Influence of Heat Treatment and Microstructure on the Corrosion Performance of Carbon Steel Line Pipe in CO_2 Environment

by

Abdullah Farhan bin Zainudin

Dissertation submitted in partial fulfillment of
the requirements for the
Bachelor of Engineering (Hons)
(Mechanical Engineering)

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CERTIFICATION OF APPROVAL

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Approved by,

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(Ir. Dr. Mokhtar bin Che Ismail)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
May 2012

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.

ABDULLAH FARHAN BIN ZAINUDIN

ABSTRACT

Hydrocarbon-transporting pipelines are intensively exposed to CO_2 corrosion. Due to high cost of corrosion-resistant alloys (CRA), carbon steel is seen to be the best option for pipelines material. During fabrication, the pipelines undergo specific heat treatments to achieve desired mechanical properties prescribed by the users. These heat treatments affect the microstructure of the pipelines. Previous studies done have discovered that the microstructure of the carbon steel offers significant influence on its corrosion behavior. The main objective of this project is to characterize the relationship between microstructures and corrosion performance of carbon steel in CO_2 environment. Meanwhile, the microstructure of different heat treatments is observed as well. The project focuses on the common materials used in pipeline construction. Pipe samples are collected from pipeline manufacturers and Centre for Corrosion Research (CCR), Universiti Teknologi PETRONAS (UTP). The samples go through sample preparation which includes sectioning, mounting, grinding, polishing and etching before examined under a microscope. The microstructure is recorded according to its corresponding heat treatment. Each sample is then put through corrosion test using glass cell in 3% NaCl solution at 50°C, pH 4, 1 bar CO_2, for two weeks. The result shows that sample with ferrite/bainite/tempered martensite microstructure, and fine ferrite/pearlite microstructure have the lowest corrosion rate. Banded ferrite/pearlite microstructure has the highest corrosion rate. Iron carbonate scale is found the most on fine ferrite/pearlite microstructure and covers most of the surface area. Fine grains anchor scale better than coarse structure does. Iron carbide or cementite influences scale adherence on the steel surface. In banded structure, the segregated distribution of cementite causes poor performance in terms of localized corrosion. In other microstructures, cementite is more evenly distributed. For hydrocarbons transportation, fine ferrite/pearlite microstructure, or ferrite/bainite/tempered martensite microstructure is recommended for better corrosion resistance.

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CHAPTER 1: INTRODUCTION

1.1 Background of Study

Offshore pipelines are commonly constructed of carbon steel, recognizing its economic and strength advantages. Although there are corrosion resistant alloys (CRA) or stainless steels which are superior in terms of resistance to corrosion, their utilizations are not economically justified. Carbon steel line pipe used in oil and gas production and transmission, is manufactured in accordance with American Petroleum Institute (API) specification 5L, does not have a closely specified elemental composition and microstructure (D. Clover, B. Kinsella, B. Pejcic and R. De Marco, 2004). Line pipe fabricated according to this specification may be as-rolled, normalized, normalized and tempered, subcritically stress relieved, subcritically age-hardened or quenched and tempered. Consequently, line pipes of the same grade may have variations in their compositional and microstructural properties, dependent upon the manufacturers. These variations may lead to substantial differences in the corrosion resistance of steel line pipe (D. Clover, B. Kinsella, B. Pejcic and R. De Marco, 2004).

Since carbon dioxide corrosion has become a major problem in oil and gas field, various studies have been done to understand its mechanisms, identify the factors affecting, and come up with suitable solutions or mitigations. In addition to temperature, pH, CO_2 partial pressure, and shear stress, studies have shown that the steel microstructure may also affect the corrosion rate. Several studies have been done by various authors on the influence of different microstructures on the corrosion performance of carbon steels. In order to understand better, studies are done on different grades of carbon steel.

1.2 Problem Statement

Carbon dioxide corrosion is a major problem in oil and gas industry. Carbon steel offshore pipelines are intensively exposed to CO_2 environment hence are very susceptible to CO_2 corrosion. The successful utilization of carbon steel line pipes in CO_2 corrosion environment requires not only effective corrosion mitigation but also careful selection of line pipe heat treatment specification. Appropriate heat treatment specification is critical as microstructures and alloying elements also influence the corrosion behavior of carbon steel. Depending upon the steel’s composition, thermal history, and mechanical history, its microstructure differs significantly. This project will investigate the effects of several heat treatments on the microstructures of selected grades of carbon steel and their subsequent influence on the corrosion performance in CO_2 environment.

1.3 Objectives

The objectives of this project are:

1. To characterize microstructures of common heat treatment specifications.
2. To study the influence of heat treatment of carbon steel line pipe on its corrosion performance in CO_2 environment.
3. To evaluate the relationship between microstructures and CO_2 corrosion performance.

1.4 Scope of Study

This project essentially focuses on the corrosion performance of carbon steels with different microstructures in CO_2 environment. The corrosion performance is evaluated under approximated actual conditions of operating pipelines. The materials tested are carbon steels which pipelines are typically made of. The corrosion performance is assessed in terms of corrosion rate and presence of corrosion film. The expected outcome to be achieved is the correlation between heat treatment, microstructure, and corrosion performance. This correlation may help in understanding the best material that is suitable to be used in oil and gas transportation.

CHAPTER 2: LITERATURE REVIEW AND THEORY

2.1 Carbon Dioxide (CO_2) Corrosion Mechanism

In oil and gas industry, carbon dioxide (CO_2) corrosion is caused by the CO_2 contained within the hydrocarbons transported by the pipelines. The carbon dioxide dissolves in the seawater to form carbonic acid.

$$CO_2 + H_2O \rightarrow H_2CO_3$$

The carbonic acid will ionize to form hydrogen and bicarbonate ions.

$$H_2CO_3 \rightarrow H^+ + HCO_3^-$$

The bicarbonate ions then further ionize to form hydrogen and carbonate ions.

$$HCO_3^- \rightarrow H^+ + HCO_3^{2-}$$

Bicarbonate ion can also produce further carbonic acid by a disproportional reaction:

$$2HCO_3^- \rightarrow H_2CO_3 + CO_3^-$$

This produces a further source of hydrogen ions. There are 3 possible cathodic reactions:

$$2H^+ + 2e^- \rightarrow H_2$$

$$2H_2CO_3 + 2e^- \rightarrow H_2 + 2HCO_3^-$$

$$2HCO_3^- + 2e^- \rightarrow H_2 + 2CO_3^{2-}$$

Though carbonic acid is a weak acid, sufficient quantities may be able to help accelerate the corrosion process. The anodic reaction is:

$$Fe \rightarrow Fe^{2+} + 2e^-$$

The Fe^{2+} and CO_3^{2-} combine to form iron carbonate, $FeCO_3$, a corrosion layer on the steel surface. This layer can be protective to the steel surface and may influence the corrosion performance of the steel.

2.2 Heat Treatments of Steel

2.2.1 Austenizing

Austenizing is a process of heating steel to a temperature within the austenite or austenite + Fe_3C region. Hardening of steel requires a change in crystal structure from the body-centered cubic (BCC) form present at room temperature to face-centered cubic (FCC) (Kenneth G. Budinski and Michael K. Budinski, 2010, p. 401). By referring to the iron-carbon equilibrium diagram, the temperature to which the steel should be heated based on the carbon content can be known, in order to obtain the FCC structure. To keep the carbon trapped in the crystal structure, quenching is required after austenizing.

2.2.2 Quenching

According to Kenneth G. Budinski and Michael K. Budinski (2010), quenching normally is accomplished by rapidly removing the part from the furnace (after it has soaked for sufficient time to reach the required temperature) and immersing it in agitated oil or water (p. 401). Hardenability is the term used to describe the ease of a steel to transform into a hardened structure on quenching. Some steels can be hardened by just removing it from the furnace and allowing it to cool by convection at room temperature. This process is known as normalizing and normally produces ferrite and pearlite microstructure. The rate of quenching is influenced by the fluid media used and the degree of agitation in the media. The most severe are water quench, followed by oil, molten salt, and gas quenching. Addition of other medium into the fluid media may also affect the cooling rate. The resulting microstructure is influenced by the cooling rate. For example, rapid quenching in water usually results in martensite microstructure.

2.2.3 Annealing

Annealing is accomplished by heating steel to its austenizing temperature and then slowly cooling to prevent the formation of a hardened structure. At annealing temperature, the structure transforms to austenite or austenite plus cementite. Slow cooling will produce ferrite and pearlite, or pearlite and cementite microstructure. Softening can occur in annealing by diffusion.

