CORROSION AND FATIGUE BEHAVIOUR OF WELDED AUSTENITIC STAINLESS STEEL IN DIFFERENT ENVIRONMENTS

BY

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DECLARATION

I hereby declare the work in this thesis titled **"CORROSION AND FATIGUE BEHAVIOURS OF WELDED AUSTENITIC STAINLESS STEEL IN DIFFERENT ENVIRONMENTS"** was performed by me in the Department of Mechanical Engineering under the supervision of Dr. D.S. Yawas and Prof. S.Y Aku.

The information derived from the literature has been duly acknowledged in the text and a list of references provided. No part of this work has been presented for another degree or diploma in any institution.

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CERTIFICATION

This thesis titled **"CORROSION AND FATIGUE BEHAVIOURS OF WELDED AUSTENITIC STAINLESS STEEL IN DIFFERENT ENVIRONMENTS"** meets the regulations governing the award of degree of Master of Science of the Ahmadu Bello University and was approved for its contribution to knowledge and literary presentation.

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DEDICATION

This thesis is dedicated to Almighty God, in whom I found grace to complete this work.

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My profound gratitude goes first to Almighty God for His grace, mercy, guidance, protection and inspiration towards the success of my program.

I am sincerely grateful for the effort of my supervisors; Dr. D.S. Yawas and Prof. S.Y. Aku for their academic and moral contributions towards the success of this work.

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ABSTRACT

This research work was carried out to study the effects of seawater, 0.5M hydrochloric acid and wet steam corrosive media on the fatigue strength and microstructure of welded austenitic stainless steel. The immersion time in the corrosive media was 30 days to simulate the effect on stainless steel structures/equipment in offshore and food processing applications. Annealing heat treatment was carried out on the samples. The weight loss method was used for the corrosion tests. From the results of the corrosion test, seawater sample has highest weight loss of 4.0mg for the heat treated samples and 4.5mg for the unheat-treated samples. For the different media used, seawater sample has the highest corrosion rate of $0.64x10^{-6}$ mm/yr for the heat treated samples and $0.72x10^{-6}$ mm/yr for the unheat-treated samples. The findings from the fatigue tests show that seawater corroded specimen have lower fatigue stress of $0.5x10^{-5}$ N/mm^2 for the heat treated sample and $0.1x10^{-5}$ N/mm^2 for the unheat-treated sample compared to the corresponding hydrochloric acid and steam samples. The post welding heat treatment was found to increase the mechanical properties of the austenitic stainless steel especially tensile strength but it reduces the transformation and thermal stresses of the samples. These findings were further corroborated by the microstructural examination of the stainless steel specimen.

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NOMENCLATURE

A – Area of immersions (cm^2)

AISI – American Iron and Steel Institute

ASTM – American Society for Testing and Materials

C – Concentration

CO_2 – Carbon dioxide

Cr – Chromium

Cr_2O_3 – Chromium Oxide

CR – Corrosion Rate

CS – Carbon Steel

$CuSo_4$ – Copper Sulphate

$FeCl_2$ – Iron Chloride

HAZ – Heat Affected Zone

HCl – Hydrochloric acid

K – Constant

L – Length

M – Mole

Mn – Manganese

$M_{23}Cr_6$ – Carbon complexes

NaOH – Sodium hydroxide

N_f – fatigue failure

N_i – Number of cycles require to initiate a crack

Ni – Nickel

N_p – Number of cycles require to propagate the crack

r – radius

SCC – Stress Corrosion Cracking

SiC – Silicon Carbide

SS – Stainless Steel

t – Period of immersion (hrs)

V – Volume

W – Weight loss (mg)

ρ – Density of material(g/cm^3)

σ_m– mean stress

σ_{max} – maximunm stress

σ_{min}– minimum stress

CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND TO THE STUDY

Fatigue failure is of great importance as it is well attested to by the large percentage of failures in machine elements. Metal fatigue is caused by repeated cycles of load. It is a progressive localized damage due to fluctuating stresses and strains on the material. From estimates made, over 80% of failures in machines are as a result of fatigue failure**,** (www.materialsengineer.com/CA-fatigue.htm).

In rotating equipment, a great deal of 300 series stainless steels are used and as a result we often experience several types of corrosion. Stainless steel generally exhibits excellent corrosion resistance, but pitting can cause catastrophic failuresin structures made of the alloy (Peter and Vito, 1985).

Corrosion is chemically induced damage to a material that results in deterioration of the material and its properties. This may result in failure of the component. Several factors should be considered during a failure analysis to determine the effect corrosion played in the failure (www.materialsengineer.com/CA-corrosion.htm).

Sudhakar (2005) studied an orthopaedic implant (nail for shinbone) made of 315L stainless steel that failed prematurely to determine the root cause for the fracture. Detailed scanning electron microscopy was carried out to conclusively establish the evidence. Based on the results of extensive fracture analysis as well as the background information provided on the implant, it was found that the stainless steel nail failed by the mechanism of ductile fracture facilitated by the

presence of non-metallic inclusions.

1.2 STATEMENT OF RESEARCH PROBLEM

Corrosion remains one of the most severe limitations to the use of various steels in chemical and petrochemical industries. Millions of dollars are lost each year because of corrosion. Much of this loss is due to the corrosion of iron and steel, although many other metals may corrode as well. The problem with iron as well as many other metals is that the oxide formed by oxidation does not firmly adhere to the surface of the metal and flakes off easily causing pitting. Extensive pitting eventually causes structural weakness and disintegration of metal (Iliyasu, 2012).

Though austenitic stainless steel is said to be resistant to corrosion, this has however not been established in all environments and conditions. Despite the introduction of polymers and composites in recent years, metals remain important in structures because of their strength, stiffness, toughness and tolerance of high temperature. The noble metals, such as gold and platinum are an exception to this, but they are too rare for common use. Although one of the main reasons why stainless steels are used is corrosion resistance, they do in fact suffer from certain types of corrosion in some environments and care must be taken to select a grade which will be suitable for the application. Corrosion can cause a variety of problems, depending on the application and the environment (Iliyasu, 2012).

1.3 PRESENT RESEARCH WORK

The study considers the effects of seawater, 0.5M hydrochloric acid and wet steam corrosive media on the fatigue strength and microstructure of welded

austenitic stainless steel. The immersion time in the corrosive media was 30 days to stimulate the effect on structures/equipment in offshore and food processing applications. Annealing heat treatment was carried out on the samples and the weight loss method was used for the corrosion tests. Seawater sample has the highest weight loss and corrosion rate for both the heat-treated and unheat-treated samples. The findings from the fatigue tests show that seawater corroded sample have lower fatigue strength. These findings were further corroborated by the microstructural examination of the stainless steel samples.

1.4 AIM AND OBJECTIVES OF THE PROJECT

The aim of the study is to investigate the corrosion fatigue strength of welded austenitic stainless steel in different corrosive media (seawater, hydrochloric acid and steam) under heat treatment.

The specific objectives are:

(1) To establish the chemical composition of austenitic stainless steel.

(2) To study the corrosion behavior of welded austenitic stainless steels in different corrosive media using the weight loss method.

(3) To investigate the fatigue strength of the welded stainless steel in the different corrosive media.

(4) To examine the microstructures of the as-received, the heat treated and corroded stainless steels.

(5) To examine the effect of annealing heat treatment on the welded stainless steel.

1.5 SIGNIFICANCE OF THE STUDY

Stainless steels are widely used by different industries such as oil and gas (onshore and offshore), pulp and paper, marine, automobile, food processing industries and for construction of storage tanks or processing vessels.

Therefore, there should be means of influencing and controlling the properties of these stainless steel materials while in use under corrosive media with a view of improving their performance.

The significance of this study is therefore to help in solving the corrosion fatigue problems usually faced when using stainless steel in different corrosive media and hence recommend conditions under which stainless steels may be used.

1.6SCOPE OF THE STUDY

This research work will cover the following areas:

(i) The use of lathe machine to machine the stainless steel to ASTM standard code D3166-99 for fatigue tests.

(ii) The use of metal inert gas welding.

(iii) Annealing heat treatment of the welded austenitic stainless steel.

(iv) The use of weight loss technique in determining the corrosion rate.

(iv) The use of grinding, polishing and etching for microstructure examination of the samples.

CHAPTER TWO

LITERATURE REVIEW

2.1FATIGUE

Fatigue failures occur in engineering materials whereby a material (metal) fails when subjected to repetitive dynamic or fluctuating stresses/loads much lower than thoserequired to cause fracture on single applications. Loads which can cause fatigue failure apart from axial can be those in shear or bending (Peter and Vito, 1985).

Corrosion is the reaction of metallic material with its environment which gradually leads to deterioration or destruction of the material. Corrosion is one of the factors that greatly influence fatigue (Fontana, 1998).

Stainless steels are widely used by different industries such as oil and gas (onshore and offshore), pulp and paper, marine, automobile, food processing industries and for construction of storage tanks(www.materialsengineer.com/DA-Food-Processing.htm).

2.2 NATURE OF FATIGUE FAILURE

Fatigue failure occurs through formation of a crack at some local spot or nucleus on the surface of the part after a certain number of load reversals and this is propagated gradually across the part. Hence, complete failure occurs when the remaining section becomes so small that it can no longer carry the applied load.

Fatigue fracture occurs through crack initiation, crack propagation and fast fracture.

A fatigue failure that was observed in an ordinary tensile test of a ductile

material occurred usually even in brittle metals. This shows that in establishing fatigue life of a particular part, the loading environment of the part and specific details of design are of great importance.Hence, fatigue problems provide a better opportunity to study the effects of corrosion as related to fatigue failure which brought about the term "corrosion fatigue" (Peter and Vito, 1985; Callister, 2007).

2.3 FATIGUE LIFE

The fatigue life of a material is the total number of stress cycles that will cause fatigue failure (N_f) at some specified stress amplitude. This involves three stages, which consist of crack initiation (N_i), crack propagation (N_p) and fast fracture.

In term of the number of cycles, fatigue life is stated as (Peter and Vito, 1985):

$$N_f = N_i + N_p' \quad \text{.....1}$$

where; N_i = number of cycles required to initiate a crack.

N_p = number of cycles required to propagate the crack.

The fast fracture stage is not included in N_f, because it is the terminal phase in fatigue failure of material and contributes very little to the total life since it occurs very quickly.

The stress level contributes mainly to the relative number of cycles involved in the initiation and propagation stages. This is because at low stress levels many cycles are required to develop and initiate a crack and as the stress level increases, the crack initiation stage (N_i) decreases. So, most of the fatigue life is utilized in crack initiation at low stress levels.

Also, cracks form early in the fatigue life, at relatively high stress levels.

Fatigue failure is divided into two categories;

Low-cycle fatigue: This is associated with high stress cycles usually less than 10^5 cycles.

High-cycle fatigue: This is associated with low stress cycles usually above 10^5 cycles, (Peter and Vito, 1985).

2.4 FACTORS AFFECTING FATIGUE LIFE

The major factors that affect the fatigue life of a material are:

mechanical, environmental and micro structural factors.

2.4.1 Mechanical Factors

This group of factors deals with the geometrical and mechanical considerations of fatigue life. The major mechanical factors are: *surface effect, stress concentration and mean stress.*

i. **Surface effect:** In most fatigue tests and service applications, the maximum stress tends to occur at the surface of a material. Therefore in addition to stress concentration, fatigue life is especially sensitive to surface conditions. Such conditions could be surface finish (roughness) surface properties and residual stress.

ii. **Stress concentration:** The area effect of stress concentration is to reduce fatigue life. Stress raisers consist of keyways, fillets, grooves, holes, or threaded regions. Any notch or discontinuity can produce a stress concentrating effect.

iii. **Mean stress:** The stress amplitude (σ_a) exerts the major influence on fatigue life. However, the mean stress (σ_m) also strongly affects fatigue life

and is given by (Peter and Vito, 1985; Callister, 2007):

$$\sigma_m = \frac{\sigma_{max} + \sigma_{min}}{2} \quad \ldots\ldots (2)$$

2.4.2 Environmental Factors

The environmental factors that affect fatigue life include; thermal effects, contact fatigue and corrosive effects.

i. **Thermal effects:** The effect of temperature on fatigue behavior can be considered from two different viewpoints: fatigue at a constant temperature (elevated or low), and fatigue due to thermal cycling (also known as the thermal fatigue).

ii. **Contact fatigue:** Failure due to contact fatigue occurs when surfaces contact repeatedly. The stresses developed are more complex than those in standard bending or axial fatigue. In many cases, the areas of contact tend to be small and the resultant stresses are quite large. Contact fatigue generally produces three types of damage: surface pitting, subsurface pitting, and spalling.

iii. **Corrosion fatigue:** This may be considered as a special case of general fatigue, with certain modifying effects resulting from the environment. But the combination of corrosive environment and cyclic stressing can be much more detrimental than either factor acting separately. The time or cycles necessary for fatigue crack initiation can be markedly reduced by corrosion reaction that creates pits, or other surface damage. In

addition, fatigue crack propagation rate can be significantly increased by the corrosive environment, (Peter and Vito, 1985; Afolabi, 2007).

2.4.3 Microstructural Factors

Though increase in fatigue life through changes in microstructure may be perhaps inconsequential, it still plays an important role in the fatigue behavior of engineering materials. Such micro- structural factors may include; grain size, second – phase particles and micro-structural orientation, (Omotade, 2005).

2.5 STAINLESS STEEL

Stainless steels are in wide use for resisting corrosion. There are many alloys of this kind and the proper alloy must be selected for its intended purpose. Chromium is the principal alloying element and occurs in percentages from 11.5 to 30. In some alloys nickel is also present in amounts up to 14%. Close control must be exercised on the carbon content of these materials. The chromium forms a layer of chromium oxide (Cr_2O_3) when exposed to oxygen and the layer is not visible contributing to the shiny appearance of the metal surface.

They are high alloy steels which possess high corrosion and oxidation resistance, making them widely used for resisting corrosion. These characteristics make stainless steel to have numerous applications in nuclear plants, petrochemical industries and food processing units, pulp and paper manufacturing plants. The alloys can be milled into sheets, bars, plates,

wire and tubing to be used for cookware, cutlery, surgical instruments and building materials, (Rollason, 1973; Iliyasu, 2012).

There are different types of stainless steel giving rise to different degrees of corrosion resistance behavior characteristics of the different grades.

There are four categories, these are a*ustenitic, martensitic, ferritic and precipitation hardening stainless steels.*

2.5.1 Austenitic stainless steels

About one-half of all stainless steels produced are austenitic. They are chromium nickel steels with minimum chromium-nickel content of 25%. That is a minimum of 8% nickel and 17% chromium which make the steel completely austenitic in the presence of low carbon contents. The nickel is used principally to increase the ease of fabrication. These steels are widely used in chemical engineering plants and for cutting, due to their best possible properties, though they are costly. Also at rapid cooling from high temperature, the carbides can be retained in solid solution; this makes them possess better corrosion resistance, (William, 1990).

2.5.2 Martensitic Stainless Steels

These steels can be hardened by quenching and then tempered to get the desired mechanical properties. They contain about 11.5% to 18% chromium with smaller amounts of carbon. They are the cheapest among the family of stainless steels and are capable of developing a martensitic

structure. For corrosion resistance the surface must be smooth with all scale and foreign matter removed, (Spott, 1985; William, 1990).

2.5.3 Ferritic Stainless Steels

The forming and drawing properties of these materials resemble those of mild steel. They are similar to martensitic steels, are magnetic in all conditions with very good corrosion resistance and cannot be hardened by heat treatment. They are used in architectural work, automobile rims, hardware and equipment in chemical and food industries. They also have many applications in high-temperature service, (Spott, 1985; Iliyasu 2012).

2.5.4 Precipitation Hardening Stainless Steels

They are strengthened by precipitation of intermetallic compounds. In addition to nickel and chromium, they contain copper, aluminum and molybdenum, (William, 1990).

