

# **CHAPTER 1**

## **INTRODUCTION**

### **1.1 Background and Problem Statement**

Corrosion is a common problem faced by the industry nowadays. Several factors may lead to this severe issue such as corrosion of stainless steel. Corrosion on the stainless steel usually cause lot of money that need to put in maintaining the equipments such as boiler and heater tube. Visual examinations on the sample will show us the causes that make the failure on the heater tube.

This heater tube consists of stainless steel, ceramic, and carbon steel layers. During power generation process, carbon steel inside the tube heater was corroded by unknown caused. However, a few possible causes have been assumed such as a crack occurrence at the stainless steel surface and over heating of the tube heater. At the end of this project, we can identify the real cause of the failure of this tube heater.

### **1.2 Objectives**

Basically, this project has two primary objectives:

- To study and identify what elements that causes the corrosion of the water tube heater.
- To be able to analyze the results of experiments done on the corroded water tube heater

### **1.3 Scope of Study**

- Study the type of corrosion that caused the stainless steel to corrode
- Study the methodology used in sample testing
- Study of failure analysis in steel

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Water-Formed and Steam-Formed Deposits**

##### General description

The term deposits refer to materials that originate elsewhere and are conveyed to a deposition site. Deposits cannot be defined as corrosion products formed in a place, although corrosion products form elsewhere and then deposited qualify. Oxides formed from boiler/heater metal are not deposits unless they have been moved from their origination sites. This distinction is fundamental.

Heater deposits come from four sources: water borne materials, treatment chemicals, corrosion products, and contaminants. Deposits from these sources may interact to increase deposition rates, to produce a more tenacious layer, and to serve as nucleation sites for deposit formation. Such species include metal oxides, copper, phosphates, carbonates, silicates, sulfates, and contaminants, as well as variety of organic and inorganic compounds.

One deposition process involves the concentration of soluble and insoluble species in a thin bordering the metal surface during steam bubble formation (Figure 2.1). Material segregates at the steam/water interface, moves along the interface, and is deposited at the bubble base the bubble grows. Other deposit mechanisms involve precipitation from solution and settling of large particular matter. Inverse-temperature solubility leads to deposition where heat transfer is great.

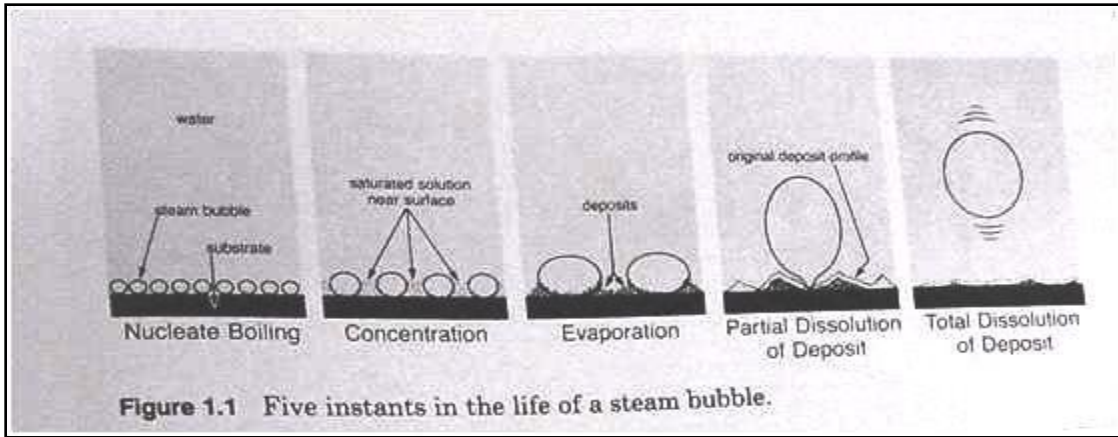


Figure 2.1 Five instant in the life of a steam bubble

The tendency to form deposits is related to localized heat input, water turbulence, and water composition at or near the tube wall. When a steam bubble becomes dislodged from a tube wall, the deposits are washed with water. The rate at which the deposits builds depend on the rates of bubble formation and the effective solubility of the deposit. In cases of high heat input, a stable steam blanket can form and cause concentration of water-soluble material (Figure 2.2). Steam blanket deposits do not redissolve, because blanketing also results from surface irregularities, which disturb water flow. Downstream of such irregularities, low pressure areas are formed, favoring steam buildup and consequently deposit formation.

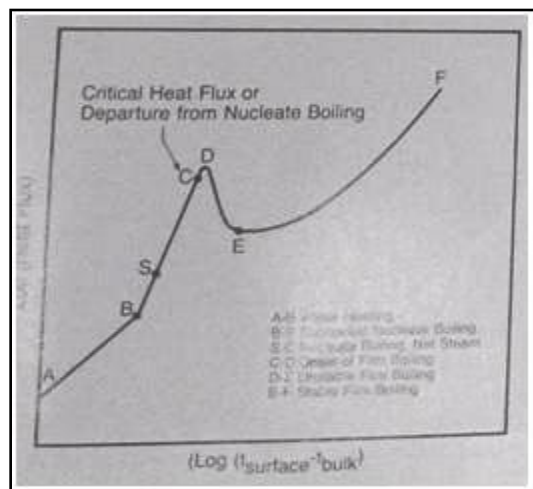


Figure 2.2 Heat transfer to water and steam in a heated flow channel. Relation of heat flux to temperature difference between channel wall and bulk water

## Locations

Deposit can occur anywhere water or steam is present in a boiler. While wall and screen tubes are usually the most heavily fouled, roof and floor tubes often contain deposits as well. Superheaters and reheaters frequently contain deposits that are formed elsewhere and are carried into the systems with boiler water. Steam is not often generated in economizers. Deposited in these tubes are usually made up of corrosion products moved from their originates sites.

Deposition can be substantial during steam generations. Tube orientation can influence the location and amount of deposition. Deposits are usually heaviest on the hot side of steam-generating tubes. Because of steam channeling, accumulations are often heavier on top portions of horizontal and slanted tubes. Also, deposition often occurs immediately downstream from circumferential-weld backing rings, which disturb flow and are favored sites for steam blanketing. Because deposits tend to concentrate in the hottest regions of steam-generator tubes those tubes near the bottom rear well of boilers using chain-grate stokers, and screen tubes are susceptible to deposition. Coarse particular matter is likely to be found in horizontal runs and where flow velocity is small.

Most economizers are designed to operate without producing steam. Waterborne deposits usually enter the economizer from sources such as the returned condensate (usually not polished). Oxides formed as a result of elevated oxygen concentrations prior to or inside the economizer may be moved ad deposited in the economizer.

Mud and steam drums often contain deposits. Because drums readily accessible, a visual inspection can provide many details about water chemistry and deposition processes. For example, sparkling black magnetite crystal may precipitate in steam drums when iron is released by the decomposition of organic complexing agents.

