

SAMARIUM STRONTIUM COBALT OXIDE – SAMARIUM DOPED CERIA
CARBONATE (SSC-SDCC) AS A NEW POTENTIAL COMPOSITE CATHODE
MATERIAL FOR LOW TEMPERATURE SOLID OXIDE FUEL CELL



PTTA UTHM
PERPUSTAKAAN TUNKU TUN AMINAH

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This thesis has been examined on date 22 November 2020 and is sufficient in fulfilling the scope and quality for the purpose of awarding the Degree of Doctor of Philosophy in Mechanical Engineering.

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Universiti Tun Hussein Onn Malaysia

MAY 2021

Dedicated to my beloved late father, mother, husband and family
Allahyarham Mohammad Bin Ibrahim and Siti Ramlah Binti Othman,

My honorable supervisor,
Assoc. Prof. Dr. Sufizar Binti Ahmad



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ABSTRACT

Systematic research regarding SSC-SDC carbonate (SSC-SDCC) as composite cathode is limited despite great chemical compatibility and cell performance achieved with other low temperature solid oxide fuel cell (LTSOFC) components. LTSOFC based on SDCC electrolyte display lower of area specific resistance (ASR) with high value of stability and porosity. This study focuses to investigate the correlation of powder composition and calcination temperature on the chemical compatibility, microstructures, physical, mechanical, thermal and electrochemical performance of SSC-SDCC composite cathode. SSC-SDCC composite powders with the weight ratios of SSC toward SDCC of 50:50 (SSCB55), 60:40 (SSCB64), and 70:30 (SSCB73) were achieved using high energy ball milling (HEBM). All powders were calcined at 600, 650, 700 and 750 °C, pelletized and sintered at 600 °C. Characterization include the chemical and structural, thermogravimetric, thermal expansion coefficient (TEC), morphologies and porosity were carried out. Electrochemical impedance spectroscopy (EIS) was conducted in air. The chemical compatibility and morphologies of powder and pellet, thermal expansion and porosity were mostly affected by the calcination temperature as compared to powder composition. SSCB55 was selected for EIS evaluation due for the optimum chemical compatibility with lesser appearance of secondary phase. From EIS results, the area specific resistance (ASR) value at operating temperatures 600 °C were ten times smaller than the ASR value offered at operating temperatures 450 °C. The smallest ASR value was 5.06 Ωcm^2 with the activation energy (E_a) of 3.99 eV from composite cathode calcined at temperatures 750 °C. In conclusion, this study significantly contributes to the investigation of SSC-SDCC composite as potential cathode material for economical, high stability and promising energy converter devices in low temperature solid oxide fuel cell (LTSOFC).



ABSTRAK

Penyelidikan yang sistematik berkenaan dengan SSC-SDC karbonat (SSC-SDCC) komposit katod masih terbatas walaupun keserasian kimia dan prestasi sel yang unggul telah dicapai dengan komponen sel fuel oksida pepejal bersuhu rendah (LTSOFC) yang lain. LTSOFC berdasarkan SDCC elektrolit mempamerkan rintangan luas spesifik (ASR) terendah dengan nilai tinggi kestabilan dan keliangan. Kajian ini tertumpu kepada penyiasatan terhadap hubungan di antara komposisi serbuk dan suhu kalsin dengan mengkaji keserasian kimia, morfologi, fizikal, mekanikal, terma dan prestasi elektrokimia bagi SSC-SDCC komposit katod. Serbuk SSS-SDCC komposit katod pada nisbah peratus berat SSC terhadap SDCC, 50:50 (SSCB55), 60:40 (SSCB64), dan 70:30 (SSCB73) telah diperoleh dengan menggunakan teknik pengisaran bebola bertenaga tinggi (HEBM). Kesemua serbuk telah dikalsin pada suhu 600, 650, 700 dan 750 °C, dipelet dan disinter pada suhu 600 °C. Pencirian melibatkan struktur kimia, termogravimetri, fizikal pekali pengembangan terma (TEC), morfologi dan keliangan telah dilakukan. Spektroskopi impedans elektrokimia (EIS) telah dijalankan dalam udara terbuka. Faktor kalsin lebih banyak mempengaruhi struktur kimia dan morfologi serbuk dan pelet, sifat pengembangan terma dan keliangan berbanding komposisi serbuk. SSCB55 dipilih untuk kajian penilaian prestasi spektroskopi impedans elektrokimia berikutan memiliki tahap optimum keserasian kimia dengan kehadiran jumlah fasa sekunder yang paling sedikit. Dari keputusan spektroskopi impedans elektrokimia, rintangan luas spesifik (ASR) pada suhu operasi 600 °C adalah sepuluh kali ganda lebih kecil berbanding pada suhu operasi 450 °C. Nilai rintangan luas spesifik yang paling rendah iaitu $5.06 \Omega\text{cm}^2$ dengan tenaga pengaktifan (E_a) 3.99 eV telah berjaya dicapai bagi sampel yang dikalsin pada suhu 750 °C. Kesimpulannya, kajian ini memberikan sumbangan yang penting dalam perkembangan SSC-SDCC sebagai komposit katod yang berpotensi untuk ekonomi, berkestabilan tinggi, serta menjanjikan fungsi sebagai alat tukaran tenaga dalam sel fuel oksida bersuhu rendah (LTSOFC).



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LIST OF SYMBOLS AND ABBREVIATIONS

AFC	-	Alkaline fuel cell
ASR	-	Area specific resistance
BSCF	-	Barium strontium cobalt ferrite
C	-	Carbon
CO ₂	-	Carbon dioxide
Ce	-	Cerium
E _a	-	Activation energy
EDS	-	Energy dispersive spectroscopy
EIS	-	Electrochemical impedance spectroscopy
FESEM-	-	Field emission scanning electron microscopy
FTIR	-	Fourier transform infrared
FWHM-	-	Full width at half maximum
GDC	-	Gadolinium-doped ceria
h	-	hour
HEBM-	-	High energy ball milling
H ₂	-	Hydrogen
HF	-	High frequency
HT	-	High temperature
HV	-	Hardness Vickers
IT	-	Intermediate temperature
LEBM-	-	Low energy ball milling
LF	-	Low frequency
Li	-	Lithium
LSCF	-	Lanthanum strontium cobalt ferrite
LT	-	Low temperature
MF	-	Medium frequency
NiO	-	Nickel Oxide



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Ni	-	Nickel
MCF	-	Molten carbonate fuel cell
Na	-	Natrium
O ₂	-	Oxygen
OCV	-	Open circuit voltage
R _Ω	-	Ohmic resistance
R _p	-	Polarisation resistance
PEMFC	-	Proton exchange membrane fuel cell
PAFC	-	Phosphoric acid fuel cell
SOFC	-	Solid oxide fuel cell
SDC	-	Samarium-doped ceria
SDCC	-	Samarium-doped ceria carbonate
SEM	-	Scanning Electron Microscopy
Sm	-	Samarium
SSC	-	Samarium strontium cobalt oxide/cobaltite
TEC	-	Thermal expansion coefficient
TPB	-	Triple phase boundary
XRD	-	X-ray diffraction
YSZ	-	Ytria stabilised zirconia



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CHAPTER 1

INTRODUCTION

1.1 Background of research

The world average temperature is rising due to the greenhouse effect triggering global warming and affecting climate change in many countries as in Asia, Africa and Europe. Levels of greenhouse gases rise promptly, especially undesirable carbon dioxide formed from combustion process of fossil fuels. Production and distribution of energy disrupt all sectors of the global world economy. The growing industrialization of the world currently demands on sustainable and highly efficient energy productivity. Without foremost technological developments, energy demand is not satisfied (Abdalla *et al.*, 2018). Fuel cells have been identified as a promising energy conversion processes, alternatively to conventional fossil fuel power generation. Fuel cells have the distinct interest over these traditional generative techniques in that they can directly convert fuel into electricity without the requirement for combustion (Sartori *et al.*, 2017). They are environmentally friendly and clean yet they can greatly diminish the generation of nasty carbon dioxide.

