

**EFFECTS OF ARC WELDING CURRENT AND HEAT TREATMENT ON CORROSION OF WELDED MILD  
STEEL PLATES FOR UNDERGROUND TANK**

***BY***

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**(MEng/SEET/2017/7181)**

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**SEPTEMBER, 2021**

## Abstract

Corrosion is a major concern for owners and managers of petrol stations. This is because corrosion failure will adversely affect the lifespan of tanks. Leakages or ruptures caused by corrosion are commonly-identified problems in buried mild steel tanks which can lead to catastrophic failures, causing significant socio-economic losses for consumers and water utilities. Unlike in the pipelines where periodic inspection of the buried pipelines is vital, in the case of underground tanks appropriate mitigating measures via construction is preferable. The development of corrosion mitigation techniques, corrosion-induced deterioration of tanks remains a major problem globally, and billions of dollars are spent every year on the repair and cleaning of leakages into water/soil. Corrosion may alter the mechanical properties of metals due to chemical and micro structural changes at macro, micro or nano levels which impact their service lives. Up to present time, there has not been sufficient measured to mitigate corrosion for underground storage tanks (UST). Researchers have not explored this area comprehensively. This study showed that Corrosion mitigation in UST can best be achieved through proper development of production procedure in the joining of the mild steel plates. This was achieved through variation of arc welding current with electrode types and subsequent post heat treatment. A current of 80A, 90A and 100A were varied with Electrodes E6010, E6013 and E7018 respectively. A total of 54 coupons were gotten in all. Twenty-seven (27) coupons were subjected to post heat treatment. Nine coupons in each case of the post heat treated and non-heat treated ones were taken for micro structural examination and the rest were subjected to corrosion in aqueous acidified sandy soil in in bath for 60 days. It was observed that the heat treated coupons has a more refined grain sizes than non-heat treated counterparts and equally more corrosion resistance. Thus, it was established as a framework for UST construction using 5mm plate that the following stages be followed: preparation of 5mm plate; measurement and cutting of plate; and carrying out butt welding using current of 100A and E7018 electrode. Then, heat treatment of weld and HAZ to a temperature of 900°C, application of appropriate coatings, burying in a well prepared underground, covering with final backing sand (sandy soil only).

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## ABBREVIATIONS, GLOSSARIES AND SYMBOLS

HAZ	Heat Affected Zone
UST	Underground Storage Tanks
A	Amperes
g	Grammes
cm	Centimeter
%	Percentage
MMAW	Manual Metal arc Welding
E	Electrode
IGA	Intergranular Attack
MIC	Microbiological Induced Corrosion
SCC	Stress Corrosion Cracking
F	Ferrite
Fe	Iron
BDMAC	Benzyldimethylammonium chloride
E1	Welding Electrode E6010
E2	Welding Electrode E6013
E3	Welding Electrode E7018

C <sub>80</sub>	Current 80 A
C <sub>90</sub>	Current 90 A
C <sub>100</sub>	Current 100 A

## CHAPTER ONE

### 1.0 INTRODUCTION

#### 1.1. Background of the Study

In many industries today including diesel tank fabrication plants, engineers are concerned with efficient weldment and ways to prolong the life of the structures. According to Wema (2013), depending on the size of parts, welding had shown to be the best means of joining metals together. The reasons being that weld joints have high strength and are air tight for tanks and vessels. Also, weld structures can be altered easily and economically. It has also been shown that welded joints have considerable corrosion resistance when compared to other joining processes and different types of joints are possible in weld joining processes.

Mild steel plates are welded together in the construction or production of underground storage tank (UST). The weld joints and the Heat Affected Zone (HAZ) performs an important role in the behaviour of the UST in application. HAZ is the adjacent zone to the weld metal. It is a region of the base metal which is not melted but its microstructure and mechanical properties have been changed by welding heats. The microstructural features of the welds and the HAZ are responsible for any emerging properties in the weldment.

Mild steel plates like other metallic materials, when subjected to acidic environment are bound to corrode. Corrosion, which is the deterioration of a metal due to chemical reactions within it and the surrounding environment. It is severe at the welded and HAZ than the parent material due to compositional variation (Foss, 2008). The cyclic heating and cooling of the welding process alters the surface composition and microstructure of welds

compared to the adjacent base metal. The effects of segregation in macro and micro scales, precipitation of secondary phase, formation of unmixed zone, recrystallization and grain growth in HAZ results in variation in composition. The compositional differences create a potential difference between the welded portion and the parent material. In UST, the percolation of water cannot be exempted, the water molecule can percolate the microscopic pits and crack of any exposed metal. The hydrogen atoms in water molecules is capable of combining with other element or compound like carbon iv oxide ( $\text{CO}_2$ ) to produce acids, which can eventually cause more steel surface to be exposed. When chloride or carbonates ions are present, like in the case of salt water, the corrosion is likely to take place faster (Schmitt, 2006).

The corrosion of UST is a serious challenge to the industry due to the enormity of consequences. Apart from the losses that would be incurred by the tank owners for reinstallation and clean up, there is fuel loss and the environment is equally contaminated. The contamination is in two folds. Firstly, the contamination of the fuel in the tank which can result to poor efficiency of the fuel. In the second place, the fuel leaking into the soil may percolate into surrounding drinking water resulting to pollution. It is unfortunate that in Nigeria no effort or attention has been paid to this industrial problem. The American Environmental Protection Agency EPA has been investigating underground fuel storage tank leakages and also instituted several measures to curb this challenge. EPA (2016) of February research report on corrosion in underground tanks storing diesel fuel shows moderate and severe corrosion of metal components. For every 42 storage tanks examined, 35 (83 percent – of examined diesel fuel tanks) were found to moderately or severely corroded. Since the corrosion may potentially lead to contaminations in the environment



and many UST owners may be unaware their systems are affected, EPA usually alert owners.

This project essence is to determine the best combination of welding parameters that will give the most efficient corrosion resistance in our USTs. This is achievable through proper choice of welding parameters which is the chief means of tank fabrication and post heat treatment by normalizing to reduce the lock up stresses in the welded portion and HAZ.

## **1.2 Statement of the Problem**

Over the last decade, there has been a tremendous increase in demand of petroleum products like petrol, kerosene and diesel. This is as a result of the emerging need of this products in fast growing transport sector and household use. These products are usually lifted from the refinery or depot through heavy duty trucks and dispatched to underground tanks in fuel station where end-users purchase the products. This has led to the development and construction of many Fuel stations. Assessment of these established service stations have shown that majority of them has shut down operations due to problem of leakages in the underground tanks. The cost of exhuming tanks for reconstruction can be exasperating to the management. This has made many of the investors to loose huge sums of money and turning down investment opportunity in the sector. Leakages from underground tanks can lead to toxicity of crops and beverages which is not safe to human life.

Therefore, corrosion in underground tank mostly made of welded mild steel plates must be tackled to meet the ranging high demand of petroleum products. The welded portion and the HAZ are usually more prone to corrosion failure than any other parts of parent materials of the mild steel plate. Hence, careful selection of best combination of arc

welding current and electrode type that can give the optimum corrosion resistance is a necessity.

### **1.3 Aim and Objectives of the Study**

The aim of the research is to determine the effects of arc welding current and heat treatment on corrosion of welded mild steel plates for underground tank. The objectives are outlined as follows:

- (1) To investigate the effect of arc welding current on corrosion propagation of mild steel plate for underground tank.
- (2) To investigate effect of post-heat treatment on weldment and HAZ to mitigate corrosion failure in UST.
- (3) To develop a production procedure for fabrication of UST.

### **1.4 Justificatiion of the Study**

Most construction engineers, fabricators as well as designers use components and parts made from mild steel like plates and rods. In underground application, these comes in contact with salty water to form oxides on the surface leading to corrosion. Hence, corrosion is a general phenomenon. However, the compromised nature of the welded joints and the HAZ makes it more prone to corrosion to have a faster corrosion rate. This research will help to reduce the rusting of steel tanks and connected piping which is capable of causing a release of a regulated substance and also eliminate the associating cost with environmental clean-ups resulting from release by the UST.

## **1.5 Scope of the Study and Limitation**

Diverse engineering materials are available for usage in underground tank fabrication. The use of fibre materials has been found suitable in most advanced countries for underground tanks fabrication. However, due to different environmental conditions, economic suitability and technology factors, mild steel plate still remains the predominant material for underground tank construction in developing societies. This study is therefore confined to mild steel of 5 mm plate and a current range of 80 A to 100 A (80 A, 90 A and 100 A) only. Also in the corrosion aspect, this study is only concern with the best combinations of electrode and arc welding current to produce the most resistance to corrosion mild steel.

There were few limitations to this study. First, the study did not consider mild steel plates of  $< 5$  mm and arc welding current of  $< 80$  A and  $> 100$  A respectively. Also, the study did not consider the possibility of heat treating a large fabricated underground tank.

## CHAPTER TWO

### 2.0 LITERATURE REVIEW

#### 2.1 Theoretical Fundamentals

A theoretical fundamental of the issues as they relate to the main topic of this study has been conducted and will be presented below. It covers four main areas: mild steel plate for UST, arc welding process, and corrosion mechanism in mild steel.

##### 2.1.1 Mild Steel Plate for UST and its properties

Mild steel which can also be referred as plain carbon steel and low carbon steel is a carbon steel with 0.05% to 0.25% carbon, 0.70% manganese, 0.1% to 0.5% silicon and bits of other elements like phosphorus. It is considered to be the most predominant form of steel due to its lower price and wider range of applications. Its ductility and weldability make it suitable in underground tanks and other structural applications. The density of mild steel in approximation is  $7850 \text{ kg/m}^3$  ( $7.85 \text{ g/cm}^3$ ) and its Young modulus is  $29 \times 10^6 \text{ psi}$  (200 GPa). Mild steels have quite lower tensile strengths, while they are cheap, malleable; and surface hardness is capable of being increased via carburizing (Callister, 1997).

##### 2.1.2 Arc – Welding Processes

Arc welding is the highest widely applied methods among the welding processes and is the most highly acclaimed best, more economical and practical process of jointing metals. The heat needed to melt both the electrode and the base metal is generated from the arc by the flow current in the circuit. It is worthy of note that a wire of a bigger diameter will require more

quantity of heat to melt and therefore resulting in greater current. Thus, the amperage of the welding machine is increased (Ohring, 1995).

To create a welding circuit, one must have a source of electric power and a welding processes in which the welding machine is a part. The arc welding process involves using an appropriate electrode which is attach to the ground cable of the work piece while carrying out adjustment of the electric current to “strike an arc” that is, to create an extreme current which can jump between the electrodes and the base metals. The electrode is then moved along the line of the metal to be welded, giving enough period for the arc to melt the metal and not too slow to cause over flooding of the welded joint. The liquefied metal from the electrode is deposited in the joint and together with molten metal; edges solidify to form a solid job. The electrode is selected to produce the arc according to job specifications.

There are many arc-welding processes. They include: carbon arc, metal arc, argon arc, atomic arc, plasma arc, and sub-merged arc, welding processes. Manual metal is predominantly use for UST construction (Lange, 1995).

### **2.1.2.1 Manual Metal Arc Welding**

This welding type is one of the most commonly used methods for fabricating mild steel components. The factors that influence the weld quality are joint configuration, base metal chemistry, filler metal chemistry, position of welding, preheating, post heating, electrical characteristics like welding current, voltage and welding speed as well as the welding process adopted.

In manual metal arc welding (MMAW), consumable electrode is used to lay the weld. The welding power supply generates electric current in the nature of alternating current or direct

current which produce electric arc between the metals to be weld and the electrode. Upon laying of weld, the flux coatings of the electrodes degenerates, releasing fumes which serve as shield and also provides a zone of slag that protects welded area from atmospherically contaminants (Askeland, 2015).

Coatings also serve the following purposes.

- (a) The formation of a fast hardening slag layer, which protect the welded layer.
- (b) The formation of inert environs, which shields the pool metal from atmospheric nitrogen and oxygen.
- (c) The addition of deoxidisers and other alloying elements to replace those lost during the welding operation.
- (d) The addition of refiners to improve the grain configuration of the weld metal.

The versatility and simplicity of manual metal arc welding equipment and operation has made it to be ranked among world's commonest welding process. It predominates other welding processes in both repair and maintenance industries. Although, flux-cord arc welding is becoming popular, MMAW continues to extensively find application in the industrial fabrications and construction of steel structures. The mechanism is mainly to join iron and steels (Wema, 2013).

In striking the arc, a very light touch of electrode holder with the base metal is made, then electrode contact with the workpiece through slight pulling backward. This produces the arc and result in melting of the workpiece.

### 2.1.2.2 Current ranges for MMAW Electrodes

The core wires of electrodes for MMAW are generally 250 to 450 mm height and have a diameter within the range of 2.5 to 6.3 mm. As current is passed through the core wire, its resistance results in the generation of heat. If the temperature rises too high, the following undesirable effects may occur.

- (i) Melting of the electrode prematurely.
- (ii) Evaporation of moisture from the compounds in lengths of the core wire uncovered.
- (iii) Oxidation of some of the alloying elements affects the composition of the resultant weld.

There is also a lower limit of amount, below which the arc becomes unstable. Table 2.1 shows a typical current ranges for MMAW electrodes. The actual values, however, depends on the manufacturers' specifications.

**Table 2.1: Current range for MMAW electrode**

Cores wire Diameter (mm)	Current (Amperes)	
	Maximum	Minimum
2.5	90	50
3.2	130	65
4	185	75
5	250	80
6	315	120

(source: ASM, 1987)

### **2.1.2.3 Electrode selection**

Maximum strength on a welded joint can usually be made with any of a varieties of electrodes. The choice of the best electrode for maximum weld efficiency should be based on joint requirements. The joints are classified as freeze; fill and follow.

The freeze joints are joints welded vertically and overhead. The weld metal must freeze fast to keep the molten metal from spilling off of the joint. It is suitable for 3/16" to 5/8" (0.48 mm) plates. Fast freeze electrodes are also applicable for plates thicker than 5/8". The fill freeze low – hydrogen electrodes are used because of its high deposition rate and fewer large beads. The fill – joints are for groove, flat horizontal fillets and lap welds in plates over 3/16 (0.48 mm) thick. They require fast – full electrodes with high deposition rate to fill – up electrodes only weld flat and slightly down joints (15 maximum). Steeply inclined fill joints are best welded with fill and freeze electrodes.

