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A Study Review On Coating For Intermediate Temperature Solid Oxide Fuel Cell (SOFC) And Low Temperature Sofc Using Electro-Phoretic Depositon (EPD) As Interconnect Coating Technique

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Abstract: Solid Oxide Fuel Cell (SOFC) is considered by many developed countries as an alternative solution of energy in near future. The potential and promises shown by it has led to many developments of SOFC to maximize the productivity and durability. Different materials, design and fabrication technologies have been developed and tested to make it cost effective and stable. But the most advancement that have been made is the reduce of temperature of SOFC from high temperature to low temperature. This thesis is focused on the difference of temperature of SOFC, categorized into two, Intermediate Temperature SOFC (IT-SOFC) and Low Temperature SOFC(LT-SOFC). While both has the same interconnect coating technique, the characterization of both type of SOFC are different. The characterization of SOFC can be seen under three different tests, which are Xray Diffraction Analysis (XRD), Coating thickness test via Scanning Electron Microscope (SEM) and a test using Area Specific Resistance (ASR) via Electro Impedance Spectroscopy (EIS).

Keywords: Solid Oxide Fuel Cell (Sofc), Intermediate Temperature Sofc (It-Sofc), Low Temperature Sofc (Lt-Sofc), Xray Diffraction Analysis (Xrd), Scanning Electron Microscope (Sem). Area Specific Resistance (Asr), Electro Impedance Spectroscopy (Eis)

1. Introduction

Solid oxide fuel cell (SOFC) technology has been studied in details and is still in development as a type of power generator of many kinds of application. The promises and potential of this technology due to its production of electricity that is clean and effective from a variety of fuels is too good to be

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ignored. There are two types of SOFC, which are Intermediate Temperature SOFC (IT-SOFC) and Low Temperature SOFC (LT-SOFC). (Minh 2004)

. The main technique used to prepare the spinal coatings is Electrophoretic Deposition (EPD). It is a coating technique that is affordable to all and also the one that is used widely all over the world for the ceramic coatings' fabrication. EPD has benefits that edge them over other techniques of deposition for instance its simplicity, the capacity to deposit coating be it a complex shaped substrate, great rate deposition and easy scalability even for mass production. Moreover, the EPD technique also provide the user with an easy control of coating thickness and morphology through adjustment of the deposition time and applied potential. (Kim et al. 2011).

An LT-SOFC operates at (350-650°C). An LT-SOFC has its own benefit compared to IT-SOFC like quicker start up, and cost effective. The fast development of technology for LT-SOFC in the field of automotive relies on consumption of conventional fuels (e. g., gasoline, diesel and ethanol) that is of adapting to the infrastructure of the supply of the fuel existed. In short, LT-SOFCs are able to produce higher "well-to-wheels" fuel conversion efficiencies and as well have advantage of higher fuel density. (Singh et al. 2017)

An IT-SOFC operates at temperature higher than LT-SOFC. An IT-SOFC is the general SOFC used all around the world. It does not need any precious substance for it to operate, which make it unique than other fuel cell. Just like LT-SOFC, IT-SOFC also has its own benefit and weaknesses that can be found after testing it in laboratories. (Irshad et al. 2016)

2. Methodology

Procedures and testing in this thesis such as preparation of sample and coating technique as well as the apparatus used in it will be dissected and explained in detail. The tests will be conducted to measure the characterization of LT-SOFC and IT-SOFC by using Xray Diffraction Analysis (XRD), Scanning Electron Microscopic (SEM) and Electro Impedance Spectroscopy (EIS). The first test to characterize the SOFC would be XR which is for identification of crystalline materials and analysis of unit cell dimension. After that, coating thickness is tested by using SEM and lastly the Area Specific Resistance (ASR) is tested by using EIS. All the tests conducted are on both IT-SOFC and LT-SOFC.

2.1 Fabrication of Interconnect

As for the process of fabrication, the coating technique chosen and used was electrophoretic deposition (EPD). The method was done to manufacture the stainless steel SS430 interconnects of SOFC. Besides, there are several other requirements needed before doing the EPD technique, for example, steel substrate's preparation of aqueous suspension and the arrangement of EPD setup process.

2.2 Methods

2.2.1 Phase Analysis via X-Ray Diffraction (XRD)

To analyze the phase of crystalline structure and the identification, the test conducted was via XRD. XRD functions by using incident of X-rays to irradiate material. Next, it will measure the intensities and scattering angles of the X-rays that leave the material.