Superheaters deposits ate caused by carryover of boiler water, sometimes associated with foaming or high water levels. Such deposits will usually be concentrated near the superheater inlet or in nearby pendent U-bends. Contaminated attemperation water can also add deposits immediately downstream from the introduction point. Chop scale and

exfoliated oxide particles can be blown through the superheater, accumulating in pendant U-bents, or even more seriously, can be carried into turbines.

## **2.2 Scanning Electron Microscope**

### General Description

The scanning electron microscope (SEM) is a type of electron microscope that creates various images by focusing a high energy beam of electrons onto the surface of a sample and detecting signals from the interaction of the incident electrons with the sample's surface. The type of signals gathered in a SEM varies and can include secondary electrons, characteristic x-rays, and back scattered electrons. In a SEM, these signals come not only from the primary beam impinging upon the sample, but from other interactions within the sample near the surface. The SEM is capable of producing high-resolution images of a sample surface in its primary use mode, secondary electron imaging. Due to the manner in which this image is created, SEM images have great depth of field yielding a characteristic three-dimensional appearance useful for understanding the surface structure of a sample. This great depth of field and the wide range of magnifications are the most familiar imaging mode for specimens in the SEM. Characteristic x-rays are emitted when the primary beam causes the ejection of inner shell electrons from the sample and are used to tell the elemental composition of the sample. The back-scattered electrons emitted from the sample may be used alone to form an image or in conjunction with the characteristic x-rays as atomic number contrast clues to the elemental composition of the sample.

### Principles

In a typical SEM, electrons are thermionically emitted from a tungsten or lanthanum hexaboride ( $\text{LaB}_6$ ) cathode and are accelerated towards an anode; alternatively, electrons can be emitted via field emission (FE). Tungsten is used because it has the highest melting point and lowest vapour pressure of all metals, thereby allowing it to be heated for electron emission. The electron beam, which typically has an energy ranging from a

few hundred eV to 100 keV, is focused by one or two condenser lenses into a beam with a very fine focal spot sized 0.4 nm to 5 nm. The beam passes through pairs of scanning coils or pairs of deflector plates in the electron optical column, typically in the objective lens, which deflect the beam horizontally and vertically so that it scans in a raster fashion over a rectangular area of the sample surface. When the primary electron beam interacts with the sample, the electrons lose energy by repeated scattering and absorption within a teardrop-shaped volume of the specimen known as the interaction volume, which extends from less than 100 nm to around 5  $\mu\text{m}$  into the surface. The size of the interaction volume depends on the electrons' landing energy, the atomic number of the specimen and the specimen's density. The energy exchange between the electron beam and the sample results in the emission of electrons and electromagnetic radiation, which can be detected to produce an image

### Sample Preparation

Specimens that are studied in the SEM can be divided into two categories, namely conductors and non-conductors. Several factors to consider during all specimen preparation and these are given below:

1. The size and weight necessitate some reduction of the specimen to fit holder and to ease specimen manipulation for observation.
2. The specimen should be able to withstand the high vacuum of the SEM as it might be deformed.
3. It should be clean and dry, i.e. free of dust, moisture, oils and grease as their presence can lead to charging, contamination and longer pump down times.
4. Porous samples will also take a long time to pump down.
5. Coat non-conductors to prevent charging.
6. There should be good electrical connection between the surface of the specimen and specimen stub or holder.

### Detection of secondary electrons

The most common imaging mode monitors low energy (<50 eV) secondary electrons. Due to their low energy, these electrons originate within a few nanometers from the surface. The electrons are detected by an Everhart-Thornley detector which is a type of scintillator-photomultiplier device and the resulting signal is rendered into a two-dimensional intensity distribution that can be viewed and saved as a Digital image. This process relies on a raster-scanned primary beam. The brightness of the signal depends on the number of secondary electrons reaching the detector. If the beam enters the sample perpendicular to the surface, then the activated region is uniform about the axis of the beam and a certain number of electrons "escape" from within the sample. As the angle of incidence increases, the "escape" distance of one side of the beam will decrease, and more secondary electrons will be emitted. Thus steep surfaces and edges tend to be brighter than flat surfaces, which results in images with a well-defined, three-dimensional appearance. Using this technique, resolutions less than 1 nm are possible.

### Detection of backscattered electrons

Backscattered electrons consist of high-energy electrons originating in the electron beam, that are reflected or back-scattered out of the specimen interaction volume. Backscattered electrons may be used to detect contrast between areas with different chemical compositions, especially when the average atomic number of the various regions is different, since the brightness of the BSE image tends to increase with the atomic number.

Backscattered electrons can also be used to form electron backscatter diffraction (EBSD) image. This image can be used to determine the crystallographic structure of the specimen.

There are fewer backscattered electrons emitted from a sample than secondary electrons. The number of backscattered electrons leaving the sample surface upward might be significantly lower than those that follow trajectories toward the sides. Additionally, in contrast to the case with secondary electrons, the collection efficiency of backscattered

electrons cannot be significantly improved by a positive bias common on Everhart-Thornley detectors. This detector positioned on one side of the sample has low collection efficiency for backscattered electrons due to small acceptance angles. The use of a dedicated backscattered electron detector above the sample in a "doughnut" type arrangement, with the electron beam passing through the hole of the doughnut, greatly increases the solid angle of collection and allows for the detection of more backscattered electrons.

### X-ray Microanalysis

X-rays, which are also produced by the interaction of electrons with the sample, may also be detected in an SEM equipped for energy-dispersive X-ray spectroscopy or wavelength dispersive X-ray spectroscopy

## **2.3 Long-Term Overheating**

### General Description

Long-term overheating is a condition in which metal temperatures exceed design limits for days, weeks, months, or longer. This type of overheating is the cause of more boiler failures than any other mechanism. Because steel loses much strength at elevated temperatures, rupture caused by normal internal pressure becomes more likely as temperature rises.

The maximum allowable design temperature is primarily a function of tube metallurgy. As the amount of alloying element, particularly chromium and molybdenum, is increased, higher temperature can be tolerated. Alloy tubes are therefore frequently used in superheaters and reheaters (Table 2.1). Long-term overheating depends on temperature, length of time at temperature, and tube metallurgy.



Steel Alloy	Approximate Scaling Temperature	
	°F	°C
Carbon	1025	550
18Cr – 8Ni	1600	870
18Cr – 8Ni, Ti	1700	930
25Cr – 12Ni	2000	1100

Table 2.1 Temperature at which Thermal Oxidation Becomes Excessive

### Creep Rupture (Stress Rupture)

Creep is the terms used to describe the tendency of a ‘solid’ material to slowly move or deform permanently to relieve stresses. It occurs as a result of long term exposure to levels of stress that are below the yield strength or ultimate strength of the material. Creep is more severe in materials that are subjected to heat for long periods, and near the melting point. It is often observed in glasses. Creep is a monotonically increasing function of temperature.

