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DESIGN & CHARACTERIZATION OF RESISTANCE-BASED CORROSION UNDER-INSULATION SENSOR

AMIRA SHAKINA BINTI AMIR SHARIFFUDIN

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By

Amira Shakina Binti Amir Shariffudin

16339

Dissertation submitted in partial fulfillment of

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Universiti Teknologi PETRONAS

Seri Iskandar

32610 Tronoh

Perak Darul Ridzuan

Malaysia

CERTIFICATION OF APPROVAL

Design & Characterization of resistance-based corrosion under-insulation sensor

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A project dissertation submitted to the

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Approved by,

____

AP DR ZAINAL ARIF BURHANUDIN

UNIVERSITI TEKNOLGI PETRONAS

TRONOH, PERAK

January 2016

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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AMIRA SHAKINA BINTI AMIR SHARIFFUDIN

ABSTRACT

Corrosion under insulation or composite wrappings has become a major concern in aging petrochemical plants. It can produce unseen pits and cracks that could lead to catastrophic failures with hours of downtime and millions of ringgits in losses. There are available sensors designed to detect this type of corrosion, however, it cannot give the actual metal loss as required by the engineers. In this work, a resistance-based corrosion sensor is proposed for this purpose. The sensor is going to be designed and developed from iron (Fe) compound thin films. This sensor is going to be integrated into a wireless system consisting of a transponder and integration. The sensor design requirement is to have a resistance of 200 Ω to 2000 Ω. The shape and features may vary depending on the requirement of the transponder. The sensor is fabricated using thermal evaporator and PCB technology with the calculated dimension of connector and finger, the resistance obtained is 1863.69 Ω. The elements within the sensor is detected by Energy-dispersive X-ray spectroscopy (EDX) is about 24% iron (Fe) with the rest consists of carbon (C), aluminum (Al) and oxygen (O_2). Upon exposure to a corrosive droplet, the resistance is modulated to few mega ohm. Correlation with LPR also showed the time that sensor element is cut off, the corrosion current becomes zero and at the similar approximate time, the resistance goes to several mega ohm. Successful development of the sensor could pave the way for a wireless sensor solution capable of sensing and monitoring corrosion under insulation.

ACKNOWLEDGEMENT

Alhamdulillah, first and foremost the author would like to thank Allah the Almighty for his blessings that gives the opportunity and ability to complete this research 'Design and Characterization of resistance-based corrosion under insulation sensor', even with many challenges faced.

I would like to grab this opportunity to show much appreciation to AP. Dr. Zainal Arif Burhanudin for his endless support and guidance in assisting me throughout this study. All his knowledge, advice and encouragement have given me so much energy and desire to complete this study. It actually has been an honor for me to learn and work under his supervision as his wide experiences in this field.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

In 1978, the National Bureau of Standards reported to the U.S. Congress that the cost of corrosion in the United States in 1975 was 70 billion dollars plus or minus 30%, and that about 15% of this loss was avoidable. This economic loss represented about 4% of the gross national product [1]. According to the European Major Accident Reporting System (MARS), corrosion was responsible for 21.5% of all incidents that were reported, and cost over $2.2 billion in property damage [2]. In a long term, corrosion will gradually reduce the performance of mechanical components and structures.

In this study, one of the most crucial problematic forms of corrosion was considered: corrosion under insulation.

Corrosion under insulation (CUI) is recognized as a major corrosion problem, which costs the oil & gas, petrochemical and chemical industries millions of dollars a year in inspection, repair and maintenance costs [3]. The covered of the insulation material over the pipe that encourage the build up of moisture and not allowing the evaporation process occur. Hence, it would accelerate the corrosion rate. As shown in Figure 2, CUI can occur under any type of insulation depending on the type of metal which is insulated or other related factors. For example, rockwool, fiberglass, or other traditional types of insulation promote corrosion, and also act as a carrier and spread the corrosion to other areas. Intruding of water through insulation could initiate the corrosion because of water may get trapped inside insulation as shown in Figure 1. Further, absence or poor condition of coating the insulation and elevated temperature may promote the CUI [4]. Thermal insulation is a necessary component of the pipeline system and is designed to save energy and control process temperatures.

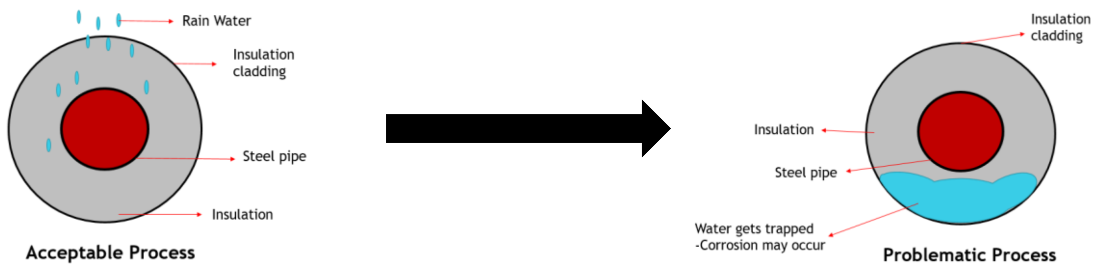


Figure 1: The CUI Cycle

In the previous research paper, common technique being introduced for corrosion inspection are non-destructive evaluation (NDE) techniques, including visual, ultrasonic, radiographic, electromagnetic and thermographic inspection. However, still try to find the best way to detect the corrosion under insulation. In order to solve this problem, a new technology will be developed to overcome the issue which is Resistance-based Sensor. All the characterization of this sensor will be investigated in every aspect such as the design and fabrication of sensor to obtain best result.

Figure 2: Pathway for CUI

1.2 Electrochemical Corrosion

Many times corrosion occurs with just one type of metal present, but the corrosion cell model is still applicable. Any metal will have microscopic impurities on its surface, especially due to its refinement and production. These impurities may form an anode and cathode known as local action cells. Anode is the part of the metal that corrodes as shown in Figure 3. At the anode, metal iron, Fe is lost to the water and becomes oxidized to Fe^{2+} ion, as stated in the Eq (1). As a result, formation of Fe^{2+} ion, two electrons are released to flow through the steel to the cathodic area. Anode is the site of oxidation process where electrons are lost. The transferred electrons will combine with water molecule and oxygen to form hydroxyl ions which is undergo reduction process in the Eq (2). At the same time, the resulting ions react with Fe^{2+} ion to form mixture of hydrous iron oxide, $Fe(OH)_2$ as shown in Eq (3) which is known as rust. Anode action causes pitting of the iron. Electrochemical cell action driven by the energy of oxidation continues the corrosion process. Illustration of corrosion mechanism is shown in Figure 3.

This type of chemical reaction called as metal oxidation. The metal atoms release electrons and become positive ions as shown in the equation below.

$$Fe \rightarrow Fe^{2+} + 2e^- \qquad \text{Eq [1]}$$

In the following equation, cathode action reduces oxygen from air, forming hydroxide ions. The type of chemical reaction that receive electrons called as reduction process.

$$2H_2O + O_2 + 4e^- \rightarrow 4OH^- \qquad \text{Eq [2]}$$

The hydroxide ion from the cathodic area reacts with the iron ion Fe^{2+}. The hydroxyl ion has an area of high electron density. Therefore, Fe^{2+} react with the electrons form hydroxyl ion to create the following reaction.

$$Fe^{2+} + 2OH^- \rightarrow Fe(OH)_2 \qquad \text{Eq [3]}$$

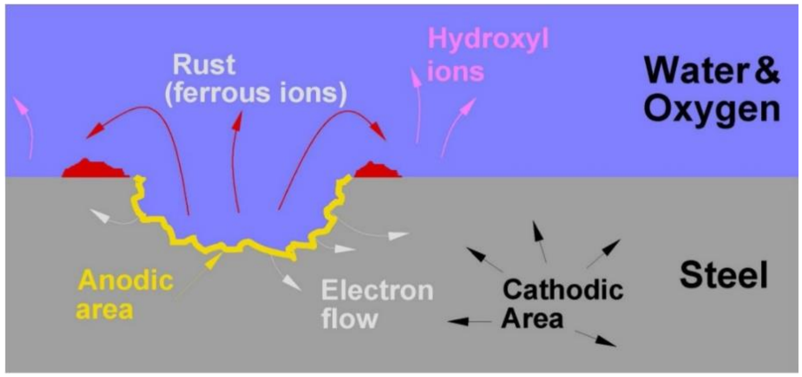


Figure 3: Schematic diagram of electrochemical process

1.3 Flow of Electrons and Electrical Current

There are four elements necessary for corrosion to occur in a corrosion cell such as anode, cathode, electrolyte and return current path between electrodes. Anode, is being placed in electrolyte consists of positively charged ion. Then, it will release the electrons to the cathodic reaction. Meanwhile, the corrosion current flowing from anodic side to the cathodic side as well as voltage or potential difference. At the same time, positive and negative charge ions are flowing within electrolyte as shown in Figure 4.

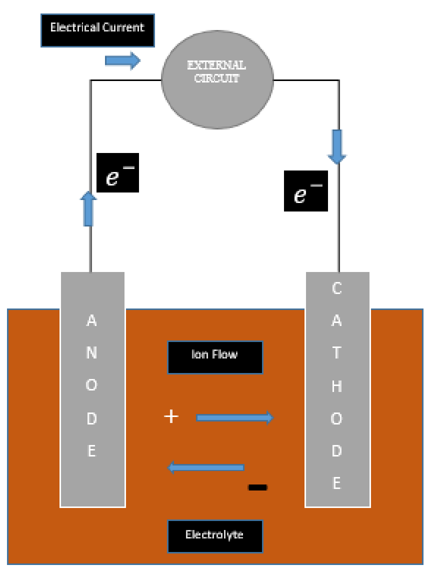


Figure 4: Corrosion cell showing flow of electrons and electrical current

1.4 Problem Statement

According to the statistics given by Exxon Mobil Chemical in September 2003, the highest incidence of leaks in the refining and chemical industries is due to CUI and not to process corrosion which contributed up to 40% to 60% of piping maintenance costs [5]. Based on local refinery company, Loss of containment (LOC) in aging petrochemical plant is more than 20% caused by CUI. Non-severe LOC can be wrapped with composite wrappings. Integrity of the pipe and the wraps, however, still need to be monitored. Therefore, sensor for corrosion under insulations/wraps need to be developed.

1.5 Objective

The objective of this research is to develop a resistance-based sensor for CUI detection. The sub-objectives are:-

1.5.1 To design a 200 Ω - 2000 Ω resistance-based sensor.

1.5.2 To fabricate the sensor using PCB or other available technology in UTP.

1.5.3 To measure the change in resistance of the fabricated sensor when it is corroded.

1.6 Scope of Study

The scope of the study is limited to the characterization of the sensor in a 0.5% concentration of NaCl solution. For actual application, the sensor should be deployed and characterized underneath the thermal insulation or composite wrappings.

