CHARACTERIZATION OF REFORMER TUBE CREEP DAMAGE BY ULTRASONIC ATTENUATION PATTERN AND MICROSTRUCTURAL CHANGES

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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CERTIFICATION OF ORIGINALITY

With this I clarify that this report was originally produced except the specified references and acknowledgement and the original work contained herein have not been undertaken or done by unspecified sources or persons.

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ABSTRACT

Creep damage is a critical degradation mechanism in reformer tubes which experienced prolonged service periods at elevated temperature. Thus, continuous monitoring of creep is vital in ensuring plant integrity. Since creep damage relates to the changes in microstructure, characterization of creep using common ultrasonic attenuation and metallography techniques can be used. This project utilized the attenuation of ultrasonic wave in different tube samples and changes within the microstructure in characterizing the level of creep damage. The study was based on a failed reformer HK-40 catalyst tube by which the tube was cross-sectioned to the thickness corresponding to exposed temperature calculated based on heat transfer formulation. The sample was characterized in terms of ultrasonic wave attenuation, metallography and micro hardness. Results had shown that tube sample which had been exposed to higher temperature produced higher attenuation of ultrasonic wave, significant increase in carbide formation and hardness value while revealing a decrease in grain size than the sample exposed to lower temperature. Based on the results, it is clear that tube sample which had been exposed to higher temperature exhibits the characteristic of creep damage in tertiary stage, while the tube sample exposed to lower temperature are proved to be in secondary stage. In conclusion, creep damage can be evaluated based on ultrasonic wave attenuation and microstructural analysis.

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In the name of Allah, Most Gracious, Most Merciful

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1.0 INTRODUCTION

1.1 Background of Study

Creep damage is a critical degradation mechanism in reformer tubes which experienced prolonged service periods at elevated temperature. This time-dependent deformation of materials is often a key factor not only in the design of components used in the power generation industry, particularly in fossil fuel plants and nuclear reactors, but also in the assessment of their remaining life. Typical examples of failures are found in power plant parts such as catalyst and reformer tubes, turbine rotors and casings. As the demands on industry grow, prolonged service lives of machinery and the ability to perform at higher operating temperatures have become necessary. At present, lifetime prediction can only be made with large uncertainty [1]. This is because the creep process is critically sensitive to the microstructures of the materials as well as to the details of operational environment history. In-service assessment of the damage state is thus important for ensuring safe operation, predicting the remaining life, and promoting a life extension program.

It is reasonable to assume that, in components subjected to more or less uniformly distributed mechanical and thermal stresses, creep damage will also be widespread (volumetric creep) and multiple cracks will generally appear over a relatively large area [2]. This type of creep damage is particularly important in power plants which are approaching their design life, or indeed operating beyond it. Recently, there was case related to this study where a damaged catalyst tube in PETRONAS Fertilizer Kedah (PFK) chemical plant is operated at the temperature around 910°C inside a reformer.

After four years of service, the tube had ruptured before it reaches the theoretical life expectancy of 100,000 hours due to the formation of creep damage where the tube had then been replaced after the reformer had been shutdown. However, the issue remains where it is vital to accurately predict the remaining lives of these tubes. Among the consequences of improper assessment of creep damage can be categorized in regard of financial and safety. Financially, untimely retubing process causes unnecessary capital losses while unscheduled equipment shutdown reduces plant productivity. For safety concerns, it is clear that creeping tubes will bulge, split and rupture and in severe cases, high pressure and temperature will cause a blow down to the equipment.

Currently, there are several inspection methods able to assess the condition of creep damage in the tube. In this study, a simpler and conventional inspection method via non-destructive tests (NDT) namely ultrasonic (UT) test and microstructure study will be used. In most of the previous researches applying ultrasonic method to the evaluation of creep damage in metal structural components, the decrease of ultrasonic attenuation due to the formation of creep cavity was reported. Measurement of ultrasonic attenuation, together with improved theoretical models, allows the porosity of the material and the grain size and concentration of cavities to be determined. In this research, it was attempted to evaluate the correlation between the results and data obtained from a microstructure study so as to indicate the usefulness of the method for in-service evaluation of creep damage.

1.2 Problem Statement

Continuous monitoring of creep is critical in ensuring plant integrity. There is a need for a simplified methodology using common non-destructive testing to be used to assist plant engineer to characterize creep damage.

1.3 Objectives and Scope of Project

To evaluate creep damage based on microstructural degradation in relation to exposed temperature in terms of :-

a) Ultrasonic wave attenuation analysis and
b) Microstructural changes analysis

This project covers the assessment of creep damage behavior and its characterization by ultrasonic attenuation and microstructure study. Ultrasonic testing parameter will be modeled using the difference of its attenuation. Preparation of samples from creep material will be done to study the microstructures as to determine the grain sizes and presence of creep voids. Equipments to be used in the project consist of:-

1. Ultrasonic Testing Flaw Detector (UTFD)
2. Optical microscope
3. Lathe machine
4. Linear Hacksaw Machine
5. Micro Hardness Testing Machine

1.4 Relevancy of the Project

This project covers the area of study where relationship between material degradation and results of non-destructive tests are examined. Proper analysis of this study will help the author to put theoretical knowledge learned throughout the course of Mechanical Engineering into practical use. In directly, positive results from this study will be able to give Inspectors and Reliability Engineers in power plants and industries a new insight on how to measure and observed creep damage efficiently without the need to consult a third-party consultant.

1.5 Feasibility of the Project

The overall timeliness of this project covers the duration of two semesters (8 months) and is broken down into two subjects namely Final Year Project I and II. By referring to the Gantt chart and key milestones provided in chapter 3.2, this project is expected to be completed over the course of 8 months. Basically, the overall project combine both research and laboratory work. For the research, the resources can be obtained mainly from UTP Information Resources centre and internet. Many books and journals can be found from both sources. For the laboratory work, all the materials and equipment needed can be accessed within UTP facilities in timely manner. The project is expected to be done smoothly if the procedures are followed accordingly.

2.0 LITERATURE REVIEW / THEORY

2.1 Creep Damage Mechanism on Catalyst Tube

Time dependent plastic deformation of materials under elevated temperature subjected to a constant stress for an extended period is generally denoted by the term creep, one of the main leading causes of catastrophic failure of components operating inside high-temperature equipments [2]. Many extensive reviews of creep mechanisms are available in various books such as Fundamental of Creep in Metals and Alloys [3] and Fatigue and Durability of Materials at High Temperatures [4]. It is fundamental to note that creep occurs even if the applied stress is much lower than the room temperature yield stress. It can be seen that under this condition, one can classify three different stages of creep life characterized by a curve in a plot of strain versus time graph as in Figure 2.1. These stages of creep lives can be segmented into three zones knows as primary (Stage I), secondary (Stage II) and tertiary (Stage III) creep [1].

