CHAPTER 1

INTRODUCTION

1.1 Background of Study

Furnaces at Ethylene (M) Sdn Bhd is ethane cracking heater which are used to crack ethane to produce ethylene gas and other useful byproduct which is mainly fuel hydrogen gas. The cracking heater is gas fired and the fuel gas is hydrogen and methane-off gas. [1]

There are six units of furnaces at Ethylene (M) Sdn Bhd plant (ET-O-F-101, ET-O-F-102, ET-O-F-103, ET-O-F-104, ET-O-F-105 and ET-O-F-106). The tubes' material is nickel based iron (NiCr) and the manufacturing process is casting. The furnace feed composition is 93.1% mol Ethane at flow rate of 20 T/hr. [1]

The furnace tubes are scheduled to be replaced for every 6 years. However, after 3 to 4 years in operation, the failure rate of the tubes increasing due to creep and carburization. One furnace tube was removed from operation due to bulging, after 1 year of operation.

1.2 Problem Statement

Furnace tube prematurely failed after 1 years of operation. Total replacement of furnace tubes are scheduled after 6 years of operation. The last total replacement of the tubes was done in June 2008. The tube experienced bulging and cracks, suspected due to overheating. Failure analysis to identify the causes of failure is to be done.

Periodical inspection of furnace tube was done by Operation Department of Ethylene (M) Sdn Bhd for ET-O-F-105. Abnormal conditions were detected before the furnace tubes were removed for inspection. Bulging of tube was observed and the failed tube was then sent for failure analysis.

1.3 Objective and Scope of Study

The objectives of the study are as follows:

a. To conduct tests and experiments on failed furnace tube to collect data relevant to failure. The tests and experiments include background analysis, visual and preliminary examination, microscopic examination, fractography examination, chemical analysis and hardness testing.
b. To analyze data obtained from tests and experiments to identify causes of failure to the component.
c. To suggest recommendations to avoid similar incidents in the future.

CHAPTER 2

LITERATURE REVIEW

Service failures of components and structures have been increasingly experienced in several industries. While some failure may be trivial, some may have serious consequences such as fatalities, injuries, damage to property, shutdowns, loss of production and ecological problems. [(2)] In this case study, furnace failure can cause unplanned shutdown to Ethylene (M) Sdn Bhd plant which will cause loss of production. Besides, furnace failure can also cause explosion which can cause injury and fatality. [(1)]

According to Asset Life Study Report by Group Technology Solution, function of cracking heater of Ethylene (M) Sdn Bhd is to crack ethane to produce ethylene and by-products (mainly H2 fuel gas). Feed Composition is 93.1% mol Ethane at flow rate of 20 T/hr. Ethane feed enters convection section (60 ºC) at Fd Prht zone. Preheated feed is diluted with steam (220 ºC & 7 bar) and subsequent convection heat at the Max Preheat and L Max Preheat zone. Then, the gas enters Radiant coil (675-700 ºC, 2.5 barg) where the ethane gas is cracked. [(1)]

$$C_2H_6 \longrightarrow C_2H_4 + H_2$$

It is well known that producing ethylene will generates free carbon according to the reaction [(4)] :

$$C_2H_4 \longleftrightarrow CH_4 + C$$

This production of carbon can cause carburization. Carburization occurs when Carbon from the environment combines at elevated temperature primary with Carbide and other carbide formers (Nb, W, Mo, Ti, etc) present in the alloy. The carbides may be quite complex and from within the grains and along the grain boundaries. They are hard and brittle. The overall effect is a drastic reduction of ductility at elevated temperatures, a reduction of ductility at elevated temperatures, a reduction of oxidation resistance, and an adverse affect on creep strength. Carburization is common in ethylene cracking furnaces due to the presence of high tube metal temperature and high Carbon potential associated with hydrocarbon feedstock. The process is unpredictable and non-uniform in nature. [(4)]

From the literature, ethylene cracking tubes which are made from austenitic steel HP grade are usually designed for a normal life of 100,000 hours, (11.4 years). [(3)] Their actual service life, however, varies from 30,000 to 180,000 hours, depending on service conditions and quality of the material. For Ethylene (M) Sdn Bhd, the scheduled total replacement of furnace tube is 6 years. [(1)] Though this replacement may result in coils being removed with significant remnant life still available, it would eliminate unplanned failures and allow re-tubing to be scheduled, budgeted and managed as a routine maintenance activity. The furnace radiant tubes subjected to creep and carburization.

The nominal chemical composition [(5)] as given by the manufacturer is as below:

Table 2.1 Chemical Composition for Furnace Tube

Element	Percentage wt%
C	0.40 – 0.45
Si	1.5-2.0
Mn	1.50 Max
P	0.030 Max
S	0.030 Max
Ni	34.0 – 37.0
Cr	24.0 – 27.0
Nb	0.80 – 1.20
Ti	0.30 Max
Zr	0.30 Max

Mo	0.50 Max
N	0.10 Max
Al	0.0 Max
W	0.30 Max
Zn	0.005 Max
Pb	0.005 Max
As	0.005 Max
Sn	0.005 Max
Te	0.005 Max
Sb	0.005 Max
V	0.10 Max
Cu	0.25 Max

The furnace is regularly inspected to detect abnormalities in its operation and avoid production lost of the plant. The regular furnace inspection practiced for ET-O-F-105: (1)

i. Tube Metal Temperature survey (once a month)
ii. Furnace Wall Temperature survey (once a month)
iii. Tube outside diameter Measurement (Magnetic permeability)
iv. Regression (measuring tube elongation)
v. Visual inspection of the refractory conditions (Visual inspection of the external areas)

The failed furnace tube will undergo Failure Analysis process. Failure Analysis is a combination of various disciplines. According to Ramachandran, Raghuram, Krishnan, Bhaumik,(2005) and Davidson(2008), several steps and analysis are required to be completed for a Failure Analysis. (2) (6)

1. Collecting Background Information (2) (6)

   Before conducting Failure Analysis, relevant background information is collected. This facilitates the developing of a complete case history about the failure. [2] [4]

The information to be collected is:

i. Information about the failed components
ii. Information about the failure itself.

2. Specimen collection[2]

   Specimen is collected from site for further laboratory examination. Specimen is handled very carefully as they can provide useful information about the mode and mechanism of the failure.

3. Preliminary Examination[2] [6]

   Visual examination of the component and the fracture surface will give information on the presence of debris and corrosion products; change in surface colour, abrasion, and rub marks. This preliminary examination is best carried out by naked eye or with low-power microscope. Findings should by documented by using sketches and photographs.

4. Nondestructive Examination[2] [6]

   Nondestructive Examination is conducted to detect at an early stage subsurface flaws and internal flaws in the component, their type, size, orientation and location. In this project, Dye Penetrant Testing is used. Dye Penetrant Testing can detect discontinuities open to the surface or in the subsurface.