CHAPTER 2

LITERATURE REVIEW

2.1 Literature Review

Corrosion under Insulation (CUI) is a worldwide problem especially to the oil and gas industry. The main cause of this phenomena is a pipeline failure. The pipeline is being covered with the insulation and the insulation itself being protected from the environment by certain materials. Therefore, the main issue is when the high operating temperature of pipe and equipment increases the activity of corrosion [6]. It will not only effect to the maintenance's schedule but also lead to serious health and safety issue. Therefore, investigator decided to create a group that able to manage the CUI problem which are European Federation of Corrosion (EFC), WP13 (Corrosion in Oil and Gas Production) and WP15 (Corrosion in the Refinery Industry). In 1990s, they formed a group known as NACE (National Association of Corrosion Engineers) to investigate and prevent the CUI problem [7].

There are many techniques of inspection that have been developed before this such as Profile Radiography, Ultrasonic thickness measurement, Insulation Removal, Infrared and Neutron Backscatter. Mostly the techniques above are non-destructive method, require high cost and generally not accurate enough. Profile Radiography is an effective evaluation method, but becomes challenging in piping because of the size. It limited to the small area of pipe and involving health risk because of radiation. Next, Linear Polarization Resistance (LPR) is one of the equipment to measure the rate of corrosion in real time. The fact that, LPR could not detect the corrosion under insulation itself [8].

Then, the other method now available is Real time X-Ray. It is proven the equipment is safe, fast and effective method of inspecting pipe. However, it could not measure the metal loss under insulation [9]. The other technique is Visual Inspection. It required to remove the insulation and it is impossible to measure remaining wall thickness. Many investigation have been made to discuss the methods to detect the corrosion under the insulation such as Magnetostrictive Sensor (MsS) Technology as shown in Figure 5 [10]. Basically, the MsS consists of a coil and a bias magnet. It can detect the corrosion under insulation without removing all involved insulation except placing the MsS Sensor when CUI suspected. It is generate and detect time-varying stresses in ferromagnetic materials [11]. The equipment used during experiment includes a signal preamplifier, an AC-to-DC converter and personal computer. As a result, MsS Technology success identify the corrosion mechanism during the experiment.

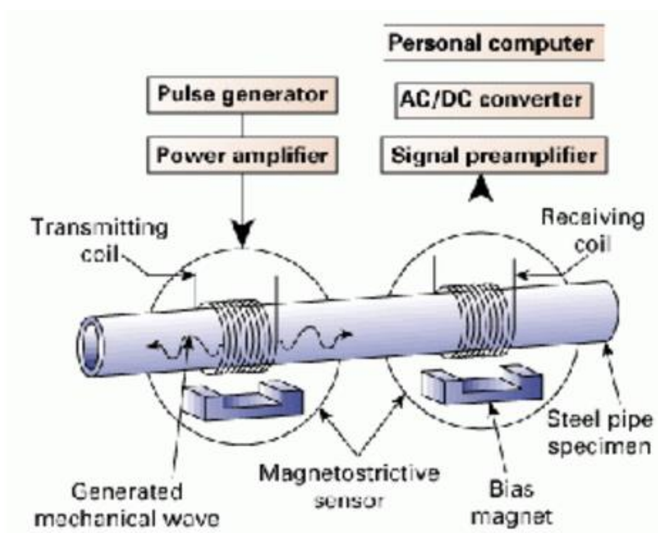


Figure 5: Magnetostrictive Sensor

Then, a widely used method is X-Ray Computed Tomography as shown in Figure 6. The X-Ray tube being attached on the pipe which mounted with screen to allow X-ray system scan every different spot area of pipe to get accurate result. It able to detect the internal and external corrosion and produce high resolution image. However, it is non-portable and cannot used for on-site inspection [12].

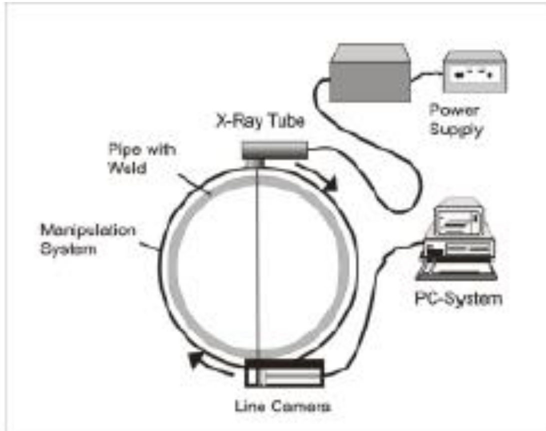


Figure 6: X-Ray Computed Tomography Principal

In the previous research, Passive Surface Acoustic Wave (SAW) Sensor [13] was developed to monitor the corrosion on steel in concrete structure as shown in Figure 7. It is an intelligent and wireless sensor. During experiment, the sensor is placing in 0.6 M NaCl SPS solution and connected with difference value of resistance. Then, reading of anodic current, resistance, voltage potential and sensor RF signal strength as corrosion sensor data collection. However, the purpose of SAW sensor is to detect the corrosion on surface of material rather than corrosion under insulation itself.

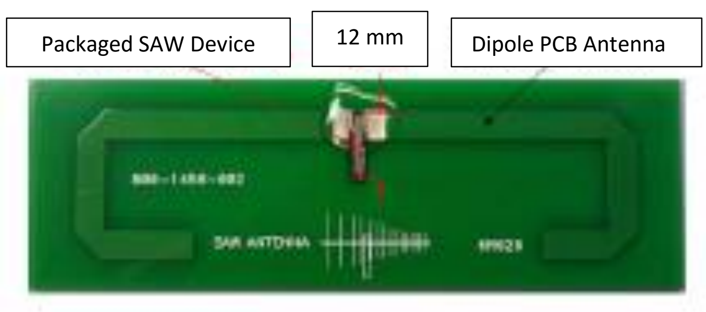


Figure 7: Passive Wireless SAW Sensor

In early work by Francois Ayello [14], he shown three different techniques to detect CUI such as Direct Impedance Measurement, Galvanic Couple with Active RFID tags and Wi-Fi based motes connected to 2-wire electrodes. They are using several techniques such as impedance measurement by using two concrete carbon steel rings placed under the insulation, development of Galvanic Sensor to trigger radio frequency identification (RFID) tag for water detection and Wi-Fi motes sensor for monitoring the pipe's impedance. The objective of these technique to create an innovative sensor using one of the methods above. Based on result, all the method success detecting the CUI. In 2000, Joe M Galbraith introduce Electromagnetic waves to detect the location of corrosion damage in insulated or buried piping [15]. However, it cannot be used to establish the amount of metal loss in an area that have suffered corrosion.

The work reported in this paper to propose a new technique that can detect the corrosion under insulation which is Resistance-based sensor. The purpose of this research to measure the change in resistance of the fabricated sensor when it is exposed to a corrosive environment. Estimation result, depends on the corrosion's rate flowing through it.

As shown in table below, advantages and disadvantage for every inspection technique in this study.

TECHNIQUES	ADVANTAGES	DISADVANTAGES
X-Ray Computed Tomography	Capability to give 3D image of specimen, showing exactly where the corrosion is located	Cannot use for on-site inspection
Magnetostrictive Sensor	Very sensitive to corrosion-type defects and can be used for 100% volumetric inspection of a long segment of piping from a single sensor location.	Require removal of insulation
Infrared	It provides temperature information to detect the presence of moisture or water in insulation.Can be applied in operation	Must be utilized another NDT(Non-Destructive) system to verify CUI
Passive Wireless Surface Acoustic Wave (SAW) Sensor	Viable, Passive, Wireless and Non-Intrusive corrosion sensor	-It is utilized only for concrete structure -RF Signals do not propagate through water or submerged concrete

Table 1: Advantages and Disadvantages of Inspection Techniques

CHAPTER 3

METHODOLOGY

3.1 Flow of the Research

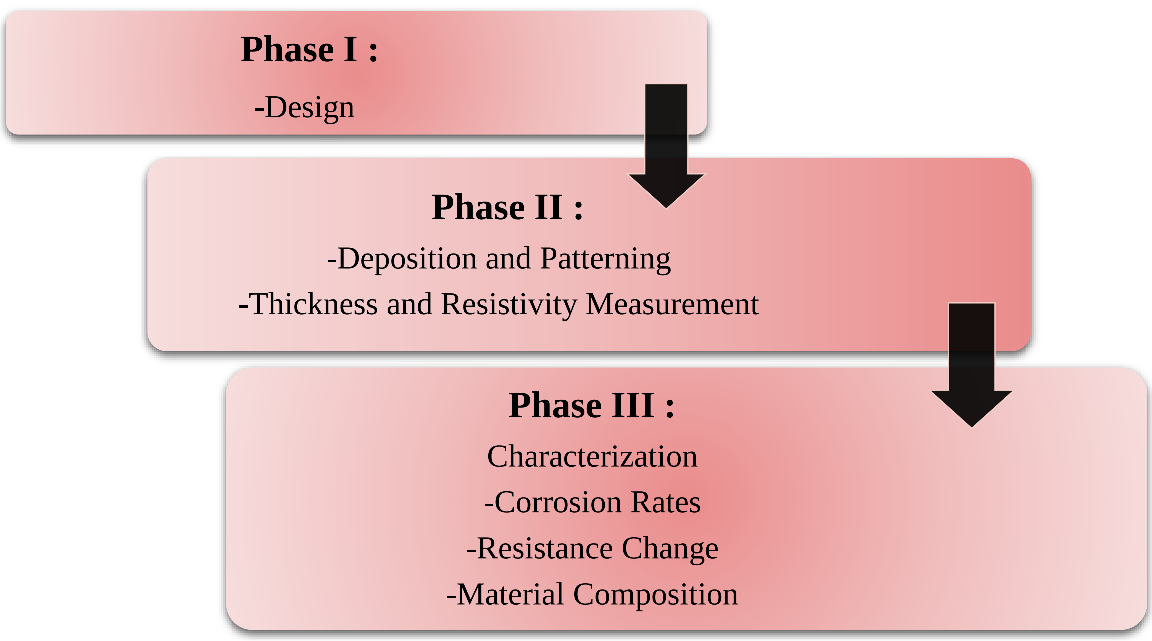


Figure 8: Flow of the Research

3.2 Phase I

3.2.1 Design Requirement

Designed a CUI sensor based on wireless transponder requirement which is in range of 200Ω-2000Ω. The material that author used in this research was iron (Fe) because it is similar with pipeline material.

3.2.2 Method to determine Resistance value

The factor of material and shape give result on the resistance of object based on the mathematical equation [4]. The resistance increases as:

- The length of the iron(Fe) film increases
- The thickness of iron(Fe) film decreases

The conducting ability of a material is often indicated by its resistivity [16].