Figure 1: Equipment use for Phase Analysis

2.2.2 Surface Morphology Analysis via Scanning Electron Microscope (SEM)

The Scanning Electron Microscope (SEM) is a type of electron microscope that, by scanning with high emitting electrons, imaged the sample surface. The electrons that interact with the sample's atoms create a signal containing information about the sample's morphology characterization. Energy Dispersive X-ray spectroscopy (EDS), is a technique of analytical that is used for the elemental analysis or characterization of chemical of a sample. EDS is the most common addition to the SEM. The energy level for EDS systems varies from 1.0 to 220 KeV, which measures the composite thin films ' elementary structure or chemical characterization. When EDS detectors with thin protective layers work in high vacuum systems, allow 44 lighter elements down to B to be analyzed. The depth of study not on the main electron beam, depending on the path length of the X-ray. EDS signals may come from the depth of 0.5 μm or more as a result. It has the ability to detect the characteristic X-ray of all elements above F in the periodic table due to the pulse counting feature of the EDS system. Both of the analyses are conducted on the $(\text{MnCO})_3\text{O}_4$ powder, and on the coated SS430 before and after oxidation.



Figure 2: Equipment used for surface morphology and elemental analysis

2.2.3 Area Specific Resistance via Electrochemical Impedance Spectroscopy (EIS)

Electrochemical Impedance spectroscopy (EIS) is the technique most favorable to use in measuring the Area Specific Resistance and impedance of certain coatings of samples. Impedance spectroscopy is a method to measure the impedance of a system depending of the AC potential frequency. As it varies the frequency of the load, it is able differentiate the features of the materials in regards of its properties of electrical and physical. The data can be attained using impedance analyzer of Hewlett Packard. Moreover, Frequency range and voltage for the technique were also adjustable. SS430 foil samples with Ti evaporated onto both surfaces were heated until the temperature reaches 400°C. 1cm² area of gold is then used to cover the resultant layer on each side and then it is located in a jig at the end of the rods between two platinum spheres. The Hewlett Packard spectrum analyzer able to control the voltage, current and frequency. Consisted of 2 layers of Ti on each surface, the sample was analyzed. The sample is then heated to 450°C for a duration of 4 days in stages of increasing temperature of 50°C, 200°C, 350°C, 400°C, and 450°C and later dwelled at each temperature for 30 mins whereas spectroscopy was done between 1Hz to 106Hz with the help of A.C. amplitude of 5 mv. By dividing the total resistance by two, system resistance values were adjusted to get the resistance of one layer, then the appropriate lead resistance subtracted for that temperature in question. Lead resistances were obtained by performing EIS test without a sample at various temperatures. Lead resistance was found to run from 1.56 Ω at 300oC to 2.00 Ω at 900°C.



Figure 3: Equipment use for Area Specific Resistance (ASR)

3. Results and Discussion

3.1 Area Specific Resistance (ASR) Measurement Review

The main objective of this study is the ASR measurement and after all the characterization analysis are reviewed, the substrate undergoes Electro Impedance Test to find out the measurement of ASR by using the machine. The ASR value reviewed from the previous journals and studies are categorized into two aspects, between IT-SOFC and LT-SOFC

3.2 IT-SOFC

The first reference reviewed was using the substrate of SS304 and the coating of Co-40Mn as the coating material. Voltage of 5 V applied for this analysis. Figure 4.6 shows the measurement of ASR

of the prepared (CoMn)3O4 oxide coatings oxidized at temperature of 800 C for a period of 100 h and 300 h respectively. From the trend observed from the ASR values after 100 h and 300 h, it can be said that it is similar as the data decreases while the temperature increases. At temperature of 800°C, the ASR value measured is about 13 mΩ cm² for (CoMn)3O4 oxide coatings oxidized for 100 h and 16 mΩ cm² for 300 h, which increase a little bit with oxidation time. (Jing et al. 2013)