5. Microscopic Examination[2] [6]

   Detailed examination of the fracture surface is to be carried out at different levels of magnification and resolution using optical and electron microscope.

6. Chemical Analysis[2] [6]

   Chemical Analysis of samples from the component provides information regarding any deviation from standard specifications, composition inhomogeneities, impurities, inclusions, segregations, and so forth. It can also helps in identifying the nature of corrosion product, coatings, external debris, and so on. Analysis on microscopic levels provides information about the nature of inclusions, phases and surface layers.

7. Mechanical Properties Testing[(2) (6)]

   Evaluation of the mechanical properties of component is an important step in Failure Analysis. This process enables the investigator to judge whether the material with which the component is made meets the strength specifications and whether the component was capable of withstanding the service stresses. If the size of failed component permits, sample can be taken from the component, and conventional mechanical testing can be done by standard test procedure

8. Analysis of Data[(2) (6)]

   The most crucial part in Failure Analysis is the consolidation and systematic connection of all data obtained in the tests. The result of tests and examination must be compared against the specifications and deviations, if any, and should be carefully considered as possible contributing factors. Information such as initiation site or sites, length of crack, its propagation path and speed, and the nature and direction of load acting on the component can be obtained through proper analysis. Role of other factors such as temperature, corrosion, wear, component history, etc will be clearer with analysis. In due course, the sequence of failure would get established, differentiating between primary cause of failure event and the consequential secondary failure and damages. In complex system, a number of causes may be seemed relevant. Detailed investigation into each of these causes may be time consuming and expensive. Systematic logical deduction technique called the fault tree technique is very helpful in identifying the basic events whose combinations could lead to the failure.

9. Preparation of report[2]

Documentation is extremely important for effective communication of results of failure analysis. The report should be clear and contain the logic behind the conclusions.

The following components form the essential sections of the report:

i. Description of the failed component
ii. Circumstances leading to the failure
iii. Operational parameters and conditions at the time of failure
iv. Background history
v. Visual examination of general physics features
vi. Laboratory investigations: Metallurgical, mechanical, chemical and other tests and their results.
vii. Discussion
viii. Conclusions and recommendations
ix. References
x. Summary

CHAPTER 3

METHODOLOGY

3.1 Project Flow Chart

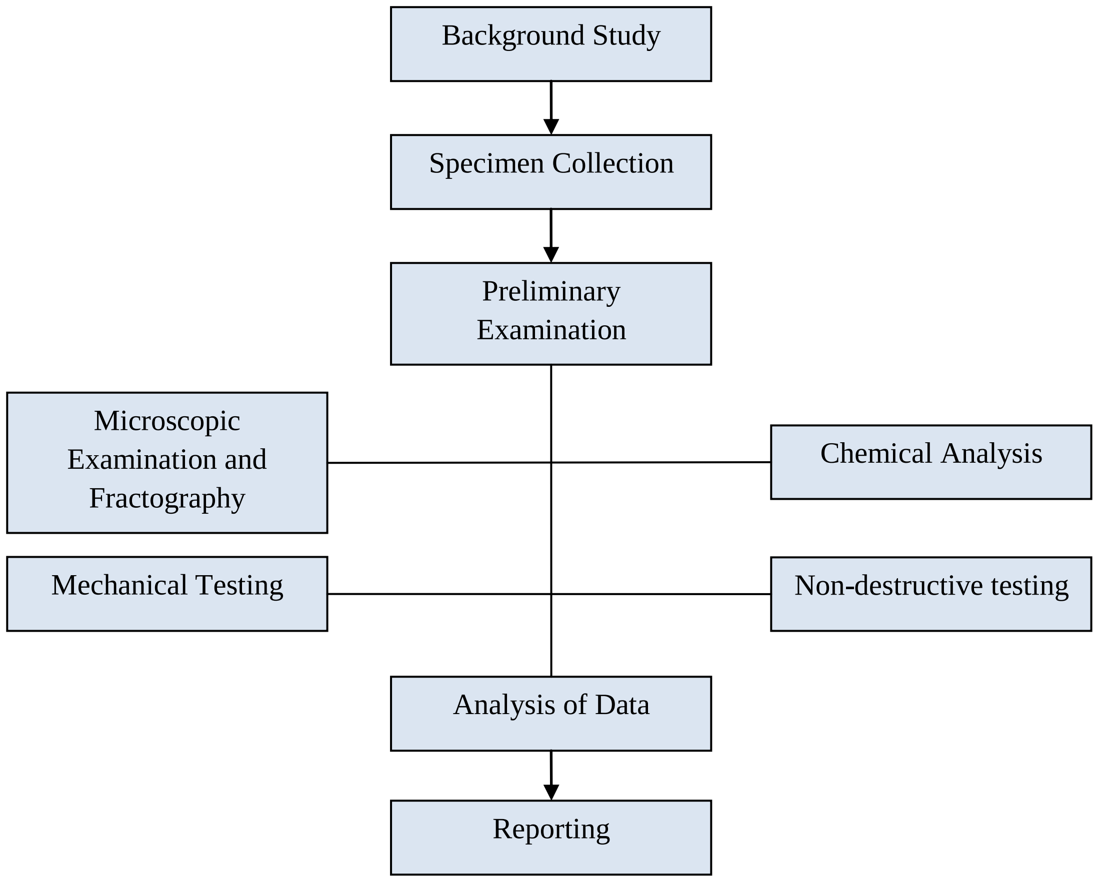


Figure 3.1: Project Flow Chart

3. 2 Research Activities

Non-Destructive Testing using Dye Penentrant Testing was done to detect surface and sub-surface crack of the component. The Dye Penetrant Testing was done at the outer surface of the tube. The samples from as received failed component were prepared and were used for microscopic examination using optical microscope. The microscopic examinations analyzed the material and detected any defect at microscopic level.

Fractography was conducted on the fracture surface in an SEM. Chemical analysis by Energy-dispersive X-ray spectroscopy (EDX) in a Scanning Electron Microscope (SEM) was used to verify the tube material.

Rockwell hardness tests were used to evaluate the mechanical hardness. The difference in mechanical hardness between internal surface and middle section were compared and analyzed.

