**CHAPTER ONE**

**INTRODUCTION**

**1.1 Background of the study**

Mild steel is a type of carbon steel with low carbon content, it is also called low carbon steel. It is the most common type of steel because its price is relatively low while it provides material properties that are acceptable for many applications. It ranges generally between 0.05% -0.25% by weight. It is not an alloy steel therefore doesn’t contain large number of other elements besides mild steel. The common method on how mid steel is formed is by the combination of iron ore and coal melted in a blast furnace, then solidified in a rectangular shape. Hot rolling or cold drawing will then be used to bring mild steel to its desired shape. Its common applications are found in structural steel, signs, cars, furniture and decorations, wire, fencing, nails amongst others.

Technically, mild steel contains an alloy majorly consisting iron at a greater percentage than other constituents like manganese, phosphorus etc. it is fairly malleable and ductile. It can be shaped by hammering and pressed while hot but cannot be hardened by heat treatment (Talbot and Talbot 1998). However, in this study a common mild steel specimen having carbon content 0.05%- 0.30% was chosen to undergo some heat treatment processes in order to compare an improvement in microstructure, properties and ultimately its performance in service. This commercial mild steel was chosen because of its well-known properties and its wide range of applications.

For this study, two basic post-production processing were employed. Generally, heat treatment is a series of altering operations involving the preheating below lower critical temperature of the metal and cooling it in an appropriate media while still in its solid state (T.S Blessing, 2019). This is done basically to change the metals mechanical properties and sometimes its chemical properties so that it becomes more useful and workable. By heat treating a metal it can be stronger, harder or more resistant to impact on the other hand it can make a metal softer and more ductile (J.R Davis, 2001).

Annealing involves heating a material above its recrystallization temperature, maintaining the suitable temperature for an amount of time and then cooling it slowly. Annealing provides solution to stress cracking and intergranular disorder. In normalizing process, the process of heating the steel to about 40 degrees Celsius above its upper critical temperature limit held at this temperature for some time and then cooled in air.

Hydrochloric acid is one of the most difficult and common acids to handle from the standpoint of corrosion and construction materials therefore, extreme care is required in the selection of materials to handle the acid even in its least concentrated state. The acid is corrosive to most metals and alloys and the rate of corrosion on the metals will be dependent on the concentration of the acid that is, the higher the concentration of the acid the faster corrosion occurs on the metal.

**1.2 Aim and Objective**

The aim of this study is to investigate the corrosion behavior of annealed and normalized mild steel in HCL as well as the effects or change in its micro structure before and after immersion in HCL at different concentrations of the acid.

**CHAPTER TWO**

**LITERATURE REVIEW**

**2.1 Mild steel and its components**

Mild steel is the most widely used steel which is not brittle and relatively cheap, it possesses enough strength but not readily tempered or hardened. It is made up of carbon and other non-metals. Some of its properties are:

1. The modulus of elasticity calculated for the industry grade mild steel is 210,000 Mpa. It has an average density of about 7860 kg/m3.
2. Mild steel is a great conductor of electricity. So it can be used easily in the welding process.
3. Because of its malleability, mild steel can be used for constructing pipelines and other construction materials. Even domestic cook wares are made of mild steel. It is ductile and not brittle but hard.
4. Mild steel can be easily magnetized because of its ferromagnetic properties. So electrical devices can be made of mild steel.
5. Mild steel is very much suitable as structural steel. Different automobile manufacturers also use mild steel for making the body and parts of the vehicle.
6. Mild steel can be easily machined in the lathe, shaper, drilling or milling machine. Its hardness can be increased by the application of carbon.
7. Mild steel is very much prone to rust because it has high amount of carbon. When rust free products are needed people prefer stainless steel over mild steel.