2.6 HEAT TREATMENT

Heat treatment is an operation involving the heating of the solid metal to a definite temperature followed by cooling at suitable rates in order to obtain certain physical properties which are associated with changes in the nature, form, size and distribution of the micro constituents, (Sudhakar, 2005).

The purpose of heat treatment is:

- To soften the steel, improve its machinability.
- To relieve internal stresses induced by some previous treatment.

- To remove coarseness of grain and
- To improve its ductility.

The types of heat treatment include;

2.6.1Normalizing

This involves heating the steel to 30-50^{o}C above the upper critical temperature followed by cooling in still air. Also when metal is heated to very high temperatures as for forging, it may lose its toughness. To remedy this defect, steel is slowly heated to about 1000^{o}C and allowed to cool in air.

2.6.2Quenching

This involves heating steel in a furnace to a temperature above its upper critical temperature for a specific period, followed by cooling in oil or water in order to bring about the formation of martensite.

2.6.3 Annealing

This refers to a heat treatment in which a metal is exposed to an elevated temperature for an extended period of time and then slowly cooled in the furnace. Annealing is carried out to relieve stresses, increase softness, ductility and toughness and to produce a specific micro structure as shown in figure 2.1.

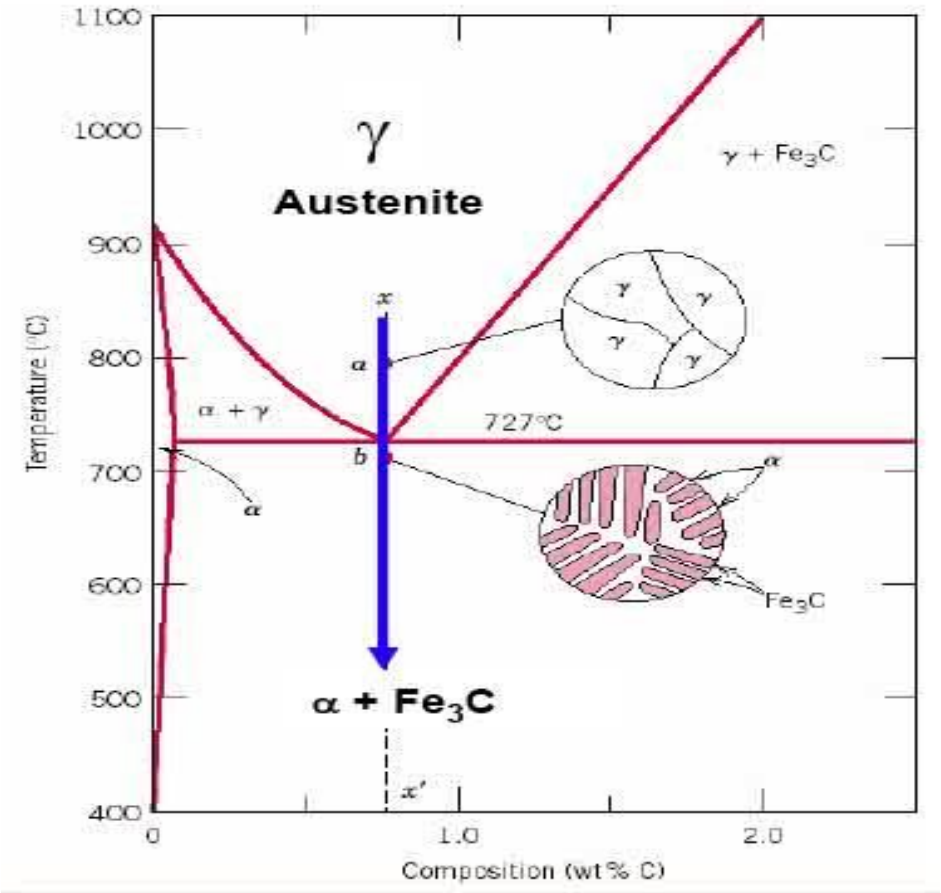


Figure 2.1: Iron-Carbon phase diagram showing the annealing cooling rate of stainless steel(http://www.navaching.com/forge/cooling.html)

2.6.4 Tempering

This is done by heating a martensitic steel to a temperature below the lower critical temperature followed by cooling in air. It is normally done at a temperature between 200^{o}C and 700^{o}C. It also allows the formation of martensite by diffusional process and removes internal stresses set up during quenching, (Callister, 2007; Rajan et al, 1988).

2.7 CORROSION

Corrosion can be defined as the chemical reaction between materials, glasses, ions, polymeric solids and even composites with environments that embrace liquid metals, gases, non-aqueous electrolytes and non-aqueous solutions, (Illiyasu, 2012).

It is the deterioration of material and loss of its properties as a result of reactions and interactions with its environment. Materials corrode at various rates and the extent of corrosion damage depends on interrelated variables involved, (Mars and Norbert, 1978; Callister, 2007).

2.7.1 Forms of Corrosion

The appearance of the corroded metal is the basis ofclassifications and depends on its nature and the prevailing environmental conditions. Some of the forms of corrosion are;

2.7.1.1Inter Granular Corrosion

This occurs when the grain boundaries within an alloy are chemically dissimilar to the grains and the small bulk of the grain boundaries become the cathode.

2.7.1.2Galvanic Corrosion

This is corrosion in the presence of an electrolyte; two dissimilar metals are electrically connected and current flows through them. The anode corrodes in preference to the cathode.

2.7.1.3Pitting Corrosion

This is corrosion with a highly localized attack of metals at a specific area resulting in small pits that penetrate into the metal which lead to perforation, for instance pitting of stainless steel in the presence of chlorine ions.

2.7.1.4 Uniform Corrosion

This is the corrosion that is characterized by chemical or electrochemical reaction that proceeds uniformly over the entire exposed surface area.

2.7.1.5 Crevice Corrosion

This is the form of corrosion that occurs in the small space between two adjoining surfaces as a result of differences in oxygen concentration or metallic ion concentration in that region.

2.7.1.6 Stress Corrosion

This is corrosion where materials deteriorate due to combined action of a tensile stress either applied or residual and a corrosive environment. Metals lose strength as a result of the simultaneous effects of tensile stress and corrosion to an extent greater than the sum of the effects of stress and corrosion acting separately.

The corrosion rate of materials immersed in a corrosive medium for a period of a time is given as, (Callister, 2007):

$$C\,R = \frac{87.6W}{\rho At}\ (mm/yr) \quad \text{.....3}$$

where;

CR = Corrosion rate (mm/yr)

W = weight loss (mg)

ρ = density of material (g/cm^3)

A = area of immersion (cm^2)

t = period of immersion (hrs)

2.7.2 Corrosion of Stainless Steel

Stainless steels get their corrosion resistance by the formation of a very thin surface in the presence of oxygen and they usually have poor resistance to low-oxygen environments such as under deposits, in mud, or in tight place called crevices. But in seawater, the passive film is attacked and destroyed by the chloride ions from the salt.

Austenitic stainless steels usually have better sea water resistance especially alloys with AISI numbers from 300 and above which contain more nickel than those with numbers below 300. They have good corrosion resistance and are ferrite stainless steels. When exposed to fresh and sea water, they suffer mild surface rusting.

All stainless steels suffer from pitting or crevice corrosion when immersed in sea water except the best special alloy stainless steels which are

normally used for impeller blades and propellers because they corrode less when seawater flows fast past them, (Denny, 1996).

2.8 WELDING

Welding can be defined as the union of metals by fusion, diffusion or surface alloying through the application of heat and, or pressure with or without a filler metal. It may also be a metallurgical bond accomplished by the attracting forces between atoms. Welding processes are performed in controlled environments or shielded by an inert atmosphere, (Denny, 1996; Evan, 1981).

2.8.1 Welding Process

It involves fusion and non-fusion welding of metals.

2.8.1.1 Fusion Welding of Metals

In fusion welding, a filler material of similar composition to the metal being joined may also be melted. There are very many fusion-welding processes and the heat necessary for welding may be obtained from a chemical reaction or by using electrical power.

The arc welding process is outstanding among the three as it is relatively affordable and offers reasonable quality welds.

2.8.1.2 WELDING PROCEDURES

There are many procedures, some of which are:

A. SHIELDED METAL ARC WELDING

There are two basic types of electrode coatings. There are the lime types indicated by the suffix 15 and the titanium types designated by the

suffix 16. The lime type electrodes are used only with direct current as positive electrode (reversed polarity). The titanium-coated electrode with the suffix 16 can be used with alternating current and with direct current as positive electrode. Both coatings are of the low-hydrogen type and both are used in all positions. However, the type 16 is smoother, has more welder appeal, and operates better in the flat position. The lime type electrodes are more crack resistant and are slightly better for out-of-position welding. The width of weaving should be limited to two-and-one-half (2.5) times the diameter of the electrode core wire. Covered electrodes for shielded metal arc welding must be stored at normal room temperatures in a dry area. These electrode coatings, of low hydrogen type, are susceptible to moisture pickup. Once the electrode box has been opened, the electrodes should be kept in a dry box until ready to be used, (Aiyelero, 2005).

B. GAS TUNGSTEN ARC WELDING

This process is widely used for thinner sections of stainless steel. 2% tungsten is recommended and the electrode should be ground to a taper. Argon is normally used for gas shielding; however, argon-helium mixtures are sometimes used for automatic applications.

C. GAS METAL ARC WELDING

This process is widely used for thicker materials since it is a faster welding process. The spray transfer mode is used for flat position welding and this requires the use of argon for shielding with 2% or 5% oxygen or

special mixtures. The oxygen helps in producing better wetting action on the edges of the weld. Short-circuiting transfer can also be used on thinner materials. In this case CO_2 shielding or the 25% CO_2 plus 75% argon mixture is used. The argon-oxygen mixture can also be used with small-diameter electrode wires. With extra low-carbon electrode wires and CO_2 shielding the amount of carbon pickup will increase slightly. This should be related to the service life of the weldment. If corrosion resistance is a major factor,CO_2 gas orCO_2-argon mixture should not be used. For all welding operations, the weld area should be clean and free from all foreign materials, oil, paint, dirt, etc. The welding arc should be as short as possible when using any of the arc processes, (Aiyelero, 2005).

D. METAL INERT GAS WELDING (MIG)

Metal inert gas welding is the welding process in which the weld is shielded by an external gas (argon, helium, CO_2, argon + oxygen or other gas mixtures). Consumable electrode wire, having chemical composition similar to that of the parent material, is continuously fed from a spool to the arc zone. The arc heats and melts both the work pieces edges and the electrode wire; the molten electrode material is supplied to the surfaces of the work piece, fills the weld pool and forms a joint.

Due to automatic feeding of the filling wire (electrode) the process is referred to as a semi-automatic. The operator controls only the torch positioning and speed, (Denny, 1996; Aiyelero, 2005).

2.8.2 Welding Filler Metals

The selection of the filler metal alloy for welding stainless steels is based on the composition of the stainless steel. Various stainless steel filler metal alloys are normally available as covered electrodes and as bare solid wires. Recently flux-cored electrode wires have been developed for welding stainless steels.

Filler metal alloy for welding the various stainless steel base metals are:

Cr-Ni-Mn (A1S1 No. 308);

Cr-Ni-Austenitic (A1S1 No. 309, 310, 316, 317, 347);

Cr-Martensitic (A1S1 No. 410, 430);

Cr-Ferritic (A1S1 No. 410, 430, 309, 502).

It is possible to weld several different stainless base metals with the same filler metal.

2.8.3 Weldability of Stainless Steel

We may define weldability of a metal as its capacity to be welded into a specific structure that has certain properties and characteristics that will satisfactorily meet its service requirements. It involves a large number of variables, making generalization difficult. These variables include material characteristics such as alloying elements, impurities, inclusions, grain structure, and professing history of the base metal and filler metal.

Because of the melt solidification and micro structural changes involved, a true knowledge of the phase diagram and the response of the

metal or alloy to elevated temperature over a period of time are essential. Also influencing weldability are the mechanical and physical properties such as strength, toughness, ductility, notch sensitivity, modulus of elasticity, specific heat, melting point, thermal expansion and surface tension characteristics of the molten metal.

Preparation of surfaces for welding is important, as the nature and properties of surface oxide films and absorbed gases the welding process employs significantly affect the temperatures developed and their distribution in the weld zone. Other factors are shielding gases, fluxes, moisture content of the coating on electrodes, welding speed, welding position, cooling rate, preheating and post welding techniques (Denny, 1996; Aiyelero, 2005).

2.8.4 Pre-Heating Temperature

This is the temperature at which the metal is heat treated before welding; usually at low range temperature distribution, increasing this parameter has the same effect as that of power input, and also reduces the magnitude of shrinkage stress which is required where distortion and metallurgical transformation control are desired.

2.8.5 Welding Speed

This is the speed with which the electrode transverses the work piece. Increasing this parameter increases the steepness of the temperature gradient and hence a faster cooling rate results. This follows from the fact that at higher speed, weld energy is very quickly delivered to a very small portion

of the work piece. So that the adjacent metal has enough time to get heated up by diffusion.

2.8.6 Post Weld Treatment

This is the temperature at which the weld is heat treated after the welding operation. It is usually required to improve impact resistance, reduces residual stresses and provides greater uniformity in the metallurgical structure across the weld.

2.8.7 The Metallurgy of Welding

In welding, heating and cooling occur as essential steps in any of these processes. Some metals are melted while some parts are subjected to temperatures above those at which phase transformation(s) or grain growth occur. This produces metallurgical effects that are usually not desirable.

The state of the weld at solidification depends strongly on the rate of removal of coatings produced by the electrode, therefore it is necessary to decide whether a fresh weld is to be quenched, cooled slowly or an extra layer allowed cooling normally in air. In the center of the weld is a zone that is made up primarily of weld metal that has solidified from the molten stage. The grain structure in this zone may be fine or coarse and dendritic depending on the type and volume of weld metal and the cooling rate. When the metal solidities, it cools from the outside inwards and the crystal grows towards the center. In most cases, the resulting structure is dentritic. The junction of the dendrites in the center is weak and contains segregated

components and impurities; next to the weld metal zone is the ever present and undesirable heat affected zone (HAZ) (Denny, 1996; Aiyelero, 2005).

2.8.8 Heat Affected Zone (HAZ)

The HAZ is within the base metal itself it has a microstructure different from that of the base metal after welding, because it has been subjected to elevated temperatures for a period of time during welding.

This zone is the area surrounding the weld whose microstructure has been affected by the heat generated by the welding operation. The hotter a structure becomes during welding, the larger the HAZ, the larger the area in which the microstructure of the material is affected leading to greater distortion and other service problems.

These variations in structure of the HAZ cause some property gradients and usually set up internal stresses; these stresses aid corrosion and metal fatigue and may lead to early failure. Beyond the HAZ is the base metal that was not heated sufficiently to cause any micro structural changes. The microstructure of the weld varies and is complex, but it can be normalized to an extent by heat treatment.

The influence of welding on some materials can act simultaneously as both a rapid heating and cooling and an annealing cycle depending on the part of the HAZ referred to, thus the effects of weld heat on the HAZ is discussed below, (Denny, 1996; Aiyelero, 2005).

2.8.9 Problem of Heat Affected Zone During Welding of Stainless Steel

There might be the problem of carbide precipitation in the HAZ of the metal in welding of austenitic stainless steels. During welding these steels are heated in the range of 450-850^{o}C, which is the sensitizing temperature range, or cooled slowly through that range, carbon is precipitated from that solution mainly at grain boundaries and unites with chromium to form Cr- rich carbides. The chromium content of the adjacent metal is thus reduced, and corrosion resistance is seriously impaired.

This phenomenon is termed carbide precipitation and the type of corrosion it promotes is known as intergranular corrosion. It can be minimized by water quenching from 1050^{o}C after welding. This treatment takes the carbides back into solid solution. Reducing the carbon content and decreases the amount of carbide precipitation at the grain boundaries. Addition of stabilizing elements, titanium and niobium, which have stronger affinity for carbon than chromium and forming elements, Si, Mo and W reduce carbon precipitation, (Denny, 1996; Aiyelero, 2005).

