Simplified Creep Assessment of Catalyst Tube

By

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Dissertation submitted in partial fulfillment of

the requirements for the

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CERTIFICATION OF APPROVAL

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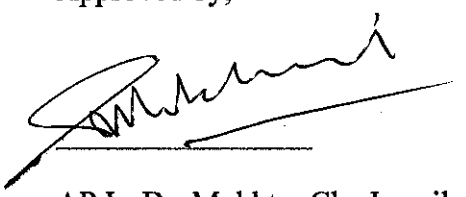

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MAY 2011

CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

Muhammad Khairuddin Bin Hashim

ABSTRACT

Creep monitoring and assessment of catalyst tube is critical to avoid catastrophic failure. Although there are many creep assessment methodologies available, the methods not only costly but also sophisticated. Thus, there is a need to develop a simplified creep assessment method to assist plant engineer. The main objective of this study is to develop a method of Non-Destructive Testing and microstructure study methodologies to monitor creep in catalyst tube. A sample of damage catalyst tube has been taken from PETRONAS Fertilizer Kedah and the tube ruptured early before it reaches the expected life of the tube and suspected due to creep damage Ultrasonic testing and metallography study has been chosen as the methodologies to monitor the creep on the catalyst tube. By using ultrasonic flaw detector, the ultrasonic velocity has been determined. While by doing metallography study, the microstructure is obtained from optical microscope and hardness value from microstructure Vickers hardness. The result showed that there is decreasing in ultrasonic velocity values with increasing values of grain size. Besides that, increasing of hardness value also associated with the precipitation of carbide. Thus, the objective for this study is achieved by monitoring the velocity in ultrasonic testing and the microstructure for reformer tube evaluation. This technique has a potential to assess the creep damages and remaining life of the reformer tube.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

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In the name of ALLAH S.W.T, the most merciful and compassionate, praise to ALLAH, he is the almighty, eternal blessing and peace upon the Glory of the Universe, our beloved Prophet Muhammad (S.A.W), and his family and companions.

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CHAPTER 1

INTRODUCTION

1.1 PROJECT BACKGROUND

A sample of damage catalyst tube has been taken from PETRONAS Fertilizer Kedah. It been used in a furnace that operated at 773°C. The tube has been replaced after 4 years in service due to creep damage. The tube had ruptured before it reaches the expected life of the tube which is 100,000 hours. It is important to avoid such failures by replacing tubes in a timely manner during planned maintenance shutdowns. In order to replace catalyst tubes before they fail but not when they still have significant useful life, an accurate, reliable method of assessing tube life is required. Before the remaining lives of tubes can be calculated, their current condition must be determined. Current assessment method relies on accurate analysis of microstructure degradation combining both non-destructive testing and replica metallography to predict remaining life of the tube. This method requires expertise usually done by third party consultant. Normal inspection during plant shutdown is using non-destructive testing and replica metallography but the data obtained usually not fully utilized to determine the condition of the tube. This is due to the lack of the technique. In this study, ultrasonic testing, hardness test, and microstructure study will be used to evaluate the tube.

1.2 PROBLEM STATEMENT

There is a need to develop a non-destructive test and microstructure study methodologies to monitor creep on the catalyst tube to assist the plant inspector in the creep assessment.

1.3 OBJECTIVES AND SCOPE OF STUDY

The main objectives this project is:

i. To develop a method of Non-Destructive Testing and microstructure study methodologies to monitor creep in catalyst tube.

ii. To study the relationship between ultrasonic sounds propagation in creep damage material by using ultrasonic testing and microstructure study.

The scope of study for this project is to do an inspection on a tube which operates at the high temperature process. The inspection will be done using ultrasonic method. Microstructures of the tube will be studied to determine what type of damage mechanism involved in the tube. The equipments that will be used in this project are:

i. Ultrasonic flaw detector

ii. Metallography equipment

iii. Optical microscope

iv. Hardness Test

CHAPTER 2

LITERATURE REVIEW / THEORY

2.1 INTRODUCTION

The optimization of the costs of industrial plants maintenance by preventing non-scheduled outages or extending service lifetime is increasingly important. Catalyst tube is critical to many high temperature processes in refining and chemical plants. Life management is usually dominated by the service capability of the radiant catalyst tubes. Their lives are limited by creep, driven by a combination of internal pressure stress and through-wall thermal stresses generated by operational factory. Although the service life may be estimated making use of theoretical models, due to the degradation process complexity, still it is necessary to characterize the materials of concern by NDE methods.

2.2 CATALYST TUBE

Catalyst tubes are generally made from creep resistant austenitic steel HK grade (25 Cr, 20 Ni, 0.4C) or HP grade (26 Cr, 35 Ni, 0.4 C). Both alloy are primarily used in corrosive gases at extremely high temperature under oxidizing condition and their high nickel content ensure resistance to stress-corrosion cracking at high temperature [1]. Although the furnace tubes are usually designed for a normal life of 100,000 h (11.4 years), their actual service life, however, varies from 30,000 to 180,000 hours, depending on the service conditions and of course on the quality of materials [2]. This tube is a high-alloy tubes which made by the centrifugal casting process intended for use under pressure at high temperatures. During plant operation, this tube are filled with catalyst, with gases are passing through the catalyst at extremely high temperature. For this tube, failures at high temperature due to deformation caused by creep are not uncommon. Creep is a tendency of solid material to slowly move or deform permanently under the influence of stresses [3]. This creep mechanism occurred due to harsh

environment in which the tube was exposed, such as at elevated temperature. Being able to identify and locate such damage at its early stage of growth is essential for safe plant operation. Due to this inevitable damage, over the past two decades much research has been done in this area.

2.3 CREEP MECHANISM IN CATALYST TUBE

Creep is the time-dependent plastic deformation which occurs when a material is subjected to a constant stress and temperature for an extended period and which may lead to catastrophic failure of components that are operating at elevated temperatures [3]. The basic mechanism involved in causing creep damage is the generation of vacancies (missing atoms) produced during diffusion of species under stress on an atomic scale [4]. Creep, arising as a consequence of the high operating temperatures and stress, primarily governs the life of catalyst tubes in gas reformer service [2]. In addition to the pressure stress, thermal stresses are introduced during plant start-up, shut-down and upset. It is the decay of these stresses at high temperature during operation, by the creep process, that results in creep damage and reduced tube life. The more rapid the rate of temperature change during start-up, shut-down or upset conditions, the higher the magnitude of thermal stress introduced into the catalyst tube and hence greater the degree of creep damage which occurs during subsequent operation. Creep is a progressive damage process, which produces a physical change in diameter of the catalyst tube with increased service time. As creep progresses, voids will nucleate at grain boundaries within the material and with further service exposure the creep process initiates micro-cracks in the tube [5]. In catalyst tubes, the thermal stress is maximized close to the internal surface and it is at this location that the creep damage starts. Once cracks form they will subsequently propagate by creep from the internal to external surface of the tube. Then inner wall starts broken, the tube begins to tear as the cracks grow outwards until they reach the outside. The final rupture occurs as a longitudinal split.

Temperature excursions or overheating can also cause a sudden and dramatic reduction in tube life. Whenever tubes are heated and cooled (thermal cycling), stresses are

temporarily increased and this leads to an acceleration of creep damage. In extreme cases, cooling or heating can be too rapid, leading to thermal shock failure.