The design are summarized in the mathematical equation called Governing Equation:

$$R = \rho \frac{L}{A} \qquad \text{Eq [4]}$$

where,

R is the electrical resistance of a uniform specimen measured in ohms

A is the sectional area in m^2

L is the length of the piece of material measured in metres, m

ρ is the resistivity of the material in Ohm(meters)

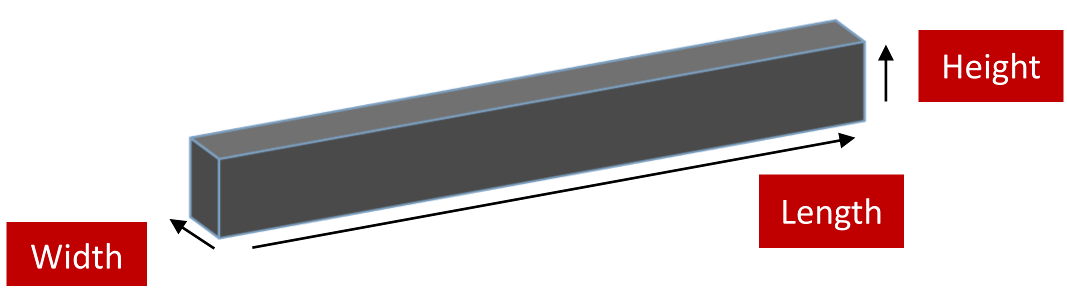


Figure 9: Dimension of iron (Fe)

3.3 Phase II

3.3.1 Deposition of iron (Fe) Powder

There have been a technique being proposed to deposit iron (Fe) powder onto FR4 substrate. The technique is using Thermal Evaporator Physical Vapor Deposition (PVD). Preparation of sample size is four unit FR4 substrate with dimension of 0.05 m x 0.05 m and being placed inside vacuum chamber. Small quantity of iron (Fe) powder is poured into Tungsten Boat, opposite with the FR4 substrate.

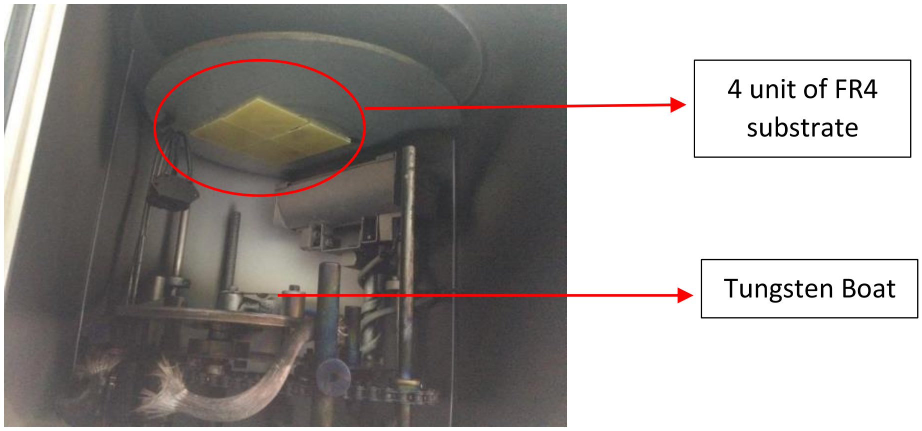


Figure 10: PVD's Vacuum Chamber

Figure 11: PVD

3.3.2 Patterning of Resistance-based Sensor

The next step is patterning the iron (Fe) deposited. The fabrication process divided by two parts which is software part and hardware part.

3.3.2.1 Software's Part

Design the sensor using Easy Applicable Graphical Layout Editor (EAGLE) software. Every dimensions in mm unit.

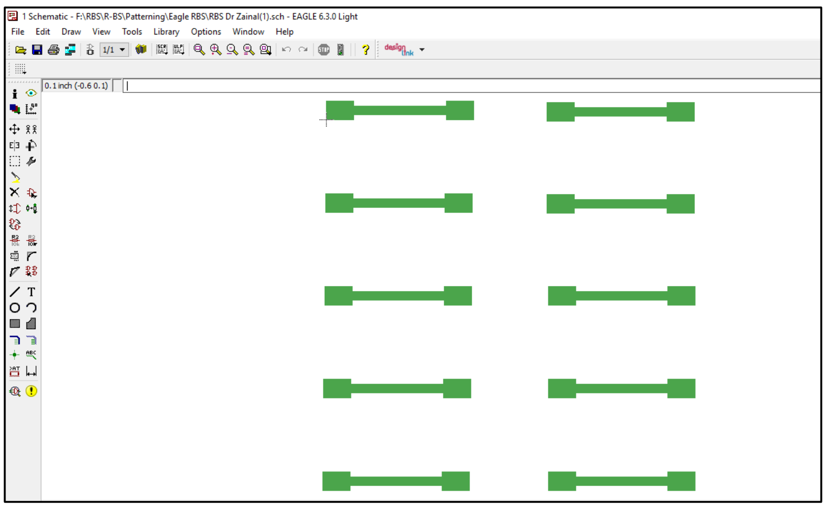


Figure 12: Schematic Diagram of RBS Sensor

3.3.3 Resistivity Measurement

3.3.3.1 Four Point Probing Measurement

Four Point Probing is a method for measuring resistivity of a semiconductor samples. In order to measure the resistivity, four points of contact must be made with the probe and the substance. In this case, the current goes through the outer probes, and the difference in voltage is measured between the two inner probes. Through this process, the resistance can be calculated. As shown in figure below, the Pro-4 is connected to a computer and power source/voltmeter. The computer automatically controls the Keithley 2400 (current/voltage source) series. A V/I value is taken and recorded.

Preparation of sample size is about 0.01m x 0.01m for placing under probe head.

Figure 13: 4 Point Probing System

3.3.4 Thickness Measurement

3.3.4.1 Scanning Electron Microscope (SEM)

SEM is a microscope that uses electrons instead of light to form an image. The SEM operator can adjust the beam to control magnification as well as determine the surface area to be scanned. The cross section area of sensor were exposed to the electron beam. Preparation of sample size is about 0.01 m x 0.01 m and being placed inside the SEM chamber.

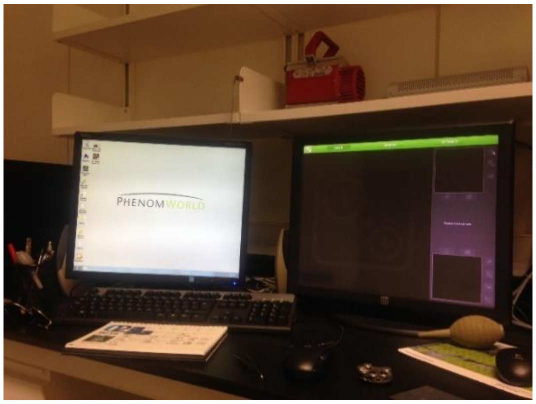


Figure 14: SEM Operation

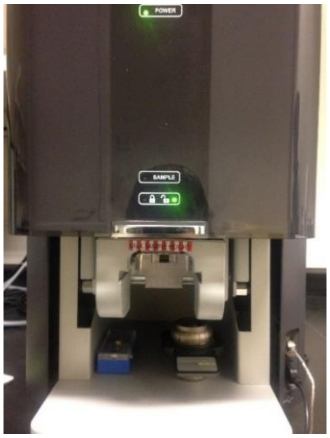


Figure 15: SEM Chamber

3.4 Phase III

3.4.1 Determination of Corrosion Rates

In this experiment, the Resistance-based sensor was tested for corrosion sensitivity in 0.5% concentration of Sodium Chloride (NaCl) solution. The top of the beaker is being covered and the ends of wire is connected with the counter wire. Then, there are two electrodes dipped in 0.5% sodium chloride (NaCl) solution which is Reference Electrode and Working Electrode. Determination of Corrosion Rate is analyzed by Applied Corrosion Monitoring Instrumentation (ACM) instrument which is included Linear Polarization Resistance (LPR) function. The equipment is presented in the figure below.

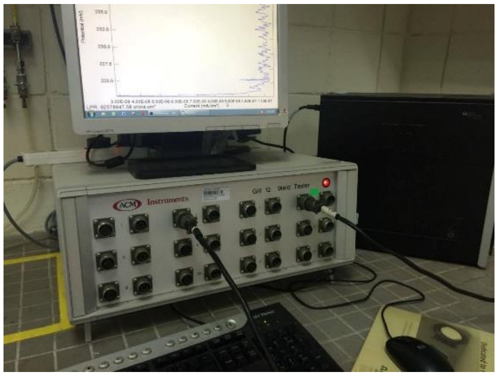


Figure 16: ACM Instruments

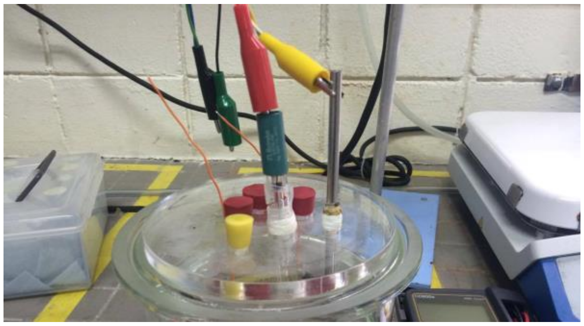


Figure 17: Set up LPR

3.4.2 Determination of Resistance Change

The author is performed automatic data logging for resistance measurement by using U1600B Handheld Oscilloscopes. To utilize this function, do the following steps:

1. Press Logger button to access data logger mode.

2. Press F1 to select the following multimeter functions for data logging:

- Ohmeter (displayed as “Ohm”)

3. After selecting the multimeter function, press F2 to select the sub-function of each multimeter function, listed as below:

- Ohmeter
  - Resistance

4. Press F3 to select the measurement type:

- Average Value

PC link 2.51 software allows author to remotely access U1600B handheld oscilloscope from a PC via USB connection to monitor on a real-time basis.

In this situation, the sensor have been connected using U1162A Alligator Clips with Handheld Oscilloscope.

Figure 18: PC and U1600B Handheld Oscilloscope

There are two samples that being prepared which is Sensor A and Sensor B.

The core wire connection is stick with Wire Glue (Conductive Glue) at both connectors. It is very important to leave the wire glue to dry sufficiently (at least 15 minutes). Then, the process was carried out with a drop of Sodium Chloride (NaCl) solution for each sample in between two wires. Then, add another drop of NaCl after it is completely dried. The resistance's flowing through Fe trace is being measured in every 10 seconds. Observe and record the resistance's changes and iron (Fe) trace.

3.4.3 Composition of Material

3.4.3.1 Energy-Dispersive X-Ray Spectroscopy (EDX)

The samples are analyzed under the EDX to view the material compositions present on it. The EDX test is done on two different condition of samples. The sample description is as follows:

Sample 1: Freshly deposited.

Sample 2: Exposed to 0.5 % NaCl solution (LPR).