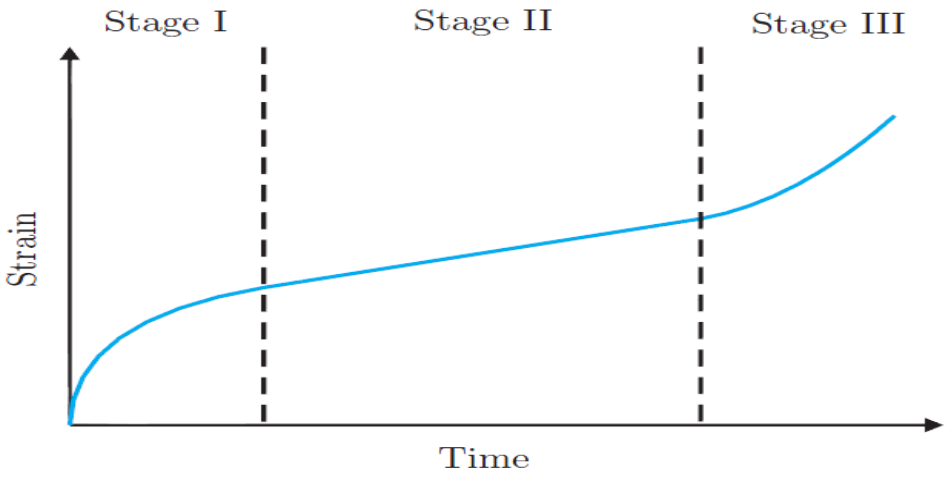

Figure 2.1: Stages of creep life [4]

During Stage I, the material initially reacted to a load by experiencing instantaneous strain deformation followed by a decrease of the strain rate [4]. In this state, primary creep occurs early in tube life, and its rate decreases as progressive work-hardening inhibits dislocation movement.

After the primary creep, Stage II, secondary creep will then takes place [4]. This is the condition under which the materials are designed to operate. Known as steady-state creep, the tube's elongates while the diameter decreases at a constant slow rate.

Following that long a period of constant strain rate in secondary creep, the strain rate is noted to accelerate again, eventually causing rupture of materials in Stage III [1]. Tertiary creep is the final creep stage, as at this point, cracks within the structure are forming at its highest rate. Martinez R., et al. [5] in his research of creep damage in reformer tubes suggests that creep damage first develops voids, coalesced voids, and then micro cracks and macro cracks with the increasing of creep damage stages.

2.2 Non-Destructive Testing (NDT)

Non-destructive testing (NDT) is an extensive collection of analysis methods performed in science and industries by the use of noninvasive techniques to determine the integrity of a material, component or structure or quantitatively measure some characteristic of an object [6]. NDT is a quality assurance management tool which can give impressive results and is necessary to ensure safety and reliability when used correctly. The demands for NDT are crucial on operation units when the operating conditions are harsher where high temperature and pressure are involved, while ensuring employee's safety, unit reliability and product quality are maintained all together. NDT also provides a convenient solution to manufacturers and operators as it can be applied to on-stream equipment as representative of current condition without the need to alter the material and components integrity. Repeated tests will also be the other benefits of NDT for the purposes of evaluation material degradation over time [6].

2.2.1 Analysis of Ultrasonic Wave Attenuation

Throughout NDT applications in lab and field conditions, UT had recorded a long success history in terms of a tool for early detection of creep damage. Techniques based on measurements of the back-wall echo, namely wave attenuation, will give results averaged over its entire thickness of the bulk object, and can therefore be used for the evaluation of volumetric creep [7]. Basically, ultrasonic wave is transmitted through the work piece until it is reflected by a surface, discontinuity or other obstruction. Reflected wave is converted from an ultrasonic wave to an electrical signal by the transducer and finally, time history of ultrasonic wave activity is used to identify discontinuities in the material [7].

Among the major applications of UT is that it is used to measure part thickness as a method for corrosion monitoring and quality control. The attenuation and velocity changes in acoustic wave assist will further assist inspectors in evaluation of material properties and in service damage such as creep [8]. This method is particularly chosen as it is the most accurate and rapid method for single sided access during inspection.

Ogi et al. [9] monitored the ultrasonic wave attenuation of fatigued steel samples and observed that early damage indications could be picked up by the ultrasonic technique. Ohtani et al. [10] evaluated creep damage accumulation in steel welds using the second harmonic non-linear ultrasonic method and micro-structural observations. Using an immersion technique, they observed increased amplitudes of harmonics around the heat-affected zone (HAZ), which corresponded to the regions of high creep void density, as confirmed through microscopy.

Martinez R., et al. [5] in his study of correlating ultrasonic attenuation and microscopy of crept Centrifugal cast austenitic material had shown that material with creep damage will change the ultrasonic attenuation from its standard value, depending on the severity of the damage. Attenuation in ultrasonic wave greatly assists evaluation process of material properties and in service damage where high attenuation is related to high level of creep damage and low attenuation to low level of creep damage.

The difference in ultrasonic wave amplitudes acting on different level of creep damage is shown in Figure 2.2 and 2.3 [5].

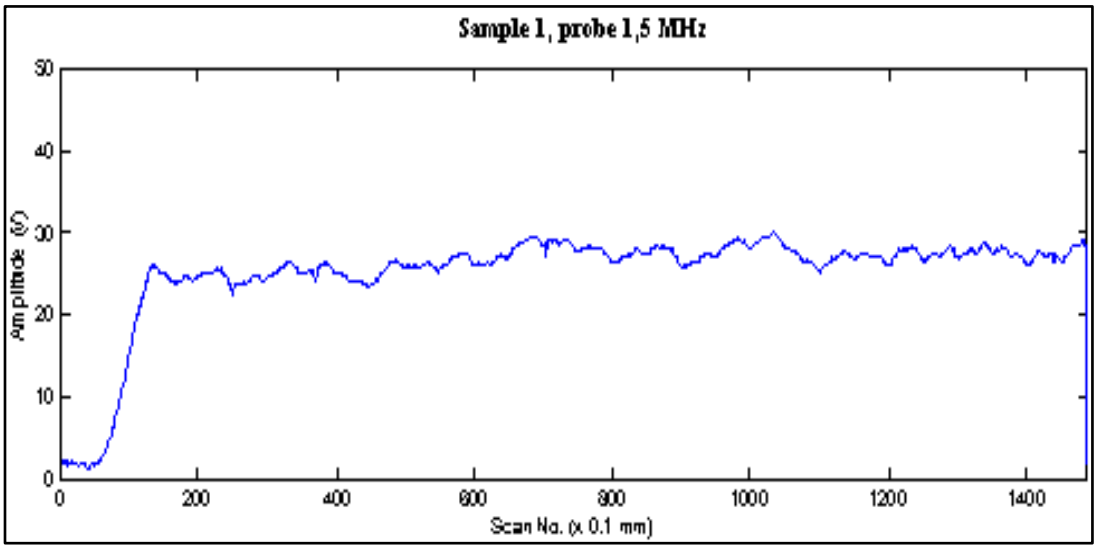

Figure 2.2: Sample 1, secondary creep voids (low damage degree) [5]