The utilization of nanoscience in energy sector, specifically in various kinds of fuel cells which can be used ultimately and potentially application at different scales, solid oxide fuel cells (SOFCs) have arose as energy conversion devices in succeeding high efficiency of over 70% with regeneration. SOFC is constructed from a dense of electrolyte layer that is sandwiched between two porous an electrodes which are cathode and anode (Singh *et al.*, 2017). Huge of valuable electricity yields from SOFC by the reaction occurred between a fuel and an oxidant reactant through an ion



conducting in solid electrolyte layer, with an additional amount of respected heat energy. (Kan *et al.*, 2016). Scientifically, the electrons generated through the oxidation of fuel at anode had been used for oxygen reduction at the cathode, made the circulation external circuit complete. Hence, the electricity energy is gained by the stream of electrons in the external circuit. There are numerous researches in cultivating the components toward SOFC such as cathode, electrolyte, anode and the manufacturing technology. However, for versatile materials to be apply in SOFC devices, each component in the cell need more progresses and investigations with superior enhanced properties (Rahman *et al.*, 2012; Agun *et al.*, 2014; Afzal *et al.*, 2015; Gao *et al.*, 2017; Ng, Rahman, & Somalu, 2018).

Recently, cobaltite perovskite oxide with $A_{1-x}B_xCoO_{3-\delta}$ structure, has been used as intermediate temperature SOFC cathode material as it required on high conductivity and promising catalytic activity. These factors have made several cobaltite perovskite oxide such as samarium strontium cobalt oxide, $Sm_{0.5}Sr_{0.5}CoO_{3-\delta}$ (SSC), lanthanum strontium cobalt oxide, $La_{0.5}Sr_{0.5}CoO_{3-\delta}$ (LSC), lanthanum strontium cobalt ferrite oxide $La_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF), and barium strontium cobalt ferrite oxide, $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ (BSCF) (Lee *et al.*, 2011; Li *et al.*, 2014; Ritzmann, 2016) are good candidates to be used as cathode material. For better cathode material, it must achieve several requirements such as high electronic and ionic conductivity, a matched thermal expansion coefficient (TEC), chemical compatibility with the electrolyte and interconnect materials, moderate porosity and stability under an oxidizing atmosphere. (Mahato *et al.*, 2015). Today, the convention is to use an electronically conductive perovskite material mixed with the electrolyte of choice, thus extending the triple phase boundary (TPB) area where the electrocatalytic activity occurs throughout the electrode and drastically improving the performance of SOFC (Duong & Mumm, 2013).

Among these, samarium and strontium cobalt oxide, $Sm_{0.5}Sr_{0.5}CoO_{3-\delta}$ (SSC) is regarded as one of the most promising cathode materials (Lv *et al.*, 2007; Gao *et al.*, 2009; Bansal & Wise, 2012; Lee *et al.*, 2015; Deng *et al.*, 2017). It is because it shows a higher ionic conductivity than that of lanthanum strontium manganate, $La_{0.8}Sr_{0.2}MnO_{3-\delta}$ (LSM), exhibits extraordinary electronic conductivity than pure barium strontium cobalt ferrite $Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3-\delta}$ (BSCF), higher of surface oxygen exchange rate compared to lanthanum strontium cobalt ferrite, $La_{0.5}Sr_{0.5}Co_{0.2}Fe_{0.8}O_{3-\delta}$ (LSCF) and reveals a good catalysis for oxygen reduction and



is particularly compatible with the ceria based electrolytes (Chang *et al.*, 2011; Chen *et al.*, 2011; Dong *et al.*, 2012; Gao *et al.*, 2017). However, SSC itself is having a large thermal expansion coefficient (TEC) which can be improved by incorporation SSC with samarium doped cerium carbonate (SDCC) electrolyte (Wang, Chen, & Shao, 2012; Ahmadrezaei *et al.*, 2014). Up to now, seldom research has been carried out on the study of SSC-SDCC composite cathode as a new cathode material for LTSOFC (K. Gao, Liu, Wang, Liang, & Xiong, 2016; Deng *et al.*, 2017; S. Lee *et al.*, 2018). The use of SDCC may also help in the performances of this SSC-SDCC composite cathode LTSOFC since carbonate universally exhibits good compatibility with many cathodes composite powders (Muhammed *et al.*, 2013; Mohammad *et al.*, 2017).

Nonetheless, a coupled of scientists Baur and Preis has been successfully developed the first model of SOFC in 1937, which can be operated at 1000 °C. Ever since most commonly used SOFC models operating with high temperatures between 800 °C and 1000 °C or known as HTSOFC. This fact leads to material degradation issues at great speed and incurs high maintenance costs, thus hindering their economic feasibility (Sartori *et al.*, 2017). Research interests have been directed also to develop SOFC operating from the intermediate temperatures (ITSOFC) between 600 °C and 800 °C and low temperatures (LTSOFC) from 400 to 600 °C (Jing *et al.*, 2013; Chen Xia *et al.*, 2016). By lowering the temperature of operation, a wider range of materials can be used, offers more rapid start-up and shut-down and reduced corrosion rate of metallic components in SOFC, as well as the advantage of greatly simplified system operating requirements.

This research is conducted for the purpose of improving the cathode which can be operate at low temperature SOFC (LTSOFC). LTSOFC significantly designed to enhance the compatibility, manufacturing cost and stability with many materials (Baharuddin *et al.*, 2017). However, electrochemical performance of SOFC system substantially decreased with a reduced operating temperature, due to the increased on polarization resistance. The polarization resistance are closely related with the interfacial resistance namely cathode-electrolyte or anode-electrolyte (Ali *et al.*, 2018). The cathode has been the centre of the focus in the electrode development largely because oxygen reduction is more difficult to activate in SOFCs operating at commercially relevant temperatures (Mahato *et al.*, 2015). It is critically important to investigate new potential cathode materials or novel cathode microstructures with low



polarization loss to maintain sufficient high electrochemical activity to enable SOFC operating at lower temperatures (Ding *et al.*, 2014).

