

**Comparative Study of the Microstructure,  
Mechanical and Thermal Properties of  
Nitrided and Base Ferritic SS439 Stainless Steel Rod**

By

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Progress report submitted in partial fulfilment of  
the requirements for the  
Bachelor of Engineering (Hons)  
(Mechanical Engineering)

SEPTEMBER 2013

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**CERTIFICATION OF APPROVAL**

**COMPARATIVE STUDY OF THE MICROSTRUCTURE AND  
MECHANICAL AND THERMAL PROPERTIES OF  
NITRIDED AND BASE FERRITIC SS439 STAINLESS STEEL ROD**

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FAISAL BIN ISMAIL

A project dissertation submitted to the

Mechanical Engineering Programme

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in partial fulfilment of the requirement for the

BACHELOR OF ENGINEERING (Hons)

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Approved by,

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(Dr. Patthi Bin Hussain)

Date: 20 DECEMBER 2013

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

**SEPTEMBER 2013**

## **CERTIFICATION OF ORIGINALITY**

This is to certify that I, Faisal Bin Ismail (I/C No : 911130-03-5525), 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.

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## **ABSTRACT**

A thick hydrogen coating layer and dark colour of SS439 were obtained when it is nitrided at high temperature (1100 Celsius). A thin hydrogen coating layer and brighter colour (blue) of SS439 were obtained when it is nitrided at low temperature (500 Celsius). Most of ferrite phase is present based on the microstructure of base SS439 at 100x and 500x magnification. Martensite phase is present in the high temperature nitrided microstructure of the SS439 as well as the ferrite phase. This is due to the heat applied and the penetration of hydrogen into the SS439. Hardness test on the three different samples are done using the Rockwell hardness machine at scale B. The load is set to a constant of 100kgf and 1/16" steel ball is used. Result shows that different samples nitrided at different temperature yield different hardness value. The samples are heated up to 100 Celsius to determine the length of its expansion and to calculate the thermal coefficient of the samples. Three different samples are tested using three point bending test to investigate its stiffness. High temperature nitrided sample shows a 59% of increase in stiffness value.

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# TABLE OF CONTENTS

<b>Abstract</b> .....	<b>i</b>
<b>Acknowledgement</b> .....	<b>ii</b>
<b>List of Figures</b> .....	<b>iv</b>
<b>List of Tables</b> .....	<b>v</b>
<b>Abbreviations and Nomenclature</b> .....	<b>vi</b>
<b>Chapter 1; Introduction</b> .....	<b>1</b>
1.0 Background of Study .....	1
1.1 Problem Statement .....	2
1.2 Objective of Study .....	2
1.3 Scope of Study.....	3
<b>Chapter 2; Literature review</b> .....	<b>4</b>
<b>Chapter 3; Methodology</b> .....	<b>7</b>
3.0 Abrasive Cutter .....	8
3.1 Mounting.....	9
3.2 Grinding and polishing .....	9
3.3 Etchant preparation .....	10
3.4 Etching.....	10
3.5 Optical microscope .....	10
3.6 Nitriding .....	11
3.7 Rockwell hardness test .....	11
3.8 Thermal expansion test.....	13
3.9 Three point bending test.....	15
<b>Chapter 4; Results and discussions</b> .....	<b>16</b>
<b>Chapter 5; Conclusion and recommendations</b> .....	<b>25</b>
<b>References</b> .....	<b>26</b>
<b>Appendices</b> .....	<b>28</b>

## LIST OF FIGURES

Figure 1; White layer and diffusion layer of steel .....	4
Figure 2; Schematic illustration of gas nitriding.....	5
Figure 3; Rockwell hardness test .....	11
Figure 4; Types of sample.....	16
Figure 5; Mounted samples .....	16
Figure 6; Microstructure of Base SS439 at 100x magnification .....	17
Figure 7; Microstructure of Base SS439 at 500x magnification .....	17
Figure 8; Microstructure of Low Temperature Nitrided SS439 at 100x magnification .....	18
Figure 9; Microstructure of Low Temperature Nitrided SS439 at 500x magnification .....	18
Figure 10; Microstructure of High Temperature Nitrided SS439 at 100x magnification .....	19
Figure 11; Microstructure of High Temperature Nitrided SS439 at 500x magnification .....	19
Figure 12; Graph of Rockwell hardness value of three different samples .....	21
Figure 13; Graph of stiffness for three different samples .....	23
Figure 14; Mounting machine .....	29
Figure 15; Nitriding furnace .....	29
Figure 16; Heating / Drying machine .....	29
Figure 17; Kalling's solution .....	30
Figure 18; Fume hood.....	30
Figure 19; Grinding and polishing machine.....	30
Figure 20; Abrasive cutter machine.....	31
Figure 21; Optical Microscope .....	31

## LIST OF TABLES

Table 1; Types of sample and dimensions.....	8
Table 2; Sample data of hardness test .....	12
Table 2; Sample data of thermal expansion test .....	13
Table 4; Sample data of three point bending test .....	14
Table 5; Rockwell hardness value of Base SS439 .....	20
Table 6; Rockwell hardness value of Low Temp. Nitrided SS439.....	20
Table 4; Sample data of three point bending test .....	14
Table 5; Rockwell hardness value of Base SS439 .....	20
Table 6; Rockwell hardness value of Low Temp. Nitrided SS439.....	20
Table 7; Rockwell hardness value of High Temp. Nitrided SS439.....	20
Table 8; Result of the linear thermal expansion test.....	22
Table 9; Deflection of three different samples .....	23



## ABBREVIATIONS AND NOMENCLATURES

wt %	weightage percentage
SS	stainless steel
Fe	iron
N	nitrogen
C	carbon
NH <sub>3</sub>	ammonia
Cr	chromium
Ni	nickel
Ti	titanium
mm	millimetre
psi	pounds per square inch
CuCl <sub>2</sub>	copper chloride
HCl	hydrochloric acid
°C	celsius
mmHg	millimetre mercury (pressure)
VHN	vickers hardness value
k	stiffness (N/mm)
W	weight (kg)
$\delta$	deflection (mm)

# CHAPTER 1

## INTRODUCTION

### 1.0 Background of Study

Heat treatment is a broad term use for all methods of hardening the steel by using heat. There are few methods under heat treatment to harden the steel like tempering, annealing and case hardening [1]. Case hardening allows the interior of the steel retains its original properties only the outer surface is hardened. Case hardening is a process with variety of techniques used to improve the steel properties, by diffusing carbon (carburization) or nitrogen (nitriding) or boron (boriding) into the outer layer of the steel at high temperature and then heat treating the surface layer to the desired hardness without affecting the softer, tough interior of the part [2]. Traditional case hardening steels are unalloyed or low-alloy steels with typical carbon content of 0.15-0.20 wt % [3]. The steels are expected to have an initial microstructure which is a mixture of ferrite and pearlite. The surface of the component is wear resistant where the core remains tough and ductile [4]. The process of nitriding start in early 1900's by Dr. Adolph Fry, to improve the wear and corrosion resistance of some metal as well as to improve the fatigue endurance of the steel parts [5].