The result is shown under Results and Discussion section.

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Introduction

For overall, this project is presented as having three phase implementation phases. Give an idea for resistance-based sensor design in the first phase. The fabrication process in the second phase. The third phase is the involvement in a measurement of where the characterization of the sensor is done in CCR lab and Communication Lab.

4.2 Phase I

4.2.1 Design's Outcome

Consists 1 unit of a finger and 2 units of connectors to achieve total resistance in a range of 200 Ω-2000 Ω.

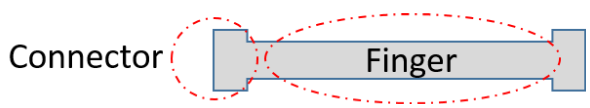


Figure 19: Design of Resistance-Based Sensor

4.2.1.1 Resistivity and Conductivity Calculation

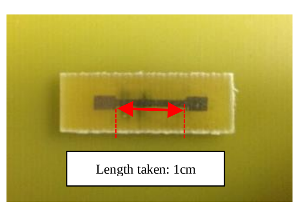


Figure 20: Resistance-Based Sensor

Resistance and Resistivity of sensor measured using Digital Handheld Oscilloscope

No of Sensor	Resistance value (kΩ) (Length of Fe trace : 0.01 m)		Resistivity ($\Omega.m$)	
Sensor 1	1.330	Max Value: 2.087 kΩ	$4.788e^{-04}\Omega.m$	Max Value : $7.513e^{-04}\Omega.m$
Sensor 2	2.087		$7.513e^{-04}\Omega.m$	
Sensor 3	1.540		$5.544e^{-04}\Omega.m$	
Sensor 4	1.455		$5.238e^{-04}\Omega.m$	
Sensor 5	0.794		$2.858e^{-04}\Omega.m$	
Sensor 6	1.110		$3.996e^{-04}\Omega.m$	
Sensor 7	1.548		$5.572e^{-04}\Omega.m$	
Sensor 8	1.290		$4.644e^{-04}\Omega.m$	
Sensor 9	1.420		$5.112e^{-04}\Omega.m$	
Sensor 10	1.765	Min Value: 0.794 kΩ	$6.354e^{-04}\Omega.m$	Min Value : $2.858e^{-04}\Omega.m$
	Average value : 1.434 kΩ		Average value : $5.161e^{-4}$ Ω.m	

Table 2: Resistance value with length 0.001 m

Based on formulae below:

To calculate Resistivity, ρ:

$$\rho = \frac{AR}{l} \qquad \text{Eq [5]}$$

where;

R is the electrical resistance of a uniform specimen of the material measured in ohms

A is the cross-sectional area of specimen measured in m^2

ρ is the resistivity if the material in Ω.m

l is the length of the piece of material measured in meters, m

To calculate Conductivity, σ:

$$\sigma = \frac{1}{\rho} \qquad \text{Eq [6]}$$

where;

ρ is the resistivity if the material in Ω.m

σ is the conductivity of the material in siemens per metre, S/m

Sensor 1

$$\rho_1 = \frac{[(0.001m \times 3.60e^{-06}) \times 1330\Omega]}{0.01m} \quad \text{Eq [7]}$$

$$\rho = 4.788e^{-04}\Omega.m$$

$$\sigma_1 = \frac{1}{4.788e^{-04}} \quad \text{Eq [8]}$$

$$\sigma_1 = 2.088e^{03}s/m$$

Sensor 2

$$\rho_2 = \frac{[(0.001m \times 3.60e^{-06}) \times 2087\Omega]}{0.01m} \quad \text{Eq [9]}$$

$$\rho_{2\,=}\ 7.513e^{-04}\Omega.m$$

$$\sigma_2 = \frac{1}{7.513e^{-04}} \quad \text{Eq [10]}$$

$$\sigma_2 = 1.331e^{03}s/m$$

Sensor 3

$$\rho_3 = \frac{[(0.001m \times 3.60e^{-06}) \times 1540\Omega]}{0.01m} \quad \text{Eq [11]}$$

$$\rho_{3\,=}\ 5.544e^{-04}\Omega.m$$

$$\sigma_3 = \frac{1}{5.544e^{-04}} \quad \text{Eq [12]}$$

$$\sigma_3 = 1.803e^{03}s/m$$

Sensor 4

$$\rho_4 = \frac{[(0.001m \times 3.60e^{-06}) \times 1455\Omega]}{0.01m} \qquad \text{Eq [13]}$$

$$\rho_{4\,=}\, 5.238e^{-04}\Omega.m$$

$$\sigma_4 = \frac{1}{5.238e^{-04}} \qquad \text{Eq [14]}$$

$$\sigma_4 = 1.909e^{03} s/m$$

Sensor 5

$$\rho_5 = \frac{[(0.001m \times 3.60e^{-06}) \times 794\Omega]}{0.01m} \qquad \text{Eq [15]}$$

$$\rho_{5\,=}\, 2.858e^{-04}\Omega.m$$

$$\sigma_5 = \frac{1}{2.855e^{-04}} \qquad \text{Eq [16]}$$

$$\sigma_5 = 3.498e^{03} s/m$$

Sensor 6

$$\rho_6 = \frac{[(0.001m \times 3.60e^{-06}) \times 1110\Omega]}{0.01m} \qquad \text{Eq [17]}$$

$$\rho_{6\,=}\, 3.996e^{-04}\Omega.m$$

$$\sigma_6 = \frac{1}{3.996e^{-04}} \qquad \text{Eq [18]}$$

$$\sigma_6 = 2.502e^{03} s/m$$

Sensor 7

$$\rho_7 = \frac{[(0.001m \times 3.60e^{-06}) \times 1548\Omega]}{0.01m} \qquad \text{Eq [19]}$$

$$\rho_{7\,=}\, 5.572e^{-04}\Omega.m$$

$$\sigma_7 = \frac{1}{5.572e^{-04}} \quad \text{Eq [20]}$$

$$\sigma_7 = 1.794e^{03} s/m$$

Sensor 8

$$\rho_8 = \frac{[(0.001m \times 3.60e^{-06}) \times 1290\Omega]}{0.01m} \quad \text{Eq [21]}$$

$$\rho_{8\,=}\, 4.644e^{-04}\Omega.m$$

$$\sigma_8 = \frac{1}{4.644e^{-04}} \quad \text{Eq [22]}$$

$$\sigma_8 = 2.153e^{03} s/m$$

Sensor 9

$$\rho_9 = \frac{[(0.001m \times 3.60e^{-06}) \times 1420\Omega]}{0.01m} \quad \text{Eq [23]}$$

$$\rho_{9\,=}\, 5.112e^{-04}\Omega.m$$

$$\sigma_9 = \frac{1}{5.112e^{-04}} \quad \text{Eq [24]}$$

$$\sigma_9 = 1.956e^{03} s/m$$

Sensor 10

$$\rho_{10} = \frac{[(0.001m \times 3.60e^{-06}) \times 1765\Omega]}{0.01m} \quad \text{Eq [25]}$$

$$\rho_{10\,=}\, 6.354e^{-04}\Omega.m$$

$$\sigma_{10} = \frac{1}{6.354e^{-04}} \quad \text{Eq [26]}$$

$$\sigma_{10} = 1.573e^{03} s/m$$

Total average of Resistivity, ρ:

$$Avg_{\rho} = 5.161e^{-4}\ \Omega.\mathrm{m}$$

Total average of Conductivity, σ:

$$Avg_{\sigma} = 2.060e^{3}\ \mathrm{S/m}$$

The expected total resistance value is calculated as follow:

where:

- ρ(Ωm) → Resistivity of iron(Fe)
- H(m) → Thickness of iron(Fe)
- W(m) → Width of iron(Fe)
- L(m) → Length of iron(Fe)
- R(Ω) → Resistance value

Calculating resistance of **finger** Eq [27]:

ρ (Ωm) = 5.161e-04
H (m) = 3.60e-06
W (m) = 0.001
L (m) = 0.01

$$R = p\frac{L}{A} \rightarrow 5.161\text{e-}04 \times \left(\frac{0.01}{3.60\text{e}-06 \times 0.001}\right) = R \qquad \text{Eq [27]}$$

R = 1433.61 Ω

Calculating resistance of **connector** Eq [28]:

$$R = p\frac{L}{A} \rightarrow 5.161\text{e-}04 \times \left(\frac{0.003}{3.60\text{e}-06 \times 0.002}\right) = R \qquad \text{Eq [28]}$$

R = 215.04 Ω

Thus,

The resistance value of 2 unit of connectors:

$$R = 215.04\ \Omega \times 2\ \text{unit}$$

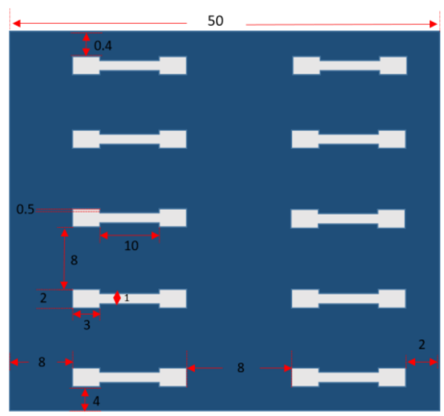
$$R = 430.08\ \Omega$$

ρ(Ωm)	H(m)	W(m)	L(m)	R(Ω)
Finger				
5.161E-04	3.60E-06	0.001	0.01	1433.61
Connector				
5.161E-04	3.60E-06	0.002	0.003	215.04

Table 3: Finger and Connector's Measurement

Finger Unit	Connector Unit	Resistance of Finger(Ω)	Resistance of Connector(Ω)	Total Resistance Value(Ω)
1	2	1433.61	215.04	1863.69

Table 4: Total resistance value of RBS



*All dimension in mm (unit)

Figure 21: Resistance-Based Sensor's Measurement

The resistance value of a material varies from 0.794 kΩ - 2.087 kΩ due to uneven deposition of iron (Fe) powder. However, the total resistance may achieve in range of 200Ω - 2000Ω with the current design.