The rate of this deformation is a function of the material properties, exposure time, exposure temperature and the applied load (stress). Depending on the magnitude of the applied stress and its duration, the deformation may become so large that a component can no longer perform its function — for example creep of a turbine blade will cause the blade to contact the casing, resulting in the failure of the blade. Creep is usually of concern to engineers and metallurgists when evaluating components that operate under high stresses or high temperatures. Creep is not necessarily a failure mode, but is instead a deformation mechanism. Moderate creep in concrete is sometimes welcomed because it relieves tensile stresses that otherwise may have led to cracking.

Creep rupture is a form of long-term overheating damage that usually produces a thick-lipped rupture at the apex of a bulge. Creep produces slow plastic deformation and eventual coalescence of microvoids in metal during overheating. Often a small

longitudinal fissure will be present at the apex of a heavily oxidized bulge. The rupture will usually have blunt and slightly ragged edges. Similar, but smaller, longitudinally oriented ruptures and fissures may exist nearby.

## **2.4 Pitting Corrosion**

### General description

Pitting is a form of extremely localized attack that results in holes in the metal. These holes may be small or large in diameter, but in most cases they are relatively small. Pits are sometimes isolated or so close together that they look like a rough surface diameter about the same or less than a depth.

It is often difficult to detect pits because of their small size and because of the varying depths and numbers of pits that may occur under identical conditions. Pitting is also difficult to predict by laboratory tests. Sometimes the pits require a long time – several months or a year – to show up in actual service. Pitting is particularly vicious because it is a localized and intense form of corrosion, failures often occur with extreme suddenness.

### Pit Shape and growth

Pits usually grow in the direction of gravity. Most pits develop and grow downward from horizontal surfaces. Lesser numbers start on vertical surfaces, and only rarely do pits grow upward from the bottom of horizontal surfaces.

Pitting usually requires an extended initiation period before visible pits appear. This period ranges from months to years, depending on both the specific metal and the corrosive. Once started, however, a pit penetrates the metal at an ever-increasing rate. In addition, pits tend to undermine or undercut the surface as they grow.

### Autocatalytic Nature of Pitting

A corrosion pit is a unique type of anodic reaction. It is an autocatalytic process. That is, the corrosion processes within a pit produce conditions which are both stimulating and necessary for the continuing activity of the pit. As the example, Metal M is being pitted by an aerated sodium chloride solution. Rapid dissolution occurs within the pit, while

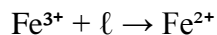
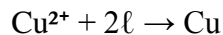
oxygen reduction takes place on adjacent surfaces. This process is self-stimulating and self – propagating. The rapid dissolution of metal within the pit tends to produce an excess of positive charge in this area, resulting in the migration of chloride ions to maintain electroneutrality.

### Solution Composition

From a practical standpoint, most pitting failures are caused by chloride and chlorine-containing ions. Chlorides are present in varying degrees in most waters and solutions made with water. Much equipment operates in seawater and brackish waters. Hypochlorites (bleaches) are difficult to handle because of their strong pitting tendencies. Mechanisms for pitting by chlorides are controversial and not well established. Perhaps the best explanation is the acid-forming tendency of chloride salts and the high strength of its free acid (HCL). Most pitting is associated with halide ions, with chlorides, bromides, and hypochlorites being the most prevalent. Fluorides and iodides have comparatively little pitting tendencies.

Oxidizing metal ion with chlorides are aggressive pitters. Cupric, ferric, and mercuric halides are extremely aggressive. Even our most corrosion-resistant alloys can be pitted by  $\text{CuCl}_2$  and  $\text{FeCl}_3$ . Halides of the nonoxidizing metal ions cause pitting but a much lesser degree of aggressiveness.

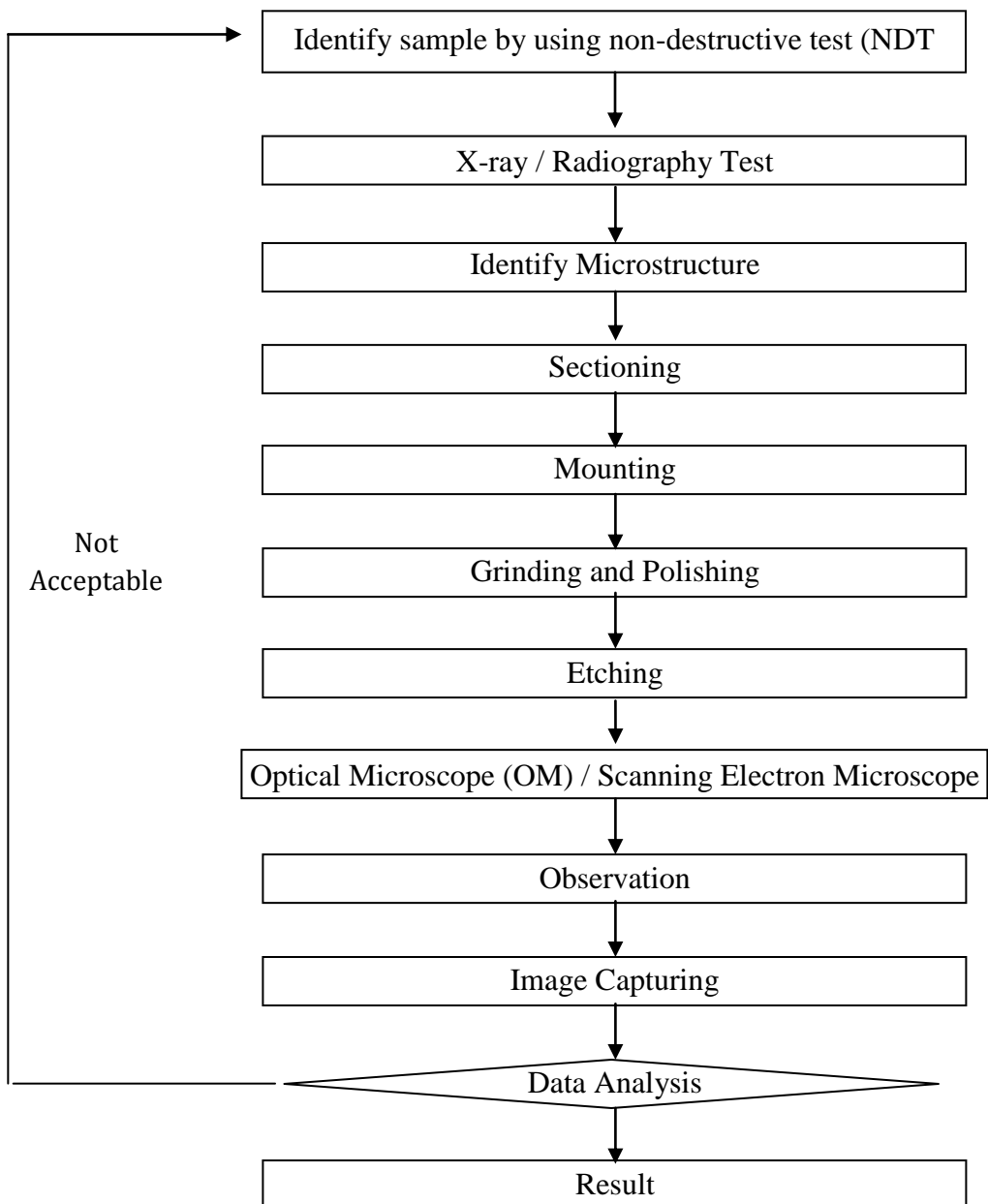
Cupric and ferric chlorides do not require the presence of oxygen to promote attack because their cations can be cathodically reduced. These ions are reducible as follows:



