THE EFFECT OF NITRIDING A MARTENSITIC STAINLESS STEEL WITH RESPECT TO TEMPERATURE VARIATION

BY

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CERTIFICATION OF APPROVAL

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Approved by,

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TRONOH, PERAK

AUGUST 2011

CERTIFICATION OF ORIGINALITY

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

MOHD ZULFADZLI BIN ZAHARI

ABSTRACT

This project is a study on solution nitriding of martensitic stainless steel. It consists of project's background, literature reviews, methodology, and all the process and procedures related to the project. Nitriding is one of the heat treatment method used for strengthening the material by diffusing the nitrogen into the surface of martensitic stainless steel. The objective of this project is to study the effect of nitriding of AISI 410 martensitic stainless steel at different temperature which are 950°C, 1000°C and 1050°C under nitriding time of 6 hours. Next is to evaluate the changes in the microstructure surface and also to evaluate the hardness of the nitrided martensitic stainless steel. In order to achieve the objectives, the metallographic study, and hardness test was performed. Field Emission Scanning Electron Microscope (FESEM), Optical microscope (OM), Vickers Microhardness tester has been used to study the nitride surface. Results from the hardness test shows an increment in the hardness value of the nitride samples from 254HV (as received) to 385.1HV (nitrided at 1050°C). Based on that, nitrogen diffuses into the surface better at high temperature. Hence, it strengthens and also improves the hardness of the martensitic stainless steel.

ACKNOWLEDGEMENT

First and foremost, I would like to express my praises to ALLAH for His blessing.

My deepest appreciation and gratitude is extended to my supervisor, AP Dr. Patthi Hussain for all his teachings, guidance, supervision and supports from the preliminary to the final report enable me to develop an understanding of the subject. It has been a hardship for you, sorry and thank you so much.

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CHAPTER 1

INTRODUCTION

1.1 Background of Project

Stainless steel is a common name for metal alloys that consist of 10.5% or more Chromium (Cr) and more than 50% Iron (Fe). Generally, the chromium content in stainless steel prevents corrosion take place. The chromium works by reacting with oxygen to form a tough, adherent, invisible, passive layer of chromium oxide film on the steel surface. If damaged mechanically or chemically, these films will self healing as long as it has enough oxygen.

There is thought that proper heat treatment can improve the stainless steel surface properties and it is proven since gas nitriding has been performed as early as in 1958, particularly for these kinds of alloys. Gas nitriding can be divided into low temperature and high temperature gas nitriding where both of them is performed by heating the material under nitrogenous atmosphere with respect to the temperature requirements. During the treatment, nitrogen will diffuse into stainless steel and increase the material hardness.

1.2 Problem Statement

Martensitic stainless steel may have the highest strength as compared to the other three types of stainless steel. However due to its brittleness, there is limitation in its application. Hence, any equipment that is made of martensitic stainless steel won't last for a long period of time. Therefore, this type of steel need to be improved especially in terms of its hardness and toughness. Nitriding procedures are chosen in order to overcome the problem. During the procedure take place, the sample is being heated at high temperature and slow cool will be done.

1.3 Objective

- To study the effect of nitriding of AISI 410 martensitic stainless steel at different temperature which are 950°C, 1000°C and 1050°C. The nitriding time is 8 hours.
- To analyze the changes in microstructure of the martensitic stainless steel samples.
- To evaluate the materials hardness (before and after the nitriding process) of the martensitic stainless.

1.4 Scope of study

The project of determining the effect of nitriding a martensitic stainless steel will be focusing on the microstructure area. Nitriding is one of the heat treatment processes where the sample is heated at high temperature under the nitrogenous atmosphere. Nitrogen will diffuse into the stainless steel and it will improve or increase its hardness. All evaluation will be made on the samples cross section area. Four samples will be prepared for 4 different nitriding temperatures and each of the samples will be cut in cross-sectional for microstrucutre analysis and also hardness testing.

In order to evaluate and analyze the microstructure of the samples which has been heat treated at different temperature, Optical Microscope (OM). Field Emission Scanning Electron Microscope (FESEM), and also the Energy Dispersive X-ray Spectroscopy (EDX) are used. Each of them is used for elemental identification across the sample cross section and also to evaluate the nitrogen composition across the sample.

CHAPTER 2

LITERATURE REVIEW

2.1 Overview of Martensitic Stainless Steel

Martensitic stainless steels are alloys of chromium and carbon that possess a martensitic crystal structure in the hardened condition. They are ferromagnetic, hardenable by heat treatments, more brittle and are less resistant to corrosion than some other stainless steel. Chromium content usually does not exceed 18%, while carbon content may exceed 1.0%. The chromium and carbon contents are adjusted to ensure a martensitic structure after hardening. Excess carbides may be present to enhance wear resistance. Martensitic stainless steels are capable of being heat treated in such way that martensite in the prime micro constituent [1]. Additions of alloying elements in significant concentrations produce remarkable alterations in the iron-iron carbide phase diagram.

Based on study of martensitic stainless steels, they are analogous to low alloy or carbon steels. Martensitic stainless steels also have a similar structure to the ferritic steels. Due to the adding up of carbon, these types of stainless steels can be hardened and strengthened by heat treatment. Generally, the main alloying element is chromium, typically in the range of 12 to 15%, molybdenum (0.2-1%), no nickel, except for two grades, and 0.1-1.2% carbon.

Their structures are "body-centered tetragonal" (bct) [2] and they are classed as a "hard" ferro-magnetic group. In the annealed condition, they have tensile yield strengths of about 275 MPa and so they are usually machined, cold formed, or cold worked in this condition. The strength obtained by heat treatment depends on the carbon content of the alloy. Increasing the carbon content increases the strength and hardness potential but decreases ductility and toughness. The higher carbon grades are capable of being heat treated to hardnesses of 60 HRC.

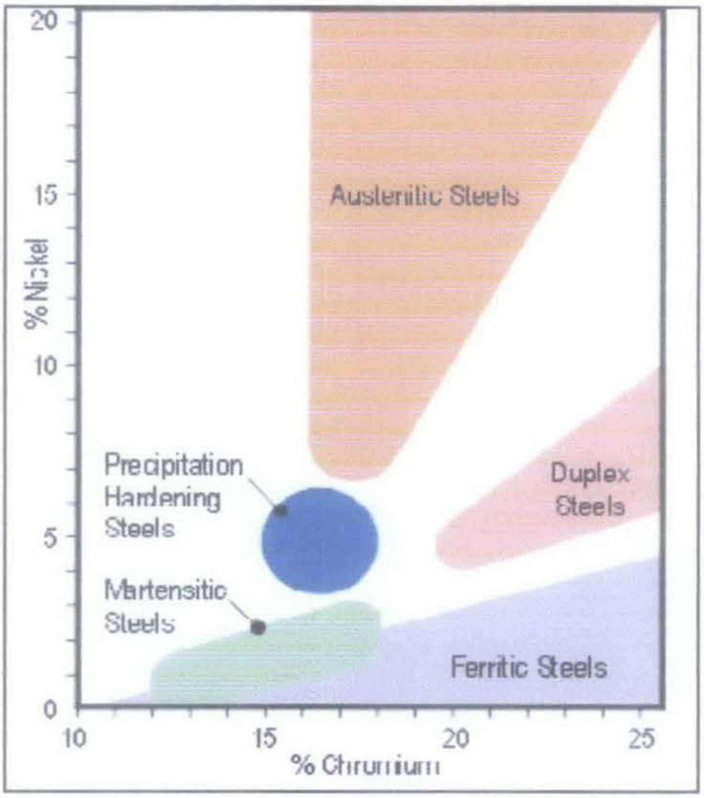

Figure 2.1: Families of stainless steel [1]

2.2 Grades of Martensitic Stainless Steel

Martensitic grades of stainless steel were developed in order to provide a group of stainless alloys that would be corrosion resistant and hardenable by heat treating. The martensitic grades are mainly used where hardness, strength and wear resistance are required. Below are the examples of martensitic grades that are available in the market:

- **Type 410**

  Basic martensitic grade, containing the lowest alloy content of the three basic stainless steels (304,430 and 410). Low cost and heat treatable stainless steel. Used widely where corrosion environment is not severe (air, water, some chemicals and food acids). Typical applications include highly stressed parts needing the combination of strength and corrosion resistance such as fasteners.

- **Type 420**

  Contains increased carbon to improve mechanical properties. Typical applications include surgical instruments.

- **Type 431**

  Contains increased chromium for greater corrosion resistance and good mechanical properties. Typical applications include high strength parts such as valves and pumps.

- **Type 440**

  Further increased in chromium and carbon to improve its toughness and corrosion resistance. Typical applications include instruments.

2.3 Martensitic Stainless Steel Properties

Out of 4 types of the available grades, there are 2 types of grades that are considered as the most common types. They are AISI 410 and AISI420. Below are the mechanical properties of those two types of stainless steel and its composition:

2.3.1 AISI 410

Typical compositions for this martensitic stainless steel grade are given in Table 2.1.

Table 2.1: Composition range for AISI 410 grade

Grade		C	Mn	Si	P	S	Cr	Mo	Ni	N
	Min.	-	-	-	-	-	11.5			
410	Max.	0.15	1.00	1.00	0.040	0.030	13.5	-	0.75	-

Typical mechanical properties for this martensitic stainless steel grade are given in Table 2.2.

Table 2.2: Mechanical properties of AISI 410 grade

Tempering Temperature (°C)	Tensile Strength (Mpa)	Yield Strength 0.2% Proof (MPa)	Elongation (%in 50mm)	Hardness Brinell (HB)	Impact Charpy V (J)
Annealed *	480 min	275 min	16 min	-	-
204	1310	1000	16	388	30
316	1240	960	14	325	36
427	1405	950	16	401	#
538	985	730	16	321	#
593	870	675	20	255	39
650	755	575	23	225	80

Note:

* Annealed properties are specified for Condition A of ASTM A276, for cold finished bar.

Due to associated low impact resistance this steel should not be tempered in the range 425-600°C

- **Corrosion Resistance**

410 resist dry atmosphere, fresh water, mild alkalies and acids, food, steam and hot gases. This stainless steel grade must be hardened for maximum heat and corrosion resistance and its performance is best with good or smooth surface finish. However, this grade is less corrosion resistance as compared to austenitic grades and also grade 430.

- **Heat Resistance**

Good resistance to scaling up to approximately 650°C, but generally not recommended for use in temperatures between 400 and 580°C. This is because of the reduction in mechanical properties for this particular stainless steel.

- **Machining**

In the annealed or highly tempered conditions grade 410 is relatively easily machined, but if hardened to above 30HRC machining becomes more difficult.

2.3.2 AISI 420

Typical compositions for this martensitic stainless steel grade are given in Table 2.3.

Table 2.3: Composition range for AISI 420 grade

Grade		C	Mn	Si	P	S	Cr	Mo	Ni	N
420	Min.	0.15	-	-	-	-	12.0			
	Max.	-	1.00	1.00	0.040	0.030	14.0	-	-	-

Typical mechanical properties for this martensitic stainless steel grade are given in Table 2.4.

