Influence of Heat Treatment Process on Carbon Steel Pipe Corrosion in The Presence of Carbon Dioxide, CO_2 and Acetic Acid, HAc

by

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CERTIFICATION OF APPROVAL

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Approved by,

____

(Dr. KEE KOK ENG)

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TRONOH, PERAK

Jan 2015

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 acknowledgments, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

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MUHAMMAD ADHAM BIN ADNAN

ABSTARCT

Carbon steel is the cheapest and the most versatile materials for pipeline. During fabrication of the pipeline, carbon steel will undergo specific heat treatment to serve their specific purposed. Heat treatment is a process of modification of the microstructure by heating and cooling of the metals to achieve desired physical and mechanical properties. In carbon dioxide (CO_2) environment, the corrosion product, Iron Carbonate ($FeCO_3$) will form as the iron ion (FE^{2+}) in the carbon steel reacted with the bicarbonate anion (CO_3^{2-}) from the dissolved CO_2. Previous study has shown that difference in heat treatment will give difference in corrosion rate. In the presence of Acetic Acid (HAc), it will act as catalyst for CO_2 corrosion where it will affected the pH value. Thus it will cause the corrosion product to from faster. The objective of this project, it is to study the influence of heat treatment process on carbon steel pipe corrosion in the presence of Carbon Dioxide and Acetic acid. The experiments were conducted by using an X-65 Carbon Steel pipe. The sample have undergo further heat treatment process which were annealing, normalizing and quenching. The corrosion rate measuring technique used was Linear Polarization Resistance (LPR) and Weight Lost (WL). Three-electrode glass cell experiment and immersion test were conducted. An investigation by using Optical microscope was conducted to determine the microstructure of each samples. For annealing, the microstructure appear to be coarse pearlite, normalizing to be fine pearlite while quenching form a martensite microstructure. From the analysis of corrosion rate, annealing showed the lowest corrosion rate compared to other type of heat treatment. This concluded that annealing have the highest corrosion resistance. The result also showed that 1000ppm of acetic acid caused the corrosion rate to increase up to 50 percent for each sample regardless of the heat treatment applied.

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Finally my deepest gratitude to my family that have been my backbone in supporting me for my future success. They have been my motivation for me during thick and thin. With their prayers and encouragement, I have manage to keep moving forward to my future success.

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CHAPTER 1.
INTRODUCTION

1.1 Background

In oil and gas industries, crude oil is transported by using carbon steel pipes. According to Ramsdale (2006) and NACE (1979), there are three main reasons for choosing carbon steel pipe. First is the safety and durability of the carbon steel. It is subjected to high in resistance to shock and vibration. Furthermore, it also environmentally friendly as it will not rot even buried underground. Finally, it is cost effective as it is cheaper to stainless steel and easier to handle in shaping and forming the dimension desired.

Referring to NavaChing (2009), heat treatment is a process of heating and cooling metals to achieve desired physical and mechanical properties. These properties are achieved through modification of the microstructure of the metal. There are various kinds of heat treatment which give different microstructure profile such as coarse pearlite, fine pearlite and martensite. This is due to various kind of specification in transporting the crude oil where some a higher strength of pipe is needed and some to be much more flexible.

However, carbon steels are very prone to corrosion in environments containing carbon dioxide, CO_2. In CO_2 environment, the iron ion (Fe^{2+}) in the pipe will reacted with the bicarbonate anion (CO_3^{2-}) from the dissolved CO_2 gas. It will cause a formation of an Iron Carbonate ($FeCO_3$) layer which can form a protective layer. If the layer disturbed, it can causes rises to the failure of pipelines and equipment which resulting in economic loss and accidents. This is due to, leakage of the pipe that have the potential to induce fire accident and environmental pollution.

Gunaltun and Larrey (2000) stated that weak organic acids usually present in oil and gas production and transportation systems. Acetic acid (HAc), one type organic acid, has contributes up to 50–90% of the organic acids. It has been approved by Gulbrandsen (2007) that HAc can cause corrosion failures resulted from localized attack such as pitting corrosion and mesa attack. It also been cited that according to Crolet (1999), Carbon Dioxide and Acetic acid can be considered as contributor for the deterioration of pipeline in oil and gas industries.

1.2 Problem Statement

To successfully utilize the carbon steel pipe, appropriate heat treatment need to be selected based on the application of the carbon steel pipe. For example, annealing is mainly focusing on the elbow of a pipelines, normalizing mainly on long transportation pipe while quenching more focusing on high small high pressure pipe such as injection point. Previous study has shown that difference in heat treatment will give difference in corrosion rate. Carbon steel pipelines for transporting the crude oil are exposed to the Carbon Dioxide, CO_2 environment. Therefore, these pipelines are prone to CO_2 corrosion. In CO_2 environment, the corrosion product, Iron Carbonate ($FeCO_3$) will form as the iron ion (FE^{2+}) in the carbon steel reacted with the bicarbonate anion (CO_3^{2-}) from the dissolved CO_2. In the presence of Acetic Acid (HAc), it will act as catalyst for CO_2 corrosion where it will affected the corrosion rate.

1.3 Objective and Scope of Study

The objective of this project is to study the influence of Heat Treatment Process on Carbon Steel Pipe Corrosion in the presence of Carbon Dioxide (CO_2) and Acetic Acid.
The scope of study are:-

- To study different heat treatment (annealing, normalizing and quenching) on microstructure surface profile.
- To study the influence of different heat treatment on Carbon Dioxide corrosion resistance.
- To study the influence of the presence of Acetic Acid in Carbon Dioxide corrosion resistance.

CHAPTER 2.
LITERATURE REVIEW

2.1. Heat Treatment

As according to NavaChing (2009), heat treatment is a process of heating and cooling metals to achieve desired physical and mechanical properties through modification of their crystalline structure. There are many variables that been considered such as the temperature, length of time, and rate of cooling after heat treatment. These variables will give a significant impact to the properties of the metal. Heat treatment processes are done to increasing the strength or hardness, to increase the toughness, improving ductility and maximizing corrosion resistance (NavaChing, 2009).

Carbon steel is one of metal alloy. It formed from a result of combining iron and carbon together. The common maximum percentages of composition in carbon steel are 1.65 percent for manganese and 0.6 percent for copper and silicon. For the copper percentage, it must be at least 0.4 percent (Wojes, 2014). The example of heat treatment that been applied to oil and gas industries are annealing, normalizing, and tempering (Smith, 2006, Wojes 2014 and Ramsdale 2006).

2.1.1 Annealing

As discussed from Smith (2006), Wojes (2014) and Ramsdale (2006), annealing is one of heat treatment for softening materials. This process is for required changes in properties. It is important in machinability, mechanical or electrical properties, and dimensional stability. Annealing process consists of heating the steel to high temperature or near the critical temperature above the recrystallization temperature. The heated steel is allowed to cool slowly for the annealing process to occur correctly. The microstructure for full annealing is coarse pearlite.

Annealing has several uses. It can increase ductility and alleviate internal stresses that contribute to brittleness. Annealing also increase toughness and homogeneity of metals. Annealed carbon steel pipe in industry is used for bending, flaring, injection tubing and to cover electrical furnace.

2.1.2 Normalizing

Normalizing is a heat treatment process also for softening material. The main different of annealing and normalizing is, normalizing does not produce the uniform material properties compared to annealing (Ramsdale, 2006). The process of normalizing consist of a material undergo heating in a specific temperature and then cool down process which is faster than annealing. For normalizing, the microstructure of the metal is fine pearlite.

Ramsdale (2006) also add that, normalizing will refines the grain size and improves the uniformity of microstructure and properties of hot rolled steel. Normalizing is used in plate mills and in the production of large forgings. For example are railroad wheels and axles. This process is less expensive than annealing.

2.1.3 Quenching

As discussed by Smith (2006) and Ramsdale (2006) quenching process is one of heat treatment that typically been carried out is quenching. The process is done by heating the steel above the eutectoid temperature undergo rapid cooling to obtain the desired mechanical properties. Quenching process is similar to normalizing and annealing process, the different is only on the cooling rate.

There are several type of quenching such as water quench and oil quench. Water quench is a cooling rate by immersed into water while oil quench in oil. For water quench, the microstructure is mainly martensite while oil quench it martensite and pearlite.