1.2 Problem statement

Lowering the operation temperature has caused a substantial performance decline resulting from cathode polarization resistance. To minimize the issues, the study on composite cathodes incorporate with perovskite material systems, which optimizable operated at a lower temperature should be done. These systems also display lower of polarization resistances, lower activation energies, increase the triple phase boundary (TPB) area, hence improves the performance of cell (Newby *et al.*, 2015; Afzal *et al.*, 2015). Undeniably, processing method of starting composite cathode powder also provides a significant impact on the cell performance. It includes the parameters of milling method, calcinations and sintering. Microstructures properties of SSC-SDCC starting composite cathode powders prepared by HEBM method need to be clarify since the limitation information up to this date. HEBM is still rare and it is a feasible method to acquire a fine, homogeneous and well distributed powder which is indispensable for the better triple phase boundary (TPB) (Torknik *et al.*, 2014).

Samarium strontium cobalt oxide (SSC) is regarded as one of the utmost auspicious perovskite composite cathode materials. It is because SSC exhibits astonishing electronic conductivity, a higher ionic conductivity and is predominantly compatible with the ceria based electrolytes (Chang *et al.*, 2011; Chang *et al.*, 2015). Unfortunately, SSC is having a large thermal expansion coefficient (TEC) which approximately $20.0 \times 10^{-6} \text{K}^{-1}$ which leads to the rapid degradation and high reactivity with some electrolytes, during the performance operating at high temperature (Lv *et al.*, 2007; Dong *et al.*, 2012; Ni *et al.*, 2017). Previous authors had proven those drawbacks can be minimized by adding SSC with a small amount of samarium doped ceria (SDC) electrolyte and leads to increase composite cathode performance by improving the TPB value (Chang *et al.*, 2011; Lee *et al.*, 2015). No previous study has given sufficient information regarding incorporation of SSC with samarium doped cerium carbonate (SDCC) composite electrolyte.

Understanding the nature of SSC itself, particularly the influence of secondary phase on the stability issues during milling processes and how it is can be affected by

the atmosphere during calcination, sintering and performance is inquisitive. Besides that, previous work revealed the presence of impurities or secondary phases from SSC based composite cathode has to be clearly investigated (Bansal & Wise, 2012; Li *et al.*, 2014), because one of the crucial requirements of the composite cathode is the chemical compatibility needs to be maintained up to cell operation. Researcher believes that SDCC may ascend the performances of SSC-SDCC composite cathode, since accomplished authors of Huang *et al.*, (2010) & Wu *et al.*, (2016), reported the carbonate element universally reveals good compatibility with numerous cathodes composite powders. Reported from other authors that SOFC based on SDCC composite electrolyte display better of ionic conductivity value (10^{-2} to 1.0 Scm^{-1}), high value on stability and porosity, lower of area specific resistance (ASR) and adequate power densities (117.9 to 120.4 mWcm^{-2}), at the intermediate temperature of SOFC (Rahman *et al.*, 2012; Rahman *et al.*, 2014; Ali *et al.*, 2015; Hoa, 2017) . Likewise, the influences of composition and microstructures properties itself moreover crucial. Microstructural composite powder includes of particle size and porosity portrays vital impact toward TPB. Comprehension of TPB might be attained from the smaller (nanosized) of particle of the composite powder (Shimada *et al.*, 2016).

Finally, the researchers have been widely and continuously developed SOFCs for more than 40 years. Evolution of SOFCs has brought great success on reducing the operating temperature to enlarge the choice of cathode material such as SSC perovskite composite cathode. This material has shown high performance and potential as the cathode material for SOFCs especially between the intermediate and low temperature (Chang *et al.*, 2015; Gao *et al.*, 2017). SSC has been revealed as a potential cathode material and is particularly compatible with the ceria carbonate based electrolytes, SDCC (Deng *et al.*, 2017). This research is conducted for the purpose of improving the SSC based composite cathode which can be operate at low temperature solid oxide fuel cell ($450 - 600 \text{ }^{\circ}\text{C}$) by optimization the pristine properties with incorporation with the SDCC composite electrolyte. In conclusion, the properties of new cathode materials SSC-SDCC will be discussed in this present work. The structure stability, TPB aspect, thermal expansion coefficient, cathode conductivity and cathodic polarization resistance will be studied from the perspective of potential composite cathodes for LTSOFC applications.



1.3 Objective

This research work is focused in developing new potential composite cathode material showing stable and good performance in LTSOFCs. Correspondingly, the objectives to be achieved by this research are specified as follows:

1. To investigate SSC-SDCC composite cathode from different composite compositions and calcination temperatures, via high energy ball milling (HEBM) method.
2. To examine the influence of secondary phase of SSC on the SSC-SDCC powder by the clarification from the phase formation identification (XRD) of the milled and calcined powders.
3. To elucidate the correlation between carbonate, composite cathode compositions and calcination temperatures on the chemical, microstructural, physical, mechanical and thermal properties on the SSC-SDCC composite cathode powders and pellets.
4. To evaluate the EIS performance on the symmetrical cell of SSC-SDCC/SDCC/SSC-SDCC composite cathode in low temperature solid oxide fuel cell (400 -600 °C).



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1.4 Scope of Study

With the perspective of objectives as mention earlier, this dissertation scope study has been divided into five major parts. Starting from an early preparation of composite powder until the performance of composite cathode powder at low temperature of solid oxide fuel cell. Details are clarified in the next section below.

1. SSC (cathode), SDC (electrolyte), lithium carbonate (Li_2CO_3), sodium carbonate (Na_2CO_3) has been used to produce all the composite cathodes. The binary ratio of (Li/Na) carbonate (67 mol.% Li_2CO_3 and 33 mol.% Na_2CO_3) and SDC powder has been mixed thoroughly by low energy (wet) ball milling for 24 hours, to produce SDCC composite electrolyte.
2. Three groups of SSC-SDCC composite cathode powder with different composition of SDCC composite electrolyte were produced using high energy ball milling (HEBM). The composite cathodes were ball milled for 2 hours and followed by calcination process at 600, 650, 700 and 750 °C for 1 hour.
3. The SSC-SDCC composite cathode pellets and rods prepared by using uniaxial pressing at 11 MPa and both has been sintered at 600 °C for 1 hour with heating rate of 5°C/min
4. Characterization of the powder and pellet/rod samples:
 - a) After milling (before calcination)
 - i) Particle morphology and distribution of particle by using FESEM and SEM/EDS
 - ii) Phase analysis and crystallite size by using XRD
 - iii) Presence of amorphous carbonate phase by using FTIR
 - iv) Thermal analysis (weight loss) by using TGA/DTA
 - b) After calcinations
 - i) Particle morphology and element distribution of particle by using FESEM and SEM/EDS
 - ii) Phase analysis and crystallite size by using XRD
 - iii) Presence of amorphous carbonate phase by using FTIR
 - iv) Thermal analysis (weight loss) by using TGA/DTA
 - c) After sintering (composite cathode pellet/rod)
 - i) Particle morphology and distribution of particle by using SEM/EDS



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- ii) Density and porosity by using Archimedes principle
 - iii) Hardness testing analysis by using Vickers indentation hardness measurements
 - iv) Thermal expansion coefficient by using dilatometer
5. SSC-SDCC/SDCC/SSC-SDCC composite cathode performance using electrochemical impedance (EIS) at operating temperatures from 450-600 °C for LTSOFC application.