4.3 Phase II

4.3.1 Deposition of iron (Fe) powder

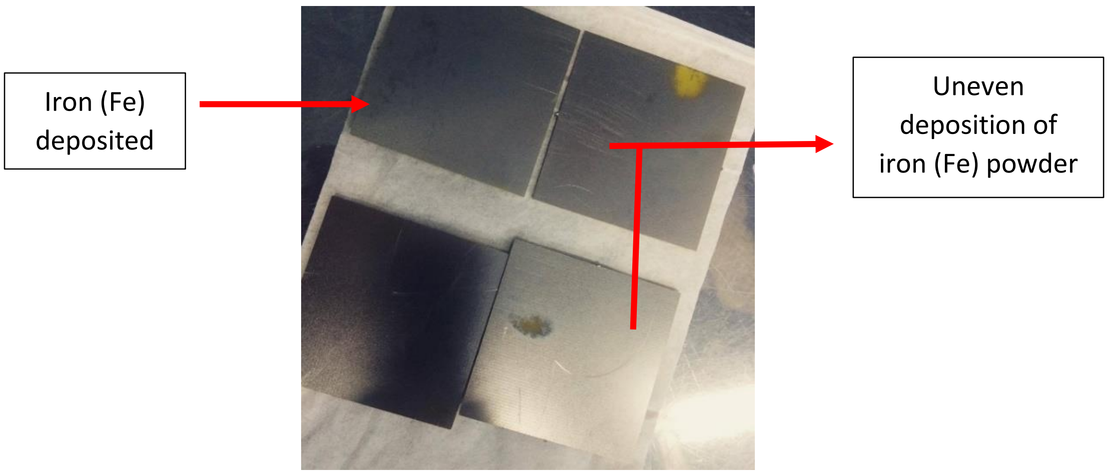


Figure 22: Iron (Fe) powder deposited onto FR4 Substrate

Preparation of sample size is 0.05m x 0.05m. The remaining two is unevenly deposited onto the FR4 substrate.

4.3.2 Patterning of iron (Fe) deposited

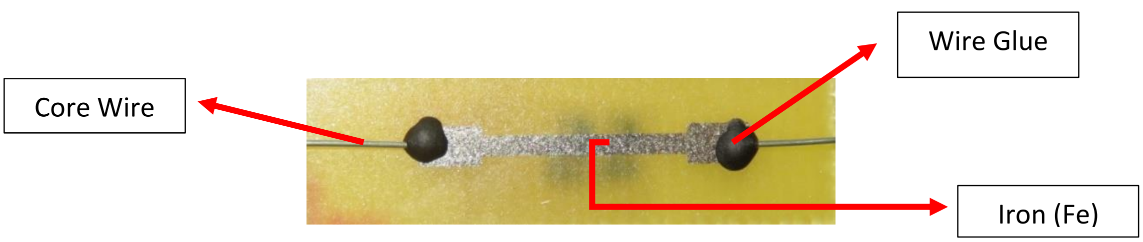


Figure 23: Resistance-Based Sensor (After deposition)

Patterning process is done at PCB Lab. 10 unit of sensor per sample is performed with dimension of:

- **Connector** : 0.003m x 0.002m
- **Finger** : 0.01m x 0.001m

4.3.3 Resistivity Measurement

4.3.3.1 4 Point Probing Measurement

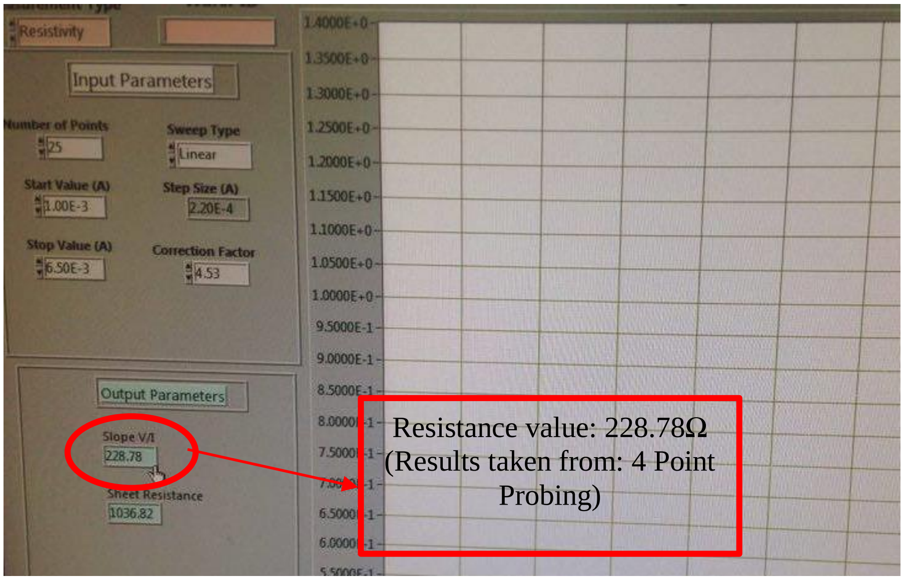


Figure 24: Slope V/I

- Given the formula to calculate Resistivity, ρ:

$$\rho = \frac{V}{I}\left(\frac{A}{L}\right)[\Omega.\text{m}] \qquad \text{Eq [29]}$$

where

V	=	Voltage (V)
I	=	Current (A)
A	=	Cross-Sectional Area (m^2)
L	=	Length (m)
ρ	=	Resistivity [Ω.m]

- Given details:
  - ➢ Resistance Value (Slope$\frac{V}{I}$) = 228.78 Ω
  - ➢ Needle Spacing, L = 0.00159 m

$$\rho = 228.78\left(\frac{0.001 \times 3.60e{-}06}{0.00159}\right) \qquad \text{Eq [30]}$$

Resistivity, ρ = 5.18e-04 Ωm

The resistivity of 4 Point Probing Measurement is approximately same with the average value of resistivity that have been calculated.

$$\% \text{ Error} = \left|\frac{5.181e^{-4} - 5.161e^{-4}}{5.181e^{-4}}\right| \times 100$$

$$= 0.37\ \%, \text{less than } 1\% \ (<1)$$

4.3.4 Thickness Measurement

4.3.4.1 Scanning Electron Microscope (SEM)

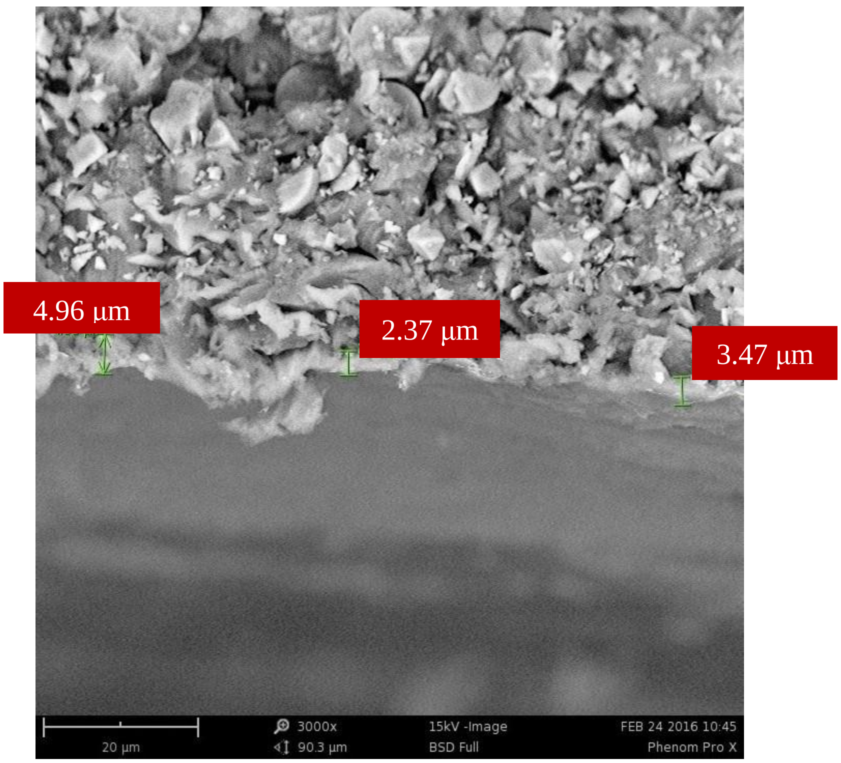


Figure 25: SEM micrograph

Average value of thickness:

$$\text{Avg} = \frac{4.96\ \mu\text{m} + 2.37\ \mu\text{m} + 3.47\mu\text{m}}{3} \qquad \text{Eq [31]}$$

Avg value = 3.60e-06 µm

4.4 Phase III

4.4.1 Corrosion Rates

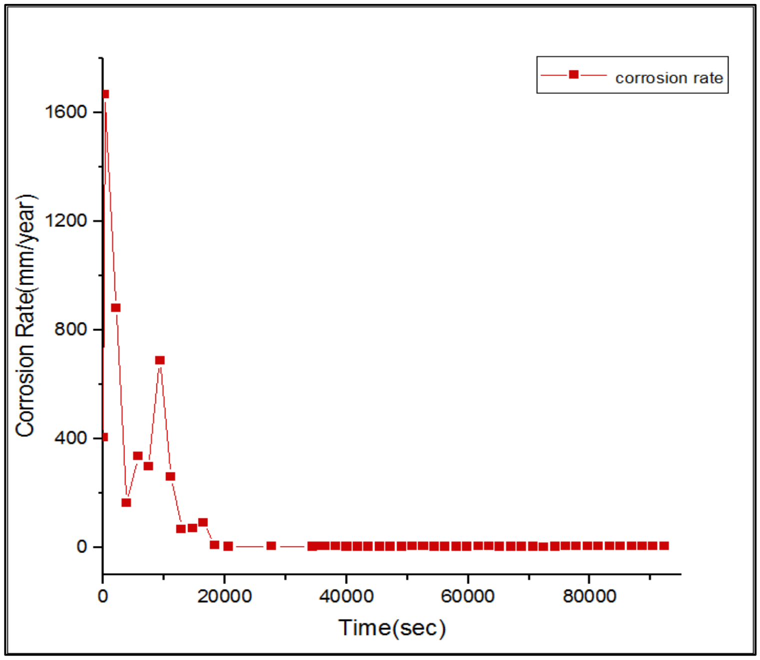


Figure 26: Corrosion rate from LPR method of iron (Fe) in 0.5% concentration of NaCl

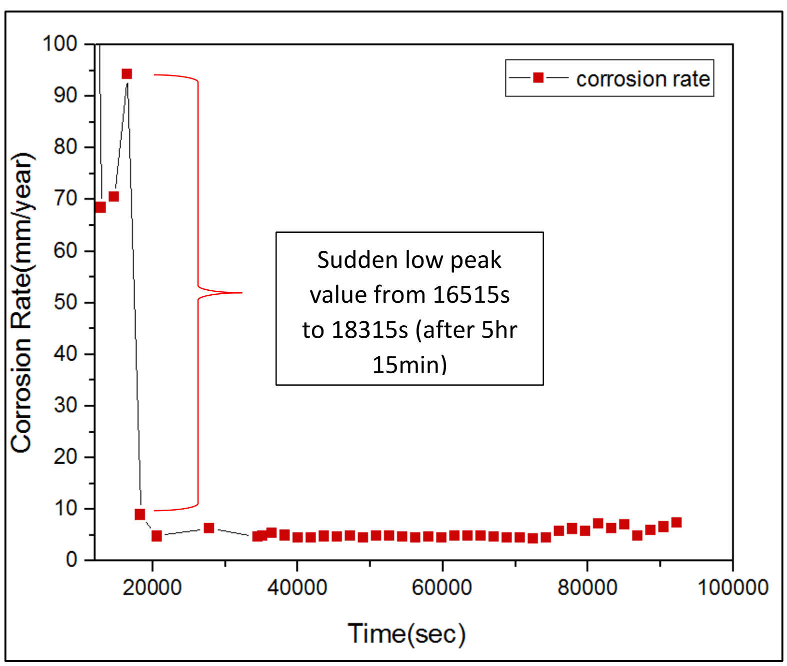


Figure 27: Corrosion rate from LPR method of iron (Fe) in 0.5% concentration of NaCl

Total Time taken of experiment: 25 hour 37 minutes

Initial Reading of Resistance Value: 1.171 kΩ

As shown in the figure above, it clearly shows that the corrosion rate slowly increasing from zero seconds to 313.5 seconds. Then, it is slowly drops every 30 minutes and keep increasing back in period of time. At 9314 seconds, the corrosion rate gradually decrease until it reach 14715 seconds. However, after 5 hours 15 minutes, sudden low peak value of corrosion rate at 18315 seconds until the end of the experiment. It shows that the corrosion process slow compared than the first 5 hours reading due to iron (Fe) trace is totally cut off. Hence, the resistance will be high due to low of corrosion rate.

