ENDURANCE INVESTIGATION OF NITRIDE FERRITIC STAINLESS STEEL SS439 UNDER TORSIONAL LOADING

By Aliff Idham Bin Mohammad Latpi

Supervised by Assoc. Prof. Dr Patthi Bin Hussain

A dissertation report submitted in partial fulfillment of the requirements for the Bachelor of Engineering (Hons) (Mechanical Engineering)

SEPTEMBER 2013

Universiti Teknologi PETRONAS Bandar Seri Iskandar 31750 Tronoh Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

ENDURANCE INVESTIGATION OF NITRIDE FERRITIC STAINLESS STEEL SS439 UNDER TORSIONAL LOADING

By Aliff Idham Bin Mohammad Latpi

A project dissertation submitted to the Mechanical Engineering Programme Universiti Teknologi PETRONAS in partial fulfilment of the requirement for the BACHELOR OF ENGINEERING (Hons) (MECHANICAL ENGINEERING)

Approved by,

(Assoc. Prof. Dr Patthi Bin Hussain)

Universiti Teknologi PETRONAS Bandar Seri Iskandar 31750 Tronoh Perak Darul Ridzuan

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 the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.

(ALIFF IDHAM BIN MOHAMMAD LATPI) Mechanical Engineering Department, Universiti Teknologi PETRONAS.

ABSTRACT

The author's Final Year Project, which entitled Endurance investigation of Nitrided Ferritic SS439 Stainless Steel under Torsional Load. The purpose of the project is to investigate the effect of nitriding on the endurance of the nitride ferritic SS439 stainless steel under torsional loading condition. Nitriding is a thermochemical surface treatment for strengthen steel purpose. In the process, nitrogen is transferred and may diffuse into the steel at a certain temperature. Nitriding process recently becomes one of the attractive case-hardening processes for steel due to its metallurgical achievement like high surface hardness, improvement in fatigue life and increased wear resistance and anti-galling properties. The scope of this project includes application of nitriding process to the Ferritic Stainless Steel SS439 and endurance investigation for the samples before and after the nitriding process. In this project, low and high temperature gas nitriding has been applied to ferritic stainless steel SS439. Microstructure, hardness testing, and also fatigue testing has been done to the base, low and high temperature nitride SS439 to compare the phase changes happened to the SS439, and also to know the endurance of the material after treated with the nitriding process. The overall result shown that high temperature nitriding has higher efficiency than low temperature nitriding to the ferritic stainless steel SS439. In hardness test, hardness value for high temperature nitride is higher than low temperature nitride on SS439 on the sample surface and below the surface. The torsional fatigue testing proved that the endurance of nitride SS439 has been improved from the base SS439. High temperature nitride SS439 is higher endurance compared to low temperature nitride SS439 with cycle to failure up to 500000 under 10N loading.

ACKNOWLEDGEMENT

First and foremost, I would like to express my greatest gratitude to *Allah S.W.T* for His blessings during my strenuous times and for His gift of the health and the ability for me to complete this Final Year Project within these two semesters. I have been received generous supports from several persons who contributed in different ways to the process of completion of this project.

I would like to take this opportunity to express my profound gratitude and deep regard to my supervisor, Ir Haji Idris Bin Ibrahim and Assoc. Prof. Dr Patthi Bin Hussain for his exemplary guidance, monitoring and constant encouragement throughout the course of this project. The blessing, guidance and help given by him time to time shall carry me a long way in the journey of life on which I am about to embark.

I also take this opportunity to express my sense of gratitude to Dr Syuaib Mohamed for his tireless advices, supports and guidance, which helped me in completing this project especially during handling the furnace for nitring process. My deep appreciation goes to Dr Saravanan A/L Karuppanan and Mr Kamal Ariff Bin Zainal Abidin for their co-operation and willingness to evaluate my project works. I highly appreciated their invaluable criticism and guidance.

Besides, I am obliged to the technicians for their valuable information, cooperation and assistance during my period of completing the project. This project would not have been completed without receiving help from them. Lastly, my special thanks to my colleagues and friends for their constant encouragement without which this project would not be possible.

TABLE OF CONTENT

CHAPTER 1 - INTRODUCTION

1.1 Background of Study	1-2
1.2 Problem Statement	2
1.3 Objective	3
1.4 Scope of Study	3
1.5 Relevancy of Study	3
1.6 Feasibility of Study	4

CHAPTER 2 LITERATURE REVIEW

2.1 Ferritic Stainless Steel SS439	5
2.2 Fundamental of Nitriding Process	5-6
2.2.1 Gas Nitriding	6-10
2.3 Reliability Analysis	10-11