3.3 Tool and Equipment

Dye Penetrant Testing

i. Dye Penetrant
ii. Cleaner
iii. Developer

Microscopic Examination and Fractography

i. Scanning Electron Microscope
ii. Optical Microscope

Mechanical Properties Testing

i. Rockwell Hardness Testing Machine

Chemical Analysis

i. Energy-dispersive X ray spectroscopy (EDX) in Scanning Electron Microscope (SEM)

3.4 Gantt Chart

Table 3.1: Gantt chart

Research Activity/ Week	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**	**9**	**10**	**11**	**12**	**13**	**14**
Initial Study	■	■												
Sample Collection		■												
Preliminary Examination		■	■											
NDT				■	■									
Hardness Testing						■	■							
Micrographic Testing							■	■						
Fractography								■	■					
Chemical Analysis								■	■					
Analysis of Data										■	■			
Reporting												■	■	■

CHAPTER 4

RESULT AND DISCUSSION

4.1 Background Analysis

The collected information about the furnace tube is as follows:

Table 4.1: Background study of ET-O-F-105's tube failure

General Information	
Plant	Ethylene (M) Sdn Bhd
Tag Number	ET-O-F-105 (Coil 6)
Design Code	API 530/ ASME VIII Div 1
Design Parameters	
Material	Nickel based alloy (25-35 CrNi)
Design Life	100,000 hours
Design Operating Temperature	1100ºC
Operating Temperature	692 ºC (Outlet), 841ºC (Inlet)
Tube thickness	6.55 mm
Manufacturing Information	
Manufacturer	Schmidt-Clemens
Manufacturing process	Casting
Failure Information	
Component major maintenance history	Total radiant tubes replacement conducted in 2003 and 2008. Replacement of furnace linings carried out in 2006.
Date of failure	30th May 2009
Detail of incident	Periodical inspection was done. Abnormal temperature detected. Tube removed and replaced.

4.2 Preliminary Examination/Visual Inspection

The received impeller was analyzed to detect any abnormalities at visible defects at the sample.

Figure 4.1: Tube bulge as received sample (1)

Figure 4.2: Furnace tube from upper view

Figure 4.3: Internal cracks

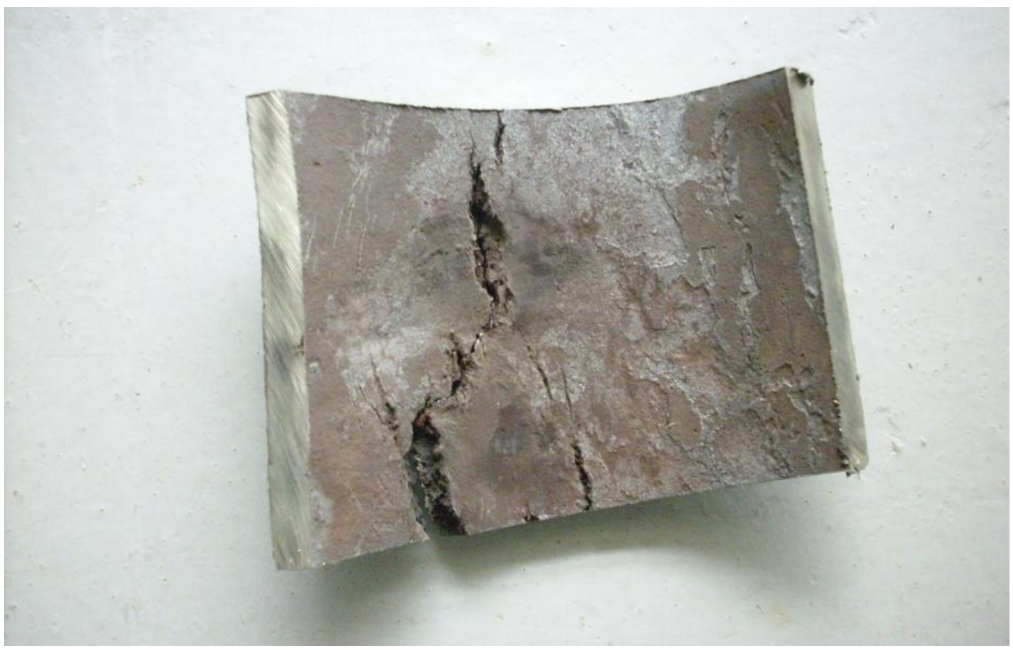


Figure 4.4: Internal cracks

Figure 4.5: Internal cracks

Figure 4.6: Penetrated internal crack

Figure 4.1 shows the actual bulge and rupture of as received sample. Figure 4.2 shows tube diameter is now increased and elliptical. This is a big amount of deformation. It is consistent, however, with a tube that has operated at higher than normal temperatures for short periods.

Figure 4.3, 4.4 and 4.5 shows the crack viewed on the tube inner surface. The crack is discontinuous in inner surface.

Figure 4.6 shows penetrated internal crack. The penetrated crack is longer and bigger in inner surface than that in outer surface which indicates the crack originated from inner surface. There is no indication of any localized damage in the form of pits. No wall thinning and plastic deformation is observed near the crack which reveals that the crack is brittle in nature.

The stresses resulting from gas pressure in the ethylene production processes are relatively low and it is well known that the most common failure modes of furnace components are longitudinal creep-rupture and carburization. [4]

4.3 Non-Destructive Testing

Dye Penetration Test was done to detect surface and sub-surface defects of the sample. Dye Penetration Testing's result on external surface of the tube is shown below.

Table 4.2: Non-Destructive Testing Result

Result	**Discussion**
	No crack detected at one side of the tube even though minor bulging is detected at this side of tube.
	Three cracks detected at the other side of the furnace tube where major bulging is detected. The cracks' lengths are about 3.0-3.5 cm. No minor cracks detected at other part of the furnace tube.

The test only covers the outer surface of the tube. However, from the visual examination, internal cracks can be detected. There are no minor cracks at the outer surface of the tube.

The result of this test concludes that the cracks originated from the inner surface and penetrated to the outer surface at some point where the failure is most severe.

4.4 Hardness Testing

Hardness test was done to examine the hardness of the sample. Hardness test was done at the inner surface of the sample and the middle section of the sample. The results for hardness test for inner surface are as below:

Table 4.3: Hardness Testing For Inner Surface

Reading	Sample 1 (HRC)	Sample 2 (HRC)	Sample 3 (HRC)
1	31.0	31.6	30.8
2	30.9	32.0	31.7
3	30.8	32.1	32.0
4	32.0	32.7	30.5
5	30.9	31.6	31.0
Average	**31.12**	**32.0**	**31.2**

Average reading for all samples is 31.44 HRC which corresponding to HV 310.88.