2.2 Influence of Heat Treatment.

According to NavaChing, (2009), there are three main structures of steel austenite, ferrite and martensite. For normalizing and annealing and quenching the different of the cooling rate can be seen clearly in the Figure 1. From the graph, there are five different cooling paths from A to D. For all the processes, the steel is heated until pass the Eutectoid temperature. For annealing the cooling process is very slow compared to normalizing. At the bottom of the cooling paths is a description of the resultant steel structure. It becomes apparent that the cooling path chosen determines the microstructure and properties of the steel.

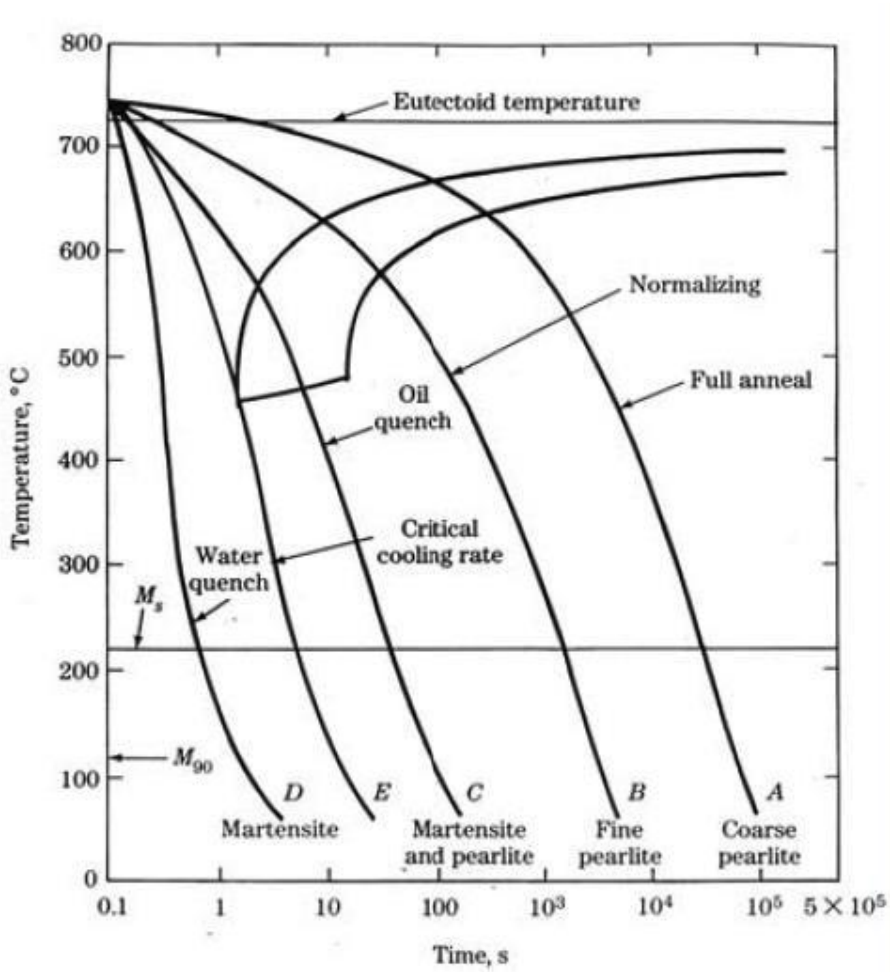

Figure 1: Cooling Path (NavaChing, 2009)

2.3 Influence of Carbon Dioxide

Carbon Dioxide, CO_2 corrosion, is considered as a “sweet corrosion,” where this type of corrosion is one of common problem that occurs in industries (Brondel, 2000 and Treseder 1998). A dry CO_2 gas is not corrosive at the temperatures within the oil and gas production. The CO_2 needs to be dissolved into an aqueous phase to promote an electrochemical reaction between steel and the contacting aqueous phase to become corrosive. It is soluble in water and brines. Moreover, it has a similar solubility in both the gaseous and liquid hydrocarbon phases. Thus, for the solubility to occur, the presence of hydrocarbon phase may provide a ready reservoir of CO_2 to change into aqueous phase.CO_2 is usually present in produced fluids.

There have been extensive works investigating the formation of FeCO3 scale and its protection of the substrate steel in CO2-containing environment without HAc, and a number of corrosion mechanisms and corrosion rate predicting models have been developed in this area (Gulbrandsen, 2007; Kermani and Morshed, 2003)

2.4 Influence of Acetic Acid

Acetic Acid is also known as organic acids. It present in production fluids and been considered to significantly influence and complement CO_2 corrosion (Crolet, 1999). The influence has been shown to occur systematically in all field conditions where CO_2 corrosion was observed. According to Crolet (1999), addition of acetic acid (HAc) to the test environment reduces the protectiveness of the films and increases the sensitivity to mesa attack. This attributes to a lower Fe^{2+} super saturation in the corrosion film and at the steel surface. Thus it suggested that a reduction in film stability can be observed when the concentration of HAc in the solution is increased.

2.5 General Overview of the Corrosion Process

As according to Kermain and Morshed (2003), a dry CO_2 gas is not corrosive at the temperatures within the oil and gas production. The CO_2 needs to be dissolved into an aqueous phase to promote an electrochemical reaction.

Carbon Dioxide Hydration:

$$CO_2\,(g) + H2O \rightarrow H_2CO_3$$

Then it will dissociated to several form

Carbonic acid dissociation $H_2CO_3 \leftrightarrow H^+ + CO_3^-$

Bicarbonate anion dissociation $HCO_3^- \leftrightarrow H^+ + CO_3^{2-}$

Water dissociation $H_2O \leftrightarrow H^+ + OH^-$

As for the metal of carbon steel, the ferrous ion will form

$$Fe\,(s) \rightarrow Fe^{2+} + 2e^-$$

According to Crolet (1999), the Acetic Acid, HAc, in formation water will enhance the cathodic reaction due to the direct reduction of HAC

$$HAc + e^- \rightarrow Ac^- + H^+$$

This shows that it contributing the H+ for acidifies the formation water. With lower pH value will enhance the corrosion rate. At a low pH and presence of CO_2, the HCO_3^- and CO_3^{2-} contents decrease.

$$CO_2 + H_2O + Ac^- \rightarrow HCO_3- + HAc$$

As consequences, the FeCO3 super saturation decrease, resulting in difficulty in achievement of FeCO3 saturation.

Carbon Dioxide corrosion overall chemical reaction

$$CO_2 + H_2O + Fe \rightarrow FeCO_3 + H_2$$

CHAPTER 3.
METHODOLOGY

The project have been divided into two phase. The flow chart in Figure 2, shows the researches methodology stages of this project in FYP 1 and FYP 2 period. For this project, Carbon Steel X65 is selected for all experimentation.