CHAPTER 3 METHODOLOGY AND PROJECT ACTIVITIES

3.1 Flow Chart	12
3.2 Project Activities	13-20

CHAPTER 4 GANTT CHART AND KEY MILESTONES

4.1 Gantt Chart	21
4.2 Key Milestones	22

CHAPTER 6 CONCLUSION	1
----------------------	---

LIST OF FIGURES

Figure 2.0: Schematic compound layer and diffusion zone structure of nitride iron/steel
Figure 2.1: Microstructure of ferrite and martensite
Figure 2.2: Partially changed phase after nitriding process
Figure 2.3: Fully transformed phase after nitriding process
Figure 2.4: Hardness vs Depth below surface graph10
Figure 3.0: Project flow chart12
Figure 3.1: Samples detail dimension13
Figure 3.2: Gas nitriding equipment setting14
Figure 3.3: Fatigue testing machine15
Figure 3.4: Indentation of Vickers hardness testing16
Figure 3.5: Abrassive Cutter
Figure 3.6: Automatic mounting machine
Figure 3.7: Grinder and polisher machine
Figure 3.8: Optical microscope
Figure 5.0: Microstructure of Base SS439 sample24
Figure 5.1: Microstructure of Low Temperature Nitriding SS439 sample25
Figure 5.2: Microstructure of High Temperature Nitriding SS439 sample26
Figure 5.3: Rockwell Hardness value comparison
Figure 5.4: Hardness as the function of the depth below the surface after nitriding treatment for 10 hours
Figure 5.5: Endurance of base, low temperature nitride, high temperature nitride SS439

LIST OF TABLE

Table 2.0: Chemical composition of ferritic stainless steel SS439	5
Table 3.0: Quantity of sample for each category experiment	13
Table 3.1: Parameters for nitriding process	14
Table 3.2: Sample mounting parameters	17
Table 5.0: Rockwell Hardness Value for Base SS439	27
Table 5.1: Rockwell Hardness Value for Low Temperature Nitriding SS439	27
Table 5.2: Rockwell Hardness Value for High Temperature Nitriding SS439	27
Table 5.3: Vickers Hardness Value for Base SS439	29
Table 5.4: Vickers Hardness Value for Low Temperature Nitriding SS439	29
Table 5.5: Vickers Hardness Value for High Temperature Nitriding SS439.	29
Table 5.6: Cycle to failure data for Base SS439	31
Table 5.7: Cycle to failure data for Low Temperature Nitriding SS439	31
Table 5.8: Cycle to failure data for High Temperature Nitriding SS439.	31

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Stainless steels are widely used in industrial sectors nowadays such as oil and gas, chemical, food, and other industries. It is because of corrosion-resistant properties of the steel. However, low hardness and poor wear resistant sometimes limit its industrial application. In this study, Stainless Steel SS439 will be used as the sample in the experiment that will be carried out. SS439 has special attention to chemical composition combined with special processing give this SS439 optimum formability that is reproducible, coil after coil and heat after heat. Engineering surface techniques is required in industry to improve workpiece performance and thus increase in wear resistance.

Heat treatment is one of the surface engineering techniques where it modified the microstructure of the materials. The resulting phase transformation influences mechanical properties like strength, ductility, toughness, hardness and wear resistance. Heat treatment purpose is to increase service life of a product by increasing its strength or hardness, or prepare the material for improved manufacturability. Hardening, annealing, normalizing, tempering, and surface hardening are examples of heat treatment processes. There are many techniques that can be categorize under surface hardening such as nitriding, carburizing, flame hardening, and many more. However this project will only using nitriding as the surface hardening process to the Stainless Steel SS439.

The nitriding process was developed in the early 1900s and it is continues to play an important role in many industrial application. There are various type of nitriding process such as gas nitriding, plasma nitriding, and salt bath nitriding. In this study, the author will be focused on gas nitriding process for the Stainless Steel SS439. Nitriding is a ferritic thermochemical method of diffusing nitrogen into the surface of steels and cast irons. This diffusion process is based on the solubility of nitrogen in iron and the solubility is temperature dependent and also with other parameters. The metal that has been through the nitriding process will improved in wear and enhance in term of fatigue endurance.

Reliability is the probability that the item will perform it required function under given condition within time interval [8]. Until this time present, there are not so much and widely documented regarding the endurance and reliability of the nitride ferritic Stainless Steel SS439. So that the reliability of the nitrided material under dynamic loading will be studied in this project..

1.2 Problem Statement

Ferritic stainless steel SS439 is not one the most commonly used materials in industrial application nowadays as compared to the austenitic SS316 or SS304 grade stainless steel. However Ferritic Stainless Steel SS439 has potential to widely used in all sectors in industrial because of its good thermal properties, but the steels is non-hardenable. Nitriding is one of the cases hardening process producing a nitride ferritic stainless steel with high hardness characteristic at the expense of other mechanical and thermal properties and possible improving of the reliability of the material. Thus far, not many literatures can be found investigating Reliability analysis of Nitrided Ferritic SS439 Stainless Steel Rods under torsional loading conditions. Therefore, this study will investigate the effect of niriding on the endurance of the Nitrided Ferritic SS439 stainless steel rod under torsional loading conditions. The study is required since there are not widely literature can be found in this topic and result from this study will help expand the application of the nitride ferritic SS439 stainless steel in the industry.

1.3 Objective

The objectives of this project are:

- 1. To investigate the effect of nitriding on the endurance of the nitride Ferritic Stainless Steel SS439 under torsional loading.
- 2. To compare the hardness and microstructure between unnitrided and nitrided Ferritic Stainless Steel SS439.

1.4 Scope of Study

The study will be focused on information and data gathering for literature review of the nitriding process, all the process parameter, and the procedure for the gas nitriding process. The study also will include the chemical composition, microstructure profile of the unnitrided SS439. The sources of the information can be from books, journals, websites, technical paper, research paper, articles, and thesis. Gas nitriding process and lab works will be carried out for the samples SS439 Stainless. Nitrided Stainless Steel SS439's chemical composition and microstructure assessment also will be carried out to compare the two elements before it is nitride. Hardness test will be done in order to compare the hardness value between nitride and unnitrided samples. Then fatigue testing will be done to the samples to do analysis for the effect of nitriding on the endurance of the nitrided ferritic SS439 stainless steel rod. Modeling is not covered in this study.