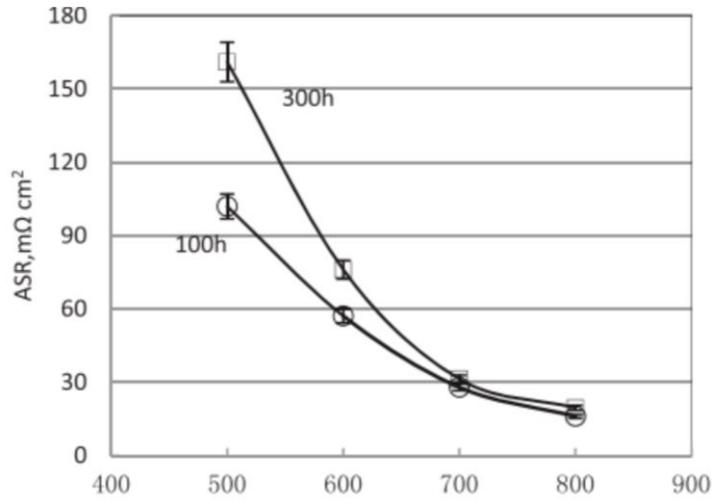


Figure 4: ASR of the as-prepared (CoMn)3O4 oxide coatings oxidized at 8000 C for 100 h and 300 h (Jing et al. 2013)

From the second journal, the study was using SS430 as the substrate coated with Co-Ni-O as the coating material. Figure 4.7 shows the measurement of ASR of bare SS430 and Co-Ni-O SS430 at from 50 h to 600 h as the oxidation time increase. As the time of oxidation increases, an oxide scale is formed that would stumble the continuous growth and therefore can be seen from the value of ASR as the bare SS430 increases slowly with time and finally at oxidation of 600h, reaches a constant value of 2.4 Ω cm². For Co-Ni-O SS430 specimen, the ASR approaches a value lower than the bare SS430 specimen which is 0.9 Ω cm². Moreover, the Co-Ni-O SS430 specimen has a low and stable ASR about 0.1 Ω cm² compared with the bare SS430 specimen which is 1.2 Ω cm² in initial 100 h. The ASR starts to increase parabolically as the oxidation time increase from 100 h to 600 h. (Cheng et al., 2017).

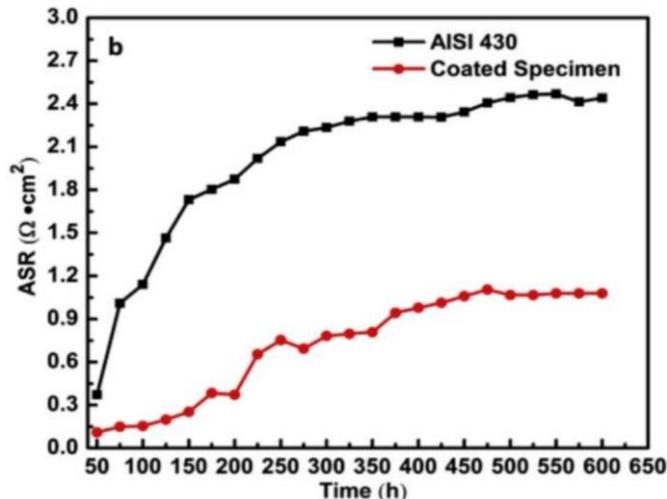


Figure 5:ASR result of bare SS430 and Co-Ni-O SS430 at different oxidation time. (Patakangas et al. 2014)

For the last journal reviewed, the ASR value of the coating and oxide at temperature of 800°C was estimated to be 6.13 m Ωcm² after a 185-h oxidation. It is well within the target ASR level of 100 m Ωcm² with the requirements for SOFC interconnecting coatings (Kim et al. 2011). Table 4.5 shows the summary of the previous researches:

Table 1: Result of ASR for IT-SOFC

Author, year	Details of SOFC	Summary of ASR analysis
(Jing et al. 2013)	-Substrate: SS304 -Coating material: Co-40Mn -Deposition time: 1.2 min -Voltage: 5 V -Oxidized for 100 h and 300 h -Temperature: 8000 ° C	- ASR for 100 h oxidation: 13 mΩ cm ² - ASR for 300 h oxidation: 16 mΩ cm ²
(Patakangas et al. 2014)	-Substrate: SS430 -Coating material: Co-Ni-O -Oxidized at 8000 ° C for 100 h	- specimen of Co-Ni-O 430 SS has a low and stable ASR (0.1 Ω cm ²) - bare 430 SS specimen has ASR about 1.2 Ω cm ²
(Kim et al. 2011)	-Crofer 22 APU as substrates -Suspension media of a mixture of ethanol and acetone are used for the EPD -Applied voltage of 20 V -Deposition time is 10min -Cu _{1.3} Mn _{1.7} O ₄ as coating material	-Coating at 800 °C is estimated to be 6.13 m Ωcm ² after 185 h of oxidation