Gas nitriding is a thermochemical surface treatment in which nitrogen is transferred from an ammonia atmosphere into the surface of steels at temperatures within the ferrite and carbide phase region [6, 7]. In order to improve the wear resistance, the steel or metal need to be nitrided so the outer layer of the metal hardened thus improve in their characteristics. With current technology and new founding, nitriding process is developed into several ways to achieve it. Gas nitriding process is among few nitriding process exists nowadays. The gas nitriding process is widely used in industry to improve the mechanical and thermal properties of the metals. Gas nitriding can be done at low temperature (500°C) or at high temperature (1100°C). All other parameters are the same except for the final temperature is different. Different temperature will yield different results of properties of the stainless steel 439 rod.

Stainless steels are stainless because of their Chromium content (minimum 10.5%). This result in resistance to wet corrosion and high temperature oxidation [8]. There are five types of stainless steel; ferritic, austenitic, martensitic, duplex and precipitation-hardening [9, 10]. Stainless

steel 439 has few characteristic properties like good resistance to uniform corrosion, excellent resistance to stress corrosion resistance (SCC), good weld ability, excellent machinability, good formability and good price range. Stainless steel 439 has quite a range of applications; automotive exhaust system components, catering and appliances, domestic water heater, elevator and indoor claddings, tubes, storage tank, and bus and wagon frames. The characteristics and applications can be varied once the stainless steel is heat treated or nitrided. This type of stainless steel is ferritic with a great finish quality and non-hardenable plain Chromium [11]. Apart from that, SS439 also has good chemical property that is resistance to nitric attack thus it is suitable to be used in chemical application.

### **1.1 Problem Statement**

Ferritic stainless steel ss439 is not widely or commonly used materials in industrial application. The problem arise is how to improve the characteristic of these metal? Whether use heat treatment or case hardening process and how to achieve the required objective? Thus far, not many literatures can be found investigating the microstructure, mechanical properties and thermal properties correlation of this metal. Hence yield to a question on how to proceed with the project. The SS439 need to be tested? Or only proceed with heat treatment or case hardening process? Will the result from this project will help expand the application of the nitrided ferritic ss439 stainless steel in the industry? Will this project give a good result or not?

### **1.2 Objective of the study**

Investigate the microstructure, mechanical properties and thermal properties of Base and Nitrided Ferritic SS439 Grade Stainless Steel Rod and correlate the microstructure profile and the mechanical properties and thermal properties.

### **1.3 Scope of study**

First six months of this study will cover the analysis of data as well as data collection from articles, journals, books, websites, research papers and thesis. The study will be specific on the microstructure profile, mechanical properties and thermal properties (thermal expansion if possible) of the base and nitrided ss439 rod as well as the testing method to examine their characteristics and properties. Next six months will be focused on lab work to test the characteristics and properties of the base and nitrided ss439 rod. Tensile test, fatigue test and hardness test will be done on the samples and last but not least, compile the data into a report.

## CHAPTER 2

### LITERATURE REVIEWS

Nitriding process is one of the heat treatment process which thermo chemically treat the surface of steel or metal in which nitrogen is transferred from a medium into the steel at high temperature completely within the ferrite and carbide region [6, 12, 13]. The nitriding process will create a white layer (compound layer) on the surface of the steel or metal that consists of ' $\epsilon$ - $\text{Fe}_2(\text{N,C})_{1-x}$ ' and ' $\gamma'$ - $\text{Fe}_4\text{N}$ ' phases [14]. Nitrogen from outside dissolves in the ferrite lattice at the nitriding temperature in the compound layer (known as diffusion zone or case). According to articles [6, 15, 16], alloy precipitates also formed in the diffusion zone. In addition, the compound layer results in improvement of the wear and corrosion resistance of the steel or metal. The compound layer also enhances the fatigue endurance of the metal as nitriding process minimizes distortion and deformation of the heat treated parts.

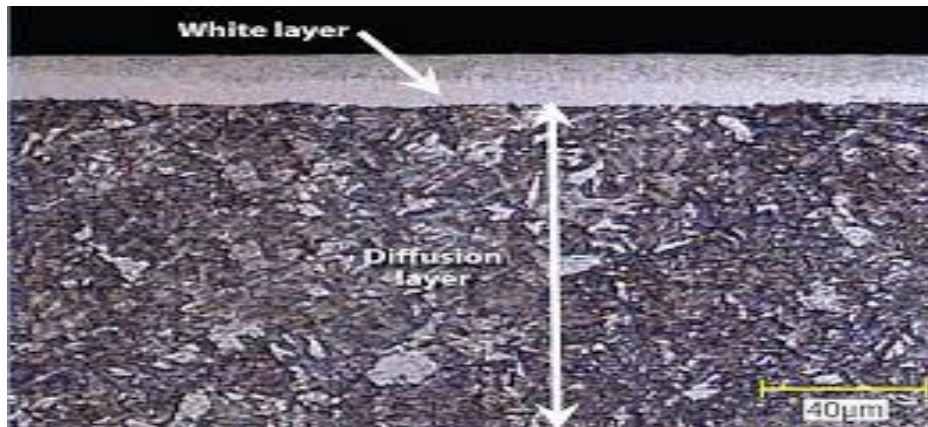


Figure 1 ; white layer and diffusion layer on the steel surface

Figure 1 shows the white layer and diffusion layer on the steel surface as discussed. Key controls parameters used to control the nitriding process will determine the output of the process. As a general of thumb [17];

- As the nitriding potential increase, the case depth and the white layer also increase.
- As the temperature increase, the case depth and white layer also increase.
- Increasing the residual ammonia and decreasing the dissociation measured will increase the case depth and the white layer.

At high temperature, iron lattice allow more space for nitrogen atoms to diffuse into the metal. Typical temperature for nitriding process is between 900 Celsius and 1100 Celsius. In gas nitriding process, nitrogen is exposed to the steel surface in a controlled atmosphere and in contact with ammonia ( $\text{NH}_3$ ) and the temperature is controlled. However, nitriding at high temperature (1100 Celsius) for a long period of time may induce serious deterioration of the substrate in many families of materials [18]. When nitriding at low temperature, components can be nitrided in the fully hardened and tempered condition without the core properties being affected. Another advantages of low temperature nitriding is the low risk of distortion and also consume less energy in the process.

As stated before, nitriding is a process which nitrogen is transferred from an ammonia ( $\text{NH}_3$ ) atmosphere into the steel at a temperature in the ferrite and carbide region [6, 12, 13]. In gas nitriding,  $\text{NH}_3$  is used as nitrogen-providing medium because it has high chemical potential of nitrogen.  $\text{NH}_3$  is transferred onto the solid-gas surface and dissociated into active nitrogen atoms and hydrogen gas by using metal surface as the catalyst. After that, nitrogen atoms diffuse into the metal and nitrides are formed.