1.5 Relevancy of Study

- 1. Few researches done on the nitriding process to the ferritic stainless steel SS439.
- 2. Ferritic Stainless Steel SS439 have high heat resistance.

1.6 Feasibility of Study

- 1. Specified objective and scope of studies
- 2. This project can be done within the time frame given.
- 3. All equipment needed are available in UTP laboratory.

CHAPTER 2

LITERATURE REVIEW

2.1 Ferritic Stainless Steel SS439

SS439 stainless steel is one of the widely used ferritic stainless steels in industrial. The steel has well both oxidation and corrosion and heat resistant [11,12]. Special attention to chemical composition combined with special processing give this SS439 optimum formability that is reproducible, coil after coil and heat after heat [11].

Examples of applications for the SS439 in industrial are cold and hot water tanks, boilers, heat exchangers, and solar water heaters. Besides, SS430 also used for home and office equipment such as kitchen ware, sinks, hot water services, stoves, trolleys, food handling equipment, etc [12].

Composition	Percentage
Carbon	0.025
Silicon	0.75
Chromium	17.00 - 19.00
Manganese	0.50
Nickel	0.50
Titanium	0.50

Table 2.0: Chemical composition of ferritic stainless steel SS439 [11]

2.2 Fundamental of nitriding process.

Nitriding is a thermochemical surface treatment for strengthen steel purpose [3,4]. In the process, nitrogen is transferred and may diffuse into the steel at a certain temperature. The nitrogen precipitated in the form of nitrite in the steel. A compound layer known as white layer is formed on the steel's surface. The white layer consist of Fe2(N,C)1-x and Fe4N phases [2,3,4]. Below the compound layer is known as

diffusion zone, where the nitrogen from the outside are dissolves interstitially in the ferrite lattice at the nitriding temperature.

Nitriding process recently becomes one of the attractive case-hardening processes for steel due to its metallurgical achievement like high surface hardness, improvement in fatigue life and increased wear resistance and anti-galling properties [1,2]. Nitriding produces less distortion and deformation at the heat treated parts because of relatively low temperature employed in the process [4]. There are various nitriding processes that have been implemented in the steel case hardening. The processes can be classified as gas nitriding, plasma nitriding, and salt bath nitriding. Nitriding is an important surface heat treatment for ferritic steels and can be widely used. Therefore, this project will be focused on gas nitriding process.



T=570°C(ferritic) ↓↓↓N from medium

Figure 2.0: Schematic compound layer and diffusion zone structure of nitride iron/steel [4].

2.2.1 Gas Nitriding

Gas nitriding is a case hardening process whereby nitrogen is introduced into the surface of a solid ferrous alloy by holding the metal at suitable temperature in contact with nitrogenous gas, which is usually ammonia [2]. 495 to 565 °C are conventional nitriding temperature for steels and considered as low-temperature gas nitriding [3]. Ammonia is an unstable thermodynamic state and decomposes to nitrogen and hydrogen at that particular temperature. Nitrogen atoms released have the opportunity to either chemically react with or diffuse between iron atoms [2]. Compound layer or so called white layer are formed at the surface, it is the formation of iron nitrides [2,3]. The compound layers are combination of FexN and CrxN, which are nitrogen solid solution and nitrides. Nascent nitrogen atoms interstially diffuse into octahedral interstices of BCC structured iron known as diffusion zone, it is beneath the compound layer [2].

There is high temperature gas nitriding, also known as solution nitriding. The process temperature is 1000 to 1200 °C exposed in nitrogen gas atmosphere [3,6]. High temperature gas nitriding is new method of nitrogen addition involving diffusion process for nitrogen to permeate the surface of stainless steel through heat treatment in N2 atmosphere at high temperature. After the process, hardened case will be obtained after being quenched. Strength and hardness of the steel will be increase by the nitrogen diffusion that occurs. This high temperature gas nitriding is widely used for improving the hardness of martensitic, austenitic, ferritic, and duplex stainless steels [3,6,7]. High temperature nitriding is more effective and more recognizes compared to low temperature gas nitriding in term of nitrogen diffusion [3].

Temperature and period factor during the gas nitriding becomes a very important parameter to the process. This is because it can affect the surface changes, nitrogen content and corrosion resistance [6]. It takes time to change phase from ferrite to martensite during the process. The time taken is about 10 hours at 1100 °C for 1.2mm thickness steel sample for the martensite phase depth to increase and changed the ferrite to the whole martensite phase [6]. The situation had shown that growth kinetics of the nitride layer depending on the process time and also nitriding potential [7]. Example of optical micrographs of Ferritic Stainless Steel after the high temperature gas nitriding at two different temperature and period are as on the figure below:



Figure 2.1: Microstructure of ferrite and martensite

For the figure 2.1, the nitriding process occurs at 1100 °C for 15 minutes. The near surface layer of about 130 μ m of martensitetic phase is observed, while at further depth, there is still in ferrite phase [6].



Figure 2.2: Partially changed phase after nitriding process.

