Investigation on Sensitization of Nitrided AISI 316L Austenitic Stainless Steel at Intermediate Temperature

by

Mohd Amri Azri Bin Zulkifli

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

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Universiti Teknologi PETRONAS
Bandar Seri Iskandar
31750 Tronoh
Perak Darul Ridzuan

CERTIFICATION OF APPROVAL

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Approved by,

_____

(AP Dr.Patthi Bin Hussain)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
SEPTEMBER 2013

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.

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MOHD AMRI AZRI BIN ZULKIFLI

ABSTRACT

Austenitic stainless steel is widely used around the globe especially in Oil and Gas industry and it gives about more than 60% of total stainless steel production. However, the application of this material is severely limited by very poor friction and wear behavior. Investigations have been performed to improve surface hardness of ASS by heat treatment thus enlarging their possibility of wider application, but led significant loss of its corrosion resistance. This tendency occurs due to the sensitization effect. The purpose of this investigation is to determine the effects of sensitization on the mechanical properties of ASS especially on hardness. Besides that, this project also will determine the patterns and differences of microstructure and chemical composition for both untreated and nitrided Austenitic Stainless Steel. Theoretically, sensitization is occurred due to the present of nitride and carbide precipitate at the grain boundaries. Heat treatment at intermediate temperatures which are at 600°C, 700°C, and 800°C will be used as the manipulating variable while 4 hours holding time as constant variable. The environment gas for this treatment is nitrogen and it was purged into the furnace where the steel is located. There will be 1 mechanical test being conducted towards the sample and it is Vickers Microhardness Test. This investigation also strongly supported by conducting nitrided layer morphology by utilizing Optical Microscope (OM) and Scanning Electron Microscope (SEM). Crystallographic structure of the nitrided steels also will be investigated by using X-ray Diffraction (XRD) machine while Energy Dispersive X-ray (EDX) analysis is used to check on the chemical composition of the samples at various spots. From the result, it can be summarized that hardness is gradually decreasing with depth profiles of the cross section and the sensitization of chromium nitride is increasing with nitriding temperature. All in all, the characteristics of chromium/nitride precipitation at the grain boundaries and Vickers Microhardness Test are verified.

ACKNOWLEDGEMENT

I would like to thank Allah S.W.T (the greatest God) for His guidance for giving me strength throughout the whole period of the Final Year Project. I am so praised to Allah S.W.T for His blessings for giving me an opportunity to meet the objectives of the Final Year Project itself. Without His guidance and blessings, the objectives of the Final Year Project could not be met.

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Last but not least, I would like to thank everyone that is involved throughout the completion of the Final Year Project, which I consider them as my respective family members. All the knowledge, experiences, advices, positive values that has been showed throughout the whole period of program is useful for me in the future. Without support and motivation that has been given to me, I would be struggling to handle the entire tasks and responsibilities that have been given to me.

-Mohd Amri Azri Bin Zulkifli-

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CHAPTER 1

INTRODUCTION

1.1 Background Study

Austenitic Stainless Steel with excellent corrosion resistance and good weldability have wide applications in industry especially in oil and gas industry. These iron-based alloys contain high level of chromium which form protective oxide film on the surface hence resisting the corrosion. The oxide film regenerates when damaged, thus making the steel stainless. However, during welding process or heat treatment, carbide and nitride precipitation might be happened because they are insoluble at that certain temperature. In order for the precipitation to occur, it needs chromium from the surrounding metal and this will lead to chromium-depleted zones at the boundaries. This depleted zones will be the focus point for the intense corrosion to occur and known as sensitization. Sensitization usually happens at an intermediate temperature typically at 500°C to 900°C. (Skrabski, 2010). Sensitization will affect the hardness and the corrosion resistance of the Austenitic Stainless Steel.

1.2 Problem Statement

Austenitic Stainless Steel (ASS) is widely used in all industries for various types of applications starting from agriculture up to the aerospace. This is due to its high resistance to corrosion because the formation of a thin, transparent chromium oxide coating that is naturally occurred. ASS is defined as a steel containing at least 11% chromium. It can protect the metal from many corrosive environment including sea water, certain acids and

moist air. Normally these films are free of pores, but their stability may be weakened locally. It therefore has different properties in areas where the steel surface is altered due to grain boundaries precipitates. This heterogeneous is very dangerous since it weakens steel without much change in the outward appearance.

But then, this ASS will be much expose to sensitization effect during welding and heat treatment process. Sensitization effect actually the formation of chromium nitride and it will precipitate at the grain boundaries. As the chromium constituents decrease, it cannot form into chromium oxide or passive layer as the corrosion resistance. Thus, corrosion resistance decrease and lead to intergranular corrosion. (Shu-Xin Li, 2013). High concentration of chromium nitride decrease locally the chromium content in the region that is adjacent to these chromium rich precipitates. Since the chromium diffuses much slowly than carbon, these is not enough time for the chromium to diffuse to the nitride from all over the grains, so, in the region that is near to grain boundaries, chromium content will be lower the theoretical composition.

1.3 Objective

There are 3 purposes of this project which are:

- To determine the characteristics of the sensitization of nitrided 316L Austenitic Stainless Steel by OM, SEM, EDX and XRD means.
- To determine the hardness and strength of nitrided 316L Austenitic Stainless Steel at different temperature and depth profiles at the cross section.
- To observe and compare of any microstructure changes of the samples before and after been nitrided.

1.4 Scope of Study

This project covers on the conducting the gas nitriding at different temperatures on 316L Austenitic Stainless Steel and will investigate the characteristics of sensitization after nitriding process. Beside that, Vickers Microhardness Test will be determined. Non-treated 316L Austenitic Stainless Steel sample will be used as a reference material for the comparison purpose.

The conventional intermediate temperature gas nitriding process will be conducted in a horizontal tube furnace for many times. This gas nitriding process will be following strictly to ASTM A355 standard specifications.

In this experiment, some analysis tools will be used such as Glancing X-Ray Diffraction (XRD), Optical Microscopy (OM), Scanning Electron Microscope (SEM), Energy Dispersion X-ray (EDX), and Vickers Microhardness Test. This hardness tests have been done by following and referring to the standard of ASTM E-92, Standard Test Method for Vickers Hardness of Metallic Materials.

After the required data is acquired, the project is considered as complete. The result obtained can be used for future work.

1.5 Relevancy of Project

From this project, nitriding treatment as a surface hardening technique can lead to the significance as a scientific reference to heat treatment industries in improving their process design. But then, sensitization might be occur during this process at intermediate temperature around 500°C to 900 °C and can lead to intergranular corrosion. Somehow, this sensitization can be vanished by appropriate methods. Besides that, degree of sensitization (DOS) is also related to hardness and corrosion resistance of materials. Hardness of material will increase while corrosion resistance decrease as the DOS is increasing.

1.6 Feasibility of Project

This project is analyzed to be feasible where the laboratory equipment is all provided in the university. The implementations of the experiments follow the theories, which become the fundamental to complete the project. The allocation of financial cost is sufficient for this project. Moreover, the project is conducted with postgraduate student where this become the medium to share some data and information in order to achieve the project completion successfully. Therefore, author could ensure the feasibility of this project within the given period.

CHAPTER 2

LITERATURE REVIEW

2.1 Stainless Steel

Stainless steels are meant as iron based alloys containing at least 10.5% chromium and a maximum of 1.2% carbon. (Skrabski, 2010). Stainless steel is well known as the best resistance to corrosion in a multitude of different environment. Corrosion resistance is not a natural property of a material, but it is actually come from the reaction between the surrounding medium such as air and the material's surface. Indeed, resistance to corrosion of the stainless steel is due to passive surface film which protects the alloy from the surrounding medium. (Europian Standard EN-10088, 2010). This passive film is formed by the reaction of chromium with oxygen and moisture in the environment and is usually about 2-3 nanometers thickness. It is a non-porous and continuous layer which means if it is broken, it will self-healing under the normal conditions by chromium reacts rapidly with oxygen and moisture in the environment. (Wallen, 1997).

Chromium plays an essential role in the formation of the passive layer. In order to passive layer to occur, minimum 10.5% of chromium is needed and the effectiveness of it is depends on the percentage of chromium present in certain stainless steel. The higher the percentage of chromium, the higher the corrosion resistance. (Europian Standard EN-10088, 2010).

Increasing the chromium from 10.5% to 18% (a typical level of Austenitic Stainless Steel) provides greater corrosion resistance. This is because, the stability of the passive layer is increase. Besides that, the corrosion resistance can be further improved by alloying the steel both with chromium and nickel. (Europian Standard EN-10088, 2010).

Molybdenum as an alloying element to stainless steel acts in a twofold ways. Firstly, it can provides the formation of the passive layer even in small amount and secondly it can stabilizes the passive film in the presence of the halides (chlorides). By having this two function, it can improves the resistance to pitting and crevice corrosion in neutral and acid chloride solution. (Newson, 2008)

2.2 Austenitic Stainless Steel

Austenitic Stainless Steel consist of chromium (16-26%), nickel (8-12%) and iron. (Wallen, 1997). Other alloying elements such as molybdenum may be added or modified according to the desired properties to produce more effective corrosion resistance. The austenitic group contains more grades and are used in greater quantities, than any other category of stainless steel like ferritic and martensitic. (Wallen, 1997). Austenitic grades do not exhibit a yield point. They offer excellent formability and their response to deformation can be controlled by chemical composition. (Newson, 2008). They are not subject to an impact transition at low temperatures and possess high toughness to cryogenic temperatures. They exhibit greater thermal expansion and heat capacity, with lower thermal conductivity than other stainless or conventional steels. They are generally readily welded, but better care is required in the selection of consumables and practices for more highly alloyed grades. Austenitic stainless steels are often described as non-magnetic, but may become slightly magnetic when machined or worked. (Europian Standard EN-10088, 2010)

Austenitic stainless steel is in the face-centered cubic (F.C.C) atomic structure. The formation of this atomic structure is initially from the adding of nickel into the ferrite stainless steel, which is formerly in the atomic structure of body-centered cubic (B.C.C). (Dyson, 2002). The formation of F.C.C atomic structure gives more planes for the flow of dislocations and low level of interstitial elements which provides the austenitic stainless steel good ductility. (Newson, 2008)

In order to distinguish the different between the steel and cast iron, this could be referred on the Iron Carbon Equilibrium Diagram. In the austenitic structure, the carbon contains

are lower which compare to the cast iron. Carbon becomes an essential alloying addition in steel. The increasing of carbon will increase the hardness and strength of the steel. (International Molybdenum Association, 2003). However, as mention before, austenitic stainless steel 316L, which is low carbon content, need to maintain below 0.03%. This is the reason why the inherent austenitic structure is not good in wear and surface hardness because of less carbon content.

Austenitic, or 200 and 300 series, stainless steels have an austenitic crystalline structure, which is a face-centered cubic crystal structure. For 300 series, there are several grades such as Type 316 and Type 304. These grades differ in the physical and chemical properties. However, there is also different in straight grades, L grades, and H grades.

Straight grades define the content of austenitic stainless steel that have maximum of 0.08% carbon. (A to Z of Steel Terminology, 2011). For example Type 316. However, for L grades, for instance Type 316L, this grade indicates low carbon of austenitic stainless steel. The percentage of carbon is maintained maximum up to 0.03%. (Tverberg, 2011). This is because to avoid carbide precipitation or known as sensitization effect. At high temperature, carbon could react with chromium and gather at grain boundaries. This could reduce the formation of passive layer (chromium oxide) as the corrosion resistance to the surface. This is why the amount of carbon needs to be controlled for type 316L. Moreover, the lower carbon content for this type of steel possesses a good weldability because sensitization effect could be prevented at high temperature of welding process (E. Menthe, 1999).