In other words, they are electron acceptors. This is one reason ferric chloride is widely used in pitting studies. The reaction are not appreciably affected by the presence or absence of oxygen.

Pitting can be prevented or reduced in many instances by the presence of hydroxide, chromate, or silicate salts. However, these substances tend to accelerate pitting when present in small concentrations.

**CHAPTER 3**  
**METHODOLOGY**



### 3.1 Sample Preparation

For non-destructive test (NDT), three samples has been prepared





Sample	Picture
ST 101	
ST 102	
ST 103	
ST104	

Table 3.1 Sample preparation

### 3.2 Non-Destructive Testing

#### General Description

Non-Destructive Testing (NDT), Non-Destructive evaluation (NDE) and Non-Destructive inspection (NDI) are the terms used in this connection to represent the techniques that are based on the application of physical principles employed for the purpose of determining the characteristics of materials or components or systems and for detecting and assessing the in homogeneities and harmful defects without impairing the usefulness of such materials or components or systems.

NDT plays an important role not only in the quality control of the finished product but also during various stages of manufacturing. NDT is also used for condition monitoring of various items during operation to predict and assess the remaining life of the component while retaining its structural integrity. Table 3.2 describes the advantages and the limitation of non-destructive testing over destructive testing.

<b>Destructive Test</b>	<b>Non-Destructive Test</b>
<p><u>Advantages</u></p> <ol style="list-style-type: none"> <li>1. Measurements are direct and reliable</li> <li>2. Usually quantitative measurements</li> <li>3. Correlation between test measurements and material properties are direct</li> </ol> <p><u>Limitations</u></p> <ol style="list-style-type: none"> <li>1. Tests are not made on the objects directly. Hence correlation between the sample specimen used and object needs to be proved</li> <li>2. A single test may measure only one or few of the properties.</li> <li>3. Inservice testing is not possible</li> <li>4. Measurement of properties over a cumulative period of time cannot readily be possible</li> <li>5. Preparation of the test specimen is costly</li> <li>6. Time requirement are generally high</li> </ol>	<p><u>Limitations</u></p> <ol style="list-style-type: none"> <li>1. Measurements are indirect reliability is to be verified.</li> <li>2. Usually qualitative measurements can also be done quantitatively.</li> <li>3. Skill judgment and experience are required to interpret indications.</li> </ol> <p><u>Advantages</u></p> <ol style="list-style-type: none"> <li>1. Test is made directly on the object. 100% testing on actual component is possible.</li> <li>2. Many NDT methods can be applied on the same part and hence many or all properties of interest can be measured.</li> <li>3. Inservice testing is possible.</li> <li>4. Repeated checks over a period of time are possible.</li> <li>5. Very little preparation is sufficient.</li> <li>6. Most test methods are rapid.</li> </ol>

Table 3.2 Advantages and the limitation of non-destructive testing over destructive testing.

The principal NDT methods are Visual or Optical inspection, Dye penetrant testing, Magnetic particle testing, Eddy Current testing, Radiographic testing and Ultrasonic testing.

### Liquid Penetrant Testing

Penetrant inspection utilizes the natural accumulation of a fluid around a discontinuity to create a recognizable indication of a crack or other surface opening. Capillary action attracts the fluid to the discontinuity as compared to its surroundings.

### Physical Principles

Penetrant inspection depends mainly on the stability of a liquid to wet the surface of a solid work piece or specimen and flow over that surface to form a continuous and reasonably uniform coating, thus penetrating into cavities that are open to the surface. The ability of a given liquid to flow over a surface and enter surface cavities mainly depends on the surface tension and capillary action.

Capillary action is the phenomenon of rise or depression of liquid in narrow cavities. Visible light or ultraviolet light is required for inspection of penetrant indications. Initially, the only detection method used was the unaided observation by the eye of the inspector under visible light.

Fluorescence describes the release of light energy by some substances when they are excited by external radiation such as ultraviolet light. In penetrant inspection, when the particles in fluid are struck by the incident ultraviolet light, they are excited to a higher energy level.

### Procedure for Penetrant Testing

#### Cleaning

The most important part in penetrant testing is initial cleaning to the surface are to be inspected. Scale, flakes, paint, dirt, grease and another chemical that are not cleaned from the surface will tend to accumulate the penetrant. This leads to either masking of real indications or defect indications where none exist.





Figure 3.1 Cleaner

### Penetrant application

The fluid should spread freely and evenly over the surface and move into the crack. The dwell time, which is the amount of time required to move into the crack will vary depending on the crack size and shape characteristics and also the environment conditions such as temperature and surface inclination. A minimum dwell time of about 20 to 30 min may be allowed after penetrant application.



Figure 3.2 Liquid penetrant

### Removal to Excess Penetrant

The desired result is that the surface is completely clear of penetrant. Yet, the crack/defect retain all the penetrant that entered into it.

### Applications of Developer

After removing the excess penetrant, a thin coating of developer is applied over the surface to draw the penetrant out of the crack and increase its visibility. Developer also cover the surface with a colour that provides good visual contrast to the penetrant. This increase the visibility of the defect.



Figure 3.3 Developer

### Inspection and Evaluation

The last step is the scanning of the surface for indications. The scanning may be carried out under visible light conditions or with ultraviolet or laser incident light and the defect recognition may be made with the human eye or with automated optical scanners.

Each Indication that appears should be evaluated. It may actually unacceptable; it may be worse that it appears; it may be false; it may be real but non relevant. Because penetrant inspection provides only indication corresponding to surface discontinuities, its severity cannot always be determined at first glance.