Tube failures resulting from known service problems, such as splitting caused by serious overheating [5], are easily recognized. However, even under good, well-controlled service conditions, all reformer tubes will slowly undergo creep damage. Therefore, over and above process controls, it is advisable to monitor the service conditions and quality of the tubes themselves. This involves regular tube skin temperature readings to ensure that the design tube skin temperature is not exceeded, and at every opportunity for inspection as much information as possible should be gathered on the mechanical state of the tubes.

Creep strain is a time-dependent phenomenon. In reformer tubes the strain generally exhibits itself by an increase in tube diameter, which accompanies the crack propagation and growth [4]. The stages of creep life can be shown in a plot of strain against time. The curve can be segmented into three zones known as primary (I), secondary (II) and tertiary (III) creep [3].

Stage (I): Primary creep occurs early in tube life, and its rate decreases as progressive work-hardening inhibits dislocation movement.

Stage (II). Secondary or steady-state creep is the condition under which the tubes are designed to operate. The diameter increases at a very constant slow rate.

Stage (III). Tertiary creep is the final stage, during which deformation accelerates. At this point cracks within the structure are getting larger.

Initiation and growth of creep induced cracks from the internal sub-surface of the catalyst tube clearly makes Non Destructive Evaluation (NDE) more difficult. However, it has been recognized for some time, that monitoring changes in catalyst tube diameter and hence creep strain, will provide an indication of the tube condition. Catalyst tubes have relatively low creep ductility and depending on the material type, strain levels in excess of 3 to 4% can be indicative of exhausted creep life and therefore this level of plastic deformation is a useful indicator for tube replacement.

Larson Miller Parameter

There are several ways in which creep data can be presented. One method is to plot laboratory test data using the Larson- Miller Parameter (LMP). The LMP is a function relating temperature and time [6]. This parameter is defined as: Creep rate could adequately be described by the Arrhenius type equation:

$$r = Ae^{-\Delta H / R * T} \quad \text{(equation 1)}$$

Where r is the creep process rate, A is a constant, R is the universal gas constant, T is the absolute temperature, and ΔH is the activation energy for the creep process. Taking the natural log of both sides:

$$\ln(r) = \ln(A) - \Delta H / R*T \quad \text{(equation 2)}$$

With some rearrangement:

$$\Delta H / R = T\,(\ln(A) - \ln(r)) \quad \text{(equation 3)}$$

Using the fact that creep rate is inversely proportional to time, the equation can be written as:

$$\Delta l / \Delta t = A' e^{-\Delta H / R * T} \quad \text{(equation 4)}$$

Taking the natural log:

$$\ln(\Delta l / \Delta t) = \ln(A') - \Delta H / R*T \quad \text{(equation 5)}$$

After some rearrangement the relation finally becomes:

$$\Delta H / R = T * (B + \ln(\Delta t)), \text{ where } B = \ln(A' / \Delta l) \quad \text{(equation 6)}$$

This equation is of the same form as the Larson-Miller relation.

$$LMP = T * (C + \log(t)) \quad \text{(equation 7)}$$

where the quantity LMP is known as the Larson-Miller parameter. Using the assumption that activation energy is independent of applied stress, the equation can be used to relate

the difference in rupture life to differences in temperature for a given stress. The material constant C is typically found to be in the range of 20 to 22 for metals.

The Larson-Miller model is used for experimental tests so that results at certain temperatures and stresses can predict rupture lives of time spans that would be impractical to reproduce in the laboratory. The Larson-Miller parameter is a means of predicting the lifetime of material vs. time and temperature using a correlative approach based on the Arrhenius rate equation. The value of the parameter is usually expressed as LMP=T(C + log t) where C is a material specific constant often approximated as 20, t is the time in hours and T is the temperature in Kelvin.

Creep-stress rupture data for high-temperature creep-resistant alloys are often plotted as log stress to rupture versus a combination of log time to rupture and temperature. One of the most common time–temperature parameters used to present this kind of data is the Larson-Miller (L.M.) parameter, which in generalized form is

P(Larson Miller) = T[log tr + C] (equation 8)

T = temperature, K or °R

tr = stress-rupture time, h

C = constant usually of order 20

According to the Larson Miller parameter, at a given stress level the log time to stress rupture plus a constant of the order of 20 multiplied by the temperature in kelvins or degrees Rankine remains constant for a given material. The relationship between stress and LMP is used to predict a probable time to the onset of creep-rupture failure [6].

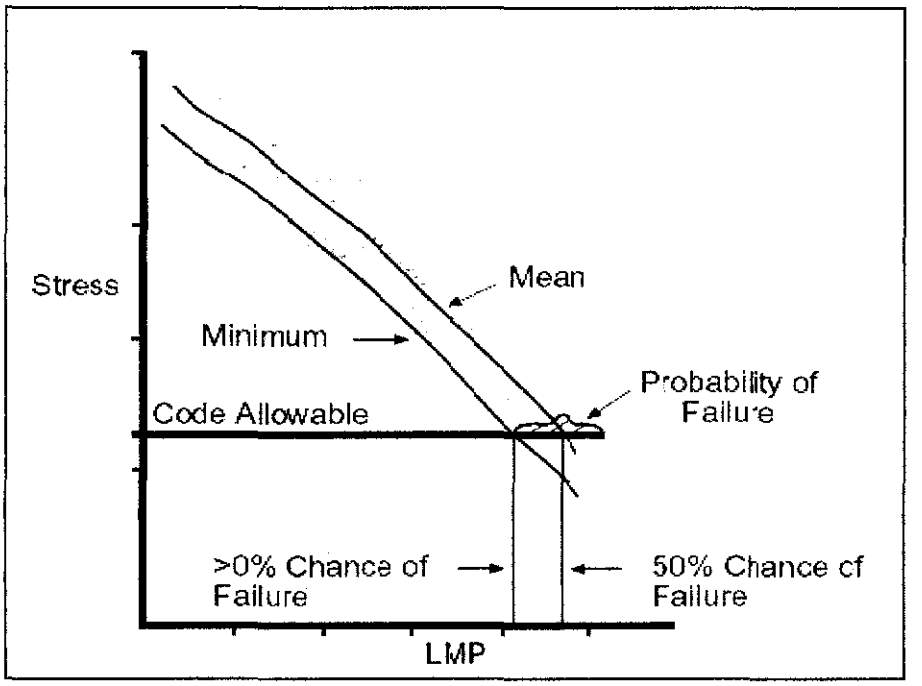

Figure 1: Stress v. LMP plot illustrating the statistical distribution of failure for a specific classification or grade of tubing.

2.4 NON-DESTRUCTIVE TESTING FOR DETECTING CREEP IN CATALYST TUBE

Nondestructive testing (NDT) is a wide group of analysis techniques used in science and industry to evaluate the properties of a material, component or system without causing damage. Because NDT does not permanently alter the article being inspected, it is a highly-valuable technique that can save both money and time in product evaluation, troubleshooting, and research. Common NDT methods have been used in detecting creep mechanism include:

a) Radiographic testing

b) Replication

c) Eddy current testing

d) Ultrasonic testing

2.4.1 RADIOGRAPHIC TESTING (RT)

Random radiographic examination is normally used as a supplementary technique to confirm the presence of severe cases of creep damage. It is reasonable to expect to locate such damage when it has extended 50% in the thru wall direction, if the tubes are filled with catalyst and isotopes are used instead of an X-Ray tube [7]. Although using RT provides an improved quality image, it is not normally employed, because of practical conditions on site.