For H grades, the range of carbon content is minimum 0.04% and maximum 0.10%. This grade is used for extreme temperature where the high carbon could give some strength to the stainless steel. The example grade is Type 316H. (Roll, 2011)

2.3 Gas Nitriding Process

Nitriding is a surface hardening technique by the diffusion of nitrogen into the surface layers and the change of chemical compositions of the steel. (G. A. Collins, 1995). There

are several advantages by nitriding treatment, which are to improve mechanical properties, tribological properties and corrosion properties. These improvements are due to the structural changes and high dimension stability by nitriding treatment on the steel. (A. S. Hamdy, 2011)

There are different methods of nitriding process. Salt-bath nitriding is a method with the nitrogen in the cyanide salt as medium for nitriding. (A. Triwiyanto, 2009). While the method of plasma or ion nitriding is involved with diffusion of nitrogen atoms into the metal surface in the presence of plasma environment. (Z. L. Zhang, 2011) and (Liang Wang, 2010) stated that nitriding of austenitic stainless steels by the use of plasma, ion implantation, gas and combination processes already used for many years. The treatment is conducted where the gases accumulate in the furnace and diffuse to the tempered surface product with a gas atmosphere control. The nitriding gas resource that could be used is pure nitrogen, nitrogen/argon gas mixture, nitrogen / hydrogen gas mixture or ammonia gases. There are some advantages from this process where hardness is achieved without the oil, water or air quench. Another advantage is surface hardening is accomplished in a nitrogen atmosphere and could prevents scaling and discoloration. (Z. L. Zhang, 2011). This process also can easily form a hardened layer on the surface of material and it is not expensive as those newly developed technologies.

For this project, author will use ammonia as the gas resource for nitriding. Nitrogen is introduced into the surface steel by holding the metal at a suitable temperature in contact by ammonia. Thus, ammonia will disassociate into gas into hydrogen and nitrogen on the surface steel. Nitrogen then diffuses from the surface into the core of the material at the certain temperature range, which could bring the formation of alloy nitrides in nitrogen diffusion zone. (Mridha, 2006)

In order to accelerate diffusion on austenitic stainless steel, the steel will be treated at relatively intermediate temperature, about 500-900 °C. However, the formation of chromium nitride/carbide could occur during the diffusion at this temperature range. As the results, chromium nitride/carbide might be precipitated into the grain boundary and

the passive layer which is chromium oxide (Cr2O3) will unable to be produced and reduced the corrosion resistance property of stainless steel. This phenomenon is known as sensitization effect.

2.4 Sensitization of Austenitic Stainless Steel

Austenitic stainless steels may become sensitized if they are heat treated or used at temperatures in the range 500 – 900°C. The heat affected zones (HAZ) of welds may also be sensitized in some circumstances.

Chromium carbides form in the grain boundaries of some austenitic stainless steels in the temperature range 500 – 900°C. As the diffusion of chromium (Cr) is slow, it cannot diffuse from the body of the grains to replace the Cr which has gone into the carbides. A lower Cr film along the grain boundary is established. (N. Parvathavarthini, 2010). The grain boundary has lower corrosion resistance, and may be attacked in an environment the steel would normally resist. The steel is said to be sensitized, and is susceptible to intergranular corrosion (also called grain boundary attack). A complete steel component may be affected after service or heat treatment in the critical temperature range, or part of the heat affected zone of a weld may suffer the problem. (Skrabski, 2010)

This classical explanation of sensitization is based on the precipitation of carbides and nitrides. Nitrides and carbonitrides also form, and the nitrogen content of the steel must be taken into account. The susceptibility of the steel is related to the sum of the carbon and nitrogen contents. (P. Atanda, 2010). The figures below are the examples of chromium depleted zone, intergranular corrosion, unsensitized and highly sensitized stainless steels and they are courtesy from (Skrabski, 2010)

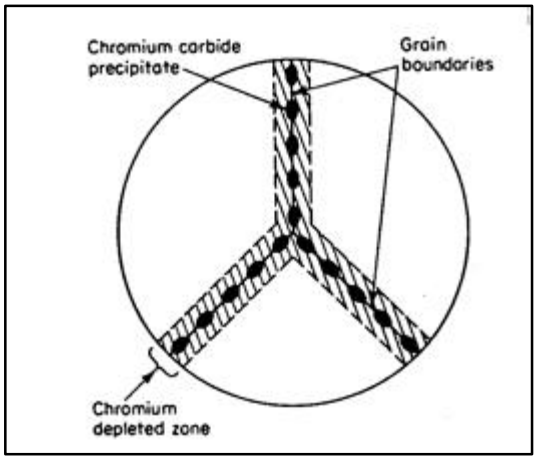


Figure 1: Chromium Depleted Zone

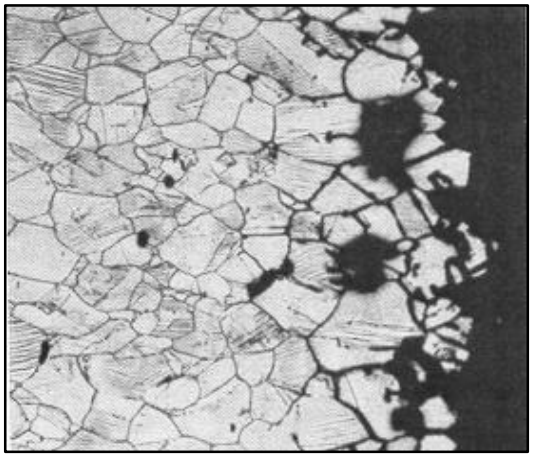


Figure 2: Parent Plate of Intergranular Corrosion.

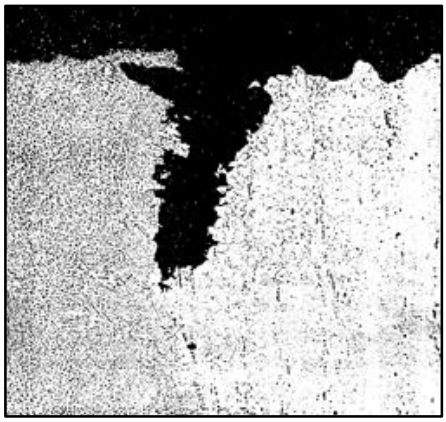


Figure 3: Heat Affected Zone of Intergranular Corrosion.

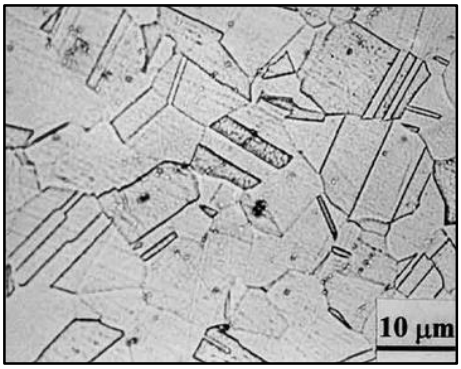


Figure 4: Unsensitized Stainless Steel

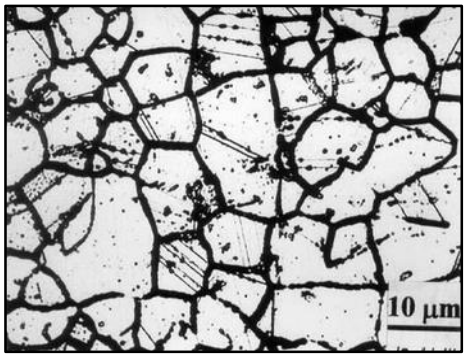


Figure 5: Highly Sensitized Stainless Steel

CHAPTER 3

METHODOLOGY

3.1 Introduction

In this chapter, the methodology on how this project will be conducted is explained in section 3.2. Sample preparation is discussed in section 3.3 and how the sample being treated with intermediate temperature gas nitriding is presented in section 3.4. Section 3.5, 3.6 and 3.7 will explain the regarding X-ray Diffraction (XRD) analysis, Microstructure Analysis and Vickers Microhardness Test respectively. Meanwhile, 3.8, 3.9.3.10 will give the details about overall project flow which are Project Flow Chart, Gantt Chart for FYP 1 and 2, and Key Milestone accordingly.

3.2 Research Methodology

In this study, AISI 316L austenitic stainless steel is used as material for analysis. This material will undergo intermediate temperature gas nitriding treatment in 3 different treatment temperature. Temperature of treatment varies at 600, 700, and 800^{o}C. In this experiment, 100% of nitrogen gas will diffuse in thermal activated environment. For layer and surface morphology region assessment of untreated and nitrided samples, Optical Microscope (OM) and Scanning Electron Microscope (SEM) instrument will be utilized while for crystallographic structure will be tested by X-ray Diffraction (XRD). This experiment will be such a loss without Energy Dispersive X-ray (EDX) analysis to check on the chemical composition of the samples at various spots. Apart from that, Vickers Microhardness Test (HV) will be used to determine the hardness at the cross section of

the samples. The flow chart in Figure 6, which is the iterative process of the experiment will be described accordingly. Data analysis and final result section are discussed later in the next chapter.

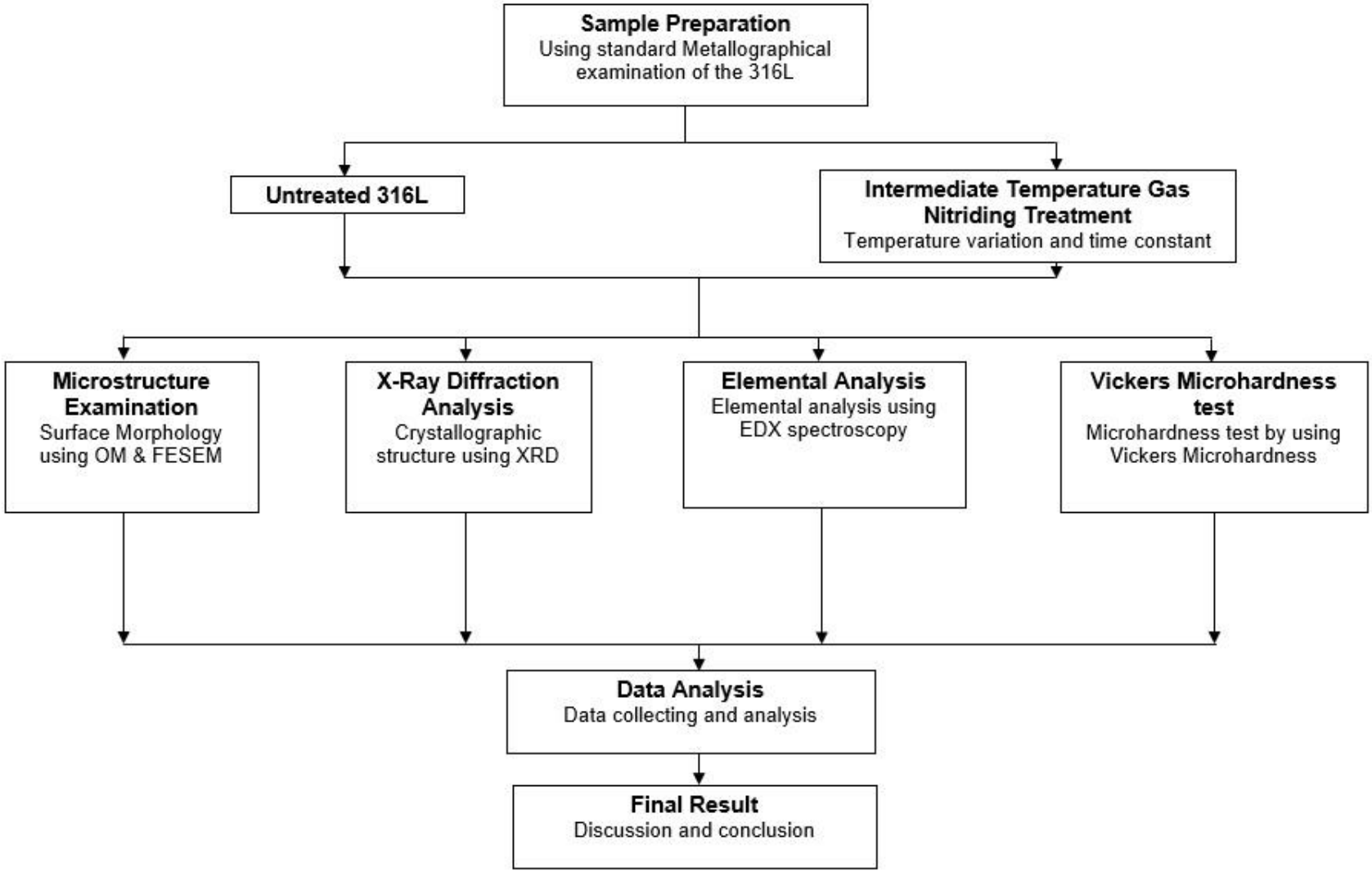


Figure 6: Methodology Flow Chart

3.3 Sample Preparation

The material that will be used is AISI 316L stainless steel and the steel supplied in the form of plate with thickness of 2mm. The plate samples will be altered by abrasive cutter into the dimension exactly 15mm x 15mm each for about 24 samples. This is because 6 samples will be used in each temperature which are 600°C, 700°C and 800°C and 6 for untreated samples. All these samples are needed in this entire project in order to complete the microstructure study, hardness test, EDX and XRD. Then all the samples surface are grind on 120, 500, 800, 1000, 1200 grit SiC papers, and then polished using 1 μm Al2O3 pastes to the mirror finish

3.4 Intermediate Temperature Gas Nitriding Process

After sample preparation is completed, all the samples are cleaned using ultrasonic cleaning to remove unwanted contaminants. Nitriding process will directly follow from ASTM A355 standard specifications. The samples that will undergo nitriding treatment will be immersed in HCl (2 M) solution for 15 minutes duration to remove the protective oxide film that commonly forms on austenitic stainless steel.