### **3.3 Radiography**

#### Basic Principle

The purpose of radiography is to show the presence and nature of defects or other structural discontinuities in the interior of the materials under examination. This technique makes use of the ability of short wavelength electromagnetic radiations, such as X-rays or gamma rays, to penetrate objects. In general, the shorter the wavelength, the greater is the penetrating power. The radiation that enters through the material, some being absorbed in the material itself and amount of absorption is a function of the density and thickness of the material. Should there be a cavity or discontinuity in the interior of the material, the beam of radiation will have less material to pass through than in solid material. The variation, if measured or recorded on film sensitive to X- or gamma radiation, produces an image that will indicate the presence of the defect.

### **3.4 Sectioning**

Sectioning is the removal of a representative sample from the parent piece by removing a suitably sized specimen from the larger mass at the desired location and orientation. The sectioning plane should be as near to the desired location as possible. The microstructure must not be altered in the process. High speed abrasive sectioning or low speed low deformation precision sectioning can be used.

In this project, the provided samples have been sectioned and the sectioning plane is in the longitudinal section of the parent sample.

### 3.5 Mounting



Figure 3.4 Hot mounting machine



Figure 3.5 Mounted samples

Mounting is a process by embedding the sample in a plastic medium for ease in manipulation and other factors such as fragility and edge preservation. The primary purpose of mounting metallographic specimens is for convenience in handling specimens of difficult shapes or sizes during the subsequent steps of metallographic preparation and examination. A secondary purpose is to protect and preserve extreme edges or surfaces defects during metallographic preparation.

Usually there are of two types of mounting methods that can be applied that is cold or hot mounting. Cold mounting is performed with a mixture of resin and hardener but hot mounting uses a certain amount of pressure and heat to mount the samples.

Equipment	SIMPLIMENT 1000, AUTOMATIC MOUNTING PRESS
Powder	Bakelite green
Heat time	2 minutes
Cool time	Cool time: 5 minutes
Pressure	4200 psi

Table 3.3 Operation Detail for Mounting

### 3.6 Grinding and Polishing



Figure 3.6 Grind and polish equipment

Grinding can be divided into two parts. Coarse grinding is to produce an initial flat surface and fine grinding is to remove the zone of deformation caused by sectioning and coarse grinding and limits the depths of deformation during this stage by proper abrasive size sequencing. All grinding steps should be performed wet provided that water has no adverse effect on any constituents of the microstructure. It is to minimize specimen heating and to prevent the abrasive from becoming loaded with metal removed from the specimen being prepared.

Polishing is the final step in producing a deformation-free surface that is flat, scratch free and mirror like in appearance. This is to observe the true microstructure for subsequent metallographic interpretation, both qualitative and quantitative. Polishing can be divided into two parts. Rough polishing is a further limitation of the deformation zone produced by fine grinding and final polishing is to remove the deformation zone produced during rough polishing. It is done with 6- or 3 $\mu$ m diamond abrasives charged onto a low nap cloths.

Sic Grit Size	Wheel Speed	Sic Grit Size	Wheel Speed
120	150	600	150

320	150	800	150
400	150	1200	150

Table 3.4 Sic grit size and wheel speed for grinding

### 3.7 Etching

Etching is the process to reveal structural details by preferential attack of a metal surface with an acid or basic chemical solution to reveal inclusions, porosity, cracks, intergranular corrosion and structural details of the samples. A table below shows the reagent medium tried, their compositions, their reaction type and methods involved:

	Composition	Type	Method	Result
Vilella's Reagent	45ml Glycerol, 30ml HCl, 15ml HNO <sub>3</sub>	Delineating	Immerse 40 to 60secs. and use several polish- etch cycle	Only poor to average results
Glyceregia	10ml Glycerol, 15ml HCl, 5ml HNO <sub>3</sub>	Delineating	Swab or immerse from 30-60secs	Good results with clear delineation of dendrites

Table 3.5 Etchants trialed on the Austenitic Stainless Steel

However, glyceregia could not be stored for a period of time due to the degradation over time and there is also a potential of changing into nitro-glycerin over time.



Figure 3.7 Glycerol, Hydrochloric acid & nitric acid

### **3.8 Image capturing**

This method will be performed by using an optical microscope (OM) and scanning electron microscope (SEM). The image of the microstructure of each sample will be captured to be characterized and analyzed. .

Scanning electron microscope was also used in this project to distinguish between the individual phases present in the microstructure. The main advantage of this method compared to the optical microscope is that it can be magnified at any magnification larger than the range of magnification from the optical microscope.





## CHAPTER 4

### RESULT & DISCUSSION

The sample have been divided into three small part which is external tube, the core, and the spiral.

#### 4.1 External Tube

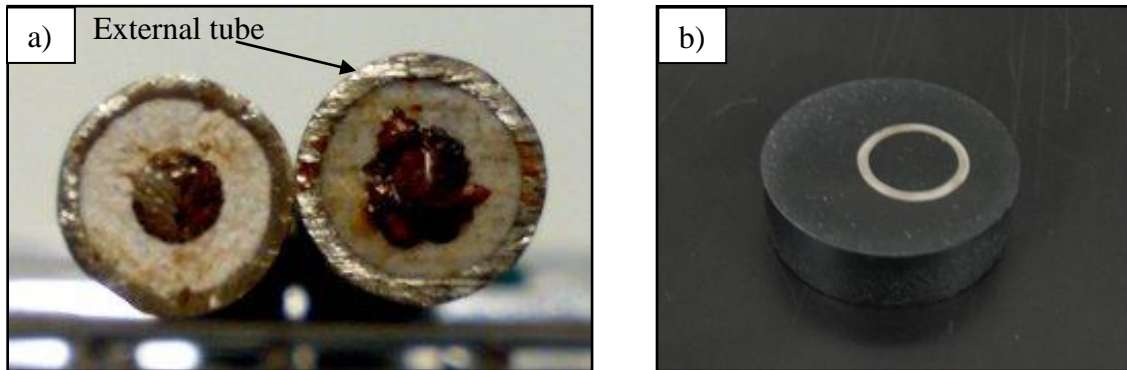


Figure 4.1 (a) Cross sectional area for heater tube; (b) Mounted sample for external tube

#### 4.1.1 Observation from Optical Microscope

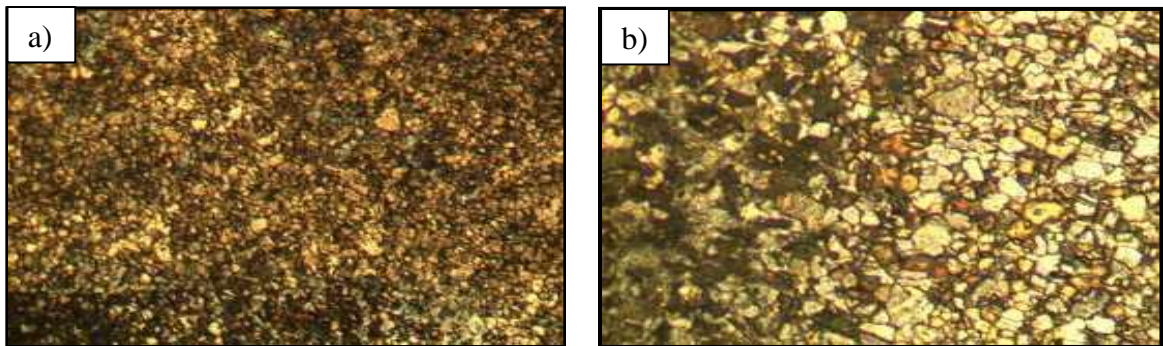


Figure 4.2 (a) Microstructure image of external tube for 20X zooming; (b) 50X zooming

Figure 4.2a and 4.2b illustrate the image of microstructure for the external tube. Figure 4.2a show magnification scales of 20 and Figure 4.2b is 50. From the images, no continues lines or cracks were found at the surface and thus no symptom of failure occurred at the sample for both 20X and 50X zoom.