For the figure 2.2, the nitriding process occurs at 1100 °C for 10 hours. About 1.2 mm of sample depth has change from ferrite to martensite phase



Figure 2.3: Fully transform phase of ferritic to martensite

Nitriding process occurs at 1050 °C for 10 hours are as shown in the figure 2.3 above. The relatively fine grain size was observed and the specimen shows the whole thickness of the steel consist of martensite phase.



Figure 2.4: Hardness vs Depth below surface graph.

Figure 2.4 shows that the hardness decreases gradually in proportional to the nitrogen content. This hardness variation is related to the nitrogen content and phases appeared on the surface layer. Therefore, nitriding process has increase the surface hardness of steels and other properties.

2.3 Reliability Analysis

In the industries, engineering analysis has advanced to improve reliability of an engineering system while considering system input uncertainties. Reliability can be defined as the probability that a system or component will perform a required function for a given period of time when used under stated operation conditions. [8,9,10]. Reliability represents safety level in industry practice and may variant due to time-variant operation condition and components deterioration throughout a product lifecycle [9]. Therefore, the capability of performing reliability analysis is very important in practical engineering applications. In product development nowadays, many companies are struggling to maintain and make it balance between the product performance and technical innovation [14]. They realize that the reliability of products is the key for the profit and also involving the quality and the company's image itself. The most common criteria to replace or repair a product in the industrial application is a technical product failure [13,14]. High reliability products are able to give benefit to user in industrials sector especially in term of economical and maintainability, as we know the cost for maintenance when include the replacement of the product will be very high. Thus, decrease the efficiency of the process and also the maintainability. In cases where the economic lifetime is much shorter than the technical lifetime, companies will easily replace fully functional products. If the reverse is the case companies will seek ways to extend the product life cycle by some strategies and technologies that involved advanced materials available. Most of the time, seek for products in high reliability is the best way rather than new product replacement to overcome the pre-mature failure hence save the capital and operational cost [14,15].

There are several of the causes of failure, and fatigue is one of the main causes of failures in various structural designs. Most of the structures in industries will subjected to dynamic loadings over a lifetime, local failures may initiate cascading failures that may lead to disproportionally large damage or can be collapse [10]. Reliability analysis cannot give the exact value data of a product life cycle, however can surely estimate it and very useful to expect the failure for maintainability. The accidents in industrials sectors will involving a huge cost and affect the socio-economic cost. So that, the reliability analysis is very important for maintenance purposes especially in order to avoid pre-mature failure to the system.

CHAPTER 3

METHODOLOGY AND PROJECT ACTIVITIES

3.1 Flow Chart



3.2 Project Activities

3.2.1 Sample Size

Samples in this project will be in dog bone shape. The reason of choosing the particular shape because for compatibility of the sample with the fatigue testing machine that will be carried out. Details information for dimensions and number of the samples is as shown in the table and figure below.

Table 3.0: Quantity of sample for each category experiment

Type of Samples	Number of Samples
Unnitrided Ferritic Stainless Steel SS439	9
Low Temperature Nitrided Ferritic Stainless Steel SS439	9
High Temperature Nitrided Ferritic Stainless Steel SS439	9



Figure 3.1: Samples detail dimension

3.2.2 Gas Nitriding Process (Low and High Temperature)

Experiment Procedure:

- 1. Gas nitriding treatment will apply to ferritic stainless steel SS439.
- 2. The gas nitriding will be carried out in a Alumina horizontal tube furnace.
- 3. Gas flow meter is attached between the nitrogen gas cylinder and furnace tube in order to adjust the flow rate and also to maintain constant flow rate during the treatment.
- 4. Gas outlet tube from the furnace tube is connected to the Drechsel bottle to slightly increase the gas pressure flown in the furnace.
- 5. The sample will be place inside the furnace tube on the sample boat. The sample must be in the middle of furnace tube right between the heating elements.
- 6. The treatment is operates at 1100°C (High Temperature) and 500°C (Low Temperature) for 10 hours.
- 7. The sample will be quenched into water after the treatment.

Other parameters for the nitriding process are as in the table below:

Parameters	Value
Pressure	50 – 100 mmHg
Gas	Purified Nitrogen
Temperature rate	10°C/min
Flow rate	1000 cm ³ /min

Table 3.1: Parameters for nitriding process.



Figure 3.2: Gas Nitriding equipment setting

3.2.3 Fatigue Testing

Experiment Procedure:

- 1. Conduct the fatigue test at room temperature using the fatigue testing machine as shown in figure below for 9 unnitrided, 9 low temperature and 9 high temperature nitriding ferritic stainless steel SS439.
- 2. Fit one end of the specimen to a motor and fit the other end to a bearing hung with a known weight, indicating the stress applied to the specimen.
- 3. Start the motor to rotate the specimen at a constant speed. The revolution counter is used to record the number of cycles to which the specimen fails.
- 4. Change the weights used and do the fatigue testing with another samples.
- 5. Repeat steps number 1 to 5 with nitride ferritic stainless steel SS439.
- 6. Construct the S-N curves of the stainless steel samples.
- 7. Compare the S-N and number of cycle to failure between unnitrided and low and high temperature nitride stainless steel SS439.