1.5 Novelty of study

Regarding the information from the literature review presented in Chapter 2, SSC-SDCC composite cathode has never been explored as a cathode component of a single cell for low temperature solid oxide fuel cell (LTSOFC) application. Despite decent power of SSC based composite cell performance achieved at high to intermediate temperature of SOFC, detailed and systematically phase stability, microstructural, physical, mechanical, thermal and electrochemical performance at low temperature SOFC are rarely reported. This study is discussing a novel finding and explains a clear understanding of SCC-SDCC as new composite cathode, based on the preparation and characterization and were intensively reported in this study.

Nonetheless, the influence of secondary phases from SSC which is strontium carbonate (strontionite) toward the phase stability and electrochemical performance at low temperature SOFC need to be clearly investigated. A comprehensive investigation regarding the influence of the secondary phase of SSC towards the phase formation analysis how it can be affect the electrochemical impedance analysis (EIS) results. As mentioned earlier, diversification on the properties of SDC composite electrolyte can be comprehended by adding with binary carbonate. The use of SDC with carbonate evidently assist in the performances of SSC-SDCC composite cathode when the operating temperatures of SOFC achieved the range between 500 to 600 °C. This new finding was deliberated transparently in this research study, beside carbonate universally exhibits a good compatibility with many composite cathodes

1.6 Summary

This chapter in brief deliberates the background of the research, problem statements, objectives and scope of study. Scope of study has been divided into four scopes includes the preparation and characterization of the sample studied. End of this chapter discussed on the significant and limitations of research work.



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CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

This chapter reviews the works reported from relevant and significant scientific journals that are correlated to the present research. General criteria, properties and behaviour of solid oxide fuel cell (SOFC) are delineated. Reviews on SSC as the composite cathode material in this present work, and SDCC as the composite electrolyte are presented concisely in this chapter.

2.2 Fuel cell

In the prior time of 1960, the International Fuel Cells (IFC) in Windsor, Connecticut, has successfully developed a fuel cell power plant for the Apollo spacecraft in the United States of America (USA). Historically, fuel cells have been discovered since scientists began searching for an alternative energy resources of fossil fuels and the first fuel cell in the world was made by Sir William Robert Grove in the early 1839 (Abdalla *et al.*, 2018). He named it “wet cell battery” or “Grove cell”, which operates by reversing the electrolysis phenomenon of water as shown in Figure 2.1 (Cassir *et al.*, 2013). “Grove cell” is defined as an electrochemical device that converts the chemical energy of fuels directly into electricity and heat by electrochemically combining the H₂, CO/H₂ or reformed hydrocarbons in fuel and an oxidant gas transported via ion conducting electrolyte. This revolutionary energy holds an enormous potential to provide a means of scalable, efficient, and environmentally

friendly power generation. An efficiency of 60% is ordinary and fuel cell is expected to be an essential and promising energy resources until the next of 21st century (Chandran & Arjunan, 2015; Jiang, 2019).

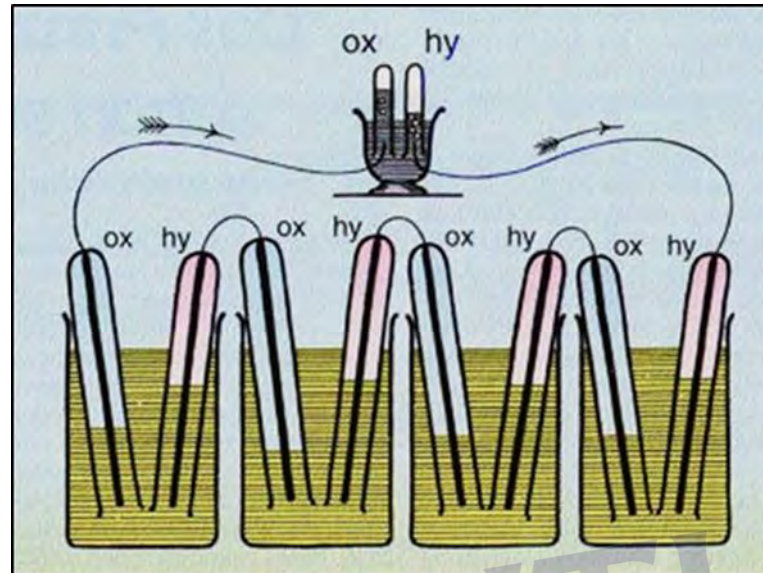


Figure 2.1: The first illustration of fuel cell developed by Sir William Robert Grove in the early 1839 (Cassir *et al.*, 2013)

Fuel cell is defined as an electrochemical device that can convert chemical energy of a fuel and an oxidant into heat and electric power. Fundamentally, it is composed of cathode, where the reduction of the oxidant occurs, anode that experiences the oxidation of fuel and electrolyte, which carries ion species between the electrodes (Kilius, 2009; Ritzmann, 2016). As compared to batteries that have to be occasionally recharged, fuel and oxidants can be consistently supplied to a fuel cell. This reaction is needed in order to generate power continuously. In the middle of the 22nd century, its development and evaluation accelerated. Several types of fuel cells are developed in the global race for conquering space and successfully approaching the consumer market in the world. However, the primary challenges to be concerned are cost and durability which can be solved by materials selection and design engineering technology (Mahato *et al.*, 2015).

In practice, fuel cells can operate in a wide range of temperatures between 50 and 1000 °C depending on the nature of the electrolyte, and are usually distinguished based on the component and construction criteria. There are many kind of fuel cells, all named after their electrolyte materials. They are categorized depending on the

nature of electrolyte. Different types of fuel cells require specific materials and fuels for different applications. Fuel cell types include; Proton Exchange Membrane Fuel Cells (PEMFCs), Direct Methanol Fuel Cells (DMFCs), Phosphoric Acid Fuel Cells (PAFCs), Alkaline Fuel Cells (AFCs), Molten Carbonate Fuel Cells (MCFCs), and Solid Oxide Fuel Cells (SOFCs). The comparisons of these major fuel cells in terms of components, fuels, capacities, costs, applications, advantages, and drawbacks are listed in Table 2.1 (Fachbereich & Geowissenschaften, 2012; Abdalla *et al.*, 2018). All kinds of fuel cells mentioned share the potential of high electrical efficiency, and reduce emissions, having unique characteristics linked to their different constituting materials and operating temperatures.

Among them, SOFCs have attracted in this research work subsequently worldwide interest due to their high energy conversion efficiency, structural integrity, easy operation, low environmental impact and high tolerance to fuels. Generally, SOFCs are cells that convert the chemical energy of hydrogen or other fuels directly into electrical energy through an electrochemical reaction with oxygen (Li, 2016). Fundamentally, this type of fuel cell is called an SOFC because of the solid electrolyte used, which is usually made of ceramics (Baharuddin *et al.*, 2013). The cleanliness and compact size of SOFCs make them especially attractive for urban settings. Compared with the other types of fuel cells, as described in Table 2.1, SOFCs present the unique characteristics of having all solid-state cell components along with reasonable operating temperature (600–1000 °C). Implementation of solid electrolyte in the SOFC has overcomes several limitations which associated with liquid electrolytes fuel cells. Such as, corrosion, electrolyte distribution, and the maintenance of stable triple phase boundary (TPB) within the electrode-electrolyte regions (Shin *et al.*, 2020). Nonetheless, because of their mainly ceramic structures, SOFCs can be configured into lightweight and compact structures unachievable with fuel cell using a liquid electrolyte (Mahato *et al.*, 2015). SOFCs can also be manufactured in relatively small, modular units. The heat released from the cell can be efficiently transferred and utilized for coal gasification or hydrocarbon reforming and also provide promising quality waste heat suitable for use in cogeneration.



