductivity of 0.131 S/cm while the electronic conductivity of LSCF/SDCC composite is as low as $9.53\text{ }10^{-4}\text{ S/cm}$ depending on LT-SOFC operation [8]. At $1000\text{ }^{\circ}\text{C}$, YSZ has an ionic conductivity of $6.62\text{ }10^{-2}\text{ S/cm}$ while LSM/YSZ has a higher conductivity of 2.27 S/cm . Based on the previous reported data, Yang [12] considers that they can be used at high temperature operations.

In general, SOFC is developed in two types of configuration namely planar and tube. Each configuration has its own advantages. The Planar type is simpler to manufacture compared to tube type. On the other hand, tube type requires smaller seal for cell stack development.

The SOFC cell manufacturing methods can be done by either conventional or non-conventional methods. The conventional methods include tape casting, slip casting, compaction or pressing, dip coating, screen printing and spraying. Whereas, for the non-conventional methods, the processes involve chemical, physical and electrochemical vapour depositions [13, 14].

Son et. al. [15] has patented the process of manufacturing an anode-supported SOFC using the screen printing method. The process can reduce the structural defects in each layer of solid oxide fuel cell and interfacial defect between the layers during the manufacturing process. Here, the used materials for anode, electrolyte, and cathode are NiO/YSZ cermet, YSZ, LSM/YSZ, respectively. The method of fabricating of NiO/YSZ anode-supported SOFC using the NiO/YSZ slurry combined with vacuum deposition also has been patented. The anode produce pore size less than 1 micron and the electrolyte thickness using YSZ was about 2 times pore size without defect [15].

Another conventional process for consolidating ceramic powder is powder compaction. The ceramic powder and the binder are filled into the mould, and then pressed at certain pressure to form green compact. Generally the density of green compact is not homogeneous. Stupkiewicz et al. [16] show the distribution of density of the green compact, the higher density is usually located at surface which is adjacent with puch. The compaction loads for the manufacturing of NiO/YSZ anode-supported SOFC may vary depending on the size and thickness. For instance, the lowest load is 21 MPa with a diameter of 57.2 mm and 3.2 mm thick [17], the medium load using material NiO/SDCC is 400 MPa with a diameter of 13 mm and thickness of 2 mm [18] while the highest load reaches 1000 MPa with a diameter of 13 mm and thickness 0.6 mm [19]. The ratios of the diameter to the thickness at low, medium and high loads are 17.9, 6.5 and 21.7 respectively.

1.2 Problem statement

A SOFC comprised of anode, electrolyte and cathode layers. In order to obtain an effective system, the cells should be manufactured as thin as possible to promote electrical transmission and minimise the electrical resistance losses. These characteristics are possible to achieve if non-conventional approaches are used. However, the manufacturing process using conventional method such as low compaction and powder spraying is a big challenge. The disadvantages of the compaction method is inhomogeneous density of green compact which cause warpage and crack during sintering [20]. Stupkiewicz et al. [16] show the

distribution of density and residual stress of ceramic material using compaction process. The higher density of compact product is located in the adjacent punch surface. Randal [21] and Rahaman [22] suggest that the ratio of the diameter to thickness is no more than one to avoid crack and warpage during sintering. Several researchers have produced sintered compact with ratio of diameter to thickness is more than one successfully. The anode-supported SOFC of NiO/SDCC was made by using load compaction of 400 MPa with diameter of 13 mm and thickness of 2 mm. The ratio of diameter to thickness is 6.5 [18]. The ratio of diameter to thickness of 35.7 has been achieved by load compaction of 94 MPa with diameter 25 mm and thickness 0.7 mm [23]. Furthermore, Kongfa [19] has produced anode-supported SOFC using load compaction of 1000 MPa with diameter of 13 mm and thickness of 0.6 mm. The ratio diameter to thickness of anode-supported SOFC is 21.7.

The issue of uniformity is of concern. The effect of non-uniform density is that the product is susceptible to cracking or warpage during the sintering process [24]. The high compaction load increase the frictional forces between powder particles and mould wall [25] resulted in the green compact is easily to crack during the sintering process, especially when the powder composition is not homogeneous in terms of its hardness.

SOFC performance is measured for its capability to generate power. The higher electricity is generated, the better the SOFC is. However, SOFC performance is influenced by several key factors. These factors include the manufacturing process, ionic conductivity material, porous electrode, thin cell and large cross-sectional areas [26, 27].

The ultrasonic process improves the homogenization of powder and powder size. The process subsequently affects the microstructure of the SOFC cell component during sintering process [28]. For instance, higher sintering process improves the density of the electrolyte that affect the performance of SOFC. Next important factor is ionic conductivity material. In order to increase the performance, the ionic conductivity should achieve the optimum value for a given operating temperature. At present, the most suitable or available materials for electrolyte were SDCC and YSZ, for LT-SOFC [8] and HT-SOFC respectively [29].

Other SOFC performance parameters are the porosity and thickness of the anode. The anode requires sufficient porosity and certain thickness to reduce electrical resistance and capable to support electrolyte and cathode, especially for

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