Figure 3.3: Fatigue Testing Machine

3.2.4 Vickers Hardness Test

Vickers hardness test procedure:

- 1. Polish the surface of unnitrided ferritic stainless steel SS439 samples by using sand paper.
- 2. Hardness measurement is carried out using Vickers hardness testing techniques on the prepared surfaces at 4 positions on each sample.
- 3. Press the indenter into the sample by an accurately controlled test force.
- 4. Maintain the force for a specific dwell time between 10 to 15 seconds. Remove the indenter once dwell time completed.
- 5. The reading of hardness value is recorded in a table.
- 6. Steps 1 to 5 are repeated for low and high nitrided ferritic stainless steel SS439.
- 7. Compare the hardness reading between unnitrided and nitride ferritic stainless steel SS439.



Figure 3.4: Indentation of Vickers hardness testing.

The formula below can be used to calculate the Vickers hardness value (VCN):

$$HV = 1.854 F / d^2$$

Where, HV = Hardness Value

F = applied force

 $d = average \ length \ of \ the \ diagonals = (d1 + d2) / 2$, mm

3.2.5 Rockwell Hardness Testing

- 1. An appropriate scale is chosen to be used.
- 2. The indenter moves down into position on the part surface
- 3. A minor load is applied and a zero reference position is established.
- 4. The major load is applied for a specified time period (dwell time) beyond zero.
- 5. The major load is released leaving the minor load applied.
- 6. The measurement is taken.

3.2.6 Abrassive cutter

- 1. Abrassive cutter is used to cut the samples into smaller dimension.
- 2. Samples is clamped and lock to make sure it is at fixed position while cutting process.
- 3. Close the machine cover for safety during cutting process.
- 4. Take out the samples after the cutting process finished.



Figure 3.5: Abrasive Cutter

3.2.7 Sample Mounting

- 1. Sample is placed at the stages together with phenolic powder.
- 2. Settle down the sample and push the start button for the process.
- 3. The parameter for this mounting process are as in the table below:

Table 3.2: Sample mounting parameters.

Parameter	Description
Heating time	3 minutes
Cooling time	2 minutes
Pressure	4000 psi



Figure 3.6: Automatic Mounting Machine

3.2.8 Grinding and Polishing

1. Switch on the motor and the tap water on the grinding machine.

2. Start grinding the sample with P400 grinding paper for about 5 to 8 minutes.

3. Change the grinding paper to other grade in increasing grade order. (P800, P1200,P1500,P2400, and P2500).

4. Lastly, polish the sample with the finest polish paper. The diamond paste is applied at the paper before start polish until mirror light is obtained from the sample.



Figure 3.7: Grinder and Polisher Machine

3.2.9 Etching

1. Kalling solution is prepared for the etching process.

2. 100 ml of hydrochloric acid, 100 ml of ethanol, and 5 grams of copper(II) sulphate are mixed in one beaker to produce the Kalling solution.

3. Sample is washed with tap water following by the ethanol to remove the water from the sample surface.

4. The sample is immersed in Kalling solution for 45 second until the sample surface become dull.

5. Sample is taken out from the solution and washed with tap water followed by ethanol again.

6. Let the sample dry before proceed to the optical microscope.

3.2.10 Optical Microscope

- 1. Clear the surface of unnitrided ferritic stainless steel SS430 and make sure it is in dry condition.
- 2. Switch on the optical microscope and all computer system.
- 3. Place the sample under the magnifying glass. Focus and capture the image, save it in the computer.
- 4. Repeat steps 1 to 3 for low and high temperature nitride ferritic stainless steel SS439.
- 5. Switch off the optical microscope.
- 6. Compare the results image between unnitrided and nitride ferritic stainless steel SS439.



Figure 3.8: Optical Microscope

CHAPTER 4

GANTT CHART AND KEY MILESTONES

4.1 Gantt Chart

Project		Week																											
Activities								FYF	21													F	YP 2						
	1 2 3 4 5 6 7 8 9 10 11 12 13 14									15	16	17	18	19	20	21	22	23	24	25	26	27	28						
Project draft																													
Literature																													
review																													
Preparation of																													
extended																													
proposal																													
Extended																													
proposal																													
submission																													
Experiment																													
procedure																													
development																													
Receiving																													
Material																													
Sample																													
preparation																													
Proposal																													
defense																													
Preparation of																													
interim report																													
Interim report																													
submission																													
Lab experiment																													
Correlation for																													
reliability and																													
performance																													
Documentation																													
Final report																													
submission																													

Legend:



4.2 Key Milestones

- Extended proposal submission
 - Submission of the extended proposal to supervisor and FYP1 coordinator.
- Receiving Sample Stainless Steel SS439.
 - Receiving samples from supplier with suitable dimension for experimental purpose.
- ✤ Interim report submission.
 - Complete the interim report and submit to supervisor and FYP1 coordinator.
- ✤ Final report/technical paper submission and VIVA assessment.
 - Complete all documentation of the project and submit to supervisor and FYP2 coordinator.
 - Viva session with supervisor, internal and external examiner.

CHAPTER 5

RESULT AND DISCUSSION

5.1 Microstructure

Figures below shows Optical Microscope (OM) image of the cross-section through the surface and the interior of the Ferritic Stainless Steel SS439. Figure 5.0 is the OM image for base SS439 and Figure 5.1 is the low temperature nitride and high temperature nitride is referred to Figure 5.2. The treated layers was clearly visible. Based on the captured image of the microstructure, the modified layer of the SS439 ferritic stainless steel appear in the samples after ething with Kalling's soulution. The diffusion affected zone was developed and observed from the image.