According to Kenneth G. Budinski and Michael K. Budinski (2010)

> If a part is being annealed to change the structure from hard martensite to a machinable structure such as ferrite and pearlite, the softening is accomplished by diffusion of carbon from the metastable martensite and re-solution of the carbon in austenite at the annealing temperature. Diffusion is the spontaneous movement of atoms in the crystal structure of a metal. Martensite is hard because there is an overabundance of carbon atoms trapped by quenching in a crystal structure that wants to be BCC. Diffusion of carbon in steels is controlled by temperature; by the time a martensitic steel reaches the annealing temperature, most of the carbon that was trapped in martensite has diffused out. At the annealing temperature, the structure transforms to austenite, and all the carbon goes into free cementite or into solution in austenite. (p. 417)

2.2.4 Tempering

Tempering is a low temperature heat treatment used to improve the toughness of quench-hardened steels. Kenneth G. Budinski and Michael K. Budinski (2010) stated that tempering of martensite in plain carbon steels involves diffusion of carbon atoms from martensite and the formation of carbide precipitates and concurrent formation of ferrite. Tempering also causes some retained austenite from quenching to transfer to cementite and ferrite. Tempering is usually done after quenching.

2.3 Influence of Microstructure on the Corrosion Performance of Carbon Steel

Microstructure plays an important role in determining the proper adherence of corrosion scale to the steel surface. D. A Lopez et. al. (2003) reported that the carbide phase can strengthen the film and anchor it to the steel substrate. Stegmann et al. proposed that the needle-like carbide structure provides a better anchoring surface for the $FeCO_3$ than large ferrite areas interdispersed by a few pearlite grains (as cited in D. A. Lopez, 2003).

It has been reported by D. Clover, B. Kinsella, B. Pejcic and R. De Marco (2004) that a course, banded, ferrite/pearlite microstructure lowers resistance to localised corrosion. In the banded ferrite/pearlite structure, the carbon bearing phase (pearlite) is distributed in layers whereas in the other structures the carbon-bearing phases are much more evenly distributed. This variation in the distribution of carbon-bearing phases within the steel affects the corrosion resistance. During the rolling process of pipelines, the heterogeneous regions are elongated in the direction of deformation, forming layers rich in manganese. Due to low solubility, it is segregated to the interdendritic areas during solidification of the steel. These manganese rich regions would be anodic to the bulk thus create galvanic cells that allow corrosion to occur.

M.A. Lucio-Garcia et al. (2009) found that martensitic microstructure has the highest corrosion rate up to two orders of magnitude higher than that for steel with a ferritic + bainitic or ferritic microstructure. Steel with a ferritic microstructure has the lowest corrosion rate. This is because the grain size and number of precipitated particles for steel with a martensitic microstructure is bigger than those for steels with a ferritic + bainitic or ferritic microstructures. Bigger grain size adds to the surface area for corrosion due to the fact that martensite grain boundaries are more reactive than ferrite or bainite. Meanwhile, Ueda and Takabe (1999) found that tempered martensitic structure showed lower corrosion rates than ferrite/pearlite structure, but suffered localized corrosion (as cited in D. A. Lopez, 2003). In the martensitic steel, cementite is homogeneously dispersed. Due to the lack of anchoring, the corrosion products peel off partially.

Jia Guo, Shanwu Yang, Chengjia Shang, Ying Wang and Xinlai He (2008) stated that homogeneous microstructures, proper amounts of carbon content and fine carbon-rich phases produced by appropriate processes are beneficial to the corrosion resistance of steels. Uniform distribution of fine carbon-rich phases which results from appropriate carbon content increases weathering resistance of the steel.

CHAPTER 3: METHODOLOGY

3.1 Project Flow Chart

The project takes about 29 weeks to complete. In order to ensure that the project progresses smoothly and finished on time, its flow has to be well planned. The flow chart of the project is as shown in Figure 1. The Gantt chart is presented in section 3.3.

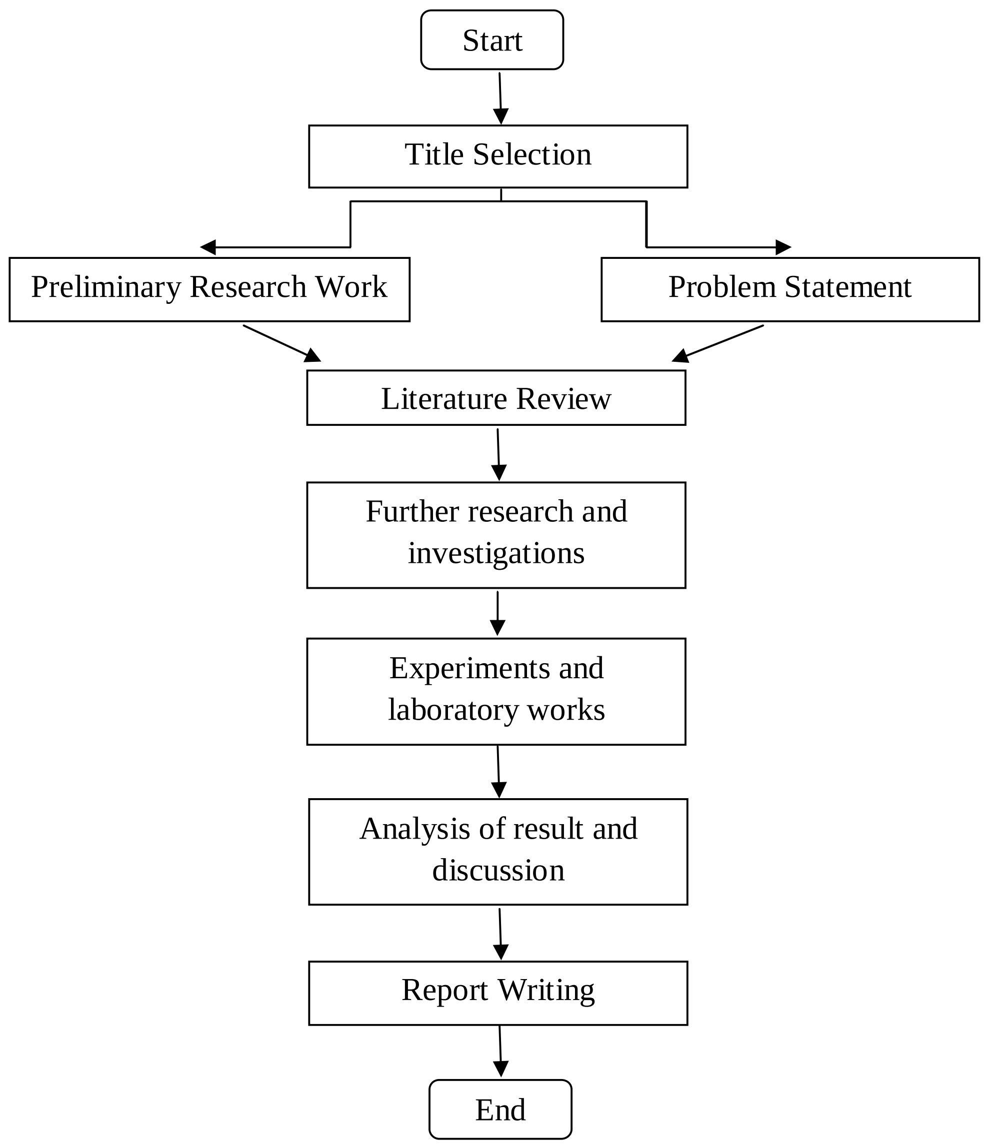

Figure 1: Project Flow Chart

3.2 Experimental Methodology

3.2.1 Sample Collection

To obtain various microstructures, samples are collected from different grades of carbon steel. Some of the samples are of the same grade but different type of heat treatments. The scope of collection is limited to grades of carbon steel which are commonly used for offshore pipelines. They are obtained from several sources, some of which from pipe manufacturers and some from Centre for Corrosion Research (CCR), Universiti Teknologi PETRONAS. The overall list of samples and their respective heat treatments is shown in Table 1. Mill certificates of the samples obtained from Kencana Petroleum are provided in the appendices.

3.2.2 Heat Treatment

One of the samples, which is API 5L X52 is heat treated in a furnace. It is heated to 900°C in 1 hour, dwelled for 1 hour, water quenched to room temperature, before heated again to 300°C. After reaching 300°C, it is let cool to room temperature inside the furnace.