The welding of sheet metal less than 3/16 (0.48 mm) thick requires electrode which weld at high speed with less skips and slag entrapments from undercuts. Fillet and lap joints in all welding positions are best performed with the Exx12 and 13 series of electrodes due to their fast – follow property. Other types of joints are done with fast freeze electrode due to its good follow freeze ability (Askeland, 2015).



The electrodes groupings and current selections are shown in Tables 2.2 and 2.3.

**Table 2.2: Electrode Grouping**

<b>SN</b>	<b>ELECTRODE</b>	<b>COATING</b>	<b>CURRENT TYPE</b>
<b>1</b>	EXX10	Organic	DC only
<b>2</b>	EXX11	Organic	AC/DC
<b>3</b>	EXX24	Rutile, approx 50 % Fe powder	AC/DC
<b>4</b>	EXX27	Mineral; approx 50 % Fe powder	AC/DC
<b>5</b>	EXX14	Rutile approx 30 % Fe powder	AC/DC
<b>6</b>	EXX12	Rutile (fast – follow)	AC/DC
<b>7</b>	EXX18	low hydrogen approx 30% Fe powder	AC/DC
<b>8</b>	EXX28	low hydrogen approx. 50 % Fe powder	AC/DC
<b>9</b>	EXXX18	low hydrogen approx 30%Fe powder	AC/DC

Source: (Kou, 2003)

**Table 2.3: Recommended current ranges.**

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ELETRODE SIZE		Flat/Horizontal	Overhead/Vertical	Max arc voltage.
inch	mm			
0.047	0.129	15 – 25	15 - 25	23
0.063	0.16	20 – 40	25 – 40	24
0.078	0.20	30 – 60	35 – 55	24
0.094	0.24	45 – 90	45 – 65	24
0.125	0.32	70 – 120	70 – 95	25
0.156	0.40	100 – 160	100 – 125	26
0.188	0.48	130 – 190	130 – 145	27
0.250	0.64	210 – 300	130 - 145	28
0.313	0.80	250 – 400	130 - 145	29

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Source: (Lange, 1995)

### **2.1.3 Production of UST**

A brief description of the various stages in the production of UST is hereby presented.

First is placing the mild steel plates on flat surface and brushing to free it of dirt and scales. This is preceded by welding and rolling processes. The welding is carried out through attaching the ground bead securely to the plate and setting the welding machine amperage to the specified current. Then, the electrode is fit into the holder and turning on the welding machine.

The hood is then dropped over the face and the arc is struck by brushing the base metal with the electrode. The distance between the electrode and the base metal was

approximately equal to the diameter of the electrode. Upon sticking of the electrode to the base metal, a quick twist was made to free the electrode. But if not, the electrode is made free through the electrode holder. Then, subsequent retry to stabilize the arc. When the arc has been stabilized, several beads are made on the plates to join them in pairs and quadrat. Each of the welded portions are adequately chipped and brushed.

When the tank has been fully welded and all external and interior accessories fully installed, it is buried in an excavated well prepared underground. The general installation procedure is based on ASTM standard.

#### **2.1.4 Corrosion**

Corrosion can be encountered in welded structure, when the properties, microstructure and composition of the welded component and HAZ differs from that of the base material. Corrosion can occur in several forms which could be general (uniform), localized, or microstructural specific attacks as the case may be. The corrosion rate associating with welds is usually higher when compared to the base metals. This is usually due to effect of combination of residual stress and microstructure alteration. Highly stressed region adjoining the welds can result in increased corrosion rate with respect to the parent metal (Salvarezza, 1980). For instance, for automotive metals, the first points where corrosion attacks take place in car bodies is the spot welds.

A lot of standard method of testing have been generated to measure corrosion susceptibility in diverse environment. Some methods of testing are accelerated to give room for long-term corrosion behaviors to be predicted from laboratory testing on short-term bases. Many of the methods can be carried out on welded components. Corrosion control of welded component is mainly a sensitive factor in the determination of the ultimate life, or service

fitness of the component. Corrosion can cover wide area when uniformly corroding a surface or concentrates locally to form cracks or pits. Corrosion is controlled by diffusion process; hence, it takes place on bare surfaces. In view of this, methods to mitigate the action of the exposed surface in the like of passivation and chromate conversion, can raise a material's ability to resist corrosion. However, few corrosion processes can take place with less visibility and unpredictably (Breakell, *et al*, 2005).

#### **2.1.4.1 Corrosion of mild steel welded joints in USTs**

One main constituent of mild steel is Iron which existed in nature as metallic oxide or salt. The oxide is more stable than pure iron. The process of refining to produce pure iron consumated a large energy input. This energy is stored and is utilized to supply the necessary driving force in returning the iron metal to its original state – an oxide or salt. The meaning of this is that iron, just like most metals are unstable and have the natural tendency to return to its original lower state or ‘corrode’. Corrosion in UST is a problem caused by nature and it is governed by energy changes (Wahid, *et al*, 1993).

#### **2.1.4.2 Forms of Corrosion**

Engineering alloys has been proven to corrode in diverse forms, some of which entail microscopic examination and others are macroscopic (seen with the bare eye). According to Fontana and Green, 1967, there are eight corrosion forms identified. Majority of them could be traced to degeneration and subsequent rupture of welded component, as enumerated beneath. The classifications were subject to corrosions' visual nature. The 9th form of corrosion which is found distinctly from others can be called microbiologically induced corrosion (MIC).

- (1) Galvanic
- (2) General
- (3) selective leaching
- (4) Crevice
- (5) Erosion
- (6) intergranular
- (7) Pitting
- (8) stress assisted
- (9) Microbiologically induced

Only basic features of the first-five of these forms of corrosion will considered in brief. Others like stress-assisted mechanism, intergranular attack (IGA) and pitting were covered in details because these ones are more predominant in welded components.

#### **2.1.4.3 Galvanic Corrosion**

When a pair of dissimilar metals with electrical or physical contact are submerged in relative electrolyte, or when the same metals are opened to electrolytes with diverse concentrations, galvanic corrosion emerges. For any galvanic-couple, the highest noble

metals (cathodes) corrodes at a slower rate and the more active metals (anodes) corrode at a very higher rate. When submerged separately, the metals corrode at their own rates. The determination of the type of metal to be used is a function of the galvanic series. Galvanic corrosion is a major concern in the underground jobs and elsewhere water that containing salts are in contacts with tanks or metal components (Johnson, 2006).

Certain factors like the relative anode size, metal type, and conditions of operation (salinity, humidity and temperature) affects galvanic-corrosion. The ratio of anodes to cathodes surface area relatively have effects on corrosion rates of the component. This corrosion can be averted most often by the usage of sacrificial anodes.

In summary, when different metals, twain in contact with corrosive and/or conductive medium, chemical potential difference emerges. The potential difference generates a flow of current between the both, when these metals in turn, are contact with each other in the presence of this same medium. By definition, the component where corrosion reaction (oxidation) take place is known as *anode*, and the component where reduction take place is known as *cathode*. Corrosion occurs in the anodic material if both components are electrically connected in a corrosive environment.

The most reactive metal (anode) will preferentially corrodes compared to the less active metal (cathode) in a galvanic couple. The attack can occur rapidly when the anode area is smaller relative to the cathode. Thus, the pertinence to ensure that parent metal is anodic to a weld metal and HAZ for environment of corrosion major challenges. In cases when the weld metal is anodic and there is large differential solution potential, high rapid attack of the weld metal is feasible. This is not a problem for some filler metal/base metal

combinations since the difference in potential is often minute. However, a recommendation has been reached, that the relative potentials of components be determined, when choosing materials for filler of different arrangements or when filler metals are of slightly different compositions than the parent metals (Breakell *et al*, 2005). This is predominantly vital if the welded component is prone to aqueous environments.

#### **2.1.4.4 General Corrosion**

Among many forms of corrosion encountered, general corrosion seems the most common and it is also the expensive (More than ten billion dollars is used annually, only in the United states to prevent or repair uniform attacks). General corrosion (Uniform attack) is known via electrochemical or chemical reactions at the surface of metal which give rise to uniform formation of corrosion products. This corrosion product, in steels' cases manifest as "rust" which is the oxidation of iron to produce  $\text{Fe}_3\text{O}_4$  (magnetite) and  $\text{Fe}_2\text{O}_3$  (hematite).

Uniform corrosion is chiefly a functions of compositions not microstructures. For cases where the weld metals and parent metals composition are almost the same (such as in homogenous and autogenous welding), the weld will only be slightly more disposed to uniform corrosion than the surrounding parent metal. More so, increasing corrosion rate in welded components are usually linked with residual stresses which can promote attacks in certain environment (De Waard, 1975).

#### **2.1.4.5 Selective leaching**

This entails the elimination of an element within alloy by corrosion mechanism. One commonest example is selective zinc depletion in brass alloys, which is generally referred

as dezincification. Welded regions are seldom prone to this form of attack except the base material.

#### **2.1.4.6 Erosion Corrosion**

This can be considered as mainly a general corrosion which is accelerated through corrosion mediums' relative motion. Many alloys and metals are prone to attack of this corrosion form. Metals like copper which are soft relatively and easily destroyed or wornout by mechanical abrasions, are mostly prone to corrosion by erosion. The welded region in most components are more vulnerable to attack of this form than the base component because of the softness (annealing) of the component emanating from weld heating cycles.

Corrosion of this form has been detected in the application of power generation, where fluid/steams are pumped with optimum velocities upon usage of impellers or rotors. The rotors and the piping system may be vulnerable to erosion corrosion. Weld pattern can be used to ameliorate erosion corrosion, in some situation. For instance, usage of 'hard-face' filler materials can locally elevate stiffness and eliminate or impede erosion connected with soft metals. For some cases, usage of filler materials with larger resistance corrosion can have likely outcome.

#### **2.1.4.7 Intergranular Corrosion**

Intergranular corrosion (IGC) is a localized attack adjacent to the grain boundaries with less or no attacks on the grains interior. The corrosion localization may be as a result of contaminations at the grain boundaries, alloy element enrichment at the boundary or



conversely, alloying element depletion. Iron enrichments of aluminum alloy grain boundary can cause IGC in a component. The depletions of Cr along grain boundaries, in austenite stainless-steels that result in IGC or intergranular attack (IGA) is a typical case. The phenomenon has been extensively studied in the alloys due to its ability to reduce the functional life of components in the power generation industries. Intergranular corrosion in 301-series austenitic stainless-steel is of utmost worry, since it takes place in environments where the alloys highly expected to exhibit exceptional resistance to corrosion. The weld HAZ is principally the dominant area of IGA in these alloys. The vulnerability to IGA emanate from local depletions of chromium next to the grain boundary, due to chromium carbide (Cr-rich;  $M_{23}C_6$ ) precipitation along the boundaries. By the time local chromium contents reduces below approximately 12.10 Wt%, the regions will witness 'stainless' cessations and this can lead to acceleration of attack. The phenomenon is still called sensitization, because the component is made 'sensitive' to IGC (Schmitt, 1978a).

#### **2.1.4.8 Pitting**

In this form, the attack is localized and results from breakage in the tiny passive oxide film which guard materials against the process of corrosion. Pits are emanated due to concentration cell, formed by discrepancy in solutions' composition, which relates with the alloying components. The compositions disparities result if the solution at a surface irregularity is distinct with the bulk solutions' compositions (Asadi, 2017). Once pit is formed, it behaves as anode, supported predominantly large cathode area. Pits have delay timing when commenced at specific microstructural features in the weld deposits (Wahid, *et al*, 1993). Pitting can also occur if component/solution combinations attain a potential that surpass a critical value, regarded as pitting-potential. The propensity for some given

alloy/solution combinations to form pit can sometimes be characterized by critical potentials for pitting, and re-passivation determined by cyclic potentiodynamic polarization techniques. Pits emerge more readily in metallurgical heterogeneous components (Savage *et al*, 1976).

Though welded metal has a higher chance of being attacked locally due to micro segregation within the dendritic layout, filler materials are now obtainable which have efficient pitting-resistance than the respective parent materials. Consumable suppliers can provide information about these filler metals. Nevertheless, even though the adequate filler material is used, pitting still takes place in unmixed zones, as depicted in Figure 2.1. Whereas in the subsurface, the corrosion attack may be substantial, there may be less observable damage on the surface of the component, (Savage *et al*, 1976).

Pitting is chiefly subjective to the metal composition and appears not to prefer welded areas over the surrounding parent material, unless exclusion of critical alloy elements has taken place. Elementally segregating in the process of weld solidification may promote localized pitting attacks (due to microscopic inhomogeneity).

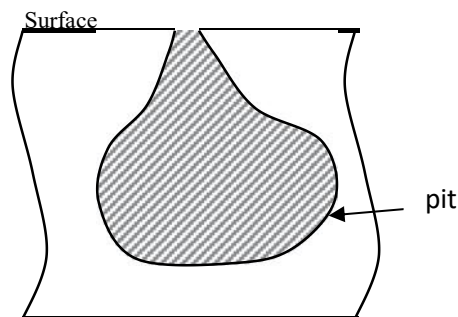


Figure 2.1: Illustration of pitting attack (source: Wahid, *et al*, 1993)

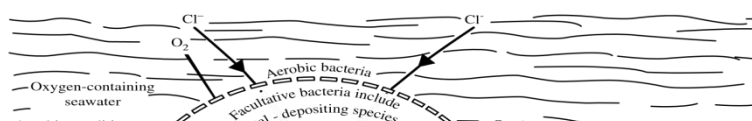
#### 2.1.4.9 Stress Corrosion Cracking

Stress corrosion cracking (SCC) is regarded as insitu cracks emanating from existence of a definite corrosive medium and tensile stress simultaneously (Wahid, *et al*, 1993). Fundamentally, all structural components including UST are prone to SCC, given the appropriate combinations of stress and environment. One oldest report about SCC was ‘season-cracking’ of brass cartridge artillery shell. Cracklings were often noticed in brass cartridge cases at the locations where cartridges were cling to the shell during seasons of heavy rainfalls or high humidity.