**2.2 Heat treatment**

Heat treating (or heat treatment) is a group of [industrial](https://en.wikipedia.org/wiki/Industrial_process) and [metal working processes](https://en.wikipedia.org/wiki/Metalworking) used to alter the [physical](https://en.wikipedia.org/wiki/Physical_property), and sometimes [chemical](https://en.wikipedia.org/wiki/Chemical_property), properties of a material. The most common application is [metallurgical](https://en.wikipedia.org/wiki/Metallurgy). Heat treatments are also used in the manufacture of many other materials, such as [glass](https://en.wikipedia.org/wiki/Glass). Heat treatment involves the use of heating or chilling, normally to extreme temperatures, to achieve the desired result such as hardening or softening of a material. Heat treatment techniques include [annealing](https://en.wikipedia.org/wiki/Annealing_%28metallurgy%29), [case hardening](https://en.wikipedia.org/wiki/Case_hardening), [precipitation strengthening](https://en.wikipedia.org/wiki/Precipitation_strengthening), [tempering](https://en.wikipedia.org/wiki/Tempering_%28metallurgy%29), [carburizing](https://en.wikipedia.org/wiki/Carburizing), normalizing and [quenching](https://en.wikipedia.org/wiki/Quench). Although the term *heat treatment* applies only to processes where the heating and cooling are done for the specific purpose of altering properties intentionally, heating and cooling often occur incidentally during other manufacturing processes such as hot forming or welding.

Metallic materials consist of a [microstructure](https://en.wikipedia.org/wiki/Microstructure) of small [crystals](https://en.wikipedia.org/wiki/Crystal) called "grains" or [crystallites](https://en.wikipedia.org/wiki/Crystallite). The nature of the grains (i.e. grain size and composition) is one of the most effective factors that can determine the overall mechanical behavior of the metal. Heat treatment provides an efficient way to manipulate the properties of the metal by controlling the rate of [diffusion](https://en.wikipedia.org/wiki/Diffusion) and the rate of cooling within the microstructure. Heat treating is often used to alter the mechanical properties of a metallic [alloy](https://en.wikipedia.org/wiki/Alloy), manipulating properties such as the [hardness](https://en.wikipedia.org/wiki/Hardness), [strength](https://en.wikipedia.org/wiki/Strength_of_materials), [toughness](https://en.wikipedia.org/wiki/Toughness), [ductility](https://en.wikipedia.org/wiki/Ductility), and [elasticity](https://en.wikipedia.org/wiki/Elasticity_%28physics%29).

There are two mechanisms that may change an alloy's properties during heat treatment: the formation of [martensite](https://en.wikipedia.org/wiki/Martensite) causes the crystals to [deform](https://en.wikipedia.org/wiki/Deformation_%28engineering%29) intrinsically, and the diffusion mechanism causes changes in the homogeneity of the [alloy](https://en.wikipedia.org/wiki/Alloy).

The crystal structure consists of atoms that are grouped in a very specific arrangement, called a lattice. In most elements, this order will rearrange itself, depending on conditions like temperature and pressure. This rearrangement, called [allotropy](https://en.wikipedia.org/wiki/Allotropy) or polymorphism, may occur several times, at many different temperatures for a particular metal. In alloys, this rearrangement may cause an element that will not normally [dissolve](https://en.wikipedia.org/wiki/Solvation) into the base metal to suddenly become [soluble](https://en.wikipedia.org/wiki/Solubility), while a reversal of the allotropy will make the elements either partially or completely insoluble.

When in the soluble state, the process of diffusion causes the atoms of the dissolved element to spread out, attempting to form a homogenous distribution within the crystals of the base metal. If the alloy is cooled to an insoluble state, the atoms of the dissolved constituents (solutes) may migrate out of the solution. This type of diffusion, called [precipitation](https://en.wikipedia.org/wiki/Precipitation_%28chemistry%29), leads to [nucleation](https://en.wikipedia.org/wiki/Nucleation), where the migrating atoms group together at the grain-boundaries. This forms a microstructure generally consisting of two or more distinct [phases](https://en.wikipedia.org/wiki/Phase_%28matter%29). For instance, steel that has been heated above the [austenizing](https://en.wikipedia.org/wiki/Austenizing) temperature (red to orange-hot, or around 1,500 °F (820 °C) to 1,600 °F (870 °C) depending on carbon content), and then cooled slowly, forms a laminated structure composed of alternating layers of [ferrite](https://en.wikipedia.org/wiki/Allotropes_of_iron) and [cementite](https://en.wikipedia.org/wiki/Cementite), becoming soft [pearlite](https://en.wikipedia.org/wiki/Pearlite). After heating the steel to the [austenite](https://en.wikipedia.org/wiki/Austenite) phase and then quenching it in water, the microstructure will be in the martensitic phase. This is due to the fact that the steel will change from the austenite phase to the martensite phase after quenching. Some pearlite or ferrite may be present if the quench did not rapidly cool off all the steel.