#### 4.1.2 Observation from Scanning Electron Microscope (SEM)

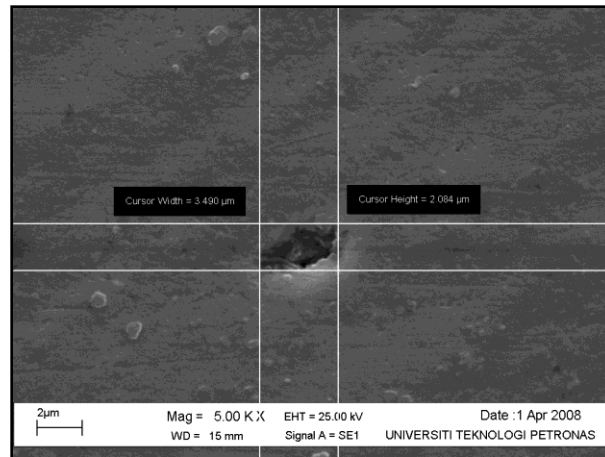


Figure 4.3 SEM Image for External Tube

From figure 4.5, 2 $\mu$ m diameters of pit occur at the surface of the external tube. This indicates pitting corrosion occurred due to dissolution of metal and the maintenance of a high degree of acidity at the bottom of the pit by the hydrolysis of the dissolved metal ions.

#### 4.1.3 EDS Analysis

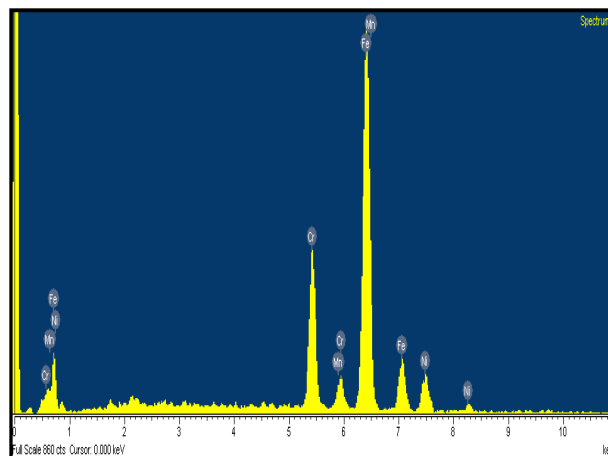


Figure 4.4 Spot Mode for external tube

From the EDS analysis, the external tube consists of three main elements which are chromium, ferum, and nickel. The percentage details are in the table below:

Element	Weight%
Cr K	18.11
Mn K	2.19
Fe K	69.25
Ni K	10.44
Totals	100.00

Table 4.1 Percentage elements for external tube

The table shows that the material consist of about 70% ferum, 19% chromium, and 10% nickel which verified that material used for the external tube is stainless steel 304.

#### 4.2 The Core

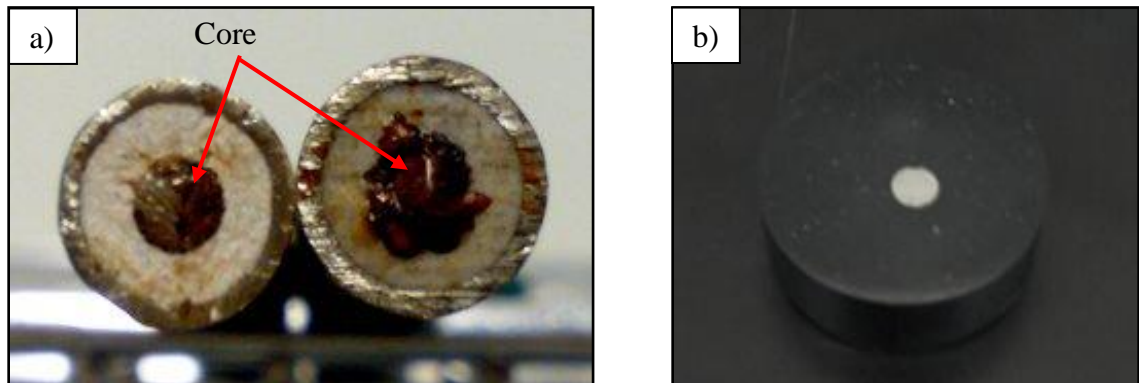


Figure 4.5 (a) The tube of the heater; (b) Mounted sample for the core

##### 4.2.1 Observation from Optical Microscope

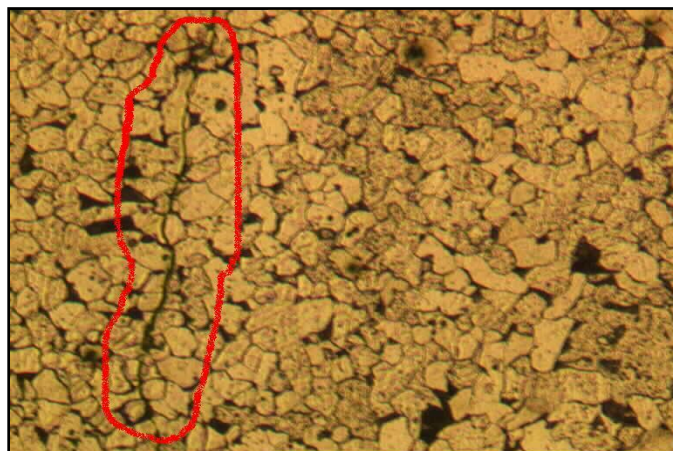


Figure 4.6 Microstructure image for internal tube

Figure 4.6 illustrate the image of microstructure for the internal tube. From the images, there was continuous line occurred (rounded with red mark) along and inside the grain boundaries. Thus there was a crack at the internal tube due to long term overheating.