Table 2.4: Mechanical properties of AISI 420 grade

Tempering Temperature (°C)	Tensile Strength (MPa)	Yield Strength 0.2% Proof (MPa)	Elongation (% in 50mm)	Hardness Brinell (HB)	Impact Charpy V (J)
Annealed *	655	345	25	241 max	-
204	1600	1360	12	444	20
316	1580	1365	14	444	19
427	1620	1420	10	461	#
538	1305	1095	15	375	#
593	1035	810	18	302	22
650	895	680	20	262	42

Note:

* Annealed tensile properties are typical for Condition A of ASTM A276; annealed hardness is the specified maximum.

Due to associated low impact resistance this steel should not be tempered in the range 425-600°C

- **Corrosion Resistance**

420 grades have good resistance in the hardened condition especially to the atmosphere, foods, fresh water and mild alkalies or acids. However, its corrosion resistance is slightly lower in the annealed condition. As for this grades, its performance also best with smooth surface finish and it also less corrosion resistant as compared to the austenitic grades. Moreover, grade 420 corrosion resistances also slightly lower than grade 410. However, this grade is sufficient enough to resist attack from food and normal washing methods, but prolonged contact with unwashed food residues can result in pitting.

- **Heat Resistance**

This grade of stainless steel is not recommended for any use in any temperatures above the relevant tempering temperature. This is due to the reduction in its mechanical properties. The scaling temperature is approximately 650^{o}C.

- **Machining**

In the annealed condition this grade is relatively easily machined, but if hardened to above 30HRC machining becomes more difficult.

2.4 Applications of Martensite Stainless Steel

In United States, low- and medium–carbon martensitic steels (type 410) have been used primarily in steam turbines, jet engines and gas turbines. While for type 420 and similar alloys are used in cutlery, valve parts, gears, shafts, and rollers. Martensitic stainless steel is also used in petroleum and petrochemical equipments. Other applications especially for higher-carbon level include cutlery, surgical and dental instruments, scissors, springs, cams and ball bearings.

2.5 Nitriding

Nitriding is one of the heat treatment methods that being used to strengthening the surface of the materials. During the nitriding, the nitrogen is being introduced and it will diffuse into the surface of the materials; martensitic stainless steel. There are three types of nitriding process methods that being applied nowadays. The first one is the gas nitriding where it used a horizontal tube furnace or fluidized bed. The second one is the liquid type or in other word is salt bath. The last one is using plasma nitriding where it used ion nitriding. Below are the common reasons for conducting the nitriding process:

- To obtain high surface hardness
- To increase wear resistance
- To improve fatigue life
- To improve corrosion resistance (except for stainless steels)
- To obtain a surface that is resistant to the softening effect of heat at temperatures up to the nitriding temperature

2.5.1 Gas Nitriding

Gas nitriding is one of the three available types of nitriding process and it is a case hardening process where nitrogen is introduced into the surface of the solid ferrous alloy by treating the sample at a suitable temperature. Under gas nitrding procedures, there are two common parameters in performing it. The first one is the low temperature gas nitriding while the others are the high temperature gas nitriding. In low temperature gas nitriding, the gas used is usually Ammonia (NH_3) where it will be disassociates into nitrogen and hydrogen when it comes into contact with the heated sample. While for the high temperature gas nitriding, it directly used nitrogen gas. The main different in result between both types of nitriding is that low temperature gas nitriding will produce a white layer at the outermost of the sample microstructure [3]. This white layer is normally called as the nitrided layer. While for the high temperature gas nitriding, there will be no white layer observed since the nitrogen will remain in solid solution [3].

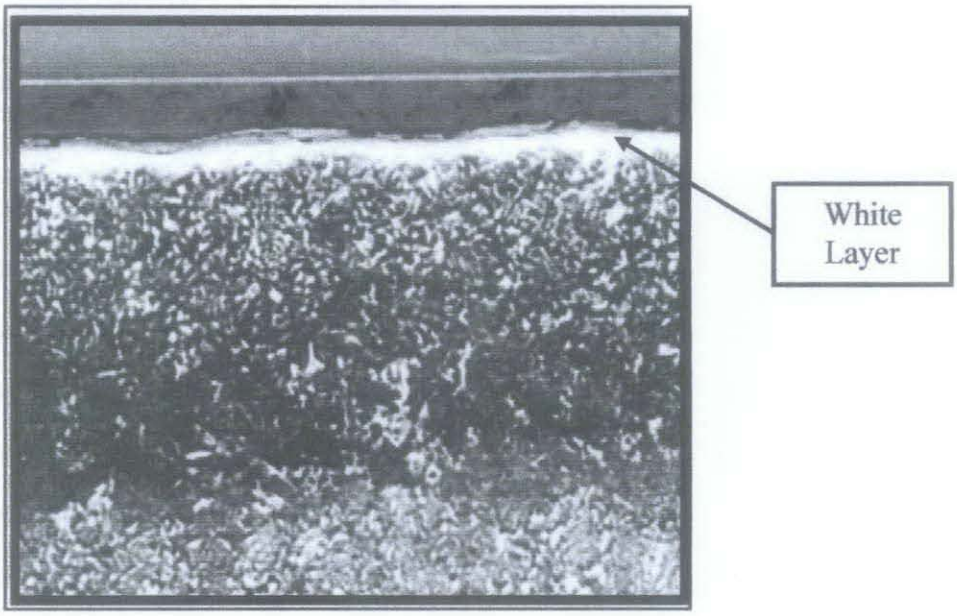

Figure 2.2: White layer [3]

2.6 Metallographic Technique

Metallographic technique is a systematic method to examine the microstructure of materials (mainly metallic materials). However, metallography does not apply to metallic materials only. Such technology can also be used to examine ceramics, polymers and semiconductors [4]. Below are the related processes in the metallography technique:

- Sectioning
- Mounting
- Grinding
- Polishing
- Etching

2.7 Previous Study

2.7.1 Modified AISI 420 using Plasma Nitriding

According to research that being made by I. Alphona, nitriding the AISI 420 martensitic stainless steel has successfully enhance the surface hardness of the material. The existence of nitrogen concentration contributed the hardness of the stainless steel. From the cross-sectional SEM micrograph for this research, it shows a case depth of 61 microns and the microstructure over the case depth is different from that within first 2-5 microns from the surface. [5]

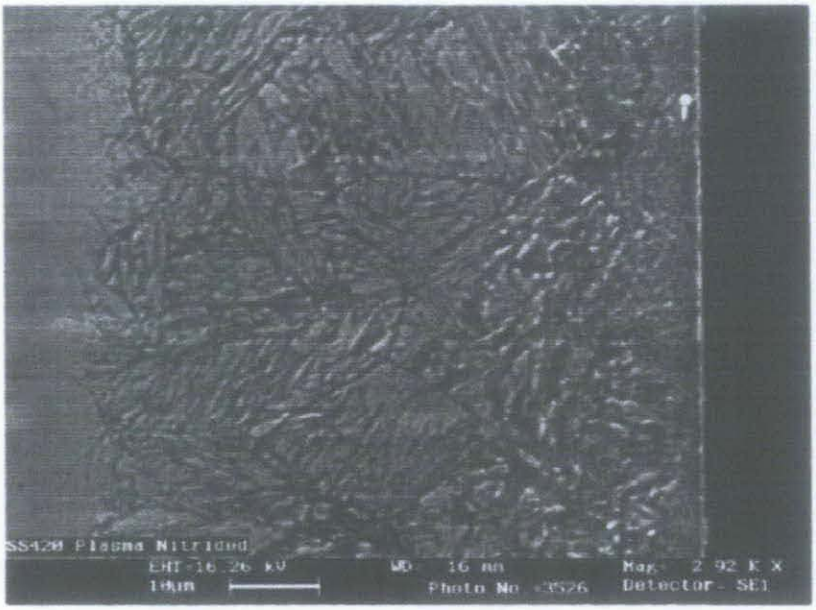

Figure 2.3: SEM picture of plasma- nitride AISI 420 [5]

A direct relation between the surface microhardness in plasma nitride AISI 420 with the nitride phase formation in the measured case depth is found. The maximum microhardness of the sample is in the range of 61 microns depth. However, as we go further the depth of the sample, its nitrogen concentration reduces very sharply, the reduce nitrogen content results in reduction of the surface hardness.

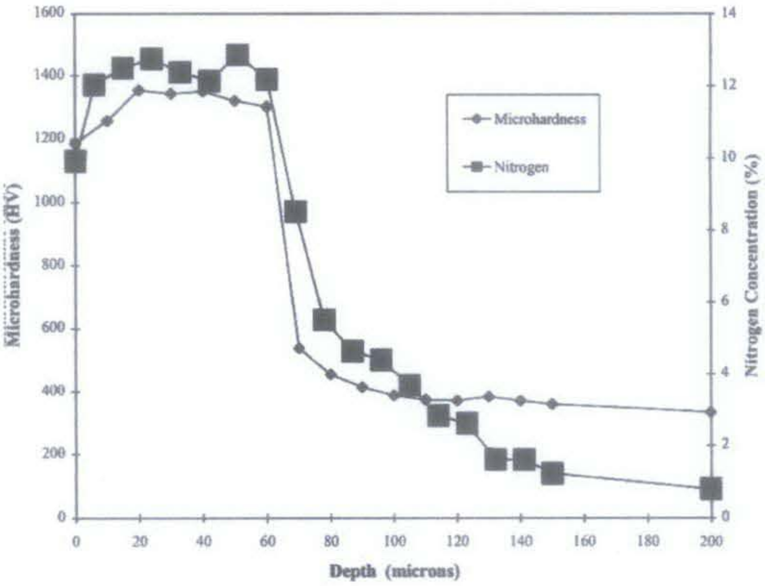

Figure 2.4: Microhardness and nitrogen concentration profile across the cross-section of plasma nitride AISI 420 [5]

Plasma nitriding was carried out on a square blocks having dimension 1 x 1 cm^2. Plasma nitriding was carried out in a 50-cm diameter stainless steel Bell jar/ vacuum chamber using a pulsed of dc glow discharge, operating at 10kHz, and at a voltage of 600 V. The samples were placed on the sample holder behaving as a cathode with respect to the chamber wall. The cathode current density was ~2 mA/cm^2. The sample was nitride for a time period of 20h at a substrate temperature of 530°C. After the completion of the nitriding, the sample was cooled in the chamber to room temperature in the presence of nitrogen flowing gas to minimize surface oxidation. (I. Alphonsa, A. Chainani, P.M Raole, B. Ganguli, P.I John, 2001)