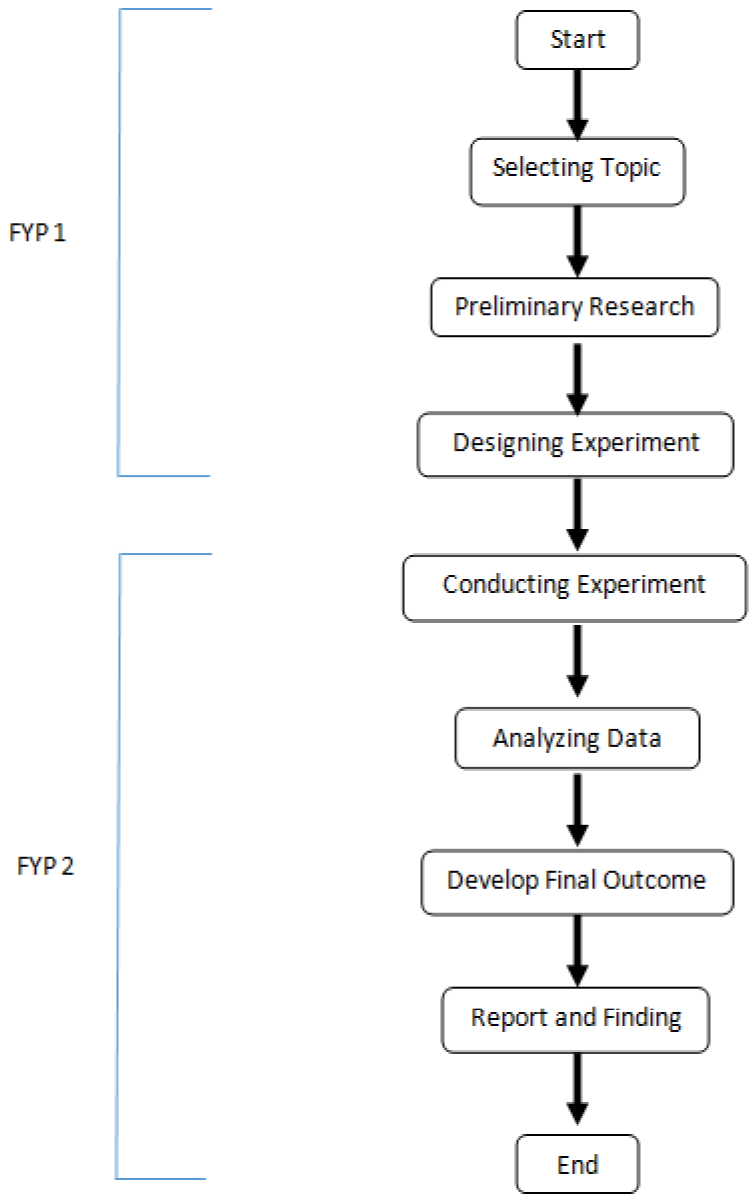

Figure 2: Flow Chart for Research Methodology

For the first phase of the Final Year Project 1 (FYP 1), it was focused on gathering data based on previous research and article. From the data gathered, some sample preparation have been done for conducting the experiment. For Final Year Project 2 (FYP 2), it was the continuation of the FYP 1. The Gantt chart Table 1: FYP 1 Gantt Chart and Table 2 shows the detailed work breakdown of the project

Table 1: FYP 1 Gantt Chart

No.	Detail/Work	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selecting of Project Topic	■	■												
2	Preliminary Research Work • Understanding Corrosion Process • Identifying type of Heat Treatment • Understanding corrosion test		■	■	■	■									
3	Completing Extended Proposal for submission						●								
4	Presentation on project proposed								●						
5	Continuing project work • Designing Possible Corrosion Test • Familiarization with Laboratory Equipment • Sample Preparation						■	■	■	■	■	■ ●	■		
6	Completing Interim Report for Submission													●	
7	Preparation for experiment in laboratory.												■	■	■
8	Meeting With Supervisor	■	■	■	■	■	■	■	■	■	■	■	■	■	■

Key Milestone ● Completion ■ Project Baseline

Table 2: FYP 2 Gantt Chart

No.	Detail/Work	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Sample Preparation • completing cutting sample to smaller pieces • conducting heat treatment • completion of Job Safety Analysis for corrosion testing • waiting for approval for approval to conducting experiment															
2	Conducting experiment • Conducting microstructural test (OM or SEM) • Sample preparation for LPR (cold mount) • Setup for weight lost and continuing with experimentation • Setup for LPR and continuing experimentation															
3	Project Work Continuous • Progress report • Completing Experiment and Collecting Data															
4	Completing Experimentation • Recording result and finding • Data analysis															
6	Completing of Dissertation and Technical Paper for submission															
8	Presenting Completed Project															

Key Milestone   Suggested Completion   Project Baseline

3.1 Sample Preparation.

For this research, Carbon Steel X65 is used. The carbon steel pipe were machined into a desired shape before undergo any experiment. 16 samples were prepared with four for each heat treatment including the untreated raw sample as the baseline. The heat treatment that have been done were annealing, normalizing and quenching. Figure 3 shows the overall step for sample preparation.

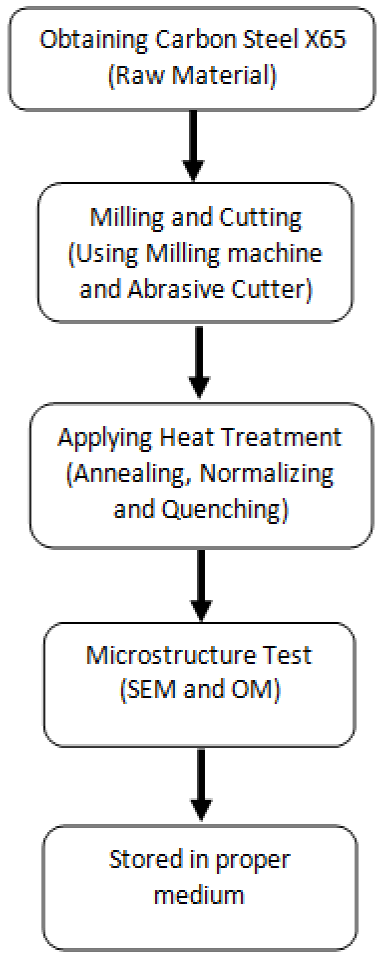

Figure 3: Sample Preparation before Conducting Corrosion Test

- Milling and Cutting Process

The obtained carbon X-65 (Figure 4) is in form of sectioned pipe shape. The carbon X-65 was undergo milling process before cut into smaller pieces. As in Figure 5, the carbon X-65 was milled by using conventional milling machine into a plate shape for ease the cutting process. The milled sample then undergo cutting process by using abrasive cutter machine. The shape of each sample were in form of cuboid with height and width of 5mm and length of 10mm. As in Figure 6, the shape of sample is in cuboid shape for ease the experiment and calculation of the surface area.

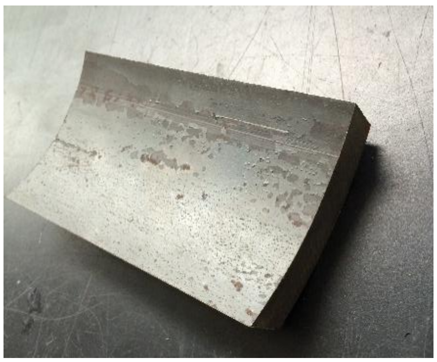

Figure 4: Raw Sample for Carbon Steel X-65

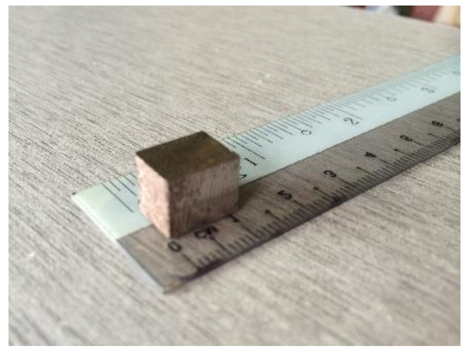

Figure 6: Cut Sample for Carbon Steel X-65

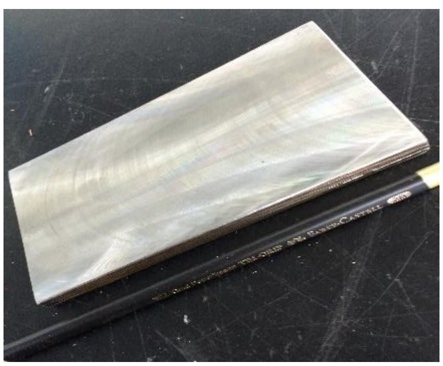

Figure 5: Milled Sample for Carbon Steel X-65

- Heat Treatment Process

The heat treatment process was done by using heat treament oven or furnace. The sample was heated until 750^oC above the eautectoid temperature and then cooldown in different rate. For annealling, the sample was heated and cooled down inside the furnace as the rate of cooling for the furnace to cool down is 24 hours to give a full annealed sample. A precaution step for not opening the furnace chamber before it reach room temperature is required to avoid thermal shcok on the sample. In Figure 7, it show the annealed samples that have been cool down inside the furnace.

For normalizing, the sample was cool down in a room temperature. A setup of brick and a holding place was required as in Figure 8. This is because the sample was in high temperature and might cause injuries if accidentally touch. Finally, for water quenched the sample was immersed inside a water. A set of clipper and metal water basin is required to immerse the samples into the water. In Figure 9, it shows that the sample was handled by using a clipper and immersed inside the water.

Figure 7: Samples are Furnace Cooled (Annealing)

Figure 9: Samples are Water-cooled

(Water Quenching)

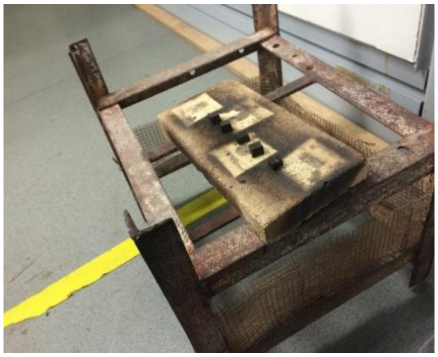

Figure 8: Sample are Air-cooled (Normalizing)

3.2 Carbon Steel X-65

The corrosion experiment was be done by using a Carbon Steel X-65. Primarily, it is used in oil and gas industries due to the strength and low cost. The manufacturing of this pipe was rolling and cooled at the room temperature (normalizing). The samples were undergo further heat treatment which were annealing, normalizing and quenching. An optical microscope investigation was conducted to determine the microstructure of each sample. The chemical composition according to Huang (2013) of carbon steel X-65 as shown in the Table 3: Chemical Composition of X-65 Carbon Steel (Huang, 2013). The mechanical properties of Carbon Steel X-65 as in Table 4.