#### 2.1.4.10 Microbiologically induced Corrosion

Microbiological induced Corrosion (MIC) arises in some wet environs where anaerobic or aerobic bacteria’s basically attack the metal components. MIC often exhibits in pitting form, in which the metals surface displays a small hole or pit and rapid attacks happens sub-surface. MIC has been often misconstrued with normal pit-corrosion over the past. Nevertheless, it can be differentiated via existence of ‘tubercules’ of biological deposit and corrosion by-product over the pits (Li *et al.*, 1991).

This corrosion forms have been detected in both seawater and freshwater, and in most instances needs oxygen dissolved water to sustain the material dissolution reactions, as depicted in Figure 2.2. As overall, the presence of a particular ‘metal-munching’ bacteria, warm-water (20–40°C), an oxygen rich environments, and specific components are needed to enhanced MIC. This type of corrosion has been observed severally over different materials, including iron and steel (Edyvean, 1984). Austenitic stainless steel seems to be specifically prone, and the existence of 2-phase austenite and ferrite



microstructures, which is not the case of most welded metals that seems to influence proneness. MIC occurs specially in the ferrite phase as revealed by researches.

Figure 2.2: Mechanism of microbiologically induced corrosion (MIC). Source (Li *et al.*, 1991).

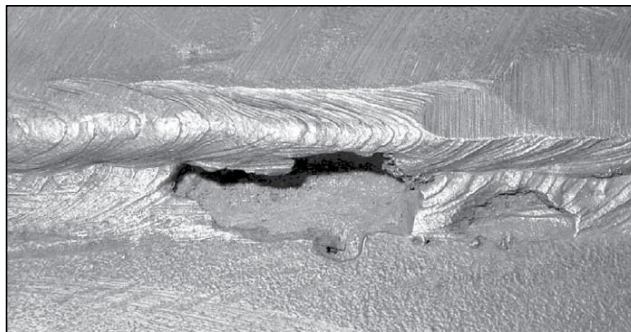


Figure 2.3: MIC attack in Type 308 weld metal Source (Li *et al.*, 1991).

A typical case of MIC that takes place during the construction of UST is depicted in Figure 2.3. It reflects a typical shielded metal arc welding from E308-16 electrodes, and joining of duo Type-304 steel plate in a butt-welding pattern. Ferrite contents of the welded component are connoted Fn-10 (~10 vol%). In the assembling process, water settlement at the bottom of the tank covering the weld were allowed as depicted in Figure 2.3. This produced the effective surrounding for MIC, as evidenced in rapidity of the welded metal attack.

## 2.2 Empirical Studies

### 2.2.1 Parameters Influencing the Quality and Strength of MMAW

The most common welding challenge faced in UST construction and other fusion welding is how to reduce the influence of welding procedure. It is usually possible to produce a weld metal with given desired properties under well observed conditions in a research laboratory or factory. However, in many circumstances on the field of application, it may not be practically possible to achieve the optimum parameters. In addition, since the MMA Welding method is very versatile; it is the most chosen method to work with in the most awkward situations. Having in mind understandably that the effects of welding parameters like pre heat treatment, welding speed, temperature and welding current, etc., should be reduced on the welded metal properties (Easterling, 2003).

#### **2.2.1.1 Welding Current**

This is one of the most prominent process variables. It controls the rate of melting of electrode, depth of fusion and the amount of melted base metal. A digging-arc is produced from extremely high current and the welded joints may melt through backing, producing burn-through. Other side defects of high current include: undercut, high narrow welded seam and extreme heat affected zone (HAZ). Too low welding current produces unbalanced arc within the cavities (Nippes, 1959).

#### **2.2.1.2 Travel Speed**

This has an important impact on the size of weld bead and penetration depth. Wetting action and high possibility of undercuts, porosity, arc blow and uneven bead shapes are reduced by too high travel speeds. Welding speed which is also referred as travel speed controls the amount of deposition of weld metal per unit length of the weld. Hence, the shape of bead is principally controlled by it. The heat input into the weld is increased by

too low travel speed, producing dense reinforcement resulting in slag inclusion. However, porosity is advantageously reduced since sufficient time is permitted for the gases to escape into the atmosphere during slag-metal reactions (Savage, 1980).

### **2.2.1.3 Arc Voltage**

This is a function of arc gap. The shape of the fusion zone and reinforcements are primarily governed by it. Too high welding voltage yields a flatter, wider and less deep penetrating weld. According to Viskanta (1998), extremely high voltage produces a hat shaped bead that is susceptible to cracking. Thick welding source of power offers constant current (CC) which may be alternating current (AC) or direct current (DC) but subject to types of electrode used. The most effective weld characteristics are mainly achieved upon DC power sources usage.

Voltage and current are means of measuring power in a welding circuit. The length of arc between the electrode and the work piece is controlled by voltage (Volts) which is governed by electrode diameter. A more practical means of measuring power in weld circuit is current expressed in amperes (Nippes, 1959).

### **2.2.1.4 Electrode Polarity**

The desired properties of the weld and the electrode being used are governed by polarity of the MMAW system. Heat buildup on the electrode results from direct current with a negatively charged electrode (DCEN), thereby increasing the electrode melting rate and reduces the weld depth. Polarity reversing such that the electrode is charged positively and the workpiece is charged negatively causes the weld penetration to increase. The polarity

changes over 100 times per second with alternating current, which creates even distribution of heat and provides a balance between penetration and electrode melting rate (Askeland, 2015).

### **2.2.1.5 Heat Input / Energy**

Heat generation by welding is governed by Energy. The heat generated is a function of current, voltage and welding speed. Equation 2.1 shows the Heat input.

$$Q = \left( \frac{V \times I \times 60}{Z \times 1000} \right) \times \text{efficiency} \quad (2.1)$$

Where

Q = Heat input (Kj/mm),

I = Current (A),

V = Voltage (V), and

Z = Welding speed (mm/min).

### **2.2.1.6 Interpass Temperature / Preheat**

This is the temperature of a workpiece prior each welding run deposition while preheat temperature is the first temperature at which a workpiece is heated. Both largely affect on the weld cooling degree. A maximum and /or a minimum interpass temperature must be stated and it is often not less than preheating temperature (Nippes, 1959).

### **2.2.2 Effect of post Heat Treatment on weldment**

Heat treatment entails heating the metal to a specified temperature, then holding the metal at this temperature for a certain period; preceded by cooling the metal according to the designated process.

The grain size, grade and shape of alloy or metal determines the temperature at which heating is done. In overall, a metal can never be heated higher above its upper critical temperature. When this is done, plastic deformation of the grains of the metal happens. The deformation is governed by the temperature at which it is heated and the chemical composition of the alloy or metal. The alloy or metal is then kept at this ascertained temperature to achieved uniform temperature through the entire mass. Time of heating is also governed by the shape and size of the components. Mainly, the period needed for heating the workpiece in approximation is 1 to 3 minutes for 1 mm thick section.

The main transformation in the properties of the workpiece happens during the process of cooling by the metal. The emerging component properties depends mainly on the degree at which the cooling happened. It also depends on the medium in which the cooling is carried out. Modern methods of cooling engage only five quenching media.

- (1) Caustic soda solution
- (2) Brine (Sodium chloride solution).
- (3) Water
- (4) Oil
- (5) Air

The different heat treatment processes mainly used in engineering practice are:



Normalizing, Annealing, Hardening, Tempering, Surface hardening and Case hardening. Only Normalizing will be considered in this review due to its peculiarity to the study review.

### **2.2.2.1 Normalizing**

Normalizing process on steel is performed for the following purposes:

- (a) For refinement of granular structures of the steel so as to enhance machinability, weldment and tensile strength.
- (b) To eradicate strains emanating from cold working processes like hammering, bending rolling, etc., which makes the metal ductile and malleable.
- (c) To eradicate dislocations caused in the internal structure of the steel resulting from hot working.
- (d) To modify certain electrical mechanical properties.

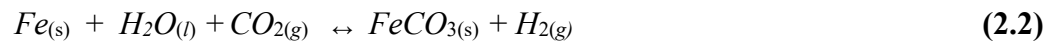
The process of normalizing involves heating the steel 30°C to 50°C beyond the upper critical temperature (for hypo – eutectoid steels) or line (for hyper eutectoid steels). This is preceded by being held at the said temperature for 15 minutes' duration and subsequently left to cool-down in still air.

This process yields homogeneous structures consisting of pearlite and cementite for hyper-eutectoid steels as well as ferrite and pearlite for hypo-eutectoid steels. The homogeneous structures results in higher ultimate tensile strength, impact strength and yield point with lesser ductility (Savage, 1980).

### 2.2.3 Mechanism, Testing and protection of Corrosion in UST

Hydrous carbon-dioxide corrosion environment is mainly envisaged in underground storage tank. It is often referred as “sweet” corrosion, in other to be distinguished it from “sour” corrosion which occurs in H<sub>2</sub>S environment though not covered in this study. The chemical and the electrochemical reactions taking place in corrosion processes are highly inter-dependent. Ability to understand their interaction has been very important in the development of mitigation parameter for CO<sub>2</sub> corrosion of mild steels.

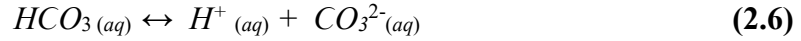
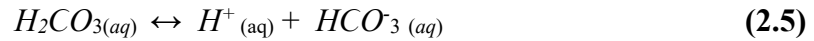
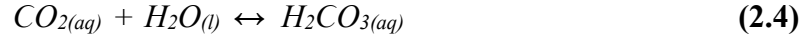
Numerous researches have been carried out to decipher the CO<sub>2</sub> corrosion mechanism over few past decades (De Ward, 1975, 1993; Bokris, 1961; Nescic, 1996, Schmitt, 2006). Reaction for general corrosion may be thus written:



These entities, H<sup>+</sup>, HCO<sub>3</sub><sup>-</sup> and H<sub>2</sub>CO<sub>3</sub> may undergoes cathodic-reduction reaction on the steel surface which balances the oxidizing iron decomposition. Mild steel corrosion in wet CO<sub>2</sub> solution without corrosion product protective formation, comprised of many process: development of reactant-species; conveyance of bulk solution reactants from the steel surface; electro-chemical reactions at the steel-surfaces; finally, transference of steel surface products the to the bulk solution.

Equations (2.3) and (2.4) depicts some major chemical reaction in CO<sub>2</sub> corrosion which includes CO<sub>2(g)</sub> water dissolution and carbonic-acid production. The carbonic-acid (H<sub>2</sub>CO<sub>3</sub>) is weak, and it dissolves in dual-steps equations (2.5) and (2.6):



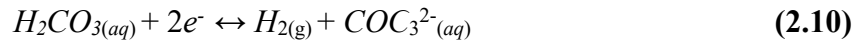
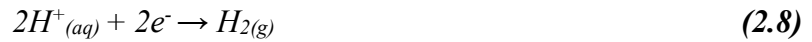


Electro-chemical reaction which take place on the surface of the steel include a cathodic reaction and an anodic reaction. The resultant product is Iron is oxidizing to ferrous iron with a release of two-electrons.



It is generally accepted that iron dissolution reaction takes place in series of sequence which includes intermediate-species (Bockris, 1961).

The widely agreed major reactions at the cathode portion are proton reduction, water reduction and direct carbonic acid reduction (Nesic, 1996).



For an aqueous CO<sub>2</sub> terrain, if the solution pH is lesser than 4, then, proton reductions (2.8) will be more. The direct reductions of carbonic acid (Equation 2.10) is very important and fulfils major roles in the determination of corrosion rate, if the solution pH, is in the ranges of  $4 < \text{pH} < 6$  (Schmitt, 1978(b); Hurlen, 1984; Nesic, 1996). The non-indirect reduction of carbonic-acid is the reason for more corrosion rates of mild

steel in wet CO<sub>2</sub> solution, than in strong acid-solution under similar pH condition (Nesic, 1996). In prediction of corrosion rate from high pH and low concentrations of wet CO<sub>2</sub>, water reduction, Equation (2.8) must be put into consideration.

### **2.2.3.1 Under Deposit Corrosion**

This is a localized form of corrosion which occurs beneath or around depositions which are found on steel tank internal surfaces (Johnson 1995). High corrosion rate may be seen underneath such solid depositions on the bottom of level lines (Espan, 2001). This type of corrosion has been cited recurrently as being liable for diverse damages found in the tank storage industries over the past recent years (Morita, 1994). The National Association of Corrosion Engineers (NACE) formed a pragmatic team aiding information dissemination among technologists and corrosion experts in respect of under deposit corrosion (Bockris, 1961).

Austere corrosion problems like pitting and crevice formation can be prompted by solid deposits in the tanks. This is because they provide an immediate environment that is physically and chemically different from the areas not within the deposit area (Li, 2009). For cases where inhibitors are contained in the tank content, these depositions of solids prevent inhibitors from getting to the surface of the metal. In the UST, the deposit composition may likely be complicated which leads to high complexity of fuel chemistry beneath those deposits for corrosion evaluation. O<sub>2</sub>, CO<sub>2</sub>, and often microbiological effects on corrosion processes are seen as the main contributing factor of failures in the UST (Achour, 2008).

A consensus has not been reached as to whether under deposit corrosion fit to “stand-alone” as corrosion process (Zhu *et al*, 2010). Within subsequent section,

advancement of under deposit corrosions will be reviewed and under deposit corrosion testing procedures will be assessed.

### **2.2.3.2 Deposits**

Deposits experienced in fuel storage tank sectors can be classified as organic or inorganic. The main predominant inorganic-deposit is sand which is in the nature of silica, SiO<sub>2</sub> (Shadley *et al*, 1998). SiO<sub>2</sub> sand is formed from the geological formulation and is conveyed with tank contents to depots. Many USTs have a volumetric concentration of sands which ranges from 1% -40% (Zhu *et al.*, 2010). Deposits that are inorganic also comprised corrosion products like carbonate of iron, oxides of iron and sulfides of iron etc., that emanate from corrosion processes and their consequence on corrosion have been investigated extensively (Johnson, 1991; Foss *et al*, 2008). Some other scales precipitation in water like barium sulfate and calcium carbonate are often referred as important parameters which interfere with corrosion and threaten the reliability of tanks.