Table 1: Details of Samples

Sample Number	Supplier	Manufacturer	Heat Treatment	Grade
1	Kencana	V&M	Normalized condition min 920°C cooling air	API 5L X42
2	Kencana	ArcelorMittal	Hot rolled above 860°C and cooled in still air	API 5L X42
3	Kencana	V&M	Normalized condition min 920°C cooling air	API 5L X42
4	CCR		Heated to 900°C, water quenched and tempered at 300°C	API 5L X52

V&M: Valllourec & Mannesmann Tubes
CCR: Centre for Corrosion Research, UTP
Kencana: Kencana Petroleum

3.2.3 Sample Preparation and Microstructural Examination

Sample preparation is done on each of the samples, involving sectioning, mounting, grinding, polishing and etching, according to this procedure:

1. The pipe is cut down to approximately 10 mm square by abrasive cutter.
2. The sectioned sample is hot-mounted with phenolics.
3. The mounted sample is grinded using grinder machine with wet silicon carbide paper. The grinding process starts with 120 grit paper, followed by 240 grit, 320 grit, 400 grit, 600 grit, 800 grit, 1200 grit and 2400 grit.
4. Diamond paste 1 micron is used to polish the sample on the rotating polishing cloth. After polishing, it is rinsed with distilled water followed by ethanol, before dried by a dryer.
5. The sample is then etched in 2% nital (nitric acid and ethanol mixture). After etching, it is rinsed with distilled water and ethanol. It is then dried by the dryer.
6. The microstructure of the sample is examined with Leica DM LM optical microscope at 100x and 500x magnification.

3.2.4 Corrosion Testing

3.2.4.1 Sample Preparation

Before corrosion test is carried out, sample preparation is necessary and done according to the following procedure, for each sample:

1. Sample is sectioned into small pieces, about 1 cm^2 each, using abrasive cutter.
2. The actual length and width of the sectioned sample are measured using digital vernier calliper. These measurements are used to calculate the surface area to be observed of the sample.
3. Copper wire is soldered to the sample and covered with a transparent tube.
4. A mixture of epoxy resin and hardener with epoxy resin-to-hardener ratio of 5:1 is weighted and slowly stirred until clear mixture colour is obtained.

5. The mounting cup is greased with release agent to ease the removal of the mounted sample.
6. The sample, along with the copper wire and the transparent tube is placed into the mounting cup. The epoxy resin mixture is then poured into the mounting cup until it covers a little bit above the tube level.
7. The sample is left for one day to allow the epoxy resin mixture to solidify.
8. After one day, the sample is removed from the mounting cup. It is labelled for easy identification.
9. The mounted sample is grinded using grinder machine with wet silicon carbide paper. The grinding process starts with 180 grit paper, followed by 240 grit, 320 grit and 600 grit.
10. After grinding, it is rinsed with distilled water followed by ethanol, before dried by a dryer.
11. Steps 9 and 10 are repeated with another sectioned piece of the same sample but without mount.

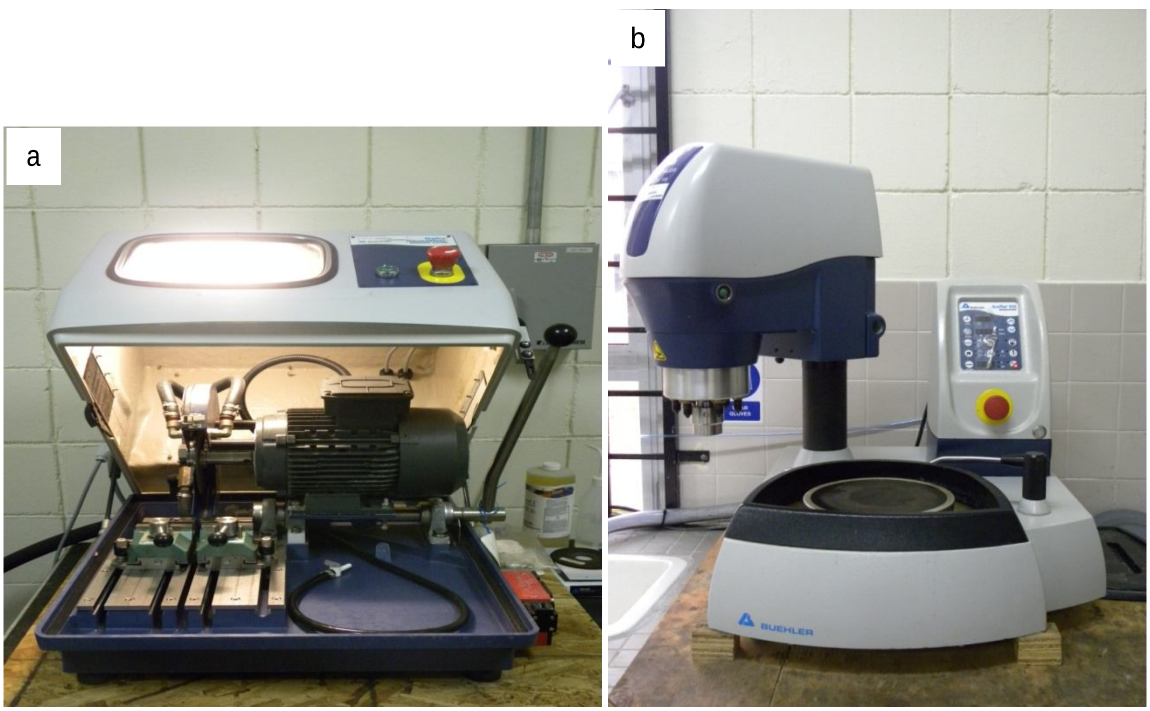

Figure 2: Equipments used for sample preparation. (a) Abrasive cutter (b) Grinder machine

3.2.4.2 Linear Polarization Resistance (LPR) Test

Glass cell is used to simulate the actual operational conditions of offshore pipelines and the CO_2 environment. The test is conducted in a static condition. Before the test is commenced, some of the necessary parameters are determined, as in Table 2.

Table 2: Test Parameters

Parameter	**Details**
Solution	3% NaCl
Temperature	50°C
De-oxygenation gas	1 bar CO_2
pH	4
Measurement Technique	LPR & EIS
Duration	2 weeks

The test follows the following procedure:

1. 30.7 grams of NaCl is weighted, mixed with 1 litre of deionised water, and stirred to achieve 3% NaCl solution.
2. The solution is purged by CO_2 gas for 1 hour to remove oxygen. The temperature is set to 130°C using hot plate to achieve solution temperature of 50°C at the end of purging process.
3. After purging process, sample is placed in the glass cell, along with other electrodes, and connected to the channels of the ACM Potentiostat instrument. The sample is left for 2 weeks.
4. Steps 1 to 3 are repeated for other samples.
5. After 2 weeks, the samples are examined under Field Emission Scanning Electron Microscope (FESEM) to view the sample surface and film formation. Elemental analysis is done with SEM – EDX.

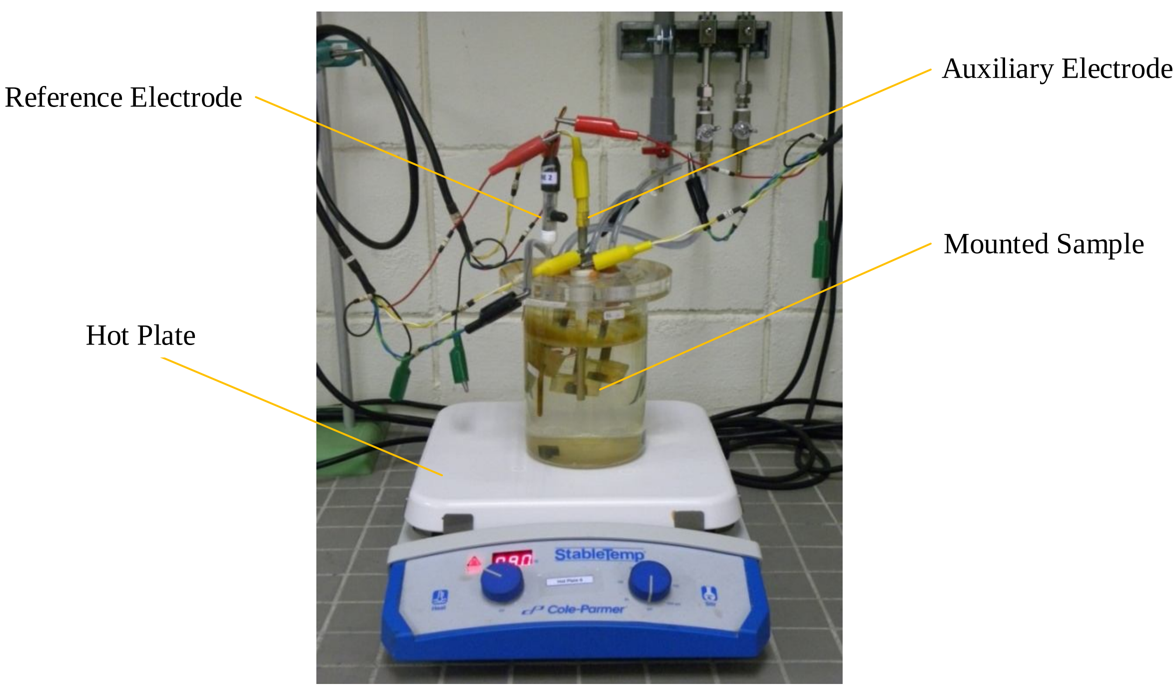

Figure 3: Equipments used. Top: Field Emission Scanning Electron Microscopy (FESEM), Bottom: Corrosion Test Setup