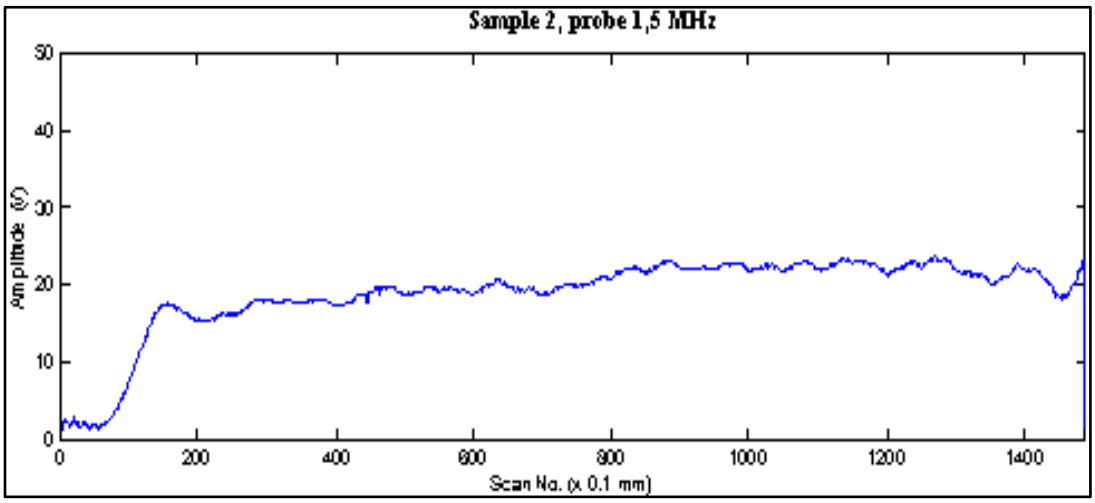

Figure 2.3: Sample 2, secondary creep aligned voids (high damage degree) [5]

However, there is some controversy on the possibility of using ultrasonic wave attenuation measurements for the detection of creep damage. This technique has been considered less promising than ultrasonic wave velocity because small variations in

grain size would produce larger variations of the attenuation coefficient than creep pores [11, 12].

Some authors claim however that the attenuation coefficient presents a larger sensitivity to creep damage than the wave velocity [13, 14]. In the work of Ohtani [10], the attenuation coefficient is shown to vary non- monotonically but with a certain relationship with creep stages: in particular, a maximum is reached at 20–60% of creep life, depending on the material.

2.2.2 Analysis of Microstructure Study

Microstructure study is the most commonly used method for creep damage detection. Its application to the study of this type of damage was found in the early 1980s [9]. The surface to be inspected must be prepared by careful polishing and etching; a film is then applied to it, softened with an appropriate solvent and left to solidify, so that it can be viewed usually with high magnification to identify flaws such as voids and microcracks. This technique can provide quantitative data on the defects, and voids of 1 mm or less can be easily distinguished on the replica in laboratory conditions [15].

It is commonly assumed that voids can be detected at a relatively early stage of creep. However, some studies have reported that in any materials, cavities may be detectable on the surface only shortly before fracture [16]. In sample preparations of replica, there are several standards can be referred. These standards are ASTM E 3-01, ASTM E 1351–01 and ISO 3057. ASTM E 3-01 gives the guideline in the preparation of metallographic specimens. ASTM E 1351–01 [11] is the standard practice for production and evaluation of metallographic replicas which proposed a description of various creep damages as shown in Table 2.1

Table 2.1: Description of damage classes [11]

Class	Structural and damage conditions	Action
0	As received, without thermal service load	None
1	Creep exposed, without cavities	None
2	Advanced creep exposure, isolated cavities	Re-inspection after approx. 20,000 hours in service
3	Creep damage, numerous oriented cavities	Re-inspection after approx. 15,000 hours in service
4	Advanced creep damage, microcracks	Re-inspection after approx. 10,000 hours in service
5	Large creep damage, macrocracks	Rejected.

3.0 METHODOLOGY

3.1 Project Flow Chart

The project is undertaken with the sample obtained from Petronas Fertilizer Kedah (PFK) involving sample preparation, ultrasonic testing and microstuctural study on tube samples, micro hardness test and finally the analysis and correlation of results. The steps are shown in Figure 3.1.

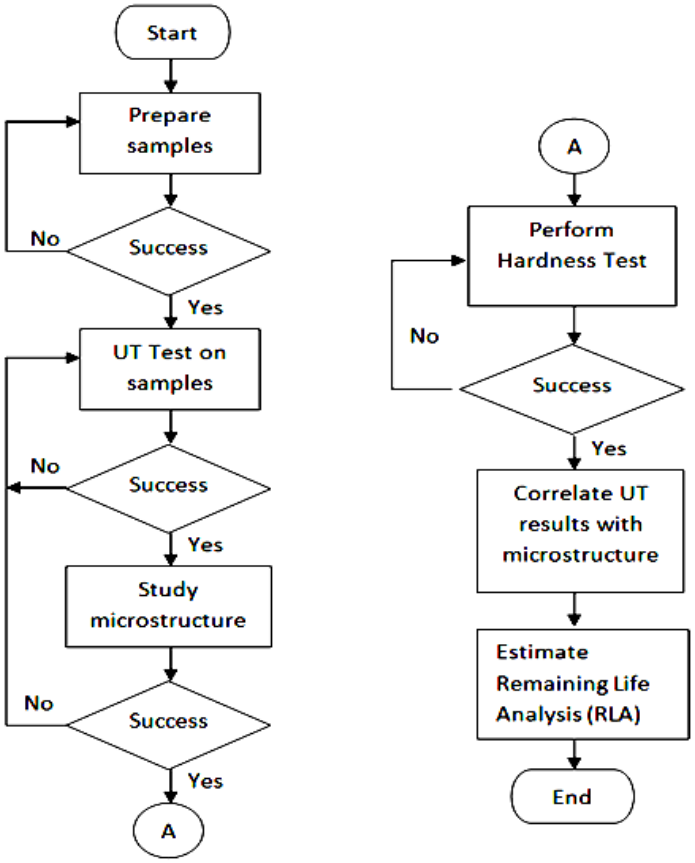

Figure 3.1: Process flow chart of this study

3.2 Sample Preparation

3.2.1 Failed catalyst tube sample from Petronas Fertilizer Kedah

Figure 3.2: Failed catalyst tube sample from PFK [18]

Information on the reformer tube sample had been collected; the tube's operating envelope and its history, material specification and physical and chemical processes taking place within the tube. The tube follows the material specification of ASTM A608 steel (HK 40 type), a standard for an austenic iron (Fe) –Chromium (Cr) –Nickel (Ni) alloy, centrifugally casted for high pressure and high temperature application. This sample is provided by Petronas Fertilizer Kedah (PFK) after having their latest 2012 plant turn-around, where several of their catalyst tubes were replaced due to occurance of creep damage after 40,000 hours of service. Further details on these data are shown in Chapter 4.1 in this report.