Another example of organic deposits which can be seen in petroleum pipelines and tanks is Biofilm. Corrosion threats from microbiological influences are usually joined with under deposit corrosion in root cause failure analysis (Zhu *et al.*, 2010). On general note, the deposits observed in the field are combination of diverse inorganic and organic deposits. This review will only consider how non-organic solid silica sand affect CO<sub>2</sub> corrosion of mild steel and its function in promulgating localized corrosion.

### 2.2.3.3 Under Deposit Corrosion Mechanisms

In the recent past years, many researches have been carried out with respect to under deposit corrosion. Nevertheless, many of the jobs done were focusing inhibitors performances determination and valuation in situations when solids settlement happens. Lesser works have been done to reveal the procedures for explanation of under deposit corrosion mechanism in tanks and pipelines. It has been hypothesized often that the under deposit corrosion problem as a result of inadequate corrosion-inhibitors' penetration to the surface of the steel, owing to host depletion of corrosion-inhibitor on the surface of the solid deposits. The diffusion of inhibitors is slow down by solid deposit barriers. Conclusion was reached sequel on observation from inhibitors performance experimentations (Shadley *et al.*, 1998).

Murray (2007) carried out series of studies on the assimilation of the alkyl BDMAC (benzyltrimethylammonium chloride); on corrosion inhibitor by solid depositions using both centrifuge and syringe technique. It was revealed from the result excellent corrosion-inhibitor propensity for both iron sulfide and silica sand; with an intake of 93 - 95% correspondingly. These authors also pointed that the assimilation affinity was a function of the surface nature and the surface-area available to the inhibitor molecules. In the determining how much of the inhibitor can be loose by unplanned assimilation, it was believed that proper comprehension of collaborations between the inhibitor's aquaphobic tailing groups and their relations with hard surfaces are very important.

Miksic *et al.*, (2009) assessed the infiltration capability of the active components in different corrosion inhibitors rather than formulating products of corrosion-inhibitors,

that often have more than one real active part. Imidazoline, quaternary-amines, and sulfur containing organic molecules were studied. Adsorption experiment was conducted on individual active corrosion inhibitor component. The results show that sulfur compound and quaternary amine penetrates the sand-column quickly than imidazoline. The authors suggestion was that amine and imidazoline shared common adsorption mechanisms, whereas sulfur compound adsorption on sand was dissimilar. The authors were of the opinion that the ultimate inhibition performance would not depend only on the penetration rate, rather on the extent of adsorption of active components on the surface of the metal; even if the fast penetrating compound like sulfur containing molecules, display better corrosion inhibition capability in the corrosion experimentations. This was realized via steady state equilibrium of the active constituents in the aqueous phase; the hydro-carbon phase; on the solid phase and the surface of the metal.

Kedadam *et al.*, (1981) still studied threats of galvanic corrosion of API-5L/X65 mild steel and the behavior of imidazoline based corrosion inhibitors in sand deposit situations. Two kinds of experimental procedures showing both scenarios obtainable in the fields were investigated. First case was sand deposition under the conditions of inadequate inhibition connoted by sand deposit before inhibitors addition. The other scenario reflects the situation of sand deposition under conditions of sufficient inhibition (at least at the plain surface) which connote cases where inhibitors are added before sand deposition. In the experimentations, a sample, uncovered with sand was coupled galvanically through a zero-resistance-ammeter (ZRA) and another sample though of the same component but adequately covered with sand deposits.

From the experimental case of sand deposits before inhibitors addition, it was seen that without coupling to a non-sand covered sample, the rate of corrosion of the specimen covered by sand was unaffected by the additions of inhibitors. For the experimental case of inhibitors addition prior sand, austere localized attacks were seen, from both case of alkyl amnio-acid based inhibitor and imidazoline based inhibitor. The least case was gotten upon addition of alkyl amnio-acid inhibitor prior sand depositions. It was noted by the authors that the potential-difference among sand covered and un-covered samples were the dynamic force for the galvanic currents, nevertheless, the measured corrosion potential from the experimentations were not discussed elaborately. From other experimental studies by Pedersen *et al.*, (2013), cetyl-trimethyl-ammonium bromide (CTAB) inhibitors were noticed to be capable of changing quartz sand's surface from preferably water-wetting to intermediate-wetting, which was alleged of altering corrosion process on the surface of the steel underneath the sand deposit.

Papavinasam (2007) and Pandarinathan (2013) studied sand deposits effects on the surface morphology of 1030-carbon steels with the use of scan electron-microscopy and graze incidence X-ray diffractometer. The inhibiting and localizing corrosion processes were discussed from the perspective of steels' surface characteristic. The result shown both iron carbide and iron carbonate were present at the surface of the steel which serves as protection to steel surfaces when sand deposits were existent. The authors submitted that galvanic couple between un-dissolved iron carbide and the steel occupies a significant function towards yielding acceleration of under deposits corrosion. The imidazoline base inhibitors were discovered to have less effect in



protecting the steels' surface under sand deposits situation. However, upon absence of corrosive inhibitors, it was discovered the authors that sand deposits result in shifting of corrosion potential of the steels' surface negatively and this was opposes the reports recorded by other researches (Murray, 2007; Miksic, 2009).

Microbiologically induced corrosion (MIC) is equally believed to play significant roles relating to under deposit corrosion, especially in circumstances where sulfur containing compounds are existent (Pandarinathan, 2013).

#### **2.2.3.4 Major Factors of Wet CO<sub>2</sub> Corrosion in Mild Steel**

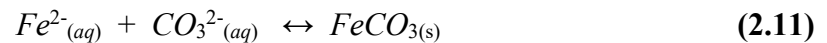
The processes of CO<sub>2</sub> corrosion of mild steels are gotten by the chemical-reactions within the tank contents, electro-chemical interactions happening at the surface of the steel in conjunction with conveyance of corrosive entities involved in all the interactions between the tank contents and the steel-surface. These processes with all reaction interacts with one other and numerous factors affects them.

##### **(a) Water Chemistry**

The key ions participating for water-chemistry analyzation of wet CO<sub>2</sub> corrosion mechanism comprises hydrogen ion H<sup>+</sup>, bicarbonate ion HCO<sub>3</sub><sup>-</sup>, carbonic-acid H<sub>2</sub>CO<sub>3</sub>, carbonate ion CO<sub>3</sub><sup>2-</sup> and ferrous iron Fe<sup>2+</sup>. Any variation of water-chemistry would significantly have effect on the process of corrosion. The H<sup>+</sup> concentrations in terms of pH have substantial and directly affects the kinetics in CO<sub>2</sub> corrosion. Lower pH, i.e., more quantity of protons which utilizes higher electrons leading to faster corrosion rate as shown in equation (2.8). Higher pH solution aids iron carbonates formation and this can significantly alter the corrosion processes (De Waard, 1975). Tanks solutions'

pH can be monitored and measured; however, the electro-chemical reactions (2.7 – 2.8) takes place through surfaces of the steel, where pH is readily difficult to be determined. Studies have revealed that if the surface of steel is covered with protecting layers, the pH of surface is one to two units more compared to the tank contents solution pH (Tanupabrungsun, 2012).

Iron carbonate precipitation as indicated in the reaction depicted underneath, likewise, has significant effects with corrosion reaction. The proportion of iron carbonate scaling is mainly a function of water-chemistry of the tank composition and temperature as well (Nesic, 2007).



### **(b) Partial pressure of CO<sub>2</sub>**

From the reactions (2.3 – 2.5), too high partial pressure of CO<sub>2</sub> results in speedy carbonic-acids formation and which produce higher concentrations of H<sub>2</sub>CO<sub>3</sub> in the solution. Thus, H<sub>2</sub>CO<sub>3</sub> reduction will formed more hydrogen, and rapid corrosion rates is achieved.

### **(c) Temperature**

All the reactions whether chemical, electrochemical or transport processes involving CO<sub>2</sub> corrosion are enhanced at high temperatures. Higher corrosion rate is obtained at elevated temperatures. Though, this is feasible if no protective layer is available on the surface of the steel. If ferrous carbonate precipitation of takes place, corrosion rate will really not be affected by temperature increase (Tanupabrungsun, 2012). Increased

temperatures facilitate ferrous carbonates formation, which changes the corrosion-kinetics and mechanisms (Schmitt, 1978b; Tanupabrungsun, 2012).

**(d) Effects of Flow**

As earlier mentioned, corrosion process is an interaction of chemical and electrochemical reactions, together with transporting of tank contents to and from within the periphery of the component. Flow promotes mass transportation process of corrosive entities to and fro on the surface of the steel, where electro-chemical reactions are occurring; thereby resulting in increased corrosion rates. Experimental observation has revealed enhanced flow condition that leads to rapid corrosion rates if there are no production of corrosion product layers (Schmitt, 2006). More so, when corrosion protective product layer is existent, the corrosion rate will be found not to be sensitive to rate of flow. Numerous researches have emerged to discover if flow could eliminate or rescind protective corrosion product layers or inhibiting thin layers (Nesic, 2003). However, consensus is yet to be reached. Corrosion processes is not only affected by flow rates but also by flow patterns and oil-water wetting system in real situations (Li *et al.*, 1991). When the flow rate is high, flow becomes an important factor for erosion-corrosion, though not common in UST (Shadley *et al*, 1998). In UST, instead of flow rate, trapped solids can deposit at the bottom of the tanks (Smart, 2009), resulting to under deposit corrosion challenges.

**2.2.3.5 Surface Corrosion Testing**

In welding construction, knowledge of corrosion behavior is pertinent in metal and filler metal choice making. While there are sufficient corrosion datas for many parent metals,

there are no adequate corrosion statistics to assist selecting best welding process and filler metal to avert corrosion attacks. In most cases, corrosion presents the hardest inherency pose to the service life-time of a welded components. Numerous examples of premature, and sometimes catastrophic failures, resulting from rapid corrosion or corrosion-related cracking are readily available.

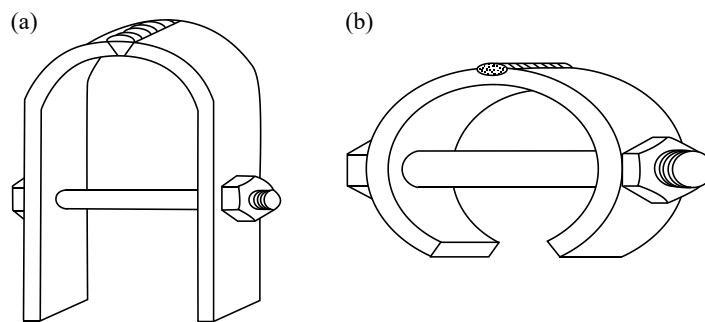
Fortunately, there are numerous corrosion testing methods developed over the years that can be used to ascertain the proneness of various corrosion forms. Most of them were already standardized via coding like the ones published by American society for Testing and Materials (ASTM) and the National Association of Corrosion Engineers (NACE). Many of the test procedures are intended for laboratory usage, and acceleration of attacks which might be encountered in real life application. They embraced: electrochemical tests, where an electrical potential is applied to the component and immersion tests, where workpieces are simply exposed to an austere environments and examined on interval basis (ASTM, 2014).

#### **2.2.3.6 Atmospheric Corrosion tests**

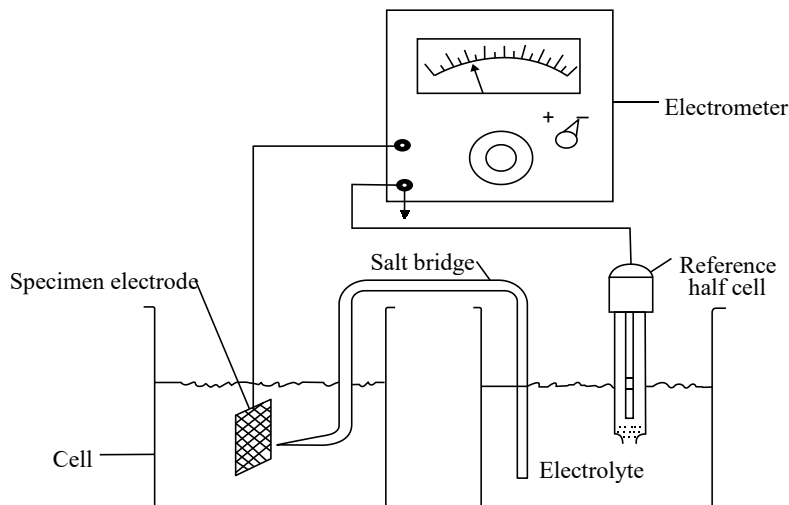
In simple exposure testing, workpieces are placed on a testing racket and observed on periodic basis. To Investigate general corrosion rate, workpieces are solely weighed and weight-loss is determined which can be translated to corrosion rate in mils/year (or mm/year). For another case, the workpiece can be observed purely by photographing to find the inception of pitting, staining, or other superficial defects. Welded components can be placed in U-bend design where the weld is bent in a linear direction to provide equivalent straining in the welded portion and HAZ.

### 2.2.3.7 Immersion Tests

For acceleration of corrosion attacks, samples are usually submerged in an aggressive solution. This test is extensively used to ascertain proneness to crevice corrosion, pitting and IGC. Many of the tests work very well for weld component workpiece. Weight-loss is a decent way to ascertain corrosion rates for base metal workpiece. For samples having welds, attacks can be insitu, and retrieval of the workpiece periodically from the solution is needed in determination IGA location. For instance, a welded Type-301 stainless steel workpiece can suffer IGC in the HAZ dully prior any attack take place in the parent material. Thermal simulation to produce sample with HAZ microstructures can use in some cases. Many kinds of immersion tests which are used to ascertain susceptibility to SCC, are also obtainable. Two common specimen-types used are the U-bend and C-ring patterns depicted in Figure 2.4 (Salvarezza, 1986). In the SCC immersion tests, the workpiece must be retrieved from the NaCl solution periodically to ascertain cracklings.