3.3 Gantt Chart and Key Milestones

3.3.1 Final Year Project 1

No.	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Selection of project topic														
2	Preliminary research work														
3	Submission of Extended Proposal Defence														
4	Proposal Defence														
5	Sample collection and continue research														
6	Submission of Interim Draft Report														
7	Submission of Interim Report														

Suggested Milestone

Process

3.3.2 Final Year Project 2

No.	Detail/Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Sample collection and preparation															
2	Corrosion Testing															
3	Submission of Progress Report															
4	Corrosion testing and result analysis															
5	Submission of Draft Report															
6	Submission of Dissertation (soft bound)															
7	Submission of Technical Paper															
8	Oral Presentation															
9	Submission of Project Dissertation															

Suggested Milestone

Process

CHAPTER 4: RESULTS AND DISCUSSIONS

4.1 Characterization of Microstructures of Various Heat Treatments

For characterization of microstructure, the samples can be divided into two groups, according to their grades, which are API 5L X42 and API 5L X52, for comparison purpose.

4.1.1 API 5L X42 Samples

Three samples (samples 1, 2 and 3) of this grade are examined. All of them are seamless pipes with normalising treatment, which is a heat treatment process where steel is austenitised before being allowed to cool in air. This process generates a microstructure of equiaxed ferrite and pearlite (D. Clover et al., 2004). Consistently, all of the samples of this grade have ferrite/pearlite microstructures. The microstructures of these samples are shown in Figure 4.

Sample 1 and sample 3 are austenitised to the same temperature (920°C) but differ in their diameter. Sample 1 is a 1 inch pipe whereas sample 3 is a 4 inches pipe. The microstructure of sample 1 is banded fine ferrite/pearlite whereas sample 3 has coarse ferrite/pearlite microstructure. The difference may be caused during the fabrication process of the line pipes. Although they undergo exactly the same heat treatment, the section of steel from which line pipe 3 was cut may have longer time allowance for the grains to grow. It was reported by D. Clover et. al. (1999) that banded microstructure is produced by preferential formation of pearlite along bands rich in manganese. More carbon-bearing phases (pearlite) are present in sample 3 as compared to sample 1. This may be due to higher manganese content in sample 3. Manganese increases the volume fraction of carbon-bearing phases present for a given carbon content (D. Clover, 1999).

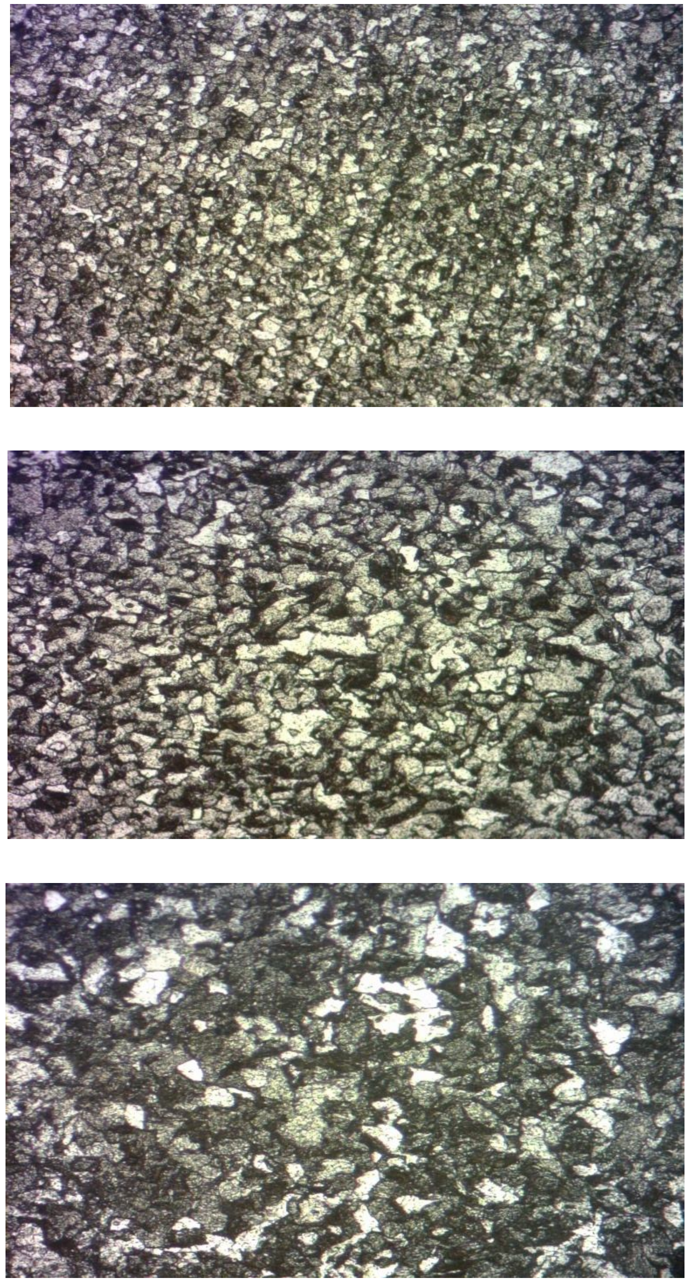

Figure 4: Microstructures of API 5L X42 samples etched in 2% Nital at 100x magnification. Top: Sample 1 (banded, fine ferrite/pearlite), Middle: Sample 2 (fine ferrite/pearlite), Bottom: Sample 3 (coarse ferrite/pearlite)

On the other hand, sample 2 is austenized to 860°C, which is lower than the other two samples, before being allowed to cool in air. This results in fine ferrite/pearlite microstructure. There are two factors that control the size of the new grains which are rate of transformation and size of the prior grains (John D. Verhoeven, 2007). Therefore, it can be understood that smaller austenite grains produce smaller ferrite/pearlite grains. To obtain smaller austenite grains, the austenizing temperature should be held as low as possible. Thus, the lower austenizing temperature, the smaller ferrite/pearlite grains that will be produced.

4.1.2 API 5L X52 Quenched & Tempered Sample

This sample has a combination of ferrite, bainite and tempered martensite microstructure, as shown in Figure 5. Rapid quenching in water prevents phase transformation, by providing a narrow window of time for the reaction to occur hence producing martensite structure. The sample is then tempered from 120°C to 300°C, allowing some of the martensite to transform into lower bainite.

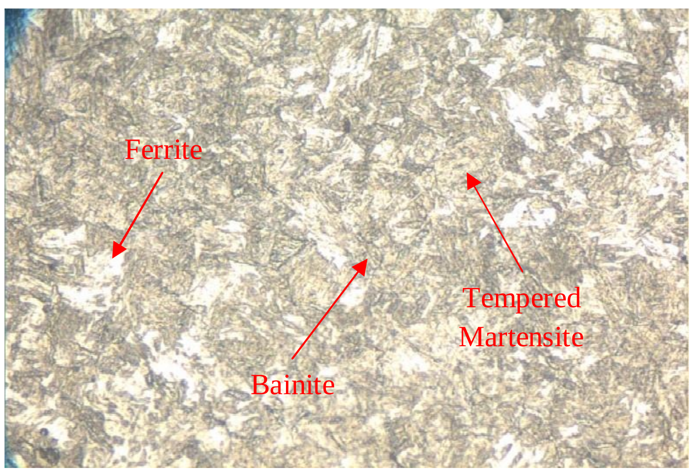

Figure 5: Microstructure of Quenched & Tempered API 5L X52 sample etched in 2% Nital at 100x magnification.

4.1.3 Summary of Microstructure of Samples

Generally, the samples are assessed in terms of the phases present, phase distribution, and grain size. The summary of microstructures of all the samples is as in Table 3.