#### 4.2.2 Observation from Scanning Electron Microscope (SEM)

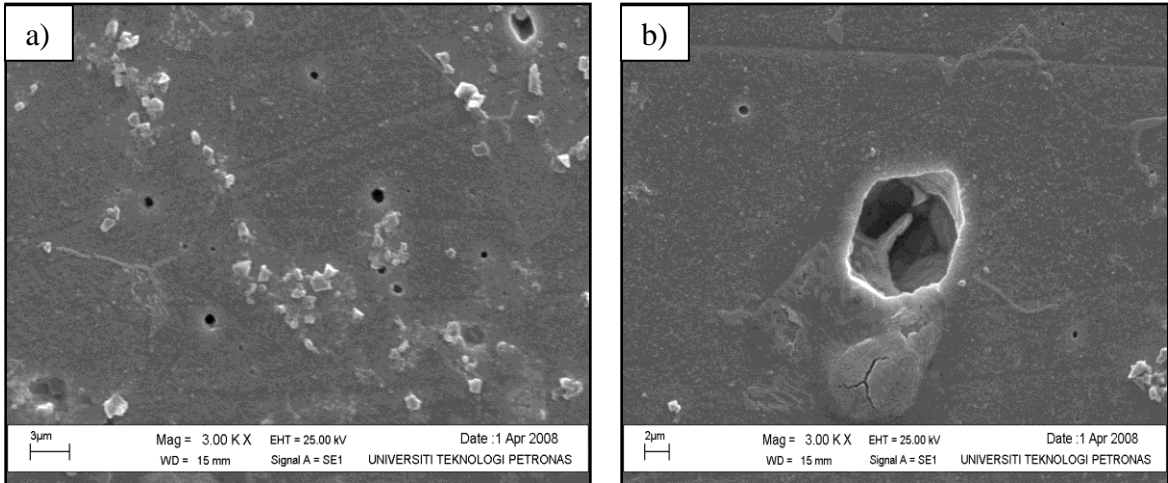


Figure 4.7 SEM image for internal tube (a) at scale 2µm (b) at scale 3µm

From the figure 4.7a, a lots of pits occurred at the sample caused by chloride and chlorine – containing ions. Oxidizing metal ions with chlorides are aggressive pitters. And at figure 4.7b, 2µm pit occur by oxidizing metal ions. The composition of the pits will be discussed further on the EDS analysis.

#### 4.2.3 EDS Analysis

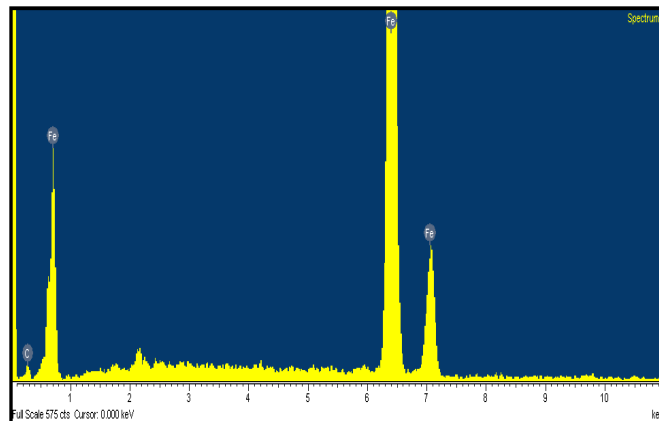


Figure 4.8 EDS analysis for internal tube

From the EDS analysis (spot mode), the internal tube consist only two elements which are Chromium and Ferum. The percentage details are in the table below:

Element	Weight %
C K	5.87
Fe K	94.13
Total	100.00

Table 4.2 Percentage elements for internal tube

The table shows that 6% Chromium and 94% Ferum used in fabrication this material which means this material is low carbon alloy.

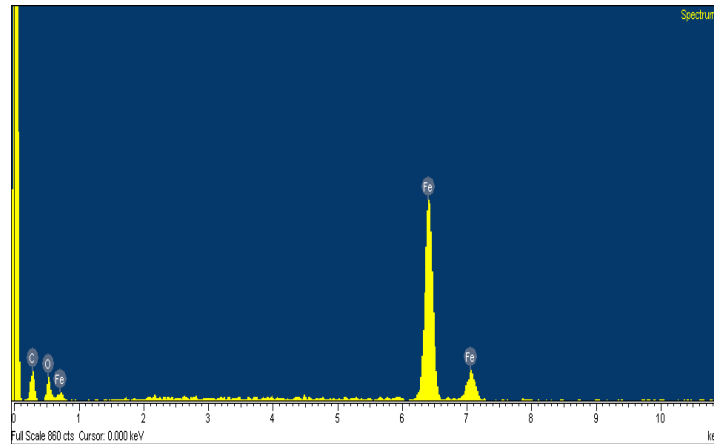


Figure 4.9 EDS analysis at the pit

From the EDS analysis, the external tube consist only three elements which are Chromium, Ferum and Oxygen. The percentage details are in the table below:

Element	Weight %
C K	30.72
O K	13.25
Fe K	56.03
Total	100.00

Table 4.3 Percentage elements at pit surface

The table shows that occurrence of oxygen in the material which mean oxidizing of metal ion happened.

### 4.3 The Spiral

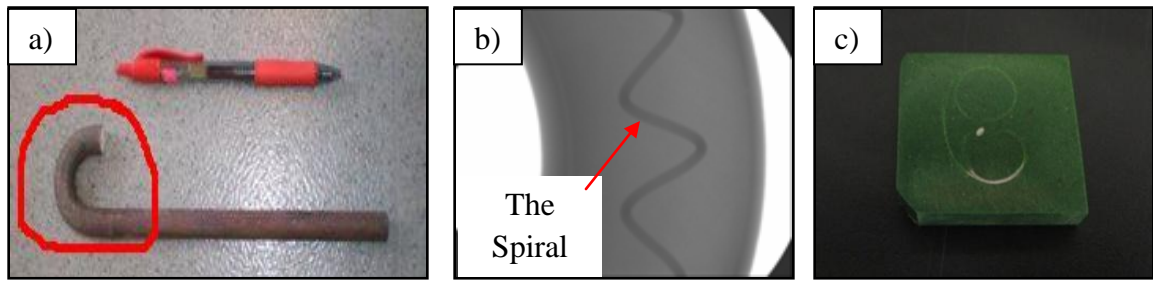


Figure 4.10 a) Internal tube at u bend of the heater; b) Results from X-ray analysis; c) Mounted sample for the spiral