**Figure 2.4:** Immersion corrosion tests for welded specimen (a) U-bend and (b) C-ring (Salvarezza, 1986).



**Figure 2.5:** Electrochemical polarization tests configuration (Salvarezza, 1986).

### 2.2.3.8 Corrosion Inhibitors

Despite resistance corrosive alloys been successful to a large extent in handling corrosion, mild steel still remains, most effective and economic material for construction of UST, processing equipment and wells for the oil and gas sectors. Though, mild steel has poor resistance to corrosion. Therefore, controlling of corrosion and mitigation is a big threat to the efficacy of mild steel been a vital component for underground tank production. A major technique in the underground corrosion control is the usage of corrosion inhibitor. In accordance with the National Association of Corrosion Engineers (NACE), corrosion inhibitor comprised ‘a substance that retards corrosion when added to an environment in little quantities’. A lot of studies on corrosion inhibition’ as protection mechanisms and applications suitability to reduce corrosion damages have been conducted over the past two decade (Hackerman, 1984; Armstrong, 1994; Paria, 2004; Achour, M, 2008). In accordance with chemical functionality, corrosion inhibitors may be grouped as passivating (anodic) or cathodic (Mercer, 1994). The passivating inhibitors slows anodic reaction,

i.e., oxidation of metal through a shift of the corrosion potential to the more positive end. On the other hand, cathodic inhibitors, retards cathodic reactions. The proposition reached was that cathodic inhibitors afford protection to the steel surface through annulling cathodic-reactions or through precipitation on the vicinities where these reactions occurs. Organic inhibitors are the most often used corrosion inhibitors (Paria, 2004). These are actively surface chemicals which can be adsorbed on surface of the steel to form shielding film of adsorbent substance. The duo, cathodic and anodic interactions are understood to be slow down by organic inhibitor (Zhang *et al*, 2001; Osman, 2003; Moretti, 2004; Popova, 2004). The magnitude of inhibition protection is a function of, adsorbent capability of chemicals on the surface of the steel. Organic inhibitors are categorized as anionic or cationic inhibitors base on the particles charges of inhibitor ions (Mercer, 1994). The surface of a metal is often negatively electrified in pH4-6 solution, and cation inhibitors like imidazoline and amines are commonly used in corrosion inhibitors preparations (Zhang *et al*, 2001; Ismail *et al*, 2010).

Inhibitors can relate with steels in diverse ways, particularly commercial corrosion inhibitors that are formulated with multiple functional group. Hence, various mechanisms instead of individual inhibition mechanism ought to be considered when corrosion inhibitor is assessed. Normally, the corrosion inhibitor introduced must be able to disperse within the surrounding water and reach the surface of the steel tank. However, an inhibition process can be influenced by a lot of factors. Presence of other surfaces than a mild steel surface can alter the effectiveness of corrosion inhibitor. This is because the surface-active compound will likely not distinguish among the different surfaces and can be adsorb by whatever surface they encountered.

### **2.2.3.9 Surface Treatments**

If surface remedies are used to slow down corrosion, extreme care must be taken to guarantee total extensiveness devoid cracks, gaps and pinhole defect. Minute flaws can serve as source of weakness, permitting corrosion to pierce the innermost and producing extreme damages even though the external protective layers seemingly remain undamaged over a given time (Salvarezza, 1980).

### **2.2.3.10 Applied Coatings**

Painting, plating, and enamel usage are the highest dominant anti-corrosion remedial methods. They function through provision of barriers to corrosion - resistant materials along the disparaging surrounding and the structural component. Apart from manufacturing and cosmetic concerns, there are substitutes in mechanical plasticity and abrasion resistance as well as elevated-temperatures. Plating often stall in thin segments and if there are more inert than the substrates (for instance, chromium on steel); the galvanic couple will make a bare surface to deteriorate faster than non-plated exteriors (Fontana, 1967). Due to this, plating with active metal such as cadmium or zinc is the best. The surface becomes unpleasant with rusting excesses, when the zinc coating is not dense enough. Service life configuration is proportionally correlated to the material coating depth. When painting is done using brush or roller, tight spaces is well needed, and spraying is suitable for well coated areas like steel decks as well as water-front application. Pliable poly-ethane coating in the like of Durabak-M26 for instance, can offer an anti-corrosive sealant with high durability slipping resisting film. Though temperature and humidity can make drying time



to differ, painted coatings are comparatively easy to apply because they require fast drying times.

#### **2.2.3.11 Reactive coatings**

In an accordance to Mercer (1994), corrosive inhibitor can usually be added to a surrounding when it is orderly (mostly in circulatory controlled systems). The chemicals produce electrical insulator or chemical impervious coats on the revealed surface of the material, to subdue electro-chemical reactions. This kind of procedures make the structure less active to scratch, and defect in the coatings can handled since additional inhibitor can accessed anytime the material is exposed. The chemistry of corrosion inhibition entails of some salt compounds in hard water (Roman water systems are best known for their mineral deposits) like phosphates, polyaniline, chromates and other conductive polymers as well as varieties of special-designated chemical like surfactant (with long-chain organic substances having ionic functional group).

#### **2.2.3.12 Anodization**

Metal alloys like those of mild steel can usually undergo external treatment. The electro-chemical surroundings of a bath are cautiously tuned so as to ensure even pores of few nanometers width in all metallic oxide films. The apertures permit the oxides to grow more than passivation condition can tolerate. A harder surface than normal surface layer is produced when the treatment ends, the pores are all sealed. Usual passivating process takes control of protecting any damage areas when this coats are itched.

Anodization is highly resistant to wearing and deterioration, thus, it is mainly used in structural exteriors and other situations where frequent contact with the surface elements

are pertinent. Because of its high resiliency, it must be cleaned regularly. When left uncleaned, plate edge streak will certainly surface. Anodizing method converts an anode to cathode through adjoining of more reactive anode in connection with it (Osman *et al*, 2003)

### **2.2.3.13 Cathodic protection**

Cathodic protection (CP) is a system of controlling the corrosion on a materials' exterior through making the surface an electro-chemical cell. This kinds of protection techniques are mainly for protection of steel components in pipelines, tanks; steel pipes and ships (NACE, 2014).

### **2.2.3.14 Sacrificial anode protection**

For efficient CP, surface potential of the steel is antipodal (pushed) of higher negative, till the metal exteriors have constant potential. With constant potential, driving-force for the corrosion propagation is stopped. In galvanic CP systems, the anode material deteriorates through the influence of the steel and in the long run it must be substituted. Polarization is driven by electrode potential difference between the anode and the cathode which is as a result of flowing current from the anodic to the cathodic. Zinc, Aluminum, magnesium and similar alloying elements are the most common sacrificial anode materials. Magnesium has the maximum driving voltage while Aluminum has the highest capability and hence, magnesium usage at higher resistance necessity. Zinc serves for multi-use which is the reason for usage in galvanization (Hackerman, 1984).

### **2.2.3.15 Impressed current cathode protection**

In large designs, galvanizing anode cannot will not effectively supply sufficient current to provides adequate protections. Impressed current cathodic protection (ICCP) system uses anodes which is linked to DC power root (like cathodic protection rectifiers). The Anodes for ICCP system are tube-like and mass rod-like shapes of different specified components. They include mixed metal oxide, high silicon cast iron, graphite niobium coated rods or platinum coated titanium and cords (Petersen, 2012)

### **2.2.3.16 Rate of Corrosion**

Weight loss method is one of the simplest experimentation for determining corrosion. This method entails exposure of a clean weighted sample of the metallic or alloying material to an active corrosion medium for a given period of time preceded by cleansing to eliminate corrosive scales and weight measurement of the sample for weight loss determination. According to Gaudet (2010), rate of corrosion (R) is calculated thus:

$$CPR = KW/dAT \quad (2.11)$$

From the equation K is a constant,  $\dot{W}$  is metal weight loss for a period of time T, A is surface metals' exposed area, and d is the metal density (in g/cm<sup>3</sup>).

Penetration depths and mechanical properties are some other main factors for the corrosion rate.

### **2.2.3.17 Tactical Considerations of Underground Corrosion of Mild Steel**

Total removal of corrosion failure tendencies, that is, corrosion reduction in well risky cases is seldom obtainable in preexisting underground tank. undoubtedly, this will

necessitate substantial design alteration, for instance, replacing current components with corrosive resisting materials or adjustment of the process (e.g., dehydration gas addition). In fresh underground tanks, such corrosion elimination mechanism will raise a major design and engineering challenges, even on costing. Therefore, an effective corrosion controlling will be embraced if the objectives of increasing failure time of mild steels more than expectant life of the tank is realistic. For these tactics to be adopted, there is need for:

- i. the service life (performance target) of the corrosion control to be defined, and
- ii. measures been put in place to guarantee the goals are achieved.

Target yielding goal might be fixed in terms of an allowable metal penetration rate. When uniform corrosion is expected, this method will mainly be applicable. On the other hand, limits might be fixed on some parameters that has indication of fluid corrosivity, for example, electrode potential in anodic and cathodic protection system, dissolve oxygen in oilfield water injection, or boiler feedwater, temperature, pH, or dewpoint. Regardless of the method applied, it is vital to obtain on-line information to make modifications as oblige. Hence, corrosion checking is important and it is an integral essential section of corrosion managing plans (Popova *et al.*, 2004).

### **2.2.3.18 Corrosion of Metal Pipes in Soil**

One of the prime objectives of this study is to mitigate corrosion of underground. Contextual knowledge of soil, the properties and how they can affect corrosion of submerged tanks are principal phase required prior carrying out studies relating to corrosion in soil. Hence, it is vital to review briefly the chemical and physical features of concern soil and their relation to corrosion in an underground tank.

Generally, soils are categorized based on their size ranges. Clay, silt, and sand are the commonly used soils which are named as such due to their inorganic contents and size. Sand can be classified as coarse (0.20-2.00 mm) or fine (0.02- 0.2 mm). The size range of silt particles is 0.002 to 0.02 mm, and clay aggregates have size range of <0.002 mm diameter to colloids matters. Another Soils classification standard which is based on the aggregate sizes is ASTM (D-2488) (2017).

Air-pore-space of soils can be seen as 'the percentage soil volume at any particular moisture contents occupied by air' (Romanoff, 1957). It measures the absorbency of the soil to water and air. Higher air-pore-space value of the soil indicates the lesser corrosion of submerged component (Romanoff, 1957). Apparent specific-gravity indicates soil minerals compactness.

With regards to soils' physical properties, mass shrinkage is the ability of soil to commence crackling upon drying and swelling if aqueous. This behavior is particular to loam and clay soil aggregates. If silt/clay soil dries up, they form crack which give room for oxygen diffusion to the tank/pipe and thus its vulnerability to increase corrosion. Due to less retention in silt and clay, the capillary-pores of these soils embrace substantial quantity of water. Water retaining capability of any soil is its moisture equivalent. Numerous ASTM standards is used in determining the soils' physical properties. ASTM (D-2488) (2017) can be used for water-retention capability of peat materials. Moisture content in effective conductive soil signifies enough ion contents and the likelihood of high reactive corrosion attacks.

Soils' chemical behaviors have effect on the corrosion of submerged tanks and pipes. Sodium, calcium, potassium, and magnesium, as well as other acidic substance like carbonates, sulfates, nitrates and bicarbonates; are the chemical compounds that has responsibility to cause corrosion. Conduction of electric current (corrosion) mainly relies on salts solubility and moisture saturation of the soil. A lot of high volume literatures on soil chemical-analysis and other analytic methods are available for reference (Papavinasam. 2007). The pH measures the soils' acidity or alkalinity, or logarithm of the reciprocal of hydrogen ion concentration. pH of 7 indicates soils' neutrality but soil below it, are acidic and above it, alkaline. Soils' pH can be measured using sensors or ASTM D2488 (2017). The underneath levels of pH connote soils' nature.

**Table 2.4: Acidity and pH Ranges**

ACIDITY	pH RANGES
Very strong alkaline	9.1 higher pH
Strong alkaline	8.5-9.0 pH
Moderate alkaline	7.9-8.4 pH
Mild alkaline	7.3-7.8 pH
Neutral	7.0 pH
Slight acidic	6.1-6.8 pH
Medium acidic	5.6-6.0 pH
Strong acidic	5.1-5.5 pH
Very strong acidic	4.5-5.0 pH
Extreme acidic	below 4.5 pH

### 2.2.3.19 Welding Practice to Minimize Corrosion

According to Petersen *et al.*, 2013 numerous approaches are available to mitigate corrosion in weldments. The most vital ones are enumerated as follows:

- (I) Material and Weld Consumable Selection. Appropriate choice of materials and welding consumables can minimize the micro- and macro-compositional disparities across the weldment and hence, reduce the galvanic effect.
- (II) Surface Preparation. Right selection of cleansing process can minimize the defectives which usually serves as locality for corrosion attack in an aggressive environment. Although, the cleansing methods can give for worry. For instance, a surface cleansed mechanically (that is, cleaning by grinding or sand blasting) may left scums on the surfaces. Another important factor is the kind of wire brushes used. Brushes that lack the formation of corrosion products capable of holding moistures is generally chosen which is stainless steels types.
- (III) Welding configuration should enhance deposits which have commonly flat beads with lesser room and minimum slag entrapments. Poor configuration generates pores which trap still liquids, resulting to crevice and pitting corrosion. Erosion corrosion can emanate from uneven welding deposits pattern which enhances wild flow in tubular tanks.
- (IV) Welding Practice. To prevent under-bead gaps, complete penetration practice is often chosen. It is important that slags should be chip off sequel each pass using power grinder or power chipping tools. When a welding procedure uses flux, the orientation of the joints must allow thorough flux removal because most flux remains are aquaphobic and corrosive.