The results for hardness test for middle section are as below:

Table 4.4: Hardness Testing For Middle Section

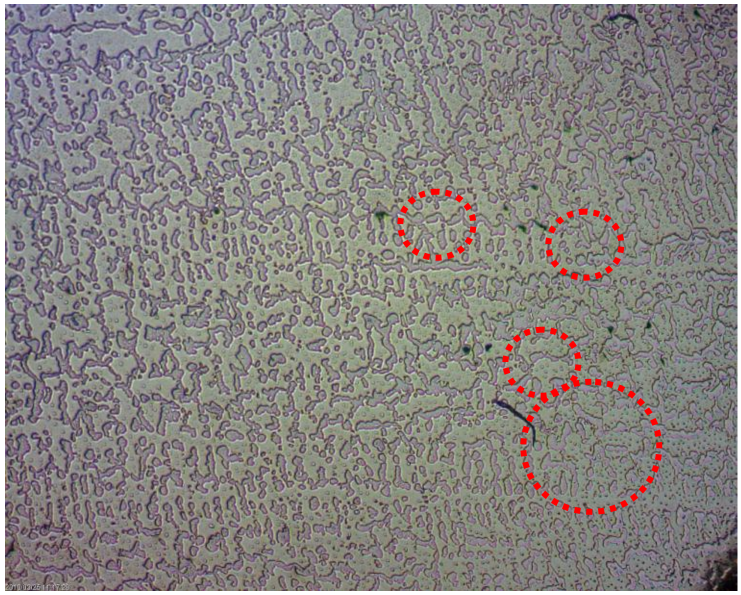
Reading	Sample 1 (HRC)	Sample 2 (HRC)	Sample 3 (HRC)
1	24.3	28.3	26.1
2	22.3	23.5	20.9
3	23.8	21.8	22.2
4	28.6	22.0	21.7
5	22.7	22.7	22.4
Average	**24.34**	**22.76**	**22.66**

Average reading for all sample is 23.54 HRC which corresponding to HV 242.75.

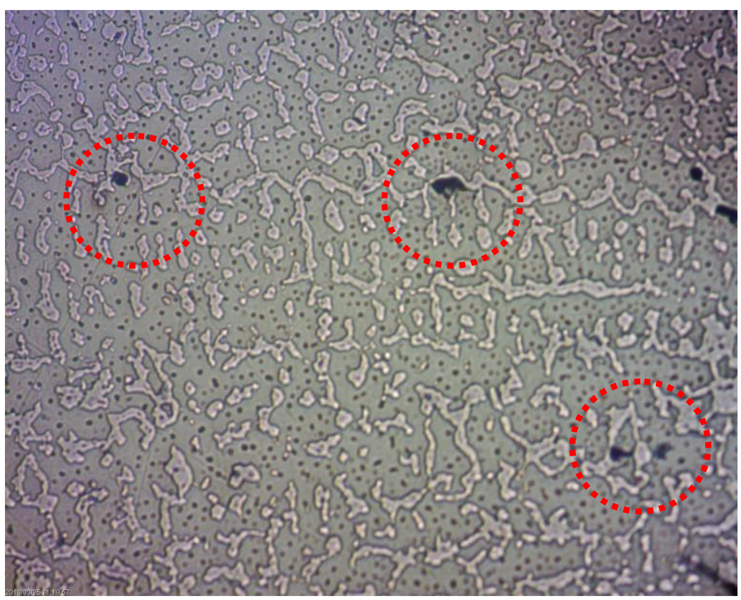
At inner surface of the tube, where carburization was most severe, the average hardness was found to be HV 310.88. Towards the outer surface however, the average hardness was reduced to HV 242.75.

Carburization is expected to the factor of this hardening. Hardening due to carburization is expected to predominate near the inner surface of the tube. According to literature, typical hardness for this type of alloy is 180-185 HV. [4]

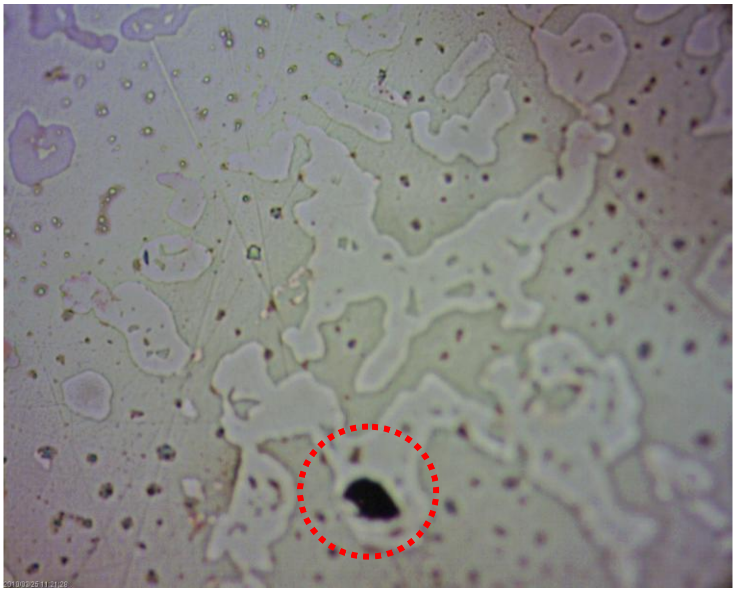
4.5 Micrographic Testing



(a) 5X Magnification



(b) 10X Magnification



(c) 50X magnification

Figure 4.7: Microstructure of failed tube

Figure 4.7 (a), (b) and (c) shows austenite matrix and a network of primary carbide of the tube, which is typical microstructure of as-cast materials of alloys with high chromium and nickel contents. However, precipitates of small blocky type are found in the austenitic grains as shown in the circles. Carbides are obviously coalesced and blocky which indicates overheating. This is in line with initial tests which lead to overheating caused by carburization.

SEM was used to further test the micrographic structure of the sample. Higher magnifications were used to analyze the grain and grain boundaries of the sample.

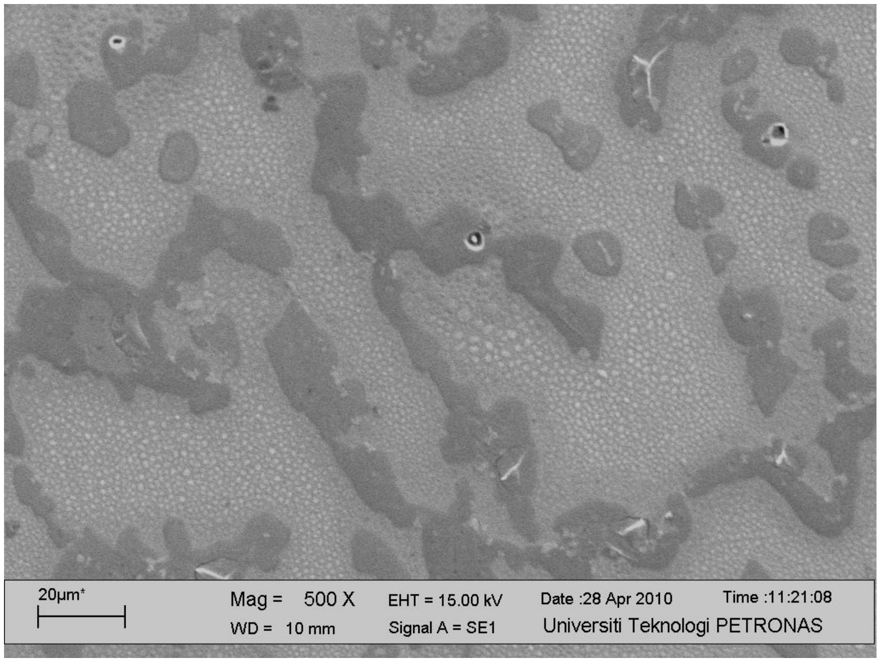


Figure 4.8: SEM image (500X magnification)