3.2.2 Calculation of Corresponding Temperature to Tube Thickness

Characterization of creep damage via ultrasonic testing and microstructure study of the tube requires it to be cut to several thicknesses, in order to determine the presence and severity of creep damage. The thickness dimension of the tube had been determined utilizing the theory of heat transfer via conduction. Heat conduction through the walls of a tube is normal to the direction of tube surface.

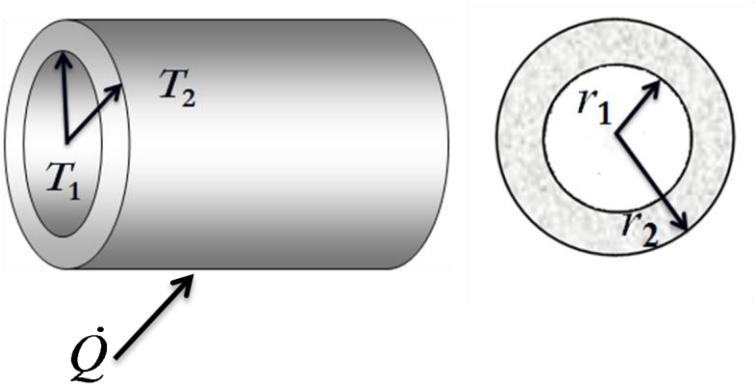

Figure 3.3: Heat conduction through a cylindrical tube [2]

Assuming steady state and one dimensional heat transfer through a tube, Equation 1 is used to determine the tube thickness and the corresponding temperatures acting on its surface.

$$\dot{Q}_{cond} = -kA\frac{T_1 - T_2}{\Delta r} \qquad \text{.....Equation 1}$$

where

$\dot{Q}$ = Heat transfer per unit time

k = Thermal conductivity

T_1 = Temperature of tube product

T_2 = Temperature of ambient

Δr = Change in thickness

Based on Equation 1, the values of temperature acting on different layers of tube surface is dependant on the difference of the tube thicknesses. Therefore, it is calculated that at at the outermost surface of the tube with thickness of 13mm, maximum exposed temperature is 910^0C, which is the ambient temperature of the reformer while at thickness of 6.5mm, the temperature is found out to be 773^0C.

3.2.3 Sample Cutting and Turning Process

Figure 3.4: Cutting of tube using linear hacksaw machine

The tube must be cut in half using a linear hacksaw machine (Figure 3.4) so that the two parts obtained can be experimented separately in ultrasonic testing and microstructure study. After that, the tube is put under turning process in order to produce a tube with no coating (bare metal) and two layers of thicknesses, 6.5mm and 13mm respectively.

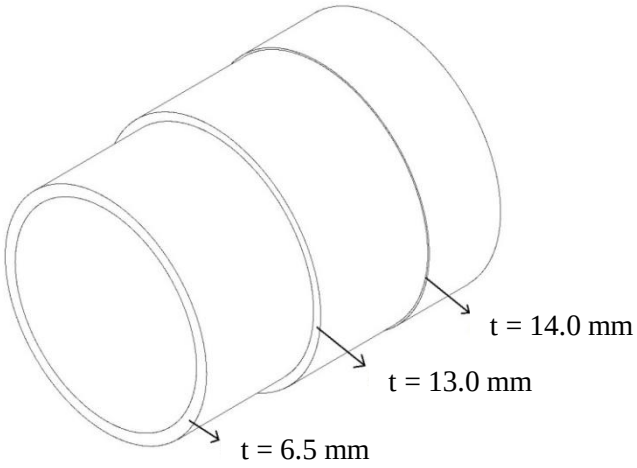

Figure 3.5: Design showing several layers of thicknesses of catalyst tube

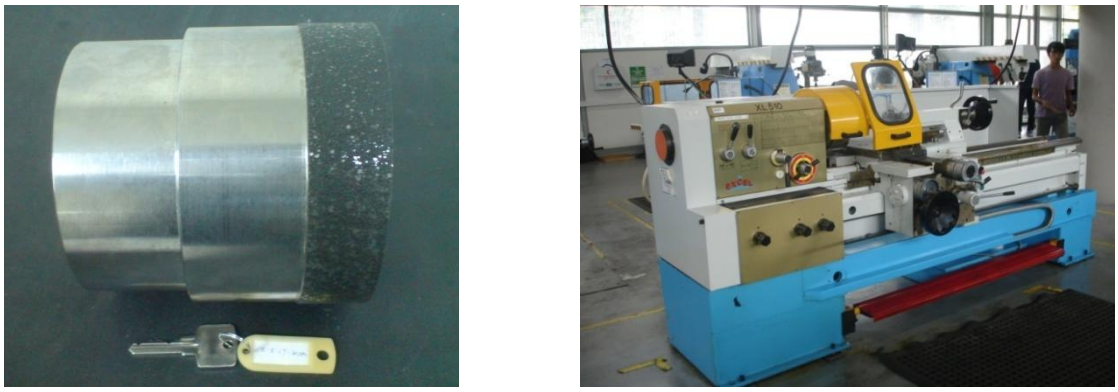

Figure 3.6: Tube after turning process via lathe machine

3.3 Ultrasonic (UT) Test on Samples

Figure 3.7: Performing UT on catalyst tube

The equipment used in this ultrasonic testing is called ultrasonic flaw detector (UTFD). The diagnostic machine display the result in the form of signal with amplitudes representing the intensity of reflection, attenuation and distance travelled, representing the arrival time and velocity of the ultrasonic wave. Without proper experimental procedure, results obtained via this method can be very unsteady and wavering. Step-by-step approach was done throughout this experiment to ensure the accuracy and consistency of the end results.

Firstly, probe selection is done whereby the 5 Megahertz (MHz) with 4 mm diameter probe is chose. This step is important as probe selection will affect the physical properties of the UT wave. By choosing the low frequency probe with 5MHz, the wave is expected to have longer wavelength, higher depth of penetration and less noise, but with less resolution.

Figure 3.8: 4mm diameter and 5MHz probe is selected

Then, the UTFD machine is calibrated using a step-wedge prior to inspection on the tube. This served as one of the most critical steps as this equipment can be reconfigured anytime at the user's will for use in variations of cases.