- (V) Weld Surface Finishing. Visual inspection should be carried out on weld deposit directly when each welding ends. Utmost corrosion resistance often requires a uniformly plain oxidized surface which is devoid of irregularities and unwanted materials. Weld deposits usually varies in coarseness and in amount of welding spatters, a need which grinding can be curtailed. Wire brushing is enough for smooth weld deposits. But in stainless steel, the presence of passivating film can alter, thereby intensifying attacks.
- (VI) Surface Coating. If the disparity in composition along the welded component can result to insitu attacks, it becomes necessary to use protecting coatings. These coatings need to conceal both the weldment and the base metal, and it usually needs distinct external preparations.
- (VII) Post-weld Heat Treatment. This is an efficient way of reducing corrosion vulnerability. This enhanced corrosion resistance is realized via a reduction in surface stress gradient which influences SCC growths. Post-weld heat treatment can aid in the transportation of hydrogen from the weldment and reduces the proneness to hydrogen crackling. This remedy can also decrease compositional quotients (that is, micro-segregation) and the corresponding micro-galvanic cell.
- (VIII) Pre-heat and Inter-Pass Temperature. Proper choice, pre-heat treatment application and inter-pass temperature control can hinder hydrogen cracks in low alloy and carbon steels.
- (IX) Passivation Treatment. This kind of treatment can improve the corrosion resistance of welded components made of stainless steels.
- (X) Avoidance of Crevices Formation. Slags that are still sticking to the weld deposits, and defects like incomplete penetration and micro-fissures can cause crevices which



can instigate a insitu concentration cell producing crevice corrosion. Proper choice of weld consumables and effective welding practice as well as thorough slag removal can mitigate this kind of corrosion damages.

- (XI) Elimination of Hydrogen Sources. With effective selection of weld consumables (that is; low hydrogen shielded metal arc welding electrode), efficient flux drying, and adequate cleaning of welded surfaces; the hydrogen-pickup can be lessened drastically.

### **2.3 Summary and Research gap**

Over the years, it is obvious that numerous researches had been conducted with a view to mitigate the corrosion of mild steel plate in UST. Some of the researches focused on effective material selection and others on the use of inhibitors. A few others focused on corrosion mitigation through welding design and practice. Despite, the success recorded so far, there has not been record of research that focused on best combination of arc welding current and electrode types that could yield optimum corrosion resistance in UST.

## **CHAPTER THREE**

### 3.0

## MATERIALS AND METHODS

### 3.1 Materials

Main material for the samples is 5 mm mild steel plate. Other items used which comprised of welding, metallography and corrosion materials are itemized in Table 3.1.

**Table 3.1: Experimental materials**

S/N	Materials (Make/Model)	Use	Location
1	Measuring tape and Vernier caliper	Samples' length, width and thickness measurement	Mechanical Workshop, FUT
2	Welding Machine	Joining of Workpiece	same
3	Hand cutting machine (Bosch)	Cutting of mild steel plate to workpiece	same
4	Filing disc	Grinding of welded work piece	same
5	Electrodes	Welding filler material	
6	Furnace (Muffle) model SX-5-12	Heat treatment	Material Lab., FUT.
7	Tongs	Removal heated specimen from furnace	same
8	Viella echant	Workpiece surface cleaning	same
9	Olymos microscope (GX-51)	Microstructural examination of selected samples	same
10	Emery papers	Sample surface polishing	Same
11	Distilled water	Washing of samples	same
12	Cylinder bath	Container for buried samples	same
13	Pipette	Measuring out acids	same
14	Plastic beakers	For containing the solutions	same
15	Conc. H <sub>2</sub> SO <sub>4</sub> analytical grade	5litres of 95% (Echant Preparation)	same
16	HCl Analytical grade	5litres of 95% (Echant Preparation)	same
17	Sodium chloride analytical grade	Soil acidification	same
18	Sodium sulphate analytical grade	Soil acidification	same
19	Sandy soil	House buried specimens	Gida Kwano

### 3.2 Methods

The experimental method comprised of sample preparation, welding of samples, post heat treatment of selected samples, and subjection to corrosion.

### **3.2.1 Experimental design and Grouping of samples:**

The factorial method for Design of Experiment was used to determine the number of samples. Based on consideration of varying two major welding parameters (current and electrode types) at three levels of currents while using three electrodes types, nine specimen or coupon is required to form a set. A set (nine) of specimen or coupon is needed in each case of heat treated and non-heat treated samples' microstructural analysis (that is eighteen coupons). Also in the case of Corrosion rate determination, a set of coupon is needed in each case of heat treated and non-heat treated samples (that is eighteen coupons). Likewise, eighteen coupons are needed for interval monitoring of corrosion rate of samples. Hence, a total of fifty-four coupons are needed for the experiment.

**Samples preparation:** This involved the purchase of mild steel plate. The plate density is  $7.85 \text{ g/cm}^3$  and was produced by Visco-Chemicals; Houston.

Characterization of work piece to ascertain the percentage composition of various elemental constituent was taken from the manufacturers' specification as depicted in Table 3.2

**Table 3.2: Chemical compositions of the mild steel sample**

C	Mn	Si	P	S	Ni	Cr	Cu	Ti	Al	Mo	Fe
0.22	1.70	0.55	0.04	0.03	0.5	0.25	0.4	0.04	0.1	0.35	95.82

(Source: Visco Chemicals, 2018)

A sheet of 5 mm mild steel plate was cut into one hundred and eight (108) test pieces each of about 50 mm x 80 mm dimension. Cutting of samples was done with Bosch hand cutting machine. To achieved this, 60 mm diameter cutting disc was fixed to the hand cutting machine and was then connected to electricity to power on. Typical cut samples are shown in Plate I.

### 3.2.2 Manual Metal Arc Welding

The workpiece ‘X’ were welded using Manual Metal Arc Welding (MMAW) at constant current 80 A (C<sub>80</sub>) with different welding Electrodes. Thirty-six workpieces welded to yield eighteen coupons (six from each case):

**Table 3.3: At arc welding of 80**

Sample Grouping	Electrode types	Electrode and welding current
X1	E1(6010)	E1C <sub>80</sub>
X2	E2(6013)	E2C <sub>80</sub>
X3	E3(7018)	E3C <sub>80</sub>

The procedure used in carrying out the MMAW in each of the cases are similar. It involves placing the steel plate flat on the bench and brushing to free it of dirt and scales. Then, attaching the ground bead securely to the plate. The welding machine (Origo arc 400 model 3161233106) amperage was set to 80 A for this particular case. The various electrode types (E1, E2, E3) were fitted into the holder, each at a time. The welding machine was put on, followed by dropping the hood over the face and striking the arc by brushing base metal with the electrode via the holder. The distance between an electrode and the base metal was approximately equal to electrode diameter. Upon striking of electrode to the base metal, a quick twist was made to free the electrode. But if not, free the electrode through the electrode holder. Then, subsequent retry to stabilize the arc. When the arc was stabilized, and several beads were laid on the sample. Chipping of the weld beads thoroughly was done with wire brush. More beads on each duo samples were laid to complete welding process, after which the welding machine was turn-off.

By still using the listed electrodes, and carrying out MMAW as earlier described while varying the current to 90A. Further eighteen (18nos) coupons were gotten from thirty-six work pieces as depicted in Table 3.4. Six samples from each case of Y1, Y2 and Y3.

**Table 3.4: At arc welding of 90 A**

Sample Grouping	Electrode types	Designation of Electrode and welding current
Y1	E1(6010)	E1C <sub>90</sub>
Y2	E2(6013)	E2C <sub>90</sub>
Y3	E3(7018)	E3C <sub>90</sub>

Similarly, by using a current of 100A, eighteen (18) coupons were also gotten. Six coupons were obtained in each cases Z1, Z2 and Z3 respectively as shown in Table 3.5

**Table 3.5: At arc welding of 100 A**

Sample Grouping	Electrode types	Electrode and welding current
Z1	E1(6010)	E1C <sub>100</sub>
Z2	E2(6013)	E2C <sub>100</sub>
Z3	E3(7018)	E3C <sub>100</sub>

The samples, welding machine, various electrode types are depicted in plate I - IV



### 3.2.3 Post heat treatment of the selected samples

Three samples from each of X3, X2, X1, Y3, Y2, Y1, Z3, Z2, and Z1 were taken for post weld heat treatment. The procedures followed is briefly discussed. First, was charging the samples (six piece at a time) into the heat treatment furnace (model: SX-5-12 Muffle furnace, US make) through the furnace door as shown in Plate V. Total of twenty-seven (27) coupons were heat treatment in all. After charging in case, the furnace was switched-on and the temperature set to 900°C. Heating was done to the set temperature and holding time was one hour to allow for phase transformation. Thereafter, in each case, the furnace would be switched –off and coupons brought out from the furnace with the aid of Tong as shown in Plate VIII.





Plate VII: Heated samples in the furnace

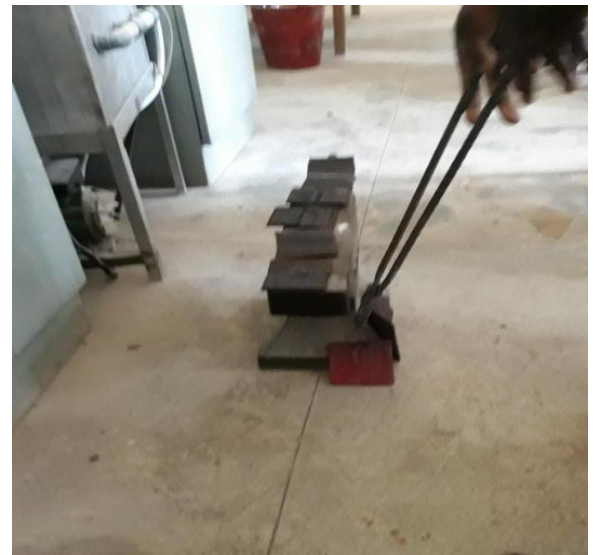


Plate VIII: Tong and the sample

### **3.2.4 Microstructural Examination of samples**

Carrying metallography and microstructural studies of all samples

#### **3.2.4.1 Metallographic Sample Preparation**



The samples (9 heat-treated and 9 non-heat treated) were sectioned and mounted on Electro-lite™, (conductive mounting compound) using the LECO PR-32 mounting press. The mounted samples were prepared and grinded (Plate VIII) using water proof silicon carbide paper of grit steps 80, 120, 150, 220, 400, 600, 800 and 1200 according to ASTM simple techniques of surface finishing.



The polishing machine (mode Plate IX: Grinding stage) to polish the samples using 6  $\mu\text{m}$ , 3  $\mu\text{m}$  and 1  $\mu\text{m}$  polish clothes, followed by a final polishing step of colloidal silica. Viella's Reagent was then used to etche the samples. Light Optical micrographs (LOM) were taken of each samples microstructure from 5x through 100x. Several etchants were attempted before finding a few that had the desired effect of showing grain boundaries and carbides in the microstructure. One etchant that was found to be successful was Viella's Reagent. The other was a solution that consisted of 1-part nitric acid, 2 parts sulfuric acid, and three parts distilled water. After polishing and etching, light optical microscopy was performed on the samples using an Olympus GX-51 scope. Images were captured at 5x to 100x magnification.





### 3.2.4.2 Scanning Electron Microscopy

A Quanta 200 microscope was used for higher magnification images of the samples. Accelerated voltage of 25 kV and a working distance of 12 mm was used to capture the images.



Plate XI: SEM and set up

### 3.2.5 Subjection of samples to active corrosive medium and observation.

The mild steel coupons (18 piece: 9 heat treated and 9 non-heat treated) were submerged in an acidic sandy soil environment for a maximum duration of 60 days. On the day 30, a replicate pairs of the submerged coupons were recovered for analysing and the final set of coupons was recovered at the last day of exposure (day 60). Observation of the buried coupons at intervals were determination of corrosion effect extent, taking place on the coupons at the end of each monitoring duration and for comparison the effect of time, on the rate of corrosion for the heat treated and non-heat-treated coupon types in an acidic sandy soil environment under study. The samples were taken to the laboratory for cleansing and analysis for weight loss and corrosion rates determination upon retrieving from the soil bath (Plate XII and XIII). Due to acidified sandy soil bath environment, the recovered

coupons did not show oil residue on them rather, they were covered with soil scales upon retrieval.

The external soil scales on the coupons was cautiously removed at the laboratory. Then, the coupons were cleansed using inhibited-acid (15% HCl) to eliminate corrosion remains on coupons' surfaces in accordance with ASTM-G1 (Standard Practice for Preparing, Cleaning, and Evaluating Corrosion-Test-Specimen). These coupons were subsequently rinsed with flowing water and preceded by placing them in an oven at temperature of 70°C for a period of 15 minutes as to dry them. Drying was followed by placing them in desiccator to cool thereafter, they were weighed to a constant mass via an electronic scale (DT 3000A, UK). Metal loss during the exposure period was ascertained by comparing the weight of retrieved coupons before cleansing to the initial measurement, the disparity indicates the loss. Equivalent average percentage weight loss (APWL) and corrosion rate (CR) were found.

According to Gaudet (2010), Corrosion rate was calculated with the assumption that uniform-corrosion occurred over the whole surfaces of the coupons. The rate of corrosion in mils per year (mpy) was calculated from the weight loss equation (3.1)

$$CR = \frac{W}{(D \times A \times t)} \times k \quad (3.1)$$

where: k = constant (87.6)

W = weight loss in grams

A = coupon area (cm<sup>2</sup>)

D = metal density in g/cm<sup>3</sup>

t = time (Hours)



Plate XII: Soil bath for non-heat treated



Plate XIII: Soil bath for heat treated

## CHAPTER FOUR

### 4.0 RESULTS AND DISCUSSION

#### 4.1 Microscopic Examination Results

The microscopic examination of the non-heat treated and heat treated samples were examined. The result obtained from microstructural examination of the samples is outlined in Plates XIV - XVII. Each plate has lists of images which comprised of the welded zone (W), the HAZ (H) and the parent material (P) or non-fusion zone.