Table 3: Chemical Composition of X-65 Carbon Steel (Huang, 2013)

Fe	C	Mn	Mo	S	Ni	Si	Cr	Nb	Al
97.53	0.13	1.16	0.16	0.009	0.36	0.26	0.14	0.017	0.0032

Table 4: Mechanical Properties of Carbon X-65 (Wang et al, 1999)

API 5L Grade	Yield Strength min. (MPa)	Tensile Strength min. (MPa)	Yield to Tensile Ratio (max.)	Elongation min. %1
X65	450	530	0.93	18

3.3 Experimental Setup

For this project, two corrosion measurement technique were used. The measurement technique were Linear Polarization Resistance (LPR) and Weight Loss (WL). The nature of the experiment is by following test matrix as in Table 5.

Table 5: Test Matrix for Sample Preparation and Corrosion experiment

Steel type	X-65 Carbon Steel			
Type of Heat Treatment	Untreated	Annealing	Normalizing	Quenching
Solution	3% NaCl, saturated with carbon dioxide			
Temperature, °C	25			
Acetic Acid, HAc Concentration (ppm)	0, 1000			
pH	6.6			
Corrosion Measurement Technique	LPR (ASTM G 3-89), WL (ASTM G 31-72)			
Duration, hours	48			
Pressure, bar	1			

3.3.1 Linear Polarization Resistance (LPR)

Linear Polarization Resistance monitoring is an effective electrochemical method of measuring corrosion. Monitoring the relationship between electrochemical potential and current generated between electrically charged electrodes in a process stream allows the calculation of the corrosion rate. The experiments were done base on three electrode glass cell. Each samples for this experiment will undergo mounting process before the experimentation take place. As referred to ASTM G1 - Standard Practice for Preparing, Cleaning, and Evaluating Corrosion Test Specimens, the sample will undergo cold mount process (Figure 10). The procedure as follows.

- The heat treated sample was grinded to remove the burn residue form on the sample.
- The sample was marked using drill to mark the soldering point.
- The sample was soldered with a copper wire and tested with voltmeter to test for conductivity. The copper wire then insulated by using a plastic tube and placed in a mount.
- An Epoxy hardener and Epoxy resin was prepared with a ratio of 1:5 and stirred slowly while avoiding bubble forming in the solution. It was stirred until the mixture form a homogenous or colourless solution.
- The solution is then poured into the mount and left for 24 hours for curing process.

For experiment, the solution for LPR was prepared referring to the test matrix. To create CO_2 environment, the solution was purged for an hour and the pH was monitored by using pH meter. Sodium Hydroxide (NaOH) was used to maintain the pH at 6.6. The temperature was maintain as the room temperature. The glass cell was setup with a working electrode (cold mounted sample), references electrode, and counter electrode. The setup is as in the Figure 11.