Table 3: Summary of Microstructure of Samples

Sample Number	Microstructure
1	Banded, fine ferrite/pearlite
2	Fine ferrite/pearlite
3	Coarse ferrite/pearlite
4	Ferrite/bainite/tempered martensite

4.2 Influence of Microstructure on Corrosion Performance

4.2.1 Corrosion Rate from Linear Polarization Resistance (LPR)

Corrosion rates of each sample were recorded every hour throughout the duration of the experiment. The graph of corrosion rate against time is shown in Figure 6. It can be seen from the graph that for all samples, the corrosion rate slowly decreases as the experiment commenced and started to increase again after one week. This may be because after one week, adherent corrosion scales formed on the surface of the samples were slightly removed. As a result, the surface became less protected as compared to the initial condition. Average corrosion rates of the samples are summarized in Table 4.

Sample with banded, fine ferrite/pearlite structure has the highest average corrosion rate among all samples. D. Clover (1999) reported that samples with a banded ferrite/pearlite structure performed poorly in terms of localised corrosion. This was due to the segregated distribution of the iron carbide or cementite phase within this steel. Cementite is cathodic to ferrite, thus ferrite will be preferentially corroded in pearlite grains. This leaves the cementite as porous mass, providing firm foundation for the iron carbonate scale thus protecting the steel surface. Too much of localized corrosion may cause the corrosion rate to increase as well. In other microstructures, the cementite is much more evenly distributed. The cementite distribution is controlled by heat treatment. Quenched steel has a more even distribution than normalized steel.

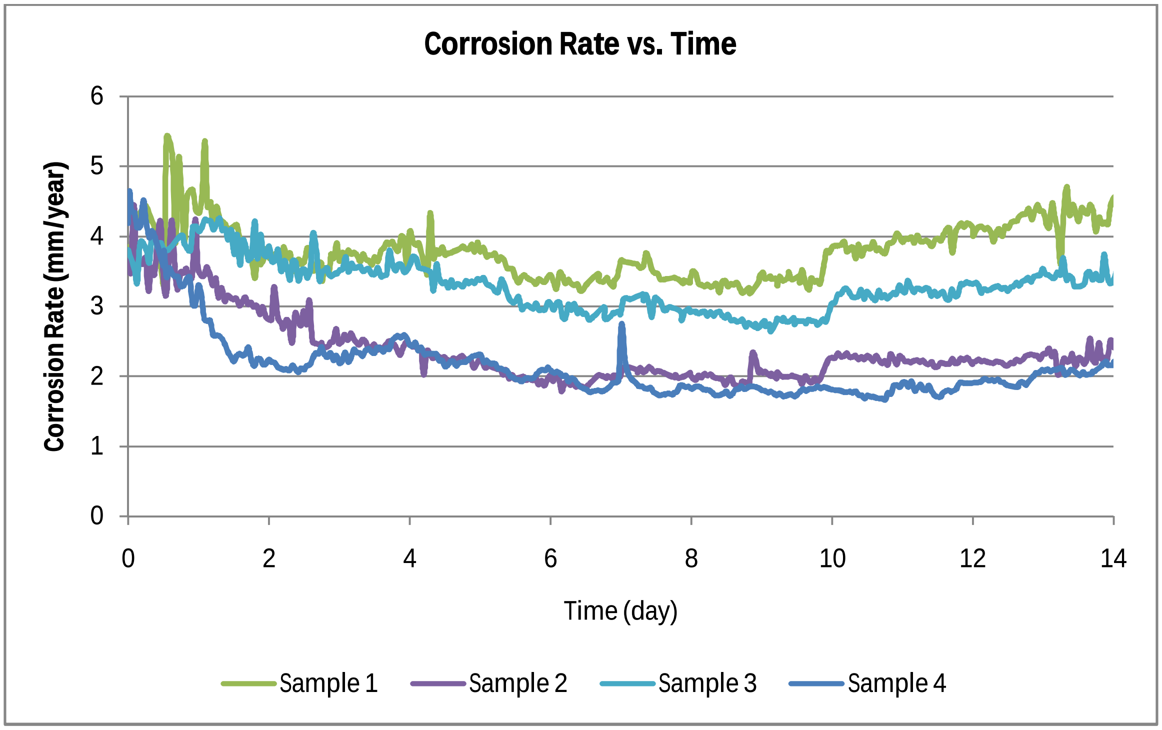

Figure 6: Corrosion rate trend of all samples in CO_2 environment with pH 4 and temperature, T = 50°C

Table 4: Summary of Corrosion Rates of Samples

Sample Number	Grade	Microstructure	Average Corrosion Rate (mm/year)
1	API 5L X42	Banded, fine ferrite/pearlite	3.8
2	API 5L X42	Fine ferrite/pearlite	2.4
3	API 5L X42	Coarse ferrite/pearlite	3.3
4	API 5L X52	Ferrite/bainite/tempered martensite	2.2

Corrosion rate of fine structure is significantly lower than that of coarse structure. When scanned by Field Emission Scanning Electron Microscope (FESEM), it was found that more iron carbonate film was present on the surface of sample 2 than it was on sample 3. Furthermore, the iron carbonate films on sample 2 surface are more evenly distributed and cover most of the surface area, whereas on sample 3 surface, some areas are not covered by the scales. This film or adherent scale provides protection against corrosion for the steel surface. FESEM images of these samples' surface are shown in Figure 7.

Ferrite/bainite/tempered martensite microstructure was observed to have the lowest average corrosion rate hence the best corrosion resistance.

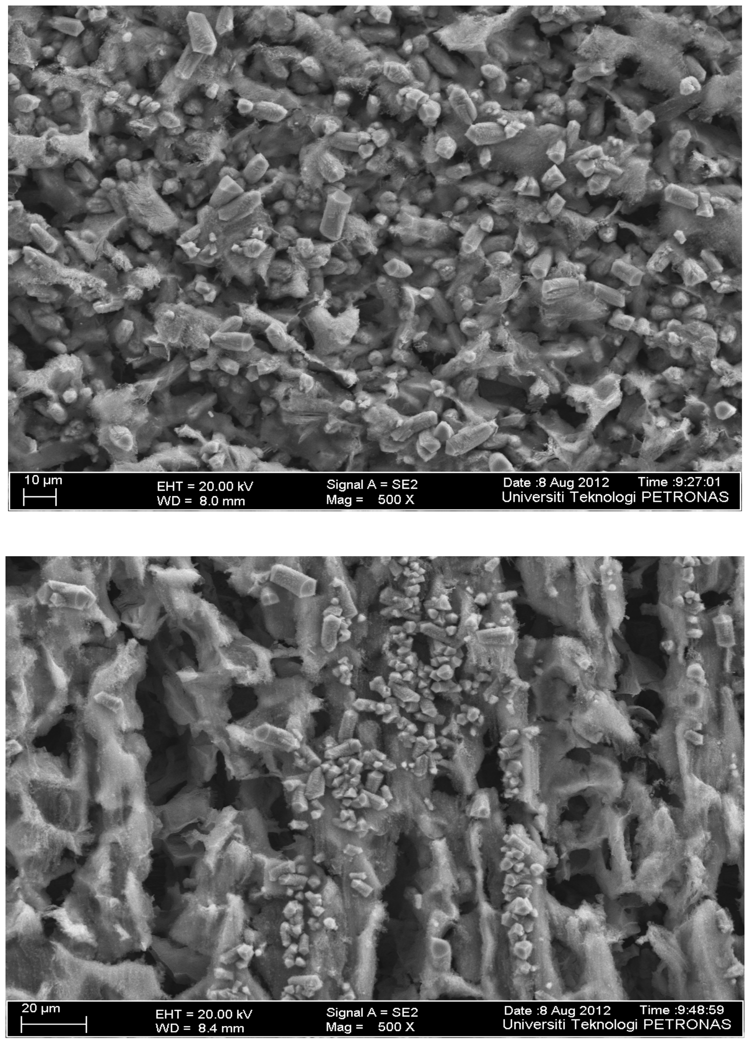

Figure 7: FESEM images of samples at 500x magnification. Top: Sample 2 (Fine ferrite/pearlite), Bottom: Sample 3 (Coarse ferrite/pearlite)

4.2.2 Corrosion Rate from Electrochemical Impedance Spectroscopy (EIS)

EIS measurement is performed three times throughout the experiment; at the beginning (0 hour), after one week and at the end (after two weeks). Based on the results, all samples indicate the same trend such that the corrosion rate declines after one week. The Nyquist plots for all samples are shown in Figure 8. The *x*-axis of the plot represents polarization resistance, R_p. The polarization resistance is inversely proportional to the corrosion rate.