3.3 LT-SOFC

The first reviewed journal for this aspect used the SS430 as the substrate coated by the materials of Cu/La doped Co-Mn. Voltage applied for the research is 5 V with temperature set in between 500-8000° C. Figure below shows the result for the measurement of ASR of Co-40Mn coating oxidized at 7500° C for 100 h and 20 h (1 and 2), Co-38Mn-2La coating oxidized at 7500° C for 100 h and 20 h (3 and 4), and Co-33Mn-17Cu coating oxidized at 7500° C for 100 h and 20 h (5 and 6). The value of ASR at temperature of 8000° C of Co-40Mn oxide coating is 79.2 mΩ cm² for duration at 20 h and 43.2 mΩ cm² for 100 h. As the oxidation time passes, the value can be obviously seen as decreasing. The value at 8000° C for the ASR of Co-38Mn-2La oxide coating is 19.6 mΩ cm² for 20 h and 24 mΩ cm² for 100 h. When the oxidation time passes once more, the value of ASR comes to increase once again, but slowly. ASR at 8000 C of Co-33Mn-17Cu oxide coating is 7.5 mΩ cm² for 20 h and 41 mΩ cm² for 100 h and the value increasing with oxidation time. (Guo et al., 2016)

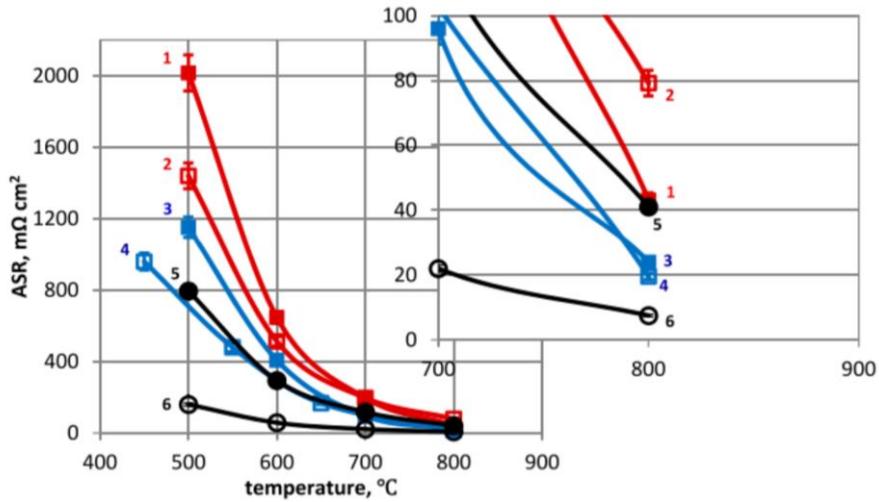


Figure 6:ASR of oxide coatings with temperature range of 500-8000 C (Guo et al., 2018)

The second journal reviewed are from the author (Zhou et al. 2008), uses SSC Perovskite as the substrate. The measurement for ASR for is taken at temperature of 550°C and 650°C. Table 4.6 shows the summary of the previous researches.

Table 2: Result of ASR for LT-SOFC

Author, date	Detail of SOFC	Summary of ASR analysis
(Guo et al., 2018)	-Substrate: SS430 -Coating material: Cu/La doped -Co-Mn coatings -Applied voltage: 5 V -Deposition time: 1.2 min -Oxidized at 750 °C in air for 20 h and 100 h -Temperature: 800°C	ASR at 8000 C of Co-40Mn oxide coating - 79.2 mΩ cm2 for 20 h - 43.2 mΩ cm2 for 100 h ASR at 8000 C of Co-38Mn-2La oxide coating - 19.6 mΩ cm2 for 20 h - 24 mΩ cm2 for 100 h ASR at 8000°C of Co-33Mn-17Cu oxide coating - 7.5 mΩ cm2 for 20 h - 41 mΩ cm2 for 100 h
(Zhou et al. 2008)	-SSC perovskite -Temperature: 650°C, 550°C, -Sc doping	At 650°C, ASR: 0.044Ω cm2 At 550°C, ASR: 0.206Ωcm2

4. Conclusion

The purpose of the study is to review journals in regards of the IT-SOFC and LT-SOFC in terms of the characteristic, advantages and disadvantages. The reviewed journals are from the study conducted 10 years ago between the year 2010 till 2020.