Then, the sample is quickly placed on the quartz and put in the horizontal tube located in the Carbolite CTF Tube Furnace for the nitriding process. Then the cycle of nitriding is set through the furnace controller. The target temperature is set to be 600°C from the room temperature with the rate of 5°C/min. Nitrogen will be purged into the furnace thus maintain the environment inert, where the oxidation of samples and furnace component can be prevented. The same steps will be repeated for 700°C and 800ºC (temperature variation of treatment).

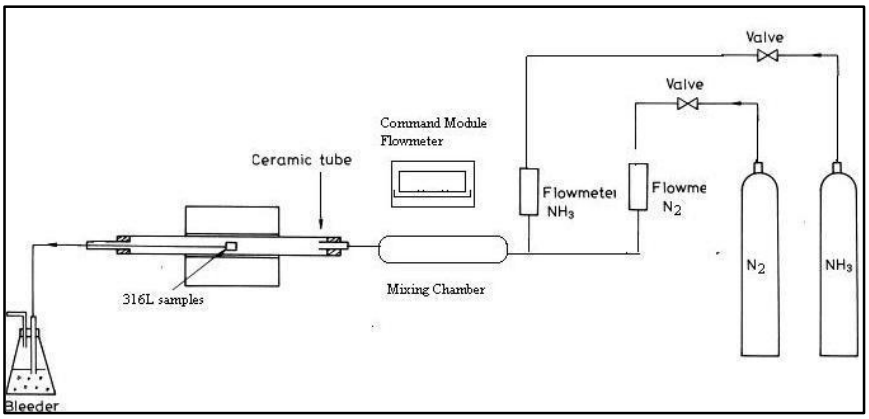


Figure 7: Schematic Illustration on Nitriding Equipment Setup

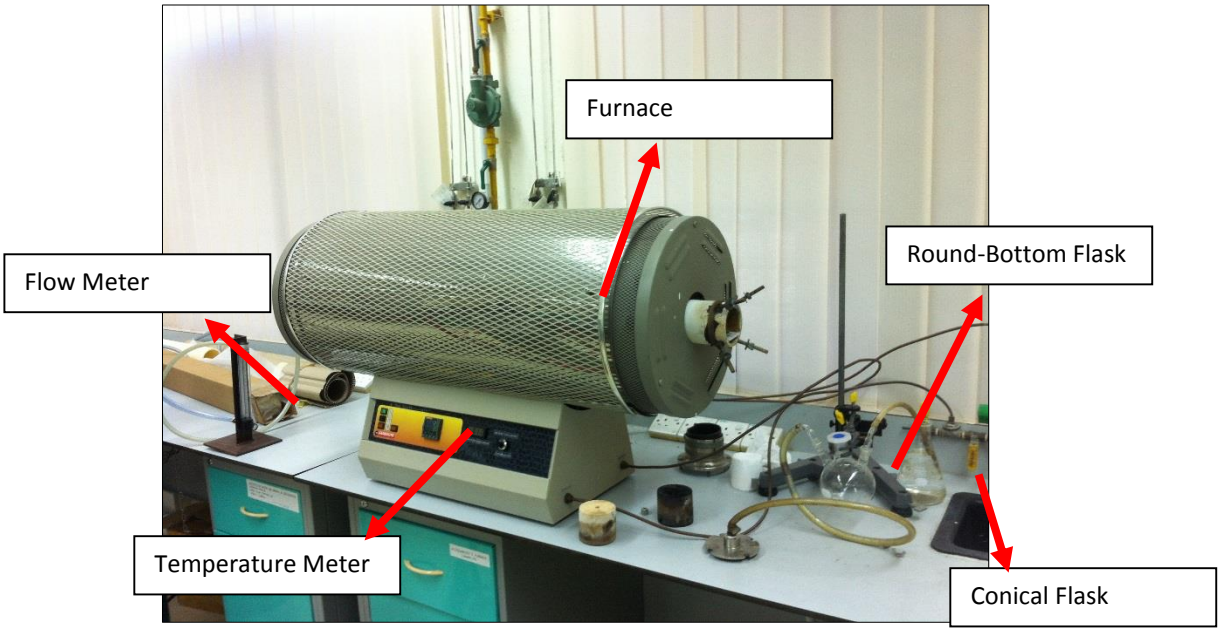


Figure 8: Nitriding Equipment Setup at Laboratory.

The amount of nitrogen gas is set to be at 100% each. This amount is obtained by setting up the Command Module for flow meter nitrogen gas. After 30 minutes, the flow meter is reduced to about 90 and 80. This is due to ensure all the atmosphere inside the furnace is 100% filled with nitrogen gas and prevent oxygen from diffusing into the furnace.

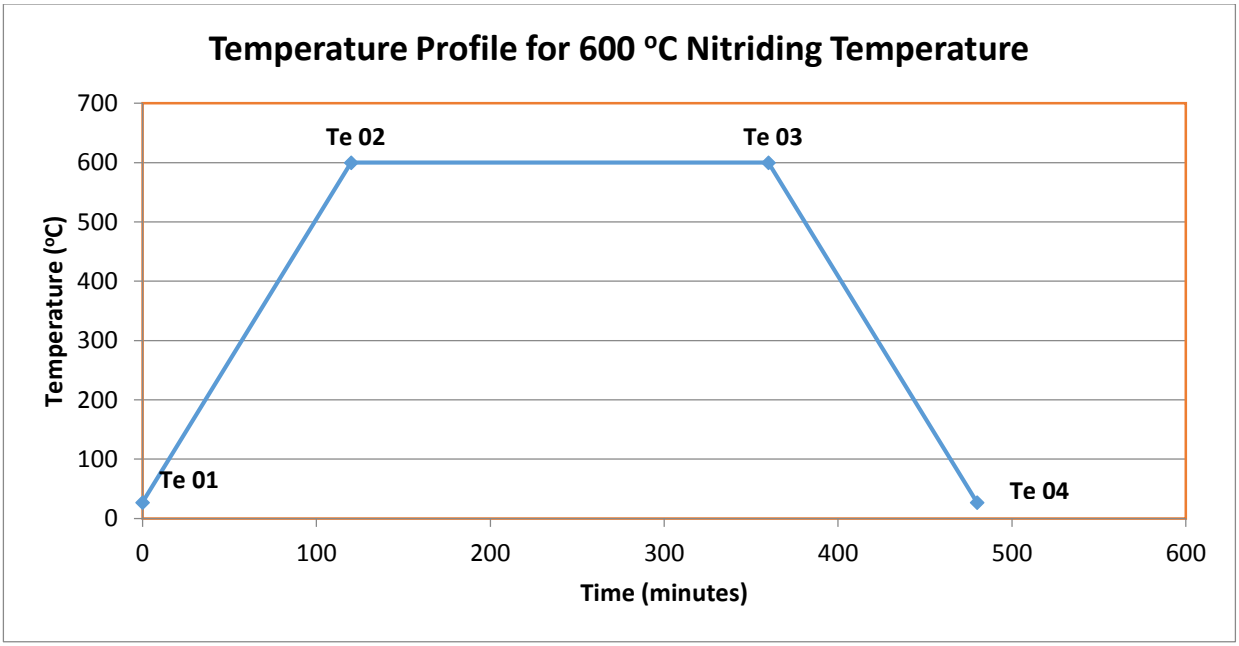


Figure 9: Temperature Profile for 600C

Table 1: Temperature Profile Details

Segment	**Details**
Temperature 01 (Te 01)	Initial Room Temperature. It can be leaved as '27°C'
Temperature 02 (Te 02)	Ramping time to reach 600°C which is 120 minutes as it increase 5°C/min
Temperature 03 (Te 03)	Holding time at 600°C for 240 mins
Temperature 04 (Te 04)	The furnace will be cool down to room temperature within 480 minutes of overall process

3.5 X-Ray Diffraction Analysis

One of the powerful and utilized non-destructive technique for characterizing crystalline materials is X-Ray Diffraction (XRD). It provides information on structures, phase, preferred crystal orientation (texture), and other structural parameters, such as average grain size, crystallanity, strain, and crystal defects. X-ray diffraction peaks are produced by constructive interference of a monochromatic beam of x-ray scattered at specific angles from each set of lattice planes in a sample. The peak intensities are determined by the distribution of atoms within the lattice. Consequently, the x-ray diffraction pattern is the fingerprint of periodic atomic arrangement in a given material. An investigation of standard database of x-ray patterns enables quick phase identification for a large variety of crystalline samples.

In the present investigation of nitriding austenitic stainless steel, the purpose of X-Ray Diffraction analysis is to determine the formation of alloying nitride subsequent to nitriding process. The X-Ray Diffraction machine that will be used for this project is located at Block 17.

3.6 Microstructure Analysis

The purpose of this test is to determine the formation of chromium nitride at the grain boundary. This test will be conducted using Optical Microscope (OM) and Scanning Electron Microscope (SEM). Before conducting the microstructure examination, the samples need to be mounted, grinded, polished and etched. Hot mounting of specimen is required so that it is more convenient during grinding and polishing. Specimen will be mounted in a resin. Bakelite is commonly used. A further advantage of that the edges of the specimen will be reasonably well polished and not “beveled” by the preparation process. After mounting, the samples need to be grinded and polished.

Then, the samples need to be etched by marbles reagent as the etchant. The purpose of etching is to reveal the grain boundary. Firstly, the specimen will be washed by using ethanol and then dried. After that, marbles reagent will be applied at the surface of the

specimen for 5 to 10 seconds. Then the specimen needs to be washed using water and ethanol. The specimen will be dried and ready for microstructure examination. Detail steps of microstructure analysis are explained in chapter 3.6.1 which is Metallography Method. Chapter 3.6.2 and 3.6.3 will discuss about the Optical Microscope (OM) and Scanning Electron Microscope (SEM) accordingly.

3.6.1 Metallography Method

A lot of steps will be conducted in order to complete this project. The apparatus and materials are ensured available in the laboratory and can perform well. Metallography is the study of a materials microstructure. Analysis of a materials microstructure aids in determining if the material has been processed correctly and is therefore a critical step for determining product reliability and for determining why a material failed. The basic steps for proper metallographic specimen preparation include: sectioning and cutting, mounting, planar grinding, rough polishing, final polishing, etching, microscopic analysis, and hardness testing. (P. Atanda, 2010)

1. **Sectioning and Cutting**

Most metallographic samples need to be sectioned to the area of interest and for easier handling. Depending upon the material, the sectioning operation can be obtained by abrasive cutting for the 316L Autenitic Stainless Steel. Proper sectioning is required to minimize damage, which may alter the microstructure and produce false metallographic characterization. During cutting process, correct selection of abrasive type, bonding, and size as well as proper cutting speed, load and coolant are very important.