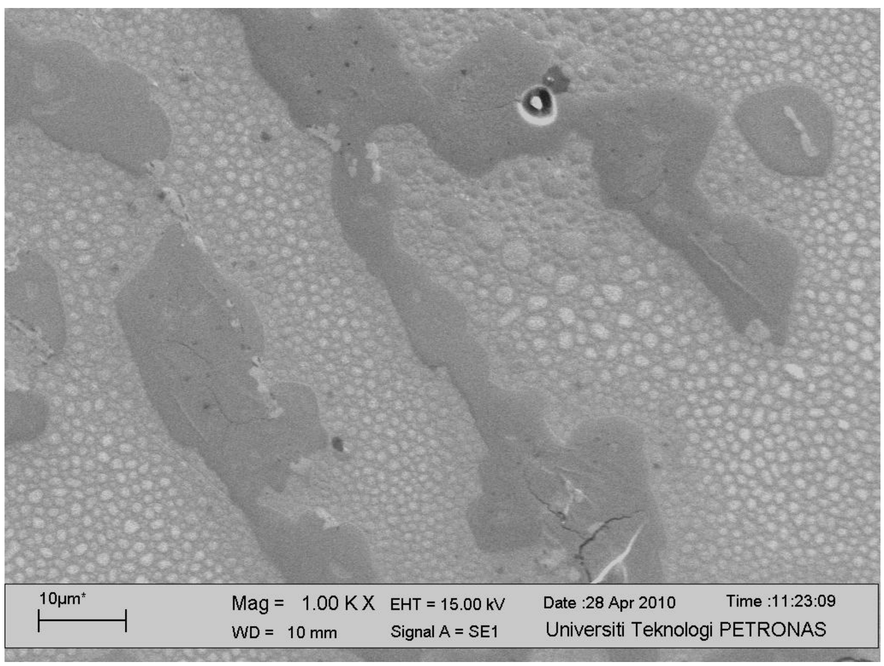


Figure 4.9: SEM image (1000X magnification)

Figure 4.8 and Figure 4.9 show SEM image from the failed tube sample. The carbide precipitation appears relatively coarse both at the austenite grain boundaries and within the matrix itself. The dark grey area is confirmed to be chromium-rich carbides from EDX analysis.

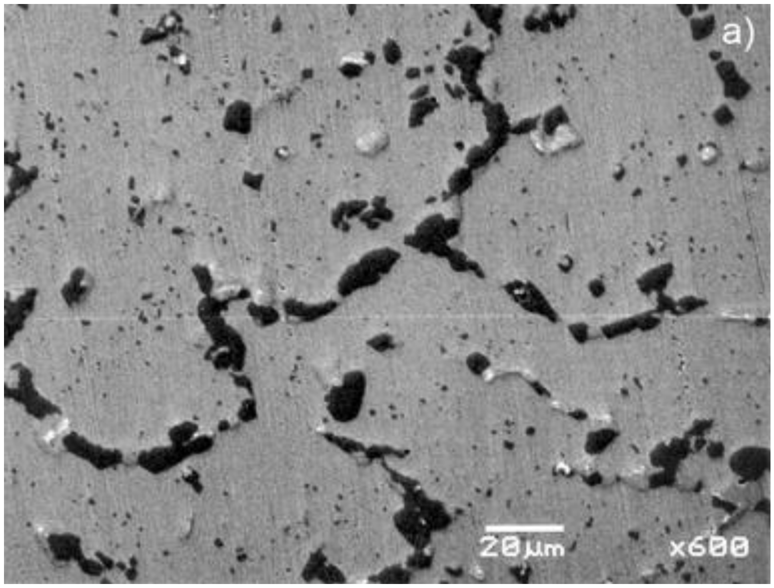


Figure 4.10: SEM image for uncarburized Nickel-Chromium alloy

From literature [4], SEM image for uncarburized Nickel-Chromium alloy is shown in Figure 4.10. Compared to the result obtained, it can be observed that chromium precipitates less coarse in uncarburized Nickel-Chromium alloy compared to the image obtained from the sample.

The sample shows a high degree of carbide precipitation at the grain boundaries and blocky carbide particles in the matrix compared to the uncarburized Nickel-Chromium alloy. Massive carbide precipitation at the austenite grain boundaries (forming a continuous lattice) and the presence of carbides as coarse blocky particles within the matrix indicate the exposure of the furnace tubes to an excessively high temperature that leads to heavy carburization.

4.6 Chemical Analysis

Chemical analysis was done using Energy Dispersive X-Ray (EDX) to verify the material chemical composition.