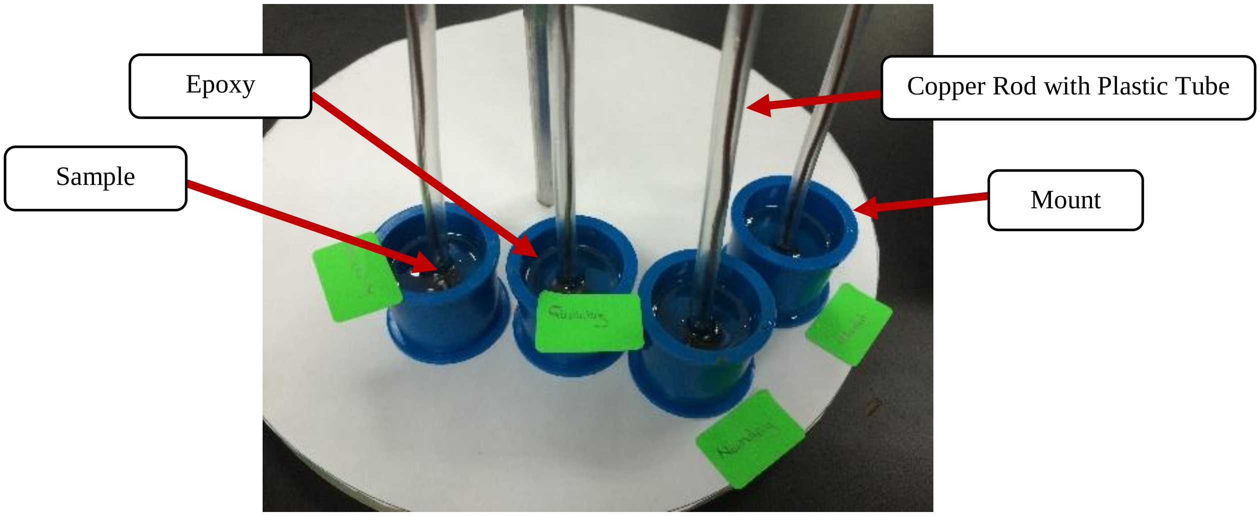

Figure 10: Cold Mounted Sample Cure for 24 hours

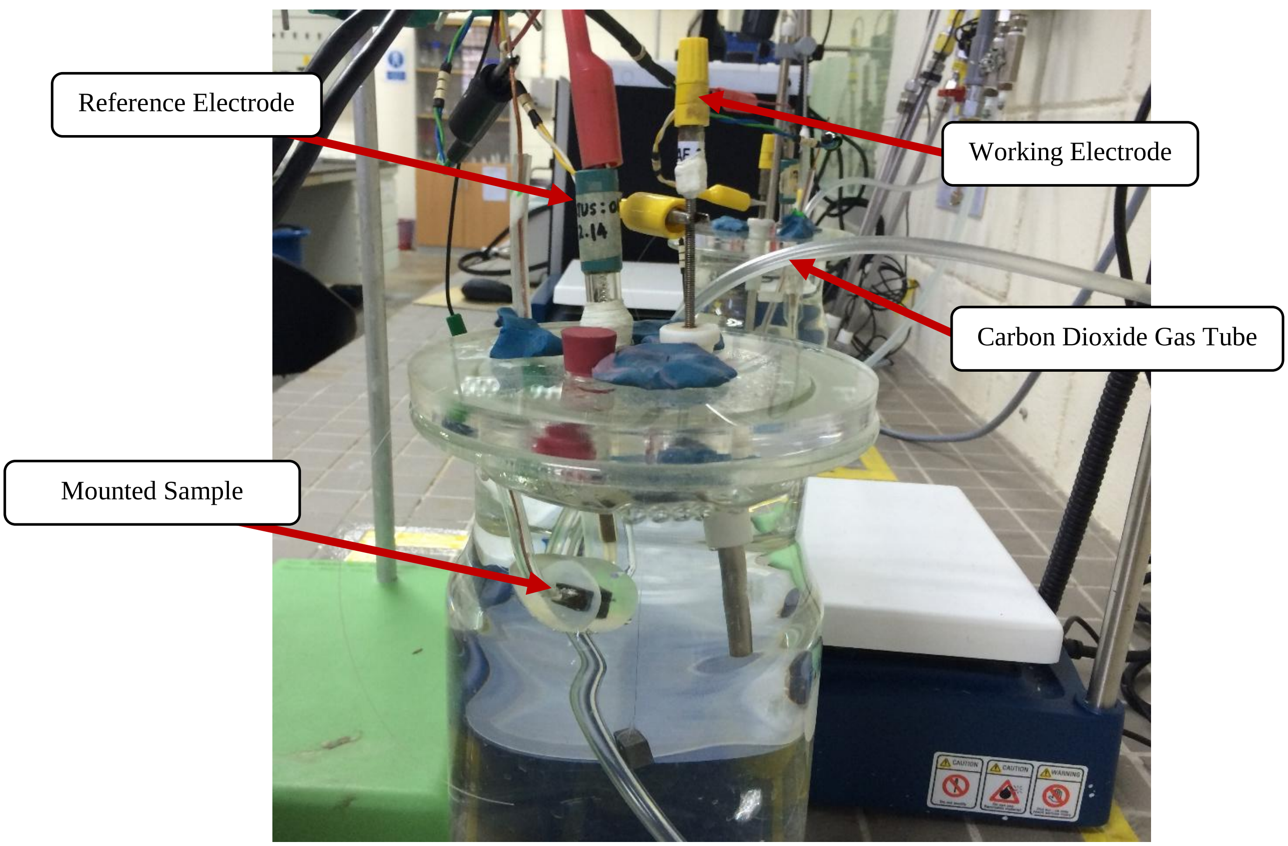

Figure 11: Experiment Setup for LPR

3.3.2 Weight Loss

Weight Loss is one of the Immersion tests. It is a measure of progress of corrosion damage obtained from the immersion length within a corrosive environment. In this test, the loss of weight is being gaged after a particular period. The solution for weight loss was prepared by referring to the test matrix. The solution was purged with CO_2 gas for an hour and the pH was monitored by using pH meter and maintain at 6.6 by using NaOH. For weight loss samples, the samples was drilled in the middle to act as a holder. The initial and final weight of each samples was determined. A string will tied the sample before being immersed into the solution. The setup of the experiment is in Figure 12.

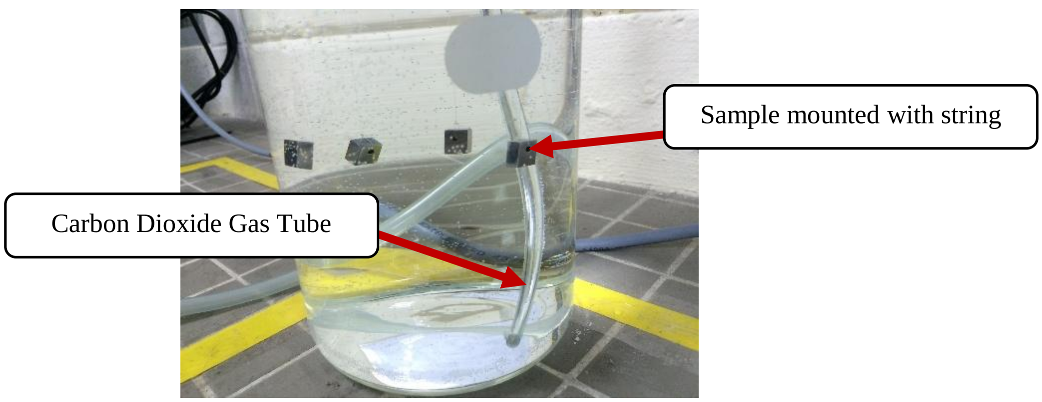

Figure 12: Experiment Setup for Weight Loss

After a period of 48 hours, by referring to ASTM G3-72(2004), the corrosion rate for weight loss was calculated. The formula as is in Equation 1

$$CR\left(\frac{mm}{yr}\right) = \frac{K \times W}{A \times T \times D} \longrightarrow \text{(Equation 1)}$$

K: 8.76×10^4 mm/yr

D: Density, 7.86 g/cm^3 Carbon Steel

A: Total Surface Area, cm^2

T: Time in Hour

W: Mass loss, g

3.3.3 Presence of Acetic acid

To create a presence of acetic acid, the acetic acid is added after the environment is CO_2 environment and pH value is 6.6. This is after an hour of purging and the pH is raised by NaOH. According to Crolet, (1999), with the presence of acetic acid, it will contribute the H+ for acidifies the formation water. Thus the pH value of the solution will be decreased as the acetic acid is added. The sample is added after the acetic acid is added.

CHAPTER 4.
RESULT AND DISCUSSION

4.1. Optical Microscope

Optical or light microscope involves transmitting visible light through or reflected from the sample through lenses that allow a magnified view of the sample. For this project, the sample image was captured digitally by using a charge-coupled device (CCD) camera to focus on the exhibit of interest the image. It is shown on a computer screen and can be recorded as figure below. For all samples, the etchant used was Nital to reveals alpha grain boundaries and constituents. The etchant will react to ferrite grain causing burn marks to appear. The sample is immersed for a few minutes before the image is captured. Figure 13, Figure 14, Figure 15 and Figure 16 shows the microstructure of each samples.

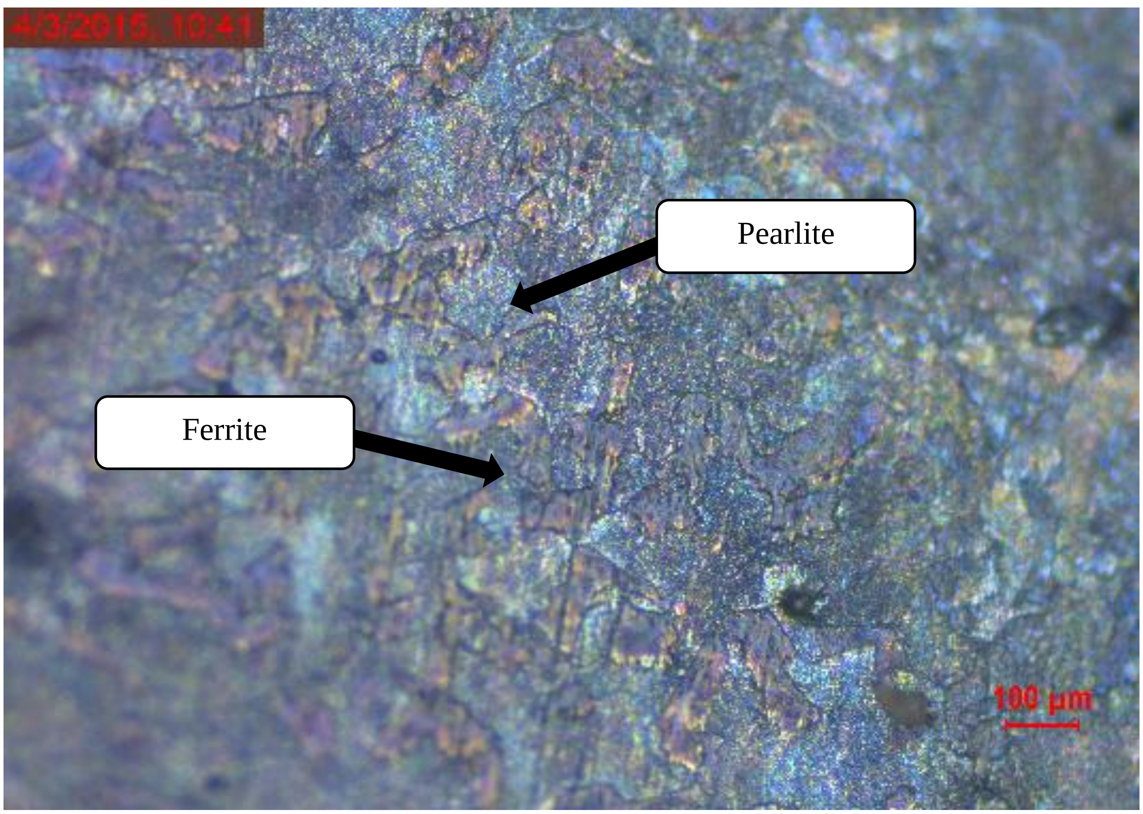

Figure 13: Microstructure of X65 Steel Sample (Untreated)