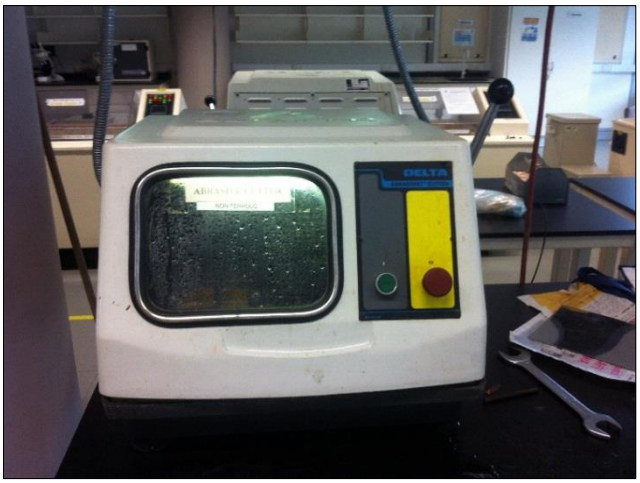


Figure 10: Outer Part of Abrasive Cutter Machine

Figure 11: Inner Part of Abrasive Cutter Machine

2. Mounting and Grinding

For the mounting purpose, each specimen is placed on the thermosetting plastic. By doing this, grinding and polishing process will be much easier as the specimens is static and not keep moving. Silicon carbide paper is used for the grinding process and flowing water is flushed to the paper as the lubricating and serve to remove away the coarse emery particles, which can possibly scratch the specimen. For the proper and clean grinding of the specimen, 120, 500, 800, 1000 and 1200 grits of silicon carbide paper are used and grinding must be done in correct order of grades.

Figure 12: Hot Mounting Press Machine

3. Polishing

Polishing cloth using alumina powder dissolved in water at a reasonable proportion is used for the polishing step. The grade for the alumina powder is about 1 micron and 0.3 micron. During the process, light pressure is applied until the surface is free from scratches. Then, the specimen is examined by using microscope with 50 to 100 magnification to ensure it is free from scratches. This examined step must be done after the specimen is cleaned and dried.

Figure 13: Grinding and Polishing Machine

4. Etching

The specimen is then etched with 10 grams $CuSO_4$ + 50ml of HCL + 50ml of H_2O also known as marbles reagent. It is washed free from any adhering polishing compound and plunged into the etching solution and igitated vigorously for 3 minutes. Then, it is quickly transferred to running water on order to wash away the etchant as fast as possible. After been washed by distilled water, it is then heated to remove the water molecules at the surface of the samples. Then, it is examined by naked eye to check what extent etching has taken place. After that, the specimen is observed under the optical microscope and photomicrographs taken.

Table 2: Marble's Reagent Etchant

Etchant	Composition	Conc.	Conditions
Marble's Reagent	Copper (II) sulphate,CuSO4	10 grams	Immerse or swab for 5-60 seconds.
	Hydrochloric acid	50 ml	
	Water	50 ml	

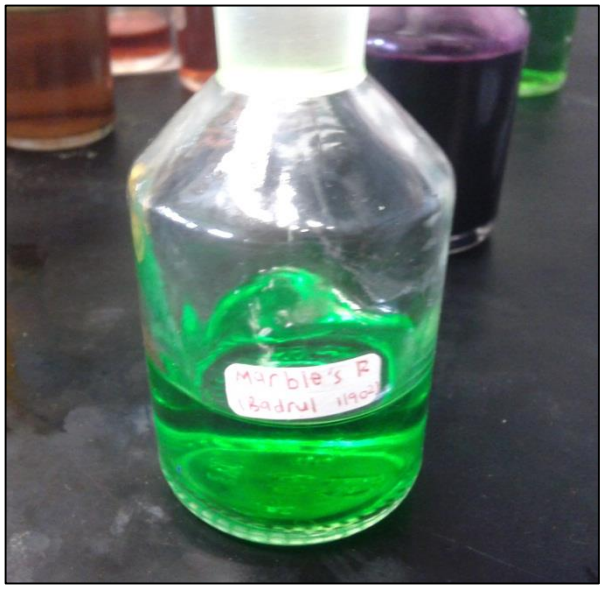


Figure 14: Marble's Reagant Solution

Figure 15: Sample Heater

3.6.2 Optical Microscope (OM)

Optical Microscope can be used in various applications and they are very powerful instrument to determine the microstructure of a great range of materials. There are some guidelines as to how to set up a specimen to be observed:

1. The specimen is mounted and placed on the stage and begin increasing the power of the light source slowly until a bright spot visible on the sample. This step should be done without looking down the eye piece.
2. With the lowest magnification lens in place focus using the coarse focus knob without looking down the microscope. Lower the objective lens close to the specimen surface, and then use the coarse focus knob to raise it until the circle of light on the specimen appears reasonably sharp. Then, looking through the eyepiece, adjust the coarse focus control. When looking down the eyepiece always use coarse focus so as to move the sample away from the objective.
3. The eyepiece distance should be adjusted to a comfortable separation and looking through the eye pieces, use the fine focus knob to bring the image to a sharp focus.

4. The image should be focused to the non-adjustable eyepiece and then the other changed such that it is also in focus.
5. To increase the magnification, slide the rotatable nosepiece around but must ensure the lens does not touch the specimen and then re-focus using the fine.
6. Once a representative area is found, and focused a digital camera can be used to take a photo and a sketch can be made.

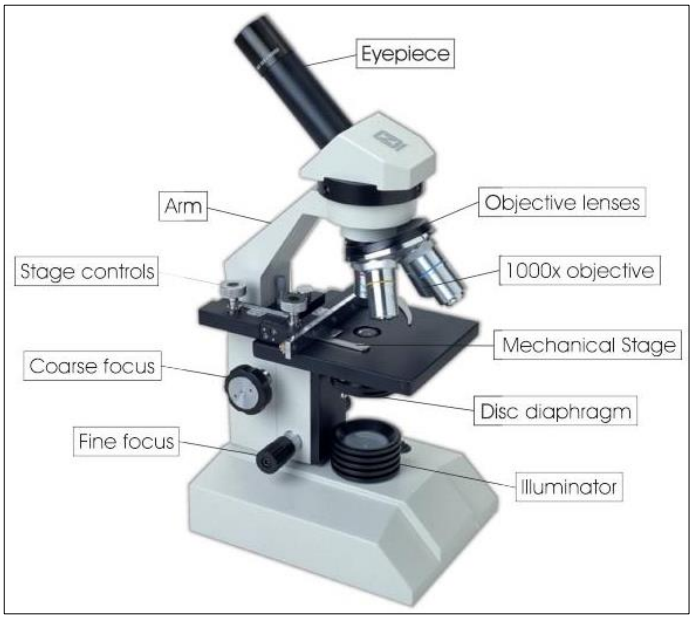


Figure 16: Optical Microscope (OM)

3.6.3 Scanning Electron Microscope (SEM)

Figure 17: Scanning Electron Microscope (SEM)

Equipment Details

Manufacturer	Carl Zeiss AG, Germany
Model	SUPRA 55VP
Acceleration Voltage	0.1-30kV
Resolution	4nm at 0.1kV 0.8nm at 30kV
Probe Current	1pA-10nA
Magnification	12-900000 X
Working Distance	1mm-50mm
VP Mode	2pa to 133pa
Signal	InLens, SE2, VPSE, and AsB

3.7 Vickers Microhardness Test

Hardness test is done to compare the difference in hardness between as received austenitic stainless steel and the nitrided one. For this testing, Vickers Microhardness Test is used. First, a preliminary test force is applied to the samples using a diamond indenter to represent the zero or reference position that breaks through the surface to reduce the effects of surface finish. Then, the major load is applied to reach the total 300gf load. This force is held for about 15 seconds to allow the elastic recovery. The indentation depth variance between preload and major load is then converted to hardness number.

Figure 18: Vickers Microhardness Test

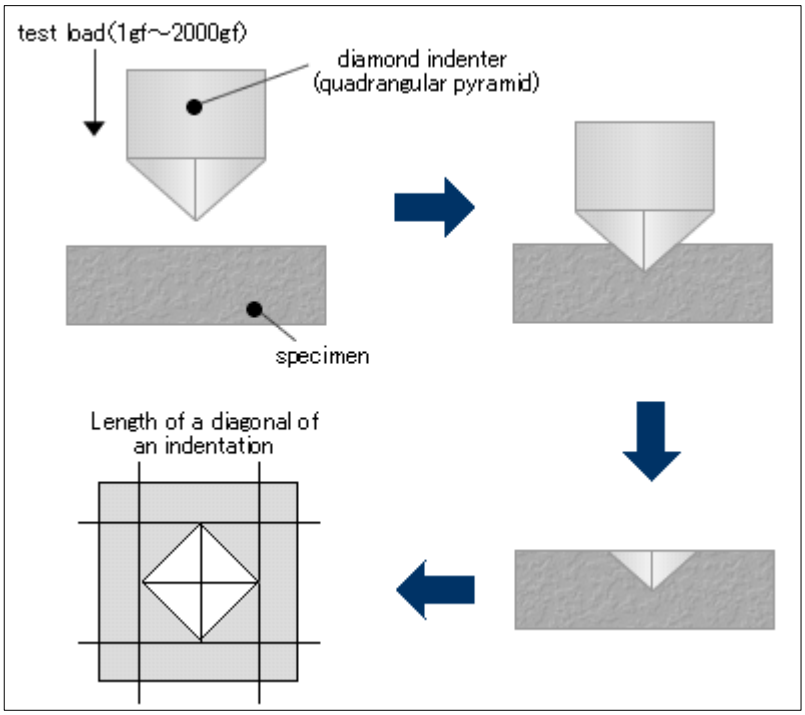
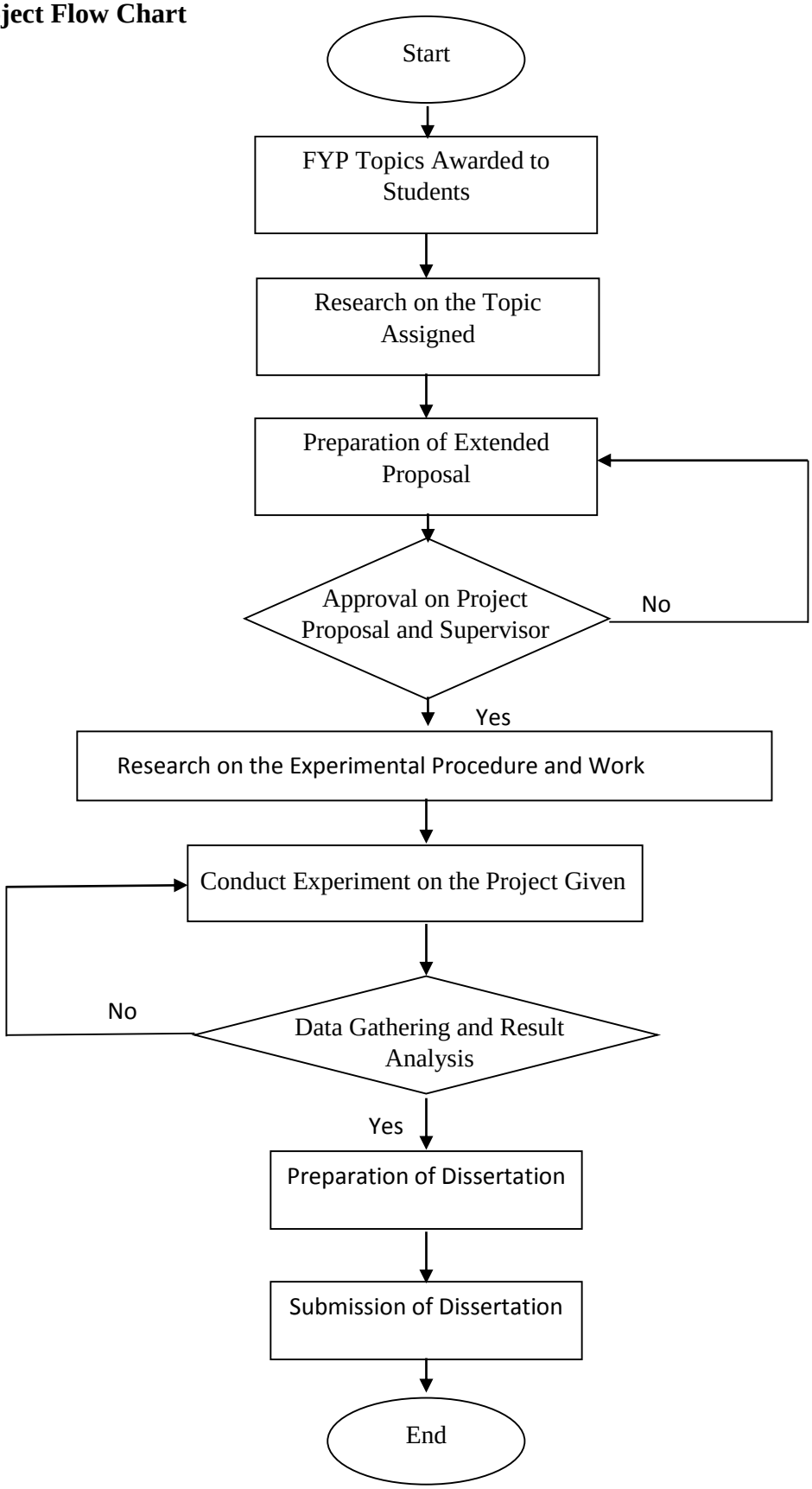