Table 2.1: List of fuel cells (Morandi, 2013 & Abdalla *et al.*, 2018)

Type	PEMFC	AFC	PAFC	MCFC	SOFC	DMFC
Electrolytes	Solid polymer membrane	Potassium hydroxide	Phosphorus	Alkaline carbonate	Ceramic Oxide	Solid polymer membrane
Anode	Hydrogen	Hydrogen	Hydrogen	Hydrogen/methane	Hydrogen/methane	Methanol
Cathode	Pure or atm oxygen	Pure oxygen	Atm oxygen	Atm oxygen	Atm oxygen	Atm oxygen
Operating temperature (°C)	50-100	50-200	~200	600-700	600-1000	50-110
Charge carrier	H ⁺	OH ⁻	H ⁺	CO ₃ ²⁻	O ²⁻	H ⁺
Fuel	Pure H ₂	Pure H ₂	Pure H ₂	H ₂ , CO, hydro-carbon	H ₂ , CO, hydro-carbon	MeOH, CO ₂
Efficiency (%)	35-60	~50	40	45-50	>50	35-40
Power Density (W/cm ²)	1.1	1.0	1.1	0.7-1.0	0.8-1.0	
Typical system size (kW)	<50	10-100	10000	100000	Up 100000	100-1000
Applications	Portable power, transportation distributed generation	Military, space, portable power	Transportation, commercial cogeneration, portable power	Transportation, utility power plants, distributed generation	Residential, utility power plants, portable power	Transportation, military, energy storage system
Advantages	High power density, quick start-up, solid non-corrosive electrolyte	High power density, quick start-up, low-cost component	Heat waste, stable electrolyte	High efficiency, fuel and catalyst flexibility, heat waste	Solid electrolyte, high efficiency, fuel and catalyst flexibility, generate high grade waste heat	Low temperatures, suitable for portable devices
Drawbacks	Expensive platinum catalyst, sensitive to fuel impurities	Expensive platinum catalyst, sensitive to CO ₂ in fuel and air, electrolyte management	Expensive platinum catalyst, Corrosive liquid electrolyte, sensitive to fuel impurities, long start-up time	High cost, Corrosive liquid electrolyte, high temperature corrosion, slow start-up, intolerance to sulphur	High cost, high temperature corrosion, slow start-up, intolerance to Sulphur	Fuel crossing from anode to cathode without producing electricity

2.2.1 Introduction to solid oxide fuel cell

Solid oxide fuel cells, SOFCs, can be considered as the “most efficient” devices invented for conversion of chemical fuels directly into electrical power. SOFCs have emerged as energy conversion devices in achieving high efficiency of over 70 % with regeneration. They are promising alternative energy conversion devices that have the potential for implementing hydrocarbon fuels that are compatible with the current infrastructure, thereby allowing small steps to be taken towards a completely “green” fuel. The former SOFC was developed in 1937 by Baur and Preis (Buerger, 2006). Since then a lot of progress has been made in terms of materials and processing of SOFCs. The advantages of SOFC are well described in Table 2.2.

Table 2.2: Advantages of SOFC (Sunarso *et al.*, 2017; Abdalla *et al.*, 2018; Raza *et al.*, 2020)

Advantage	Description
Higher electricity density and modularity	Delivers an electrical efficiency between the range up to 50 – 60 %
Virtuous fuel flexibility	Attributed with clear path to renewable and CO ² -neutral energy systems
Low emissions	Low rate of noise sign due to no combustion process and moving parts
Higher efficiencies	Theoretical efficiency of an SOFCs device can exceed 60 %
Economical maintenance	Operating periods between overhauls are markedly longer than conventional technologies
Promising power generation	Produce power with minimal pollution

2.2.2 Fundamental principle operation of solid oxide fuel cell

As a whole, solid oxide fuel cell (SOFC) is characterized by the use of solid oxide material as an electrolyte, which acts as a conductor of oxygen ions. There are three basic components of SOFC, a porous anode, an electrolyte, and a porous cathode. The anode and cathode are located in compartments that are physically separated. The electrolyte usually conducts O²⁻ ions. The schematic cross section of the operation of the solid oxide fuel cell is shown in Figure 2.2 (Mahato *et al.*, 2015; Yu *et al.*, 2016). Typically, oxygen atoms are reduced on the porous cathode surface by electrons. The oxygen ions diffuse through the electrolyte to the fuel rich and porous anode, where they react with the fuel (hydrogen), and give off electrons to an external circuit. Thus,

the final products of SOFC are electricity, heat and water (Fan *et al.*, 2018). The electrolyte is also an electron insulator, forcing the electrons to be generated at the anode to flow through an external circuit, which can be used to satisfy a load. Basically, all three layers in SOFC contain ceramic materials, except for the anode, which is a composite material consisting of ceramic and metal (cermet) (Fachbereich & Geowissenschaften, 2012). Nevertheless, the oxidation reaction also known as fuel electrode, occurs at the anode as in Equation 2.1. Meanwhile, the reduction reaction occurs at the cathode as mentioned in Equation 2.2, also known as air electrode. The overall reaction of reduction and oxidation is presented in Equation 2.3 (Mahato *et al.*, 2015).

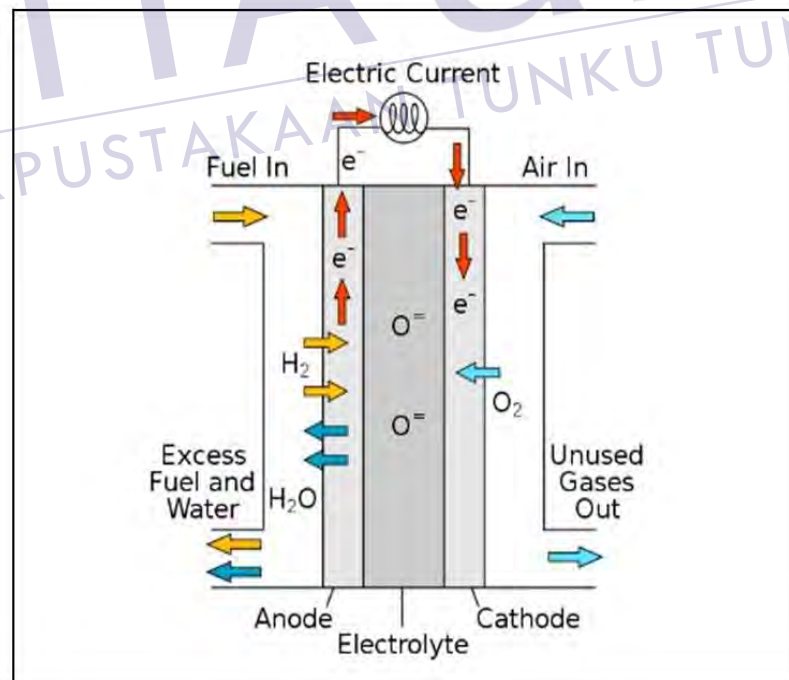


Figure 2.2: Schematic cross section diagram of the operation in SOFC using oxide-ion conducting electrolyte (Yu *et al.*, 2016)