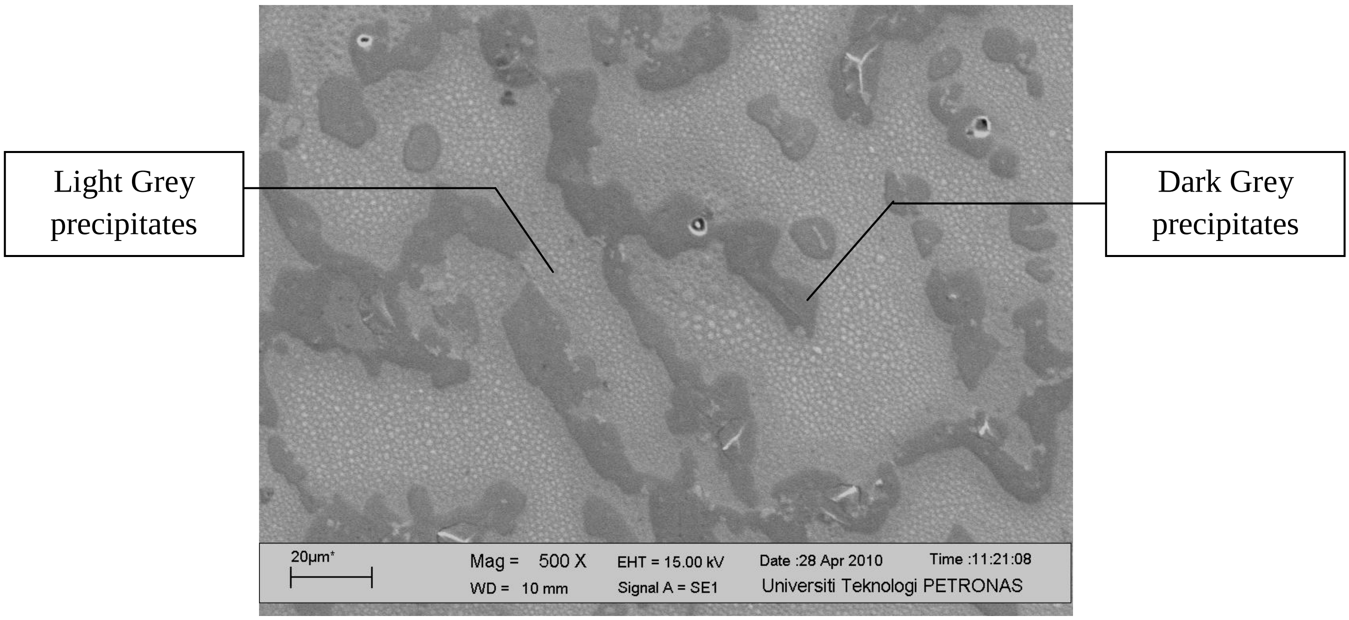


Figure 4.11: SEM image for EDX test

Energy dispersive X-Ray (EDX) spectra obtained from the dark grey region of the microscopy is as follows:

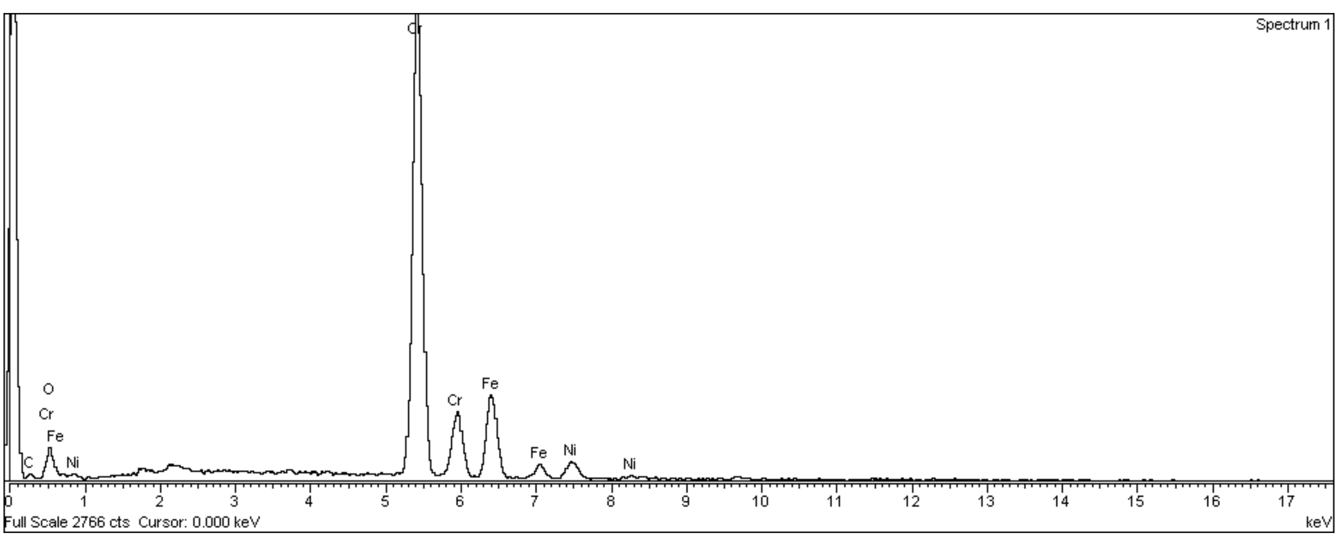


Figure 4.12: Energy dispersive X-ray spectra obtained from dark grey region

Table 4.5: Chemical composition of dark grey precipitates

Element	Weight %	Atomic %
C K	3.73	13.54
O K	3.87	10.55
Cr K	67.76	56.85
Fe K	19.57	15.29
Ni K	5.08	3.77

The Energy dispersive X-ray result shows large peak for Chromium. Chemical composition shown in Table 4.5 shows that the dark grey precipitates are chromium-rich precipitates.

Energy dispersive X-Ray (EDX) spectra obtained from the light grey region of the microscopy is as below:

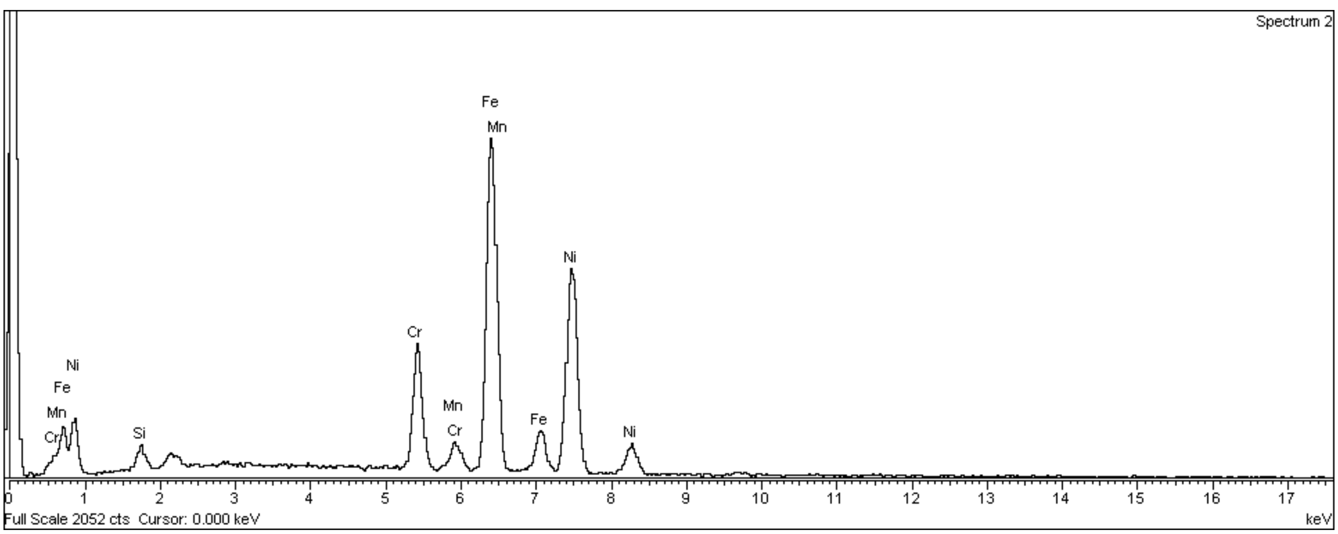


Figure 4.13: Energy dispersive X-ray spectra obtained from light grey region

Table 4.5: Chemical composition of light grey precipitates

Element	Weight%	Atomic%
Si K	2.38	4.68
Cr K	11.20	11.89
Mn K	1.21	1.22
Fe K	42.82	42.34
Fe K	35.05	18.40
Ni K	9.07	4.53

The Energy dispersive X-ray result shows large peak for Ferum. Chemical composition shown in Table 4.5 shows that the dark grey precipitates are Ferum-rich precipitates.