2.9 MICROSTRUCTURAL BEHAVIOUR

This is the arrangement of the micro grains of the austenite, martensite, ferrite, cementite and pearlite in a micro-cross section of the metal when viewed under photomicrographs.

Microscopic study of materials helps in the determination or prediction of the properties of a material. Both optical and electron

microscopes are commonly used in microscopy. When this instrument is connected in some way to a photographic device, photomicrographs of the microstructure can be taken. Before taking the photomicrographs of a specimen, the specimen is first made to undergo a series of preparation such as cutting, grinding, polishing until a mirror-like surface is obtained and then etched. The photomicrographs reveal phases such as

ferrite-which is otherwise called α–iron phase at room temperature.

Austenite-which is a result of the transformation at temperatures above the upper critical temperature.

Martensite-which is formed when austenite is rapidly cooled (or quenched) to relatively low temperature.

Cementite,is one of the elements of the transformation of austenite much harder but more brittle than ferrite.

Pearlite; a two-phase microstructure found in some steels and cast irons results from the transformation of austenite of eutectoid composition and consists of alternating layers (or lamellae) of ferrite and cementite.

It is to be noted that martensite is the hardest and strongest and the most brittle material while tempered martensite consists of extremely small and uniformly dispersed cementite particles embedded within a continuous ferrite matrix, making it nearly as strong and hard as martensite but with substantially enhanced ductility and toughness, (Fontana,1998; Hassan, 2003).

2.10 Review of Previous Researches on Heat Treatment, Welding, Corrosion and Fatigue.

Aruleba (2004) researched on the effect of isothermal tempering and time on stress corrosion failure of stainless steel rods in acidic and sea water environments. He discovered that the corrosion penetration rate of stainless steel decreases with increasing temperature and time. Also, the isothermal tempering and time can be used to control the corrosion penetration rate of stainless steel.

Loto and Ives (1994) worked on the corrosion resistance of super austenitic stainless steel in seawater. They discovered that except the type 3161, all the alloys used in the research work had high resistance to corrosion in seawater. Also to varying degrees under high stream temperature and depending on testing conditions, the alloys were found to be susceptible to crevice corrosion attack.

Aribisala (1980) worked on the effect of heat treatment on the machinability of mild steel. He found that fine-grained microstructure gives a smoother surface than coarse-grained one and also that a fine-grained microstructure is more prone to corrosion than the coarse-grained material.

Hassan (2003) worked on the evaluation of hardening and tempering characteristics of ordinary and chromium nodular cast iron and showed that water quenching may result in cracking and distortion particularly in casting of complicated shapes and therefore, in such cases oil quenching, which is

less severe than water quenching is preferred.

Oloche and Ause (2002) worked on the effect of post quenched treatment on hardness of nodular cast iron quenched in different media. They showed that cold treatment increases hardness of nodular cast iron and that tempered cold treated specimen retained appreciable hardness.

Oni (1988) worked on the effect of tempering temperature and time on stress- induced failure of low carbon steel in corrosive environments. He showed that resistance to stress corrosion failure increases with tempering temperature and time.

Adeyemi (1998) worked on the effect of heat treatment on the quality of the surface finish of turned steel rods. He observed that the surface finish/roughness numbers of the mild steel rods in annealed and normalized conditions under lubricated conditions were greatly dependent on the cutting speed. Also, higher surface roughness numbers were obtained for normalized mild steel rods than annealed mild steel rod under identical cutting speed investigation.

Hosary and Saleh (1985)worked on the synergistic effect of halides and coal tar distillation of the corrosion of mild steel in sulphuric acid. They showed that halides and coal tar distillation products inhibit corrosion.

Loto (1992) worked on the effect of inhibitors in the corrosion of mild steel in acidic and seawater environments. He revealed that inhibitors have great effect on the corrosion of mild steel in acidic and seawater

environments.

Addington and Hendricks (2002) worked on corrosion control in amine systems with 316 stainless steel. They revealed that some process operators attempt to mitigate the corrosion of stainless (SS) steel by applying the same methods used to control corrosion of carbon (CS) steel. However, the two react differently to the corrosive conditions present in an amine system.

Umoru et al (2003) worked on corrosion characteristic of NST-37-2 and NST-60-Mn rebars concrete exposed to different media. They revealed that IM HCI, IM NaOH and seawater environments are capable of causing concrete corrosion although at different rates and levels, also the NST-60Mn steel reinforcement is more resistant to concrete corrosion than the NST-37-2 steel.

Sololu (1989) investigated the effect of crude oil and seawater on fatigue strength of ST 60 Mn steel. The result obtained showed that crude oil and saline environments (seawater) lower the fatigue strength of ST 60Mn steel and the kind of heat treatment given causes further decreases in the fatigue strength.

Odebisi (2001) and Olubusi (1995) carried out research on corrosion fatigue behaviour of ST 60Mn steel in cocoa mucilage and in cassava juice respectively. They observed that tempering will enhance the fatigue strength of ST 60 Mn steel in a corrosive environment.

Oluwole (2001) worked on the effect of heat treatment on the damage ratio of corroded ST 60 Mn steel. He recommended that manganese steels like ST 60 Mn used for oil pipelines should be heat treated by normalizing prior to use in order to enhance the fatigue strength of this grade of steel and thus prevent oil spillage.

Aiyelero (2005) worked on the effect of welding and heat treatment on the mechanical properties of austenitic stainless steel. He observed that the microstructure of the as-received samples is more fine than the welded and heat treated samples. The as-received samples have higher values of impact energy.

Omotade (2005)worked on the corrosion fatigue and microstructural behaviour of stainless steel rods under varied tempering temperatures. He observed that seawater corroded specimen have lower cycles to failure compared to the corresponding cyanide corroded specimen at the same fatigue strength. Also increasing temperature was observed to increase the fatigue life of the specimen and produced a phase transformation that improved the fatigue of the stainless steel rods.

Afolabi (2007) carried out investigation to study critically the corrosion behaviour and stress corrosion cracking (SCC) of low and medium carbon steels in cassava and cocoa extracts by weight loss measurement and constant extension to fracture methods respectively. The results obtained showed that medium carbon steel is more susceptible to

corrosion than low carbon steel in both media. SCC is also more in medium carbon steel than low carbon steel in the two media under study. These deductions are due to higher carbon content in medium carbon steel coupled with various aggressive corrosion constituents contained in these media.

Iliyasu (2012) worked on the susceptibility of austenitic stainless steel to stress corrosion cracking in some aggressive environments. The results showed that at elevated temperature, the chloride solution was more damaging to austenitic stainless steel than the sulfide solution.

However no research work has been carried out on corrosion fatigue and micro-structure effects on welded austenitic stainless steel immersed separately in seawater, hydrochloric acid and wet steam environments.

Therefore, this study intends to provide more detailed information on corrosion fatigue and micro structure effects on welded austenitic stainless steel under heat treatment conditions.

CHAPTER THREE

METHODOLOGY

3.1MATERIALS

The corrosive media used in this study include:

0.5M hydrochloric acid, seawater and steam.

The etchants used are:

5g $FeCl_2$, 50ml HCl, 100ml water (for the as-received samples) and

4g $CUSO_4$, 20ml HCl, 20ml water (heat treated and corrosive).

3.2 EQUIPMENT

The equipment used in this study includes:

Metal analyser, lathe machine, gemco heat furnace, duplex fatigue testing machine, weighing balance, plastic bowls, austenitic stainless steel, metal inert gas welding machine, grinding, polishing and etching machines, photographic visual metallurgical microscope.

3.3 METHODS

3.3.1 Preparation of 0.5M HCl acid

Standard concentrated HCl acid has a density of 1.18g/cm^3 and a percentage purity of 36%. It has a molar mass of 35.5g.

To obtain the molarity of the concentrated HCl acid, the following relation was used:

$$\text{Molarity} = \frac{\text{Concentration}}{\text{Molar Mass}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 1$$

And concentration = standard volume x density x percentage purity

For 1 litre ($1000cm^3$) of standard HCl acid,

Concentration = 1000 x 1.18 x 36%

Hence, the molarity is given by:

$$\text{Molarity} = \frac{1000 \times 1.18 \times 36\%}{35.5}$$

$$= 11.97M$$

From the relation by (Callister, 2007)

$C_1V_1 = C_2V_2$,……..…2

where:

C = Concentration and

V = Volume

$500cm^3$ of 0.5M HCl acid is required,

Hence,

$11.97 \times V_1 = 0.5 \times 500$

$$V_1 = \frac{0.5 \times 500}{11.97}$$

$$= 20.80cm^3$$

Hence, $20.80cm^3$ of HCl from a standard Winchester bottle was dissolved in $500cm^3$ of distilled water to obtain the desired concentration.

3.3.2 Preparation of specimen

The work material, (austenitic stainless steel rods) were purchased from the steel market in Zaria in the form of rods and the chemical composition was carried out at Universal Steel Ikeja, Lagos using a spectrometer machine.

3.3.2.1 Metal analysing procedure

This was done to determine the elements present and their percentages in stainless steel. A 20mm x 30mm piece of standard pipe samples was made. The surface of the sample was ground in a grinding machine to obtain a smooth surface. The sample was then placed in the orifice on the machine, a pin to pin spark was impressed on the sample using a solid technique optical emissions spectrograph with 0.0001% sensitivity. The reading was taken from a direct computerized spectrometer. The procedure was repeated two times and a mean value was obtained that revealed a type 316, straight grade austenitic stainless steel as shown in the table 3.1below. A straight grade austenitic stainless contain maximum of 0.08% C while type 316 contains 16% to18% chromium and 11% to 14% Nickel.

(http://www.spiusa.com/Ref001/austenitic.htm)

Table 3.1: Chemical compositions of austenitic stainless steel

Element	C	Si	S	P	Mn	Ni	Cr	Mo	V
% by wt	0.079	0.212	0.223	0.249	1.216	14.189	17.189	1.538	0.037

Element	Cu	W	As	Sn	Co	Al	Pb	Ca	Zn	Fe
% by wt	0.155	0.416	0.150	0.158	0.134	0.008	0.004	0.0001	0.019	63.215

The austenitic stainless steel rods of 10mm diameter were cut into equal lengths and machined on the lathe machine to ASTM standard code D3166-99 for a fatigue test for AVERY 7304 fatigue testing machine as shown in figure 3.1a below. The machining was done at the production laboratory of the Mechanical Engineering Department, Ahmadu Bello University, Zaria.



Figure 3.1a: Fatigue Specimen(Mechanical Engineering Dept, Ahmadu Bello University, Zaria)

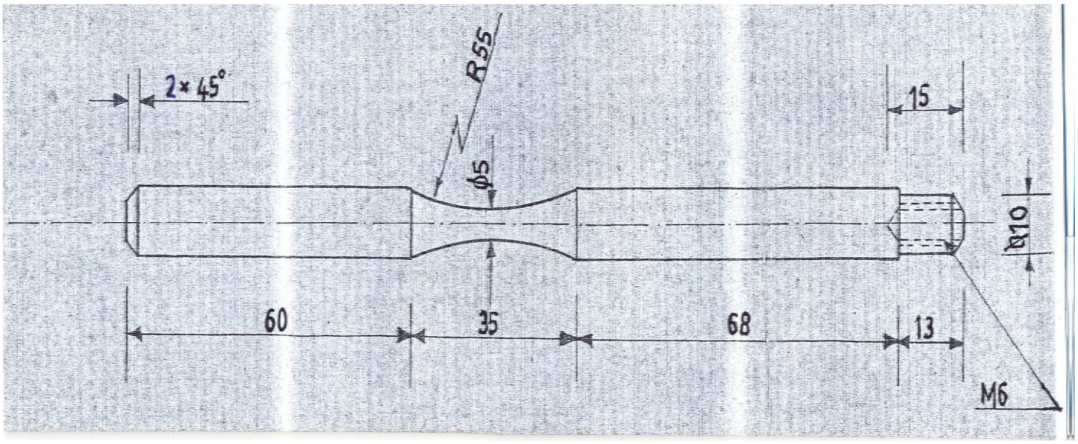


Figure 3.1b: Fatigue Specimen (schematic diagram)

3.3.3 Fatigue Testing Machine

Fatigue testing machines are classified according to the purpose or specimen to be tested, type of stress and means of producing the loads. In this work the dynamic testing machine was used as shown in the figure 3.2 and plate 3.1.

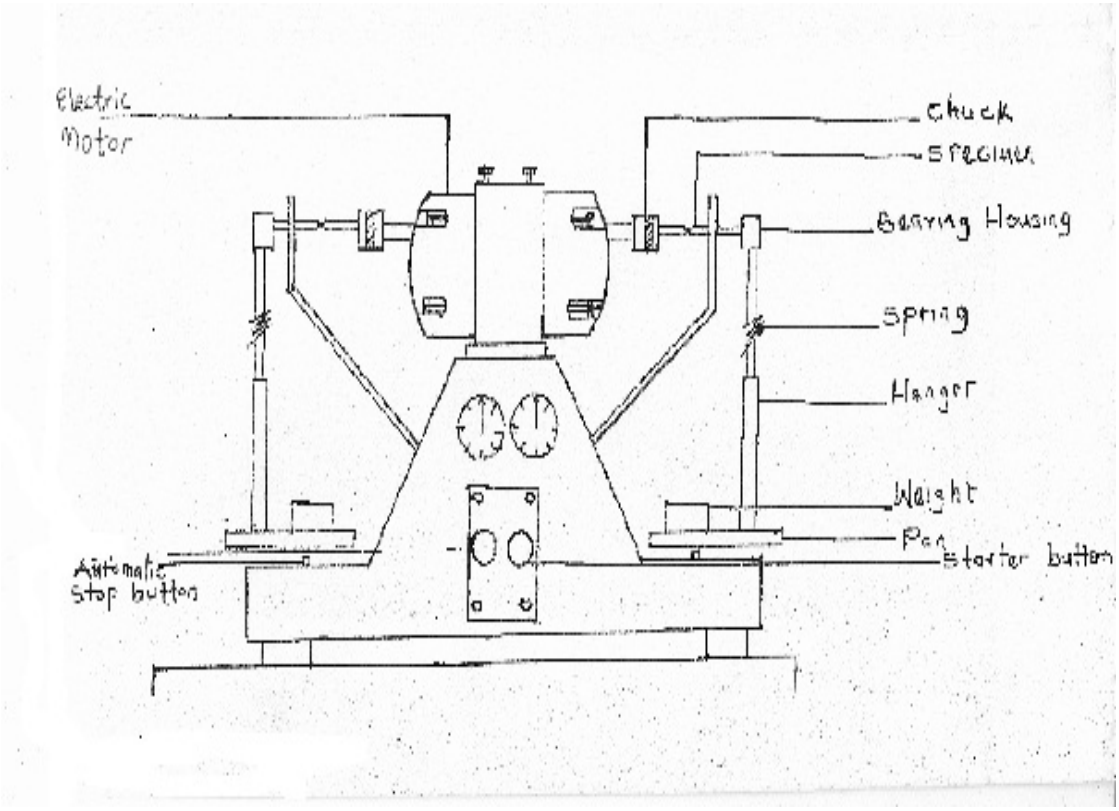


Figure 3.2: Duplex Fatigue Testing Machine (schematic diagram)



Plate 3.1: Duplex Fatigue Testing Machine (Mechanical Engineering Dept, Ahmadu Bello University, Zaria)

Machine data:

Test speed (n) – 3000 rev/min

Weight of load pans, with suspension bearings and springs-22.24N

Maximum added weight – 200.23N

Power – Electric supply

Manufacturer – Avery Ltd. Sotto Fountary Birmingham

3.3.4 TESTPROCEDURE

The corrosive media that were used for this experiment are seawater, 0.5M hydrochloric acid and steam.