Figure 19: Testing Method

3.8 Project Flow Chart



3.9 Gantt Chart

Table 3: Gantt Chart FYP1

No	Details	Weeks													
		1	2	3	4	5	6	7	8	9	10	11	12	13	14
1	Consolidation of FYP Topics														
2	Topic assignments to students														
3	Research for the topic assigned														
3	Preparation for Extended Proposal														
4	Submission of Extended Proposal														
5	Research for the experimental procedure and work														
6	Research for the material properties														
7	Preparation for Proposal Defense														
8	Proposal Defense														
9	Preparation for Interim Report														
10	Submission of Interim Report														

Table 4: Gantt Chart FYP 2

Training Activities	Week													
	1	2	3	4	5	6	7	8	9	10	11	12	13	14
Arranging Furnace														
Nitriding Process														
Microstructure Examination														
Chemical Composition Examination														
Surface Hardening Test														
Corrosion Test														
Submission of Progress Report														
Submission of Draft Report												❖		
Oral Presentation													❖	
Submission of Technical Paper														❖
Submission of Dissertation														❖

3.10 Key Milestones

Table 5: Key Milestones for FYP 1 and 2

No.	Milestone	Date
1	Submission of Extended Proposals	3 July 2013 (Week 7)
2	Proposal Defense	15-21 July 2013 (Week 9)
3	Laboratory Equipment Familiarization	August 2013
4	Laboratory Activities	August 2013
5	Submission Interim Report	19-25 August 2013 (Week 14)
6	Submission of Progress Report	Week 8
7	Submission of Draft Report	Week 12
8	Oral Presentation	Week 13
9	Submission of Technical Paper	Week 14
10	Submission of Dissertation	Week 14

CHAPTER 4

RESULT AND DISCUSSION

4.1 Layer Morphology

A lot of results were obtained from the difference of intermediate temperature gas nitriding treatment. These results should be interpreted well in order to get the correct information. Figures below show the images of the microstructure from Optical Microscope.

Figure 20 shows image of untreated samples at 100x magnification. The grains and grain boundaries can be seen clearly in this image. The dark region represents the present of the chromium. For this untreated sample, the thickness of the grain boundaries is thin and that means the chromium is distributed equally and precipitation of chromium nitride does not occur. The size of the grains is in range 5-15 µm. Microstructure image of nitrided 316L stainless steel at 600°C is shown by Figure 21. This image is taken at 500x and it shows the difference of grain boundaries between untreated and nitrided stainless steel. For the analysis, the thickness difference of the grain boundaries is due to sensitization is occurred for the nitrided stainless steel. As sensitization happened, the chromium is unequally distributed and some of it is react with nitrogen supplied through the nitriding process. As the chromium is react with nitrogen, chromium nitride produced and precipitate at the grain boundaries. That explains why there is differences in grain boundaries between these 2 samples. Several black spots in this image is formed due to over-etched during the sample preparation.

Figure 22, 23, and 24 show the microstructure images of nitrided 700°C at 500x, nitrided 800°C at 100x and nitrided 800°C at 500x magnification respectively. Figure 22 and 24

show the difference in term of grain boundaries. Nitrided 800°C having thicker grain boundaries compares to nitrided 700°C sample. This is because higher temperature allows more rapid nitrogen diffusion into the samples compares to lower temperature. Figure 23 clearly shows that concentration of black region is higher at the surface and decrease as the depth profile increase. It is mean that the rate of nitrogen diffusion is faster and rapid at the surface compares to its subsurface because nitrogen cannot diffuse much further into the cross section. All in all, diffusion of nitrogen is much higher at the surface and slowing down as it goes deeper along the cross section. It is due to pressure applied during the nitriding process cannot push nitrogen any further into the cross section of the ASS and diffusion of nitrogen is limited.

Figure 22 and 24 below clearly show the formation of twins at the microstructure. Twinning is generally considered as a deformation mechanism that is activated at high strain rates which the critical resolved shear stress for dislocation slip is high. In low Stacking Fault Energy (SFE) which is in Face Centered Cubic (FCC) structure typically observed after the activation of multiple slips system (Dyson, 2002). There are two types of twinning which are suspended and transgranular twin. In this microstructure analysis, it is transgranular twin because it has 4 parts and they are 2 sides are coherent twin planes and 2 ends are the grain boundaries. It is easier to make stacking sequence stagger thus will result on formation of stacking faults when austenite grows by migration of high angle grain boundary during the nitriding process (Newson, 2008).

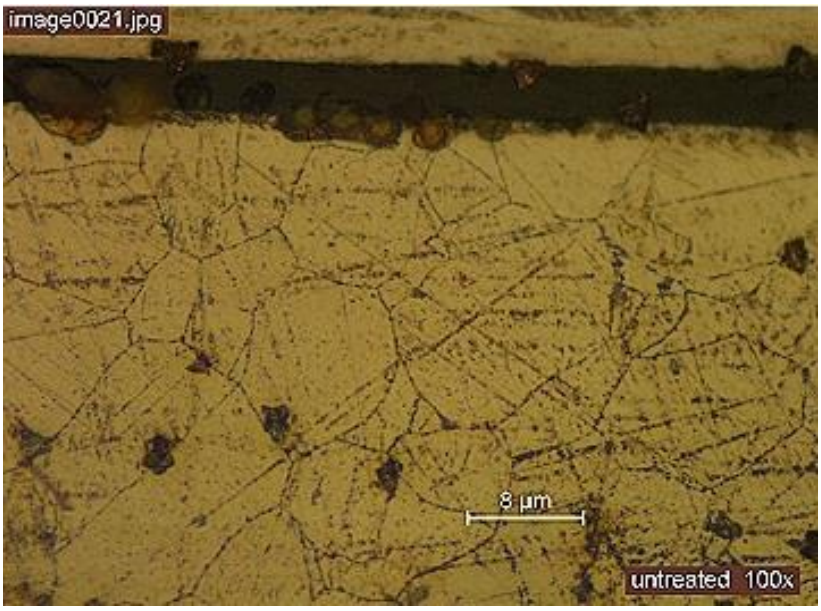


Figure 20: Optical Microscopic of Untreated Sample at 100x

Figure 21: Optical Microscopic of 600°C Nitrided 316L ASS at 500x

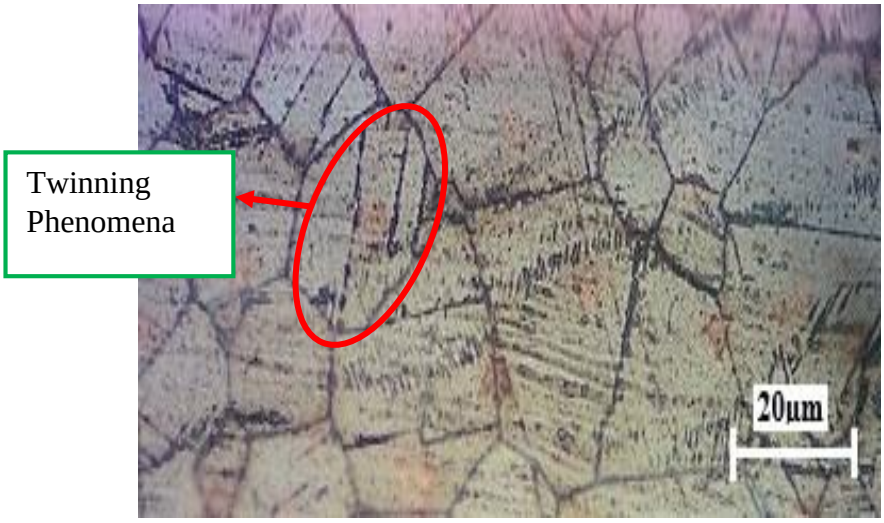


Figure 22: Optical Microscopic of 700°C Nitrided 316L ASS at 500x

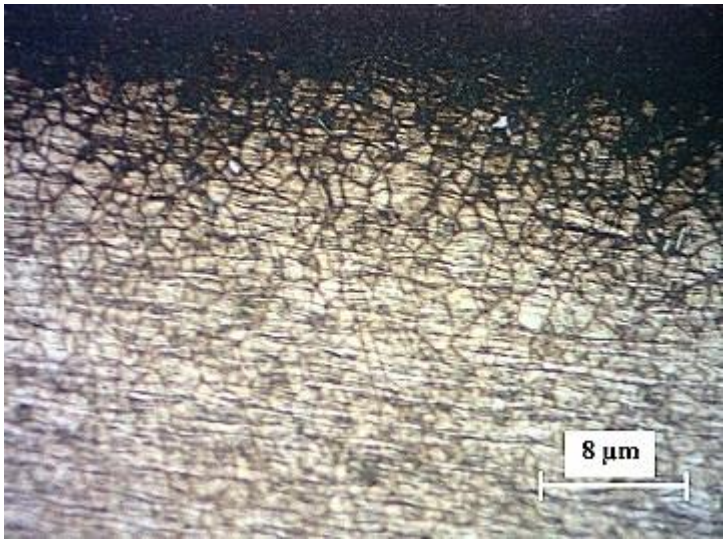


Figure 23: Optical Microscopic of 800°C Nitrided 316L ASS at 100x

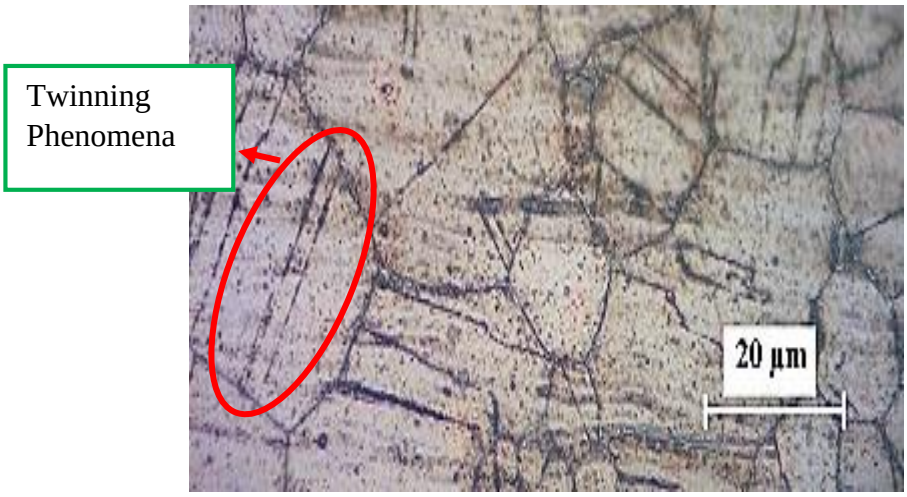


Figure 24: Optical Microscopic of 800°C Nitrided 316L ASS at 500x

4.2 Scanning Electron Microscope (SEM)

Figure 25 to 28 show the result of Scanning Electron Microscope for untreated, 600, 700, and 800°C nitrided of 316L Austenitic Stainless Steel respectively. These results were obtained after 2 hours of utilizing the SEM. Figure 25 and 26 is different is term of the grain boundary as Figure 26 shown the chromium nitride precipitation whereas Figure 25 did not. Figure 25 is clear from any white precipitate at the grain boundary as white precipitate represents the chromium nitride precipitation. The size of grain boundary of Figure 26 is larger than compare to Figure 25. Besides that, both of this figures clearly show the twinning phenomena that always appear in Austenitic Stainless Steel.