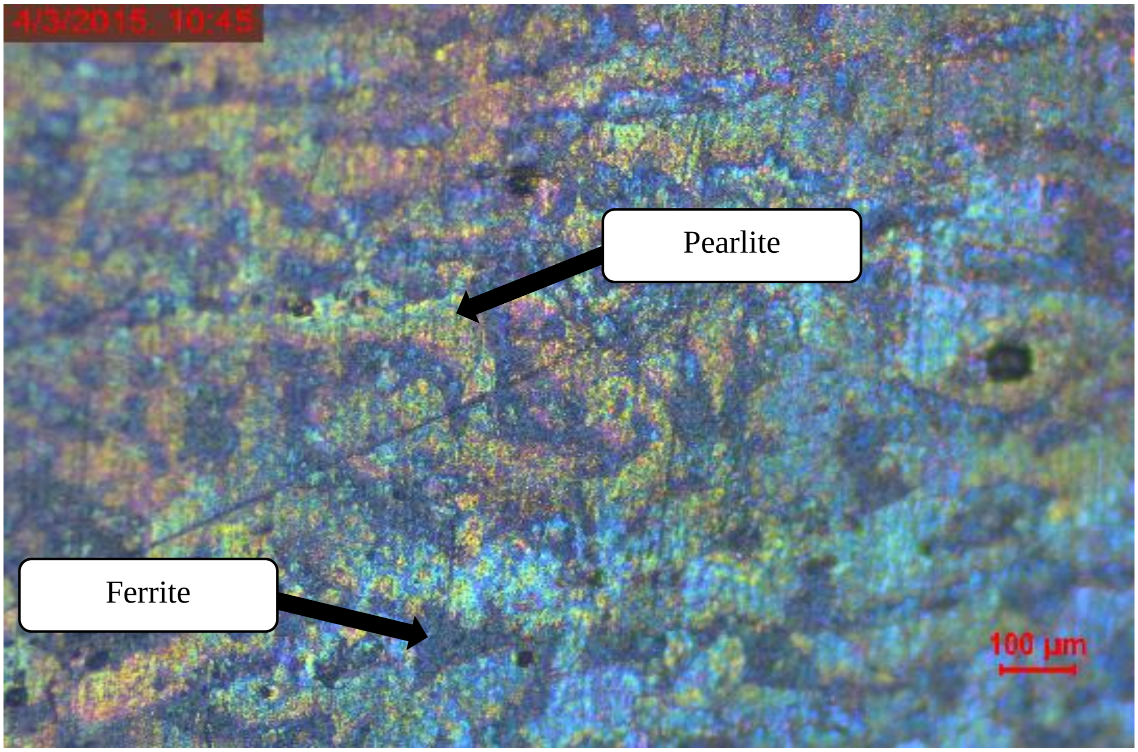

Figure 14: Microstructure of X65 Steel Sample (Annealing)

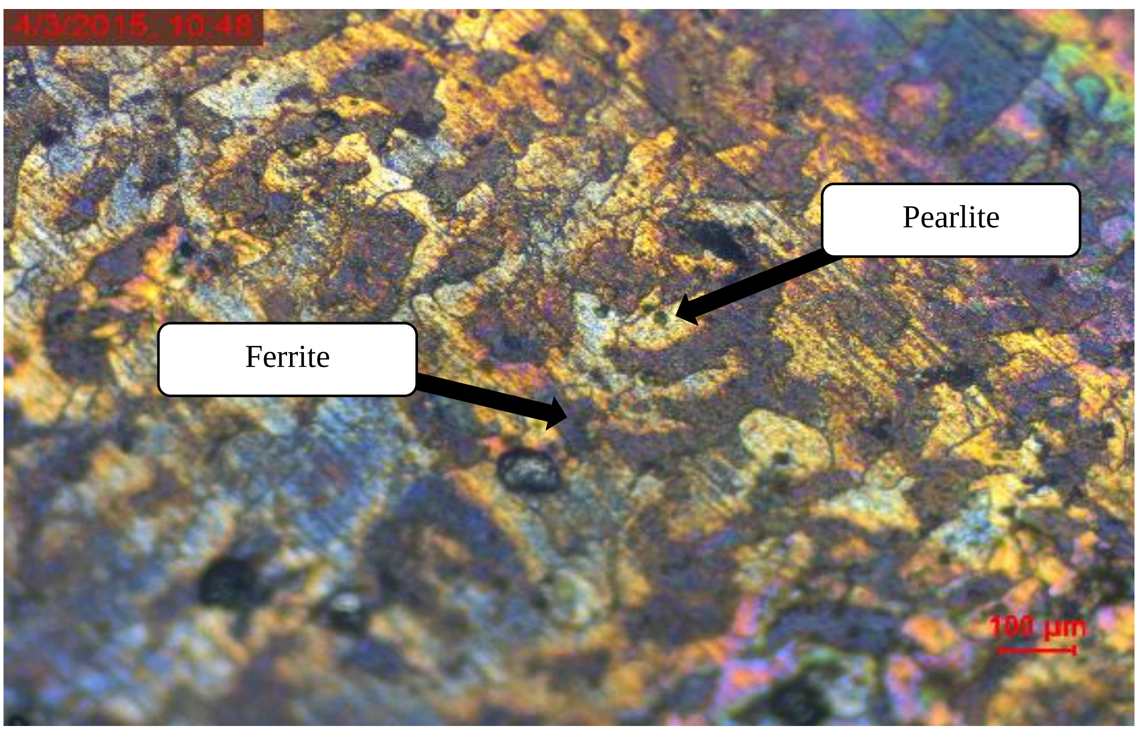

Figure 15: Microstructure of X65 Steel Sample (Normalizing)

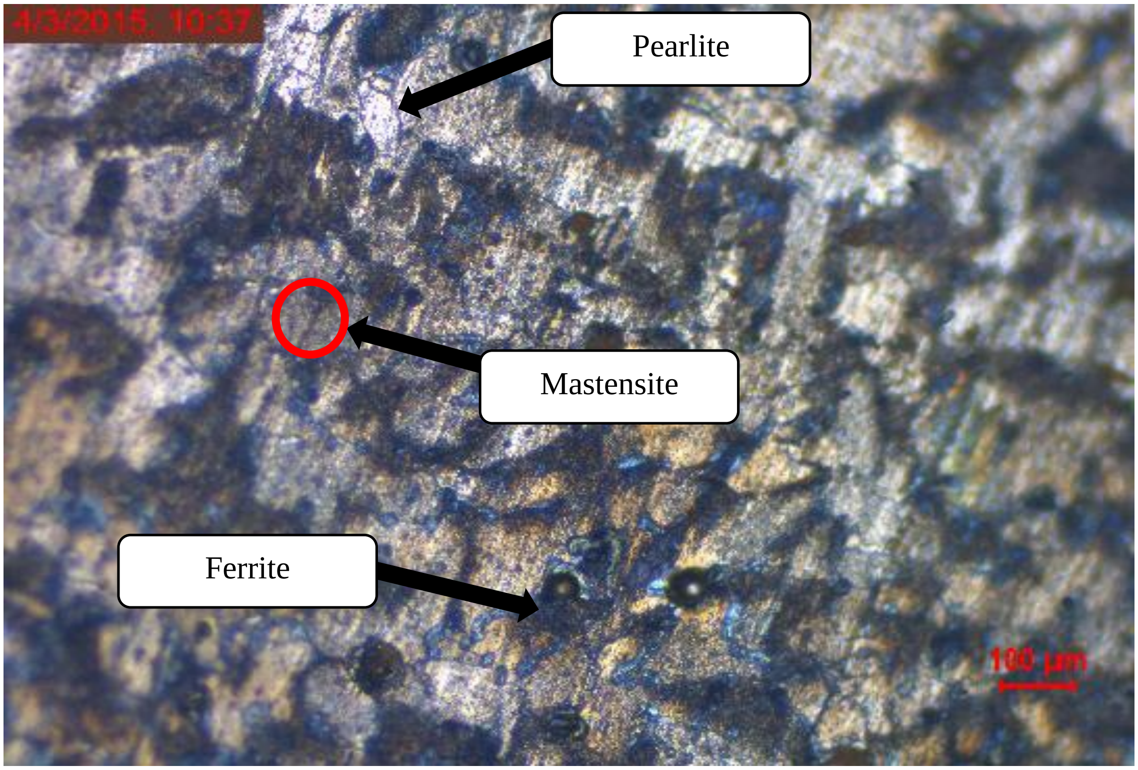

Figure 16: Microstructure of X65 Steel Sample (Quenching)

From the figure, we can study the differences of the microstructure surface on the sample. The darker color in each microstructure is ferrite while the lighter color is pearlite because of the Nital burn ferrite grain. In Figure 13, this was the original surface of the Carbon X-65. The surface is a result from the rolling and normalizing (cool down using surrounding temperature) process during the manufacturing of the pipe. With further heat treatment applied, different microstructure surface produced. For example, as in Figure 14, the ferrite grains had undergo complete recrystallization. This give a much coarse pearlite distribution on the surface. In Figure 15, the surface of the microstructure is showing a fine pearlite distribution. As in this figure, there are much shorter graphite flakes (lighter color) compared to annealing. Finally in Figure 16, the burn small like needle shape have been identified as martensite. From the microstructure, it was determined that quenching have the highest distribution of ferrite compared to annealing and normalizing. This theoretically will cause quenching to have highest corrosion rate compared to annealing and quenching as there will be more ferrite reaction with carbonate ion in the carbon dioxide corrosion.