Table 3.2: The chemical composition of natural seawater

Element	Na^+	Mg^+	Ca^{2+}	K^+	Sr^{2+}	Cl^-	SO_4^{2+}	Br^-	H_2BO_3
CONC. g/kg	10.77	1.30	0.409	0.338	0.010	19.37	2.71	0.065	0.026

The seawater having the pH value of 8 and the salinity of 3.5% (Shaaba, 2012)

The seawater (see table 3.2 for chemical composition) was obtained from bar – beach, Lagos, the 0.5M hydrochloric acid was obtained from Chemical Engineering Department, Ahmadu Bello University, Zaria and the specimen were subjected tosteam at Sunseed Nig. Ltd, Jos road, Zaria.

The specimen were machined and cut into two equal parts and then welded together using MIG (Metal Inert Gas welding) as shown in figure 3.4 below. The welding was carried out at the Centre for Energy Research and Training,Ahmadu Bello University, Zaria.

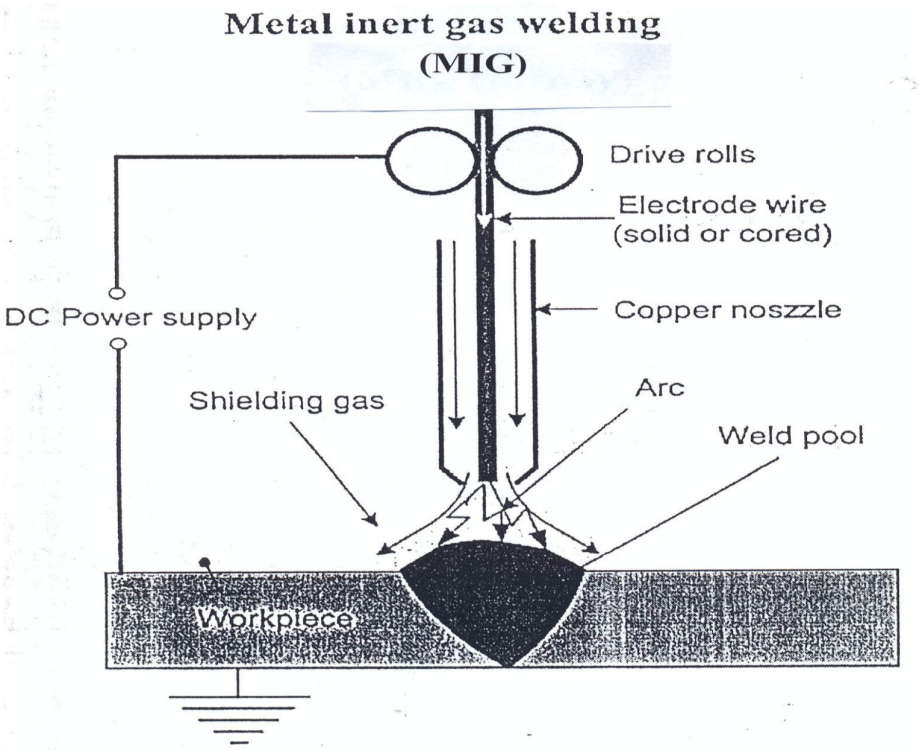


Figure 3.3: Metal Inert Gas Welding (Center for Energy and Research Institute, Ahmadu Bello University, Zaria)

Some specimen were retained in as–received condition while the others were annealed at the Industrial Development Centre (IDC), Zaria as shown in figure 3.5 below in order to relieve stresses, increase softness, ductility and toughness after welding.

3.3.5 Post-welding heat treatment

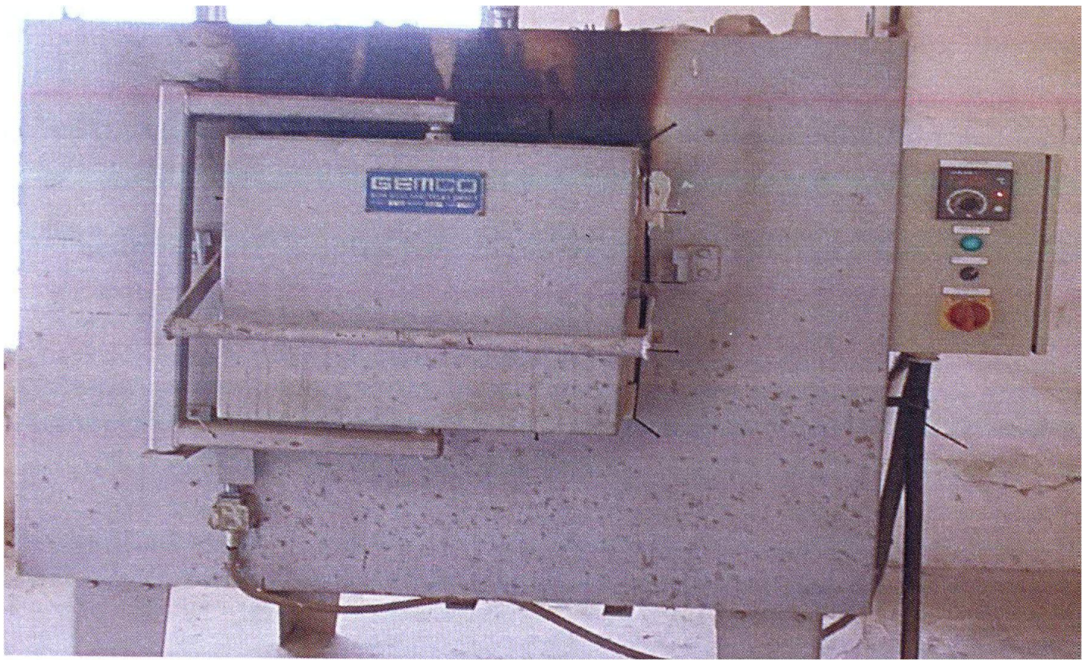


Plate 3.2:Gemco Furnace (Industrial Development Centre, Zaria)

The samples were placed in the furnace and the furnace was closed. The power was then switched on and the temperature was set at 930^0C. After attaining the set temperature, the samples were soaked for 2h after which the furnace was switched off and the samples were left to cool to room temperature in the furnace according to the method of Aiyelero (2005). The samples were then removed from the furnace.

The respective annealed specimens werethen immersed in seawater, 0.5M hydrochloric acid and steam corrosive media for 30 days.

3.4 WEIGHT LOSS EXPERIMENT

The test specimen were weighed before being immersed totally in each of the corrosive media in a plastic bowl and then covered for 30days. The test specimen were taken out of the corrosive media (one each) after every 5 days, washed with distilled water and acetone, air dried and re–weighed according to the method of Yawas (2005).

Plots of weight loss versus exposure time and corrosion rate versus exposure time are shown graphically in fig 4.1 to fig 4.5. All the experiments were performed at room temperature. The results of the tests are shown in appendix A1 to appendix A3.

3.5 FATIGUE TEST

The duplex fatigue testing machine belongs to the class of rotating bar fatigue testers. It provides for the determination of alternating bending stresses and can only be used for imposing upon the specimen pure alternating bending stresses.

The fatigue testing machine applied cyclic loads to the test specimen. Fatigue testing is a dynamic testing mode and can be used to simulate how a component/material will behave/fail under real life loading/stress conditions, (www.azon.com/materials-equipment.aspx).

An electric motor mounted on the machine rotated the two chucks at the rotor ends. These chucks were used to hold two specimen with the load placed on the pan weighing 22.24N which was hung on a spring mounted on

the specimen through the roller bearing. Rotating bending was achieved as bending is vertically downward.

There are two dial counters on the sides of the machine indicating the number of stress reversals through which each specimen has gone. After placing the specimen on the machine, it was switched on and as soon as the specimen failed, the pan which carries the weight falls down, pressing a button which automatically stops the counter and the machine when both specimens have failed.

Hence the dial reading was taken at any time after the specimen failed. The counter can record up to 10,000,000 reversals. The fatigue stress was recorded against each specimen. The results are shown in appendix A4 to appendix A6.

3.6 METALLOGRAPHY

Metallography was carried out on the as-received and annealed specimen in the following sequence of operation in order to know the effects of various tests on the microstructure.

3.6.1 Grinding

This involves the use of fine grinding wheels covered with surfaces in the sequence of 180, 240, 320, 400 and 600grit. The specimen was applied with a uniform but firm pressure against the abrasive surface back and forth in straight but even motion. The grinding sequence followed from the

coarsest (180 grit) to the finest (600 grit). For each step the grinding was done perpendicular to the previous direction and the specimen was rinsed in water to remove abrasive particles between each step and was thoroughly cleaned after the last step.

3.6.2 Polishing

This involves the use of a polimet polisher machine. At this stage, emery cloth and solution of silicon carbide (SiC) were used to remove the ridges left on the surface of the specimen after grinding. The mechanism of polishing is such that it leaves an amorphous layer of metal on the surface. When polishing, a constant drop of water and alumina polishing powder were fed to the rotating pad which was run at low speed against the specimen until a very smooth mirror-like surface was obtained.

3.6.3 Etching

This is the chemical attack on the surface of a polished metal to reveal the full structure for microscopic analysis. The etchants used are:

For the as-received samples: 5g $FeCl_2$, 50mls HCl, 100mls water.

For the remaining samples (heat treated and corroded):

4g $CUSO_4$, 20mls HCl, 20mls water.

3.6.3.1 Etching procedure

After preparation of the etchant in a beaker, a piece of cotton wool was soaked in the etchant and swabbed the polished surface of the sample for 15 minutes. Then, the photomicrography of the specimen at the point of fracture and the parent material were taken using a photographic visual metallurgical microscope at the Metallurgical and Materials Engineering Department of the Ahmadu Bello University,Zaria, (see figure 3.6 below).



Figure 3.6: Photographic visual metallurgical microscope at Metallurgical and Materials Department, Ahmadu Bello University, Zaria.

The microstructural examinations are shown in plates 4.1 to plates 4.5.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 RESULTS

The results of fatigue tests, corrosion tests and microstructural examination of the samples are shown in table A1 – A6, figures 4.1 – 4.15 and plates 4.16a – 4.20b respectively.

The corrosion rate is the rate at which a material deteriorates in a specific environment and was calculated in mm/yr using corrosion penetration rate (CPR), using the relation (Callister, 2007):

$$CPR = \frac{KW}{\rho A t} \quad \text{……..……1}$$

where:

For stainless steel

K = constant = 87.6

ρ = density of the material in $g/cm^3 = 8.03 g/cm^3$

A = Exposed area of specimen in mm^2

$= (2\pi rl + 2\pi r^2)$

$= 2\pi r (l + r)$……2

For the specimen

$r = {}^{d}/_{2}$

$= {}^{100}/_{2}$

= 50mm

l = 250mm

$$A = 2\pi \times 50 \times (250+50)$$

$=94285.7mm^2$

W = Weight lost in milligrams

= Weight before corrosion – Weight after corrosion

t= Exposure time in hours.

4.2 EFFECT OF CORROSIVE MEDIA

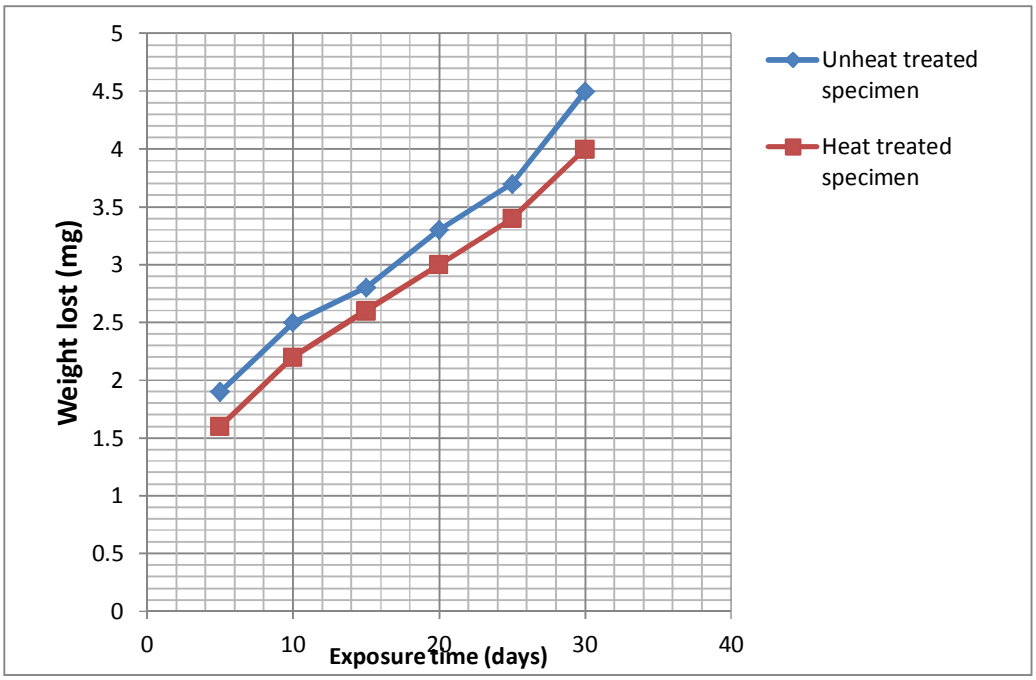


Fig 4.1 Variation of Weight Lost with Exposure Time of Austenitic Stainless Steel in Seawater.

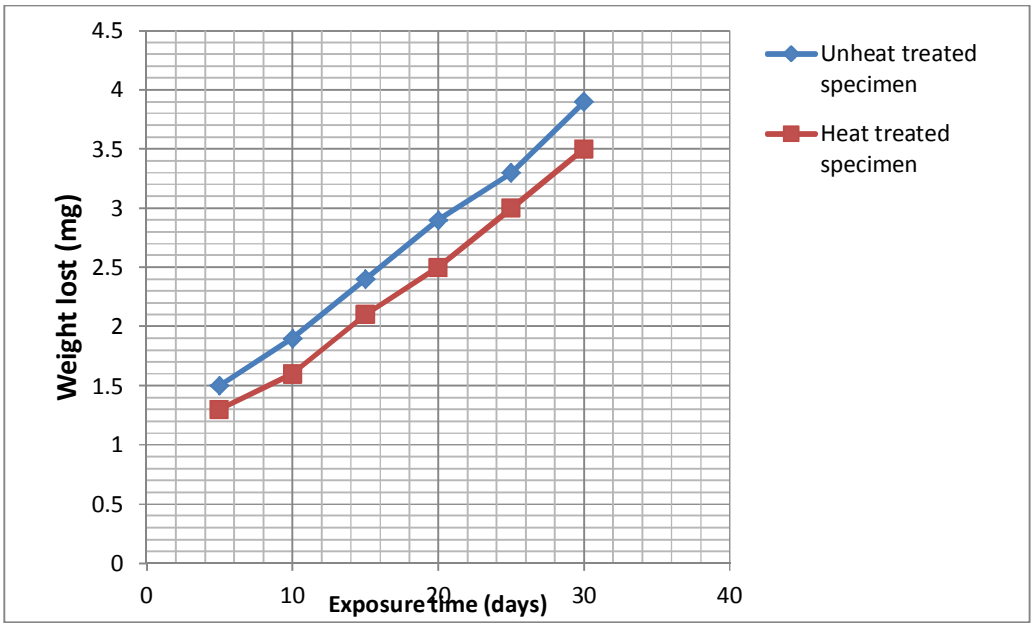


Fig 4.2 Variation of Weight Lost with Exposure Time of Austenitic Stainless Steel in 0.5M HCl.