Figure 26 and 28 show the difference in term of chromium nitride precipitate concentration as Figure 28 is higher than 26. Chromium nitride precipitate at all over the grain boundaries for 800°C nitrided 316L Austenitic Stainless Steel. This difference is much related to nitriding temperature for each ASS. Higher temperature will provide high nitrogen diffusion into the microstructure thus higher chromium nitride precipitate will occur.

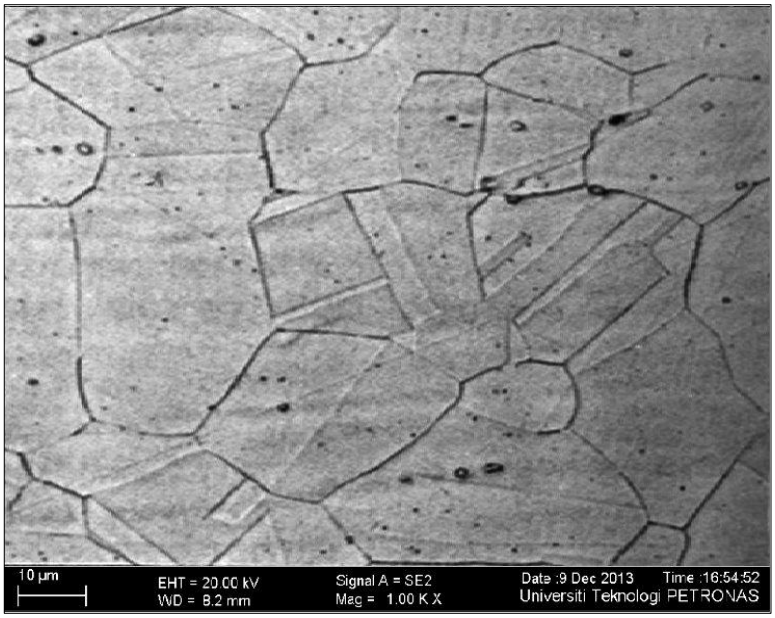


Figure 25: SEM Image of Untreatred 316L ASS

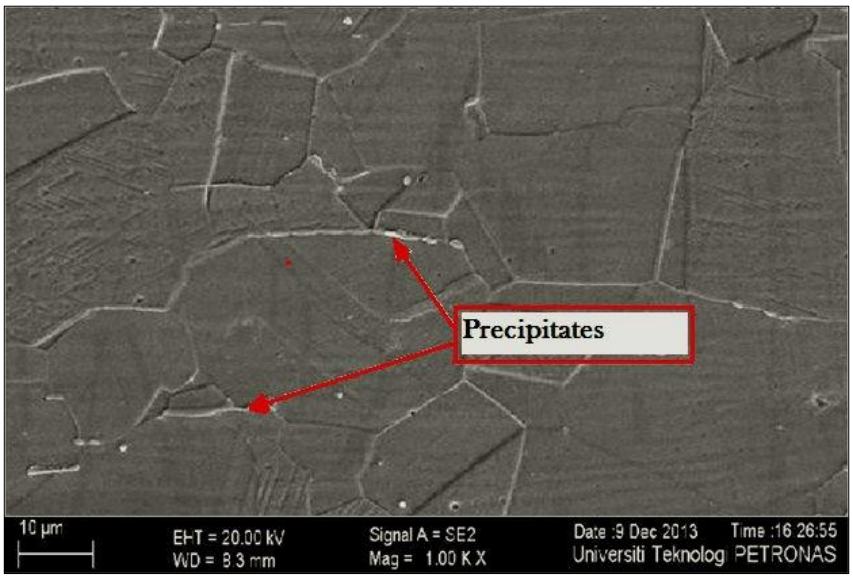


Figure 26: SEM Image of 600°C Nitrided 316L ASS

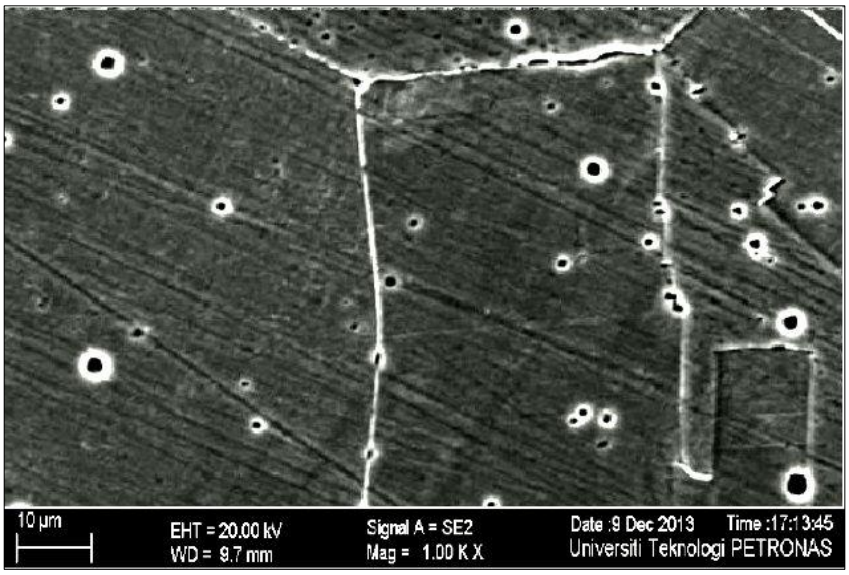


Figure 27: SEM Image of 700°C Nitrided 316L ASS

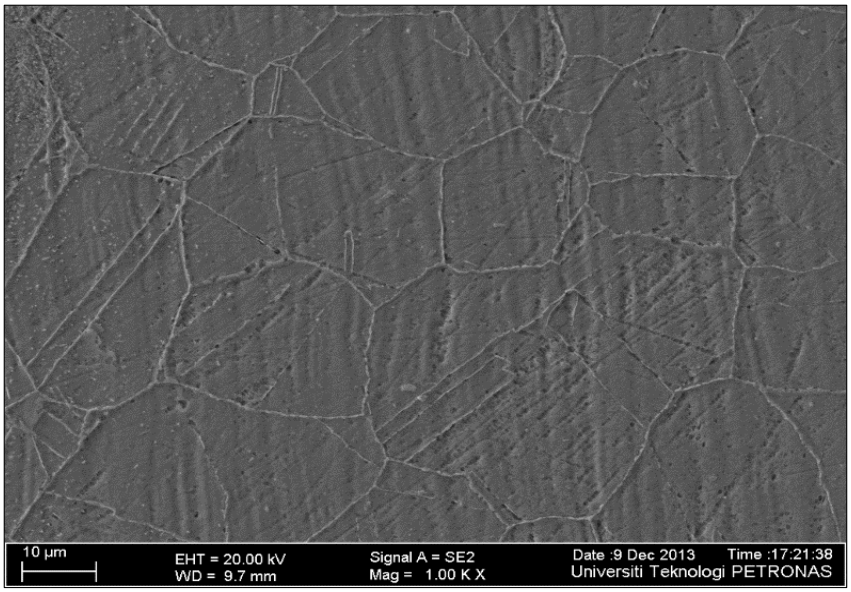


Figure 28: SEM Image of 800°C Nitrided 316L ASS

4.3 Electron Dispersive X-Ray (EDX)

Figure 29 shows the Electron Dispersive X-Ray (EDX) on 800°C nitrided 316L Austenitic Stainless Steel. There are 6 spectrums for the EDX analysis, 3 spectrums at grain boundaries and other 3 spectrums at the grain. This EDX analysis is done to determine the chemical composition at the microstructure of 316L ASS after the nitridng process and find out the difference between the Chromium, Nitrogen, and Carbon at both areas. This figure represents for all samples which are untreated sample, 600°C and 700°C nitrided ASS on how 6 spectrums are specified.

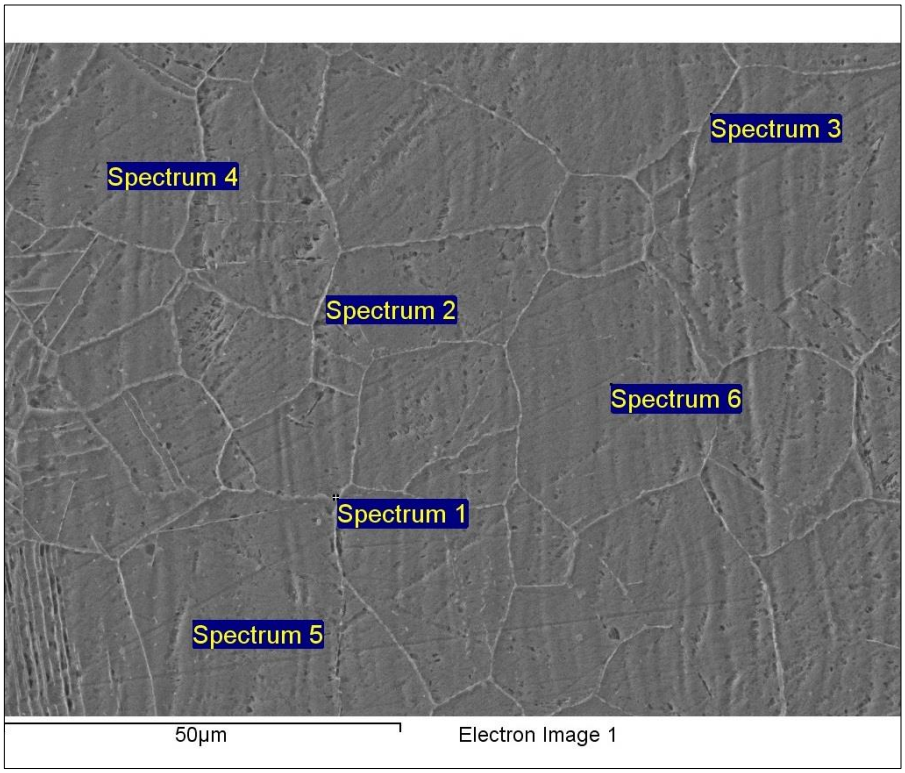


Figure 29: EDX Analysis on Chemical Composition at 6 Spectrums for 800C Nitrided 316L ASS

Table 6 shows the composition of the element in weight (%) at respective spots for the untreated sample. While table 7, 8 and 9 show the composition of the element in weight (%) for 600°C, 700°C, and 800°C nitrided 316L ASS. Spectrum 1, 2 and 3 represent the composition at the grain boundaries of the samples, and spectrum 4, 5, and 6 represent the composition at the grain. Analysis have been made on these 600°C, 700°C, and 800°C tables and found out that the composition of the Carbon (C), Nitrogen (N), and Chromium (Cr) is the greater at the grain boundaries compared to the grain area. Composition difference between grain boundaries and grain area can be clearly seen at Nitrogen composition at any nitriding temperature. For example, for 800°C sample, the first 3 spectrums give 2.73, 2.81, and 2.93 and give an average about 2.82. However, the last 3 spectrums give 1.04, 1.03, and 1.20, thus 1.09 is the average. This composition difference is about 61.24% with respect to the higher composition. This difference is due to the precipitation phenomena which is the chromium, carbon, and nitrogen are precipitated at the grain boundaries. High concentration of chromium nitride decrease locally the chromium content in the region that is adjacent to these chromium rich precipitates. Since the chromium diffuses much slowly than carbon, these is not enough time for the chromium to diffuse to the nitride from all over the grains, so, in the region that is near to grain boundaries, chromium content will be lower the theoretical composition.

