

**Correlation between Microstructure and Microhardness of
Nitrided Austenitic Stainless Steel**

by

Ahmad Ikram Bin Mohamad

13607

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

JANUARY 2015

Universiti Teknologi PETRONAS

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme,
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BACHELOR OF ENGINEERING (Hons)
(MECHANICAL)

Approved by,

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UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
JANUARY 2015

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.

AHMAD IKRAM BIN MOHAMAD

ABSTRACT

This paper will study on the effect of the duration of solution nitriding towards the microstructure and micro-hardness of AISI 316L stainless steel. The AISI 316L stainless steel will be exposed to the high temperature gas nitriding process at 1200°C by using the 50% of ammonia gas and 50% of nitrogen gas for different duration of 8, 15 and 20 hours. The nitrided AISI 316L was then undergone slow cooling process after the solution nitriding process. After that, the samples were polished and etched using Viella's etchant in order to have a better image on the microstructure for the optical micrograph observation. It was observed that there were several formation of twins occurred on the microstructure of the nitrided samples. There were two types of twins indentified on the microstructure of the samples which were transgranular twin and suspended twin. It was also noted that the grain size is increasing with the increase of duration of nitriding due to the slow cooling process. Then, the nitrided samples were analyzed using the Electron Dispersive X-ray Spectroscopy (EDS) embedded in the Phenom ProX Scanning Electron Microscope (SEM). From the EDS analysis, it was found that the nitrogen content in the nitrided austenitic stainless steel is increasing as the duration of solution nitriding is increase. The highest nitrogen content was located in the nitrided samples for 20 hour which have about 8.5 wt% of nitrogen. After that, the micro-hardness of the nitrided samples were investigated using the Vickers hardness testing machine. The micro-hardness test were conducted on the cross-sectional area of the nitrided samples. The highest reading were acquired on the surface of each nitrided samples and the reading is decreasing towards the center of all the nitrided samples. From the hardness test, it shown that the nitrided samples for 20 hours have the highest surface hardness of about 245.1 HV which is about 70% higher than the surface hardness of as-received sample. All the results obtained from all the experiments and tests conducted were analyzed to find the correlation between the microstructure and micro-hardness of the nitrided austenitic stainless steel.

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CHAPTER 1

INTRODUCTION

1.1. Background of Study

Stainless steel was developed on the early of 20th century and had become one of the most importance materials in the world recently. The stainless steel had been applied in many types of industry such as medical equipment, textile, marine engineering, architecture, chemical engineering and other industries.

Basically, any types of steels that contains more than 12% of chromium is considered as stainless steel. In order to increase the resistance towards localized corrosion, element like nickel is added into the stainless steel. The composition and heat treatment had played an important roles in determining the properties and microstructure of stainless steel. The stainless steel had been classified into three groups according to its microstructure. These groups are austenitic, martensitic and ferritic where each have a face-centered-cubic, tetragonal crystallattice and body-centered-cubic structures.

Compared to the other two groups, the austenitic stainless steel had been widely used in the industry due to its strength, corrosion resistance, weldability and ductility. However, the only drawbacks for austenitic stainless steel is the low hardness. So, in this paper, the austenitic stainless steel type AISI 316L will be exposed to solution nitriding process.

The solution nitriding is a type of gas nitriding which is done at high temperature between 1000°C to 1200°C where the material is exposed to nitrogenous environment in a vacuum condition. This heat treatment method will increase the hardness of the stainless steel due to the diffusion of nitrogen into the steel. So, in this experiment, the AISI 316L is gone through the solution nitriding process at different duration, then, the microstructure and microhardness of the stainless steel is analyzed to find the correlation between it.

1.2. Problem Statement

One of the best way to increase the hardness of stainless steel is by undergone gas nitriding process. Recent studies had been done on austenitic stainless steel which focus on the corrosion resistance, effect of coating to its strength and also effective duration of nitriding process.

Hence, this paper will study about the relationship and correlation between microstructure and microhardness of AISI 316L austenitic stainless steel when subjected to solution nitriding process at 1200°C using 50% ammonia and 50% nitrogen gas at different duration.

1.3. Objectives of the Study

Related to the above problem verified, this final year project was conducted with the following objective to investigate the correlation between microstructure and microhardness of the nitrided austenitic stainless steel

1.4. Scope of Study

This final year project covers the analysis of the austenitic stainless steel type AISI 316L subjected to heat treatment by solution nitriding at temperature of 1200°C using 50% ammonia gas and 50% nitrogen gas at duration of 8, 15 and 20 hours. The microstructure and microhardness of the nitrided austenitic stainless steel will be investigated and analyzed to find the correlation between both microstructure and microhardness.