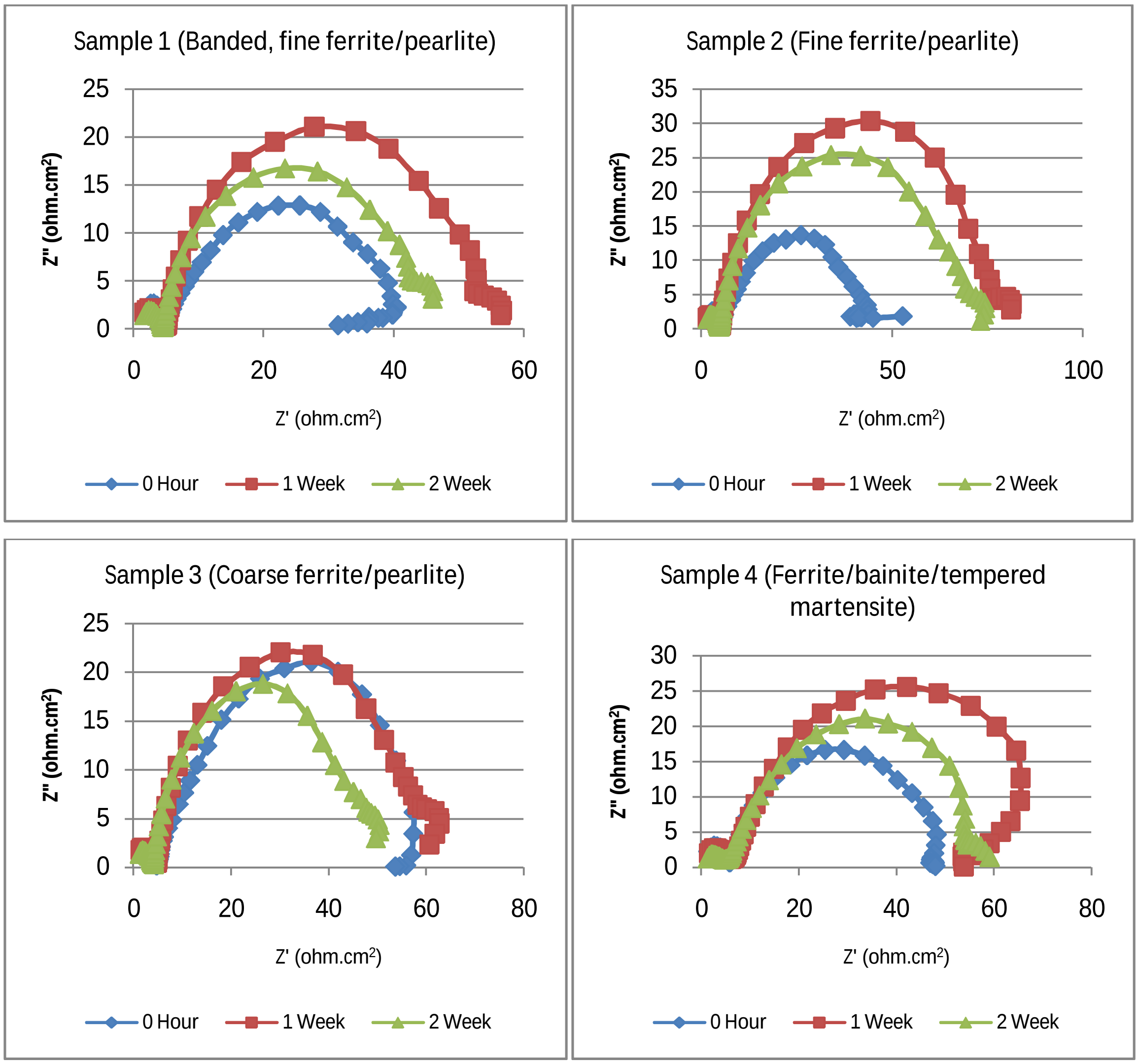

Figure 8: Nyquist plots for all samples

If the corrosion rates of all samples after two weeks are compared, the result slightly contradicts the findings in LPR. The corrosion rates from EIS after two weeks are summarized in Table 5. Corrosion rate of sample 2 (fine ferrite/pearlite) is lower than that for sample 4 (ferrite/bainite/tempered martensite). In LPR measurement, the average corrosion rate of sample 2 is slightly higher than sample 4. This suggests that fine ferrite/pearlite microstructure also has good corrosion resistance.

Table 5: Corrosion Rate after 2 Weeks

Sample Number	**Grade**	**Microstructure**	**Corrosion Rate after 2 weeks (mm/year)**
1	API 5L X42	Banded, fine ferrite/pearlite	7.6
2	API 5L X42	Fine ferrite/pearlite	4.7
3	API 5L X42	Coarse ferrite/pearlite	6.9
4	API 5L X52	Ferrite/bainite/tempered martensite	5.4

CHAPTER 5: CONCLUSION

5.1 Conclusion

The relationship between heat treatment, microstructure, and corrosion performance was investigated. The microstructure of carbon steels which are commonly used in hydrocarbon transportation mostly consists of ferrite and pearlite phases. However, it is more than just the phases that matters. The size of the grains, volume fraction of the phases and its shape may also have influence on the corrosion performance of the carbon steel. Therefore, these little variations are also taken into consideration for this project. The manganese content affects the fraction of pearlite present in a carbon steel. The higher the manganese within a steel, the larger the fraction of pearlite will result. In normalizing heat treatment, to obtain a finer ferrite/pearlite microstructure, finer austenite grains are required. For this reason, the austenizing temperature should be held as low as possible.

For this project, four samples are obtained, all with different microstructures:

Sample 1: Banded, fine ferrite/pearlite

Sample 2: Fine ferrite/pearlite

Sample 3: Coarse ferrite/pearlite

Sample 4: Ferrite/bainite/tempered martensite

From the experiment conducted, fine ferrite/pearlite and ferrite/bainite/tempered martensite microstructures show the best corrosion resistance. EIS results shows that fine ferrite/pearlite performs slightly better than ferrite/bainite/tempered martensite. It can also be concluded that fine structure resists corrosion much better than the coarse one. Morphology results from FESEM indicate that more iron carbonate scale are found on the steel surface of fine structure than the coarse one. The scales are evenly distributed and cover large portion of the surface whereas for coarse structure, some areas are not covered. Fine structure anchors scale better than the coarse one.

Carbon steel with banded ferrite/pearlite has the highest corrosion rate thus the poorest corrosion resistance. This is due to the segregated distribution of cementite within this steel. As cementite is cathodic to ferrite, preferential corrosion of ferrite within pearlite (lamellar structure of ferrite and cementite) grains leaves pores which anchor iron carbonate scale. Since the cementite is not evenly distributed, some areas are not protected against corrosion by the film. This tends to cause localized corrosion.

Studies on the influence of microstructure on the corrosion performance of carbon steel is going to be beneficial for the oil and gas industry. The understanding upon this subject will help in the decision of the best-suited material for offshore pipelines, particularly. Proper selection of corrosion-resistant material reduces the required corrosion allowance thus save costs and prolong the service life of the pipelines.

5.2 Recommendation

Several improvements can be made to this project for future work and investigation. To achieve more convincing result, more samples from each microstructure should be tested. The result would be firm and convincing conclusion can be made if all the samples of the same microstructure shows the same result. It would also be better if more variation of microstructures can be obtained. This would enlarge the scope of investigation and there may be other microstructure which has better corrosion performance.

The method of evaluation can also be improved by measuring the penetration depth to assess in terms of localized corrosion. Measurement of film thickness may also help in understanding the corrosion performance in terms of corrosion film mechanism.

REFERENCES

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1-03

V&M FRANCE (A01) TUBERIE SAINT SAULVE SAINT SAULVE ZONE INDUSTRIELLE 59880 SAINT SAULVE	VALLOUREC & MANNESMANN TUBES Vallourec Group	INSPECTION CERTIFICATE (A02) 3.1 EN 10204 : 2004 WBT / P.I. / 142 No. : 7208Sv10 (A03) Page: 1 / 5 Date: 02.08.2010

ITEM NO: 2-03
PO NO: RSSB/PO/0407/11/275
DO NO: 9734

ORIGINAL

(A12) MILL TEST CERTIFICATE	
(A01) **V&M FRANCE**	(A08.1) **V&M-Order-No.** MD1505 (A08.2) **Suborder** 856/5770 4
(A06.1) Customer PANTECH CORP SDN BHD PLO 234 JALAN TEMBAGASATU 81700 PASIR GUDANG JOHOR	(A07.1) Order-No. AR2165/03/10
(A06.2) **Orderer** /	(A07.2) **Order-No.** AR2165/03/10 **Date** 03.03.2010
	(A07.3) **Order-No.** PO/109982
(B01, B02, B04) **Description of the product**	Hot finished seamless line pipe Ends bevelled, angle 30° (+5 / -0), root face 1.6 mm (± 0.8) Inside without rust protection Outside dry varnish As rolled API spec. 5 L, PSL1, 10.2007 ASTM A 106 M - 08 / ASTM A 530 M - 04 A / ASTM A 53 M - 07 ASME SA 106, Edition 2007 / ASME SA 530 M, Edition 2007 ASME SA 53 M, Edition 2007 ASME Boiler and Pressure Vessel Code, Sect. II, Part. A, Edition 2007, Addenda 2009b Nace MR 0175 / ISO 15156-2:2003 / COR.1:2005 / EN ISO 15156-2:2003, Annexe A.2.1.2 Nace MR 0103-2005, Paragraphe 2.1 X 42 Grade B acc. to - API 5 L - ASTM A 106 M / ASME SA 106 - ASTM A 53 M / ASME SA 53 M