Figure 3.9: UTFD machine is calibrated using a step-wedge as shown

Third, UT testing is done on the layers of the tube which are of 6.5mm and 13mm thicknesses. Before adjusting probe onto the surface of the tube, it is necessary to apply grease as a couplant. Couplant here will act as a medium to focus the ultrasound wave directly to the surface of metal and to reduce scattering which could cloud the actual readings.

Figure 3.10: Grease is used as a couplant

The probe is adjusted so that most of its surface fits well onto the metal surface as to eliminate any unwanted noise. The sound energy (decibels), range (mm) and velocity (m/s) of the ultrasound wave can be adjusted manually until a consistent set of waves are seen on the display of UTFD. The velocity readings were then taken when the wave no longer fluctuates.

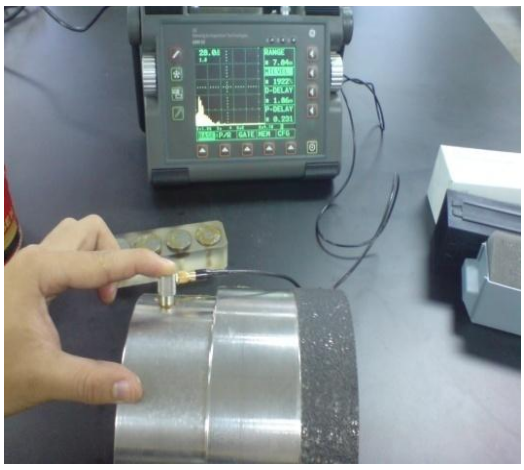

Figure 3.11: Probe is adjusted on tube

Figure 3.12: Example of consistent set of waves

3.4 Microstructure study of samples

Microstructure study of tube samples requires extensive preparation procedures in order to achieve reliable results. The steps for sample preparation are shown on Figure 3.13.

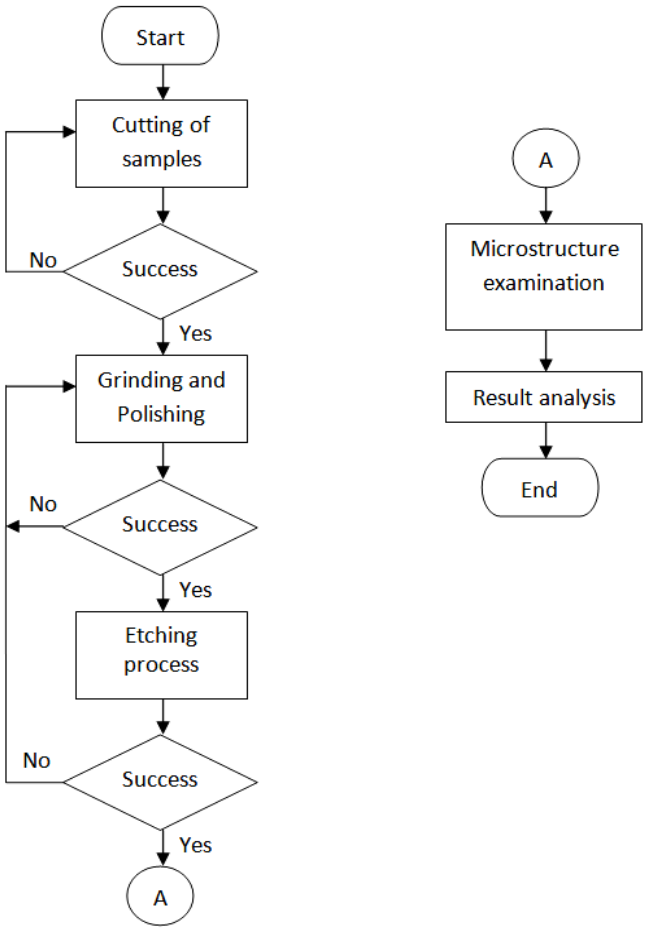

Figure 3.13: Steps for sample preparation

The cutting of samples into small rectangular sizes into dimension of 15mm x 10mm was done using a linear hacksaw machine. Then, it is ground using a grinding machine to produce a flat and smooth surface on progressively finer silicon carbide, SiC waterproof sandpapers ranging from 240 to 4000 grit while being lubricated uniformly throughout the process to avoid the samples from being overheated.

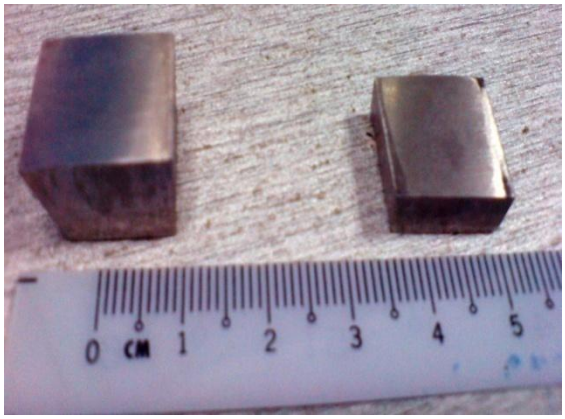

Figure 3.14: Rectangular samples

Figure 3.15: Grinding

Next, polishing process is done after the surfaces of the samples had been evenly flattened and smoothened. The purpose of polishing is to remove or disperse the scratch finely on the samples so that true microstructure can be observed. Polishing is also done using same rotating wheel-like grinding apart from using a cloth impregnated with a very fine abrasive compound made from water-based diamond particles, known as diamond paste. The samples are grounded on the on the lubricated rotating wheel. It is made sure that the sample must not be pressed on one orientation for a long time as 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 are dispersed finely and the samples exhibit mirror-like effect.

After polishing, the samples are required to be etched first before microstructure study is to be done in order to completely reveal the grain boundary and structures of the sample. The etchant chemical suitable for ASTM A608 nickel-chromium alloy is Fry's reagent as shown in Figure 3.16 [15]. It can be prepared by mixing 30ml of water (H_2O), 25ml of ethanol, 40ml of hydrochloric acid (HCL) and 5g of copper chloride ($CuCl_2$). Surface of the samples must be cleaned using alcohol and water prior to applying etchant to remove any debris. Then, the etchant chemical is applied on to the surface of the samples using a cotton bud immersed in Fry's reagent as shown in Figure 3.17.

After evenly spreading the etchant, the surface must be left for about 10-20 seconds, before washing it using water. The samples are then wiped clean and dried.