2.2.3 Solid oxide fuel cell (SOFC) geometrical classification

Due to the solid type of electrolyte in solid oxide fuel cell (SOFC), various shapes are available and thus many designs have been devised since the 1960s. According to Morandi, (2013) clarified SOFC can be classified according to the cell designed geometry. The two most common cell designs developed for SOFCs correspond to planar and tubular cell geometries. Both designs with their advantages and disadvantages have been widely explored. A planar SOFC enables simple series of electrical connections of cell components configured as flat plates as shown in Figure 2.3. The planar reveals numerous advantages as the total of power density higher compared to the tubular. The cells are stacked between bipolar plates, diminish ohmic losses, and elevate the power densities of the cells. Also, configuration of planar cell allows the use of cost-effective, easiest fabrication, and favors the processing of the thinnest electrolyte layers (Timurkutluk *et al.*, 2016). As the huge advantages from the planar cell SOFC and promising power density, this configuration has chosen to be use in this research work.

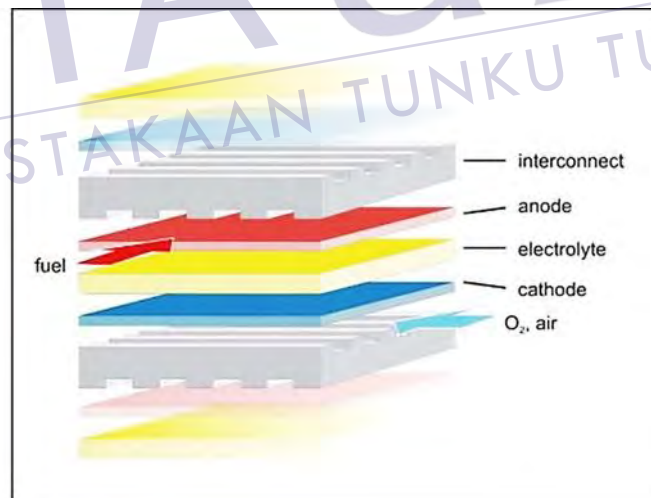


Figure 2.3: Geometrical design of planar SOFC (Timurkutluk *et al.*, 2016)

Another cell geometry regarded as a tubular is depicted in Figure 2.4. This tubular design is the acquainted type of SOFC, and it is challenging and very difficult to yield (Almar *et al.*, 2017). It has some drawbacks as presented with a low output power density in comparison with the previous uneconomical design. However, there are some compensations of tubular cells displayed with higher mechanical, good

thermal stability and it is easy to accomplish both gas seal and interconnection applications of single cells during stack manufacturing process (Egger, 2013).

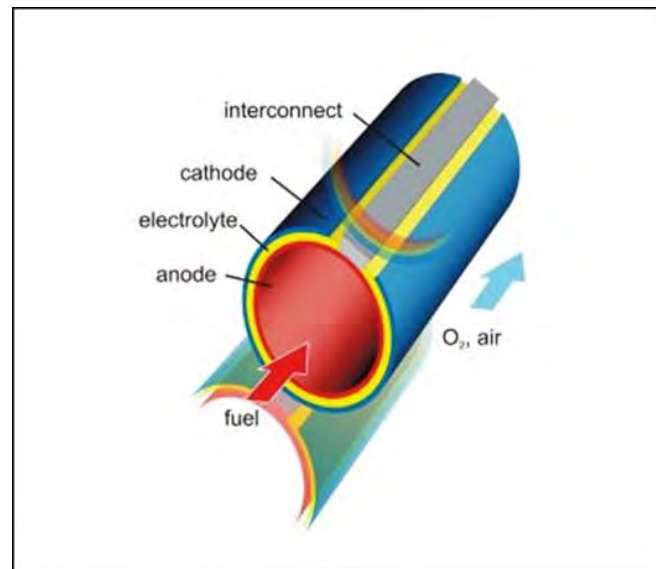


Figure 2.4: Geometrical design of tubular SOFC (Egger, 2013)

2.2.4 Low temperature solid oxide fuel cell

Initial design researches are frequently engrossed on high temperature of solid oxide fuel cell (SOFC) that operates between 800 °C and 1000 °C. However, dependability issues with typically high temperature SOFC (HTSOFC) have initiated a transferal in research pattern to emphasise on lowering the operational temperature, from 400 °C to 600 °C (Khan *et al.*, 2018). Low temperature solid oxide fuel cell (LTSOFC) operation reflects a huge range of resources particularly regarding the chosen electrolyte material that can be used while maintaining economical cost of manufacturing (Fan *et al.*, 2018).

However, there is a several unfavourable drawbacks formed from LTSOFC operation. By gradually reducing temperatures until 400 °C, the ionic conductivity of the electrolyte drops exponentially nearly three orders of magnitude, and the ohmic loss of electrolyte becomes substantial (Anwar *et al.*, 2016). Decline in temperature will also be accompanied by an increase in the losses of reaction and transport kinetics in all layers of the cell. This phenomenon also leads to an increase of in the total polarization resistance of the SOFC cathode (Ali *et al.*, 2017). There is thus a need to investigate new classes for both electrolyte and cathode materials that can maintain

high rates of oxygen transport even at reduced temperatures, and it is also significant to design a cathode with lower polarization resistance at low operating temperature of SOFC.

In order to be pragmatic in SOFC, the vital idea of LTSOFC research is to develop suitable electrolyte materials with such as based on gadolinium and ceria electrolyte which having a minimum requirement of ionic conductivity of 10^{-2} S/cm (Wang, 2012). Consequently, lower temperature operation offers much desirable advantages compares with HTSOFCs including rapid start-up and shut-down, reduces the enormous differences among the thermal expansion coefficients (TECs) of cell components, and reduction in thermal stresses in active ceramic structure cells which leads to a longer expected life time of the SOFCs system (Shimada *et al.*, 2016). Overall, LTSOFC offers a superior overall system efficiency with effective maintenance and economical cost (Sartori *et al.*, 2017). According to Shin *et al.*, (2020) revealed the potential used of 3D architectures LTOSFC with large area and powerful tools operates at low temperatures subsequently opens up opportunities to yield practical LTSOFC systems satisfying both power and durability (Shin *et al.*, 2020).

2.3 Solid oxide fuel cell systems

A solid oxide fuel cell (SOFC) cell consists of a three or multilayer ceramic composite structure as displayed in Figure 2.5 (Xia *et al.*, 2016). The gas tight solid electrolyte separates the two porous electrodes and conducts only oxygen ions from cathode to anode. At the cathode side oxygen ions are created after oxygen molecules receive electrons and migrate through the electrolyte. At the anode side, oxygen ions give off the electrons and react with fuel, which is extremely demanding (Ng *et al.*, 2018). The materials should fulfil various properties, such as electric and electro-catalytic properties, as well as chemical and thermomechanical properties.