(A11)
Letter of Credit Text
Irrevocable documentary credit
No 007LC002054 dated 2010 06-07

Hot finished seamless Line Pipe
(as per PO/109982)

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21/10/11
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BUREAU VERITAS PARIS 1828 MALAYSIA Nº 856 SOUTH EAST ASIA REGION
PANTECH CORPORATION SDN. BHD. Documentation QC Passed Raja Fauziah
22/9/10

ROCKWELL C HARDNESS <= 22 GUARANTEED

V&M FRANCE (A01) TUBERIE SAINT SAULVE SAINT SAULVE ZONE INDUSTRIELLE 59880 SAINT SAULVE	VALLOUREC & MANNESMANN TUBES **Vallourec Group**	INSPECTION CERTIFICATE (A02) 3.1 EN 10204 : 2004 WBT | P.I | 142 No. : 7208Sv10 (A03) Page: 2 / 5 Date: 02.08.2010

(A13) V&M Item	(A09) Cust. Item	(B14) Item text	(B09) Dimensions	(B10) Single length
6			NPS 1 XXS OD-Tolerance + 0.015 in - 0.015 in WT-Tolerance + 15 % - 12.5 % Inside diameter may deviate from circularity	Fixed length 6000 mm + 100 mm - 0 mm

(A13) V&M Item	(A09) Cust. Item	(B07.1) Heat	(B08) Quantity	(B11) Total length m	(B13) Weight kg
6		62836	150	905,450	4.937

(C71)

HEAT CHEMICAL ANALYSIS

For each reduction of 0,01% carbon below 0.30 %, an increase of 0,06 % manganese above the specified maximum will be permitted to a maximum of 1,35% manganese.

(B07.1) Heat	(B15) Process	C %	Si %	Mn %	P %	S %	Cr %	Mo %	Ni %	Cu %	Ti %
min	-	-	0,10	0,29	-	-	-	-	-	-	-
max	-	0,22	-	1,06	0,030	0,030	0,40	0,15	0,40	0,40	-
62836	Electric (EAF)	0,16	0,20	0,62	0,016	0,006	0,14	0,05	0,08	0,14	0,001

(B07.1) Heat	Nb/Cb %	V %	B ppm	0002 %	0014 %	1003 %
min	-	-	-	-	-	-
max	-	0,08	-	0,15	1,00	0,43
62836	0,000	0,00	3	0,00	0,41	0,31

0002	V+NB+TI
0014	CR+CU+MO+NI+V
1003	CE = C+MN/6+(CR+MO+V)/5+(NI+CU)/15

PANTECH CORPORATION SDN. BHD. Documentation QC Passed Raja Fauziah 22/9/10

Heats fully killed

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(C72)

PRODUCT CHEMICAL ANALYSIS

For each reduction of 0,01% carbon below 0.30 %, an increase of 0,06 % manganese above the specified maximum will be permitted

V&M FRANCE (A01)
TUBERIE SAINT SAULVE
SAINT SAULVE
ZONE INDUSTRIELLE
59880 SAINT SAULVE

VALLOUREC & MANNESMANN TUBES

Vallourec Group

INSPECTION CERTIFICATE (A02)
3.1 EN 10204 : 2004

WBT	P1	142

No. : 7208Sv10 (A03)

Date: 02.08.2010

(A05, Z02, Z03)

Date	02.08.2010
Validated by	Inspection Representative
	As manufacturer : Valérie DELACROIX
☎	+ (33) 3 27 23 14 56
🖷	+ (33) 3 27 23 15 25
@	valerie.delacroix@vmtubes.fr
Stamp	

Indication in parentheses correspond to attributes according to EN 10168

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PANTECH CORPORATION SDN. BHD.
Documentation QC Passed
Raja Fauziah
22/9/10

ArcelorMittal South Africa Limited
Tubular Products
Genl. Hertzog rd.
P O Box 48 Vereeniging 1930
South Africa

Telephone +27 (0)16 450 4220
Fax +27 (0)16 423 4906

ITEM NO: 2.04
PO NO: RSSB/PO/640 #11/27
DO NO: 973Y

MATERIAL TEST CERTIFICATE
SEAMLESS TUBE

Licensed under API Spec 5L and 5CT
5L - 0319
5CT - 0419

ISO 9001: 2008
DE-395997 QM
Test Certificate EN 10204:2004 TYPE 3.1

ArcelorMittal

WBT - PL - 143

Customer:	PANTECH CORPORATION SDN. BHD.
Order No:	4000012620
Certificate Reference No:	040060124910
Product:	FULLY KILLED HOT FINISHED CARBON STEEL SEAMLESS TUBES
Specification:	ISO3183:2007/API 5L:2007 L245/L290/B/X42 PSL1 ASTM A106.08B/A53B.07/ A530.04 ASME SA106.08B/SA53B.07/SA530.04
Product Marking:	ARCELORMITTAL SA ISO3183/Spec 5L-0319 MONOGRAM 05-11 ASTM/ASME A/SA106B A/SA53B 60.300 (2.374) 8.740 (0.344) 6.000 (19.700) L245/B L290/X42 PSL1 SMLS TESTED 20.7 MPa (3000 psi) CAST NO:98B1728 PROD/O NO: T7641262010

Customer Order/Contract No:	SMY6 136109
Material No:	1000023001
Cast/Heat No:	98B1728 2.04

General Information

Quantity	Mass	Dimensions			Total Length	Steel making process	Final Rolling Operation
		Tube OD	Thickness	Length			
76	5,067.680(kg)	2" 60.300(mm)	160 8.740(mm)	6.000(m)	456.000(m)	Electric Arc	Final hot rolling operation finished above 860°C and cooled in still air.
	11,172.207(lb)	2.374(")	0.344(")	19.700 (ft)	1,497.200(ft)		

Chemical Composition

R05-(Cr+Ni+Mo+Cu+V) R22-(V+Nb+Ti) R24-(Nb+V)

Element(%)	C	Si	Mn	S	P	Cr	Ni	Mo	Cu	V	Al	Ti	Sn	Ca	N	B	Nb	CE	R05	R22	R24
Minimum	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Maximum	0.200	-	1.30	0.030	0.030	0.50	0.50	0.150	0.500	-	-	-	-	-	-	-	-	0.41	-	0.15	0.060
Heat	0.190	0.27	0.84	0.003	0.008	0.10	0.10	0.026	0.090	0.003	0.027	0.001	0.006	0.0008	0.0110	0.0002	0.0020	0.37	0.32	0.01	0.005
Product	0.190	0.270	0.840	0.003	0.008	0.100	0.100	0.026	0.090	0.003	0.027	0.001	0.006	0.001	0.011	0.000	0.002	0.368	-	0.006	0.005
Product (ADD)	0.190	0.270	0.840	0.003	0.008	0.100	0.100	0.026	0.090	0.003	0.027	0.001	0.006	0.001	0.011	0.000	0.002	0.368	-	0.006	0.005

Mechanical Properties

Specification	UTS (Rm)		Yield (0.5%)		% EL
Limits	MPa	psi	MPa	psi	50mm
Minimum	415	60000	290	42000	30.0
Maximum	-	-	-	-	-
(1) Actual	497	72083	347	50328	35.0
(2) Actual	493	71503	341	49458	36.0
(3) Actual					
(4) Actual					

	UTS (Rm)		Yield (0.5%)		% EL
	MPa	psi	MPa	psi	50mm
(5) Actual					
(6) Actual					
(7) Actual					
Orientation & type of tensile test			Longitudinal, Strip		
Width of tensile piece (mm)			19mm		
Orientation of impact test piece					

	OTHER TESTS
Bending	Passed
Flattening	Passed
Hydrostatic	20700 kPa (3000 psi) - 5 sec
NDI: EMI	PASS - ASTM E570 - 12.5% NOTCH
NDI: UT	UT not required
HV 10 Kg	156 157 158

Remarks:

Material in accordance with NACE MR0175:2003/ISO15156-2:2003, MR0103:2010. Dimensions to ANSI B36.10M-2004
The material conform to the hot yield strength requirements as per ASME, Sect II, Pt D, Table Y-1, 2010
All the material conform to the visual and dimensional requirements and is made to a suitable fine grain practice