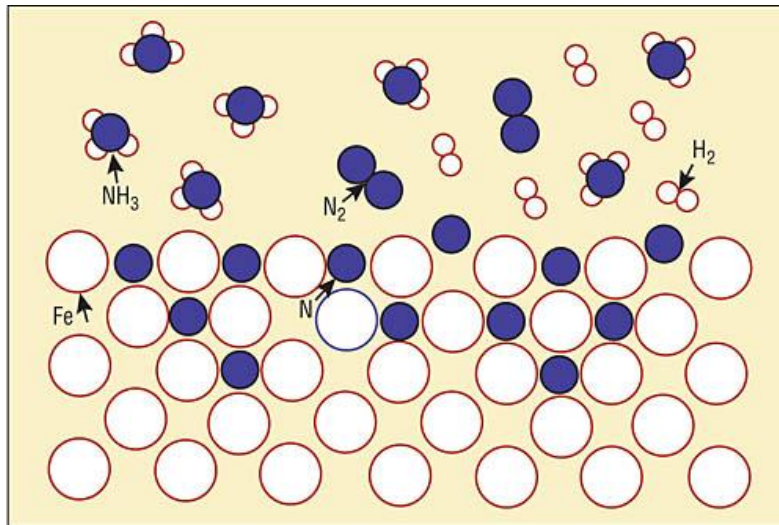


Figure 2; schematic illustration of gas nitriding

Figure 2 shows the schematic illustration of gas nitriding. Solubility of nitrogen controls the adsorption and diffusion process because the ferrite has much higher solubility for nitrogen than it does for carbon [2]. Somehow the limit of solubility of nitrogen in iron is temperature dependent. Anything beyond this will cause the surface phase formation on alloy steels to be

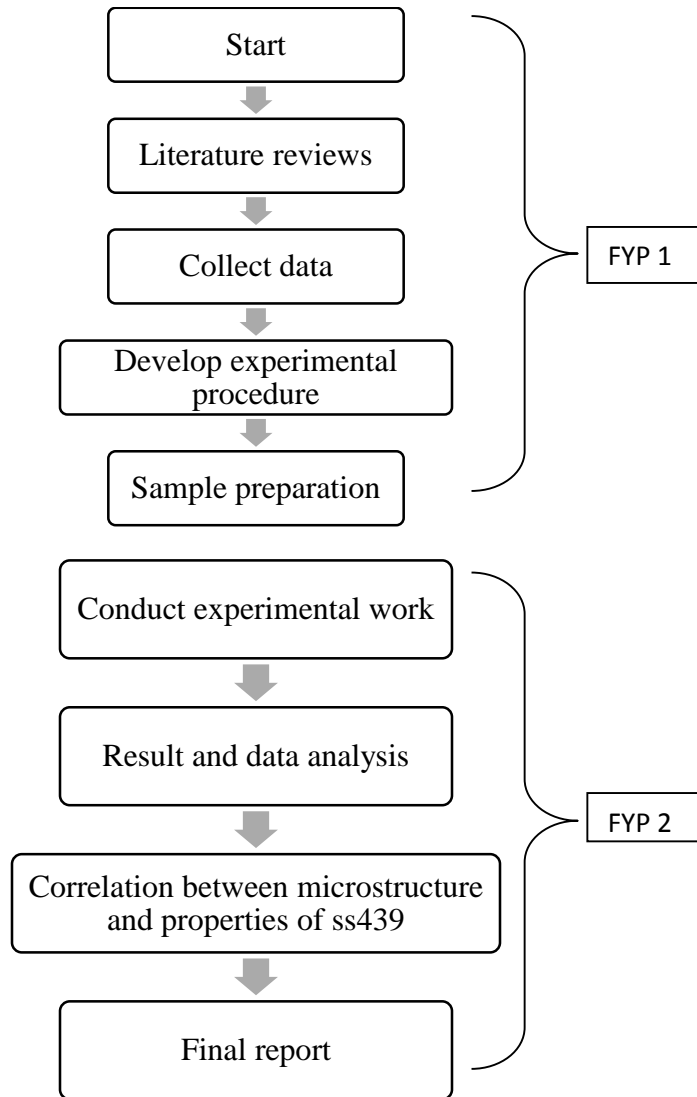
predominantly  $\epsilon$  phase. The higher the carbon content, the higher the potential for  $\epsilon$  to form. Strong nitride forming elements (e.g. aluminium and chromium) affect the hardening effect of the nitriding process.

Ferritic stainless steels are widely used due to its high corrosion resistance at room temperature and they are much cheaper than the other stainless steels. These ferritic stainless steels contain 16-30% Chromium (Cr) in their structures in respect of addition of the alloy element [19]. The ferritic stainless steels are easy to shape and can resist the atmospheric corrosion well. It is used widely in architecture, interior and exterior purposes, food industries and etc [20, 21]. Stainless steels ss439 grade is low-carbon plain chromium, ferritic stainless steel without any stabilization of carbon and nitrogen with titanium or niobium [22]. Due to the fact it has 16% Chromium make this SS439 a good resistance towards corrosive environments and also a good oxidation resistance at elevated temperature. It is ductile and can be formed using wide variety of roll forming as well as drawing and bending operations. It does not harden during cold working. Somehow SS439 not suitable for cryogenic applications as brittle fracture will occur at sub-zero temperatures due to its ferritic properties. This ferritic grade, contain only chromium and possible other elements like titanium (Ti) and it is well known as cost saving material since most of this ferritic materials have no expensive Ni additions. Stainless steel 439 can be formed into complex shape and can be joint using most conventional joining methods including welding. It is under group 3 which contain 16% -18% Cr with additional elements like Ti [18]. This 16%-18% Cr stabilized grade often has a fully ferritic microstructures at all temperatures.

## CHAPTER 3

### METHODOLOGY AND PROJECT ACTIVITIES

Basically the flow of this research is based on below flow chart;



Project activities;

In order to complete the project, many testing will be involved. Nitriding process is the main part of this study as the steel rod must be nitride to be compared with the base steel rod, thus nitriding machine will be used. A procedure to operate the machine will be developed according to the standard nitriding method. Few sample preparation machine and testing machines will be used; Mounting machine, abrasive cutter, optical microscope (stereoscopic),



hardness tester (Vickers or Rockwell), tensile testing machine and fatigue testing machine. To enhance the project flow, procedures are to be developed. Experiment and testing will be conducted with the help of the technicians.

Table 1; Types of sample and dimensions

<b>Type of sample</b>	<b>No. of sample</b>	<b>Diameter</b>	<b>Length</b>
Base	1	10mm	100mm
	5	10mm	60mm
	2	10mm	10mm
Low temperature Nitride	1	10mm	100mm
	5	10mm	60mm
	2	10mm	10mm
High temperature Nitride	1	10mm	100mm
	5	10mm	60mm
	2	10mm	10mm

### **3.0 Abrasive cutter; cutting sample**

- 1) Power on the machine. Make sure light is on and water flow on the cutter disk.
- 2) Open the casing and place the sample and lock the sample to required position.
- 3) Close the casing and switch on the blade. Cut the specimen into 10mm long.
- 4) Switch off the blade and open the casing. Unlock the sample and remove it.
- 5) Clean the sample and let it dry.
- 6) Repeat step 2-5 for other sample.
- 7) Switch of the machine and clean workplace.