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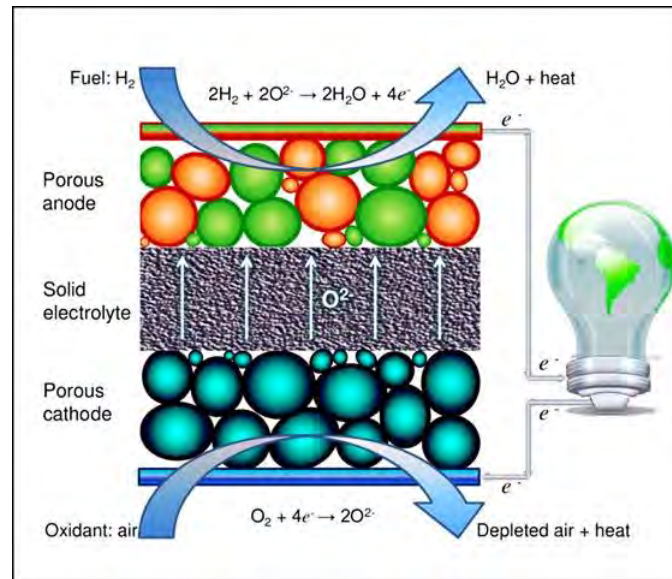


Figure 2.5: Solid oxide fuel cell schematic diagram with three layers of anode, electrolyte, and cathode (Xia *et al.*, 2016)

2.3.1 Cathode

Materials for solid oxide fuel cell (SOFC) cathodes have been well established over the past 50 years (Xia, 2018) with extensive work done to optimize conductivity, thermal stability, and to facilitate the oxygen reduction reaction. In an SOFC, the cathode functions as the site for electrochemical reduction of oxygen to occur. For this effect, the cathode must fulfil the following requirements (Mahato *et al.*, 2015):

- High electronic conductivity (~ 1000 S/cm)
- Adequate ionic conductivity (~ 0.1 S/cm)
- A matched thermal expansion coefficient (TEC) comparable to other SOFC-components ($< 20 \times 10^{-6} \text{ K}^{-1}$)
- Chemical compatibility with the electrolyte and interconnected materials
- Adequate porosity to allow gaseous oxygen to readily diffuse through the cathode to the cathode/electrolyte interface (20 – 40 %)
- Stability under an oxidizing atmosphere
- High thermal stability
- High catalytic activity for oxygen reduction reaction

As mentioned above, the mixture of ionic and electronic conductivity is required to improve the size of triple phase boundary (TPB), which is the reactive site at the cathode, where oxygen is reduced and transferred to the electrolyte (Penwell, 2014 ; Ahn, Cho, & Lee, 2019).

2.3.2 Selection of cathode materials

The main function of the cathode is to provide a reaction site for the electrochemical reduction of the oxidant. Thus, the cathode material must be stable in oxidizing environments, and have sufficient electronic conductivity (around 100 Scm^{-1}) and catalytic activity for the oxidant gas reaction at operating conditions (Chandran & Arjunan, 2015). The choice of cathode material depends on the target application, the specific ceramic electrolyte material, the desired operating temperature, and the electrochemical cell design and fabrication methods used. Furthermore, cathodes are manufactured as a porous structure to allow rapid mass transport of reactant and product gases (Xu *et al.*, 2014). SOFC cathodes should also have sufficient porosity to facilitate transportation of molecular oxygen from the gas phase to the air electrode/electrolyte interface. Cathode materials should possess high electrical conductivity and high electrocatalytic activity for oxygen reduction reaction (Chang *et al.*, 2015). Furthermore, compatibility with adjacent cell components and the chemical and dimensional stability during cell operation are very important factors. The cathode material should also be sufficiently porous to facilitate transport of oxidant to the cathode/electrolyte interface. The crucial feature for a cathode material is a high catalytic activity with respect to the dissociation of oxygen molecules that leads to low electrochemical polarization resistance. In summary, a cathode material in an SOFC has to exhibit several general properties (Yu *et al.*, 2016):

- High electronic conductivity ($>100 \text{ Scm}^{-1}$)
- Non-negligible anionic (oxygen) conductivity ($\sim 10^{-1} \text{ S/cm}$)
- Match thermal expansion coefficient (TEC)
- Suitable porosity
- Extended triple phase boundary (TPB)

In order potential cathode materials to be used in SOFC application, the incorporation of perovskite based oxides is particularly prominent. Perovskite is the original name given to calcium titanium oxide (CaTiO_3) mineral discovered in the early 1900s by the Russian scientist Gustav Rose, in honour of mineralogist Lev Perovski. It has since become a general name to describe the family of materials comprising of this structure (Abdalla *et al.*, 2018). In 1966, lanthanum strontium cobalt oxide (LSC) was the first perovskite material reported by Button and Archer to be used as cathode for SOFC. This was followed by synthesis of several perovskite materials which were subsequently tested for their usability exclusively for cathode application, and it remains the most investigated cathode material for SOFC (Yeh *et al.*, 2013; Singh *et al.*, 2017; Amar & Ahwidi, 2018). Perovskites are oxides with general formula ABO_3 in which A and B cations together possess similar net charge as depicted in Figure 2.6. The A sites are occupied with lower valence cations, mainly of rare and alkaline earth metals such as La, Sr, Ca, and Ba. Meanwhile, the B - sites are occupied with higher valence reducible transition metal cations such as Ti, Cr, Ni, Co, and Fe (Mahato *et al.*, 2015). Perovskite-type materials have been widely used as cathode materials in SOFC.

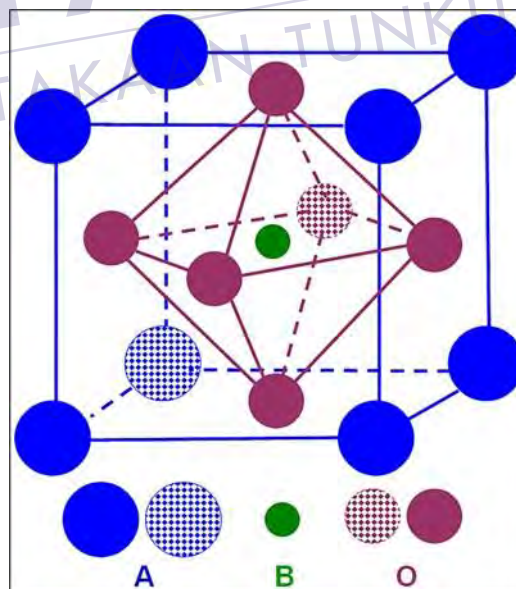


Figure 2.6: Perovskite illustration with general formula ABO_3
(Mahato *et al.*, 2015)

One of the important characteristics of the perovskite structure is its ability to conduct both electrons and ionic species, thus leading to them being labeled as Mixed