2.4.2 REPLICATION

Replication is useful for in-situ assessment of reformer tube outside surface, to detect overheating that causes micro-structural changes. Replication is a 'spot' type assessment and is normally used as a supplemental technique. Only the advanced stages of creep damage can be assessed utilizing in-situ replication, since damage occurs internally within the tube wall [7].

2.4.3 EDDY CURRENT TESTING

Eddy-current testing uses electromagnetic induction to detect flaws in conductive materials. There are several limitations, among them: 1) only conductive materials can be tested.2) the surface of the material must be accessible.3) the finish of the material may cause bad readings.4) the depth of penetration into the material is limited.5) flaws that lie parallel to the probe may be undetectable. Eddy current is applied from the outside of the reformer tube and detects the variations in flux density generated by an induced electromagnetic coil. The sensitivity of this technique is compromised when the tube material's permeability changes. Due to the environment in which the reformer tube is operated and the materials it is made of, there are significant permeability variations down the tube's length after the first day it is placed into service. The eddy current technique also has a reduced sensitivity to damage located at the inside

surface of the tube wall. In some cases, this test method may provide false positives which will prompt a perfectly good tube to be removed from service. Just the opposite may also happen, resulting in a damaged tube being left in the reformer causing in tube failure, causing an unplanned outage to make necessary repairs. The technique relies on changes in electric circuit conditions; the circuit being the instrumentation, cables, sensing coil, and the item under test [7]. As the mechanical properties of the test materials change, a change in overall circuit impedance occurs, which is displayed on an oscilloscope. By monitoring these changes, it can be inferred that creep damage is present, based on observation of the signal parameters in comparison to similar changes that occurred on known creep-damaged materials. The depth of penetration of eddy currents is primarily influenced by frequency, conductivity, and relative permeability [8].

2.4.4 ULTRASONIC TESTING (UT)

The primary ultrasonic technique utilized for the detection and estimation of creep damage is through transmission ultrasonic attenuation [8]. This technique observes the absorption of UT energy as a function of damage accumulation in the form of microvoids and cracking, both microfissures and macrocracking. Recent validations have again found that ultrasonic is more reliable at the detection and quantification of creep damage, particularly in its early stages [7].

2.4.4.1 BASIC PRINCIPLES OF ULTRASONIC TESTING

Ultrasonic Testing (UT) uses high frequency sound energy to conduct examinations and make measurements. Ultrasonic inspection can be used for flaw detection/evaluation, dimensional measurements, material characterization, and more [10]. To illustrate the general inspection principle, a typical pulse/echo inspection configuration as illustrated below will be used.

A typical UT inspection system consists of several functional units, such as the receiver, transducer, and display devices. A receiver is an electronic device that can produce high voltage electrical pulses. Driven by the receiver, the transducer generates high frequency ultrasonic energy. The sound energy is introduced and propagates through the materials in the form of waves. When there is a discontinuity in the wave path, part of the energy will be reflected back from the flaw surface. The reflected wave signal is transformed into an electrical signal by the transducer and is displayed on a screen. In the applet below, the reflected signal strength is displayed versus the time from signal generation to when a echo was received. Signal travel time can be directly related to the distance that the signal traveled. From the signal, information about the reflector location, size, orientation and other features can sometimes be gained.

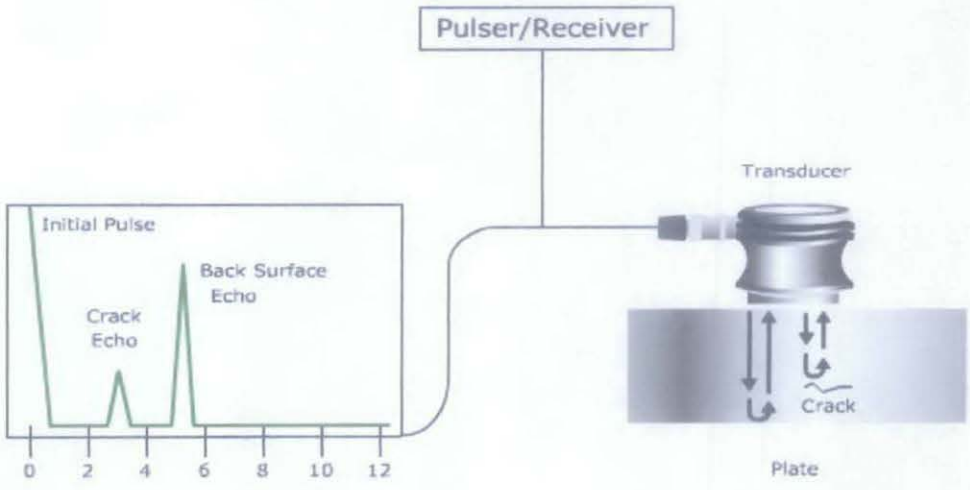

Figure 2: General inspection principle

2.4.4.2 TYPE OF DEFECTS MEASURE BY UT

- Crack
- Voids
- Porosity
- Laminations
- Unbond in materials
- Thickness variation

2.4.4.3 ADVANTAGES OF ULTRASONIC TESTING (UT)

Some of the advantages of ultrasonic inspection that are often cited include:

- It is sensitive to both surface and subsurface discontinuities.
- The depth of penetration for flaw detection or measurement is superior to other NDT methods.
- It is highly accurate in determining reflector position and estimating size and shape.
- Minimal part preparation is required.
- Electronic equipment provides instantaneous results.
- Detailed images can be produced with automated systems.
- It has other uses, such as thickness measurement, in addition to flaw detection.

2.4.4.4 CREEP DETECTION OF HK40 TUBE BY USING UT METHOD

Damage normally starts at the centre of the tube thickness and, due to operating temperature and pressure, develops to the inside surface [9]. Therefore, a volumetric method such as ultrasonic is required to detect voids. On the other hand, high anisotropy of the centrifugal cast austenitic material produces high attenuation and scattering. However a careful selection of ultrasonic variables could minimize these effects and be able to detect creep damage.

The method consists on a pair of probes arranged in pitch and catch fixture. The transmitter sends a pulse, which travels following a chord in the tube and is picked up by the receiver (see figure 2(a)).

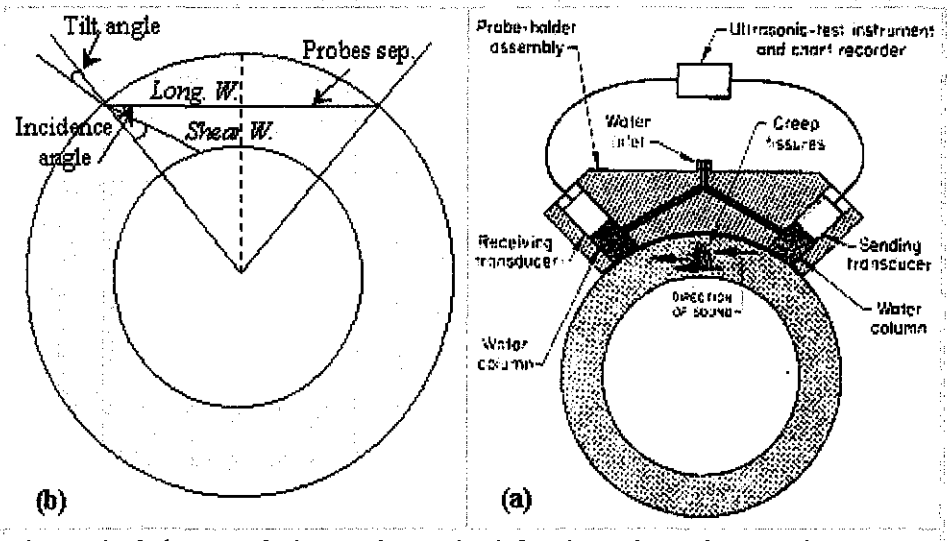

Figure 3: Scheme of ultrasonic method for detection of creep damage: a) inspection device for in-service inspection; b) fundamentals of the UT technique for reformer tubes inspection

The fixture is designed to allow the central beam passes by the mid-wall chord. Then, the wave signal received and the associated energy is affected by the insonified material structure. Presence of cavities will produce some attenuation and scattering of the ultrasonic wave, these will increase or decrease depending on the number and extent of cavities [9]. Therefore amplitude, and then energy, of the received signals is a rough

indicator of the present damage. High attenuation is associated to high level of damage and low attenuation to low level of damage.