Besides that, as the nitridng temperature is getting higher, the composition of Carbon (C), Nitrogen (N), and Chromium (Cr) also keep increasing at both area which are grain boundaries and grain. For instance, for the Nitrogen composition at 600°C at the grain boundaries are 1.19, 2.51 and 1.20 while for 800°C are 2.73, 2.81, and 2.93. 0.10, 0.10, and 0.14 are the value of the grain area for 600°C and 1.04, 1.03, and 1.20 are for 800°C. This composition difference is related to the nitriding temperature because the higher nitriding temperature, the higher the rate of nitrogen diffusion into the stainless steel. At higher temperature nitrogen will be more vigorous and diffuse faster into the stainless steel.

1) Untreated Sample

Table 6: Weight Percentage of Chemical Composition Based on EDX Analysis for Untreated Sample

Elements		C	Cr	Mn	Fe	Ni
Weight (%)	Spectrum 1	6.34	17.93	0.83	67.58	7.32
	Spectrum 2	5.97	17.67	1.11	67.69	7.56

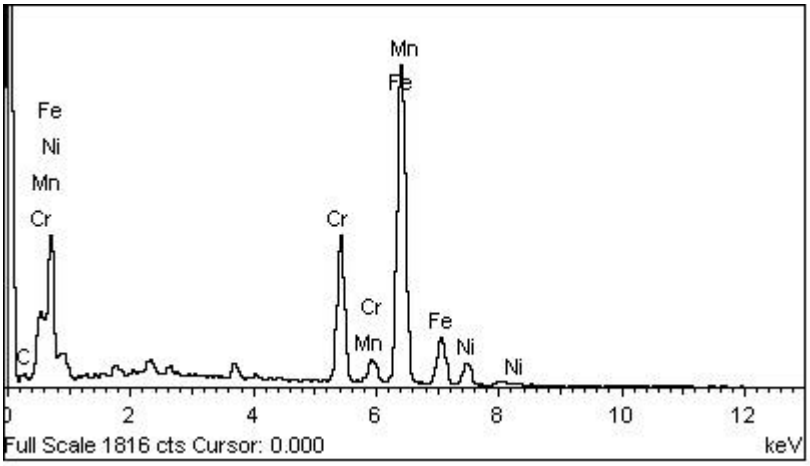


Figure 30: Graph of EDX Analysis on Chemical Composition for Untreated Sample

2) 600°C Nitrided 316L ASS

Table 7: Weight Percentage of Chemical Composition at 6 Spectrums Based on EDX Analysis for 600°C Nitrided 316L ASS

Elements		C	N	Cr	Mn	Fe	Ni
Weight (%)	Spectrum 1	6.38	1.19	17.05	0.73	66.23	8.42
	Spectrum 2	6.30	1.51	17.74	0.98	66.11	7.37
	Spectrum 3	6.58	1.20	17.21	0.87	66.18	7.96
	Spectrum 4	3.11	0.10	18.12	1.63	69.52	7.62
	Spectrum 5	5.87	0.10	17.50	1.16	65.49	9.89
	Spectrum 6	5.94	0.14	17.90	1.19	67.40	7.43

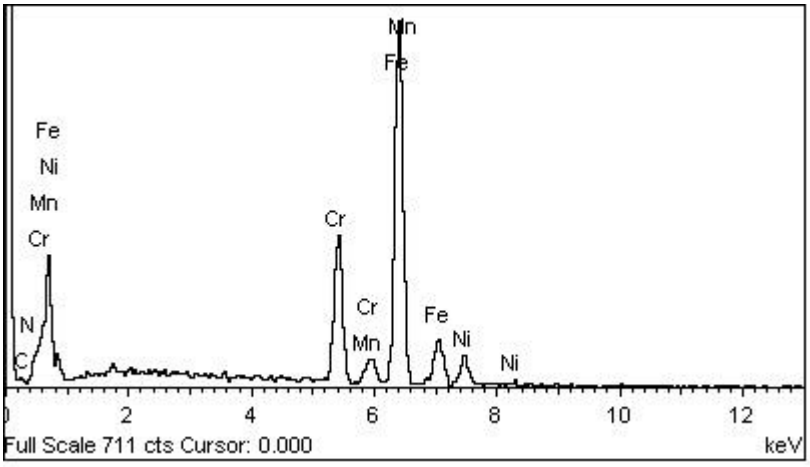


Figure 31: Graph of EDX Analysis on Chemical Composition for 600°C Nitrided 316L ASS Sample at Grain Boundary (Chromium Nitride Precipitation)

3) 700°C Nitrided 316L ASS

Table 8: Weight Percentage of Chemical Composition at 6 Spectrums Based on EDX Analysis for 700°C Nitrided 316L ASS

Elements		C	N	Cr	Mn	Fe	Ni
Weight (%)	Spectrum 1	8.93	1.93	16.25	1.50	64.89	6.49
	Spectrum 2	4.29	1.81	19.05	0.73	67.17	6.95
	Spectrum 3	5.06	2.05	18.63	0.72	66.39	7.16
	Spectrum 4	6.31	1.08	16.80	0.84	67.24	7.73
	Spectrum 5	4.54	0.98	18.76	1.44	66.64	7.63
	Spectrum 6	6.82	0.33	18.11	1.18	66.49	7.06

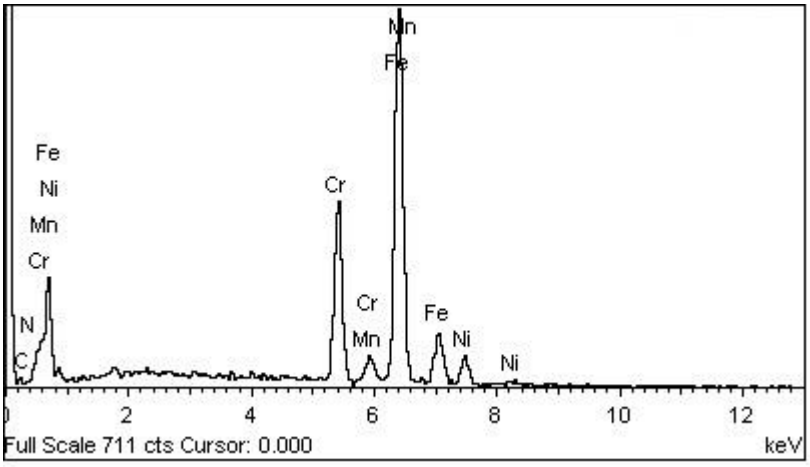


Figure 32: Graph of EDX Analysis on Chemical Composition for 700°C Nitrided 316L ASS Sample at Grain Boundary (Chromium Nitride Precipitation)

4) 800°C Nitrided 316L ASS

Table 9: Weight Percentage of Chemical Composition at 6 Spectrums Based on EDX Analysis for 800°C Nitrided 316L ASS

Elements		C	N	Cr	Mn	Fe	Ni
Weight (%)	Spectrum 1	6.34	2.73	17.81	1.42	64.19	7.51
	Spectrum 2	9.70	2.81	16.02	0.89	64.17	6.41
	Spectrum 3	5.74	2.93	16.69	1.27	66.31	7.07
	Spectrum 4	4.81	1.04	17.70	1.20	68.68	6.58
	Spectrum 5	10.07	1.03	33.09	1.18	49.73	4.90
	Spectrum 6	6.38	1.20	16.96	1.35	66.63	7.48

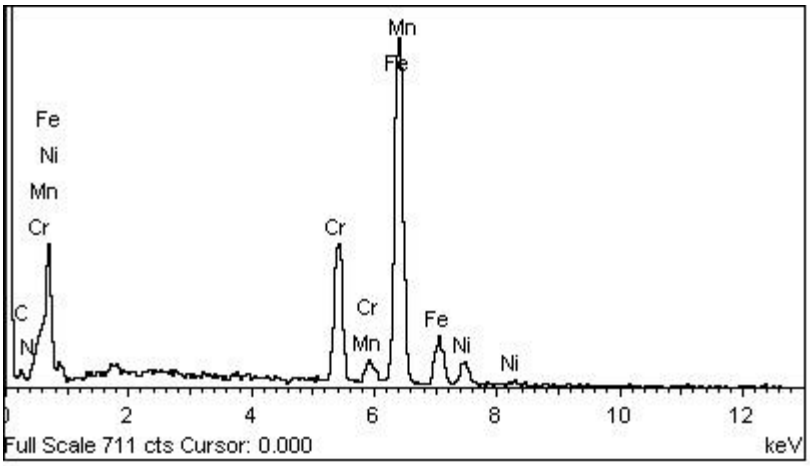


Figure 33: Graph of EDX Analysis on Chemical Composition for 800°C Nitrided 316L ASS Sample at Grain Boundary (Chromium Nitride Precipitation)

4.4 X-Ray Diffraction Analysis

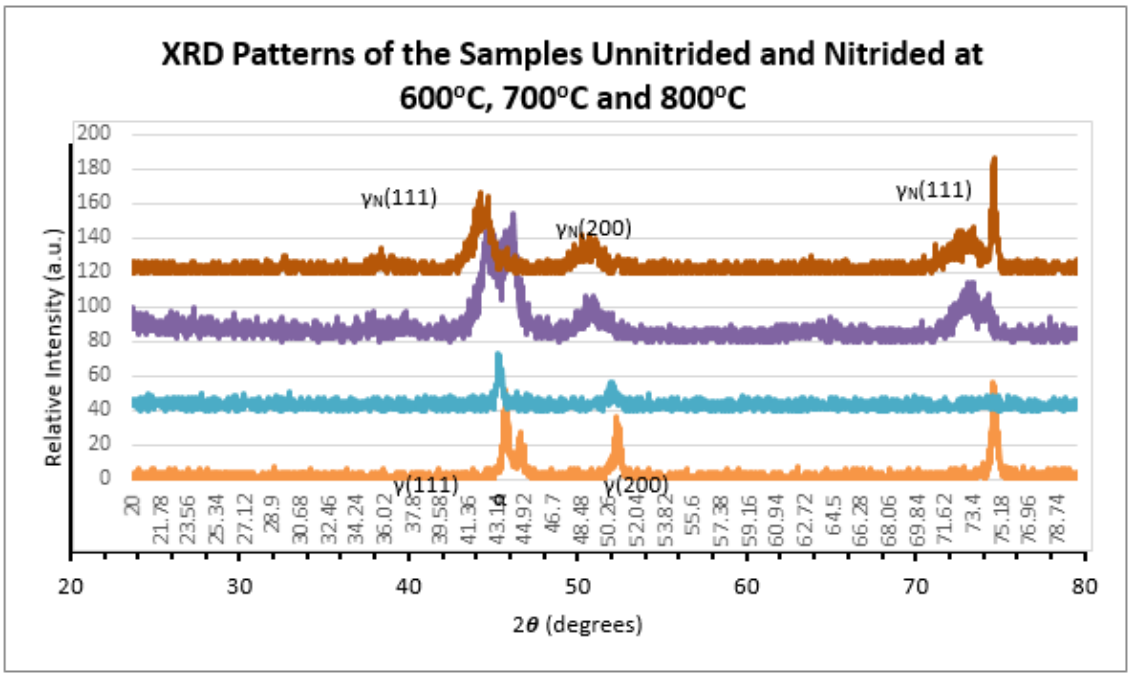


Figure 34: XRD Pattern of Untreated and Nitrided Sample

Figure 34 shows the XRD patterns of untreated and nitrided samples. Nitrided samples consist of 3 different parameters which they are different in term of nitriding temperature, 600°C, 700°C, and 800°C. XRD pattern show γ and α peaks for the untreated sample while γN(111) and γN(200) peaks for all nitrided samples. Hybrid layer were broadened and shifted to a lower angle compared with the γ(111) and γ(200) peaks from the untreated substrate. This phenomena is much related to the nitrogen and carbon supersaturation in the austenite lattice. These supersaturation of nitrogen and carbon with chromium will produce chromium rich precipitate at the grain boundaries and lead to intergranular corrosion. On top of this supersaturation is also related to the enhancement of the nitrided samples towards hardness as been confirmed with Vickers Microhardness Test.