After the low temperature nitriding treatment at 500 °C for 10 hours, the near surface layer is observed about 300µm of martensitic was observed. Below the martensitic layer, the ferrite phase was still observed in the interior in the figure 5.1. Means that the low temperature nitriding treatment is unable to change all the ferrite to martensitic phase at the SS439 samples. After the high temperature nitriding to the SS439 at 1100 °C for 10 hours, it is observed that the whole thickness of sample was changed phase from ferritic to martensitic. The diffusion of nitrogen almost contant at the particular parameter in nitriding treatment.

As conclusion for metallurgraphy, since high temperature nitriding develop fully phase changes to the SS439, the low temperature nitriding treatment at 500 °C for 10 hours can be considered as not sufficient enough to the SS439 to be nitride at the parameters. The statement can be supported by Hakan Aydin in his journal tittled "Friction characteristics of Nitrided Layers on AISI 430 Ferritic Stainless Steel Obtained by Various Nitriding Processes" stated that gas nitriding procedure at 570 °C for 13 hours was not sufficient to form a hard nitride layer on AISI 430 ferritic stainless steel surfaces.



Figure 5.0: Microstructure of Base SS439 Sample (100x Magnificent)



Figure 5.1: Microstructure of SS439 Low Temperature Nitriding Sample (100x Magnificent)



Figure 5.2: Microstructure of SS439 High Temperature Nitriding Sample (100x Magnificent)

5.2 Rockwell Hardness Testing

Rockwell Hardness Testing is used to examine the hardness at the surface of base SS439, low temperature nitride SS439, and high temperature SS439 samples. For each sample, five indent points are made to take the average Rockwell Hardness value. The details of data are as in the table below.

Sample Reference	Indentation Point	Rockwell Hardness Value	Average Value		
	А	94.4			
	В	93.4			
	С	94.2	94.4		
	D	95.5			
	Е	94.5			

Table 5.0: Rockwell Hardness Value for Base SS439

Table 5.1: Rockwell Hardness Value for Low Temperature Nitrided SS439

Sample Reference	Indentation Point	Rockwell Hardness Value	Average Value		
	А	98.6			
	В	97.9			
	С	98.5	98.3		
	D	98.5	2010		
	Ε	98.1			

Table 5.2: Rockwell Hardness Value for High Temperature Nitrided SS439

Sampla Deference	Indontation Daint	Rockwell Hardness	Average Value	
Sample Reference	Indentation Point	Value		
	А	108.3		
	В	109.5		
	С	109.7	109.4	
\bigcirc	D	109.4		
	Е	109.9		



Figure 5.3: Rockwell Hardness Value Comparison

From the testing, the result shown Rockwell Hardness Value (RHV) for Base SS439 is 94.4, low temperature nitride SS439 is 98.3, while high temperature nitride SS439 is 109.4. By comparison, LTN SS439 hardness value is slightly higher than the hardness value of base SS439 by 4.13%. There is not much increase in hardness after low temperature nitride treatment. After the SS439 is received high temperature nitriding treatment, the hardness value increased from 94.4 to 109.4, which is the value up to 15.89%. Therefore, high temperature nitriding treatment improved the SS439 in term of hardness efficiently than low temperature nitriding.

5.3 Vickers Hardness Testing

In order to know the hardness changes beneath the surface, Vickers Hardness testing is used. Hardness readings are taken at the sample cross sectional area, starting from the surface and increasing the depth by 20µm under the surface. The reading is taken for base SS439, low temperature nitride SS439, and high temperature SS439 samples. Data are recorded as in the table below.

Table 5.3: Vickers Hardness Value for Base SS439

Type of	Depth (µm)								
sample	0	20	40	60	80				
Base SS439	230.5	241.3	243.6	245.4	244.3				

Table 5.4: Vickers Hardness Value for Low Temperature Nitrided SS439

Type of	Depth (µm)								
sample	0	20	40	60	80				
LTN SS439	411.3	405.1	399.9	339.1	266.1				

Table 5.5: Vickers Hardness Value for High Temperature Nitrided SS439

Type of	Depth (µm)								
sample	0	20	40	60	80				
HTN SS439	651.7	599.4	428.9	363.2	355.0				



Figure 5.4: Hardness as the function of the depth below the surface after nitriding treatment for 10 hours.

Figure 5.4 shows the hardness variation with depth below the surface after low and high temperature nitriding treatment. The hardness decrease gradually in proportionally to the nitrogen content for high and low temperature nitriding [6]. Meanwhile for base SS439, the hardness value at the depth below surface was relatively unchanged. The highest hardness achieved at the surface of the high temperature nitride SS439 which is 651.7 and starts to decrease in hardness observed at 20μ m, 40μ m, and so on. Same goes to the low temperature nitride SS439, the hardness value observed to decrease below the surface depth. The highest hardness value also recorded at the surface which is 411.3. This hardness variation is related to the nitrogen content and phases appeared on the cross sectional depth area [6]. In low temperature nitriding treatment, not fully phase transform from ferrite to martensite induced the lower hardness. Overall hardness value for high temperature nitride is higher than low temperature nitride on SS439.