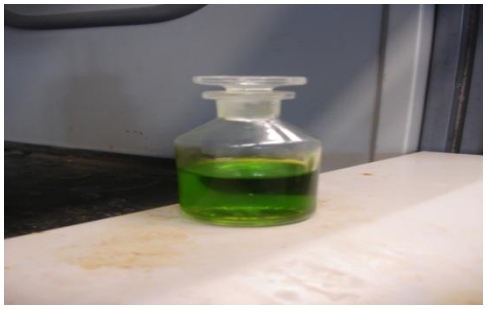

Figure 3.16: Fry's Reagent

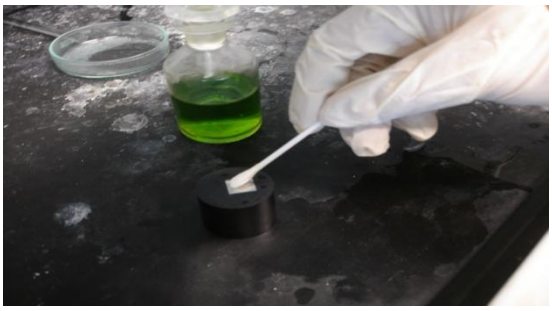

Figure 3.17: Applying etchant on surface

After the sample preparation processes, the samples are put under optical microscope with 500x magnification. Images of the grain structures are captured and will be further discussed in Chapter 4.4.

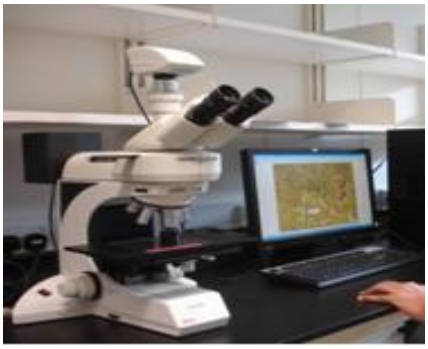

Figure 3.18: Optical microscope and microstructure study

3.5 Micro Hardness Test

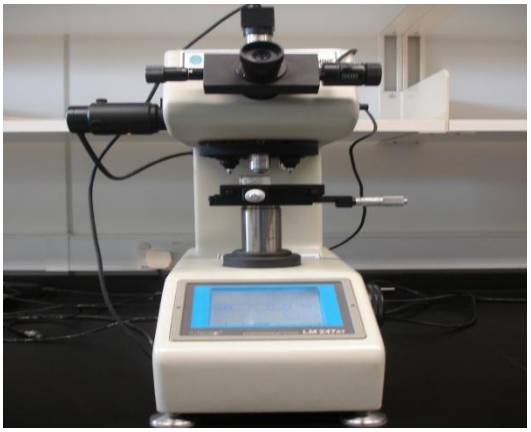

Figure 3.19: Micro Hardness Testing Machine

The usual method to achieve a hardness value is to measure the depth or area of an indentation left by an indenter of a specific shape, with a specific force applied for a specific time. The Vickers hardness test method consists of indenting the test material with a diamond indenter. The full load is normally applied for 10 to 15 seconds. The hardness measurements of the samples are taken for three times for each sample. The averages of these readings are then recorded and tabulated in Chapter 4.5.

3.6 Gantt Chart and Key Milestones

Table 3.1: Project Activities and Key Milestones for FYP 1

NO	DETAIL	WEEK														
		1	2	3	4	5	6		7	8	9	10	11	12	13	14
1	Primary Research Work		■	■	■			MID-SEMESTER BREAK								
2	Submission of Preliminary Report				●											
3	Data collection					■	■									
4	Gather research on creep mechanism in relation to current inspection methods								■	■	■	■				
5	Planning sample preparation methods and study material specifications								■	■	■	■	■	■	■	■
6	Submission of Progress Report									●						
7	Proposal Defend session											●				
8	Submission of Interim Report															●

Legends

■ ● Key Milestones

■ Process Activities

Table 3.2: Project Activities and Key Milestones for FYP 2

NO	DETAIL	WEEK														
		1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
1	Sample preparation															
2	Ultrasonic testing on the tube samples															
3	Microstructure study on the tube samples															
4	Study the relation of ultrasonic testing and microstructure study															
5	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)															

Legends

Key Milestones

Process Activities

4.0 RESULTS AND DISCUSSIONS

4.1 Data Collection of Catalyst Tube

Manufacturer's data records for the catalyst tube are shown in Table 4.1 and Table 4.2, indicating the mechanical and chemical properties of the tube.

Table 4.1: General data of catalyst tube [18]

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

Table 4.2: Chemical composition of catalyst tube [19]

Chemical Composition, %	Carbon, C	Manganese, Mn	Silicon, Si	Phosphorus, P	Sulphur, S	Molybdenum, Mo	Chromium, Cr	Ferum, Fe
Minimum	-	0.35	-	-	-	0.90	8	-
Maximum	0.20	0.65	1.00	0.04	0.045	1.20	12	balance