Many metals and non-metals exhibit a [martensite](https://en.wikipedia.org/wiki/Martensite) transformation when cooled quickly(with external media like oil, polymer, water etc.). When a metal is cooled very quickly, the insoluble atoms may not be able to migrate out of the solution in time. This is called a "diffusion less." When the crystal matrix changes to its low temperature arrangement, the atoms of the solute become trapped within the lattice. The trapped atoms prevent the crystal matrix from completely changing into its low temperature allotrope, creating shearing stresses within the lattice. When some alloys are cooled quickly, such as steel, the martensite transformation hardens the metal, while in others, like aluminum, the alloy becomes softer.

Complex heat treating schedules, or "cycles," are often devised by [metallurgists](https://en.wikipedia.org/wiki/Metallurgist) to optimize an alloy's mechanical properties. In the [aerospace](https://en.wikipedia.org/wiki/Aerospace) industry, a [super alloy](https://en.wikipedia.org/wiki/Superalloy) may undergo five or more different heat treating operations to develop the desired properties. This can lead to quality problems depending on the accuracy of the furnace's temperature controls and timer. These operations can usually be divided into several basic techniques.

**2.2.1 Annealing**

Annealing consists of heating a metal to a specific temperature and then cooling at a rate that will produce a refined [microstructure](https://en.wikipedia.org/wiki/Microstructure), either fully or partially separating the constituents. The rate of cooling is generally slow. Annealing is most often used to soften a metal for cold working, to improve machinability, or to enhance properties like [electrical conductivity](https://en.wikipedia.org/wiki/Electrical_conductivity).

In ferrous alloys, annealing is usually accomplished by heating the metal beyond the upper critical temperature and then cooling very slowly, resulting in the formation of pearlite. In both pure metals and many alloys that cannot be heat treated, annealing is used to remove the hardness caused by cold working. The metal is heated to a temperature where [recrystallization](https://en.wikipedia.org/wiki/Recrystallization_%28metallurgy%29) can occur, thereby repairing the defects caused by plastic deformation. In these metals, the rate of cooling will usually have little effect. Most non-ferrous alloys that are heat-treatable are also annealed to relieve the hardness of cold working. These may be slowly cooled to allow full precipitation of the constituents and produce a refined microstructure.

Ferrous alloys are usually either "full annealed" or "process annealed." Full annealing requires very slow cooling rates, in order to form coarse pearlite. In process annealing, the cooling rate may be faster; up to, and including normalizing. The main goal of process annealing is to produce a uniform microstructure. Non-ferrous alloys are often subjected to a variety of annealing techniques, including "recrystallization annealing," "partial annealing," "full annealing," and "final annealing." Not all annealing techniques involve recrystallization, such as stress relieving.[[21]](https://en.wikipedia.org/wiki/Heat_treating#cite_note-Dossett,_2006,_2-6-21)

**2.2.2 Normalizing**

Normalizing is a technique used to provide uniformity in grain size and composition (equating) throughout an alloy. The term is often used for ferrous alloys that have been [austenized](https://en.wikipedia.org/wiki/Austenite#Austenitization) and then cooled in open air. Normalizing not only produces pearlite, but also martensite and sometimes [bainite](https://en.wikipedia.org/wiki/Bainite), which gives harder and stronger steel, but with less ductility for the same composition than full annealing.

In normalizing process, the process of heating the steel to about 40 degrees Celsius above its upper critical temperature limit held at this temperature for some time and then cooled in air.