5.4 Torsional Fatigue Testing

Torsional fatigue testing is carried out for strength analysis for before and after nitriding process or the case hardening application to the SS439. The testing is running until the samples are failed for different load in each run. Cycle to failure reading is taken for base SS439, low temperature nitride SS439, and high temperature SS439 samples for each load. Data are recorded as in the table below.

Type of		Load								
material	10	15	20	25	30	35	40	45	50	
Base	221766	218174	207293	166615	159774	115212	60003	46151	30786	
SS439										

Table 5.7: Cycle to failure data for Low Temperature Nitrided SS439

Type of		Load									
material	10	15	20	25	30	35	40	45	50		
LTN	366906	291007	237756	225195	200210	141008	65906	51703	39666		
SS439											

Table	5 8.	Cycle t	o failure	data for	High	Temperature	Nitrided	SS439
1 abie	5.0.	Cyclet	0 ranuic	uata 101	Ingn	remperature	minucu	22422

Type of	Load									
material	10	15	20	25	30	35	40	45	50	
HTN	503048	418889	355021	346905	324644	221772	103001	82914	71228	
SS439										



Figure 5.5: Endurance of base, Low Temperature Nitride, High Temperature Nitride SS439

Figure 5.5 shows the cycle to failure of base, low and high temperature nitride SS439 under torsional loading condition. The nine samples from each category were run under torsional loading with different set of load starting from 10 Newton to 50 Newton. Base SS439 was observed can stand the cycle up to more than 200000 until it reach the failure under 10 Newton torsional load. Cycle to failure decreases gradually as the load of torsional was increased. At 50 Newton, the base SS439 only can stand around 30000 of cycle until it fail.

From the observation in the figure xx, the endurance of low temperature nitride SS439 was impressively improved from the base SS439. It was improved up to 65% and the cycle to failure at 10 Newton is about 360000. Same like base SS439, the cycle to failure decrease as the applied torsional load was increased. However, the endurance for low nitride SS439 was all more than the endurance of base SS439 under all set of torsional loading.

High temperature nitride SS439 has the higher endurance from those previous two categories. At 10 Newton of torsional loading, the cycle to failure is more than 500000. Compared to base SS439, the improved endurance is around 127% and 37% if compared to low temperature nitride SS439. Cycle to failure decreases gradually as the load of torsional was increased. At 50 Newton, the high temperature nitride SS439 can stand around 70000 of cycle until it fail.

As conclusion, the torsional fatigue testing proved that the endurance of nitride SS439 has been improved from the base SS439. High temperature nitride SS439 is higher endurance compared to low temperature nitride SS439.

CHAPTER 6 CONCLUSION

In this work, the endurance of base and nitride Ferritic Stainless Steel SS39 (Low and high temperature nitriding) was investigated using hardness test and also defining its cycle to failure by torsional fatigue testing. The phase changes of the material before and after the case hardening treatment also investigated by using optical microscope. The conclusions derived from the study can be given as follow:

Metallurgraphy analysis in this study conclude that, since high temperature nitriding develop fully phase changes to the SS439, the low temperature nitriding treatment at 500 °C for 10 hours can be considered as not sufficient enough to the SS439 to be nitride at the parameters. The statement can be supported by Hakan Aydin in his journal tittled "Friction characteristics of Nitrided Layers on AISI 430 Ferritic Stainless Steel Obtained by Various Nitriding Processes" stated that gas nitriding procedure at 570 °C for 13 hours was not sufficient to form a hard nitride layer on AISI 430 ferritic stainless steel surfaces.

After the SS439 is received high temperature nitriding treatment, the hardness value at the surface increased from 94.4 to 109.4, which is the value up to 15.89% compared to low temperature nitriding treatment that only improved the hardness only about 4.13% in the Rockwell Hardness Testing. Therefore, high temperature nitriding treatment improved the SS439 in term of hardness efficiently than low temperature nitriding. In Vickers Hardness Testing, the hardness decrease gradually in proportionally to the nitrogen content for high and low temperature nitriding. Overall hardness value for high temperature nitride is higher than low temperature nitride on SS439.

The torsional fatigue testing proved that the endurance of nitride SS439 has been improved from the base SS439. High temperature nitride SS439 is higher endurance compared to low temperature nitride SS439 with cycle to failure up to 500000 under 10N loading. Therefore, the objectives of the study have been met.

Reference

- Hakan Aydin, Ali Bayram, and Sukru Topcu, "Friction Characteristic of Nitrided Layer on AISI 430 Ferritic Stainless Steel Obtained by Various Nitriding Processs," *Material Science (Medziagotyra)*, vol. 19, No 1, 2013.
- [2] S.M. Hassani-Gangaraj and M. Guagliano, "Microstructural evolution during nitriding, finite element simulation and experimental assessment," *Applied Surface Science* 271, pp 156-163, 2013.
- [3] Hudiyo Firmanto, "Reaction Layers In Diffusion Bonded of Sialon to Ferritic Steel," Doctor of Philosophy Mechanical Engineering Universiti Teknologi PETRONAS, 2011.
- [4] Mei Yang, "Nitriding fundamental, modeling and process optimization," *Worcester Polytechnic Institute*, 2012.
- [5] Hans Berns, Andreas Kuhl, "Reduction in wear of sewage pump through solution nitriding," *Wear*, Vol 256, pp 16-20, 2004.
- [6] J.H. Sung, J.H. Kong, D.K. Yoo. H.Y. On, D.J. Lee, H.W. Lee, "Phase changes of the AISI 430 ferritic stainless steels after high-temperature gas nitriding and tempering heat treatment," *Material Science and Engineering*, Vol 489, pp 38-43, 2008.
- [7] Jerzy Ratajski, Tomasz Suszko, "Modelling of the nitriding process", *Journal of Material Processing Technology*, Vol. 95, pp. 212-217, 2008.
- [8] Javad Barabady, Uday Kumar, "Reliability analysis of mining equipment: A case study of a crushing plant at Jajarm Bauxite Mine in Iran," *Reliability Engineering and safety System*, Vol 93, pp 647-653, 2008.
- [9] Zequn Wang, Pingfeng Wang, "A new approach for reliability analysis with time-variant performance characteristics," *Reliability Engineering and System Safety*, Vol. 115, pp. 70-81, 2013.