4.2 Design Analysis of Creep Sample

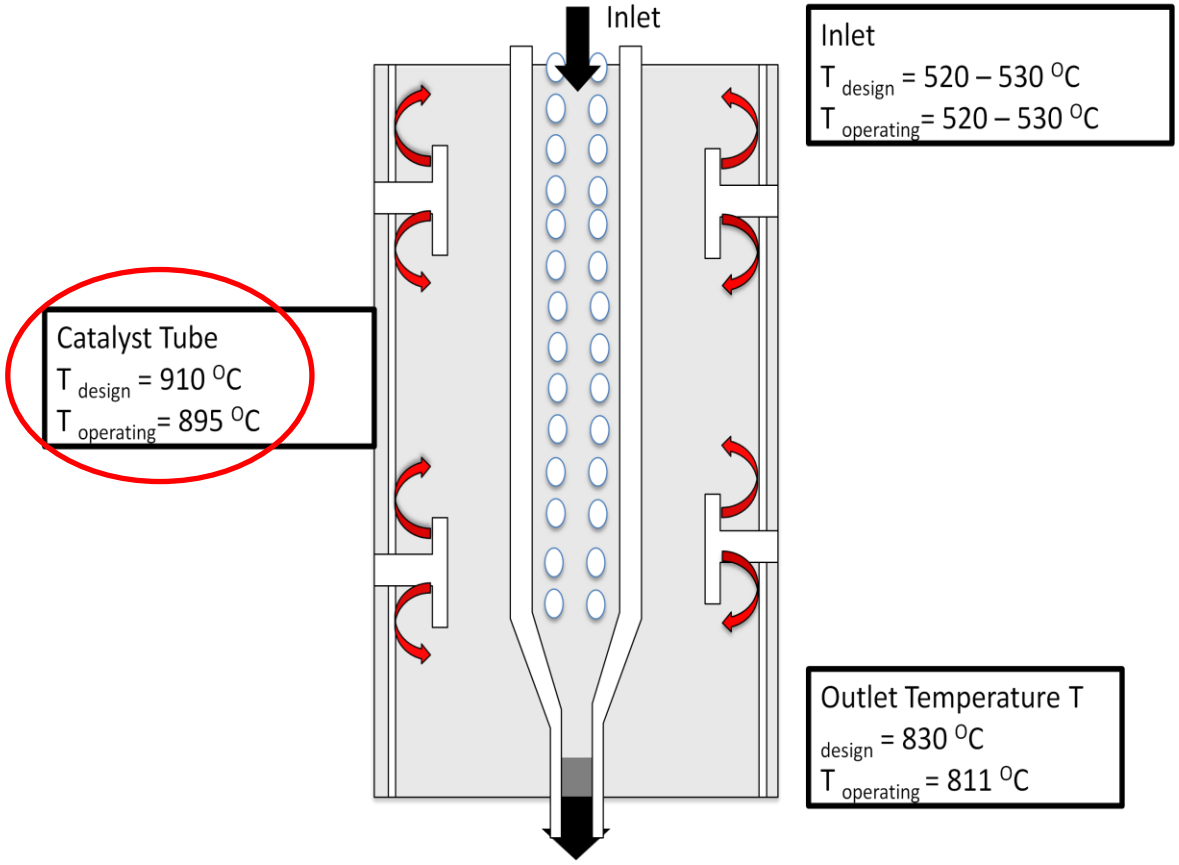

Figure 4.1: Operating conditions of PFK catalyst tube [18]

Melting point of catalyst tube, $T_m = 1510\ ^{O}C$

Creep starts when creep temperature, $T_{creep} > 0.3$ to $0.4\ T_m$

In this case, $T_{creep} = 453$ to $604\ ^{O}C$

From this, it can be shown that the operating temperature of catalyst tube had reached a maximum temperature of 910 ^{O}C, which is above the temperature where the tube is expected to creep ($T_{creep} = 453$ to $604\ ^{O}C$)

4.3 Analysis of Ultrasonic Wave Attenuation

UT test on the internal subsurface of the tube with 6.5mm thickness yielded ultrasonic wave velocity of 2009 meter per second (m/s) as shown in Figure 4.2 below.

Figure 4.2: UT velocity reading on 6.5mm thickness

UT test on the external surface of the tube with 13.0mm thickness yielded ultrasonic wave velocity of 1894 meter per second (m/s) as shown in Figure 4.3 below.

Figure 4.3: UT velocity reading on 13.0mm thickness

The values of ultrasonic velocities were found to be higher on the internal subsurface (6.5mm) of the tube, than that on the external tube surface (13.0mm). It is well known that the operating temperature environments for both these surfaces are different, where the external were exposed to much higher temperature than the other.

Preliminary analysis suggested that the severity of creep damage is higher in the external surface of the tube than the subsurface resulted from having different temperature environment. Higher creep damage will cause the grain structure of the material to increase in size, and presence of voids will be more significant. The reduced in ultrasonic wave signaled that the path taken by the sound waves are more heavily obstructed. Therefore, tube layer with higher creep damage will produce ultrasonic wave with lower velocity. However, these analyses are subjected to confirmation via microstructure study which will be executed in accordance to the Gantt Chart provided in Chapter 3.3.

4.4 Analysis of Microstructural Changes

After preparing the samples as mentioned in the previous methodology, microstructure for tube at internal subsurface (6.5mm thickness) and external tube surface (13.0mm) was revealed as in Figure 4.4 and 4.5.

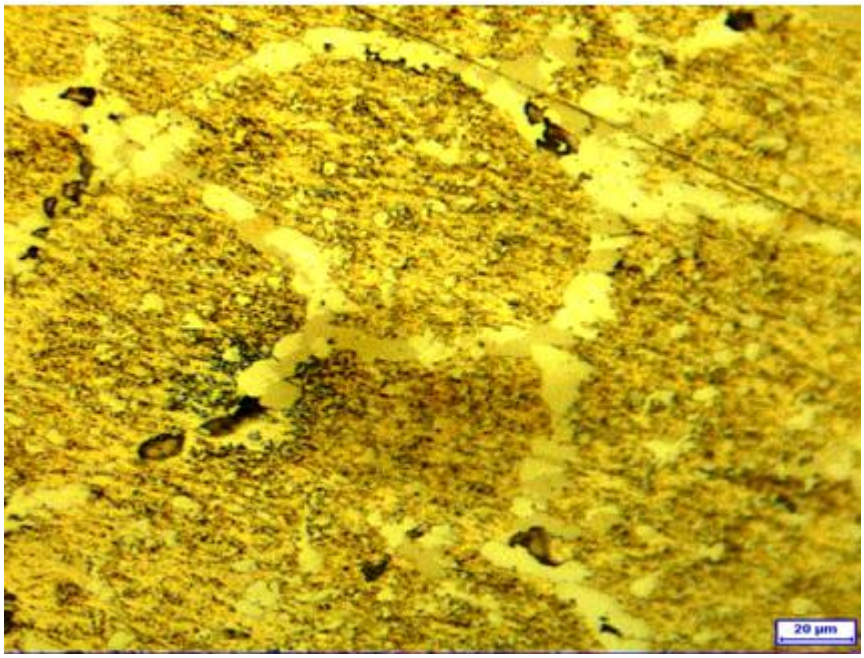

Figure 4.4: Microstructure of tube at 6.5mm thickness showing isolated voids

This part of tube section was exposed to a maximum of 773^0C, which is the temperature of the product that flows on the internal surface of the tube. The grain size on this particular layer of thickness is measured to be 7.00µm.