Due to the surface state of reformer tubes, non-contact techniques must be applied. When the UT beam goes through the water and cross the steel, the probe angle is diffracted (see figure 03, (b)). Even, mode conversion is produced (unless in the case of normal incidence), generating shear and longitudinal waves. The shear wave front has some limitations to be transmitted in this type of materials. So, it has been shown that the best probe arrangement (based on the distance between emitter and receiver probes and in the tilt angle) consists of doing the longitudinal wave cross the tubes along the middle thickness. Amplitude of the ultrasonic signal is the first information to be evaluated in order to identify damaged areas. Due to the coarse structure of the tube material, there might occur changes in the ultrasonic response from point to point, which could mask the damage detection. Then, to assess the tube material, it is studied the evolution of signal amplitude in different adjacent locations along the tube.

CHAPTER 3

METHODOLOGY / PROJECT WORK

3.1 PROJECT IDENTIFICATION

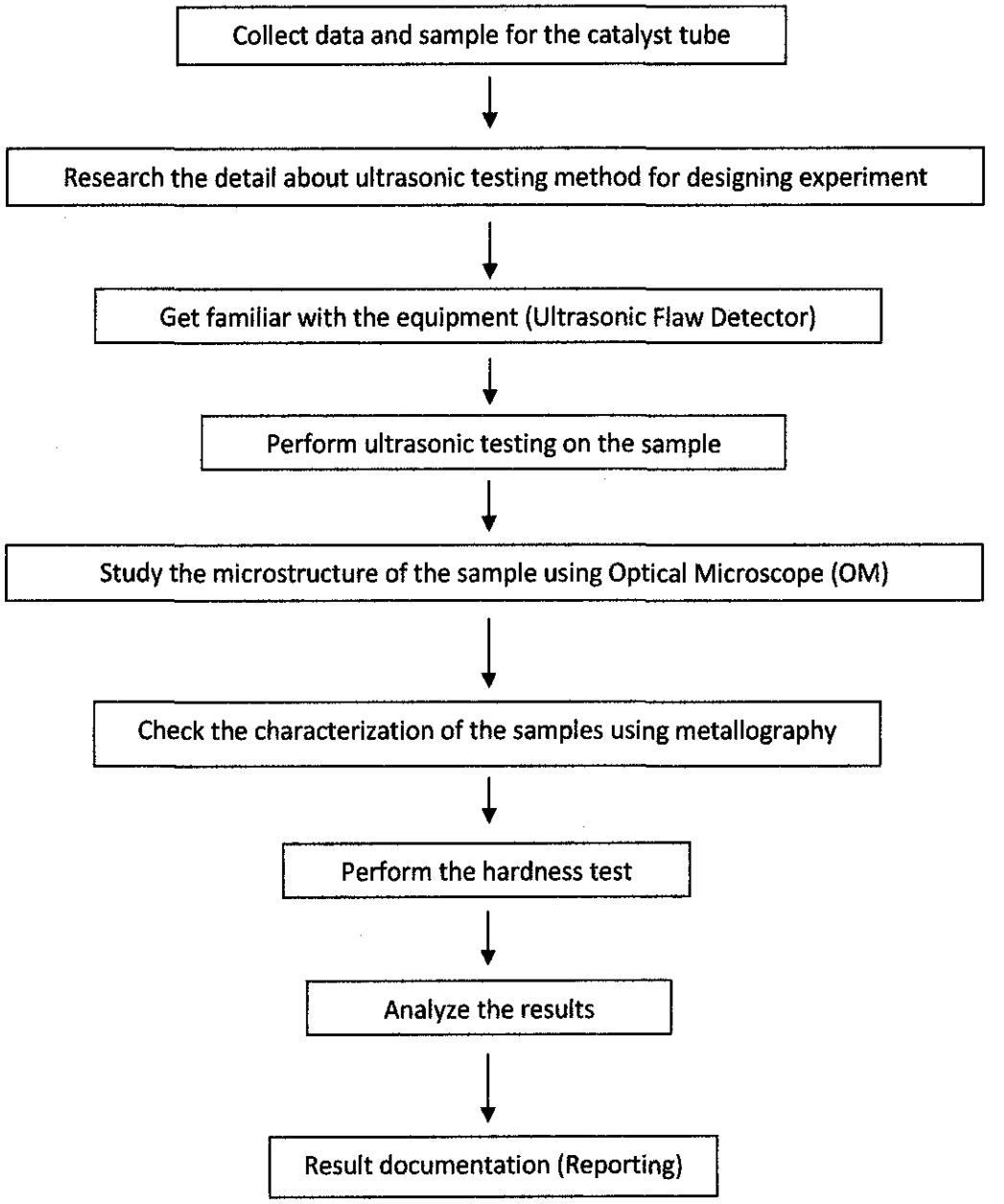

Figure 4: Process Flow Chart

3.2 MATERIAL

A608 - Standard Specification for Centrifugally Cast Iron-Chromium-Nickel High-Alloy Tubing for Pressure Application at High Temperature (34%Nickel, 20% Chromium)

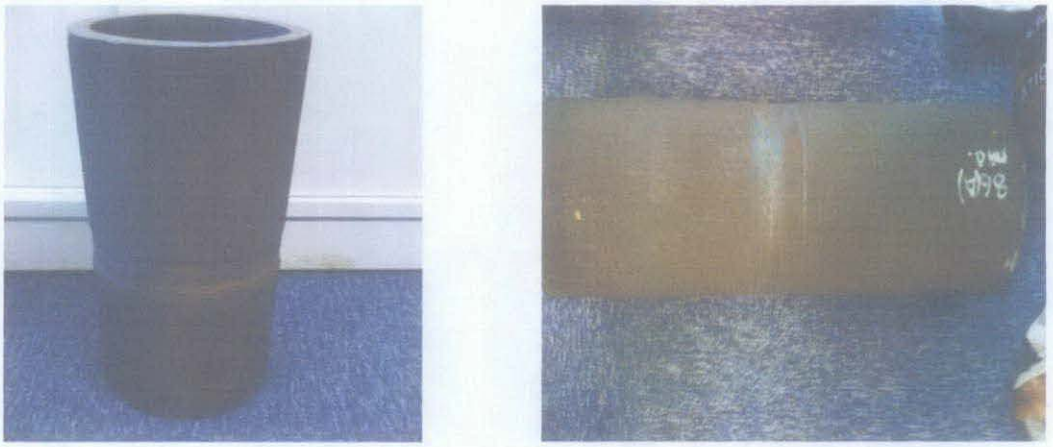

Figure 5: Catalyst tube

3.3 DATA COLLECTION

A visit to Petronas Fertilizer Kedah has been made to collect the data needed for the study. Three major data has been collected which is history of the catalyst tube, material specification of the tube and the process inside the tube. The material specification of the tube is A-608 which is a standard specification for centrifugally cast iron-chromium-nickel high alloy tubing for pressure application at high temperature. The tube was located in the furnace that operated at 772.75°C. During the 4 years operation, the tube has reached its design temperature, 910°C for two times which cause the catalyst tube to be replaced. The suspected reason of this condition was due to creep damage.