4.5 Vickers Microhardness Test Analysis

Different depth and nitriding temperature may give a trend to the profiles of thermochemically hardened stainless steel. Vickers Microhardness Test is utilized to determine the persistence of 316L Stainless Steel to the indentation under the influence of the nitriding temperature. As hypothesis for the microhardness test, the higher the value, the greater the hardness of ASS. 300gf load and 15 seconds dwell time is used in this test. The test is conducted by taking the microhardness value starting from the surface until its subsurface along the cross section of the samples

Table 10 shows the Vickers Microhardness Test value on untreated, 600°C, 700°C, and 800°C samples. Each of the sample is experimented with 11 spots along the cross section. The average Vickers Microhardness Test of untreated sample is about 148.9, the lowest between other 3 samples. 600°C, 700°C, and 800°C give 176.7, 203.5, and 253.9 respectively. This value can be analyzed as the higher the nitriding temperature, the higher the microhardness. The differences in hardness is much more related to the presence of chromium nitride/carbide at the microstructure during the nitriding process.

Figure 35 shows the Vickers Microhardness Test profiles for all samples which are untreated, 600°C, 700°C, and 800°C. It clearly shows that untreated sample which mean the sample that do not undergo nitriding process give the lowest value. On top of that, as

the nitriding temperature increase, the microhardness increase. For example, at 3mm indentation point, the Vickers Microhardness Test gives 148.1 for untreated sample while 240.9, 320.3 and 401.7 for 600°C, 700°C, and 800°C respectively. Then, the hypothesis for the Vickers Microhardness Test and Nitriding Temperature is accepted.

Table 10: Vickers Microhardness Test's Result

Indentation Point (mm)	**Vickers Microhardness Test**			
	Samples			
	Untreated	**600°C**	**700°C**	**800°C**
3	148.1	240.9	320.3	401.7
6	147.3	236.7	321.1	383.2
9	148.9	217.8	289.9	341.9
12	146.8	169.3	200.3	284.9
15	149.0	164.1	180.7	302.1
18	150.4	160.4	162.4	279.8
21	148.2	154.1	158.8	174.4
24	151.7	152.0	154.0	162.7
27	149.8	150.5	152.3	159.5
30	150.1	149.7	150.5	154.0
33	147.6	147.8	148.3	148.7
Average	148.9	176.7	203.5	253.9

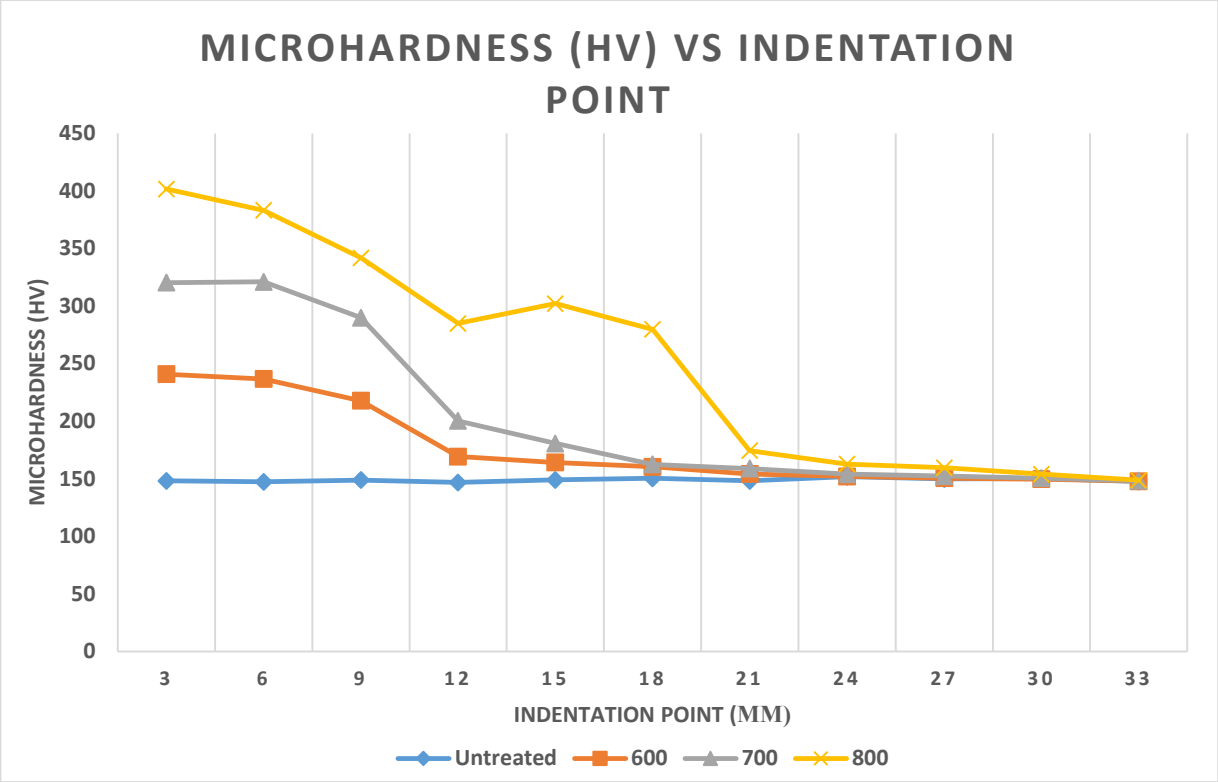


Figure 35: Microhardness (HV) vs Indentation Point

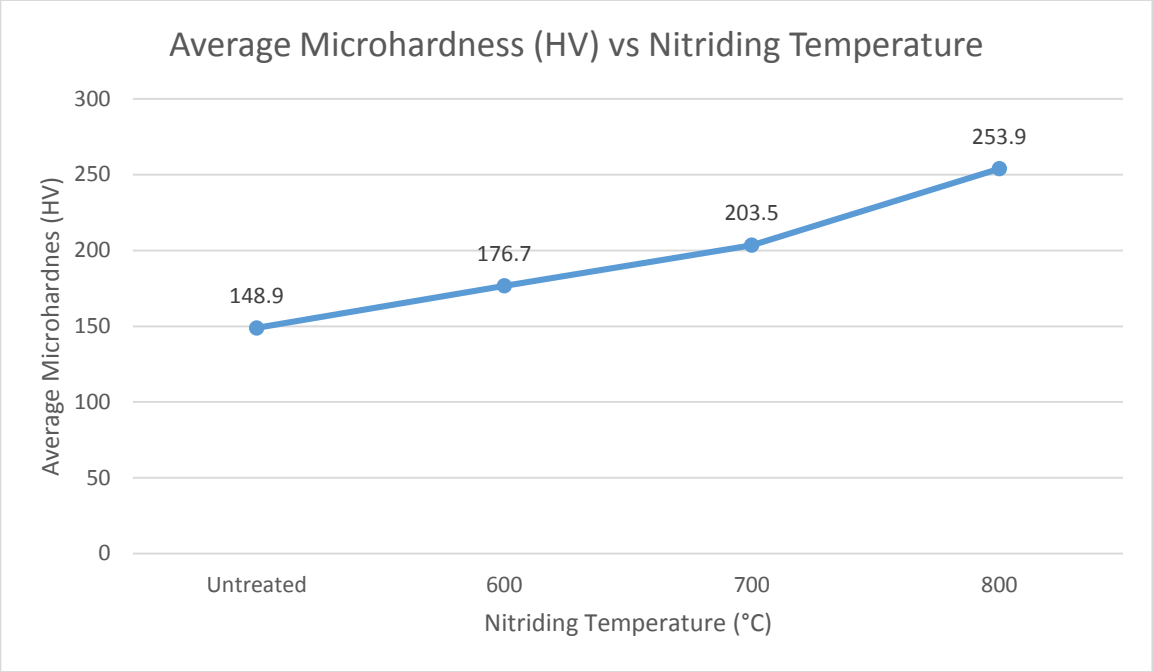


Figure 36: Average Microhardness (HV) vs Nitriding Temperature

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

All in all, the objectives for this research has been achieved which are basically to determine the characteristics of sensitization of nitrided 316L Austenitic Stainless Steel at intermediate temperature and investigate the hardness at different temperature and depth profiles. Besides, any microstructure changes of the untreated and nitrided samples also have been recorded and analyzed. Based on microscopic examination results, twinning phenomena of ASS is formed. Twinning is generally considered as a deformation mechanism that is activated at high strain rates which the critical resolved shear stress for dislocation slip is high. Next, the grain boundary at untreated sample is thinner compare to nitrided sample and higher nitriding temperature gives thicker grain boundary. As sensitization happened, the chromium is unequally distributed and some of it is react with nitrogen supplied through the nitriding process. As the chromium is react with nitrogen, chromium nitride produced and precipitate at the grain boundaries

Vicker Microhardness values increased as the nitriding temperature increased. All the nitrided samples showed microhardness value higher than untreated sample. Average Vickers Microhardness Test for untreated sample is 148.9 and give 18.7%, 36.7%, and 70.5% hardness increment percentage for 600°C, 700°C, and 800°C respectively. These increment percentage is much related to the formation of nitrided layer on the nitrided steel that successfully formed during intermediate temperature gas nitriding treatment. However, the microhardness of each nitrided sample is decreasing at depth profiles increase along the cross section.

Last but not least, based on the results from all experiments, it can be concluded that, sensitization occurred at intermediate temperature gas nitriding treatment and keep increasing at the nitriding temperature increased. Nitriding treatment also contributed to microstructural changes and hardness measurement of 316L austenitic stainless steel. As nitriding temperature increased, the microstructure experienced major changes at the grain boundary as well as the surface hardness values of samples. Hence, the objectives set up at the beginning of this project have been achieved successfully.

5.2 Recommendation

Some improvements could be done for the future research in order complete this investigation. It is recommended to:

i. Conduct corrosion test to determine the corrosion resistance of nitrided sample at intermediate temperature.
ii. Desensitization might be occur when stainless steel is treated at longer period. Determine the most optimum period of nitriding treatment for desensitization to occur could give some information of it.
iii. Modify the nitriding temperature range starts from 500°C to 900°C as sensitization is studied to occur in that temperature range and study about its microstructures, hardness, and corrosion resistance.

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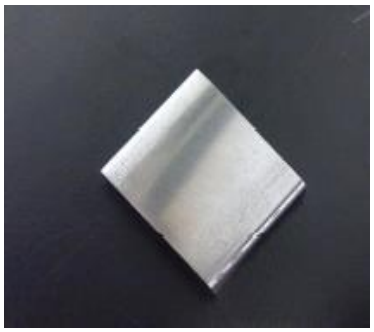
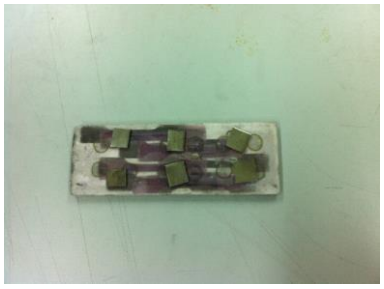
APPENDICES



Untreated 316L Stainless Steel

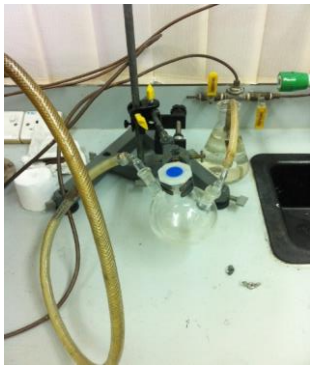
316L immersed in HCL



Sample after the Immersion

Sample in the Furnace

Bubbles Appear



Round-Bottom Flask