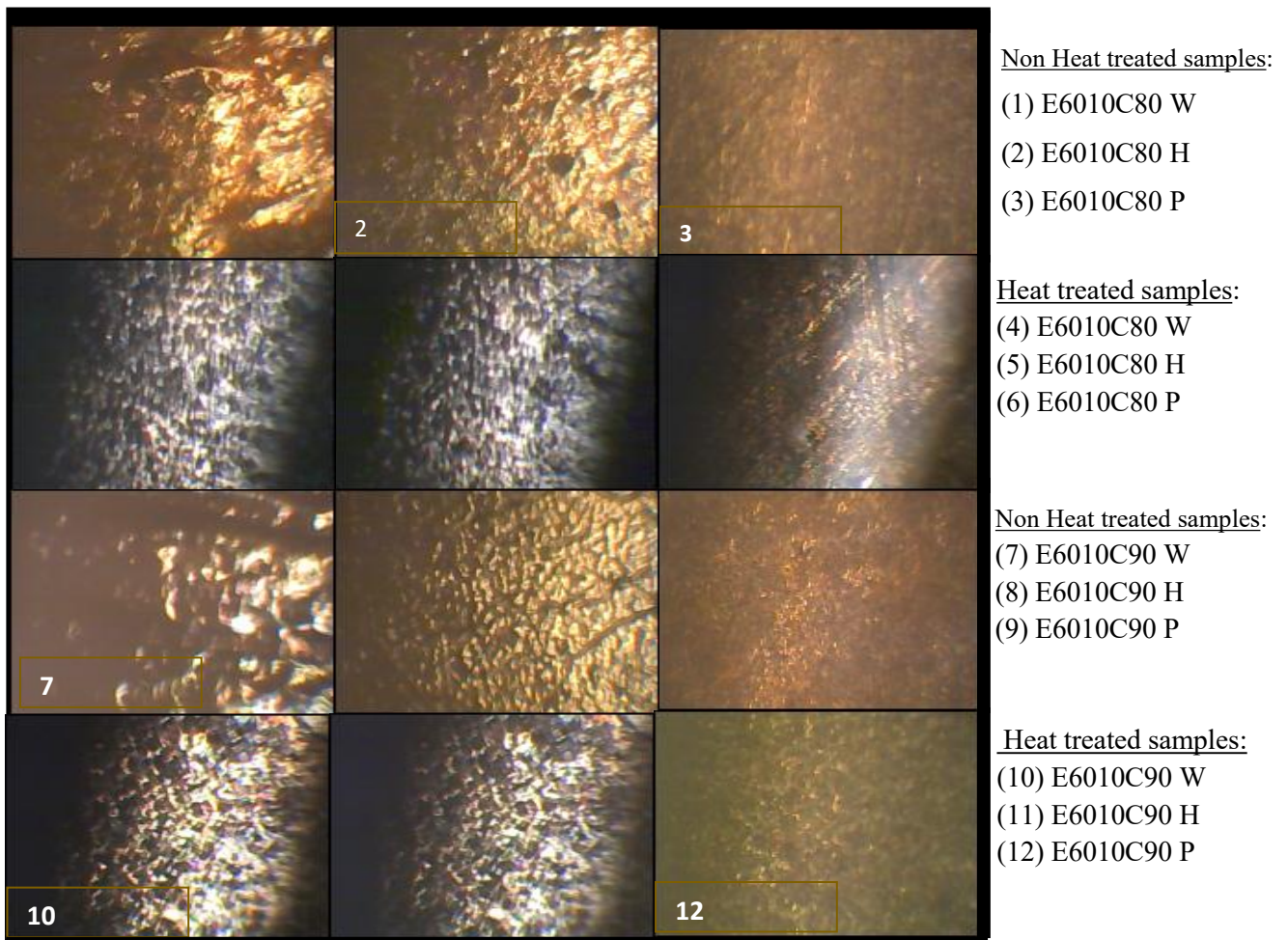
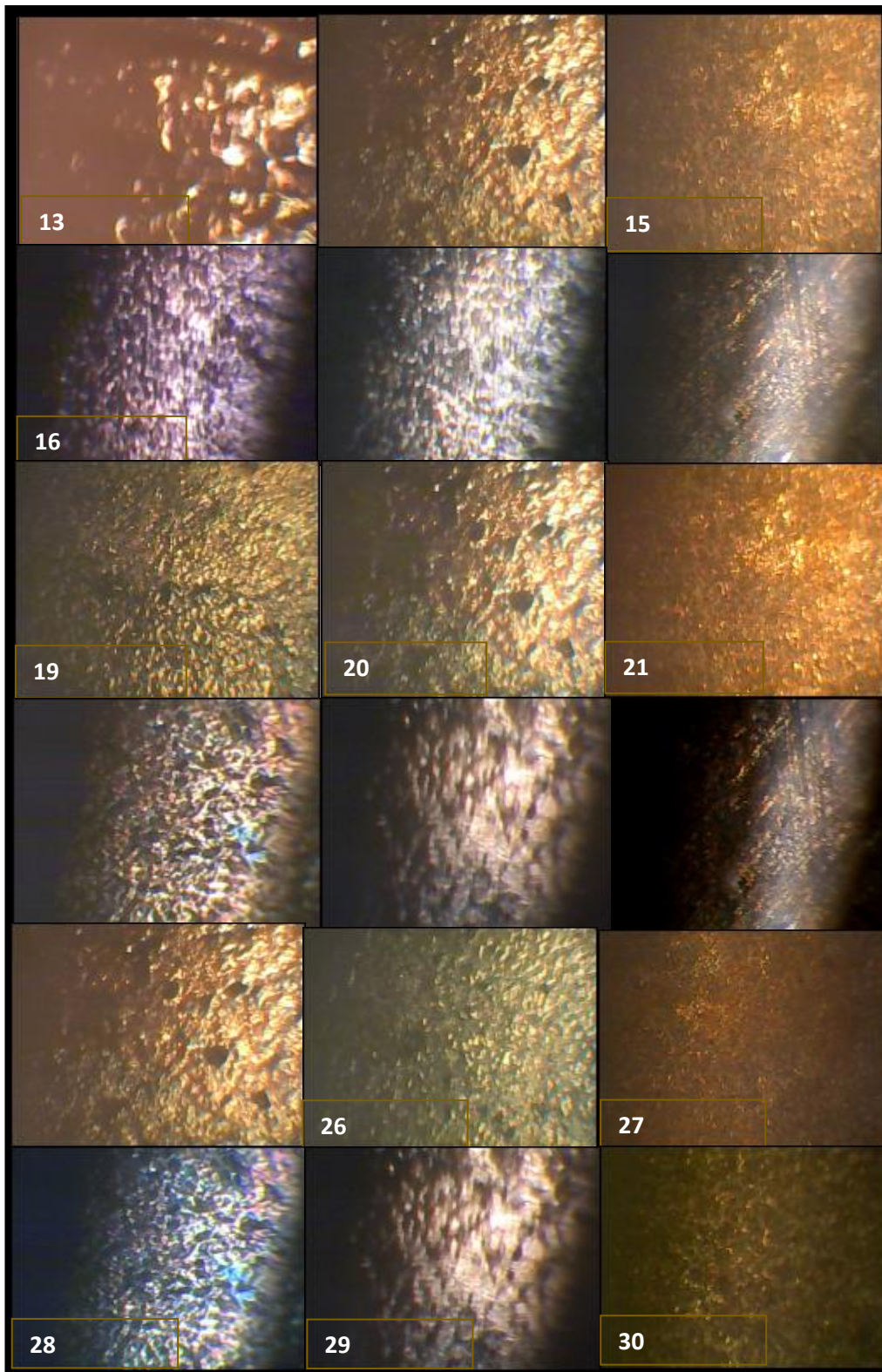


Plate XIV: Microscopic results of E6010 weld samples.



Non Heat treated samples:

- (13) E6010C100 W
- (14) E6010C100 H
- (15) E6010C100 P

Heat treated samples:

- (16) E6010C100 W
- (17) E6010C100 H
- (18) E6010C100 P

Non Heat treated samples:

- (19) E6013C80 W
- (20) E6013C80 H
- (21) E6013C80 P

Heat treated samples:

- (22) E6013C80 W
- (23) E6013C80 H
- (24) E6013C80 P

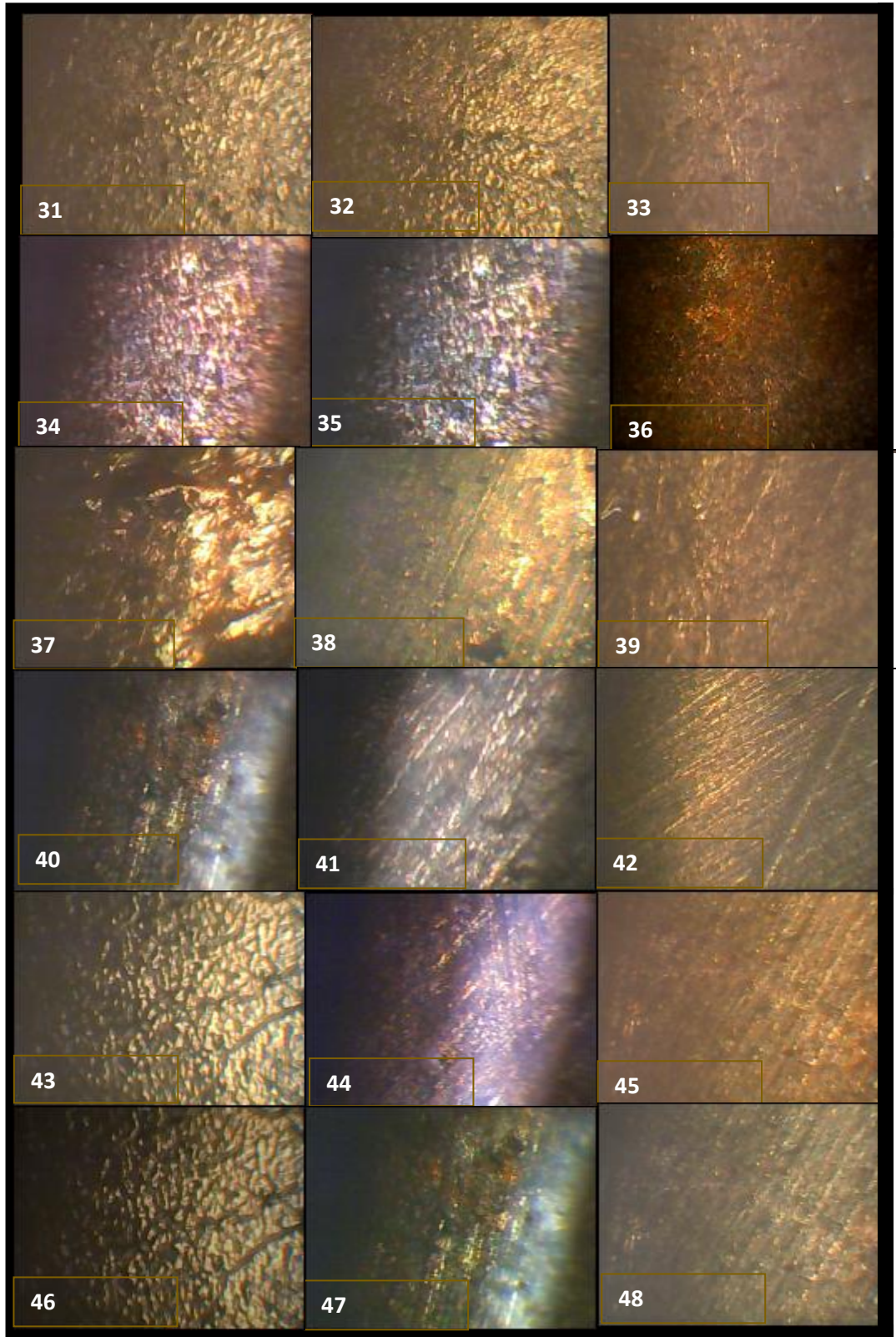
Non Heat treated samples:

- (25) E6013C90 W
- (26) E6013C90 H
- (27) E6013C90 P

Heat treated samples:

- (28) E6013C90
- (29) E6013C90 H
- (30) E6013C90 P

Plate XV: Microscopic results of E6010 and E6013 weld samples.



Non Heat treated samples:  
 (31) E6013C100 W  
 (32) E6013C90 H  
 (33) E6013C90 P

Heat treated samples:  
 (34) E6013C100W  
 (35) E6013C100 H  
 (36) E6013C100 P

Non Heat treated samples:  
 (37) E7018C80 W  
 (38) E7018C80 H  
 (39) E7018C80 P

Heat treated samples:  
 (40) E7018C90 W  
 (41) E7018C90 H  
 (42) E7018C90 P

Non Heat treated samples:  
 (43) E7013C90 W  
 (44) E7013C90 H  
 (45) E7013C90 P

Heat treated samples:  
 (46) E7013C90 W  
 (47) E7018C90 H  
 (48) E7018C90 P

Plate XVI: Microscopic results of E6013 and E7018 weld samples.



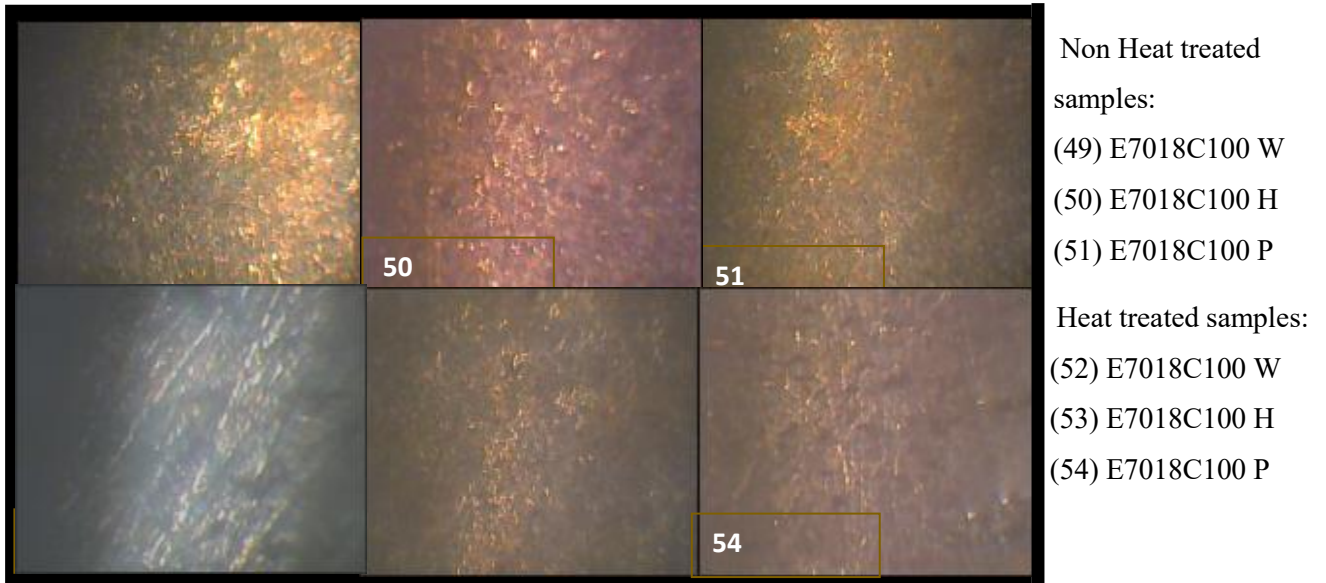


Plate XVII: Microscopic results of E7018 weld samples.