CHAPTER 3

METHODOLOGY

3.1 Project Flow

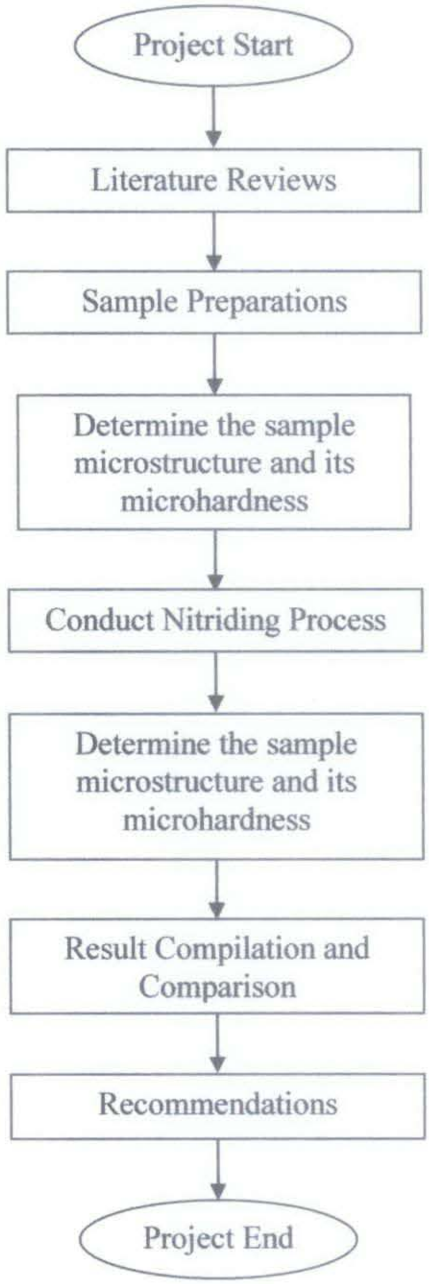

Figure 3.1: Methodology of the project

3.2 Project Phases

3.2.1 Literature Review

For the first stage of the research, understanding and identifying the problem statement and project objective is a must. Then, all the relevance information which is related to the project base is being gathered. These include the details of the stainless steel types and its mechanical properties, nitriding process and its types. Based from the research there are three methods of ntirding and only the best or the most suitable method of nitriding will be performed. A part from that, survey or studies from the related journals, articles or even from internet has been done.

3.2.2 Sample Preparations

Acquire the substrate material from Supervisor.

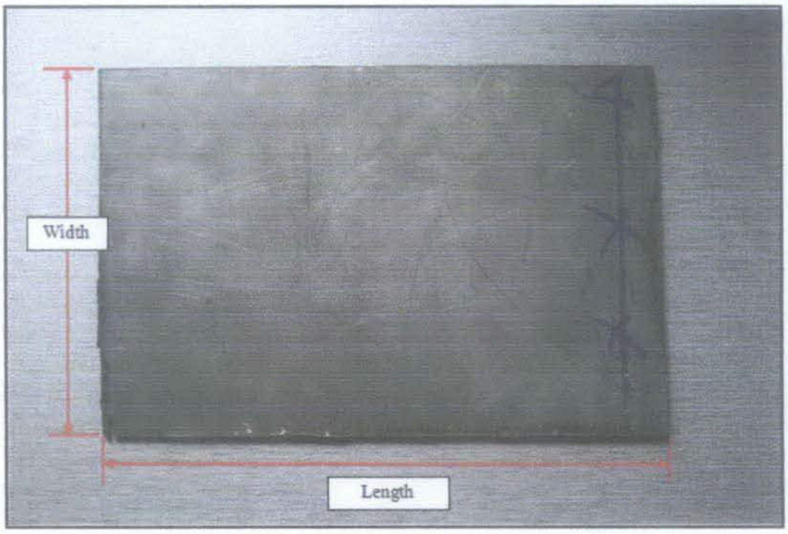

Figure 3.2: Martensitic Stainless Steel sheet

Dimension:

- Width, w = 13.2cm
- Length , l = 20.5cm
- Thickness, h = 0.2cm

From that, the substrate will be cut into smaller dimension (20mm X 20mm) using EDM Wire Cut. Raw sample is being studies using the metallographic technique and bellow are the related procedures in it:

1. Sectioning.

   Secitioning is being performed since we are only interested in evaluating or studying the microstructure at the cross section of the sample. Besides, it is also due to the size limitation of sample to be examined under optical microscope. Hence, the sample (20mm x 20mm) is being cut into half by using the abrasive machine.

Figure 3.3: Abrasive machine

2. Mounting

   Mounting is required when the sample is small or oddly shaped and the sample edge area needs to be examined. There are two types of mounting:

   i. Thermal Mounting

      The sample is embedded in thermosetting plastics at elevated temperatures and pressure.

   ii. Cold Mounting

      The sample is embedded in epoxy type of materials.

As for this project, the author used the thermal mounting process.

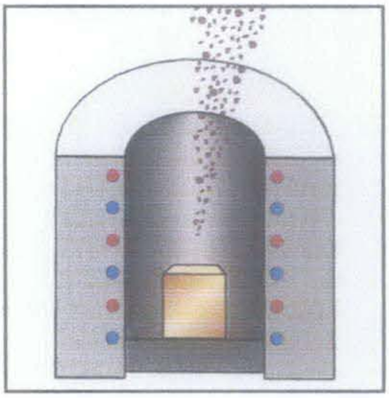

Figure 3.4: Schematic display of the sample in an Auto Mounting Pressing machine

Figure 3.5: Auto Mounting Press machine

3. Grinding

Grinding removes the damages on the surface produced by sectioning. However, grinding also produced damages which must be minimized so that subsequent grinding with finer abrasives is achieved. At the end of grinding phase, the only grinding damages present must be from the last grinding step.

Grinding materials: Abrasive paper (covered with silicon carbide grits). Series of abrasive paper is used, from coarse to fine one.

Grit sequence: 120-, 240-, 320-, 400-, 600- and etc.

Note: Initial grit size depends on the surface roughness and depth of damage from sectioning.

i. Surfaces cut by abrasive cutoff saw start with 120 to 240 grits.

ii. Surfaces cut by EDM or diamond saw start with 320 to 400 grits.

Figure 3.6: Grinding and polishing machine

4. Polishing

The sample is polished to produce a flat and scratch-free surface with high reflectivity.

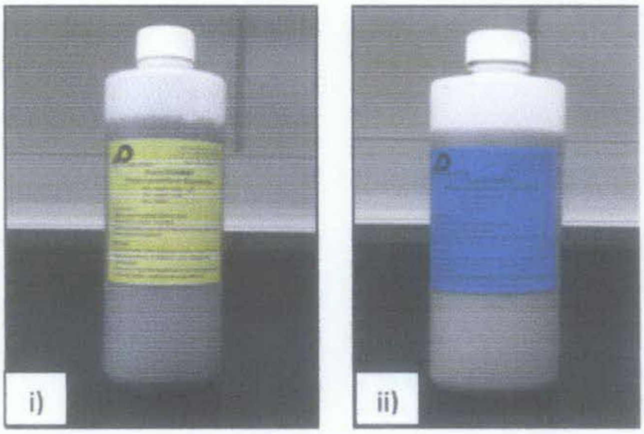

Figure 3.7: i) 3μm and ii) 1μm DIAMAT Polycrystalline Diamond

5. Etching

Method that used chemical to dissolve into the surface of materials and it will reveal the microstructure of the surface during the microscope inspection. As for the martensitic stainless steel, the most suitable etchant is Marble 5.

Figure 3.8: Marble reagent

Below are the steps or procedures for etching the sample:

i. Marble reagent is being applied evenly on the entire surface of the sample and let it be around 1 minute.

ii. Then, the sample is being washed with water.

iii. Next, washed or sprayed it with ethanol.

iv. Lastly, the sample is being dried properly and make sure that there is no leftover water on the surface.

3.2.3 Microstructure Studies and Microhardness Test

1. Microstructure studies

After all the samples are perfectly polished and etching, it will undergo microstructure examination using the Optical Microscope and Field Emission Electron Microscope machine. It is very important for the component to be in mirror finish so that it will ease the surface examination. The same goes to the etching procedures where it will reveal the microstructure of the sample. By using the Optical Microscope, the microstructure can be identified while FESEM is used to the identified the composition of the samples.

Figure 3.9: Optical Microscope

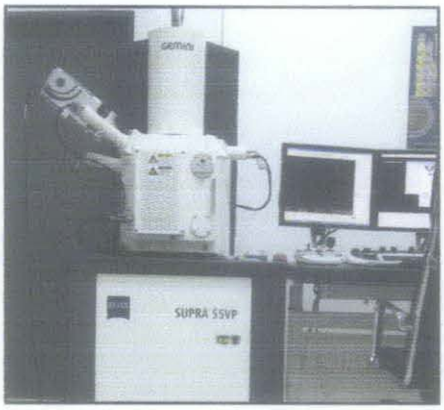

Figure 3.10: Field Emission Scanning Electron Microscope

2. Microhardness testing

For this microhardness testing, the author used the Vickers microhardness testing. In this testing, the hardness value is determined by measuring the size of the resulting unrecovered indentation with a microscope and using established formulas or conversion tables in accordance with ASTM E 384. The Vickers hardness value is recorded.

Figure 3.11: Vickers Microhardness tester

3.2.4 Conduct Nitriding Process

In this process, a Carbolite horizontal furnace tube, nitrogen supply, flow meter and a cone flask will be used. All of them are connected to each others. A flow meter is used to observe the flow of nitrogen into the tube. For this project, the author performed the nitriding process with three different temperatures; 950°C, 1000°C and 1050°C while the time interval is set to six hours each.

Figure 3.12: Carbolite horizontal furnace tube

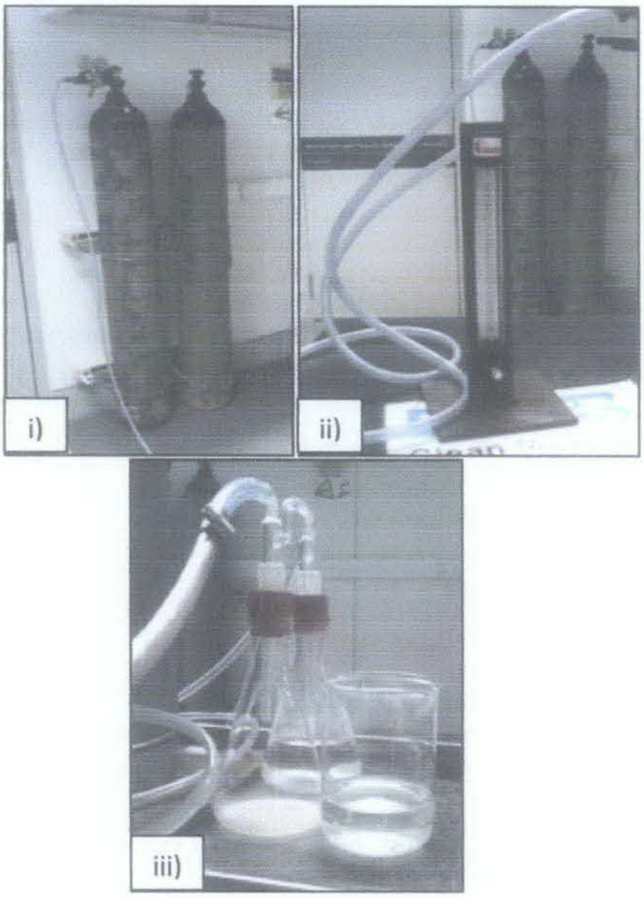

Figure 3.13: i) Nitrogen supply, ii) Flowmeter, iii) Cone flask and beaker

Below are the procedures in performing nitriding process:

1. Sample is cleaned using ultrasonic cleaning where the samples were immersed with acetone.
2. The initial weight is recorded.
3. The sample is placed in the alumina boat and inserted into the heating zone in furnace.
4. Air in furnace is purging with nitrogen for 15 minutes and at a flow rate of $1000cm^3/min$ to prevent oxidation of the samples.