Catalyst tube data:

Table 1: Data of catalyst tube

Design Temperature (°C)	**910**
Design pressure, P (bar)	**39.20**
Allowable stress, Sr ($N/mm^{2)}$	**43.00**
Tube diameter, ID (mm)	**126.80**
Stress thickness, ts (mm)	**35.09**
Minimum required thickness (mm)	**41.49**
Outside diameter, OD (mm)	**152.00**

Process data:

Table 2: Process's data

Exit gas through the catalyst tube	
H_2, mole % dry	63.96
N_2, mole % dry	1.00
CO, mole % dry	8.86
CO_2, mole % dry	13.24
CH_4, mole % dry	12.84
Ar, mole % dry	0.10
Total	**100.00**

3.4 TOOLS AND EQUIPMENT

i. Ultrasonic flaw detector

ii. Metallography equipment

iii. Optical microscope

iv. Hardness Test

3.5 ULTRASONIC SOUND PROPAGATION

3.5.1 Ultrasonic Testing Calibration

Ultrasonic equipments used to develop weld or material flaw detection and sizing, need to be calibrated prior to the inspection, so one of the mandatory calibrations is the well known time base calibration that can be defined as the process developed to establish a correspondence between the time spend by the wave to go and come (time of flight) from a known reflector and the distance traveled. First, suitable probe need to be selected. For this study, 5 Mhz probe has been used to do the calibration. The diameter for this probe is 4 cm.

Figure 6: 5 Mhz probe

Ultrasonic velocity is one of the time base calibration variables that the operator need to set to perform the calibration. In general ultrasonic velocity is considered as a constant value relative to the material. Material velocity for the block is determined which is 5920 m/s. For this study, the calibration block that been used is calibration step wedge. Ultrasonic testing calibration is done each time before the testing be done on the samples. It is important to be done first on the samples because it helps to reduce the error in ultrasonic measurement.

Figure 7: Calibration step wedge

3.5.2 Ultrasonic testing on reformer tube

Ultrasonic testing is performed on the reformer tube. The tube is inspected using pulse-echo testing method. The tube has been lathed to three layers of thickness, 7.8 mm, 9.7 mm and 12 mm. The coating is removed from the tube because it produced so much scattering while doing the measurement. The testing on the catalyst tube been done using 5 Mhz probe. Grease has been used as a couplant to be applied on tube's surface.

Figure 8: Grease as a couplant

Figure 9: UT been performed on the tube

The equipment used in this ultrasonic testing is called ultrasonic flaw detector. The diagnostic machine display the result in the form of signal with an amplitude representing the intensity of reflection and the distance, representing the arrival time of the reflection. Firstly, the range value is set-up to ensure the measurement is easy to read. Usually the range is set-up two times larger than the thickness. For this study, the sound velocity of the material is unknown. By using the known distance, the sound velocity is determined by adjusting the value of velocity to get the right signal.

Figure 10: Ultrasonic flaw detector

3.6 METALLOGRAPHY

There are several tasks that have been accomplished in order to detect the damage mechanism on the tube. One of the tasks is to study the metallography of the chromium-nickel alloy catalyst tube. In accomplishing this task; there are several activities that need to be done such as cutting of specimen, mounting, grinding and polishing, etching and study the microstructure using optical microscope.

Cutting the sample - For microstructure study, the optical microscope cannot detect the microstructure of the outside coating of the tube. So, it is necessary to remove the coating. The tube has been turning to three layer of thickness which is 8 mm, 10 mm, and 12 mm. The coating at the tube surface also been removed using lathe machine.

Figure 11: Lathe machine

Figure 12: Tube after the turning process

The tube has been cut into 2 part which is half part will use for metallography study and another half will be used for ultrasonic testing inspection. The cutting of the tube been conducted using linear hacksaw machine. After been cut to half, a piece of the nickel alloy tube was cut from the each layer of the tube using electric discharge machining. The samples size was cut around 10mm x 10 mm for convenience during grinding and polishing.

Figure 13: Linear hacksaw machine

Figure 14: 10mm x 10 mm samples

Mounting of the sample – Mounting is necessary because the samples are small and it is hard to do polishing and grinding job without mounting. Hot mounting is chose for this study and this type of mounting used heat generation in compression mounting. Black powder is used as the mounting material. The advantage of mounting is that the edges of the specimen will be reasonably well polished and not "bevelled" by the preparation process.

Figure 15: Mounting's samples

Figure 16: Hot mounting machine

Grinding - The samples are ground on progressively finer SiC waterproof papers from 300 to 4000 grit, the reason for using different grit papers is to produce a reasonably flat surface; then the samples are lubricated with water to keep it cool and to remove the grinding products. If the samples are not flat, they might be necessary to remove some material on the lathe or grinding machine first. The sample should be moved forward

and backward on the paper until the whole surface is covered with unidirectional scratches. It is then washed with running water to remove debris associated with the grade of paper used. It is then ground on the next finer paper such that the scratches produced are at right angles to those formed by the previous paper.

Figure 17: Grinding

Figure 18: Grinder/polisher

Polishing – After finished with grinding, the next step is to do polishing on the samples. The purpose of polishing is to remove or disperse the scratch finely on the samples so that true structure can be observed. Polishing also is done using same rotating wheel like grinding but it used a cloth impregnated with a very fine abrasive compound. The compounds used are diamond. The samples are grounded on the on the lubricated rotating wheel and it is important not to press the sample on one orientation for a long time because it can cause the dragging of some microstructural components on the sample's surface. After 20-30 seconds the specimen is removed and rotated through 90°in the hand, placed back on the wheel and then again oscillated. The process of polishing is continued until the scratches is removed or dispersed finely.

Etching – For 34%Nickel 20% Chromium alloy, fry's reagent is used as the etchant material. Fry's reagent consisted of 30ml H_2O, 25ml of ethanol, 40ml of HCl and 5g of copper chloride. The purpose of etching is to reveal the grain boundary and the microstructure of the sample. Firstly, the samples were cleaned using alcohol and water and then being dried on the dryer. The purpose of this cleaning is to remove any impurities on the sample's surface. After that, fry's reagent was applied at the surface of

the specimen for 5 to 10 seconds. Then the samples were washed using water and alcohol. Lastly, the samples were dried and ready for microstructure examination.

Figure 19: Etching process

Figure 20: Fry's reagent

Microstructure Examination - The next step after etching is to do a microstructure examination on the samples using optical microscope. Firstly, the sample been put on the lens and then it been adjusted till the monitor come out with a good microstructure view. The brightness of the microscope also is adjusted until a fine picture of microstructure can be seen. The pictures of the microstructure were taken by using 500 x magnifications.