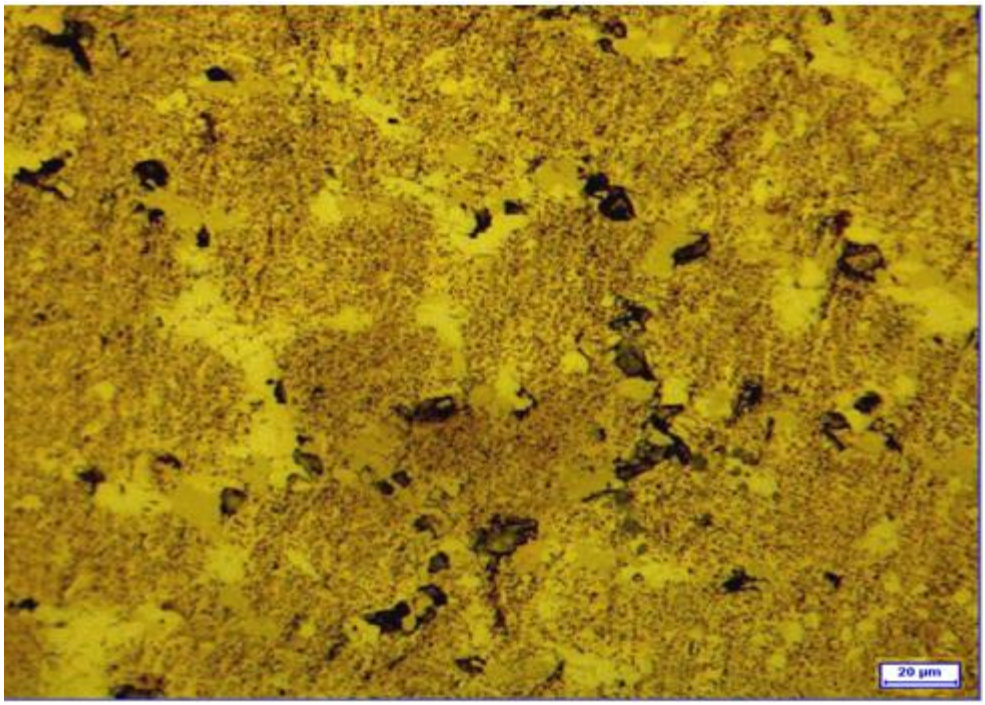

Figure 4.5: Microstructure of tube at 13.0mm thickness showing chain-like voids

This part of tube section was exposed to a maximum temperature of 910^0C, which is the temperature of the heat provided by the burner onto the external surface of the catalyst tube. The grain size on this layer of thickness was measured to be 5.50µm.

Both microstructures exhibit the structure of coagulated carbide and micro-pores at grain boundaries [20]. It is in the early stage of secondary creep, and carbides are seen to precipitate at the grain boundaries in chain-like form. With respect to time and strain, the micro-pores will further develop into micro-cavities at the grain boundaries, which will contribute to the next level of creep damage.

It can be seen that the difference in grain sizes between the samples are because of the difference in temperature acting on it. The progressive decrease in grain size with increase in temperature was due to the production of greater homogeneity and compactness of the deposited grains [15].

4.5 Analysis of Micro Hardness Test

The samples taken at 6.5mm and 13.0mm thicknesses underwent Vickers's hardness test at the load of 100gf. The average reading from three tests on each sample were recorded and tabulated as shown in Table 4.5.

Table 4.3: Readings taken from Vickers's micro hardness test

Sample taken at thickness	**6.5mm**	**13.0mm**
Reading 1	265 Hv	325 Hv
Reading 2	232 Hv	316 Hv
Reading 3	223 Hv	319 Hv
Average Reading	**240 Hv**	**320 Hv**

The hardness value of the tube material in as-cast condition is 180±6 [19]. It can be seen that after 40,000 hours of service in high pressure and high temperature environment, the hardness values of tube on both internal and external surface of the tubes had increased considerably to 240 Hv and 320 Hv, as shown if Figure 4.6. This is due to the increase of carbide precipitation along the grain boundaries which are extremely hard in nature [15]. As the temperature increases, the formation of carbides proved to be increasing and had undergone significant agglomeration as shown in the microstructure study, which explains the higher hardness value of tube sample operating at a maximum of 910^0C (external surface) than that of the sample operating at around 773^0C (internal surface).

The hardness value increased in relation to the tube condition as shown in Figure 4.6.

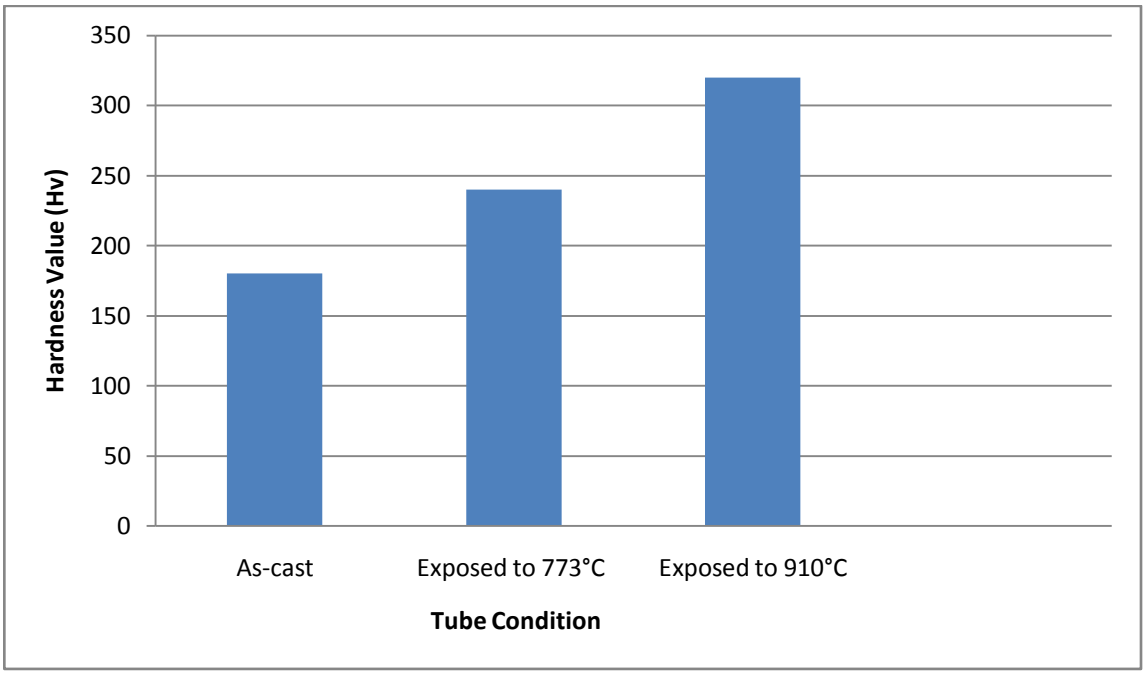

Figure 4.6: Hardness values of different tube conditions

5.0 CONCLUSION AND RECOMMENDATION

5.1 Conclusion

Evaluation of creep damage based on microstructural degradation in relation to exposed temperature can be concluded as shown below :-

1) The ultrasonic wave attenuations had been determined, and it can be seen that the outer surface of tube which was exposed to higher temperature produced lower velocity of ultrasonic wave than the internal subsurface of the tube, which was exposed to significantly lower temperature environment.

2) The study of microstructure showed that the grain size of ASTM A608 steel sample decreased with increase in temperature. Presence of coagulated carbide and micro-pores were detected. In addition, hardness value of tube sample had increased linearly with the exposed temperature. This indicates that the tube suffers from the early stage of tertiary creep damage.

5.2 Recommendation

In the future, it is very fascinating if the catalyst tube sample used from PFK which is assumed to be in secondary and early stage of tertiary creep damage could be put under accelerated creep condition to further induce it to reach later stage of tertiary creep damage until it ruptures. In doing so, extensive relation between the microstructures and wave attenuations of all three stages of creep damage can be further discussed.

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