5. Heating at $15^{o}C$ will be start immediately after the purging complete.
6. The required cycle, segment, temperature and nitriding period are set.
7. The heater will be turned on and the furnace is heated to temperature of $950^{o}C$ for six hours and nitrogen will be introduced into the furnace with flow rate of $1000cm^3/min$.
8. After six hours, the nitride sample is left to cool by slow cool.
9. Open the furnace and the sample will be taken out from it.
10. Record the final weight of the sample.
11. Repeat the procedures for temperature of $1000^{o}C$ and $1050^{o}C$.

3.2.5 Microstructure Studies and Microhardness Test

As for the microstructure studies and microhadness test of the nitirided samples; $950^{o}C$, $1000^{o}C$ and $1050^{o}C$, all the procedures from sectioning to Vickers microhardness testing is repeated. The data is recorded.

3.2.6 Result Compilation and Comparison

The result obtained from the microstructure studies and Vickers microhardness testing is recorded. Analyze the result from the Optical Microscope and FESEM examination especially in terms of the difference in microstructure of the samples. Compare the hardness of the sample between the raw sample and nitride samples.

3.2.7 Recommendations

Based from the analysis and discussion, recommendations for future works are required. This will help and guide the others in completing their projects.

3.3 Equipments and tools

Materials:

- Martensitic stainless steel

Equipments:

- Abrasive cutter
- Grinder and abrasive paper
- Polisher and water based polycrystalline diamond
- Etching reagent
- Acetone
- Ultrasonic cleaning
- Carbolite horizontal tube furnace
- Optical microscope
- FESEM – EDX (Field Emission Scanning Electron Microscope – Energy Dispersive X-ray Spectrometry)
- Vickers microhardness

3.4 Project Gantt chart

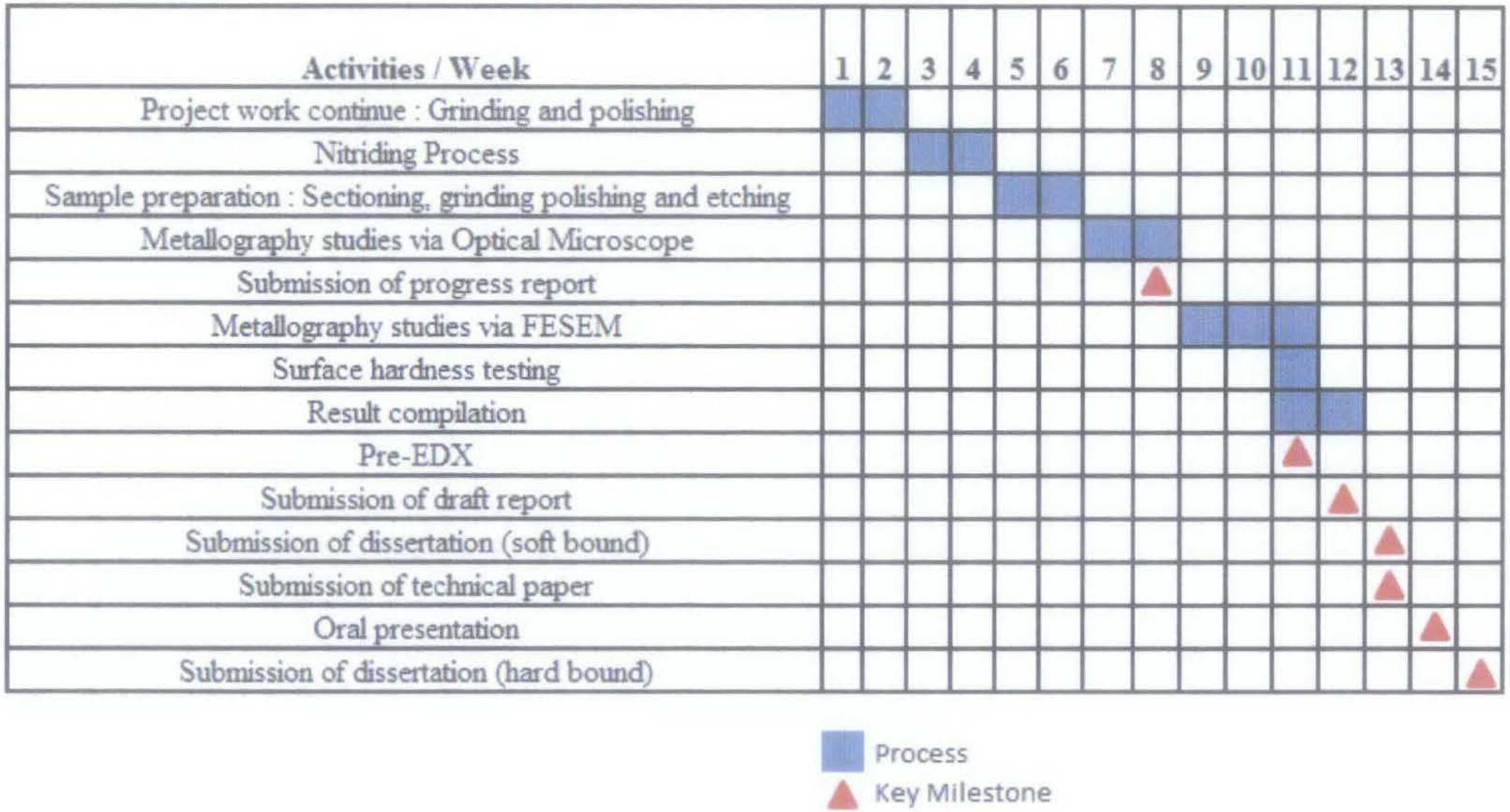

Figure 3.14: FYP II Gantt Chart

CHAPTER 4

RESULTS AND DISCUSSION

This chapter includes the experimental results of the untreated (as received) and heat treated AISI 410 stainless steel through nitriding process. It also includes the result from the sample preparations procedures, microstructure analysis and Vickers microhardness testing.

4.1 Sample Preparation Result

Four cross section of sample have been grinded, polished and etched. The conditions of the samples are as below:

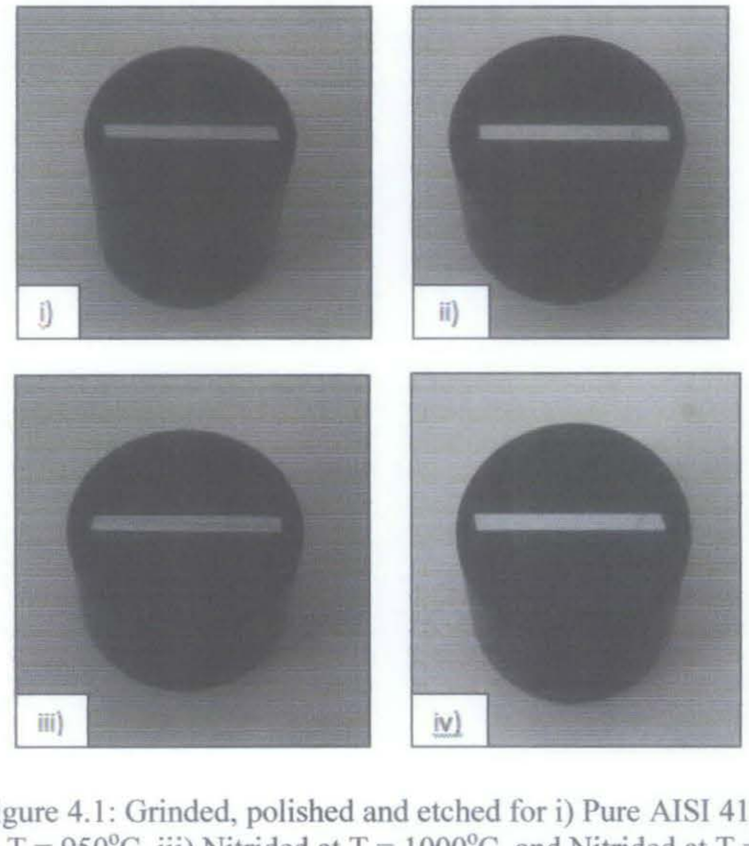

Figure 4.1: Grinded, polished and etched for i) Pure AISI 410, ii) Nitrided at T = 950°C, iii) Nitrided at T = 1000°C, and Nitrided at T = 1050°C

Between all the samples above, there is no significant different in naked eyes. This is because we are only interested in the microstructure of the samples and it only visible under the Optical Microscope and Field Emission Scanning Electron Microscope.

4.2 Untreated Stainless Steel

Figure below show the microstructure of the untreated AISI 410 stainless steel. The plate-like or needle-like grains are the martensite phases. The magnification is 500x.

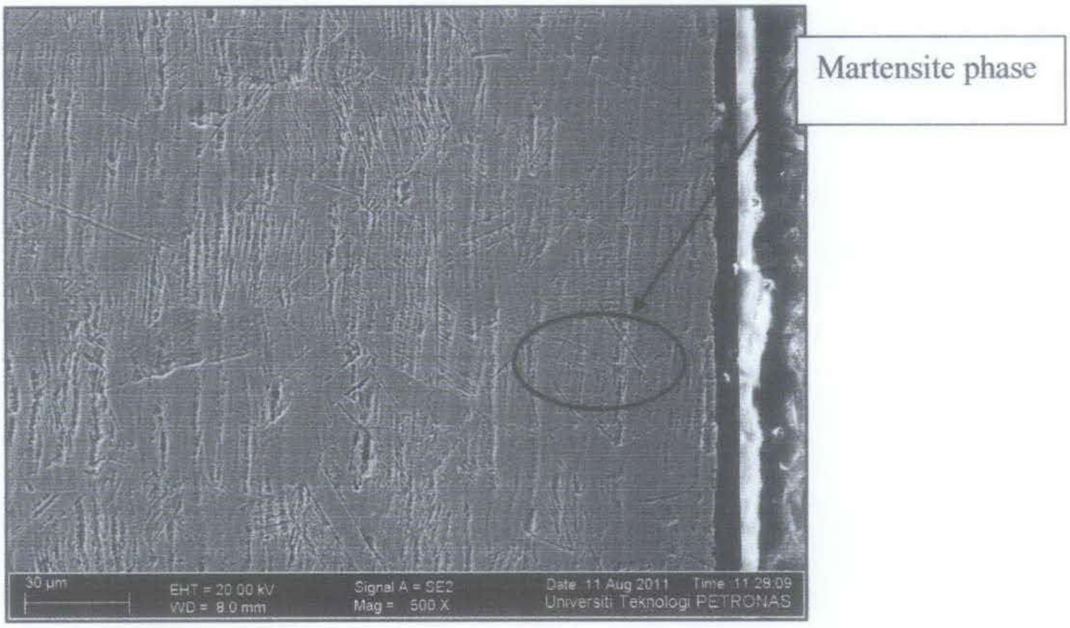

Figure 4.2: Microstructure of untreated AISI 410 stainless steel (x500)

From Table 4.1, it shows the surface hardness of untreated AISI 410 (as received). The measurement was taken at 5 different points. The load applied was 10gf and the dwell time was 15 seconds.