### **3.1 Mounting sample**

- 1) Use sample with diameter of 10 mm and length of 10mm.
- 2) Switch on the mounting machine.
- 3) Clean the mould to remove any residue or leftover
- 4) Place the surface of the sample that is to be tested. Insert suitable amount powder into the sample casing and lock the casing.
- 5) Parameters of mounting;
  - Pressure – 4000psi
  - Heating time – 3 minutes
  - Cooling time – 2 minutes
- 6) Press start cycle button on the mounting machine.
- 7) Machine will ‘beep’ when it is finish. Take out the finished mount.
- 8) Repeat step 2-6 for other samples.
- 9) Switch off the mounting machine and clean the workplace.

### **3.2 Sample grinding and polishing**

- 1) Install #400 grit sand paper on the grinding machine. Allow water to flow on the sand paper during grinding.
- 2) Switch on the grinding machine and grind the mounted sample until it has mirror like finish.
- 3) Change the grit of sand paper to #800, #1200, #1500 and #2500 and repeat step 1-2 for other sample.
- 4) Spray liquid polisher on the polisher and on the surface of the sample.
- 5) Polish until get free scratch or little scratch on the sample surface.
- 6) Proceed to final polishing using semi-liquid diamond polisher.
- 7) Clean the sample surface and apply ethanol on the surface to remove water from the surface of the sample.
- 8) Repeat step 4-7 for other sample. Clean the workplace.

### **3.3 Etchant preparation; Kalling's No.2**

- 1) Switch on the fume hood. All experiment involving chemicals must be prepared in this fume.
- 2) Prepare a clean beaker, two 100ml test tube and a stirrer.
- 3) Weight 5 grams of Copper Sulphate ( $\text{CuCl}_2$ ), 100ml Hydrochloric acid (HCl) and 100ml Ethanol.
- 4) Pour all the weighted chemicals into the beaker and stir until it completely dissolve.
- 5) Solution is ready to be used.

### **3.4 Etching sample**

- 1) Use sample that has been polished. All steps are done in fume hood and use gloves for protection.
- 2) Apply ethanol on the sample surface and put it on the heater. Let it dry.
- 3) Apply Kalling's solution on the surface of the sample by swabbing using cotton. When the surface appear dull, stop swabbing and clean the surface using water.
- 4) Apply ethanol on the sample surface and put it on heater. Let it dry.
- 5) Repeat step 1-4 for other sample. The sample is ready for the next test.
- 6) Clean the workplace. Gloves and cotton are put in the special dustbin just for chemical waste.

### **3.5 Optical microscope (OM)**

- 1) Power on the optical microscope and the computer, make sure all the cables connect properly.
- 2) Place the etched specimen on the microscope.
- 3) By using 10x magnifying glass, focus the image and capture the image and save it on the computer.
- 4) Use 50x magnifying glass, focus the image and capture the image and save it on the computer.
- 5) Repeat step 4 to 6 for another specimen.
- 6) Power off the microscope and the computer.
- 7) Images taken will be analysed and studied.

### 3.6 Nitriding

- 1) Take samples to be nitrided.
- 2) Clean the sample using cotton or cloth.
- 3) Connect all power sources to the nitriding machine. Check the gas flow and make sure the machine work properly.
- 4) Parameters ;
  - Temperature – 500°C and 1100°C
  - Time – 10hours
  - Pressure – 50 to 100mmHg
- 5) Put the sample on a mold provided and put it inside the furnace.
- 6) Use single-staged nitriding process. Temperature is set to 500 Celsius for low temperature nitriding and 1100 Celsius for high temperature nitriding.
- 7) Close furnace and start flow of ammonia gas at fast flow rate.
- 8) Set furnace temperature control at 150°C simultaneously. Heat furnace to this temperature but do not exceed.
- 9) When the furnace has been purged to the degree that 10% or less air and 90% or more ammonia are present in the retort, the furnace may be heated to the nitriding temperature 500°C or 1100°C
- 10) Maintained this rate for 4 to 10 hours. It is important that the nitriding cycle begin with a dissociation rate of about 15 to 35%.
- 11) When finish, take out the sample using heat-resistant tool.
- 12) Set up the same parameters for other sample.

### 3.7 Rockwell hardness test

- 1) Clean the specimen and make sure the machine work properly. Parameters are set as follow:
  - Testing Machine ; AFFRI 206 RTD
  - Rockwell scale ; B
  - Load ; 100kgf

Temperature ; 22.4 Celsius  
 Indenter ; Ball 1/16 “  
 Type of ball ; Steel (S)

- 2) Put the specimen on right place and switch on the Rockwell hardness test machine.
- 3) Press the indenter into the sample by an accurately controlled test force.
- 4) Maintain the force for a specific dwell time, normally 10 – 15 seconds.
- 5) After the dwell time is complete, remove the indenter, leaving an indent in the sample.
- 6) The hardness of the sample surface will be automatically calculated by the machine.
- 7) Repeat step 1-6 at five different spot on the same sample. Calculate the average hardness

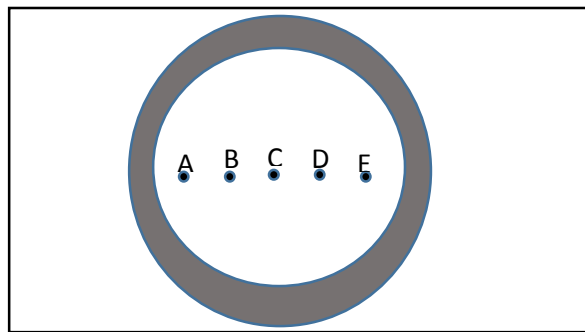


Figure 3 ; Rockwell hardness test

Table 2 ; Sample data of hardness test

Indentation point	Rockwell Hardness Value	Average Value
A		
B		
C		
D		
E		

### 3.8 Thermal expansion test

- 1) Take the first specimen.
- 2) Measure the initial length of the rod ( $L_o$ ).
- 3) Measure the initial temperature of the rod ( $T_o$ )
- 4) Heat the rod at high temperature up to 100 Celsius.
- 5) Measure the final length of the rod ( $L_f$ )
- 6) Measure the final temperature of the rod ( $T_f$ )
- 7) Record data in the table

Table 3 ; Sample data for thermal expansion

Sample	$L_o$ (m)	$L_f$ (m)	$\Delta L$ (m)	$T_o$ ( $^{\circ}\text{C}$ )	$T_f$ ( $^{\circ}\text{C}$ )	$\Delta T$ ( $^{\circ}\text{C}$ )
Base						
Low Temp.						
High Temp.						