The first objective of this study is to review to the characteristics the low temperature SOFC and intermediate temperature SOFC. For the study, the interconnects that comes into review are SS430, SS403 and Crofer 22 APU due and the coating technique used are electro-phoretic deposition (EPD). The EPD process was followed by several analysis to determine its characteristic and grouped into two aspects, intermediate temperature and low temperature. The analyses include phase analysis by XRD and surface morphology analysis by SEM. In general, the result obtained from the reviewed journals are expected for this study. Although, not all the papers have the specific result in regards of the temperature whether it is intermediate or low, the main points were taken from the journals are really looked into.

The second objective is to elucidate the performance of IT-SOFC and LT-SOFC. This objective is reviewed base on the performance from the measurement taken from the ASR method. The effect of different temperature, intermediate or low can affect the performance of the SOFC. At intermediate temperature, (IT-SOFC) measurement is much bigger than the one recorded for LT-SOFC. This can be seen as one article that investigated IT-SOFC using substrate SS430 coated with Cu/La doped Co-Mn. The research measurement for ASR recorded as follows. At 20h, the Co-Mn oxide coating is 79.2 m Ω and 43.2 m Ω when oxidized at 100 h. The Co-38Mn-2La oxide coating are 19.6 m Ω and 24 m Ω when oxidized at 20 h and 100 h respectively and lastly for Co-33Mn-17Cu oxide coating, the ASR value is 7.5 m Ω when oxidized at 20 h and 41 m Ω when oxidized at 100 h. Another study uses SS304 as the substrate and is coated with CO-40Mn, with recorded ASR measurement of at 100h oxidation is 13 m Ω and 16 m Ω for 300 h oxidation. Lastly, the last article uses Crofer22 APU as its substrates with the ASR measurement of 6.13 m Ω cm² after 185 h of oxidation

In comparison, the value for ASR using LT-SOFC are as follows. The first articles recorded the measurement of less than $\sim 0.25 \Omega$ -cm² (based on simple linear current-voltage behavior) using substrate of Ytria Stabilized Zirconia (YSZ) at 500 C. The second journal investigated how the area specific polarization resistances (ASRs) of SSC at various temperatures. The ASR is 0.044 X cm² at 650 C. The value is slightly higher than that of Ba_{0.5}Sr_{0.5}Co_{0.8}Fe_{0.2}O_{3d} (BSCF) (0.040 X cm²) tested in our previous study also shown in figure below for comparison. As the temperature decreases, however, the ASRs of SSC increased slower than those of BSCF. The ASR is only 0.206 X cm² for SSC at 550 C, which is about 52% lower than that for BSCF. In general, the value of ASR measurement in LT-SOFC is lower than it is of IT-SOFC.

The third and last objective of the study is to identify the most effective SOFC and for this objective, the study is broken down to main aspect such as the energy produced, economic factor, and the future of the SOFC itself. Although IT-SOFC can produce much more energy than LT-SOFC, the major downside of it is that it cost much more than an LT-SOFC. This is due to the factors like the equipment needed to achieve the intermediate temperature and to maintain it until the process is complete. Scientifically, a higher temperature can SOFC can also cause poisoning on cathode and degradation on cells. Furthermore, an IT-SOFC have restrictions that blocks the development and deployment of SOFC as it uses certain specific equipment and materials that is also expensive. The materials used and the operation cost is not affordable for the long term. Thus, an LT-SOFC is the one that can be seen used worldwide in the future.

The advantage of LT-SOFC than IT-SOFC is firstly, it is more reliable as it has smaller thermal mismatch than can help easier sealing, Additionally, at lower temperature, it needs less insulation and therefore has a much lower cost. Besides, as temperature decreases, wider choice of selection for

interconnect can be used. Next, with lower temperature, the maximum theoretical fuel cell efficiency increases, in contrast to the Carnot cycle. For instance, this can be seen using CO as the fuel as the maximum theoretical efficiency of an SOFC increases from 63% at 900 C to 81% at 350 C. Lastly, and perhaps the most convincing attribute of SOFC is that it takes less time to start up with less energy, which contributes much toward making this type of energy portable and transportable applications.

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