Table 4.1: Surface hardness of untreated AISI 410

Distance, µm	**Vickers Hardness, HV**
0	254.0
100	257.4
200	259.7
300	253.2
400	255.9
500	251.4
600	256.3
700	252.5

The average hardness of the untreated AISI 410 is 255.05HV which is almost the same hardness value for the AISI 410 at room temperature; 254HV.

4.5 Mass of the samples

Nitriding will cause nitrogen to diffuse into the surface of the metal. This diffusion will increase the mass of the samples. Mass of the samples before and after nitriding process is recorded. If the mass of the samples increased, it showed that there is nitrogen diffusion across the surface of the samples. Table 4.2, 4.3, and 4.4 below shows the recorded mass of the sample before and after it undergoes nitriding process at different nitriding temperatures.

Table 4.2: Mass differentiation for nitriding at 950°C

Before Nitriding	**After Nitriding**
6.2450 g	6.2470 g
6.2400 g	6.2465 g
Avg = 6.2425 g	Avg = 6.2468 g

Table 4.3: Mass differentiation for nitriding at 1000°C

Before Nitriding	**After Nitriding**
6.3340 g	6.3400 g
6.3337 g	6.3390 g
Avg = 6.3339 g	Avg = 6.3395 g

Table 4.4: Mass differentiation for nitriding at 1050°C

Before Nitriding	**After Nitriding**
6.4720 g	6.4840 g
6.4690 g	6.4870 g
Avg = 6.4705 g	Avg = 6.4855 g

4.6 Microstructural Changes Nitrided Stainless Steel

In this section, the microstructure of each sample after nitriding was revealed in accordance with the processing parameters. The observation was mainly based on the changes in the grain boundaries of the samples and the presence of the nitrided layer particularly located at the outermost of the cross sectional surface.

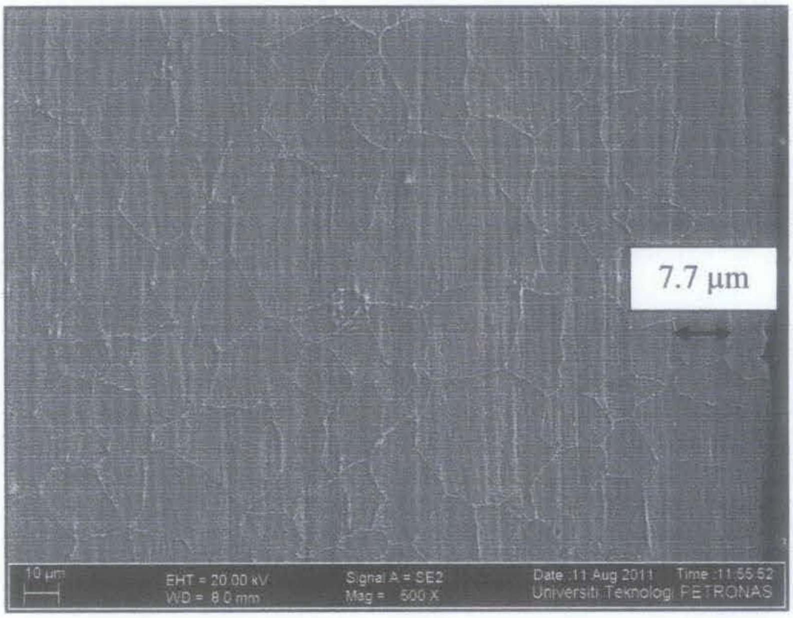

Figure 4.3: Microstructure of AISI 410 nitriding at 950°C (x500)

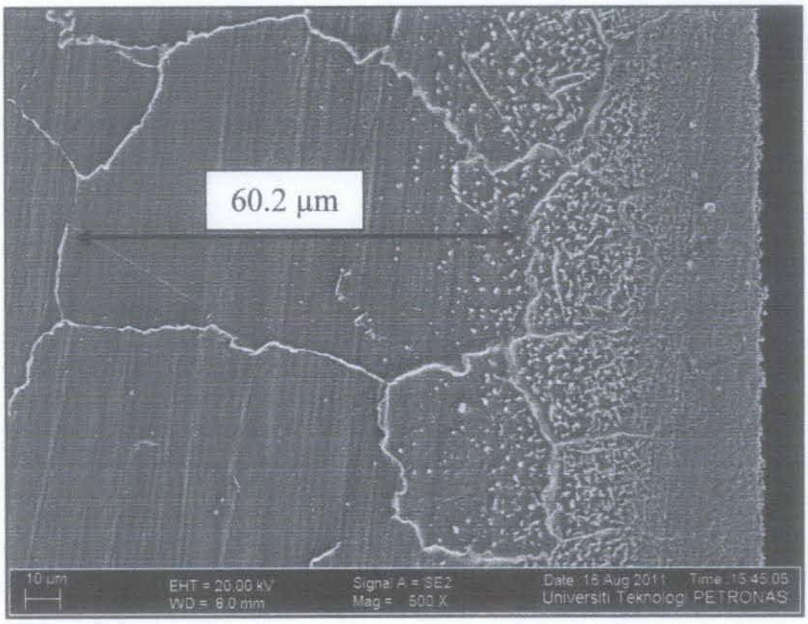

Figure 4.4: Microstructure of AISI 410 nitriding at 1050°C (x500)

From Figure 4.3 till Figure 4.5, there were significant changes in the shape of the grain boundaries of the sample. When the sample was being nitriding at 950°C, the needle-like phase or martensite phase has disappeared. This is due to the slow cool process where there is no phase transformation from austenite to martensite phase. The boundaries become more apparent, clearer, and more structured.

As the nitriding temperature increase, the grain boundaries also increase. This is proven by comparing the grain size from 950°C nitriding temperature with the grain size from 1050°C nitriding temperature. From Figure 4.3, the grain size is in the range of 7.7 μm while from the Figure 4.5, the grain size is in the range of 60.2 μm. Both of the figures are taken under 500x magnifications.

There is no white layer or nitride layer located at the outermost of the surface. The layer only visible for the low nitriding temperature process. Hence, the expected result is similar with previous study. A part from that, there were formation of precipitate near the grains boundaries of the outermost of the surface. These precipitate as a result from the formation of chromium carbide precipitates. However, these precipitate can only be identified through X-Ray Diffraction analysis.

Figure 4.5: Microstructure of AISI 410 nitriding at 1050°C (under optical microscope.

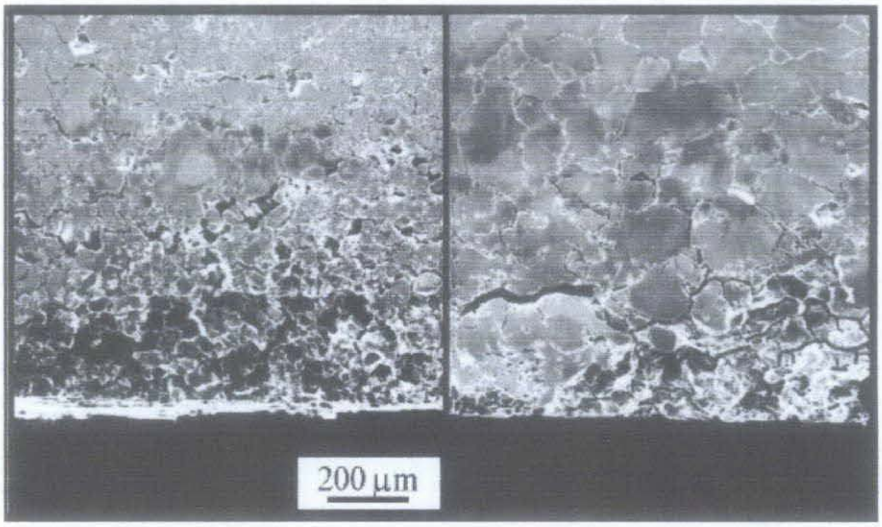

Figure 4.6: Sensitization phenomena due to intergranular corrosion

Based from the Figure 4.6 and Figure 4.7, the strucutural inside the figure is quite similar especially in term of the presence of the cracks along the grain boundaries. Based on that, the author suspects that this particular sample may experience intergranular corrosion. This is because the sample is being heat treated at the temperature of 1050°C which is above the sensitization temperature range (510°C – 790°C). Thus, the steels become "sensitize" and become prone to the intergranular attack during slow cooling to room temperature. This sensitization involves the precipitation of Cr carbide ($Cr_{23}C_6$) at the grain boundaries.