#### 4.3.1 Observation from Optical Microscope

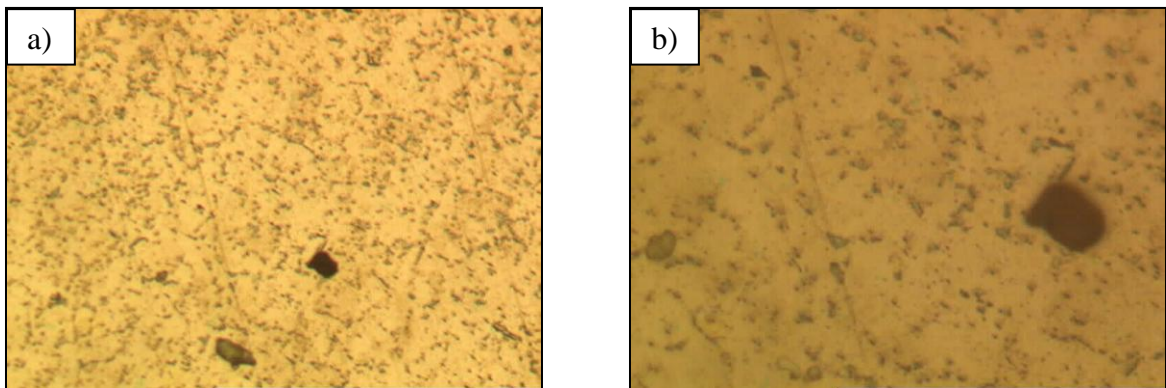


Figure 4.11 (a) Microstructure image of internal tube at the spiral for 20X zooming; (b) 50X zooming

Figure 4.11 illustrate the image of microstructure for the internal tube at the spiral. Figure 4.11a show magnification scales is 20 and Figure 4.11b is 50. From the images, no continues lines or cracks were found at the surface thus no symptom of failure occurred at the sample for both 20X and 50X.

#### 4.3.2 Observation from Scanning Electron Microscope (SEM)

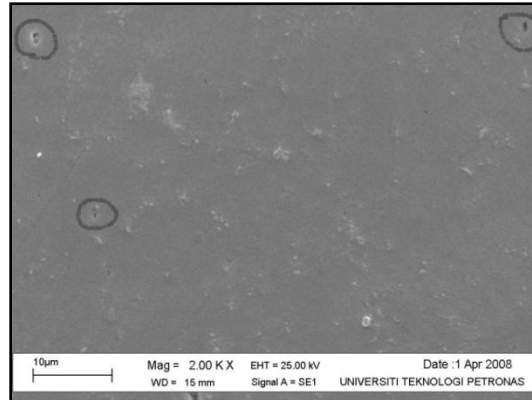


Figure 4.12 SEM image for internal tube at the spiral

From figure 4.12, only a few and small sizes of pits occur at the surface internal tube at the spiral thus no symptom of failure occurred at the spiral.

#### 4.3.3 EDS Analysis

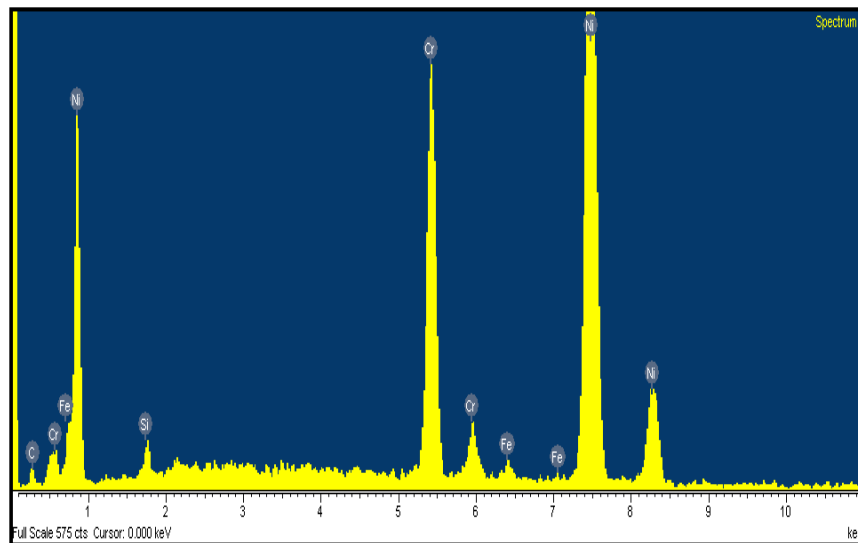


Figure 4.13 EDS analysis for internal tube at U bend

From the SRD analysis, this internal tube consists of five elements which are Carbon, Silica, Chromium, Ferum, and also Nickel. The percentage details for each element in the table below:

Element	Weight %
C K	7.35
Si K	1.49
Cr K	19.29
Fe K	0.93
Ni K	70.97
Total	100.00

Table 4.4 Percentage elements at internal tube at the spiral

From the table, the internal tube consists of three main elements which are 1% of ferum, 19% of chromium and 70% of nickel which verified that material used for the spiral is the nickel-chromium-carbon-alloys.



## CHAPTER 5

### 5.1 CONCLUSION

This water heater tube basically made from three parts which are external tube, internal tube, and internal tube at U-bend. The external tube used as a heating element meanwhile the internal tube used as detector and both of them separated by ceramic. The external and the internal tube work as thermocouple.

Basically, several processes must be done to get clear image of the microstructure and it started with sampling as representative sample from the parent piece by removing a suitably sized specimen, mounting for easier to hold the sample, grinding to produce an initial flat surface and polishing to produce a deformation-free surface, etching to reveal structural details and finally image capturing by using optical microscope and scanning electron microscope.

After all the tests have been done, the material used for the external tube was stainless steel 304 meanwhile for the internal tube was low carbon alloy. The different types of materials for the heater tube (external and internal) have cause the pitting corrosion and cracks occurred at the internal tube due to overheating and oxidizing.

## **REFERENCES:**

1. MAB4423: Failure Analysis & Non Destructive Examination – the Study Crude Storage tank Bottom Plate Failure.
2. Structural Materials Technology IV: Technomic, 2000.
3. ASTM E-45; Standard Test Methods for Determining the Inclusion Content of Steel<sup>1</sup>.
4. ASTM E 1245; Standard Practice for Determining the Inclusion or Second-Phase Constituent Content of Metals by Automatic Image Analysis<sup>1</sup>.
5. ASTM E 381; Standard Method for Macroetch Testing Steel Bars, Billets, Blooms, and Forgings.
6. Corrosion of Stainless Steel; 2<sup>nd</sup> Edition – A. John Sedriks 1996.
7. Corrosion Engineering; 3<sup>rd</sup> Edition – Mars G. Fonatana: McGraw-Hill Book Company 1987.
8. Analytical Characterization of Aluminum, Steel, and Superalloys – D. Scott MacKenzie & George E. Totten: Taylor and Francis 2006.

**APPENDICES:**

**Appendix 1** – ASTM B 167; Standard specification for Nickel-Chromium-Iron Alloys

**Appendix 2** – ASTM B 649; Standard specification for Ni-Fe-Cr-Mo-Cu Low Carbon

Allow