- [10] Young-Joo Lee, Junho Song, "Finite-element-based system reliability analysis og fatigue-induced sequencial failures," *Reliability and Safety System*, Vol. 108, pp.131-141, 2012.
- [11] www.aksteel.com, "Product Data Sheet 439 Stainless Steel", (Accessed: 2013,June, 08)
- [12] www.atlassteels.com.au, "Ferritic Stainless Steel Sheet, Coil & Plate", (Accessed: 2013,June, 08).
- [13] Man Cheol Kim, Poong Hyun Seong, "Reliability graph with general gates: an intuitive and practical method for system reliability analysis," *Reliability Engineering and System Safety*, Vol. 78, pp. 239-246, 2002.
- [14] A.C. Brombacher, P.C. Sander, P.J.M. Sonnemans, J.L. Rouvroye,
 "Manging product reliability in business processes 'under pressure'," *Reliability Engineering and System Safety*, Vol. 88, pp. 137-146, 2005.
- [15] J.H. Saleh, K. Marais, "Reliability: How much is it worth? Beyond its estimation or prediction, the (net) present value of reliability," *Reliability Engineering and System Safety*, Vol. 91, pp. 665-673, 2006.
- [16] B.Meyer, "Ferritic Cane Technol Stainless Steel AISI 439 For The Sugar Industry," Proc. Int. Soc. Sugar Cane Technol., Vol. 27, 2010.
- [17] Antonio Claret Soares Sabioni, Anne-Marie Huntz, Elizete Conceicao da Luz, Marc Mantel, Christian Haut, "Comparative Study of High Temperature Oxidation Behavior in AISI 304 and AISI 439 Stainless Steel," *Material Research*, Vol. 6, No 2, pp.179-185, 2003.
- [18] A.M. Huntz, A. Reckmann, C. Haut, C. Severac, M. Herbst, F.C.T. Resende, A.C.S. Sabioni, "Oxidation of AISI 304 and AISI 439 stainless steels," *Materials Science and Engineering*, Vol. 447, pp 266-276, 2007.
- [19] E. Skolek-Stefaniszyn, J. Kaminski, J. Sobczak, T. Wierzhon,
 "Modifying the properties of AISI 316L steel by glow discharge assited lowtemperature nitriding and oxynitriding," *Vacuum*, Vol. 85, pp. 164-169, 2010.
- [20] E. Menthe, A. Bulak, J. Olfe, A. Zimmermann, K.-T. Rie, "Improvemenf of the mechanical properties of austenitic stainless steel after plasma nitriding," *Surface and Coatings Technology*, Vol. 133, pp. 259-263, 2000.

- [21] Carlos E. Pinedo, Waldemar A. Monteiro, "On the kinetics of plasma nitriding a martensitic stainless steel type AISI 420," *Surface and Coatings Technology*, Vol. 179, pp. 119-123, 2004.
- [22] S. Ganesh Sundra Raman, M. Jayaprakash, "Influence of plasma nitriding on plain fatigue and fretting fatigue behavior of AISI 304 austenitic stainless steel," *Surface and Coatings Technology*, Vol. 201, pp. 5906-5911, 2007.
- [23] Kimiaki Nagatsuka, Akio Nishimoto, Katsuya Akamatsu, "Surface hardening of duplex stainless steel by low temperature active screen plasma nitriding," *Surface and Coatings Technology*, Vol. 205, pp. 5295-5299, 2010.
- [24] B. Larisch, U. Brusky, H.-J. Spies, "Plasma nitriding of stainless steels at low temperature," *Surface and Coatings Technology*, Vol. 116, pp. 205-211, 1999.
- [25] M. Fattah, F. Mahboubi, "Comparison of ferritic and austenitic plasma nitriding and nitrocarburizing behavior of AISI 4140 low alloy steel," *Material and Design*, Vol. 31, pp. 3915-3921, 2010.
- [26] Hiroyuki Tsujimura, Takuya Goto, Yasuhiko Ito, "Electrochemical surface nitriding of SUS 430 ferritic stainless steel," *Material Science and Engineering*, Vol A355, pp 315-319, 2003.
- [27] S.A. Nikulin, S.O. Rogachev, V.M. Khatkevich, A.B. Rozhnov, T.A. Nechaykina, "Effect of heat treatment on the structure and mechanical properties of C-Cr-Ti steel after internal nitriding," Journal of Alloys and Compounds, Vol. 564, pp. 114-116, 2013.