4.7 EDX Line Scan Mode Analysis

Based from the discussion in section 4.5, it is inappropriate for the author to just simply make the assumption that there was nitrogen diffusion across the surface of the sample. Hence, a line scan mode analysis was made just to prove and also to validate the assumption. Below are the result obtained from the line scan mode analysis:

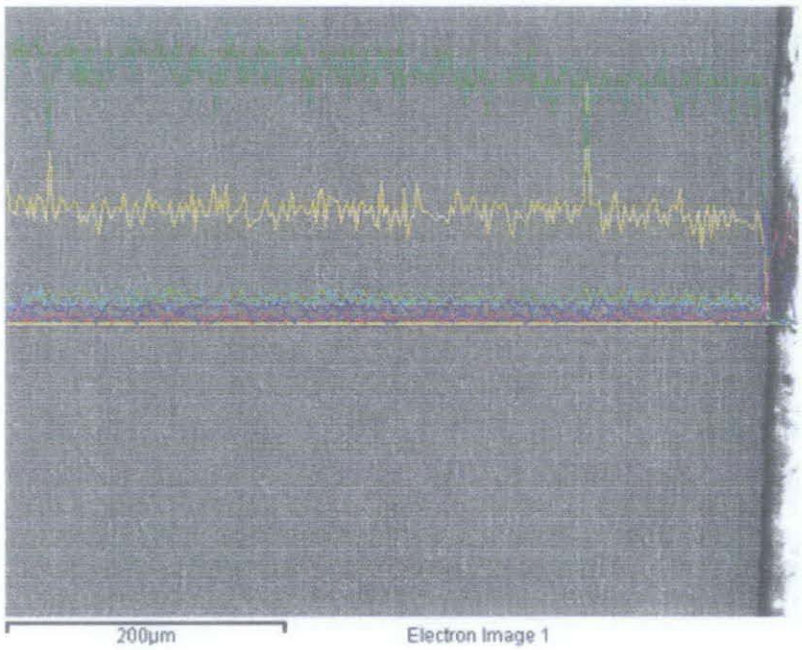

Figure 4.7: Overall line scan mode analysis of the nitrided sample at 950^{o}C

Note:

Red line: Nitrogen

Dark yellow line: Chromium

Light green line: Manganese

Green line: Iron

Light blue line: Nickel

Blue line: Copper

Purple line: Silicon

Pink line: Carbon

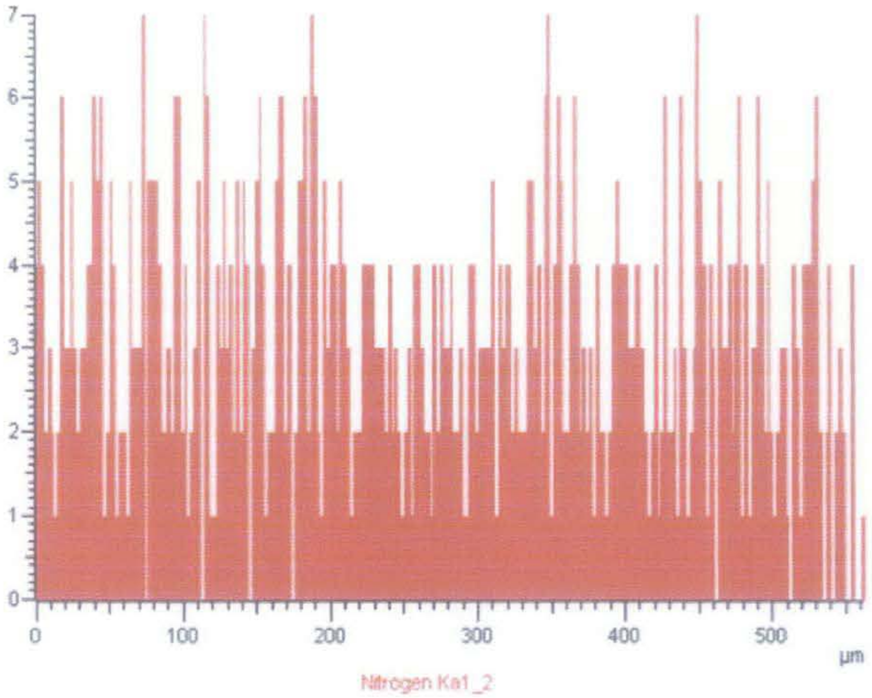

Figure 4.8: Nitrogen line scan mode analysis of the nitrided sample at 950°C

For the EDX line scan mode analysis, the SEM electron beam is scanned along a preselected line segment across the sample while x-rays are detected at discrete positions along the line. From Figure 4.7, it shows all the related elemental concentration variance for that particular sample. From Figure 4.8, it show the elemental line scan mode analysis which the nitrogen element. The overall intensity of nitrogen was measured in counts per second (cps) where it records the highest value at 7 cps. Based on that, it is sufficient enough to show and to prove that there is significant nitrogen diffusion across the surface of the sample.

4.8 Hardness Value of Nitride AISI 410 Stainless Steel

In order to evaluate the samples hardness after the heat treatment procedures, Vickers microhardness was conducted. The hardness of the samples was tested from the edge of the nitride surface till its core and the result is recorded in the table. The load used was 10gf and the dwell time was 15 seconds.

Table 4.5: Vickers hardness for nitriding at 950°C

Distance, µm	Vickers Hardness, HV
0	296.2
100	280.8
200	273.9
300	269.7
400	262.4
500	258.7
600	255.0
700	250.8

Table 4.6: Vickers hardness for nitriding at 1000°C

Distance, µm	Vickers Hardness, HV
0	320.0
100	304.9
200	283.4
300	279.8
400	260.7
500	257.8
600	256.3
700	255.1

Table 4.7: Vickers hardness for nitriding at 1050°C

Distance, µm	Vickers Hardness, HV
0	385.1
100	366.2
200	341.8
300	309.4
400	285.4
500	271.5
600	267.3
700	257.1

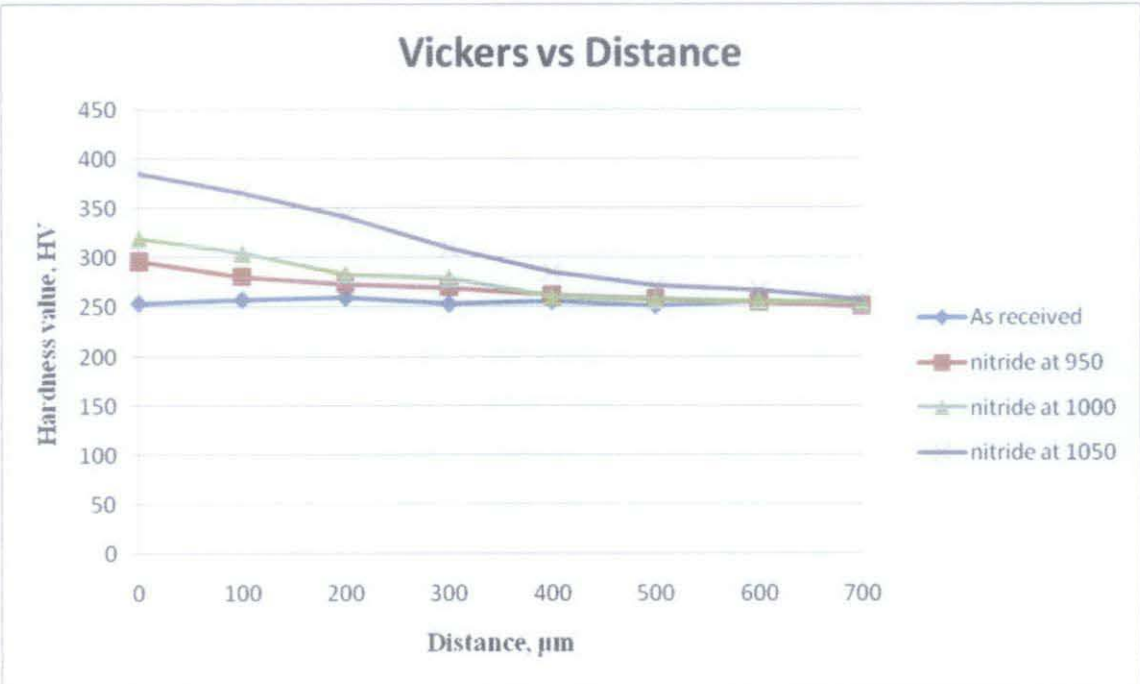

Figure 4.9: Vickers hardness vs. Distance

According to the result from Figure 4.9, the hardness for the treated AISI 410 is supposed to be lower than the hardness of the untreated AISI 410. This because of the disappearance of martensite phase. However, based from the data from Table 4.5 till Table 4.7, it shows the opposite result. This is due to the presence of the diffuse nitrogen inside the surface of the sample. This diffuse nitrogen strengthens the surface and increases the hardness of the sample.

Also based from the Table 4.5 till Table 4.7, the highest hardness of the sample is located near the edge of the sample for respective temperatures. This is due to the higher composition of nitrogen at that particular location. The hardness of the samples decreases as it moves further away from the edge of the samples. Besides that, the surface hardness also increases when the nitriding temperature is increased.

Nitriding the sample at 1050°C produce the highest surface hardness where the hardness is 385.1HV. The hardness decreases and it becomes constant when it reach the 500μm from the surface. After this distance, the hardness of the sample is in the range of the untreated AISI 410 hardness; 254HV.

CHAPTER 5

CONCLUSION AND RECOMMENDATIONS

5.1 Conclusion

Based from the result obtained for the mass of the samples, it shows that the mass of the sample increased when subjected to the nitriding process. For nitriding at 950°C, the mass increment was 0.0043 g. while for nitriding at 1000°C, the increment was 0.0056 g and the last one is 0.015 g increment. The mass increment is related to nitriding temperature where more nitrogen will diffuses at higher nitriding temperature.

In terms of micrographic analysis, we can say that when the martensitic stainless steel is subjected to high temperature gas nitriding and was slow cool, the martensite phase in its microstructure will disappeared. The grains become more apparent, and more structured. As the nitriding temperature increases, the size of the grain also increases. A part from that, there is no presence of white layer at the outermost of the sample surface. The white layer only occurs in low temperature gas nitriding.

For the hardness test result, nitriding at 1050°C shows the highest hardness among all the samples where it reached 385.1HV. Then, followed by the hardness of nitriding at 1000°C and 950°C. It shows that the higher the nitriding temperature is, the higher the hardness will be produced. However, the hardness of the sample will decrease as we move further away from the nitride surface. It will keep on reducing until it reached the average value or even lower hardness value as compared to the untreated AISI 410.

As for the conclusion, this study has managed to achieve its objectives which to analyze the changes in the microstructure of the sample and also to evaluate the hardness of the materials when subjected to high temperature gas nitriding.