- 8) Calculate the thermal expansion using the formula.

$$\frac{\Delta L}{L_o} = \alpha \Delta T$$

Where,

$\Delta L$  = change in length of sample ( $\text{m}^3$ )

$L_o$  = initial length of sample ( $\text{m}^3$ )

$\alpha$  = expansion coefficient ( $\text{m}/\text{m}^{\circ}\text{C}$ )

### 3.9 Three point bending test

- 1) Setup the bending test equipment
- 2) Put the sample on the bending test equipment
- 3) Set the dial gauge to zero
- 4) Put 200g load on the bending test equipment. Record the deflection shown by the dial gauge
- 5) Repeat step 2 – 4 using load of 400g, 600g, 800g and 1000g.
- 6) Repeat step 2-5 using different sample
- 7) Compute second moment of area,  $I_{x-x}$  for the samples using equation

$$I = \pi \frac{d^4}{64}$$

- 8) Record the deflection in a table

Table 4: Sample data for stiffness test

	Base	Low Temperature Nitrided	High Temperature Nitrided
200g			
400g			
600g			
800g			
1000g			

- 9) Calculate the stiffness of samples using equation

$$k = \frac{W}{\delta}$$

### KEY MILESTONES AND GANTT CHART

ACTIVITIES	WEEK																											
	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	29	
Meeting and discussion with supervisor																												
Project charter and draft preparation																												
Literature reviews from articles, journals, books, thesis, research papers and websites																												
Extended proposal submission*				x																								
Proposal defence presentation*							x																					
Develop experimental procedures																												
Interim report submission*													x															
samples preparation - nitriding and mounting samples																												
samples preparation - grinding and polishing																												
samples testing - optical microstructure																												
progress report submission*																					x							
samples testing - hardness,tensile,fatigue test																												
report compilation and writing																												
Pre-SEDEX presentation and poster*																								x				
draft dissertation submission*																									x			
dissertation and technical paper submission*																										x		
oral presentation*																											x	
hard bound dissertation submission*	x																											

x - key milestone



## CHAPTER 4

### RESULTS AND DISCUSSIONS

Samples for tensile test, hardness test and microstructure test have been nitrided both for low temperature nitriding (500 Celsius) and high temperature nitriding (1100 Celsius). The samples have been mounted for hardness test and for imaging its microstructure.



Figure 4 ; Types of samples



Figure 5 ; Mounted samples

Figure 4 shows the three different samples; the top one is the base SS439, the middle one is the low temperature nitrided SS439 and the bottom one is the high temperature nitride SS439.

Low temperature nitriding result in blue colour of the SS439. This is due to the low heat and also due to thin layer of the hydrogen coating that penetrate the SS349. The result shows that at low temperature, Nitrogen do not penetrate well into the surface of the SS439. High temperature nitriding give a thick hydrogen coating and a better penetration of hydrogen into the SS439. A darker colour is observed on the SS439. However the testing result is yet to be determine later.

Figure 5 shows the samples that are mounted using mounting machine. These samples will be used for hardness test and for microstructure imaging. This is the base SS439 samples and other samples have been mounted and not included in the picture as it will be shown in the appendix.

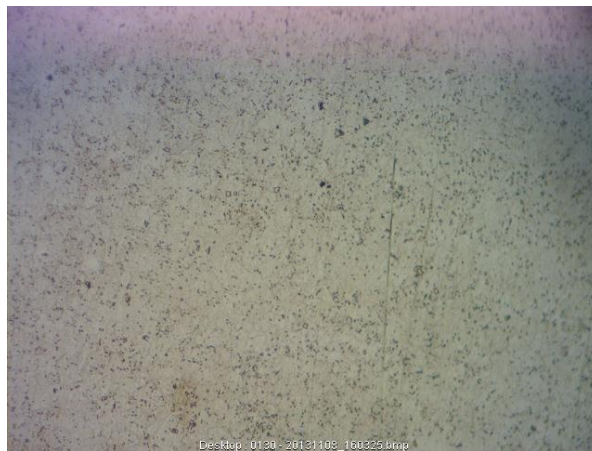


Figure 6; Microstructure of the Base SS439 at 100x magnification

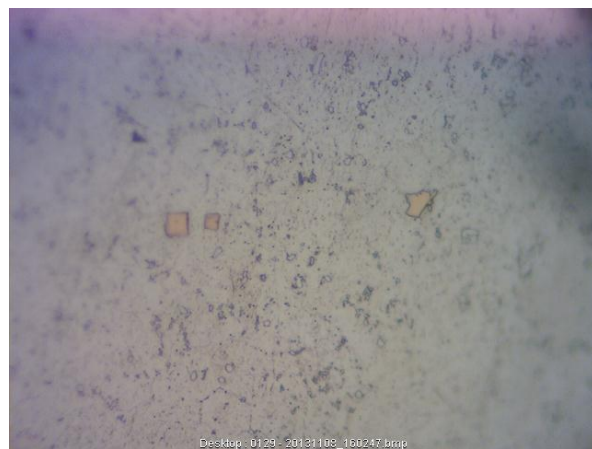


Figure 7; Microstructure of the Base SS439 at 500x magnification

Figure 6 and figure 7 show the microstructure of base SS439 at 100x and 500x magnification. The microstructure shows the present of the ferrite phase in the SS439. This result

is relevant as in the articles stated that most of the microstructure of SS439 consist of ferrite phase (J. Charles, 2008). Phase transformation do not occur because no heat treatment or case hardening is applied on the sample.

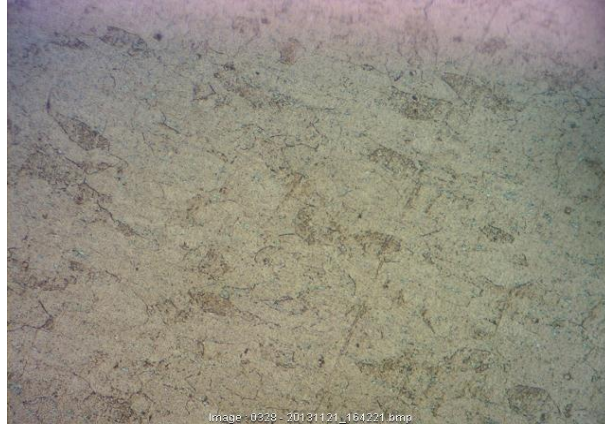


Figure 8; Microstructure of the Low Temperature nitrated SS439 at 100x magnification

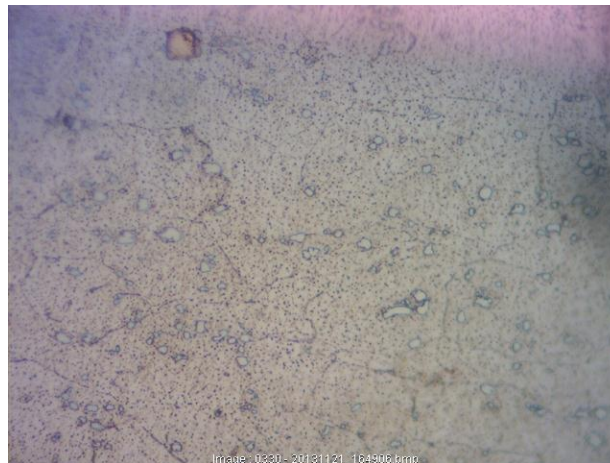


Figure 9; Microstructure of the Low Temperature nitrated SS439 at 500x magnification

Figure 8 and figure 9 show the microstructure of the low temperature nitrated SS439 at 100x and 500x magnification. The image is a bit darker than the base SS439 as the hydrogen has penetrated a bit the surface of the SS439. These microstructures show that hydrogen do not penetrate well into the SS439 at low temperature. The phase transformation from ferrite phase to martensite phase is not completed due to low penetration of hydrogen into the SS439. The hydrogen penetration only cause distortion to the SS439 grains as the phase transformation can be completed.