This result confirms the analysis of micrographic testing in the previous section, as shown in Figure 4.8 and 4.9.

Energy Dispersive X-ray (EDX) spectra obtained from the surface of the failed sample is as follows:

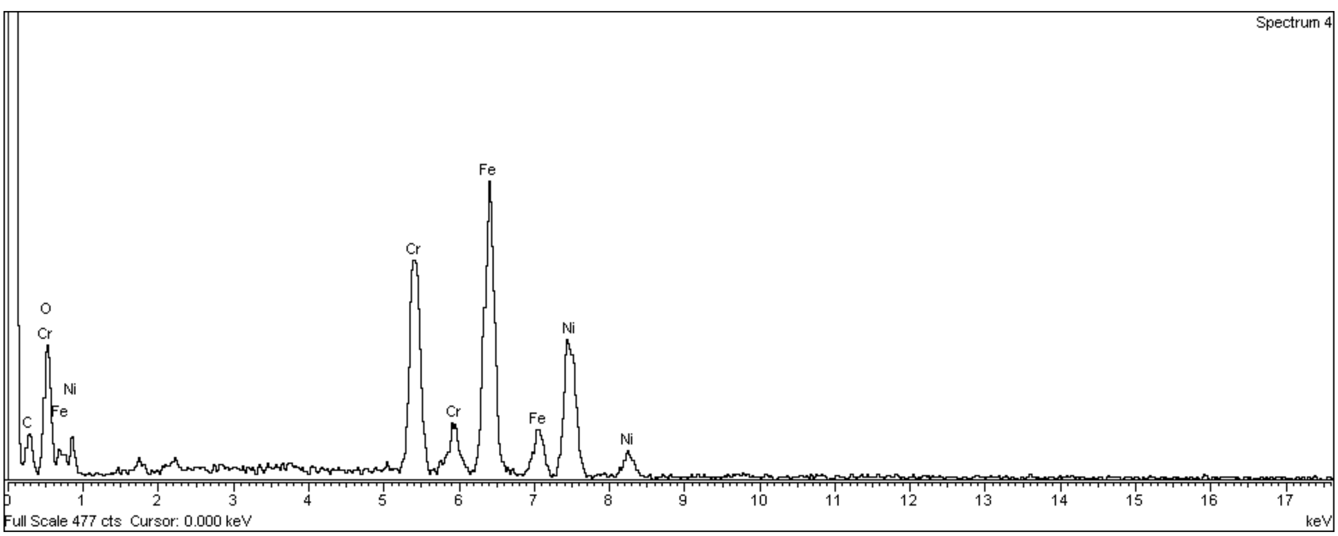


Figure 4.14: Energy dispersive X-ray spectra obtained from surface of the sample

Table 4.6: Chemical composition of failed tube sample's surface

Element	Weight%	Atomic%
C K	15.03	33.18
O K	22.32	37.00
Cr K	15.22	7.76
Fe K	27.07	12.86
Ni K	20.36	9.20

The Energy dispersive X-ray result shows the presence of Carbon. Comparing to chemical composition of the tube from the manufacturer, Carbon's percentage is much higher for the sample which is 15.03% compared to 0.40-0.45% as informed by the manufacturer.

This proved carbon's involvement in the reaction at the surface of the tube.

4.7 Fractography

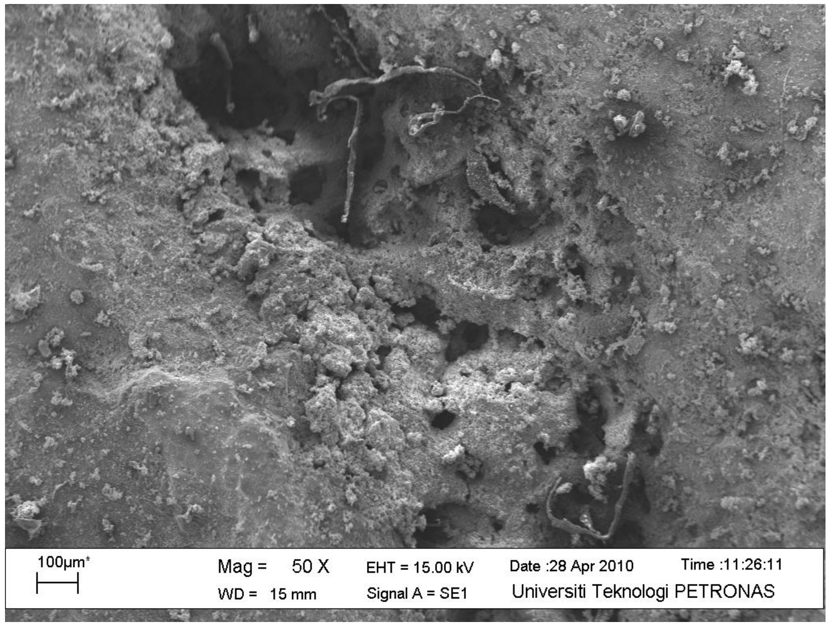


Figure 4.15: SEM image of fracture (50X magnification)

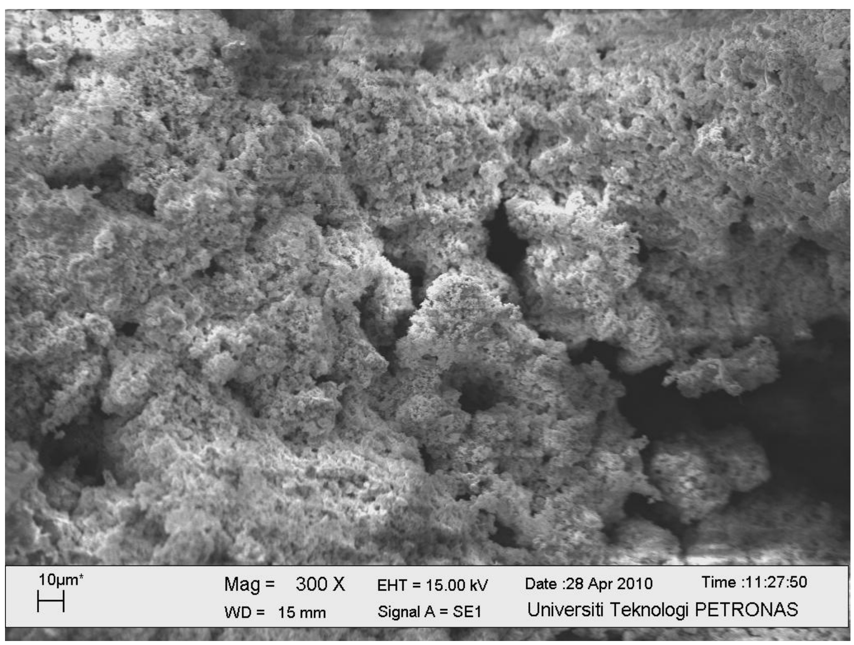


Figure 4.16: SEM image of fracture (300X magnification)

Figure 4.11 and Figure 4.12 present the fractography of the crack. The fracture mode was found to be intergranular crack.