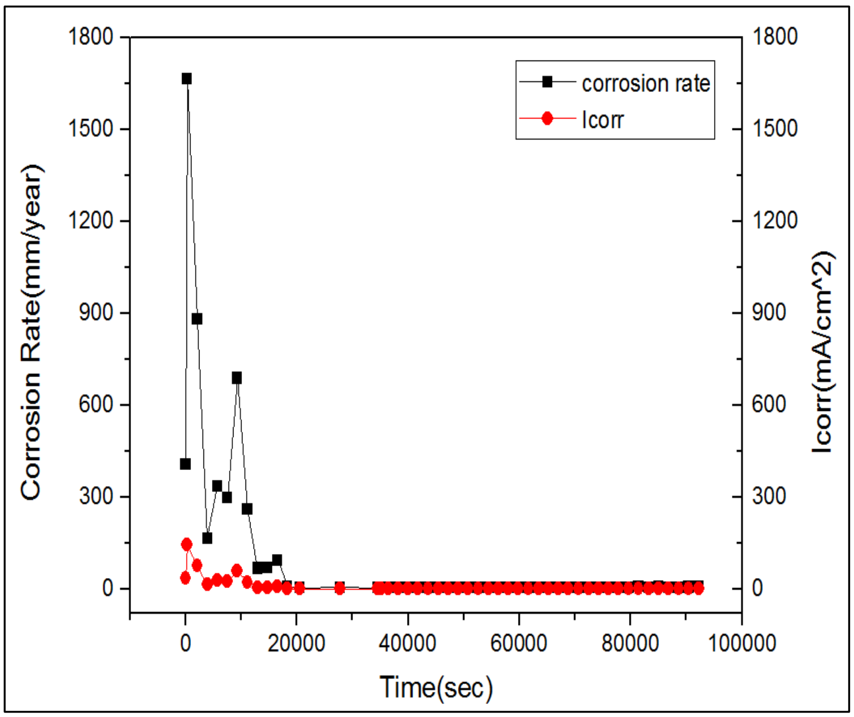


Figure 28: Corrosion rate and Corrosion current from LPR method of iron(Fe) in 0.5% concentration of NaCl

The results show that the corrosion current becomes zero at 18315s due to iron (Fe) trace is completely cut off.

The electrochemical nature of corrosion can be illustrated as shown in figure below.

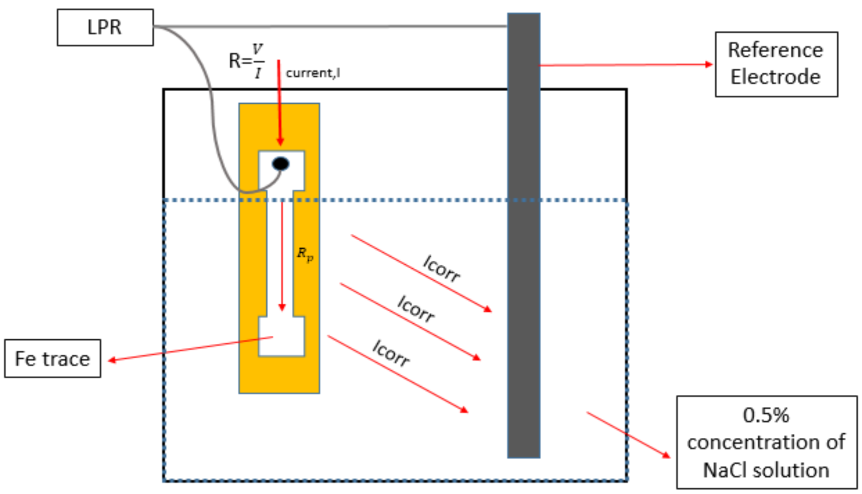


Figure 29: The electrochemical reaction beween Fe and NaCl

As shown in table below, the LPR's result after 25 hours 37 minutes.

Time(sec)	LPR ($ohm.cm^2$)	Icorr (mA/cm^2)	Corrosion Rate (mm/year)	Total metal loss (mm)	Potential (mV)
0	0.742085	35.153	407.43	0	-302.46
313.15	0.1814709	143.75	1666	0.0169627	-280.36
2113.6	0.3431229	76.028	881.16	0.0685423	-294.04
3913.8	1.8181	14.347	166.29	0.0782751	-299.78
5714	0.9005736	28.967	335.72	0.097924	-318.58
7514.1	1.0104	25.816	299.21	0.1154354	-308.44
9314.4	0.4388836	59.439	688.9	0.1557572	-305.43
11114	1.1562	22.561	261.48	0.1710616	-289.5
12914	4.4176	5.9051	68.44	0.1750673	-281.31
14715	4.2873	6.0845	70.52	0.1791949	-266
16515	3.2073	8.1335	94.268	0.1847123	-255.81
18315	33.742	0.773122	8.9604	0.1852368	-238.75
20540	62.929	0.4145395	4.8045	0.1855842	-228.57
27771	47.791	0.5458478	6.3263	0.1870717	-240.29
92228	40.78	0.6396847	7.4139	0.1979026	-248.96

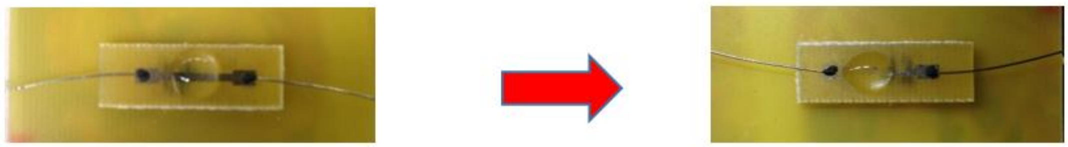
Maximum rate of corrosion process

Minimum rate of corrosion process

Table 5: LPR's Result

4.4.2 Resistance Change

4.4.2.1 Result of Sensor A



A drop of NaCl solution

Dried of NaCl solution

Figure 30:Process Flow

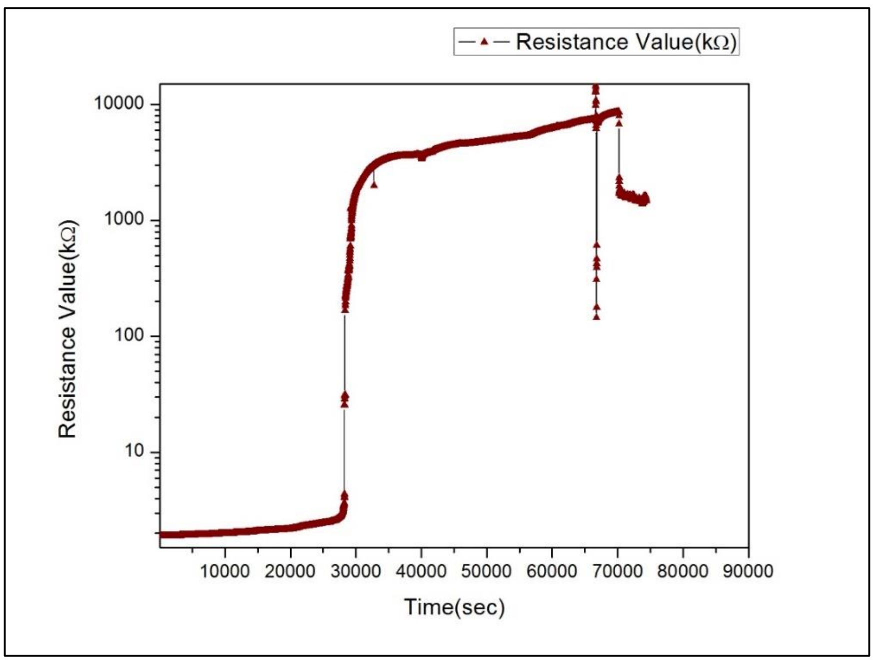


Figure 31: Resistance value versus time taken in seconds

- 1st: Add a drop of NaCl solution at 0s
- 2nd: Add a drop of NaCl solution at 7300s
- 3rd: Add a drop of NaCl solution at 15240s
- 4th: Add a drop of NaCl solution at 22480s
- Fe trace cut off at 28370s

- 5th: Add a drop of NaCl solution at 29040s
- 6th: Add a drop of NaCl solution at 70200s

Total Time taken of experiment: 20 hour 39 minutes

Initial Reading of Resistance Value: 1.905 kΩ

Based on the graph above, the resistance value is slightly increased 0.001 kΩ for every 10 seconds from zero second until 28640 seconds. Then, it can be seen that the resistance gradually increased with increasing immersion time until it reach certain times. This is due to the continuous electrochemical behavior of iron (Fe) react with corrosive agent, NaCl. At 28360 seconds, the thickness of Fe trace slowly reduced due to ionization process. Then, Fe trace is cut off within 10 seconds from 31.77kΩ to 167.4kΩ. Next, author add a drop of Sodium Chloride (NaCl) solution at 70200 seconds. Therefore, the resistance rapidly increases until it modulate few mega ohms and it started to follow the first's one hour reading of resistance. Here is a plot showing the trend of resistance value for first's 12 hour.