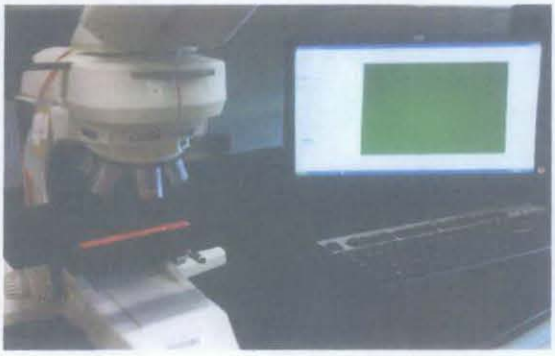

Figure 21: Optical microscope

Figure 22: Microstructure examination

Hardness test – Surface hardness of the samples is measured by using microstructure Vickers hardness. The measurement of the samples was taken for four times for each thickness's samples. The average samples were determined.

Figure 23: Microstructure Vickers hardness

CHAPTER 4

RESULTS & DISCUSSIONS

The results of the simplified creep assessment using ultrasonic testing, hardness test and microstructure information at different layers of the tube are shown below. The measurements were taken at the external surface (12mm), subsurface (9.7 mm), and inner layer (7.8 mm).

4.1 Ultrasonic testing result

4.1.1 Calibration result

Ultrasonic testing is calibrated and the result shown in Figure 24. The sound velocity for the block is 5920 m/s and it has a thickness of 10 mm. The frequency is constant which determined by the type of probe which is 5 MHz. The material used is normal carbon steel. No data is available for the sound velocity of new chromium-nickel alloy catalyst tube.

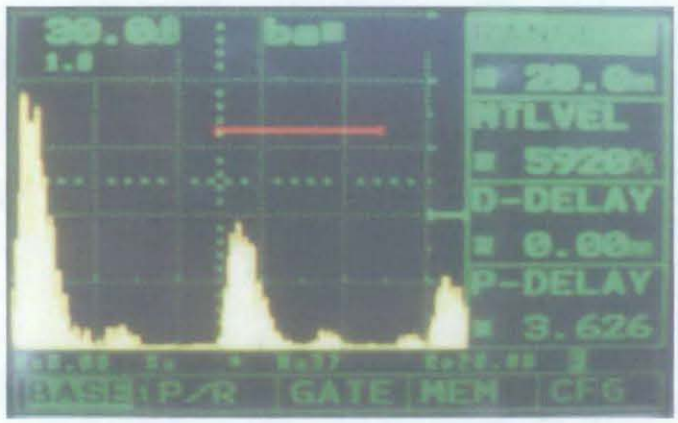

Figure 24: UT on calibration block t=10mm

4.1.2 Ultrasonic testing of creep damage tube

The ultrasonic testing of the external surface of the tube with the thickness of 12.0 mm is shown in Figure 25. The sound velocity for the sample was measured to be 6000 m/s.

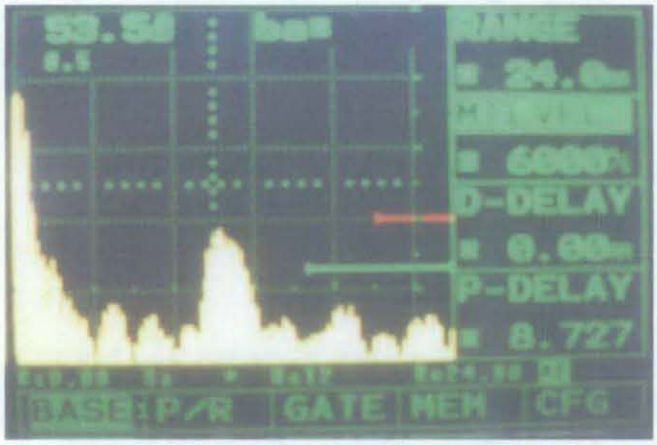

Figure 25: UT on Nickel Chromium alloy t=12mm

The ultrasonic testing of the subsurface of the tube with the thickness of 9.7 mm is shown in Figure 26. The sound velocity for the sample is measured at 5978 m/s.

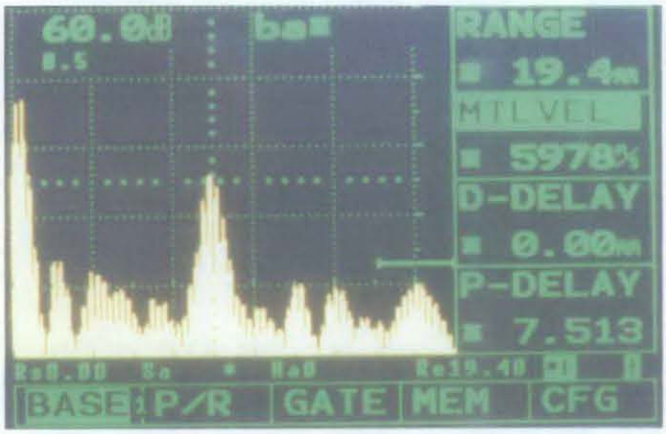

Figure 26: UT on Nickel Chromium alloy t=9.7mm

The ultrasonic testing of the inner layer of the tube with the thickness of 12.0 mm is shown in Figure 27. The sound velocity for the sample is measured at 5552 m/s.

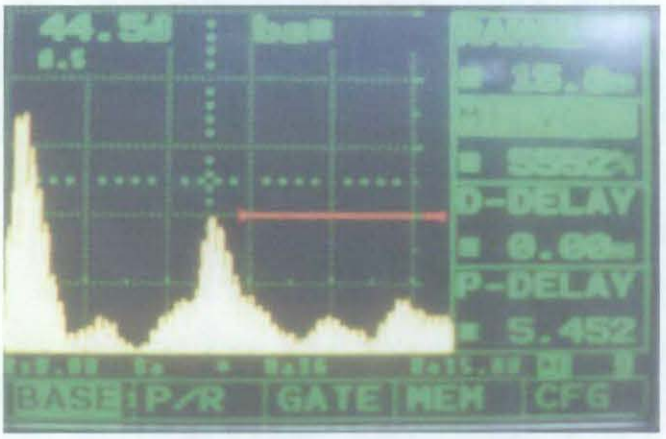

Figure 27: UT on Nickel Chromium alloy t=7.8mm

The results of the ultrasonic velocity on the different layer of thickness are summarized in Table 3 and Figure 28.

Table 3: Ultrasonic velocity

Sample thickness (mm)	**Ultrasonic velocity (m/s)**
7.9	**5552**
9.7	**5978**
12	**6000**

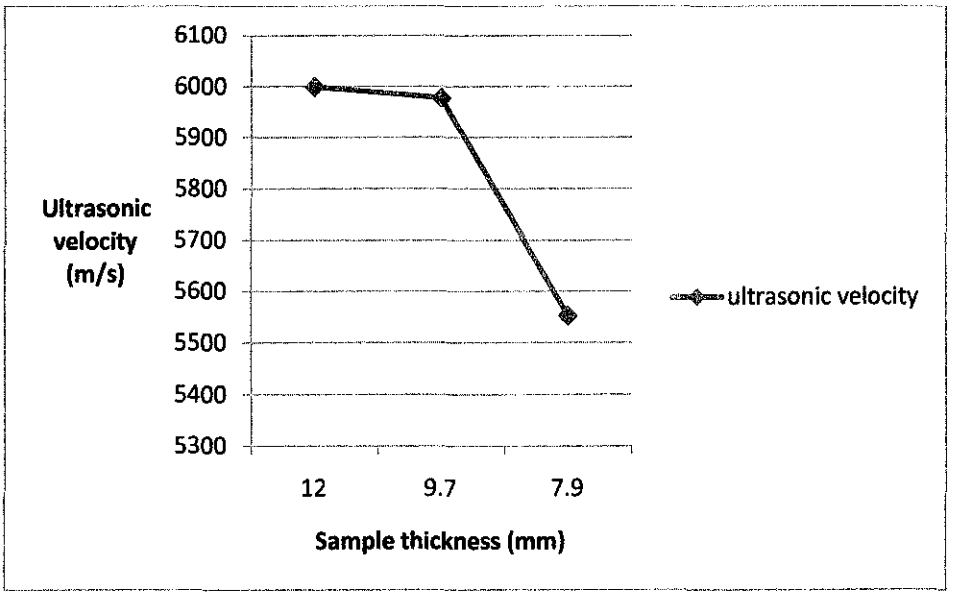

Figure 28: Ultrasonic velocity versus sample thickness

The ultrasonic velocity values are observed to be decrease with thickness, particularly for the inner layer. The values of sound velocity are influenced by the condition of the tube. The external tube and subsurface were in the same condition while the inner tube represented different condition. The condition of the tube can be determined by metallography study.