**2.3 Spark testing**

Spark testing is a method of determining the general [classification](https://en.wikipedia.org/wiki/Steel_grades) of [ferrous](https://en.wikipedia.org/wiki/Ferrous) materials. It normally entails taking a piece of metal, usually scrap, and applying it to a [grinding wheel](https://en.wikipedia.org/wiki/Grinding_wheel) in order to observe the sparks emitted. These sparks can be compared to a chart or to sparks from a known test sample to determine the classification. Spark testing also can be used to sort ferrous materials, establishing the difference from one another by noting whether the spark is the same or different.

Spark testing is used because it is quick, easy, and inexpensive. Moreover, test samples do not have to be prepared in any way, so, often, a piece of scrap is used. The main disadvantage to spark testing is its inability to identify a material positively; if positive identification is required, [chemical analysis](https://en.wikipedia.org/wiki/Chemical_analysis) must be used. The spark comparison method also damages the material being tested, at least slightly. Spark testing most often is used in [tool rooms](https://en.wikipedia.org/wiki/Tool_room), [machine shops](https://en.wikipedia.org/wiki/Machine_shop), [heat treating](https://en.wikipedia.org/wiki/Heat_treating) shops, and [foundries](https://en.wikipedia.org/wiki/Foundry).

A [bench grinder](https://en.wikipedia.org/wiki/Bench_grinder) is usually used to create the sparks, but sometimes this is not convenient, so a portable [grinder](https://en.wikipedia.org/wiki/Grinding_machine) is used. In either case, the grinding wheel must have adequate surface velocity, at least 23 m/s (4500 [surface feet per minute](https://en.wikipedia.org/wiki/Surface_feet_per_minute) (sfpm)), but should be between 38 and 58 m/s (7500–11,500 sfpm). The wheel should be coarse and hard, therefore [aluminum oxide](https://en.wikipedia.org/wiki/Aluminium_oxide) or [carborundum](https://en.wikipedia.org/wiki/Silicon_carbide) often are employed. The test area should be in an area where there is no bright light shining directly into the observer's eyes. Moreover, the grinding wheel and surrounding area should be dark so that the sparks can be observed clearly. The test sample is then touched lightly to the grinding wheel to produce the sparks.

The important spark characteristics are color, volume, nature of the spark, and length. Note that the length is dependent on the amount of pressure applied to the grinding wheel, so this can be a poor comparison tool if the pressure is not exactly the same for the samples. Also, the grinding wheel must be [dressed](https://en.wikipedia.org/wiki/Grinding_dresser) frequently to remove metallic build-up.

Compressed air method

Another less common method for creating sparks is heating up the sample to [red heat](https://en.wikipedia.org/wiki/Red_heat) and then applying compressed air to the sample. The compressed air supplies enough oxygen to ignite the sample and give off sparks. This method is more accurate than using a grinder because it will always give off sparks of the same length for the same sample. The compressed air applies in essence the same "pressure" each time. This makes observations of the spark length a much more reliable characteristic for comparison.

Automated spark testing

Automated spark testing has been developed to remove the reliance upon operator skill and experience, thereby increasing reliability. The system relies upon [spectroscopy](https://en.wikipedia.org/wiki/Spectroscopy), [spectrometry](https://en.wikipedia.org/wiki/Mass_spectrometry), and other methods to "observe" the spark pattern. It has been found that this system can determine the difference between two materials that give off sparks that are indistinguishable to the human eye.

Spark characteristics



(A) High-carbon steel
(B) Manganese steel
(C) Tungsten steel
(D) Molybdenum steel



(A) Wrought iron
(B) Mild steel
(C) Steel with 0.5 to 0.85% carbon
(D) High-carbon tool steel
(E) High-speed steel
(F) Manganese steel
(G) [Mushet steel](https://en.wikipedia.org/wiki/Mushet_steel)
(H) Special magnet steel

Wrought iron

[Wrought iron](https://en.wikipedia.org/wiki/Wrought_iron) sparks flow out in straight lines. The tails of the sparks widen out near the end, similar to a [leaf](https://en.wikipedia.org/wiki/Leaf).

Mild steel

[Mild steel](https://en.wikipedia.org/wiki/Mild_steel) sparks are similar to wrought iron's, except they will have tiny forks and their lengths will vary more. The sparks will be white in color.