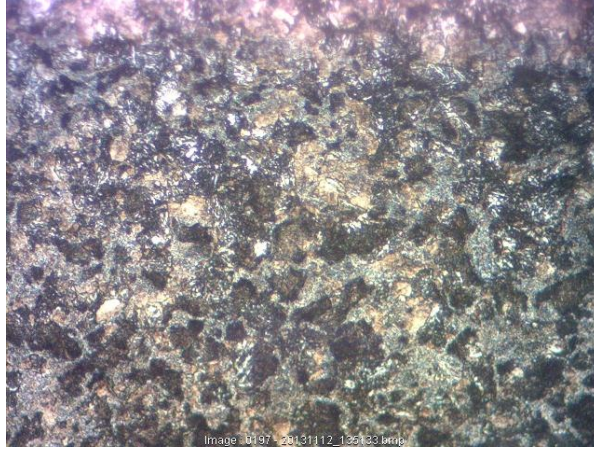


Figure 10; Microstructure of the high temperature nitrided SS439 at 100x magnification

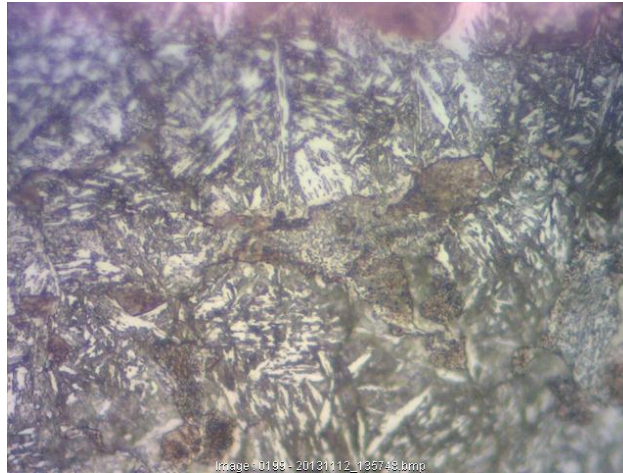


Figure 11; Microstructure of the high temperature nitrided SS439 at 500x magnification

Figure 10 and figure 11 show the microstructure of the high temperature nitrided SS439 at 100x and 500x magnification. The image is darker than the base and low temperature nitrided SS439 as the hydrogen has penetrated the surface of the SS439. These microstructures show the present of the ferrite phase as well as the martensite phase. Phase transformation from ferrite phase to martensite phase is completed. The needle look alike shape is the martensite phase (Dr. Patthi Hussain, 2013). This will result in increase of strength and harder SS439.

The mounted samples have been tested for its hardness using the Rockwell testing machine. The test is conducted with the help of a technician specific for the machine. All guidance and steps involved are based on laboratory standard. All samples have been tested using the same parameters.

Table 5; Rockwell hardness value of Base SS439

<b>Indentation Point</b>	<b>Rockwell Hardness Value</b>	<b>Average Value</b>
A	89.1	93.1
B	93.6	
C	95.1	
D	94.0	
E	93.7	

Table 6; Rockwell hardness value of Low Temperature Nitrided SS439

<b>Indentation Point</b>	<b>Rockwell Hardness Value</b>	<b>Average Value</b>
A	91.2	93.3
B	94.0	
C	94.4	
D	93.6	
E	93.3	

Table 7; Rockwell hardness value of High Temperature Nitrided SS439

<b>Indentation Point</b>	<b>Rockwell Hardness Value</b>	<b>Average Value</b>
A	108.9	109.7
B	109.4	
C	109.4	
D	110.3	
E	110.3	

Table 4, 5 and 6 show the Rockwell hardness value for Base SS439, Low Temperature Nitrided SS439 and High Temperature Nitrided SS439. The test results show different value of hardness for three different types of samples. . Low temperature nitrided SS439 has shown increase in hardness value by 0.2% and high temperature nitride SS439 has shown increase in hardness value by 18%. The result should yield increase in hardness from outer to the inner core of SS439. Low temperature nitride cause poor penetration of hydrogen, thus the hydrogen cannot penetrate deeper into the SS439. This yield decrease in hardness of the low temperature nitride SS439 as the test proceed into the inner core of the low temperature nitride SS439. At high temperature, hydrogen can penetrate into the inner core of the SS439 thus yield in increase of hardness from outer to inner core. To illustrate the differences of hardness value between the three types of samples, a graph is drawn.

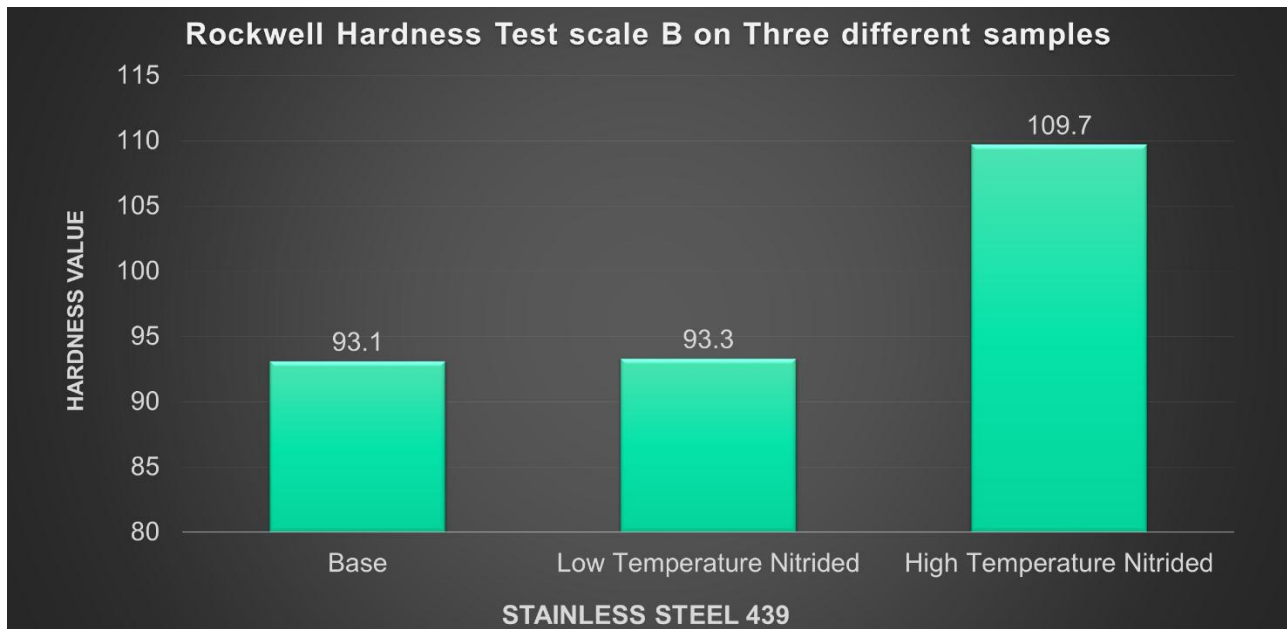


Figure 12; Graph of Rockwell hardness value of three different samples

Figure 12 shows the graph of Rockwell hardness test scale B on three different samples. Graph shows that low temperature nitride SS439 only increase its hardness by 0.02. This result is due to the poor penetration of hydrogen into the SS439. High temperature nitride SS439 shows massive increase in hardness by 16.6. This shown that nitriding the samples at high temperature yield a great increase in hardness value. Hydrogen successfully penetrate into the inner core of the SS439.