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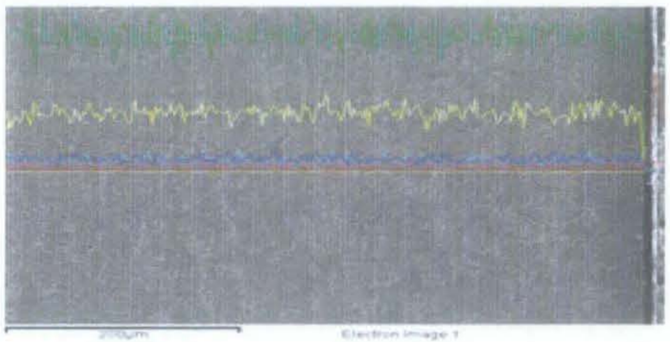
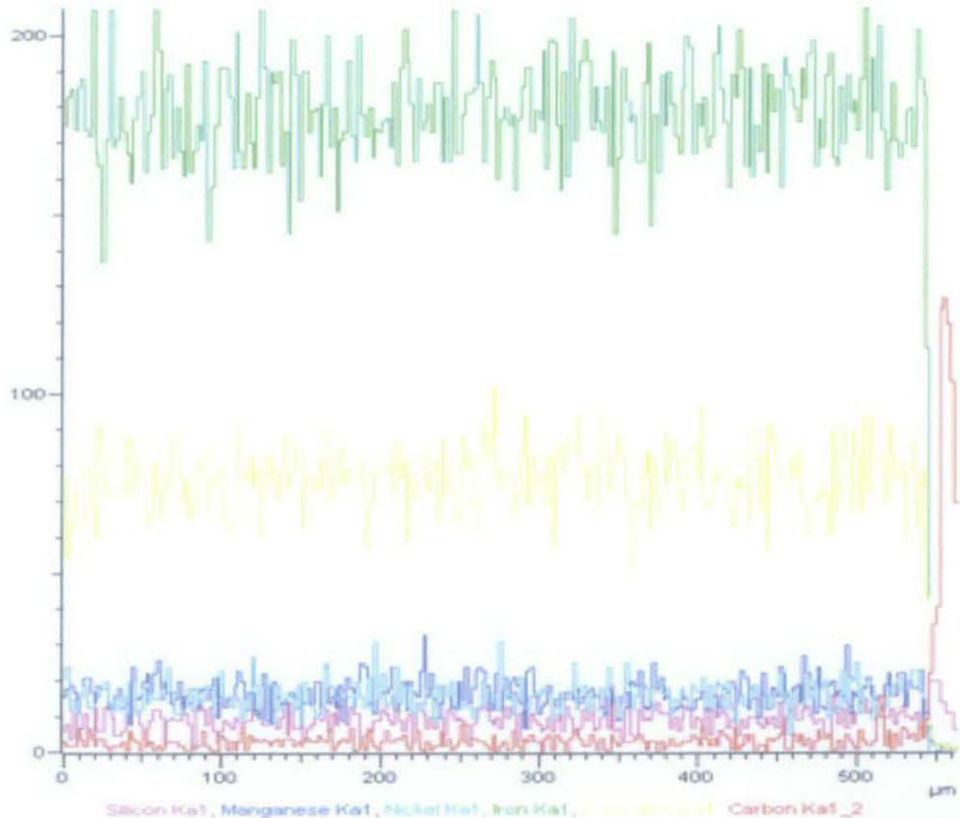
APPENDIXES

EDX Line Scan Mode Analysis – As Received sample





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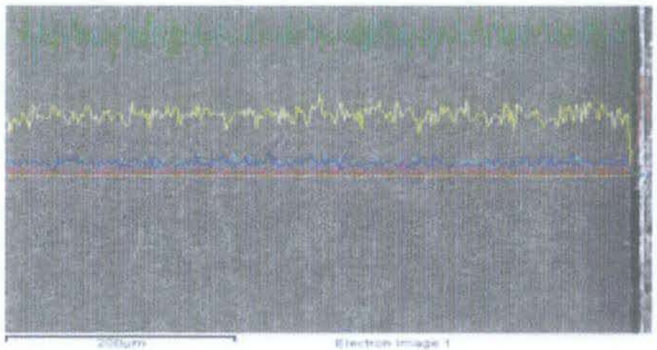

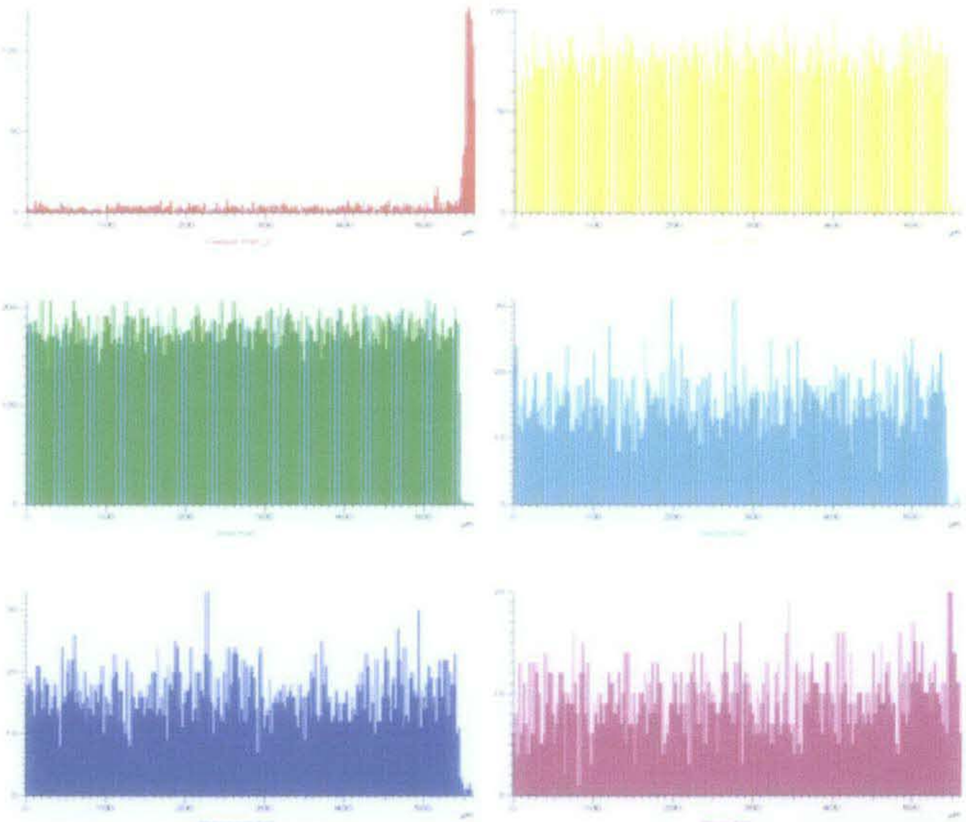

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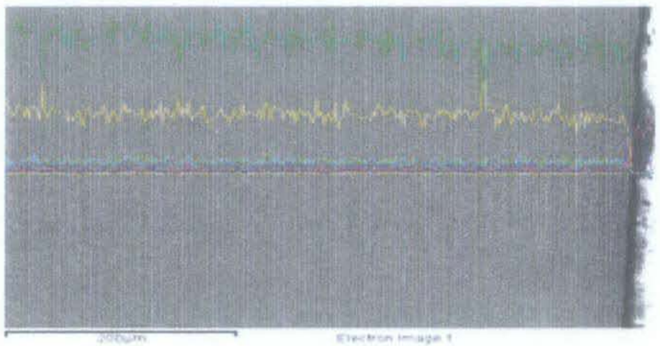
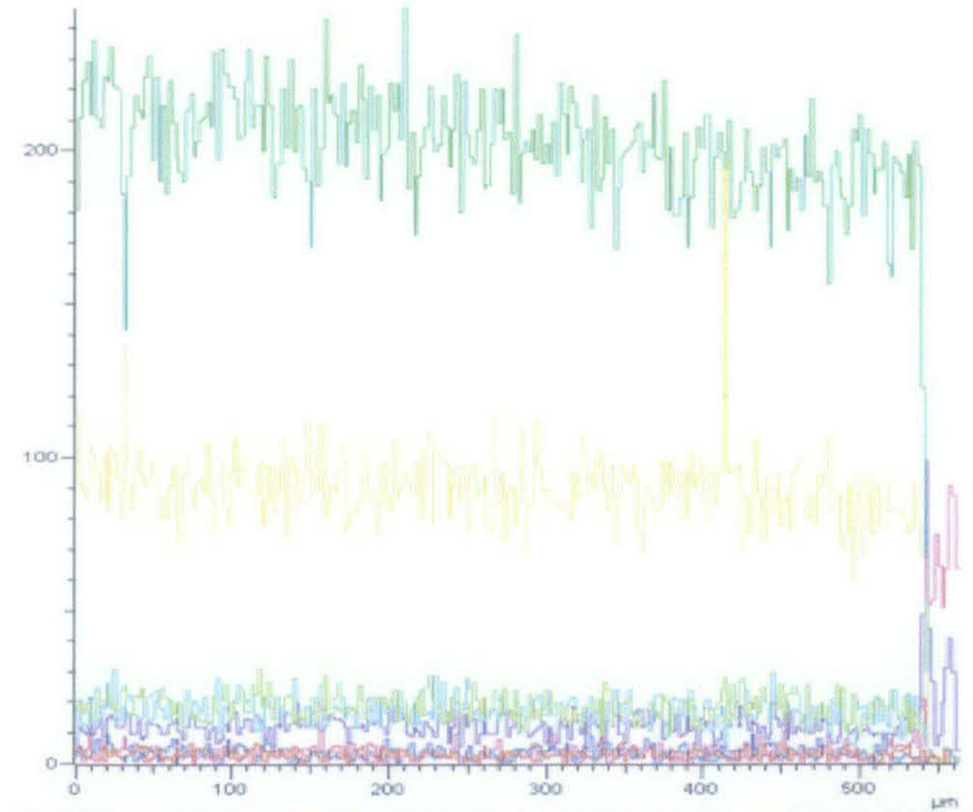
EDX Line Scan Mode Analysis – Nitride at 950°C





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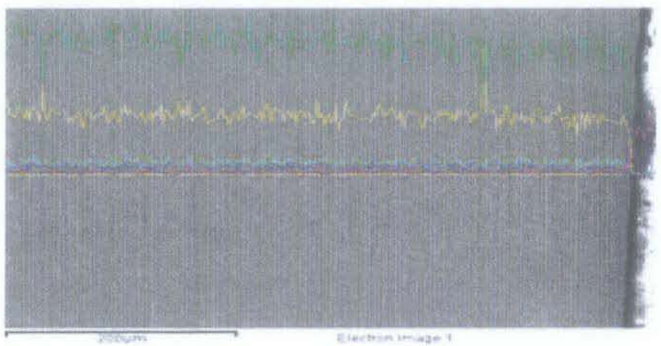