Medium-carbon steel

This steel has more forking than mild steel and a wide variety of spark lengths, with more near the grinding wheel.

High-carbon steel

High-carbon steel has a bushy spark pattern (lots of forking) that starts at the grinding wheel. The sparks are not as bright as the medium-carbon steel ones.

Manganese steel

[Manganese](https://en.wikipedia.org/wiki/Manganese) steel has medium length sparks that fork twice before ending.

High-speed steel

[High-speed steel](https://en.wikipedia.org/wiki/High-speed_steel) has a faint red spark that sparks at the tip

**2.4 SEM (Scanning Electron Microscope) analysis**

A scanning electron microscope (SEM) is a type of [electron microscope](https://en.wikipedia.org/wiki/Electron_microscope) that produces images of a sample by scanning the surface with a focused beam of [electrons](https://en.wikipedia.org/wiki/Electron). The electrons interact with [atoms](https://en.wikipedia.org/wiki/Atom) in the sample, producing various signals that contain information about the surface [topography](https://en.wikipedia.org/wiki/Topography) and composition of the sample. The electron beam is scanned in a [raster scan](https://en.wikipedia.org/wiki/Raster_scan) pattern, and the position of the beam is combined with the intensity of the detected signal to produce an image. In the most common SEM mode, [secondary electrons](https://en.wikipedia.org/wiki/Secondary_electrons) emitted by atoms excited by the electron beam are detected using a secondary electron detector ([Everhart-Thorley detector](https://en.wikipedia.org/wiki/Everhart-Thornley_detector)). The number of secondary electrons that can be detected, and thus the signal intensity, depends, among other things, on specimen topography. SEM can achieve resolution better than 1 nanometer.

Specimens are observed in high vacuum in conventional SEM, or in low vacuum or wet conditions in variable pressure or environmental SEM, and at a wide range of cryogenic or elevated temperatures with specialized instruments

**CHAPTER THREE**

**METHODOLOGY**

**3.1 Spark testing**



Spark testing of mild steel

A 20mm mild steel specimen was prepared for the spark test The spark test is made by holding a sample of the material against an abrasive wheel. By visually inspect­ing the spark stream, an experienced metalworker can identify the metals with considerable accuracy. This test is fast, economical, convenient, and easily accom­plished, and there is no requirement for special equip­ment. We can use this test for identifying metal salvaged from scrap. Identification of scrap is particularly impor­tant when selecting material for cast iron or cast steel and subjected to heat treatment processes (annealing and normalizing) in a furnace at a temperature below 300 degrees Celsius below its recrystallization temperature and then allowed to cool slowly in the furnace, in case of annealing and open air in the case of normalizing

**3.2 Corrosion test in hydrochloric acid (HCL)**

Mild steel has been used extensively in the oil, gas and chemical industries because of its outstanding mechanical properties. The use of steel is also one of the effective strategies to maximize profit and reduce cost as compared to expensive corrosion resistant alloys ([Abdel-Rehim et al., 2011](https://www.sciencedirect.com/science/article/pii/S1878535218301734%22%20%5Cl%20%22b0005), [Laamari et al., 2016](https://www.sciencedirect.com/science/article/pii/S1878535218301734%22%20%5Cl%20%22b0210), [Umoren et al., 2016](https://www.sciencedirect.com/science/article/pii/S1878535218301734%22%20%5Cl%20%22b0390)). However, this alloy still suffers from corrosion when it comes in contact with corrosive environments, especially those containing [chloride ions](https://www.sciencedirect.com/topics/chemistry/chloride) ([Dwivedi et al., 2017](https://www.sciencedirect.com/science/article/pii/S1878535218301734%22%20%5Cl%20%22b0095)). In many industrial applications related to oil and gas processing such as pipeline cleaning, pipeline/acid descaling and oil well acidizing, the use of mineral acids (usually hydrochloric acid) is still an effective method for improving productivity ([Lgaz et al., 2017a](https://www.sciencedirect.com/science/article/pii/S1878535218301734%22%20%5Cl%20%22b0220)). The heat treated mild will be immersed in five different concentrations of the acid and will be monitored for a period of forty days.