Linear thermal expansion has been calculated for all three different samples; keeping the test at constant room temperature or initial temperature of 27 Celsius and final temperature of 100 Celsius for 1 hour.

Table 8; Result of the linear thermal expansion test

<b>Sample</b>	$L_o$ (m)	$L_f$ (m)	$\Delta L$ (m)	$T_o$ (°C)	$T_f$ (°C)	$\Delta T$ (°C)	$\alpha$
Base	0.1	0.113	0.013	27	100	73	$1.78 \times 10^{-3}$
Low Temp. Nitrided	0.1	0.110	0.010	27	100	73	$1.37 \times 10^{-3}$
High Temp. Nitrided	0.1	0.103	0.003	27	100	73	$4.11 \times 10^{-4}$

Table 8 shows the results from the linear thermal expansion test. Low temperature nitrided SS439 shows not much different from the Base SS439. The difference is only by  $0.41 \times 10^{-3}$ . High temperature nitrided shows a huge difference from the Base SS439 by  $1.369 \times 10^{-3}$ . This shows that high temperature nitride SS439 has a good thermal resistance compared to Base SS439 and low temperature nitride SS439. High temperature nitrided SS439 shows a lower value of linear thermal coefficient. It shows that temperature do not greatly affect the dimension of the high temperature nitrided SS439. Thermal expansion must be taken into account when designing product with close tolerance fits as these tolerance will change as temperature change. It should also be understood that thermal expansion can cause significant stress in a component.

Table 9; Deflection of Three different samples using bending test equipment

	Base	Low Temperature Nitrided	High Temperature Nitrided
200g	0.010mm	0.008mm	0.001mm
400g	0.013mm	0.011mm	0.002mm
600g	0.017mm	0.012mm	0.005mm
800g	0.019mm	0.015mm	0.007mm
1000g	0.022mm	0.016mm	0.009mm

Table 9 shows the result from bending test for Base SS439, Low temperature nitrided SS439, and High temperature SS439. Base SS439 and Low temperature nitrided SS439 has small difference in deflection. This is because hydrogen do not penetrate well into the SS439 at low temperature thus result in low hardness of sample. Correlate with the microstructure of the Low temperature nitrided SS439, the ferrite phase do not fully transform into martensite phase thus giving the sample low hardness. Huge difference in deflection can be seen when comparing High temperature nitrided SS439 and Base SS439. Hydrogen penetrate well into the sample and yield a high hardness sample. The microstructure shown that ferrite phase has fully transform into martensite phase which cause the hardness of SS439 to increase.

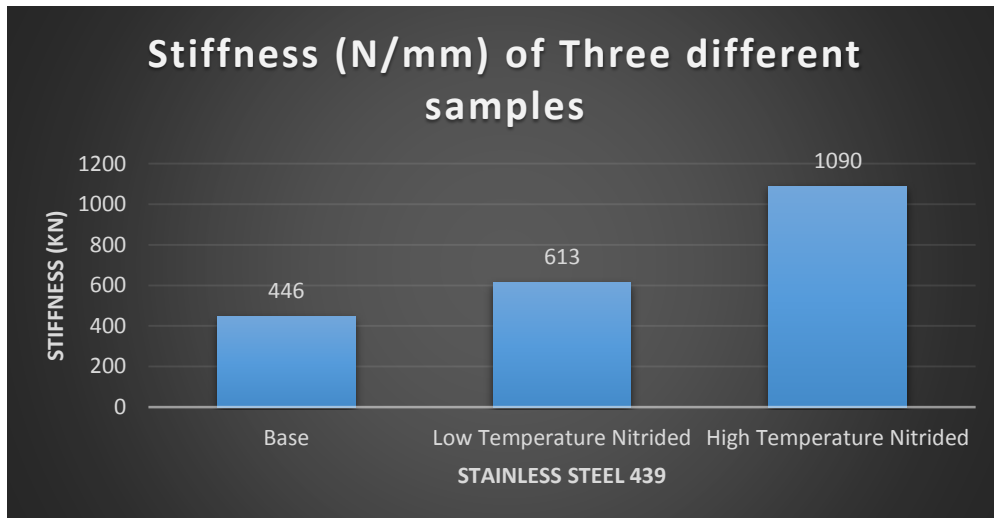


Figure 13; Graph of stiffness for three different samples



Figure 13 shows the stiffness of Base SS439, Low temperature nitrided SS439 and High temperature nitrided SS439. Low temperature nitrided SS439 increase its stiffness value by 27% which is not a huge increase. Somehow, when SS439 is nitrided at high temperature, its stiffness value increase by 59% which is a huge increase in value. It shows that, nitriding SS439 at high temperature allow the hydrogen to penetrate into the core of the SS439, thus increase the stiffness value. High stiffness value is required where the design can withstand or resist deformation in response to applied force. Suitable for its application in the kitchen where cyclic force exist, thus this high value of stiffness can prolong the life of the material. The application itself required a high stiffness value where a rigid and stiff material is needed and desired no deflection.

## CHAPTER 5

### 5.1 Conclusion

From the research and test results, this project can be concluded that different temperature result in different thickness of hydrogen coating on the SS439 and yield different results in every test. Based from observation, high temperature nitriding give thicker coating and darker colour than the low temperature nitriding with thin coating and brighter colour. The microstructure of the base SS439 shows ferrite phase is the most phase present. Low temperature nitrided SS439 shows ferrite phase fail to fully transform into martensite phase. High temperature nitrided SS439 microstructure shows ferrite phase has successfully transform into martensite phase. Low temperature nitrided SS439 shows an increase in hardness value by 0.2%. The linear thermal coefficient of the low temperature nitrided is  $1.37 \times 10^{-3}$ . Nitriding at high temperature results in increase of hardness by 18% as well as low thermal coefficient,  $\alpha = 4.11 \times 10^{-4}$ . Low temperature nitrided SS439 has increase its stiffness by 27% and high temperature nitrided SS439 has increase its stiffness by 59%. The objective of this experiment that is to investigate the properties of the SS439 has been achieved. Nitriding at high temperature yield a greater results compared to the Base SS439 and nitriding at low temperature. Therefore this research can be concluded that high temperature nitrided SS439 is way better than Base SS439 and low temperature nitrided SS439.