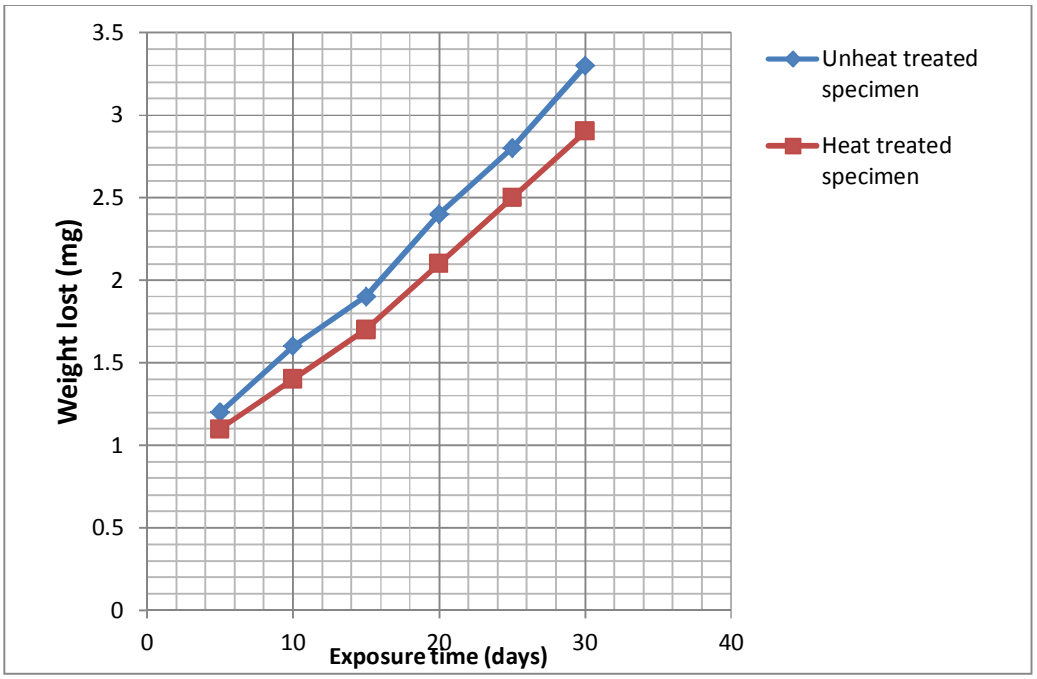


Fig 4.3 Variation of Weight Lost with Exposure Time of Austenitic Stainless Steel in Wet Steam.

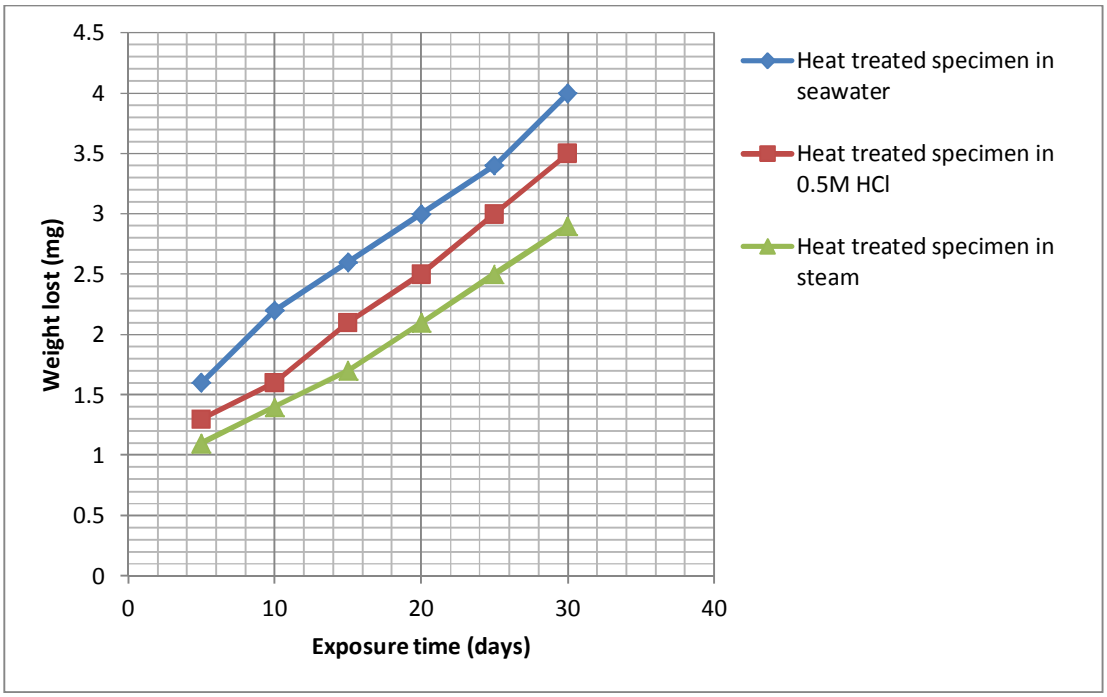


Fig 4.4 Variation of Weight Lost with Exposure Time of Heat-treated Austenitic Stainless Steel in Seawater, 0.5M HCl and Steam.

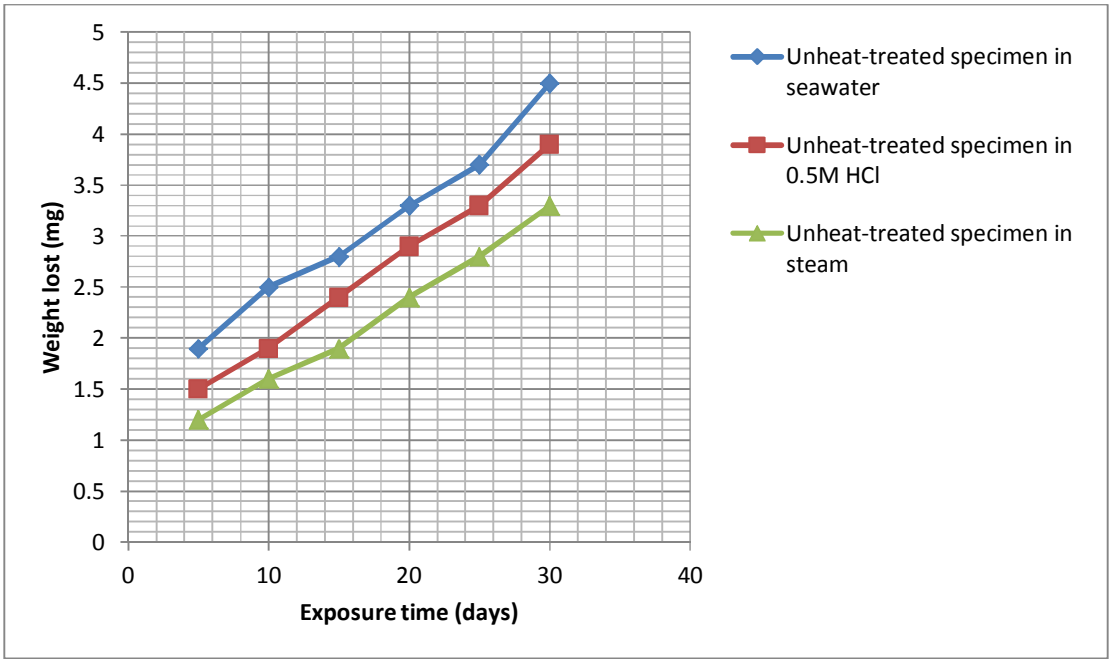


Fig 4.5 Variation of Weight Lost with Exposure Time of Unheat-treated Austenitic Stainless Steel in Seawater, 0.5M HCl and Wet Steam.

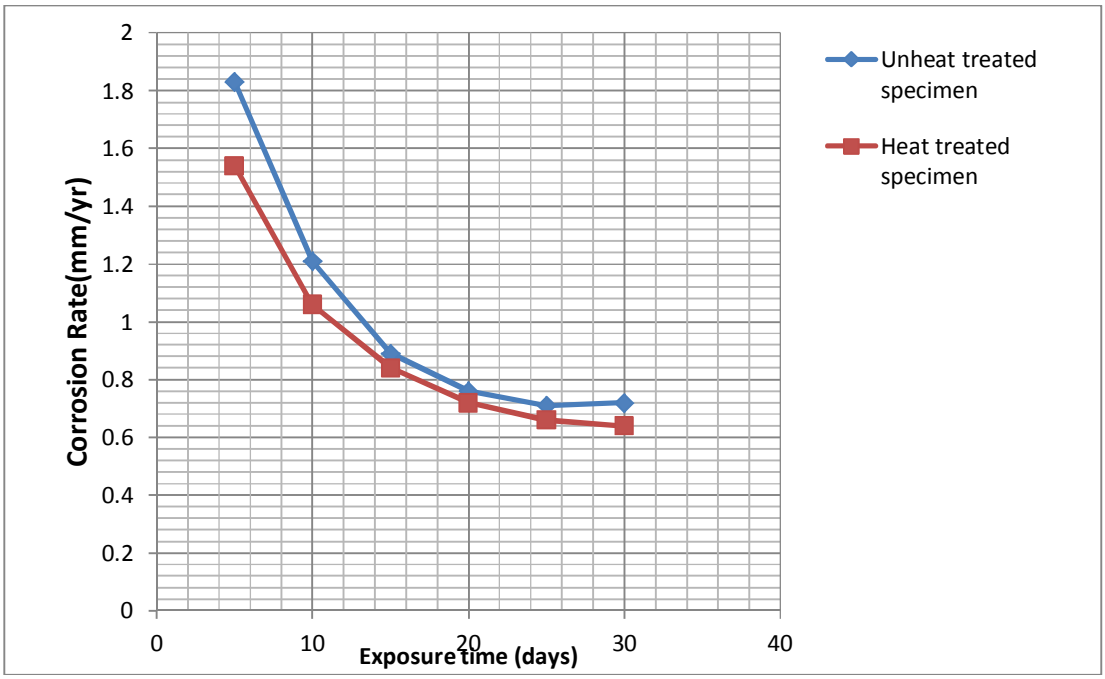


Fig 4.6 Variation of Corrosion Rate with Exposure Time of Austenitic Stainless Steel in Seawater.

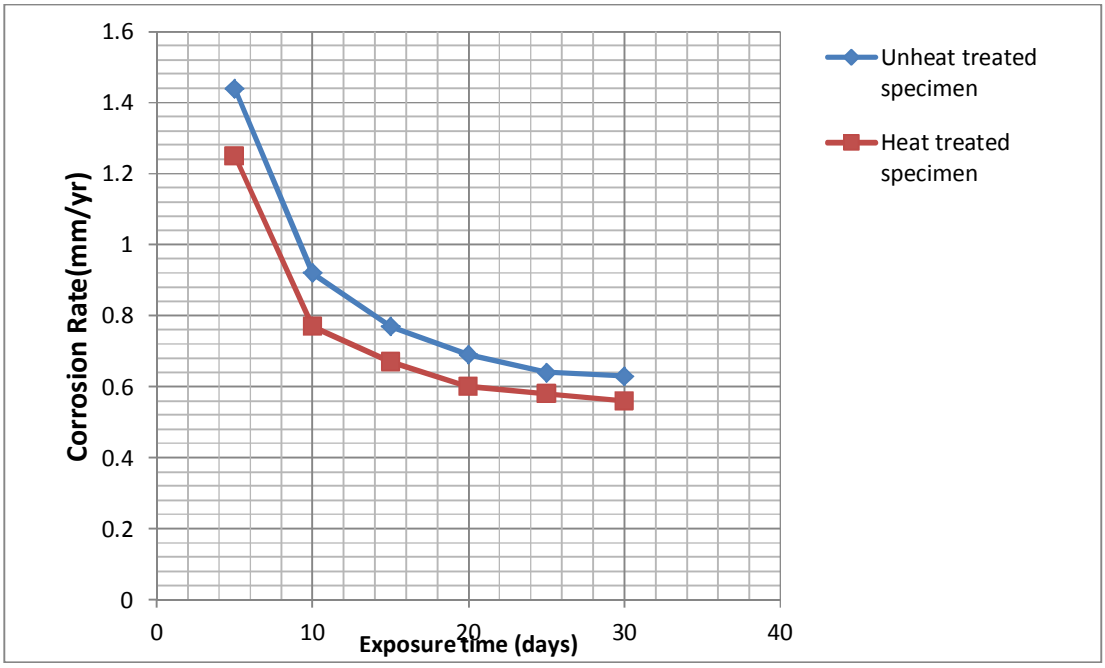


Fig 4.7 Variation of Corrosion Rate with Exposure Time of Austenitic Stainless Steel in 0.5M HCl.

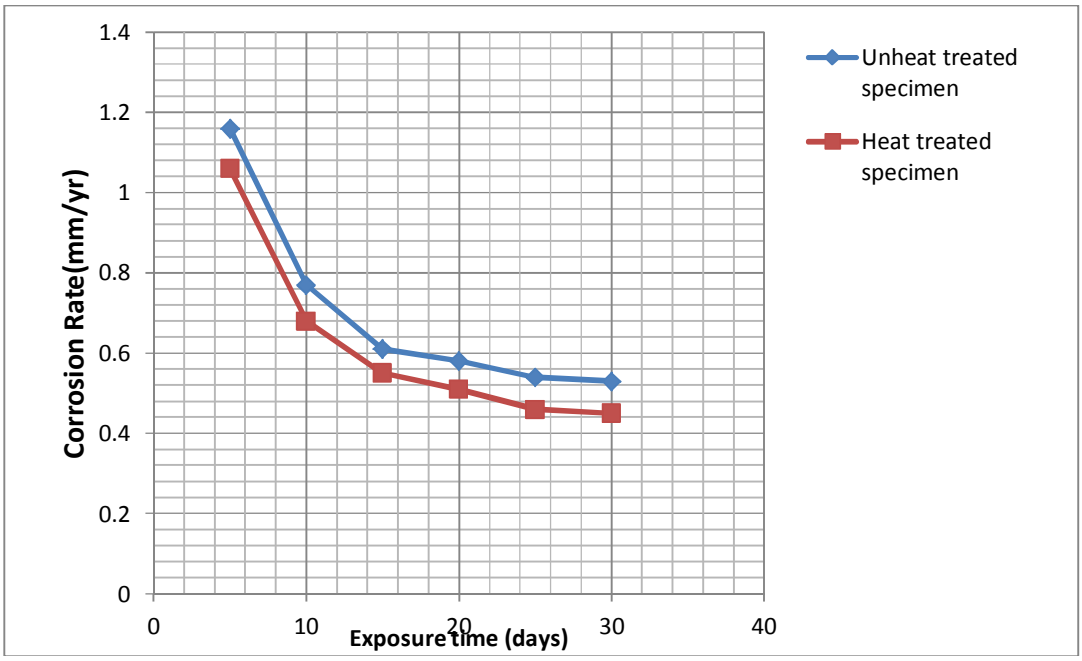


Fig 4.8 Variation of Corrosion Rate with Exposure Time of Austenitic Stainless Steel in Wet Steam.

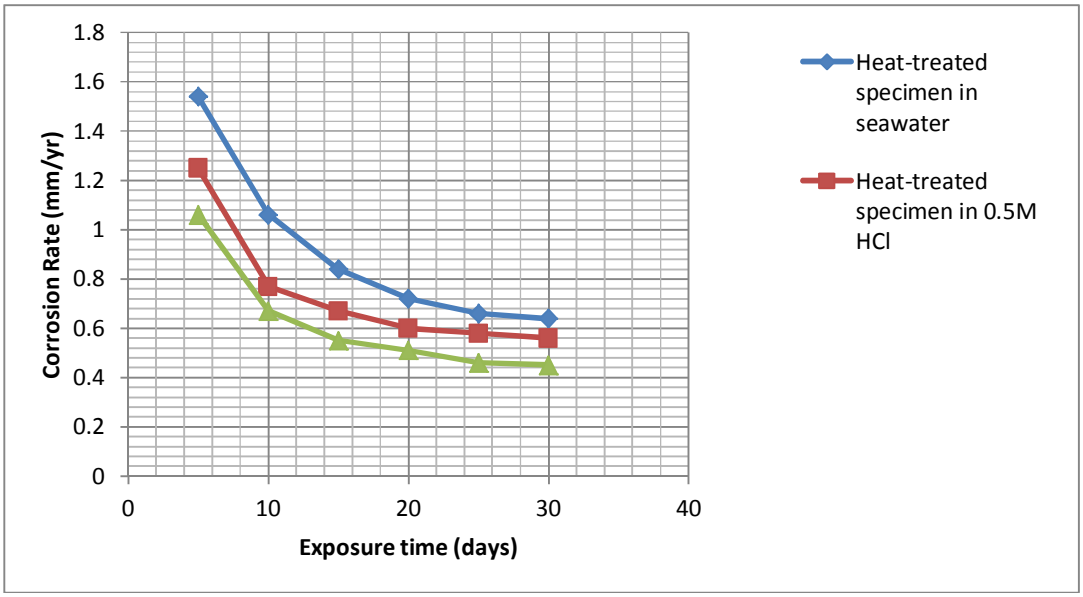


Fig 4.9 Variation of Corrosion Rate with Exposure Time of Heat-treated Austenitic Stainless Steel in Seawater, 0.5M HCl and Wet Steam.