4.2. Corrosion Rate

Linear Polarization Resistance (LPR)

From the test matrix, the LPR was done for with the presence of acetic acid and without the presence of acetic acid. The corrosion rate for each samples were recorded every hour throughout the 48 hours duration of the experimentation. Figure 17 show the corrosion rate varying with time without the presence of the acetic acid. For each samples, the corrosion rate was increased for the first 24 hours. For the next 24 hours, the corrosion rates are stabling until the end of the experimentation. It is stable as the reaction is reaching of equilibrium point where the reaction occurs in constant rate. From the figure, it also identified that different heat treatment will give different corrosion rate. For example, from the figure, annealing shown the lowest corrosion rate which is 1.3 mm/year compared to quenching which give the highest corrosion rate of 1.6mm/year. This have proved that with higher distribution of ferrite microstructure on the surface, it will cause the reaction to bicarbonate ion to occur faster.

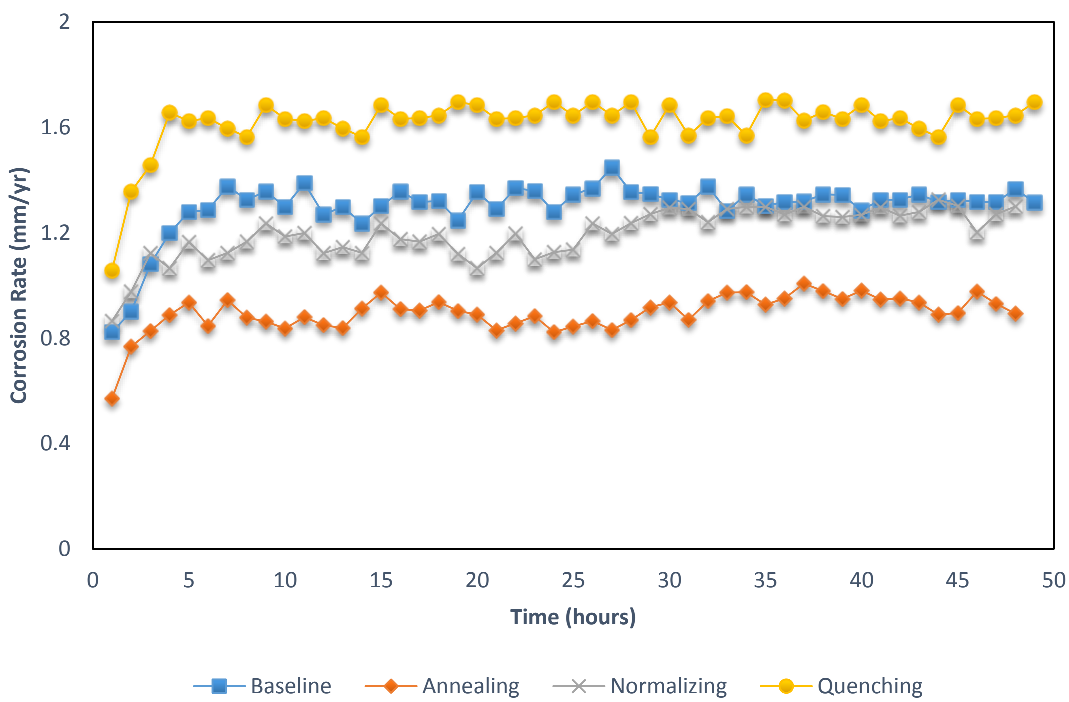

Figure 17: LPR Corrosion Rate against Time with Absence of Acetic Acid

Figure 18 show the corrosion rate again time with the presence of the acetic acid. The pattern of the corrosion rate is similar with without acetic acid. It is identified that corrosion rate is increased with the presence of acetic acid. This can be seen by comparing the corrosion rate of the baseline with absence of acetic acid and baseline with presence of acetic acid. For without acetic acid, it is shown that the corrosion rate in range of 0.8mm/yr to 1.6 mm/yr. Compared to with presence of acetic acid, the corrosion rate is in range of 1.4 mm/yr to 2.2 mm/yr. This shows that with the presence of acetic acid, the corrosion rate is increased.

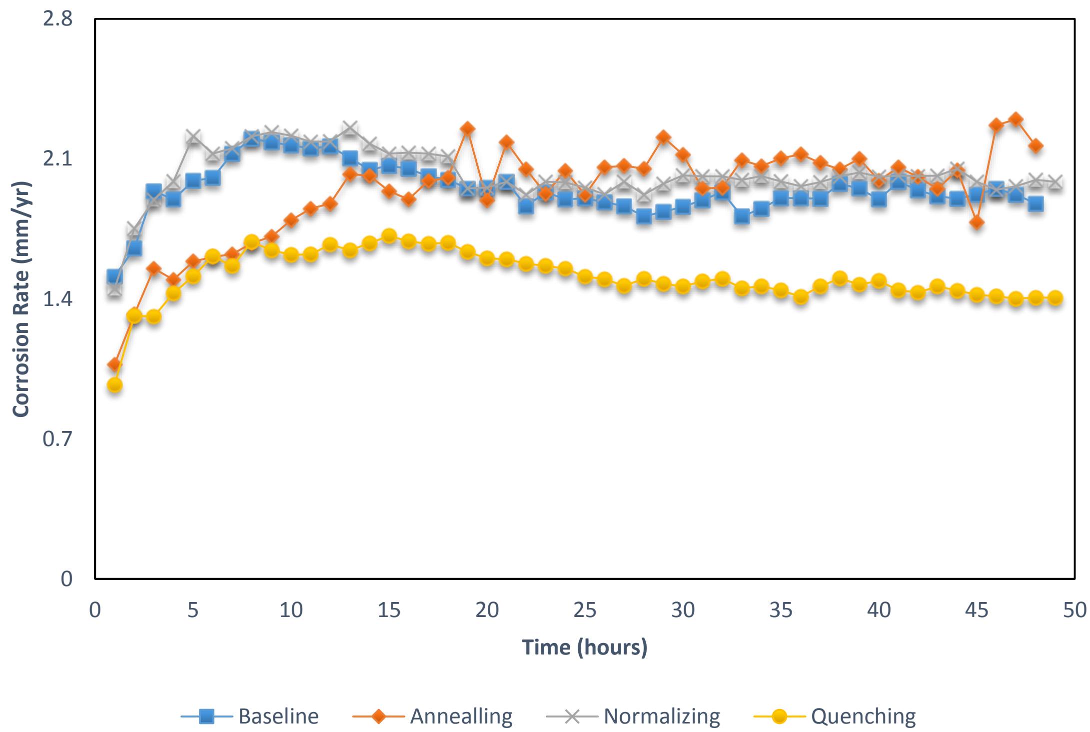

Figure 18: LPR Corrosion Rate against Time with Presence of Acetic Acid (1000ppm)

Weight Loss

From the test matrix, the weight loss experiment was done for with the presence of the acetic acid and without the presence of acetic acid. The dimension of each samples were recorded as in Table 6 and Table 7. Based on the dimension and the weight loss the corrosion rate was calculated by using Equation 1 referring to ASTM G3-72(2004).

Table 6: Dimension of Each Sample and the Calculated Corrosion Rate without Acetic Acid

Dimension/Heat Treatment	Untreated	Annealing	Normalizing	Quenching
Height, cm	0.735	0.801	0.562	0.765
Width, cm	0.868	0.844	0.872	0.843
Length, cm	1.101	1.146	1.088	1.053
Total Area, cm^2	5.098632	5.220984	4.775072	4.840506
Initial Weight, g	5.1335	5.6433	3.8944	4.8142
Final Weight, g	5.0823	5.6021	3.8619	4.7672
Weight Loss, g	0.0512	0.0412	0.0325	0.047
Corrosion Rate (mm/yr)	1.2	1.0	1.1	1.6

Table 7: Dimension of Each Sample and the Calculated Corrosion Rate with Acetic Acid (1000ppm)

Dimension/Heat Treatment	Untreated	Annealing	Normalizing	Quenching
Height, cm	0.658	0.865	0.749	0.959
Width, cm	0.844	0.826	0.849	0.857
Length, cm	1.033	1.098	1.074	1
Area, cm^2	4.598112	5.056772	4.919106	5.071726
Initial Weight, g	4.1204	5.7636	4.9657	6.1791
Final Weight, g	4.0799	5.7301	4.9251	6.1306
Weight Loss, g	0.0405	0.0335	0.0406	0.0485
Corrosion Rate (mm/yr)	2.0	1.5	1.9	2.2

From both tables, the weight loss experimentation also conclude the same as LPR technique that different heat treatment will give different corrosion rate. From table 4-1, it shows that annealing give the lowest corrosion rate of 1.0 mm/year while quenching give the highest corrosion rate of 1.6 mm/year. From Table 6 and Table 7, it also identified that with the presence of acetic acid, the corrosion rate will increased. For example, annealing shows increased up to 50% of the corrosion rate with the presence of acetic acid.