Electronic and Ionic Conductors (MIECs) (Richter *et al.*, 2009). MIEC materials have recently been investigated due to their unique ability to improve SOFC performance (Koep, 2006). Since the performance of the SOFC ultimately depends on the oxygen incorporation reaction, the reaction can proceed only where ionic and electronic species are present. MIEC materials allow simultaneous transport of both electronic and ionic species, thereby increasing the size of possible reaction sites. According to Yeh *et al.*, (2013), the entire interface from MIEC electrode (cathode) with the electrolyte demonstrates transferal of ionic oxygen species from the MIEC to the electrolyte while surface of cathode facing the air side is involved in the oxygen uptake as shown in Figure 2.7 (a-b). The uniqueness of mixed conductivity displayed from the perovskites materials permits for their use in diverse applications from catalysis to fuel cells to gas sensors (Kan *et al.*, 2016; Natile *et al.*, 2016). Therefore the understanding and optimization of perovskite conductivity as a cathode material is currently an area of high interest. MIEC perovskite represents various kind of structure, depending on the relative sizes of both A and B site cations. The distinctions of perovskite structural forms include cubic, orthorhombic, rhombohedral, and hexagonal (Sunarso *et al.*, 2008). Consequently, MIEC such as lanthanum strontium cobalt oxide, $\text{La}_{0.5}\text{Sr}_{0.5}\text{CoO}_{3-\delta}$ (LSC), lanthanum strontium cobalt ferrite oxide $\text{La}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.2}\text{Fe}_{0.8}\text{O}_{3-\delta}$ (LSCF), and barium strontium cobalt ferrite oxide, $\text{Ba}_{0.5}\text{Sr}_{0.5}\text{Co}_{0.8}\text{Fe}_{0.2}\text{O}_{3-\delta}$ (BSCF) which are exhibited as a cathode material, typically displays lower polarization and has improved performance compared to other classes of cathodes, which have already been explored (Ritzmann, 2016).

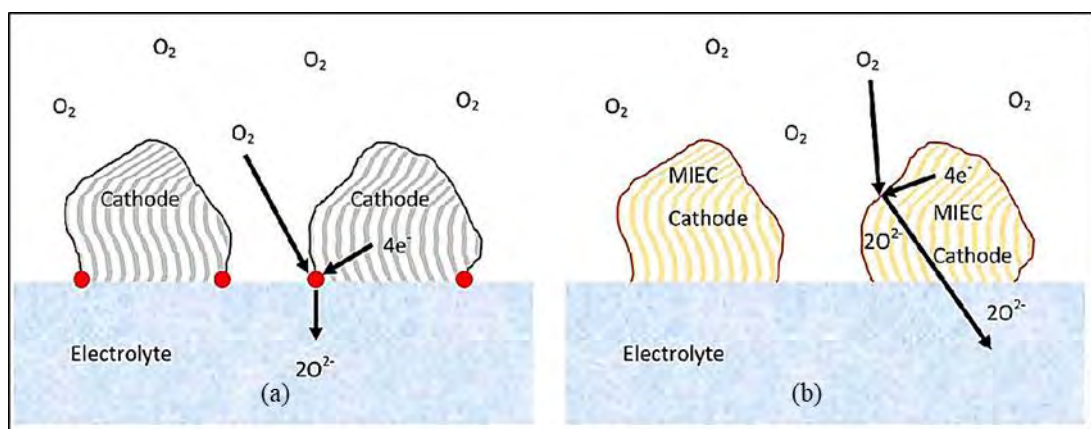


Figure 2.7: Schematic diagram transferal of ionic oxygen species to the electrolyte which from the (a) cathode material and (b) MIEC material

2.3.3 Selection of electrolytes materials

The electrolyte of solid oxide fuel cell (SOFC) is a dense layer of ceramic that should possess high ionic conductivity, that determines the power output of SOFC. As mentioned earlier, SOFC is based on the concept of oxygen ion-conducting electrolyte through which the oxide ions (O^{2-}) migrate from the cathode side to anode side where they react with the fuel to generate an electrical voltage. Enabling fast oxide ion transport in electrolyte materials is essential to facilitate the intermediate to low temperature operation of SOFCs to enhance stability and economical applications. The internal ohmic resistance (ohmic loss) of a SOFC mainly attributed by the electrolyte. These are some characteristics that the electrolyte should possess (Chronos, *et al.*, 2011; Li, 2016).

- High ionic conductivity (> than 0.1 S/cm)
- Chemically inert and structurally stable with contacting electrode, and sealing materials under ambient temperature and also under operation in highly oxidizing and reducing environment
- Negligible electronic conductivity (approximately zero)
- Must be dense with no existence of open porosity for better mechanical properties and stability
- Suitable mechanical properties
 - Thermal expansion coefficient (TEC) compatible with adjacent electrode components (cathode and anode)
- Efficient cost of starting material and fabrication

It has been noted for optimal performance of SOFC that the electrolyte should be uniformly thin to minimize loss produced from ohmic (electrolyte), and it should have high oxygen ionic conductivity. Current noteworthy electrolytes for SOFCs include fluorite structured rare earth (RE) doped ceria and RE doped zirconia with high oxygen ionic conductivities which have been frequently reported (Zuo *et al.*, 2011; Park *et al.*, 2017; Khan *et al.*, 2018), making them subjects of interest to researchers as most commonly used electrolytes in SOFCs. Yttria stabilized zirconia, YSZ has been most successfully employed as the electrolyte material for SOFCs operating at high temperatures (around 1000 °C) with ionic conductivity of 0.13 Scm^{-1} at 1000 °C

(Xia, 2018). Up to now, there has been no reliable usage of YSZ as an electrolyte on SSC based composite cathode. However, several authors have presented studies of MIEC cathode by employing YSZ electrolyte, such as LSCN with YSZ (Xu *et al.*, 2014) and LSM-YSZ (Li, 2014). To obtain low temperature SOFC, it is found that YSZ electrolyte cannot fit this request. As the operating temperature decreases, the ionic conductivity of YSZ will decrease exponentially near three orders of magnitude. In order to be applied in SOFCs, the minimum requirement of ionic conductivity is 10^{-2} S/cm and this material has been specifically appropriate for a solid electrolyte for oxygen sensors and other types of fuel cells applications.

Numerous researchers have extensively deliberated that, utmost optimal performance of SSC based composite cathode is required through combination of SSC with rare earth doped ceria based electrolytes (Guo *et al.*, 2011; Wang, 2012; Deng *et al.*, 2017). The use of an SOFC offers the choice of innumerable ceria based electrolyte from intermediate to lower temperature operations and illustrious among two types of well-known ceria based materials, which are gadolinium, Gd doped ceria (GDC) with samarium doped ceria (SDC). Xia *et al.*, (2002) investigated the properties of SSC-SDC composite cathode material and established the cathode in a composition of 70 wt.% SSC / 30 wt.% SDC30, which exhibited the highest catalytic activity. The microstructurally optimized SSC/GDC cathode composites found by Kim *et al.*, (2014) have attributed the least electrode polarization resistance in a composition of 86 wt.% SSC / 14% GDC, compared to the pure conducting SSC cathode materials.

A research from Georgia Institute of Technology (Harris, 2013) reported that composite cathode of 70 wt% SSC / 30 wt% SDC equipped from chemical vapour deposition was produced with lower value of polarization resistance. Sintered cell of NiO-SDC/SDC/SSC-SDC has attributed $0.18 \Omega\text{cm}^{-2}$ of interfacial resistance, measured between the cathode and electrolyte at 600 °C of operating temperature. Introduction of SDC materials into SSC has lower value of resistance contributed from SSC pure. Thus, ceria based electrolytes are regarded as a conceivable electrolyte applicant for the operating temperature of SOFC at 400 - 600 °C.

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