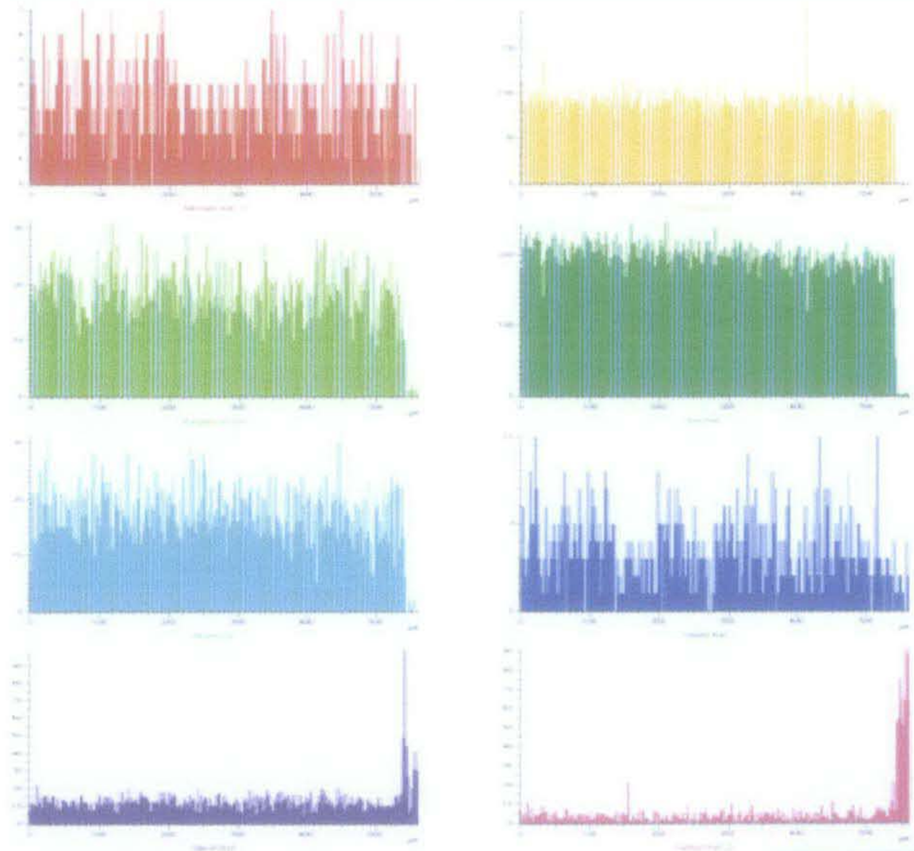

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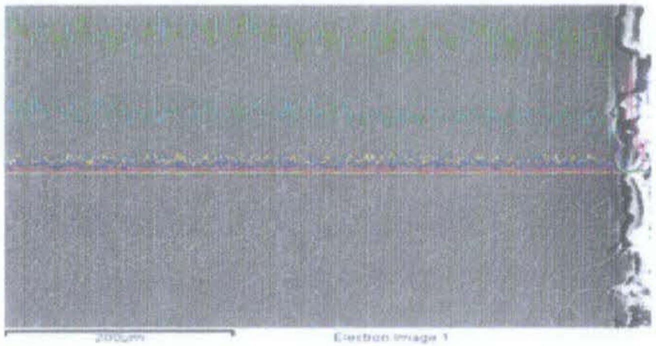

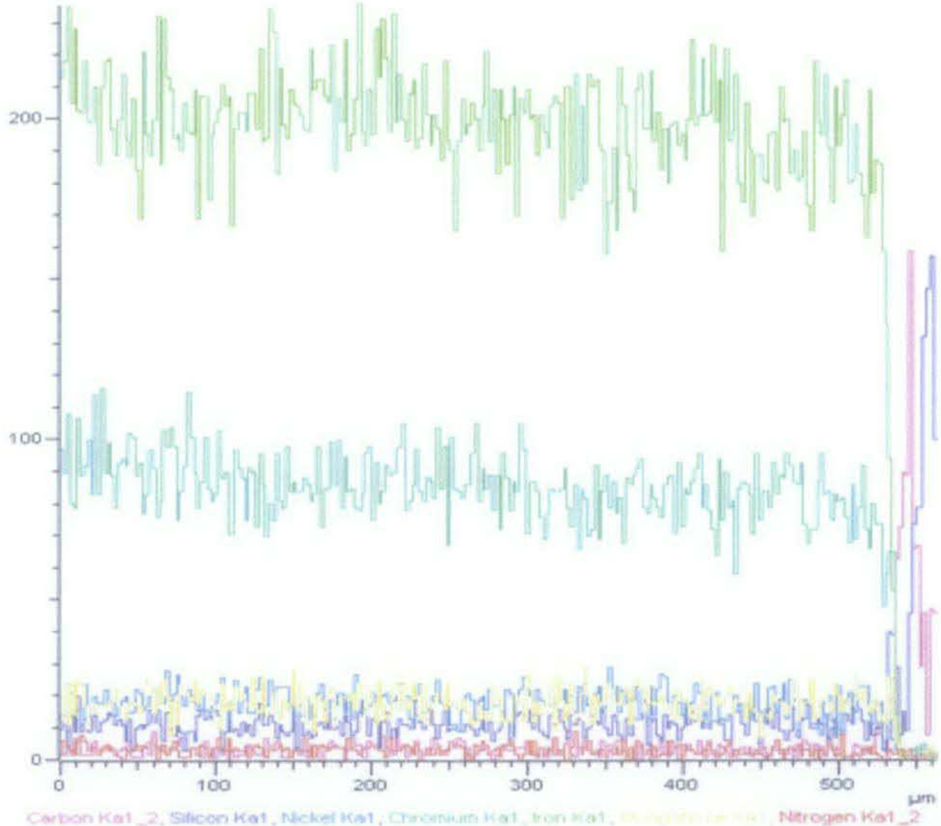

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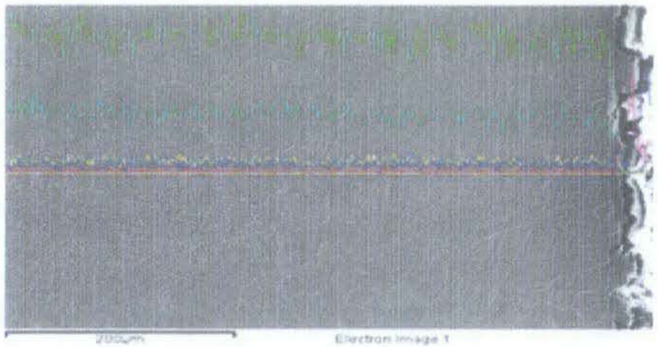

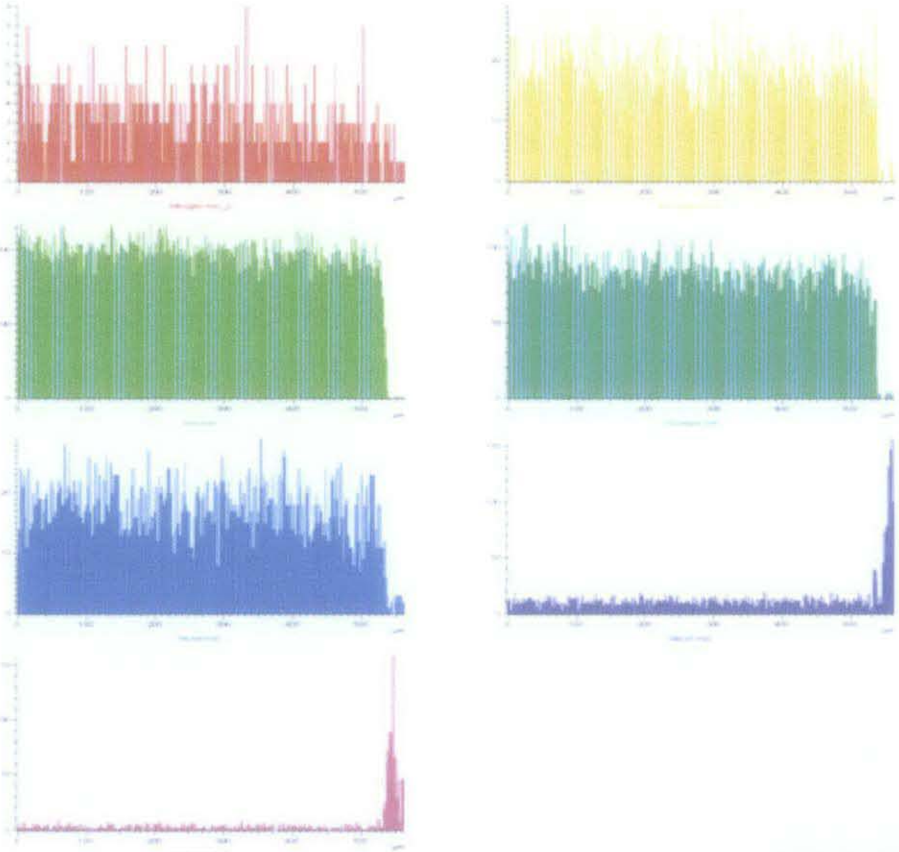

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EDX Line Scan Mode Analysis – Nitride at 1050°C

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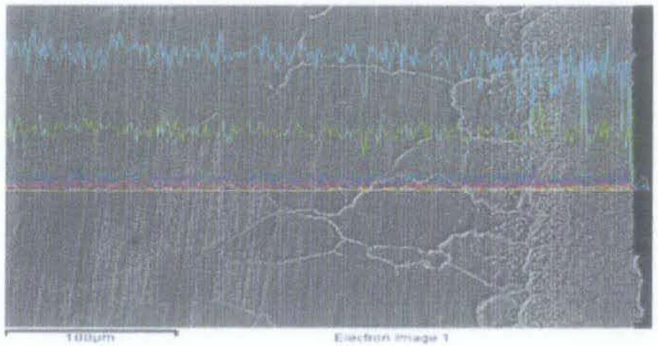

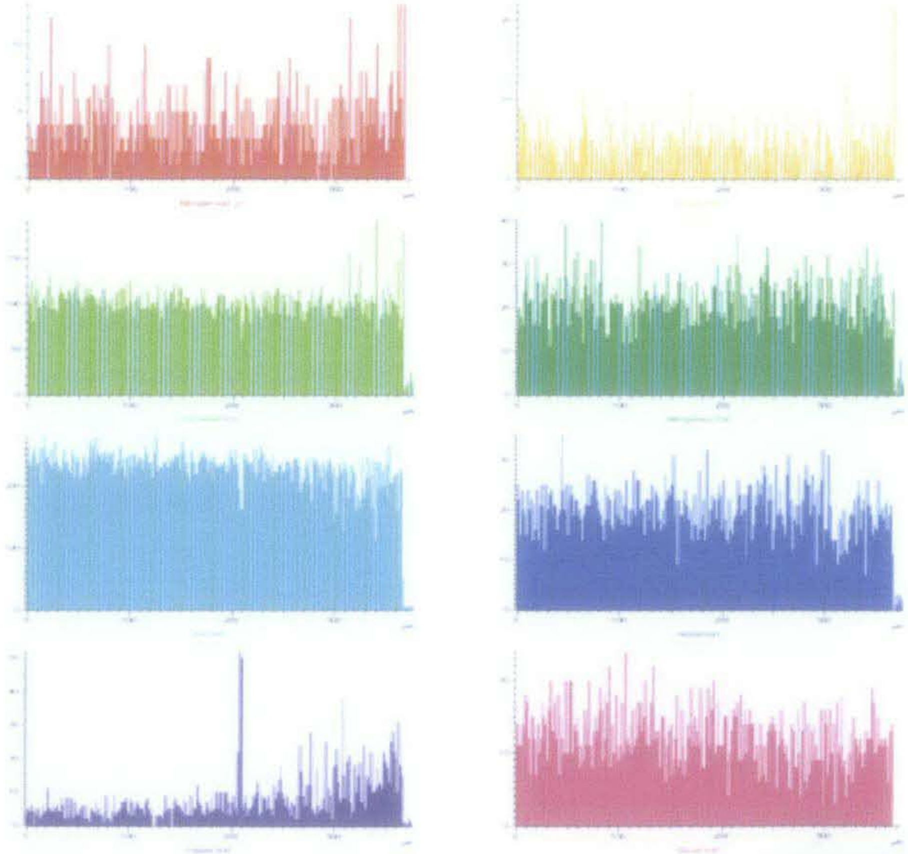

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