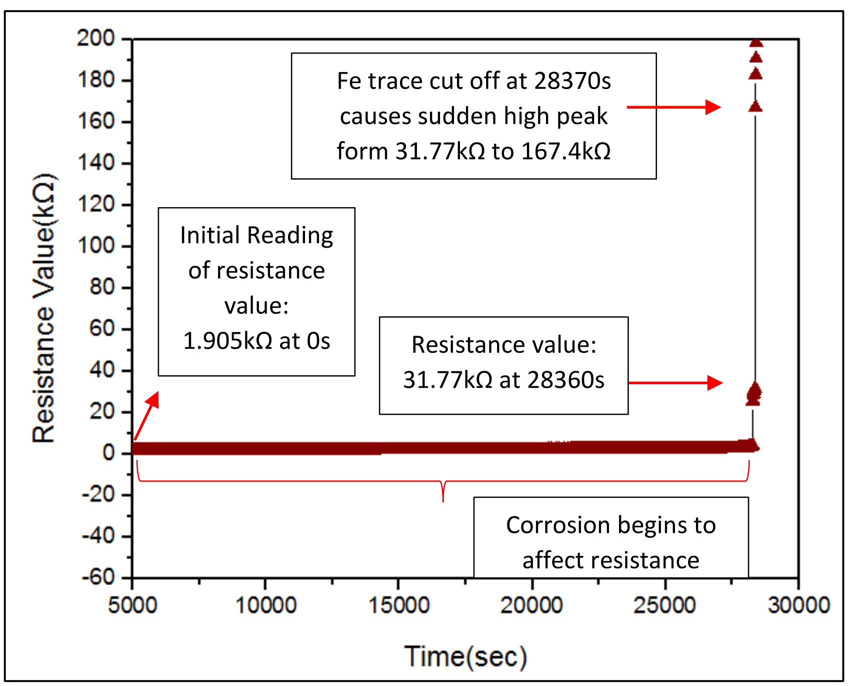


Figure 32: Resistance value versus time taken in seconds

4.4.2.2 Result of Sensor B

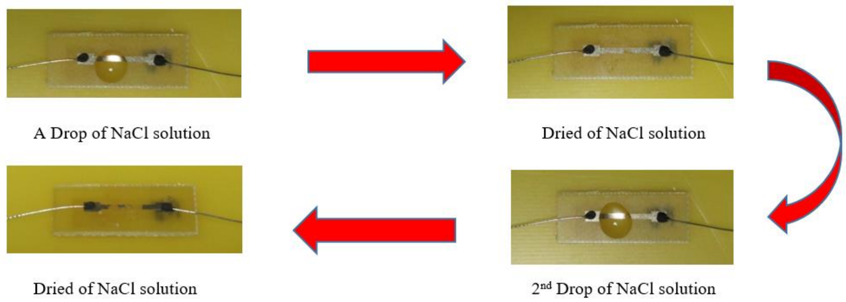


Figure 33: Process Flow

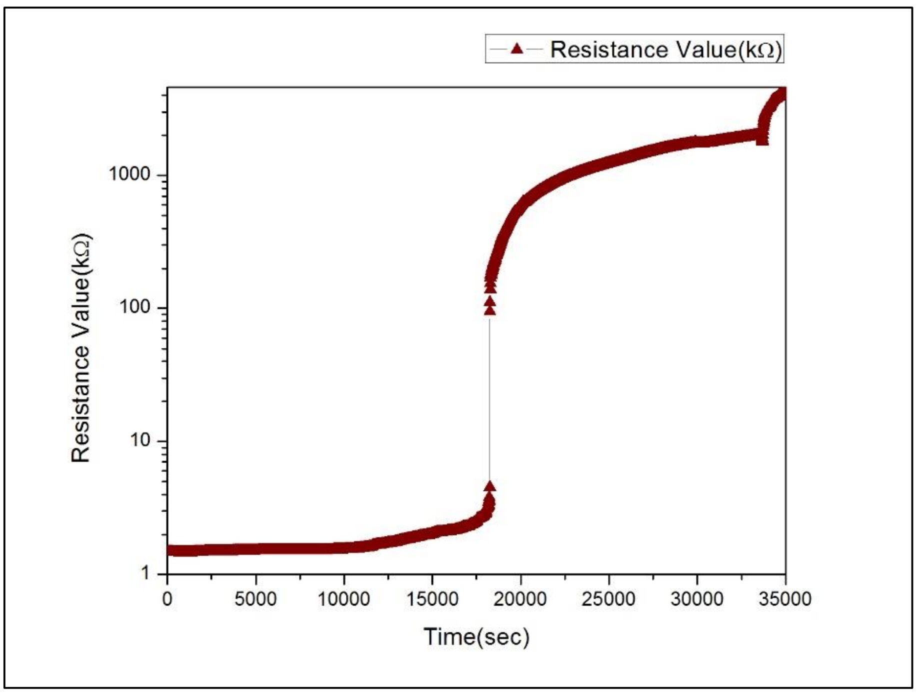


Figure 34: Resistance value versus time taken in seconds

- 1st: Add a drop of NaCl solution at 980s
- 2nd: Add a drop of NaCl solution at 16530s
- Fe trace cut off at 18260s

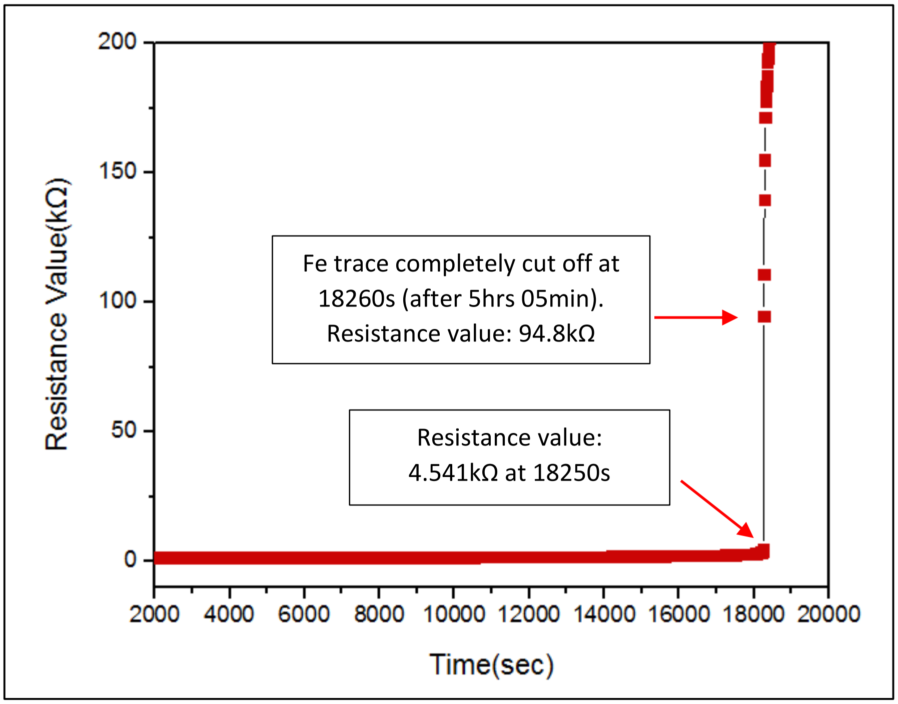


Figure 35: Resistance value versus time taken in seconds

Total Time taken of experiment: 10 hour 22 minutes

Initial Reading of Resistance Value: 1.48 kΩ

Figure 34 shows the resistance value for the first 16 minutes (970 seconds) is consistently within the range (1.48KΩ - 1.47KΩ) before drop a Sodium Chloride (NaCl) solution. At point 960 seconds, the resistance's value starts to increase after dropping a drop of Sodium Chloride (NaCl) solution. This is because, iron (Fe) initially dissolves actively in the solution. Next, it can be seen that the resistance gradually increased when Sodium Chloride (NaCl) solution partially dried until it reach certain times. Then, author add another drop of Sodium Chloride (NaCl) solution at point 16530 seconds. With increasing potential, the resistance value rises in which there is a sudden sharp rise at point 18260 seconds. It is clearly shown in Figure 35 that the Fe trace is completely cut off at the respective time. Afterwards, the resistance is rapidly increases until it reach few mega ohms.

4.4.3 Composition of Material

4.4.3.1 Freshly Deposited

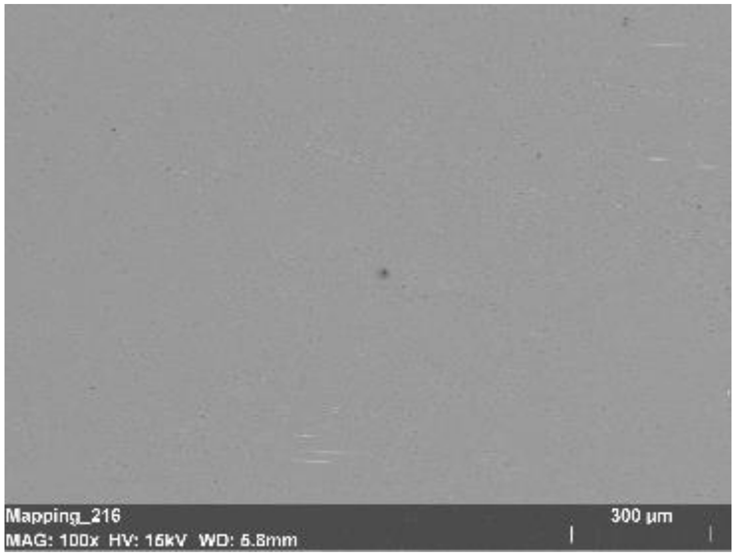


Figure 36: EDX micrograph of iron after deposition process with magnification of 100

Spectrum: Point

Element	AN	Series	norm. C [wt.%]	Atom. C [at.%]
Carbon	6	K-series	37.68	56.45
Iron	26	K-series	24.19	7.79
Oxygen	8	K-series	22.56	25.37
Aluminium	13	K-series	15.58	10.39
		Total:	100.00	100.00

Table 6: Quantitative analysis result

The microstructure of sample after deposition process is shown in the figure above. The data from table indicate the fact that the weight concentration of iron (Fe) is lower than the carbon. However, it shows other containment material which is aluminium. This is because, during deposition process, the vacuum chamber of PVD has been used for the aluminium process. Thus, it may effect to the result.

4.4.3.2 Exposed to 0.5% NaCl solution

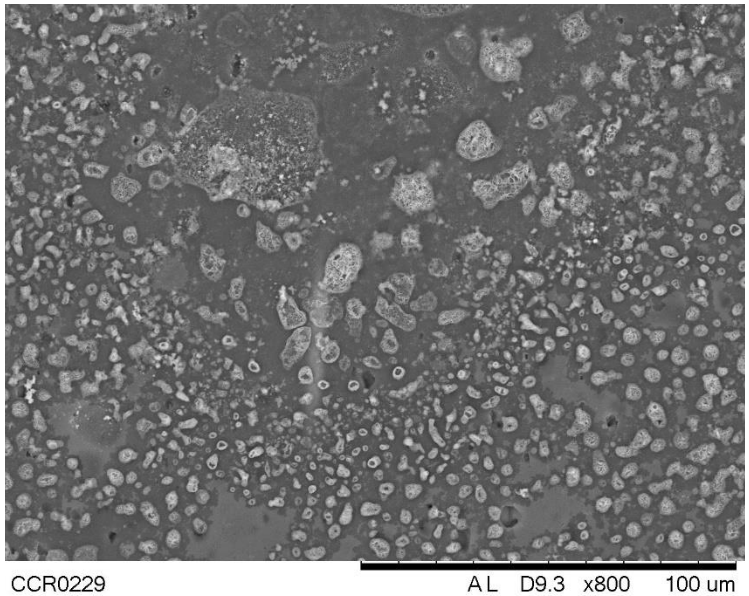


Figure 37: EDX micrograph of iron after exposes to NaCl solution with magnification of 800

Spectrum: Point

Element	AN	Series	norm. C [wt.%]	Atom. C [at.%]
Carbon	6	K-series	41.32	55.31
Oxygen	8	K-series	33.33	33.50
Iron	26	K-series	12.70	3.66
Aluminium	13	K-series	12.65	7.54
		Total:	100.00	100.00

Table 7: Quantitative analysis result

The microstructure of sample after exposes to 0.5% concentration of NaCl solution is shown in the figure above. The data from table indicates the fact that the iron (Fe) is still existed but in low weight concentration compared to the previous result.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The main conclusions that can be drawn from this research are the following:

- The resistance value varies from 794Ω - 2.087Ω due to uneven deposition process.