#### 4.2 Discussion of Microscopic Examination Results

In each of the samples, it was observed that coarseness of grains decreases from the welded portion through the HAZ to the parent material. This is in line with reviewed literatures. The samples being cut to contain the welded, HAZ and parent material areas undergone polishing and etching with 2% nital solution to detect the grain size as well as their microstructures. As contained in literatures, the microscopic examination observations also revealed phases of the tested mild steel samples to be ferrites (Fe), light yellow in colour with black cementites ( $\text{Fe}_3\text{C}$ ), as shown in Plates XIV – XVII (with various images captions 1 – 54). Further interesting, the microscopic examination images showed that the grains/boundaries in the middle portions of samples' thickness, are larger near the welded area than at the base materials. This is because cooling is less rapid at the centre than in the surface. The differences in grain size are determined by these factors. Comparison of heat treated and the non-heated samples revealed that the microstructures of the

heated treated samples were more refined. Comparison of the heated and non-heated sample still revealed the darker phase composition of heated samples. This due to transformation of phase from austenite to combined ferrite and cementite. The presence of other alloying elements like Silicon, Manganese, Nickel etc (as shown in Table 3.1) accounts for the dark appearance of the heat treated sample.

### **4.3 Corrosion Result**

Based on discussion in the literatures reviewed, lower pH in soil has been observed as a key corrosion accelerating factor for submerged metallic components. For example,  $\text{pH} < 4$  has been reportedly to extremely corrode for submerged pipes/tanks (Schwerdtfeger, 1953; Romanoff, 1957; Murray and Moran, 1989; Cast et al., 2013; Asadi and Melchers, 2017). These studies also pointed out that clay soil (fine-grained) is the best corrosive medium than other type of soils readily available (like granular). However, the reason for usage acidified sandy soil in this study is due to the recommendations by EPA as the best choice for final back filling of UST. The coupon was examined on day 30 and 60 respectively. The weight of coupons was recorded. The result obtained in the case of non-heat treated and heat treated samples is tabulated thus in Tables 4.1 and 4.2 respectively.

Table 4.1: Corrosion of non-heat treated coupons in acidified aqueous sandy soil

ELECTRODE	E6010							
CURRENT↓	length of sample	width of sample	Initial Wt(g)	DAY 30(g)	Final Wt(g) day 60	Weight Loss(g)	Surface area (cm <sup>2</sup> )	corrosion rate mm/yr
C80	50	80	233.10	233.00	232.10	1.00	4000.00	1.955E-06
C90	50	74	224.00	223.50	222.70	1.30	3700.00	2.747E-06
C100	50	72	197.80	197.15	196.20	1.60	3600.00	3.475E-06
ELECTRODE	E6013							
CURRENT↓	length of sample (cm)	width of sample (cm)	Initial Wt(g)	DAY 30(g)	Final Wt(g) DAY 60	Weight Loss(g)	Surface area (cm <sup>2</sup> )	corrosion rate mm/yr
C80	50	81	235.40	235.02	234.43	0.97	4050.00	1.873E-06
C90	50	79	230.70	230.04	229.67	1.03	3950.00	2.039E-06
C100	50	78	218.60	218.03	217.69	0.91	3900.00	1.824E-06
ELECTRODE→	E7018							
CURRENT↓	length of sample (cm)	width of sample (cm)	Initial Wt(g)	DAY 30(g)	Final Wt(g)	Weight Loss(g)	Surface area (cm <sup>2</sup> )	corrosion rate mm/yr
C80	50	79	230.60	230.01	229.65	0.95	3950.00	1.881E-06
C90	50	75	222.50	222.01	221.60	0.90	3750.00	1.877E-06
C100	50	72	197.80	222.12	196.89	0.91	3600.00	1.977E-06

Table 4.2: Corrosion of heat treated coupons in acidified aqueous sandy soil

ELECTRODE	E6010							
CURRENT↓	length of sample (cm)	width of sample (cm)	Initial Wt(g)	DAY 30(g)	Final Wt(g)	Weight Loss(g)	Surface area (cm <sup>2</sup> )	corrosion rate mm/yr
C80	50	82	238.7	238.22	237.71	0.99	4100.00	1.888E-06
C90	50	83	239.4	238.98	238.46	0.94	4150.00	1.771E-06
C100	50	79	229.4	228.91	228.51	0.89	3950.00	1.7618E-06
ELECTRODE	E6013							
CURRENT↓	length of sample (cm)	width of sample (cm)	Initial Wt(g)	DAY 30(g)	Final Wt(g)	Weight Loss(g)	Surface area (cm <sup>2</sup> )	corrosion rate mm/yr
C80	50	76	225	224.67	224.08	0.92	3800.00	1.8931E-06
C90	50	83	239.4	238.96	238.53	0.87	4150.00	1.6392E-06
C100	50	83	239.6	239.03	238.7	0.90	4150.00	1.6957E-06
ELECTRODE→	E7018							
CURRENT↓	length of sample (cm)	width of sample (cm)	Initial Wt(g)	DAY 30(g)	Final Wt(g)	Weight Loss(g)	Surface area (cm <sup>2</sup> )	corrosion rate mm/yr
C80	50	77	227.2	226.87	226.43	0.77	3850.00	1.5638E-06
C90	50	78	219	218.71	218.3	0.70	3900.00	1.4034E-06
C100	50	74	210.6	210.23	210	0.60	3700.00	1.268E-06

#### 4.4 Discussion of Corrosion Results

Physical weight-loss measurements were carried out for samples retrieved at the specified periods from the test-batches so as to ascertain the real corrosion damages and severities. The samples were cleansed of the soil debris on them. The preparation of this solution and the procedure of cleaning the samples had been reviewed. The overall appearance of the samples after removal from the test containers are shown in Plate XVIII.



Plate XVIII: Typical corroded Samples

Weight loss measurements of different samples were performed at the specified durations, and the equivalent corrosion rates were calculated, as earlier described in Section 3.2.5, Chapter 3. The weight loss of these samples of different welding electrodes were found to be moderately similar, indicating the uniformity of the tailored soil for the experimentation, along with the constancy and precision of the undertaken measurements for the test-batches. The final weight losses were used in

determination of the corrosion rates for each electrode type and were reported for analysis as depicted in Tables 4.1 and 4.2 respectively.

From Table 4.1 (the case of no-heat treated samples), upon the use of E6010 Electrode, there was a progressive increase in loss of weight as the current increases from 80 A, 90 A and 100 A. The increase weight loss from 1 g, 1.3 g and 1.6 g might be resulting from increase in heat dissipation as the current increased. This results to increase in grain size and hence, higher corrosion rate. In the case of usage of Electrode E6013, as the current was increased from 80A, 90 A to 100 A, the weight loss increased from 0.97 g to 1.03 g and reduced to 0.91 g. From the literature, it was expected that the weight loss reduced correspondingly as the current increases. The reason for the variation was simply due to differences in samples width sizes which transcended to the surface area. Upon the usage of Electrode E7018, there was similar weight loss result trend like that of Electrode E6013. Comparing the weight loss and corrosion rate of the various electrodes indicates that electrode E7018 produces lesser corrosion rate and weight loss of 0.95 g, 0.90 g and 0.91 g at current 80 A, 90 A and 100 A respectively. Then followed closely by Electrode E6013 which produced weight loss of 0.97 g, 1.09 g and 0.91 g at current 80 A, 90 A, and 100 A respectively. Electrode E6010 yielded the highest weight loss and corrosion rate. The weight loss was 1 g, 1.3 g and 1.6 g at current 80 A, 90 A, and 100 A respectively.

Form Table 4.2 (the case of heat treated samples), upon the usage of Electrode E6010, there was progressive decrease in weight loss from 0.99 g, 0.94 g and 0.89 g as the current increases from 80 A, 90 A and 100 A respectively. In the case of Electrode E6013, at current 80 A, 90 A and 100 A, the corresponding weight loss was 0.92 g, 0.87 g and 0.9 g respectively. The lowest weight loss was at 90 A instead of 100 A as expected. The reason

due to lower surface area. Upon usage of Electrode E7018, the respective weight losses were 0.77 g, 0.70 g and 0.60 g at current 80 A, 90 A and 100 A. The weight loss and corrosion rate were seen to decrease as the current increased.

Upon comparison of the non-heat treated and heat treated sample, it was observed that at various electrode types, the heat treated samples gave a lesser weight loss and corrosion rate. The reasons are not farfetched. Results have shown how large grains are more vulnerable to environmental and corrosion attacks (Popova et al. 2004). Larger grains become weaker and start corroding when exposed to a corrosive environment, acting as an anode. More so, the grains at the edge acts as a cathode which leads to anode-cathode galvanic corrosion. In the end, grain in the middle section corrode rapidly than the edge grains. In the heat treated sample, the grains were refined by the normalizing process and hence lesser corrosion than non-heat treated counterparts.

From the results, the flowchart beneath in Figure 4.2 shows the appropriate steps for production procedure in the development of UST.

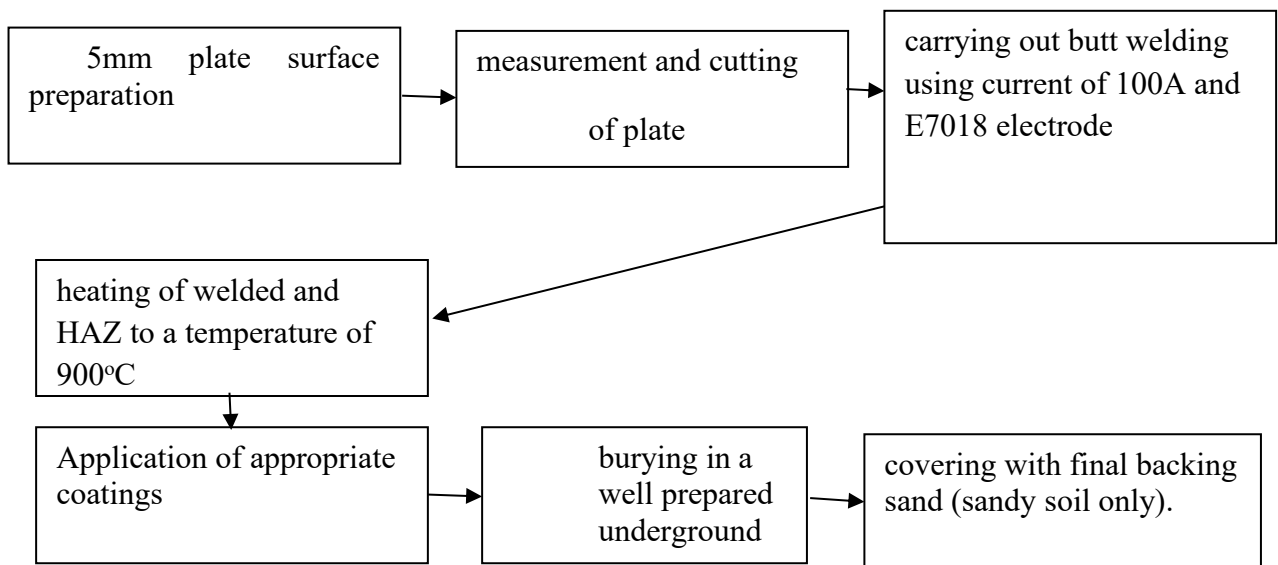


Figure 4.1 Flowchart of production procedure for development of UST.

## **CHAPTER FIVE**

### **5.0 CONCLUSIONS AND RECOMMENDATIONS**

#### **5.1 CONCLUSIONS**

Corrosion in an underground tank which are usually made of mild steel is inevitable. Therefore, deliberate effort must be made to mitigate this menace. This can be achieved through proper development of production frame work. The method of joining plates in UST construction is via mainly arc welding. The welding parameter such as current and electrode types variation, has significant effect on the microstructural properties of material which also inadvertently affects corrosion rate. A study of variation of arc welding current and electrodes in mild steel for UST and the resultant corrosion resistance were presented. Electrode E7018 and a current of 100 A were the best among the chosen parameters.

Post heat treatment improves grain refinement and mitigates corrosion in UST. For the study, all the heat treated samples irrespective of electrode types used gave a better corrosion resistance than the non-heat treated counterparts.

Thus, for 5 mm mild steel plate, the production framework or procedure for fabricating underground storage tank was successfully developed. Adherence to the developed procedure yielded the maximum corrosion resistance.

#### **5.2 RECOMMENDATIONS**

Project study is a continual course; modifications, gaps, limitations, further improvement and enhancement are always linked with effective studies in any given field of engineering or other non-engineering fields in our modern society. Every project studies needs to future enrichment. In like manner, though this study report is comprehensive as corrosion



mitigation techniques development, it has some constraints and limitations to resources and time. The following few suggestions and recommendations are proposed for further studies:

1. In this study, the sandy soil which is often used as final backing-sand was acidified to enhance corrosion, it would be interesting to use it in normal state while studying corrosion in laboratory-monitored environment. Such studies will produce better and more pragmatic corrosion mitigation framework that can prove extremely beneficial to industries dealing with UST managements.
2. In this study, the depth of coupons immersion was inconsiderate for ease in the laboratory. In field applications, tanks are submerged at depths of specified values. Hence, further studies should be carried out while varying depths of submerging to determine differential aeration effects and their corresponding corrosion rate prior developing the framework.

### **5.3 Contribution of study to knowledge**

The most effective combination of electrode and current combination of E7018 and 100A respectively was developed for welding of 5 mm mild steel plate in underground tank construction.

Development of procedural measures for corrosion mitigation in underground storage tank through proper selection of arc welding parameters and normalizing heat treatment.

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