Khin

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Quality Assurance Manager: R. Bester *Date of Issue: 2011.07.18* 13/9/11 *Certified by:* B

V&M FRANCE (A01) TUBERIE SAINT SAULVE SAINT SAULVE ZONE INDUSTRIELLE 59880 SAINT SAULVE	V&M VALLOUREC & MANNESMANN TUBES Vallourec Group	INSPECTION CERTIFICATE (A02) 3.1 EN 10204 : 2004 WB7 | PI | 381 No. : 12806Sv11 (A03) Page: 1 / 5 Date: 21.10.2011

ORIGINAL

ITEM NO: 2-5
PO NO: RSSB/PO/04071 5(R0)
DO NO: 10342

(A01) **V&M FRANCE**	(A08.1) **V&M-Order-No.** LW0457 (A08.2) **Suborder** 856/6302 1
(A06.1) Customer PANTECH CORP SDN BHD PLO 234 JALAN TEMBAGASATU 81700 PASIR GUDANG JOHOR	(A07.1) Order-No. AR2419/07/11
(A06.2) **Orderer** /	(A07.2) **Order-No.** AR2419/07/11 **Date** 05.07.2011
	(A07.3) **Order-No.** PO/125433
(B01, B02, B04) **Description of the product**	Hot finished seamless line pipe Ends bevelled, angle 30° (+5 / -0), root face 1.6 mm (± 0.8) Inside without rust protection Outside dry varnish As rolled API spec. 5 L, PSL1, 10.2007 ASTM A 106 M - 10 / ASTM A 530 M - 04a / ASTM A 53 M - 10 ASME SA 106, Edition 2010 / ASME SA 530 M, Edition 2010 ASME SA 53 M, Edition 2010 ASME Boiler and Pressure Vessel Code, Sect. II, Part. A, Edition 2010 Nace MR 0175 / ISO 15156-2:2009 EN ISO 15156-2:2009, Annex A.2.1.2 Nace MR 0103-2010, Paragraph 2.1 X 42 Grade B acc. to - API 5 L - ASTM A 106 M / ASME SA 106 - ASTM A 53 M / ASME SA 53 M
ROCKWELL C HARDNESS <= 22 GUARANTEED	

(A13) V&M Item	(A09) Cust. Item	(B14) Item text	(B09) Dimensions	(B10) Single length
10			NPS 4 SCH 40 OD-Tolerance + 0.031 in - 0.031 in WT-Tolerance + 15 % - 12.5 %	Fixed length 6000 mm + 100 mm - 0 mm

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(A13) V&M Item	(A09) Cust. Item	(B07.1) Heat	(B08) Quantity	(B11) Total length m	(B13) Weight kg
10	→	970758	48	289,810	4.668
		970759	59	356,280	5.742
			107	646,090	10.410

(C71)

HEAT CHEMICAL ANALYSIS

For each reduction of 0,01 % carbon below 0,30 %, an increase of 0,06 % manganese above the specified maximum will be permitted up to a maximum of 1,35 %

(B07.1) Heat	(B15) Process	C %	Si %	Mn %	P %	S %	Cr %	Mo %	Ni %	Sn %	Cu %
min	-	-	0,10	0,29	-	-	-	-	-	-	-
max	-	0,21	-	1,06	0,030	0,030	0,40	0,15	0,40	-	0,40
970758	Electric (EAF)	0,16	0,19	0,77	0,016	0,005	0,18	0,05	0,11	0,009	0,15
970759	Electric (EAF)	0,15	0,18	0,77	0,017	0,003	0,15	0,05	0,09	0,008	0,15

(B07.1) Heat	Al %	Ti %	Nb/Cb %	V %	N %	B ppm	0002 %	0014 %	1003 %			
min	-	-	-	-	-	-	-	-	-			
max	-	-	-	0,08	-	-	0,15	1,00	0,41			
970758	0,015	0,001	0,000	0,00	0,008	2	0,00	0,49	0,36			
970759	0,013	0,001	0,000	0,00	0,007	2	0,00	0,44	0,34			

0002	V+NB+TI
0014	CR+CU+MO+NI+V
1003	CE = C+MN/6+(CR+MO+V)/5+(NI+CU)/15

Heats fully killed

(C72)

PRODUCT CHEMICAL ANALYSIS

each reduction of 0,01 % carbon below 0,30 %, an increase of 0,06 % manganese above the specified maximum will be permitted up to a maximum of 1,35 %

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(C72)

PRODUCT CHEMICAL ANALYSIS

(B07.1) Heat	(C00.1) Test Piece	C %	Si %	Mn %	P %	S %	Cr %	Mo %	Ni %	Sn %	Cu %
min		-	0,10	0,29	-	-	-	-	-	-	-
max		0,21	-	1,06	0,030	0,030	0,40	0,15	0,40	-	0,40
970758	01FD736	0,17	0,19	0,76	0,019	0,004	0,15	0,05	0,09	0,009	0,16
970758	02FD736	0,17	0,19	0,76	0,018	0,004	0,15	0,05	0,10	0,009	0,16
970759	01FD738	0,17	0,20	0,77	0,013	0,004	0,15	0,05	0,09	0,008	0,17
970759	02FD738	0,17	0,20	0,77	0,013	0,004	0,15	0,05	0,08	0,009	0,17

(B07.1) Heat	(C00.1) Test Piece	Al %	Ti %	Nb/Cb %	V %	N %	B ppm	0002 %	0014 %	1003 %	
min		-	-	-	-	-	-	-	-	-	
max		-	-	-	0,08	-	-	0,15	1,00	0,41	
970758	01FD736	0,017	0,001	0,001	0,00	0,000	0	0,00	0,46	0,35	
970758	02FD736	0,014	0,001	0,001	0,00	0,000	0	0,00	0,46	0,35	
970759	01FD738	0,019	0,001	0,000	0,00	0,000	0	0,00	0,44	0,35	
970759	02FD738	0,020	0,001	0,000	0,00	0,000	0	0,00	0,44	0,36	

0002	V+NB+TI
0014	CR+CU+MO+NI+V
1003	CE = C+MN/6+(CR+MO+V)/5+(NI+CU)/15

(B04)

HEAT TREATMENT

NORMALIZED CONDITION MIN 920°C (1688 F) COOLING AIR

TENSILE TEST RESULTS

Type (C10.1)	Tube strip specimen
Test temperature (C03)	Room temperature
Direction (C02)	longitudinal

DOUBLE LENGTH

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FADZILLAH HARUN

V&M FRANCE (A01)	VALLOUREC & MANNESMANN TUBES	INSPECTION CERTIFICATE (A02)
TUBERIE SAINT SAULVE SAINT SAULVE ZONE INDUSTRIELLE 59880 SAINT SAULVE	Vallourec Group	3.1 EN 10204 : 2004 WB7 | P1 | 381 No. : 12806Sv11 (A03) Page: 4 / 5 Date: 21.10.2011

TENSILE TEST RESULTS

(B07.1) Heat	(C00.1) Test Piece	(C10.2) Dimension	(C11) YS	(C12) TS	(C13.2) Elong.						
		mm / mm2	$R_{p0,2}$ MPa	R_m MPa	2" %						
min max		- -	290 -	415 -	23,0 -						
970758	01FD736	18,80x6,20 117,15	316	493	32,8						
970759	01FD738	18,80x6,70 126,61	308	486	33,8						

(C10.2)

Dimension — Test piece dimensions
Test piece area

)50)

TECHNOLOGICAL AND OTHER TESTS ON SPECIMENS

Test	Conditions	Test rate	Result
Flattening test	Flattening test (specific)		Satisfactory

(D54)

OTHER TESTS ON PIPE

Test	Conditions	Test rate	Result
Hydrostatic test	18.3 MPA (183 BARS) 5 SEC	100% each lot	Satisfactory
Appearance & Dimensions	Aspect & Dimensions (spécifique)	100% each lot	Satisfactory
Residual magnetism	30 GAUSS MAX	100% each lot	Satisfactory

(A04, B06)

MARKING, IDENTIFICATION

10 Paint stenciled on one side

V & M FRANCE 5L 0061 API DATE OF MARKING A/SA53 A/SA106 114.30 X 6.02 10.79 B + X42 PSL1 S SMLS 18.3 MPA HT HEAT NUMBER SCH 40 AR2419/07/11 PO/125433 PANTECH - PORT KELANG - MALAYSIA LENGTH

(Z01)

The supplied products are in compliance with the requirements of the order

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...lication in parentheses correspond to attributes according to EN 10168

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