4.2 Metallography Result

Microstructures of different layer of thickness are obtained using optical microscope. The results are shown in Figure 29, Figure 30 and Figure 31. There are massive primary carbide in austenitic matrix and fine secondary carbides within the austenite grain. The microstructure shows the damage part of the tube which due to high temperature, secondary carbides were reduced and the inter-dendritic carbides had undergone significant agglomeration and coarsening. Microstructure also shows some random creep voids.

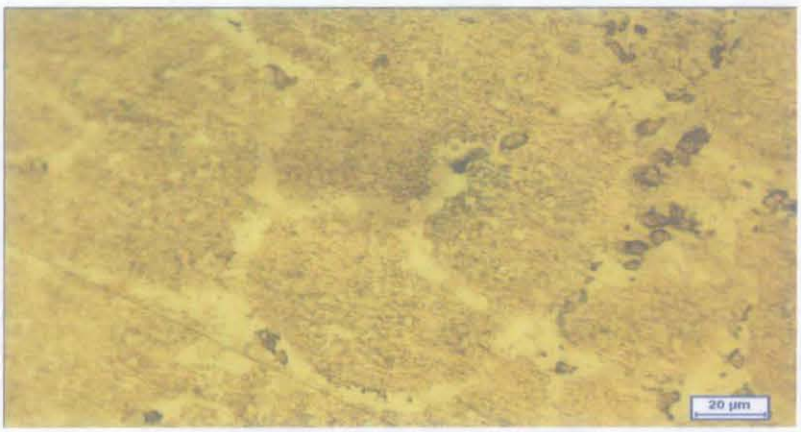

Figure 29: Nickel Chromium alloy t=7.8mm microstructure

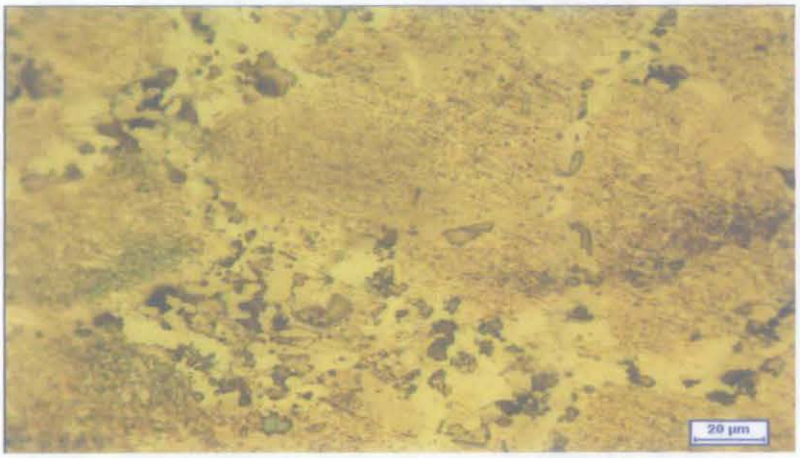

Figure 30: Nickel Chromium alloy t=9.7mm microstructure

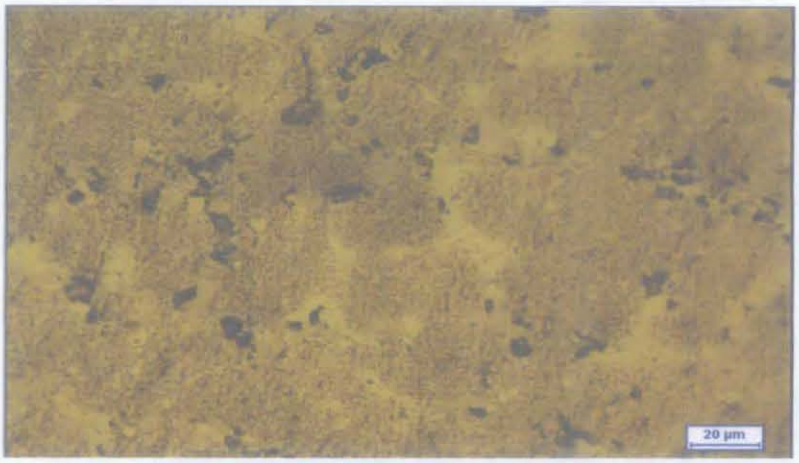

Figure 31: Nickel Chromium alloy t=12mm microstructure

The comparison of the grain size on different layer of thickness is summarized in Table 4 and Figure 32.

Table 4: Microstructure result

Sample thickness (mm)	**Grain size(µm)**
7.9	6.67
9.7	4.00
12	3.33

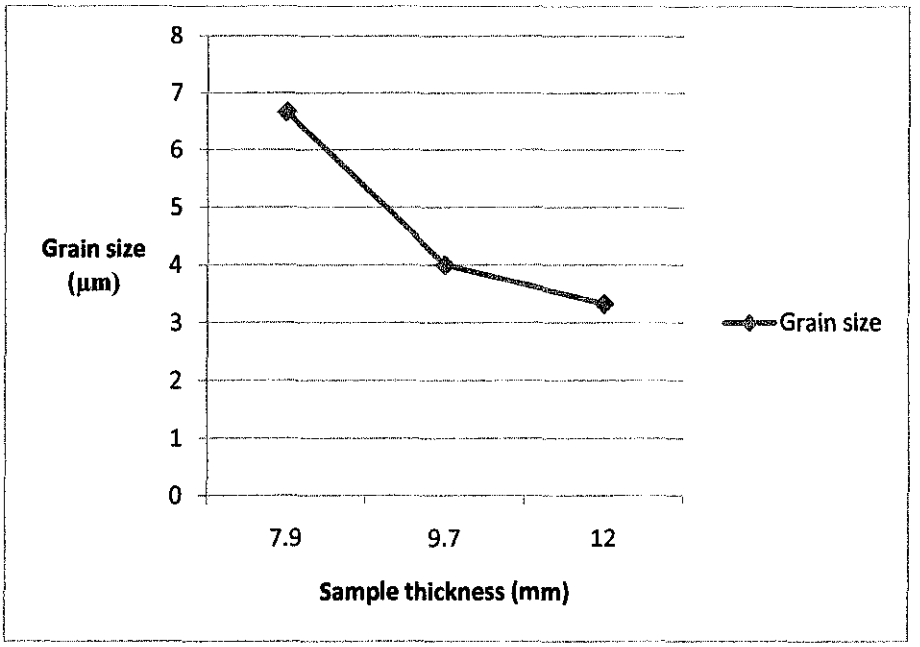

Figure 32: Grain size versus sample thickness

The grain size values are observed to be decrease with thickness. The mechanical condition of the different layer can be determined hardness test.