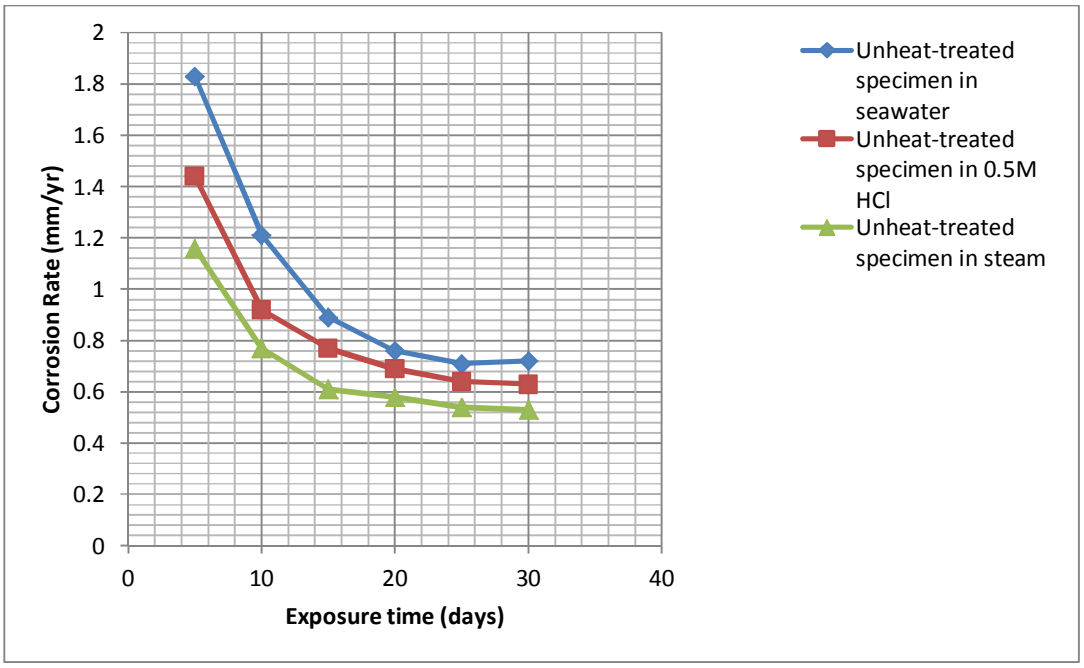


Fig 4.10 Variation of Corrosion Rate with Exposure Time of Unheat-treated Austenitic Stainless Steel in Seawater, 0.5M HCl and Wet Steam.

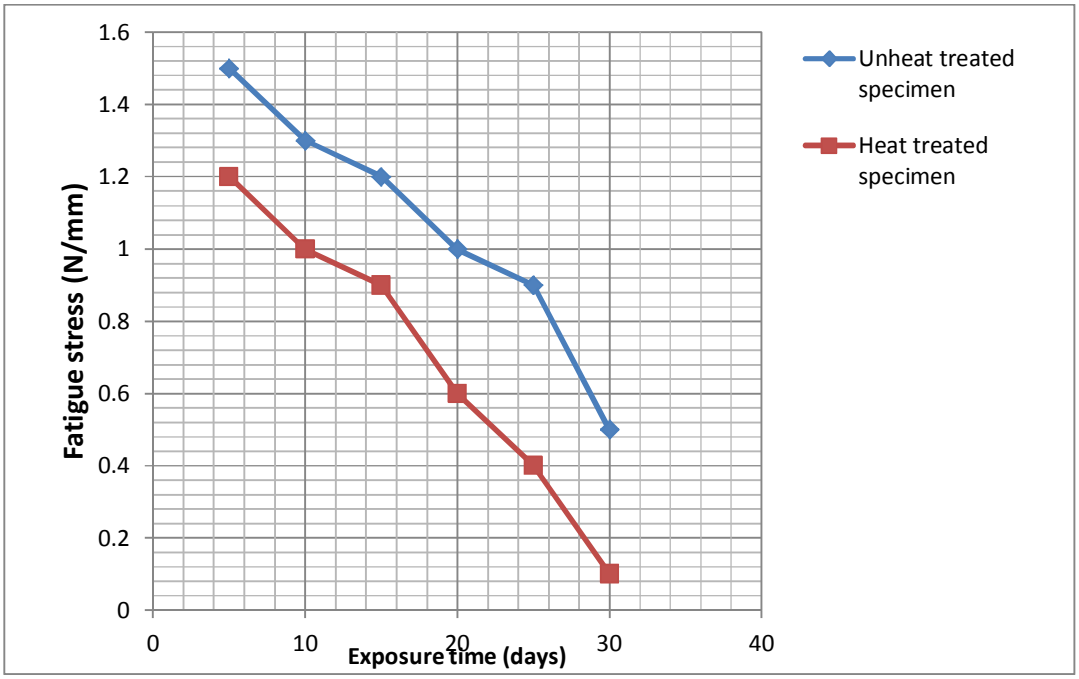


Fig 4.11 Variation of Fatigue stress with Exposure Time of Austenitic Stainless Steel in Seawater.

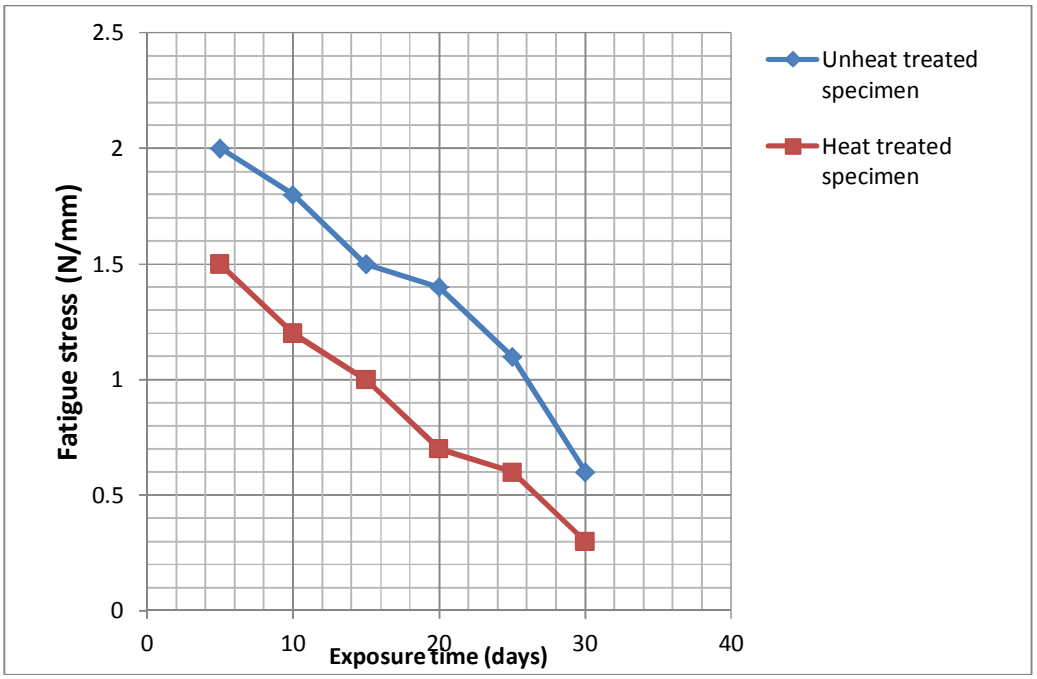


Fig 4.12 Variation of Fatigue stress with Exposure Time of Austenitic Stainless Steel in 0.5M HCl.

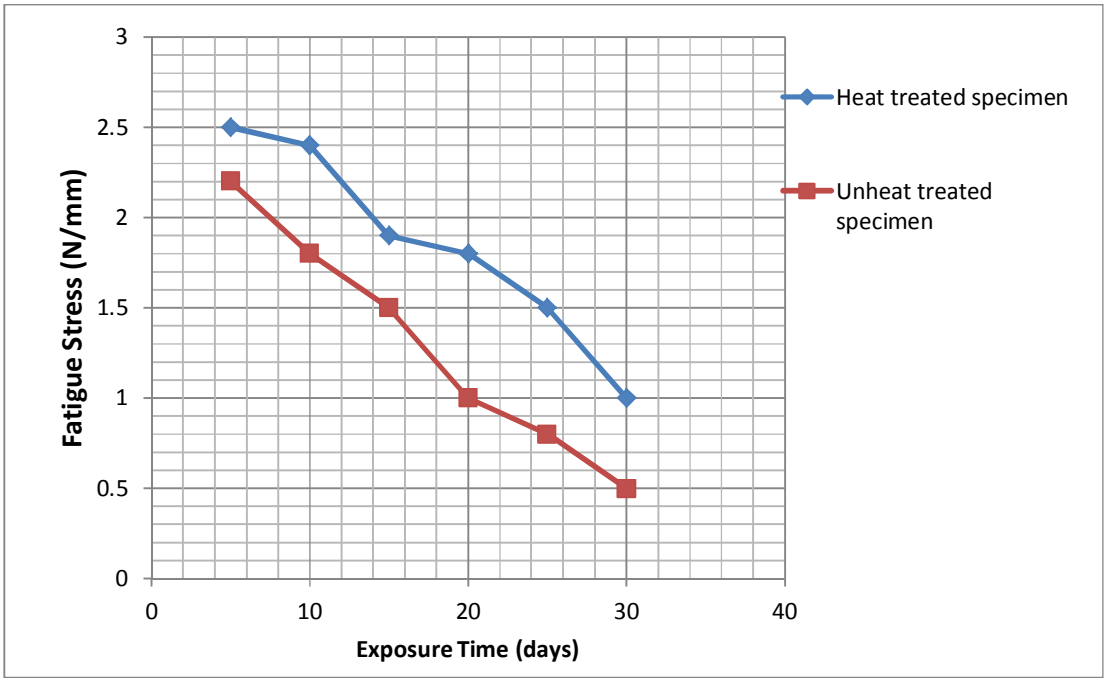


Fig 4.13 Variation of Fatigue Stress with Exposure Time of Austenitic Stainless Steel in Wet Steam.

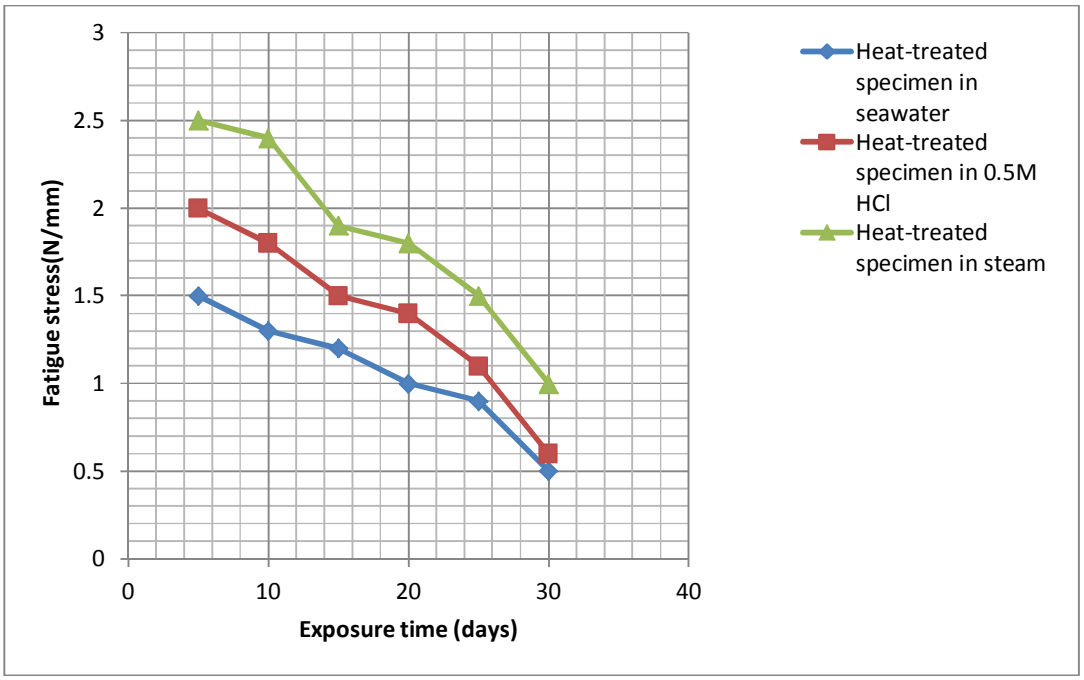


Fig 4.14 Variation of Fatigue Stress with Exposure Time of Heat treated Austenitic Stainless Steel in Seawater, 0.5M HCl and Wet Steam.

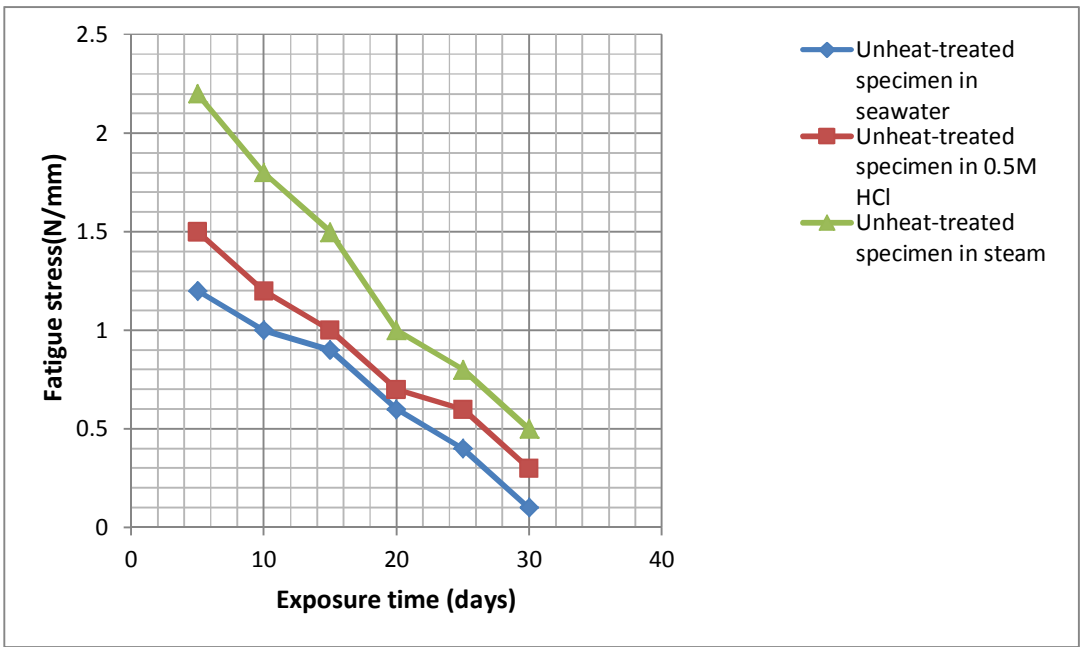


Fig 4.15 Variation of Fatigue Stress with Exposure Time of Unheat-treated Austenitic Stainless Steel in Seawater, 0.5M HCl and Wet Steam.