### 5.2 Recommendations

There are a few other aspects of this research that can be approached in order to improve the findings of this research. The suggested aspects are as follow:

- a) Nitride samples with different time period to investigate the effect of nitriding with time
- b) Vary the temperature to find the suitable temperature for the hydrogen to 100% penetrate into the SS439
- c) Test the samples with fatigue testing machine to investigate the reliability of the treated samples
- d) Investigate wear resistance and corrosion resistance for the treated samples
- e) Build a real prototype for the Base SS439 and Nitrided SS439 to compare the characteristics of the material in real situation ; e.g. automotive exhaust

## REFERENCES

- [1] Craig Fitzgerald. 2006, "Home Nitriding, Feature Article from Hemmings Sports & Exotic Car" in Hemming sports and Exotic Car Magazines.
- [2] Xiaolan Wang A. 2011, Activated atmosphere case hardening of steels by Dissertation, Degree of Doctor of Philosophy in Material Science and Engineering, Faculty of the WORCESTER POLYTECHNIC INSTITUTE.
- [3] S. Lampman, "Introduction to Surface Hardening of Steels," vol. 4, pp. 259-267, 1991.
- [4] H. Roelofs, S. Hasler. 2008, "Multiphase Structures in Case Hardening Steels following Continuous Cooling," University of Cambridge, U. K.
- [5] Badger Metal Tech. Inc Company. 2011, "History of Nitriding," USA.
- [6] Pye D. 2003, "Practical nitriding and ferritic nitrocarburizing," Materials Park, OH: ASM International.
- [7] Somers MAJ, "Thermodynamics, kinetics and microstructural evolution of the compound layer; a comparison of the states of knowledge of nitriding and nitrocarburizing," Heat Treatment of Metals, pp. 27-92, 2000.
- [8] J. Charles, J.D. Mithieux, P.O. Santacreu, L. Peguet, 2008, "The Ferritic Stainless Steel Family: The Appropriate Answer To Nickel Volatility," ArcelorMittal R&D, France.
- [9] Taskin, M., Ozan, S. and Kolukisa, S, "The Effect of the Process Temperature on the Bondability in Diffusion Bonding of AISI 430 with Nodular Cast Iron", Practical Metallography, vol. 43, (6), pp. 293-305, 2006.
- [10] Ozan, S., Taskin, M. and Kolukisa, S, "The Effect of the Process Temperature on the Bondability in Transient Liquid Phase (TLP) Diffusion Bonding of AISI 430 Ferritic Stainless Steel with Nodular(Spheroid) Cast Iron Using A Copper Interlayer", Practical Metallography, vol. 43, (11), pp. 575-585, 2006.
- [11] Stainless Steel Technical Information and Datasheet. 2013, "Stainless steel technical information," AALCO Multi-metals Company, U.K.
- [12] M. A. J. Somers, "Heat Treatment of Metals," vol. 27, pp. 92-102. 2000.
- [13] D. H. Herring, "Heat Treating Progress," pp. 17, 2002.
- [14] H. Du, M. A. J. Somers, J. Agren, "Metallurgical and Materials Transactions a Physical Metallurgy and Materials Science," pp. 195-211, 2000.

- [15] T. Bell, 1997, "Gaseous and Plasma Nitrocarburizing," Materials Park, Ohio.
- [16] T. Bell, Y. Sun, A. Suhadi, "Vacuum," vol. 59, pp. 14-23, 2000.
- [17] Karl-Michael Winter, 2009, "Gaseous Nitriding: In theory and In Real Life Technical Paper," United Process Control, Germany.
- [18] T. Bell, "Source Book on Nitriding: American Society for Metals," Metals Park, OH, 1977.
- [19] Ugur Caligului, Halil Dikbas, Mustafa Taskini, 2011, "Microstructural Characteristic of Dissimilar Welded Components (AISI 430 Ferritic-AISI 304 Austenitic Stainless Steels) by CO<sub>2</sub> Laser Beam Welding (LBW)," Firat University, Faculty of Technology, Dep. of Metallurgy and Materials Engineering, TURKEY.
- [20] Moustafa, I. M., Moustafa, M. A. and Nofal, A.A., "Carbide Formation Mechanism during Solidification and Annealing of 17% Cr-Ferritic Steel," Materials Letters, vol. 42, (6), pp. 371-379, 2000.
- [21] Taskin, M. and Caligulu, U., "The Effect on the Joining of Welding Power on the Laser Welding of Couple Steels AISI 430/1010," Firat Univ. Journal of Engineering, vol. 21, (1), pp. 11-22, 2009.
- [22] North American Stainless Company. 2011, "Flat products stainless steel grade sheet," USA.

**APPENDIX I**  
**MACHINES AND TOOLS USED**  
**MECHANICAL LABORATORY**



Figure 14; Mounting machine



Figure 15; Nitriding furnace



Figure 16; Heating / Drying machine

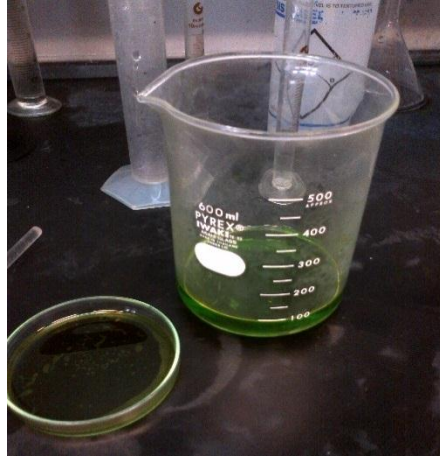


Figure 17; Kalling's solution



Figure 18; Fume hood



Figure 19; Grinding and polishing machine



Figure 20; Abrasive cutter machine

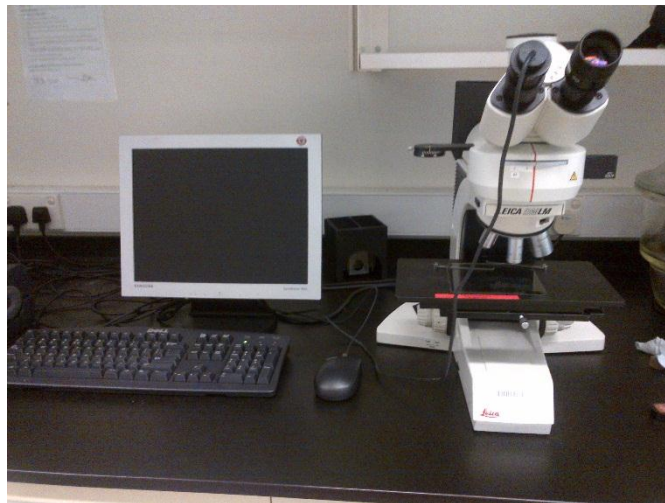


Figure 21; Optical microscope



**APPENDIX II**  
**LINEAR THERMAL EXPANSION**  
**CALCULATIONS**

$$\frac{\Delta L}{L_0} = \alpha \Delta T$$

Base SS439;

$$\frac{0.013m}{0.1m} = \alpha(73)$$

$$\alpha = 1.78 \times 10^{-3}$$

Low temperature nitride SS439;

$$\frac{0.010m}{0.1m} = \alpha(73)$$

$$\alpha = 1.37 \times 10^{-3}$$

High temperature nitride SS439;

$$\frac{0.003m}{0.1m} = \alpha(73)$$

$$\alpha = 4.11 \times 10^{-4}$$

**APPENDIX III**  
**STIFFNESS VALUE**  
**CALCULATIONS**

$$k = \frac{W}{\delta}$$

Base SS439;

$$k = \frac{1000g (9.81)}{0.022mm}$$

$$k = 446 \text{ N/mm}$$

Low temperature nitrided SS439;

$$k = \frac{1000g (9.81)}{0.016mm}$$

$$k = 613 \text{ N/mm}$$

High temperature nitrided SS439;

$$k = \frac{1000g (9.81)}{0.009mm}$$

$$k = 1090 \text{ N/m}$$