4.3 Hardness value

Vickers hardness test were performed on each reformer tube sample at 100 gf. Four reading for each sample surface were taken, in order to detect, an increase due to massive carbide precipitation. The means value from these readings for each samples are reported in Table 5. The hardness value of material in as-cast condition is 179 ± 7. The external surface layer of 12mm has the highest value which is 324.8 Hv while inner layer of 7.8 mm has smallest value which is 230.4 Hv. The results of hardness test are summarized in Table 5 and Figure 33.

Table 5: Data of Hardness Vickers Test

Thickness = 7.8 mm	**Thickness = 9.7mm**	**Thickness = 12 mm**
227.3	301.8	324.5
256.5	294.2	318.8
230.2	316.6	330.2
243.5	318.2	325.5
Average= 239.4 Hv	**Average= 307.7 Hv**	**Average= 324.8 Hv**

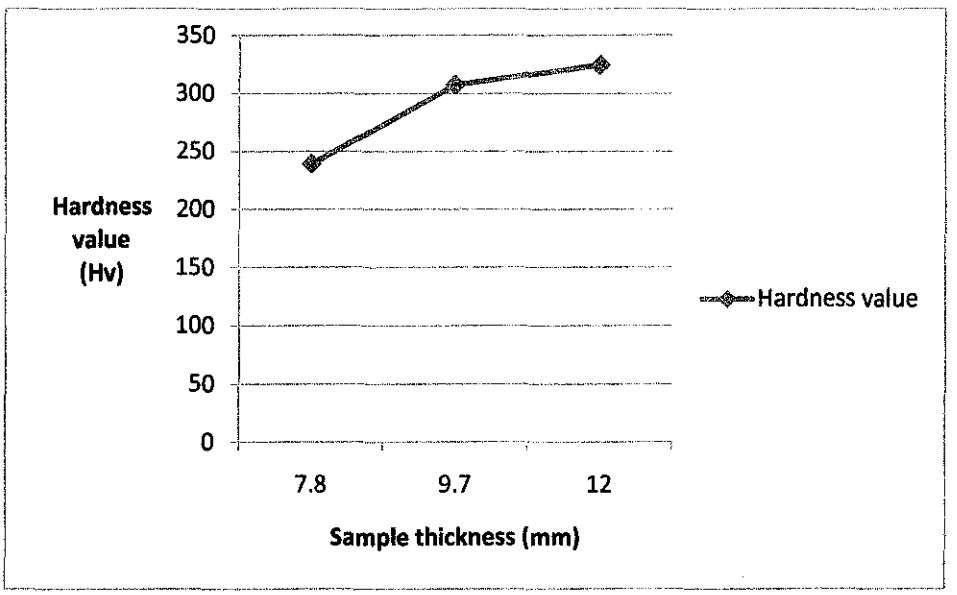

Figure 33: Hardness value versus sample thickness

The hardness values observed to be increased with thickness.

4.4 Discussion

A simplified creep assessment of the tube can be done by using ultrasonic testing, microstructure study and hardness test data. The measured data in terms of sound velocity, hardness and microstructure image can be compared with the base data of new tube. In this study, however, the base data of new tube is not obtained. Without the data, the comparison between the microstructure of virgin material and the tube cannot be done.

CHAPTER 5

CONCLUSION

Based on the ultrasonic testing and microstructure study that has been made on the nickel chromium alloy catalyst tube, the results showed that there were damage mechanism occurred on the microstructure's samples. This study has presented the potential of ultrasonic for characterizing creep damage in the reformer tube. Preliminary results using ultrasonic testing are very encouraging. Ultrasonic velocity demonstrated high sensitivity to the creep progress. There is clear relationship between the ultrasonic velocity and the grain size. Present sound velocity measurements that have differences value since significant variability exists in the samples prior to creep damage. An added advantage of ultrasonic testing can be detection of cracks deep within the specimens. For metallographic study, by using optical microscope, the data providing information about the grain size and the precipitation of carbide involved in the tube surface. The increase of hardness directly also correlated to microstructural features of the material.

Thus, the objective for this study is achieved by monitoring the velocity in ultrasonic testing and the microstructure for reformer tube evaluation. This technique has a potential to assess the creep damages of the reformer tube.

RECOMMENDATION

The study can achieve more accurate result by using more sensitive probe. The sensitivity of probe is in relation with the frequency. Suitable frequency will give more accurate result on the ultrasonic testing regarding its amplitude, wavelength and velocity. The study can also try out other ultrasonic testing methods such as immersion and contact by using dual probe or normal beam and angle beam technique. Study using ultrasonic testing to detect other damage mechanisms such as localized corrosion, erosion corrosion and thickness variation can also being carried out for future study.

MILESTONES FOR FYP 1
PROJECT TITLE: ULTRASONIC INSPECTION OF CENTRIFUGALLY CAST IRON-CHROMIUM-NICKEL HIGH ALLOY CATALYST TUBE DUE TO CREEP MECHANISM

NO	DETAIL	WEEK													
		1	2	3	4	5	6	MID-SEMESTER BREAK	7	8	9	10	11	12	13	14
1	Primary Research Work															
2	Submission of Preliminary Report				●											
3	Data collection															
4	Do research about ultrasonic testing and creep mechanism															
5	Familiarization of ultrasonic testing															
6	Submission of Progress Report									●						
7	Seminar (compulsory)									●						
8	Submission of Interim Report Final Draft															●
9	Oral Presentation(study week)															

MILESTONES FOR FYP 2

PROJECT TITLE: ULTRASONIC INSPECTION OF CENTRIFUGALLY CAST IRON-CHROMIUM-NICKEL HIGH ALLOY CATALYST TUBE DUE TO CREEP MECHANISM

No	Details/week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Perform ultrasonic testing on the tube															
2	Do metallography examination on the tube															
3	Study the relation of ultrasonic testing and metallography study															
4	Submission of progress report															
6	Submission of draft report															
7	Submission of dissertation															
8	Submission of technical paper															
9	Oral presentation															
10	Submission of project dissertation (hard bound)															

REFERENCES

[1] Ulrich Heubner, 1998, Nickel Alloys, Marcel Dekker, Inc., New York.

[2] Ashok Kumar Ray, et. al., 2002, Analysis of failed reformer tubes, *Engineering Failure Analysis 10 (2003) 351-362.*

[3] M. E. Kassner, 2009, Fundamentals of creep in metals and alloys, Elsevier Ltd., UK.

[4] Tito Luiz da Silveira, Iain Le May, 2006, Reformer furnaces: Materials, damage mechanism and assessments, *The Arabian Journal for Science and Engineering 31. 2C.*

[5] D. R. H. Jones, 2004, Creep failures of overheated boiler, superheater and reformer tubes, *Engineering Failure Analysis 11 (2004) 873-893.*

[6] T. J. Wardle, 2000, Creep rupture assessment of superheater tubes using nondestructive oxide thickness measurement, The Babcox and Wilcox Company, USA.

[7] G. R. Lobley, A. Kermad, et. al., 2003, Assesment of aged steam reformer furnace, Cape.

[8] Brian Shannon, June 2004, <http://www.ndt.net/article/mendt03/shannon/shannon.htm>

[9] R. Martinez-Ona, 2000,
<http://www.ndt.net/article/wcndt00/papers/idn238/idn238.htm>

[10] <http://www.ndt-ed.org/EducationResources/CommunityCollege/Ultrasonics/Introduction/description.htm>