Intergranular fracture can exclusively occur by creep deformation. However, it is accelerated by carburization attack which reduces the ductility of the material. If the material becomes unable to strain harden during the initial creep stage (primary creep), e.g., by intergranular cracking due to carburization, the creep rate is accelerated. [4] [7]

It can be concluded that the mode of failure of the radiant furnace tube was a combination of creep damage and carburization attack.

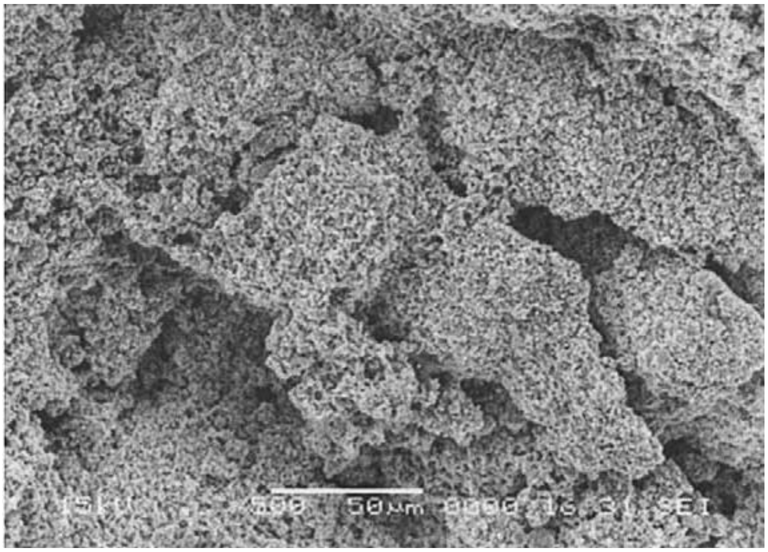


Figure 4.17: SEM image of intergranular crack

From literature [3], the intergranular crack is shown in Figure 4.17. Comparing with SEM image of the crack, the result is confirmed to be intergranular crack.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

The furnace tube examined in this study exhibited carburization.

From initial tests which are Non-Destructive Testing and Visual Inspection, it can be initially concluded that the failure is due to overheating at the bulging area. From the results presented in the earlier section it appears that tube failed due to long and high temperature exposure. However, the other sections of the same tube did not fail although they had undergone the same period of service life. Therefore, it is understood that there was some uniqueness in the failure of tube in this section. The rise in metal temperature leads to ruptures, which can be seen from the formation of bulges.

The hardness testing reveals that the internal surface of the tube has higher hardness compared to the middle section of the tube. This could be due to carburization which occurs at the internal surface of the tube. This indicates a higher a precipitation and Carbon pickup by the alloy. The decomposition of coke on the inner tube will also promote Carbon diffusion and the precipitation of secondary carbides within the alloy.

Micrographic test revealed precipitates of small blocky type are found in the austenitic grains. Carbides are obviously coalesced and blocky which indicates overheating. This is in line with initial tests which lead to overheating caused by carburization.

From the comparison between uncarburized Nickel-Chromium alloy SEM image and the sample SEM image, the sample shows a high degree of carbide precipitation at the grain boundaries and blocky carbide particles in the matrix. Massive carbide precipitation at the austenite grain boundaries (forming a continuous lattice) and the presence of carbides as coarse blocky particles within the matrix indicate the exposure of the furnace tubes to an excessively high temperature that leads to heavy carburization.

Chemical Analysis of the sample confirmed the Chromium-rich precipitates are coarser compared to Nickel-rich precipitates and Nickel-rich precipitates as shown in the micrographic testing.

Carbon is also detected at the inner surface of the sample. According to the chemical composition provided by the manufacturer, Carbon percentage is much smaller compared to the result. From the composition provided by the manufacturer, Carbon percentage is only 0.40-0.45%, while the chemical analysis shows a higher percentage which is 15.03%. This confirms Carbon's involvement in the reaction at the surface of the failed tube.

Fractography reveals intergranular crack. Intergranular attack can be caused by creep deformation and was accelerated by the attack of carburization.

Experimental data shows that the furnace tube had undergone carburization due to exposure to excessively high temperature and carbide formation from the reaction of carbon during service. Cracking can be caused by the reduction of ductility of the tube because of overheating. The overheating could lead to significant degradation in high temperature strength and ductility during service. (3)

On the other hand, with high temperature exposure, secondary carbides precipitate in the austenite matrix of heat resistant steels, reducing the elongation and ductility. Ductility is a very important property as metal should be able to deform plastically during the tube expanding. Some steel can lose more than 80% of their original ductility when they operate at high temperature. [3]

The temperature of the furnace should be monitored and controlled closely, in order to avoid over-heating during ethylene production and decoking.

5.2 Recommendation

It is recommended for Ethylene (M) Sdn Bhd to review all the parameters involved to make sure no overheating occur during process. Decoking process should also be reviewed in term of its frequency to control the deposition of coke. Decoking process which is done too frequent will could accelerate creep deformation, while in the other hand, without decoking process, coke formation will occur and disturb the process parameters.

As a long term solution, it is also recommended for Ethylene (M) Sdn Bhd to replace Nickel-Chromium alloy with an Al_2O_3-forming alloy. Most evidence points out that there is a correlation between the protective nature of the surface oxide scale developed by the alloy and its resistance to carburization. (7) Alloys that develop Al_2O_3-base scale are found to be considerably more resistant to carburization in comparison with CrO_3-forming alloys. It is believed that an Al_3O_2-base scale impedes carbon diffusion into the alloy, which considerably reduces the extent of carburization. (7)

For future work, it is recommended to review the furnace’s process parameters, the furnace structure and burning system as these aspects could also contribute to the failure. Besides, it is also recommended to study the previous failure records, frequency of failure for each furnace tubes and operating records of the furnace. These records could help the researcher to analyze other factors which could contribute to the failure.

It is also recommended for future researcher to study on other sample from other furnace unit, to compare with the furnace tube in this study. There are six units of furnace in Ethylene (M) Sdn Bhd, which could have different failure rate and system.

CHAPTER 6

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