- The resistance of sensor may achieve in range of 200Ω - 2000Ω which is the total resistance is 1863.69Ω.

- The microstructure of sample was examined by Energy-dispersive X-ray spectroscopy (EDX). The EDX test is done on two different condition of samples. From these results, it was confirmed that the weight concentration of iron (Fe) is lower than the carbon. However, observed that 15.58 % amount of aluminum is formed after deposition process. In the second sample, there is a significant difference of weight concentration for every elements after exposed to 0.5% NaCl solution. It shows that, the iron (Fe) is still existed but in low weight concentration compared to the previous result.

- As can be seen from Figure 28, sudden low peak value of corrosion rate as well as corrosion current becomes zero due to iron (Fe) trace is completely cut off after 5 hours 15 minutes. The same trend was obtained when resistance were plotted against the time (second). It shows that, the resistance of sensor is rapidly increases with increasing exposure time until it reach a few mega ohms.

- As a conclusion, the resistance-based sensor can detect corrosion.

5.2 Future Work

The resistance-based sensor is going to be integrated with the Transponder wireless system as shown in figure below.

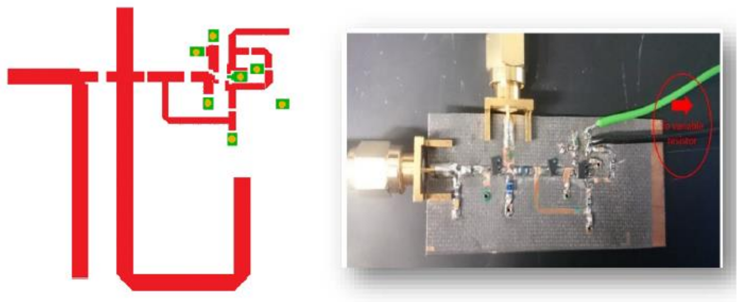


Figure 38: Transponder Circuit

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APPENDICES

1) Project Key Milestone (1st Semester)

2) Project Key Milestone (2nd Semester)

3) Gant Chart (1st Semester and 2nd Semester)

NO	Activity/Week	FYP 1														FYP 2													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	FYP1: Briefing 1																												
2	Selection Of Project Topic																												
3	FYP1: Briefing 2																												
4	Literature Review																												
5	Submission Extended Proposal																												
6	Proposal Defence																												
7	Design and Develop Fe Films																												
8	Testing																												
9	Fabrication and Patterning of Fe Films																												
10	Submission of Draft Interim Report																												
11	Submission of Interim Report																												
12	Determination of Corrosion's rate																												
13	Pre-SEDEX																												
14	Analysis of Experiment's Result																												
15	Submission of Draft Report																												
16	Submission Of Dissertation(Soft Bound)																												
17	Submission Of Technical Paper																												
18	Oral Presentation																												
19	Submission of Project																												

4) Compilation of journal related with various techniques of CUI

Title/Author	Abstract/Brief Description	Methods/Parameters	Observation/Conclusion
Title: Corrosion Under Insulation Detection Technique Year: 2013 Name: Sally Nicola Affiliation: Texas A&M Journal: NACE	-Study CUI using X-ray Computed Tomography (Non-Destructive Method)	**Exp. 1: The Accuracy of the X-Ray Tomography System** -Detecting holes with different diameters, 10 mm, 8 mm & 3 mm. **Exp. 2: The Effect on the insulation Material on the Output** -Type of insulation with diff densities: 1.High-density foam 2.Low density foam 3.Fiberglass -Wrapped around pipe with aluminum casing to test whether it could affect the X-Ray penetration through the pipe **Exp. 3: Can it detect internal corrosion?** -Scans at diff locations to capture internal corrosion	**X-Ray Computed Tomography:** -Visualize inside of opaque object -Can detect internal & external corrosion -Does not require insulation to be removed -High resolution -Can provide 4D imaging -X-Ray Tomography has shown more accurate results than Real Time Radiography Results: **1.Exp. 1** -2D imaging might be sufficient for corrosion(internal &external) detection rather than 3D **2.Exp. 2** -All diff materials does not affect/no changes based on the results **3.Exp. 3** -Successful detecting the internal corrosion **Results:** -Gave accurate results -Require to achieve faster scan time for large scale-plants -Not Portable/Cannot used for on-site inspection

Title: Integrated Sensor Networks For Corrosion Under Insulation: Monitoring, Cost Reduction, And Life Extension Strategies Year: 2011 Name: Francois Ayello Affiliation: Detnorske Veritas Res & Inno Journal : NACE	-Three different techniques to detect CUI: **1.Direct Impedance measurement** **2.Galvanic Couple with active RFID tags** **3.Wi-Fi based motes connected to 2-wire electrodes** -To create an intelligent sensor networks using one of the technique -how to model corrosion risks using large amount of data generated by sensor network	**Methods:** 1. Impedance measurement by using two concrete carbon steel rings placed under the insulation 2. Development of Galvanic Sensor to trigger radio frequency identification(RFID) tag for water detection 3. Wi-Fi motes sensor for monitoring the pipe's impedance **Techniques:** **1.Measurement impedance using metallic rings and potentiostat :** -Two electrodes placed under insulation and connect with potentiostat to measure the impedance **2.Impedance Measurement using Wi-Fi motes :** -Motes connect wirelessly to a server which collects and uploads data via a web connection -Mote inputs are connected to two thin wires electrodes(electrodes=wetness inputs, corrosion inputs and coating degradation inputs parameter) **3.Modified RFID tags connected to galvanic couple for moisture detection** -RFID tag power supply was removed and replace by Cu-Mg Galvanic Couples -Galvanic Couple wet -> RFID give signal/certain values -Galvanic Couple dry -> RFID signal inactive	-All can detect CUI but only one can be used in smart sensor networks **Results:** **1.Metallic Rings and Potentiostat:** -Direct way to detect the moisture under insulation **2.Wi-Fi Motes:** -Motes continuously collect data and store information -Available data to anyone -Easy to be structure up **3.RFID tags:** -To detect the presence of water under pipe insulation -Requires The use of an RFID reader to detect when the sensors are active.

Title: A New Inspection Technology for Detecting Corrosion Under Insulation Year: 2000 Name: Joe M Galbraith Affiliation: Profile Tech Journal: NACE	-Introduce Electromagnetic Waves (Non-Destructive)	**Methods:** **1.Dual pulse(Two synchronized pulses)** – To excite the two ends of a piping segment **2.Single pulse** – Magnetically attached pulser into the piping at either end of the tested interval	-To detect the location of corrosion damage in insulated or buried piping(CUI) **Results:** -Cannot be used to establish the amount of metal loss in an area that has suffered corrosion.
Title: Magnetostrictive Sensor Technology Proven in Process Application Year: 2000 Name: Hegeon Kwun Affiliation: Southwest Research Institute Journal : Oil & Gas Journal	-Demonstrate new technology , Magnetostrictive Sensor (MsS) technology	**Magnetostrictive Sensor (MsS):** 1.Consists of a coil and bias magnet 2.The transmitting coil applies a time-varying magnetic field that generate elastic wave via Magnetostrictive effect 3.Receive the signal using preamplifier, AC-to-DC Converter and a personal computer	-It can detect corrosion under insulation without removing all involved insulation and tubing -Produces elastic wave that travels in the wall of material -Sensitive to variations in wall thickness -Monitor processes such as corrosion -Can generate and detect time-varying stresses in ferromagnetic materials -No direct physical contact to the structural surface **Results:** -The signal shows the discrete defect signals which produced by erosion mark -Success identify the location of defects during inspection of heat exchanger tubes

Title: Corrosion Detection By Fiber Optic AE Sensor Year: 2009 Name: Yuichi Machhijma Affiliation: Sumitomo Chemical Co. & Ltd Journal : Acoustic Emission Group	-Introduced Fiber Optic AE Sensor	**Experimental Setup:** 1.Fiber Optic Sensor(FOD) located at 300,2000,3000 mm away on the pipe 2.FOD installed on the pipe via U-Shape bolt and directly attached on welded flange with a C-clamp 3.Using AE(Acoustic Emission) to view the result during FOD testing	-Non-Electric Principle **Results:** -Success detect and evaluate AE signal caused by corrosion progression using FOD
Title: A novel electrochemical Method for monitoring Corrosion Under Insulation Year: 2014 Name: Naing Naing Aung Affiliation: Nanyang Technological Universities Singapore Journal : ResearchGate	-Introduce an Electrochemically Integrated Multi-Electrode Array namely as Wire Beam Electrode(WBE) method -Combine with Noise Signature Analysis	**Method:** 1.WBE sensor fabricated from 100 metal wires by embedding wires in epoxy resin 2.Measuring the corrosion potential noise and galvanic current over WBE surface using AutoAC	-To monitor the penetration of corrosive species under CUI **Results:** 1.Proven to be capable monitoring CUI combined with noise signature analysis
Title: Passive Wireless Surface Acoustic Wave Sensors For	-Introduce Wireless and Powerless Surface Acoustic Wave (SAW) sensor	**Methods:** 1.Set up all the equipment's for data analysis 2. Accelerated Corrosion Experimental Setup and Testing	-**Focus:** Monitor on concrete structure -Passive and wireless sensor system -This paper presented measured data that

Corrosion Monitoring Of Steel In Concrete Structures Year : 2015 Name : A.R Steele Affiliation: Florida Department of Transportation Journal : NACE		-Placing the specimen with SAW sensor in 0.6M NaCl SPS Solution. Connected with different resistance value (<100Ohm and 3KOhm) with +5V Power Supply 3.Corrosion Sensor Data Collection -Measure the anodic current, resistance, voltage Potential and Sensor RF signal strength	demonstrated the functionality of passive and wireless corrosion detection. Future refinements such as size sacrificial links are needed.