4.3. Result Summary

Different type of heat treatment will give different type of microstructure. In CO_2 environment, different type of microstructure will give different type of corrosion rate. With the presence of the acetic acid, the corrosion rate performance is increased. Figure 19 and Figure 20 show the summary result for the corrosion rate performance.

From Figure 19, it shows that difference in heat treatment process give difference corrosion rate. From the result, it is shows that annealing have the lowest corrosion rate performance indicates it have highest resistance to corrosion while quenching have the highest corrosion rate which indicates lowest corrosion resistance.

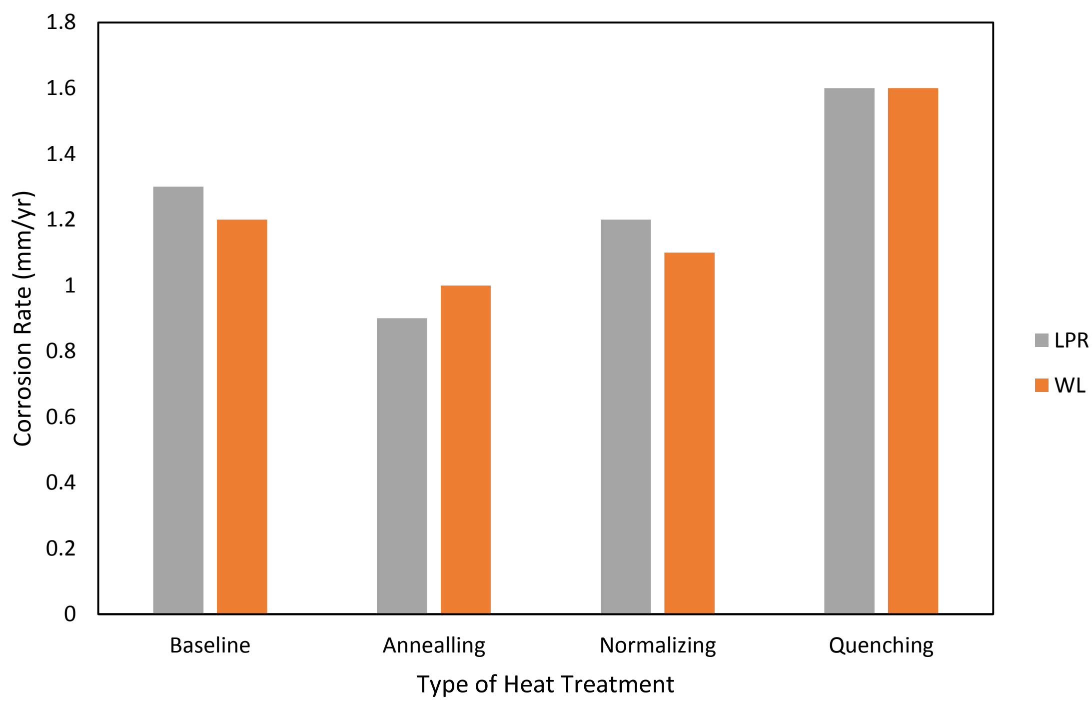

Figure 19: Summary of Corrosion Rate with Absence of Acetic Acid

Figure 20 also show that different type of heat treatment give different corrosion rate even with the presence of acetic acid. The presence of acetic acid only increased the corrosion rate of each of the sample. For example, the baseline sample in Figure 19 shows an average reading of 1.2 mm/yr while the baseline in Figure 20 shows an average of 1.95 mm/yr. This shows that the presence of acetic acid will affecting the corrosion rate regardless of the heat treatment applied.

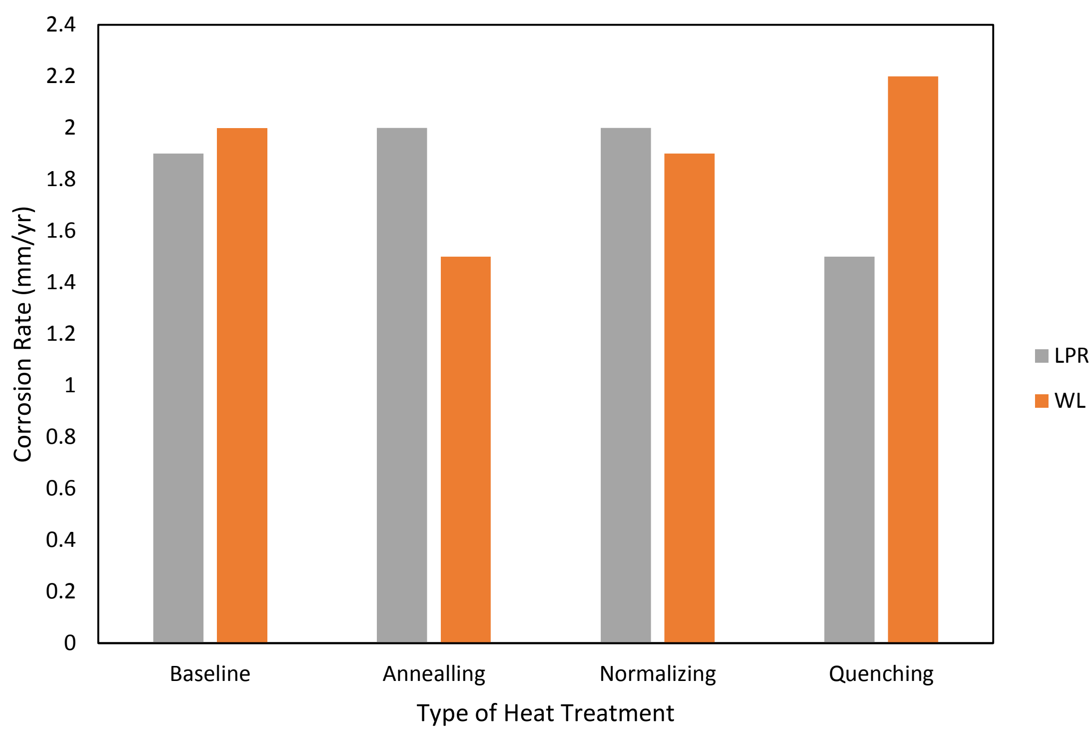

Figure 20: Summary of Corrosion Rate with Presence of Acetic Acid (1000ppm)

CHAPTER 5.
CONCLUSION AND RECOMMENDATION

For this project, it is to study the influence of heat treatment process on carbon steel pipe corrosion in the presence of carbon dioxide and acetic acid.

- From the heat treatment result obtained, the microstructure was altered by heat treatment. It was supported from the Optical Microstructure result where the annealed sample give a more coarse pearlite grain, normalized sample give a fine pearlite grain and quenching give a martensite grain.
- In Carbon Dioxide environment, different heat treatment has shown different corrosion rate on each of the sample. The annealed sample of carbon steel has the lowest corrosion rate compared to other type of heat treated sample. This shown that annealed sample have the highest corrosion resistance among normalizing and quenching.
- With the presence of acetic acid, the corrosion rate have increased. Each of the sample have shown an increased in corrosion rate regardless of the heat treatment applied.

According to past research, different kind of heat treatment will give different kind of microstructure surface of the metal. Therefore, the corrosion rate of each type of microstructure surface is different. Furthermore, with the presence of the acetic acid it will cause the Carbon Dioxide environment to be much more acidic. Therefore, it is expected the corrosion rate to be increased.

As future references, to investigate the different type of heat treatment, the sample is suggested to be various following the equipment standard in the industries today. For example a pipeline and a valve which will undergo different heat treatment to serve the purposed. Instead focusing on oil and gas industries, it is suggested to widen the area to other industries such as utilities plant or chemical plant which will give different environment to be studied. For the experiment it, it is suggested to check the equipment to be used as in laboratories, some of the equipment such as reference electrode might get contaminated by previous experiment.

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