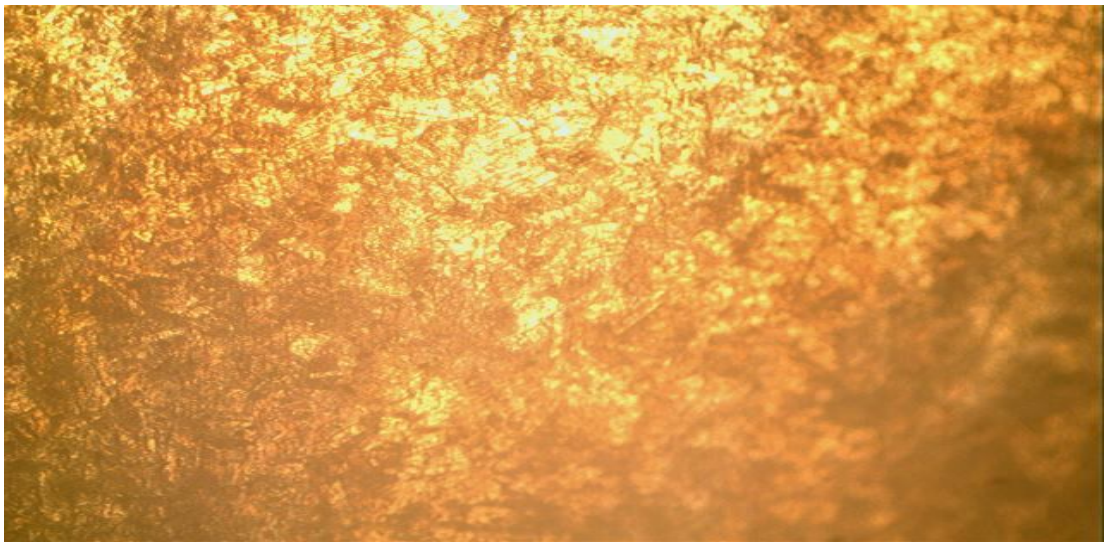


Plate 4.1a: Micrograph of the parent as-received austenitic steel showing some non-metallic inclusion (black), apha ferrite (white) in austenitic matrix (white background). 100x

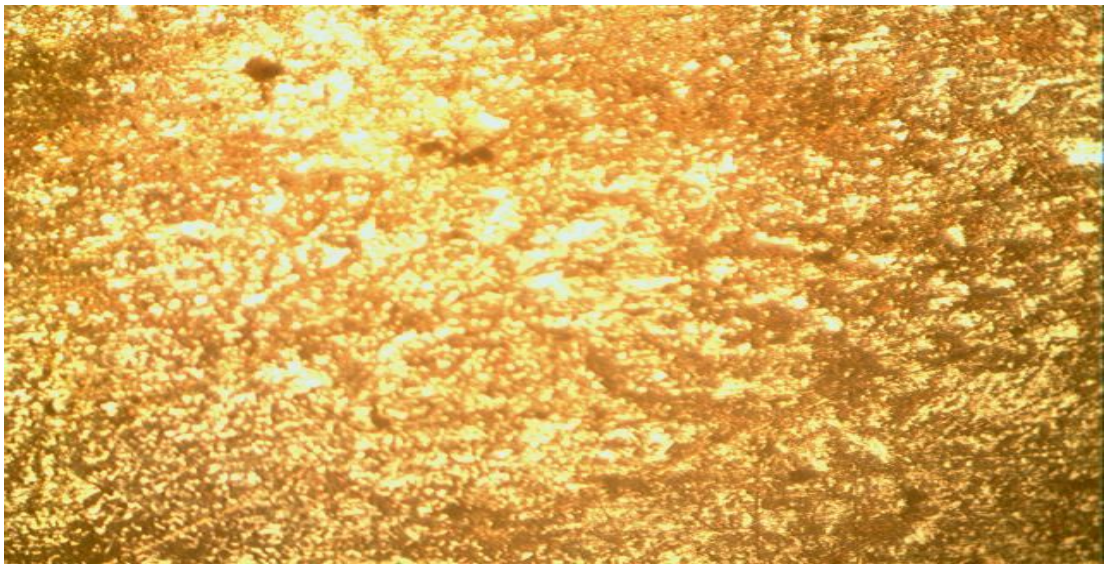


Plate 4.1b: Micrograph of the weldments of the as-received austenitic steel showing some carbide inclusion (black), delta ferrite (lacy-white) in austenitic matrix (white background). 100x

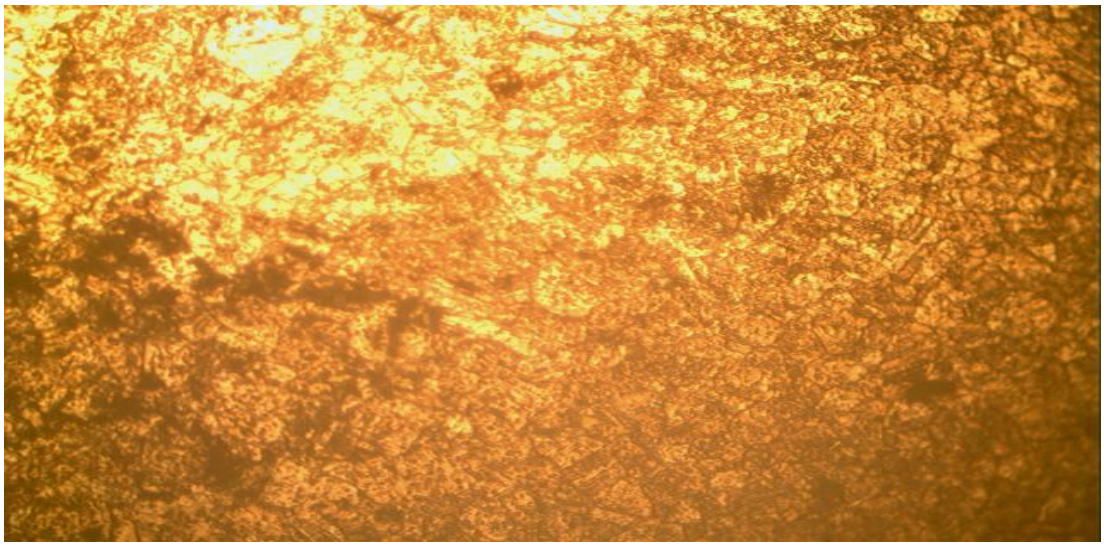


Plate 4.2a: Micrograph of parent annealed austenitic stainless steel showing some carbide inclusion ($M_{23}Cr_6$) precipitates (black), delta ferrite (white) in austenitic matrix (white background). 100x

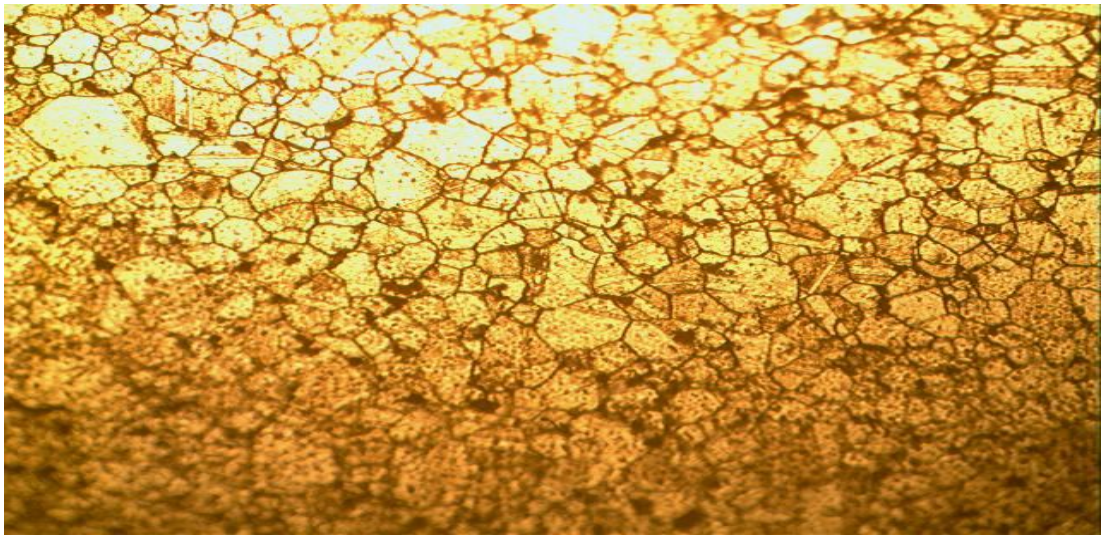


Plate 4.2b: Micrograph of the weldments of the annealed austenitic stainless steel showing high proportions of carbide ($M_{23}Cr_6$) precipitates along the grain boundaries(black), delta ferrite (acicular-white) in austenitic matrix (white background). 100x

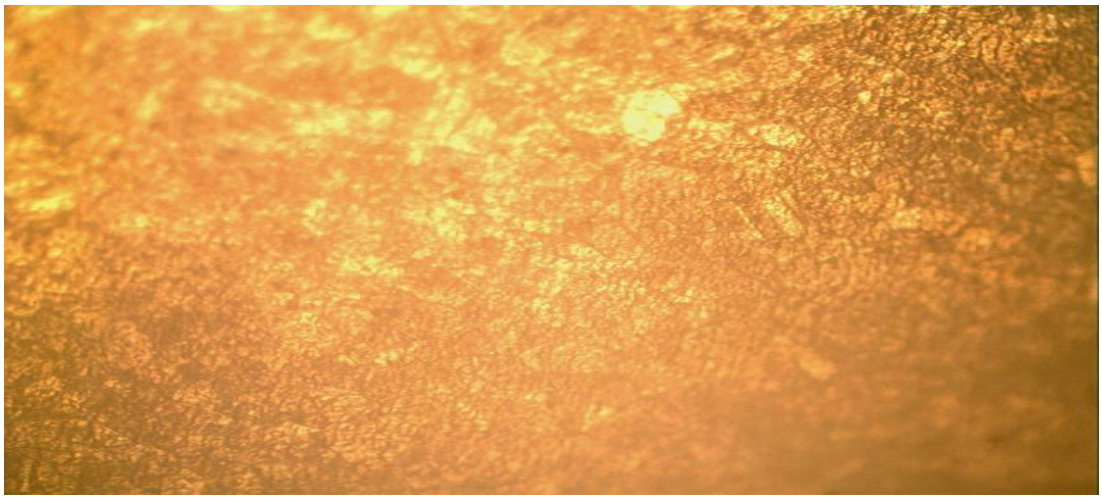


Plate 4.3a: Micrograph of the Acidic corroded parent austenitic stainless steel showing visible corrosion on the carbide ($M_{23}Cr_6$) precipitates and delta ferrite (acicular-white) but less in the austenitic matrix (white background). 100x

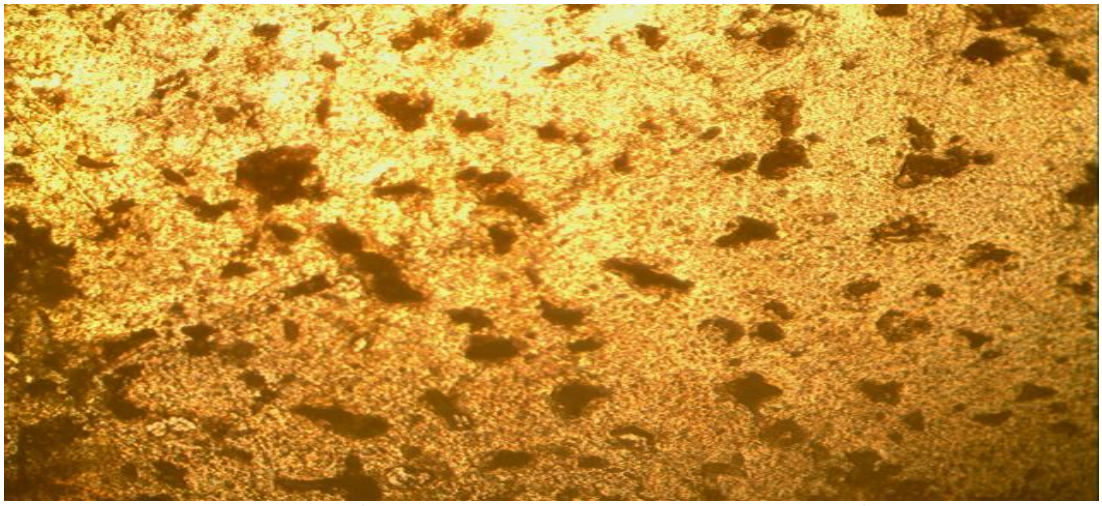


Plate 4.3b: Micrograph of the Acidic corroded weldments of the austenitic stainless steel showing high attack on the carbide ($M_{23}Cr_6$) sites (black) and sigma phase (black) but relatively less in the austenitic matrix (white background). 100x

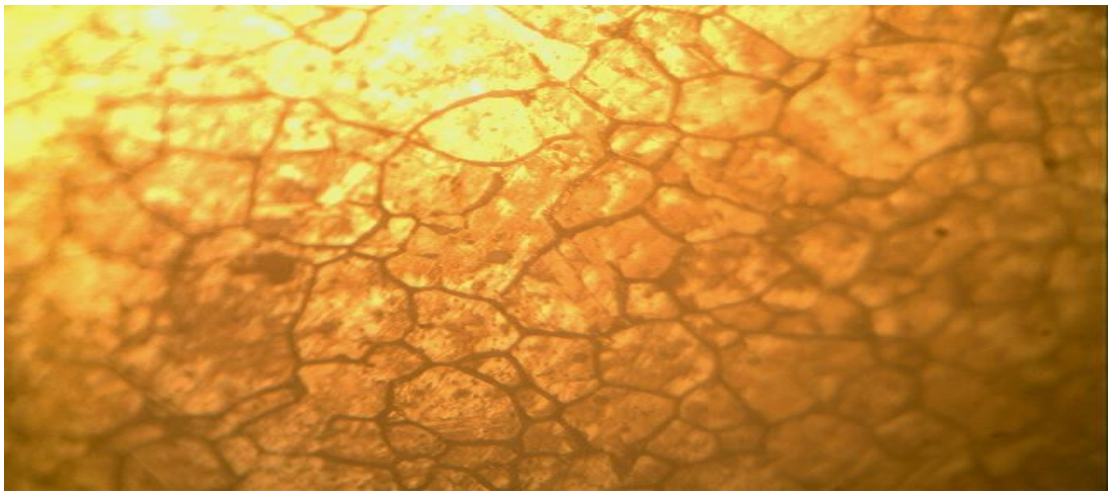


Plate 4.4a:Micrograph of the Sea water corroded parent austenitic stainless steel showing significant visible rate of attack on the carbide ($M_{23}Cr_{6)}$ sites (black) and delta ferrite (lacy-white) but probably no attack on the austenitic matrix (white background).100x

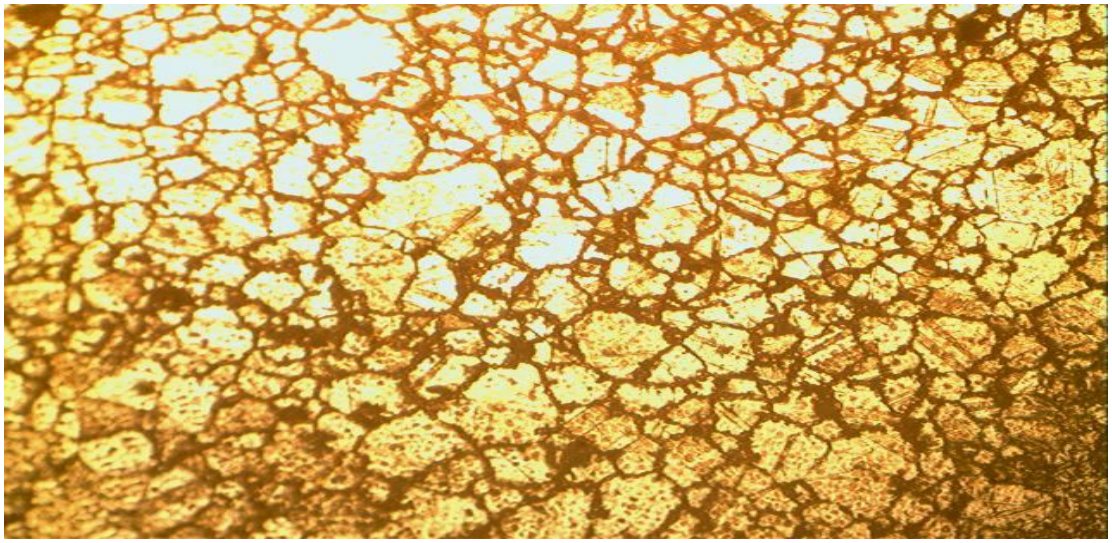


Plate 4.4b: Micrograph of the Sea water corroded weldments of the austenitic stainless steel showing significant attack on the carbide ($M_{23}Cr_6$) sites (black) and sigma phase (black) distributed along the austenitic matrix (white background). 100x

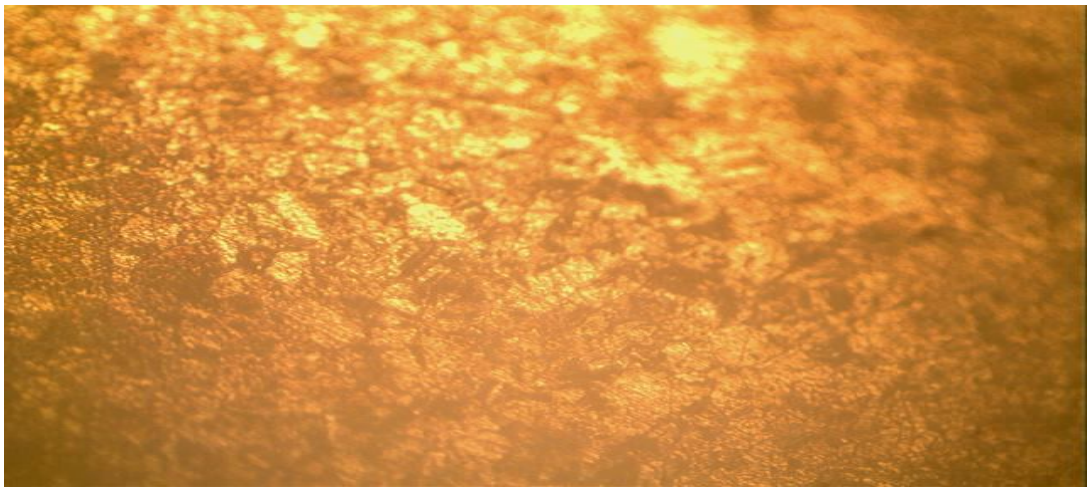


Plate 4.5a:Micrograph of the Steam corroded parent austenitic stainless steel showing visible high proportions of corroded carbide ($M_{23}Cr_6$) sites (black) and sigma phase (black) distributed along grains boundaries of the austenitic matrix (white background). 100x

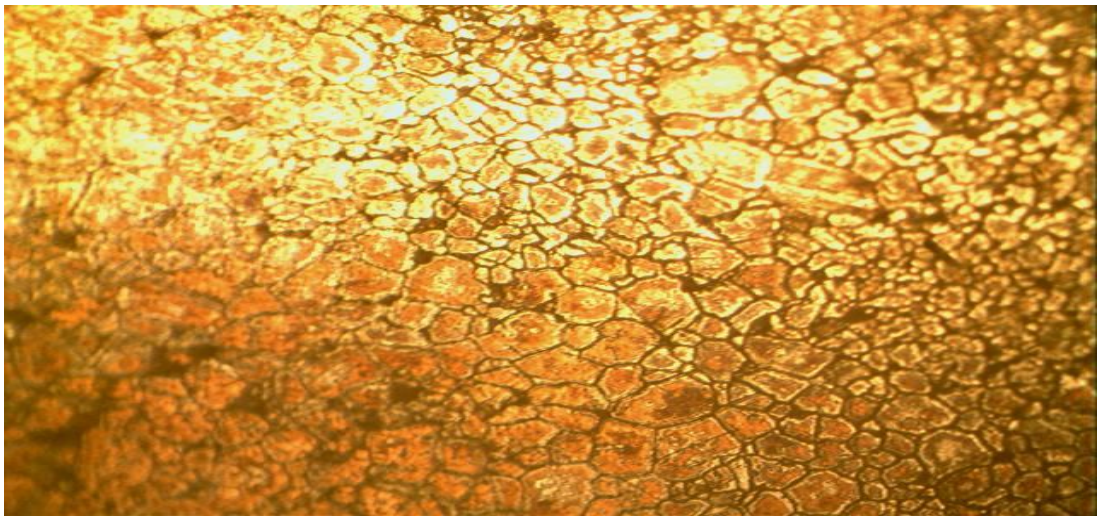


Plate 4.5b:Micrograph of the Steam corroded weldments of the austenitic stainless steel showing visible high proportions of corroded carbide ($M_{23}Cr_6$) sites (black) and sigma phase (black) distributed along the austenitic matrix (white background). 100x

4.3 DISCUSSIONS

4.3.1 Weight Loss of Austenitic Stainless Steel

Figures 4.1, 4.2 and 4.3 show variations of weight loss with exposure time of austenitic stainless steel in the different media used (seawater, 0.5M hydrochloric acid and wet steam). The graph trends show that weight loss in range of test increases with increase in exposure time. The unheat-treated samples have higher weight loss than the heat treated specimen for all the media. This is because the more time the samples spend in the corrosive media, the more they are corroded due to the formation of chromium carbide precipitates and the more weight that is lost. Also, the weight loss in seawater corroded specimen is the highest followed by acidic corroded specimen and steam corroded specimen, this is due to the presence of the chloride ions in seawater. This invariably reveals that stainless steel suffers greater corrosion in seawater due to the attack of chloride ions, this is in agreement with the findings of Aruleba, (2004), Omotade, (2005) and Afolabi, (2007).

4.3.2 Corrosion rate of Austenitic Stainless Steel

Figures 4.4, 4.5 and 4.6 show the corrosion rate with exposure time of austenitic stainless steel in the different media used.

The graphs show that corrosion rate of austenitic stainless steel decreases with increasing exposure time. Also, the unheat-treated specimen have higher corrosion rate than the heat treated specimen in all the media.

This is because as the iron on the surface corrodes, it leaves a chromium enriched surface behind, therefore the corrosion rate then would decrease as the chromium enriched surface begins to impact more passivity. It is evident from the plots that the corrosion rate of seawater is greater than that of the other media used throughout the exposure period, this collaborates the findings of Dillion, (2001) and Afolabi, (2007).

4.3.3 Fatigue Strength of Austenitic Stainless Steel

Figures 4.7, 4.8 and 4.9 show variations of the fatigue strength with exposure time for austenitic stainless steel in the different media used.

The graphs show thatfatigue strength decreases as the exposure time increases. Fatigue has to do with initiation and propagation of cracks and flaws at the weldment and there is formation of chromium carbide precipitates in the corrosive media which assumes the anodic and cathodic sites, because of the formation of cracks, which in turn reduces the fatigue strength. And the steam specimen has greater strength to failure followed by the specimen corroded in hydrochloric acid and lastly the specimen corroded in seawater.This revealed that stainless steel suffers greater corrosion in seawater due to the attack of chloride ions, this invariably reduces the fatigue strength, which is in agreement with the findings of Iliyasu, (2012), Omotade, (2005) and Afolabi, (2007).

4.3.4 Microstructure

Plates 4.1a, 4.2a, 4.3a, 4.4a and 4.5a show the microstructures of as-received specimen, heat treated specimen, seawater corroded specimen, acidic specimen and steam corroded specimen for the base material of austenitic stainless steel

Plates 4.1b, 4.2b, 4.3b, 4.4b and 4.5b show the microstructures of as-received specimen, heat treated specimen, seawater corroded specimen, acidic specimen andsteam corroded specimen for the weldments of austenitic stainless steel.

In plate 4.1a, the micrograph of the parent as-received austenitic steel shows some non-metallic inclusions (black), apha ferrite (white) in austenitic matrix (white background).

Plate 4.1b shows the micrograph of the weldments of the as-received austenitic steelshowing some carbide inclusions (black), delta ferrite (lacy-white) in austenitic matrix (white background). The lacy-ferrite microstructure emerges probably due to greater ferrite contents and/or a characteristic cooling time after the welding procedure.

In plate 4.2a,the micrograph of parent annealed austenitic stainless steel shows some carbide ($M_{23}Cr_6$), precipitate inclusions (black), delta ferrite (white) in austenitic matrix (white background). The grains are larger than those of the control sample probably because of the heat treatment which allowed time for grain growth and re-crystallization.

Plate 4.2b shows the micrograph of the weldments of the annealed austenitic stainless steel. It shows high proportions of carbide ($M_{23}Cr_6$) precipitates along the grain boundaries (black), delta ferrite (acicular-white) in austenitic matrix (white background).

In plate4.3a, the micrograph of the acidic corroded austenitic stainless steel shows visible corrosion on the carbide ($M_{23}Cr_6$) precipitates and delta ferrite (acicular-white) but less in the austenitic matrix (white background).

Plate 4.3b shows micrograph of the acid corroded weldment of the austenitic stainless steel revealing high attack on the carbide ($M_{23}Cr_6$) sites (black) and sigma phase (black) but relatively less in the austenitic matrix (white background). The grain sizes are larger than those of the as-received specimen. The grain boundaries are also clearly defined. This is probably the effect of the hydrochloric acid on the surface of the material.

In plate 4.4a, the micrograph of the seawater corroded austenitic stainless steel shows significant visible rate of attack on the carbide($M_{23}Cr_6$) sites (black) and delta ferrite (lacy-white) but little or no attack on the austenitic matrix (white background).

In plate 4.4b, the micrograph of the seawater corroded weldment of the austenitic stainless steel shows significant attack on the carbide ($M_{23}Cr_6$) sites (black) and sigma phase (black) distributed along the austenitic matrix (white background). Seawater has a rigorous effect on the cooling rate. This is

probably the reason why there are fewer ferrite regions in the microstructure. And generally, the corrosion rate is highest in seawater.

In plate 4.5a, the micrograph of the wet steam corroded austenitic stainless steel shows visible high proportions of corroded carbide ($M_{23}Cr_6$) sites (black) and sigma phase (black) distributed along grain boundaries of the austenitic matrix (white background).

In plate 4.5b, the micrograph of the wet steam corroded weldments of the austenitic stainless steel shows visible high proportions of corroded carbide ($M_{23}Cr_6$) sites (black) and sigma phase (black) distributed along the austenitic matrix (white background), where the M_{23} represents the carbide complexes that are formed. This research is in agreement with earlier researches by Rajasekhar, et al (1997), Zhang, et al, (2001) and Lakshminarayanan, et al (2009),

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1 CONCLUSIONS

From the results and discussion in this work, the following conclusions can be drawn:

1.The weight loss of austenitic stainless steel increases with increase in exposure time and seawater specimen has the highest weight loss owing to the presence of chloride ions.The corrosion rate of austenitic stainless steel has been found to decrease with increase in the exposure time and seawater specimen have the highest corrosion rate followed by acidic specimen and then wet steam specimen.

2.The fatigue strengthwas found to decrease with an increase in exposure time and steam corroded specimen has greater resistance to fatigue failure than other media used.

3.Post welding heat treatment of annealingwhich is grain refinement was found to increase the mechanical properties of the austenitic stainless steel especially tensile strength and also reduced transformation and thermal stresses.

4.The microstructure of the parent as-received austenitic stainless steel reveals alpha ferrite (white) in the austenitic matrix while the weldment reveals delta ferrite (lacy-white) in the austenitic matrix. Also, the microstructure of the media used gives predominantly carbide (M_{23} Cr_6)

precipitates in the austenitic matrix while the weldment reveals high proportions of carbide precipitates along the grain boundaries.

5.Finally, austenitic stainless steel, though possessing a high degree of resistance to corrosion, still suffers corrosion to some extent when used in corrosive environment and even suffers double when there is a tendency to fatigue in corrosive environment.

5.2 RECOMMENDATIONS

The following recommendations can be made:

i. Since, not much work has yet been done on corrosion fatigue; it is recommended that more attention be shifted to work on corrosion fatigue of engineering materials as fatigue has proven to be the largest cause of failure of engineering structures.

ii. The investigation may be carried out at higher concentrations of the acid to see its effect on the stainless steel.

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APPENDIX A

TABLE A1: Austenitic Stainless Steel in Seawater

Time of Immersion (days)	Initial Weight (g)	Final Weight (g)		Weight loss (mg)		CPR (mm/yr)x10^{-6}	
		Heat treated	Unheat treated	Heat treated	Unheat treated	Heat treated	Unheat treated
5	144.6515	144.6499	144.6496	1.6	1.9	1.54	1.83
10	145.3782	145.3760	145.3757	2.2	2.5	1.06	1.21
15	146.4812	146.4786	146.4784	2.6	2.8	0.84	0.89
20	144.0620	144.0590	144.0587	3.0	3.3	0.72	0.76
25	144.6620	144.6586	144.6583	3.4	3.7	0.66	0.71
30	144.6882	144.6842	144.6837	4.0	4.5	0.64	0.72

TABLE A2: Austenitic Stainless Steel in 0.5M HCl

Time of Immersion (days)	Initial Weight (g)	Final Weight (g)		Weight loss (mg)		CPR (mm/yr)x 10^{-6}	
		Heat treated	Unheat treated	Heat treated	Unheat treated	Heat treated	Unheat treated
5	144.4878	144.4865	144.4863	1.3	1.5	1.25	1.44
10	145.1132	145.1116	145.1113	1.6	1.9	0.77	0.92
15	144.9120	144.9099	144.9096	2.1	2.4	0.67	0.77
20	144.8972	144.8947	144.8943	2.5	2.9	0.60	0.69
25	144.3292	144.3262	144.3259	3.0	3.3	0.58	0.64
30	142.6882	144.6846	142.6843	3.5	3.9	0.56	0.63

TABLE A3: Austenitic Stainless Steel in Wet Steam

Time of Immersion (days)	Initial Weight (g)	Final Weight (g)		Weight loss (mg)		CPR (mm/yr)x 10^{-6}	
		Heat treated	Unheat treated	Heat treated	Unheat treated	Heat treated	Unheat treated
5	145.7825	145.7814	145.7813	1.1	1.2	1.06	1.16
10	146.6805	146.6791	146.6789	1.4	1.6	0.67	0.77
15	144.2469	144.2452	144.2450	1.7	1.9	0.55	0.61
20	142.8146	142.8125	142.8122	2.1	2.4	0.51	0.58
25	146.3098	146.3073	146.3070	2.5	2.8	0.46	0.54
30	143.9804	143.9775	143.9771	2.9	3.3	0.45	0.53

TABLE A4: Fatigue stress of Austenitic Stainless Steel in Seawater.

Time of Immersion (days)	Fatigue stress $x10^5$ (N/mm)	
	Heat treated	Unheat treated
5	1.5	1.2
10	1.3	1.0
15	1.2	0.9
20	1.0	0.6
25	0.9	0.4
30	0.5	0.1

TABLE A5: Fatigue stress of Austenitic Stainless Steel in 0.5M HCl.

Time of Immersion (days)	Fatigue stress $x10^5$ (N/mm)	
	Heat treated	Unheat treated
5	2.0	1.5
10	1.8	1.2
15	1.5	1.0
20	1.4	0.7
25	1.1	0.6
30	0.6	0.3

TABLE A6: Fatigue stress of Austenitic Stainless Steel in Wet steam.

Time of Immersion (days)	Fatigue stress $x10^5$ (N/mm)	
	Heat treated	Unheat treated
5	2.5	2.2
10	2.4	1.8
15	1.9	1.5
20	1.8	1.0
25	1.5	0.8
30	1.0	0.5