1.5. Relevancy and Feasibility of Study

This study is relevant because the austenitic stainless steel had been widely used in many industries. So, by knowing the relationship between the microstructure and microhardness of solution nitrided austenitic stainless steel, it will help to increase the applications of the austenitic stainless steel in the industries. Furthermore, it is also feasible to carry out this study due to the availability of all equipment needed to finish this study in the university. Thus, there is no need to rent any equipment from the outside. Hence, it can save a lot of time and costs.

CHAPTER 2

LITERATURE REVIEW AND THEORY

2.1. Stainless Steel

Normally, the classification of stainless steel is according to its microstructure. One of the element in stainless steel is chromium which function is to prevent the oxidation of the steel by forming a protective film on the surface of the stainless steel. Recent study shows that the amount of chromium in stainless steel is at least 11% compared from other element [1].

Based on the Figure 2.1 below, it shows that at temperature of about 1050°C the solubility limit of Cr is about 13% in the γ phase. Thus, the steel itself is in the γ phase where the austenite will change to martensite due to the high temperature condition for the martensite start temperature [2]. Hence, the steel will be in the martensitic condition at room temperature.

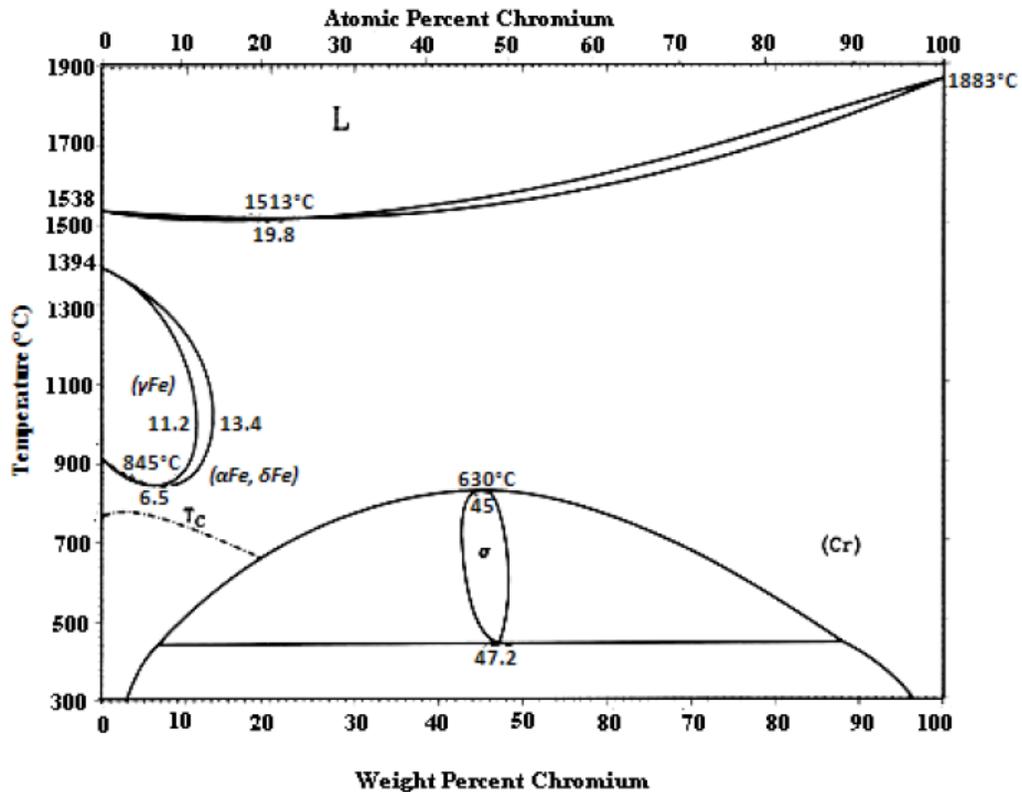


Figure 2.1: Fe-Cr equilibrium diagram [1]

The increase in the content of chromium in the stainless steel will cause the transformation of austenite to ferrite. Thus, the stainless steel will be at ferrite condition at room temperature. In order to prevent this from happen, the nickel is add into the stainless steel to keep the austenite condition at higher content of chromium due to the ability of the nickel that is a powerful former of austenite structure. The Figure 2.2 below shows the ability of the nickel in stabilizing the austenite. So, the insertion of only 9% nickel was enough to keep the stainless steel in the austenite form even at a high content of chromium. As a result, the temperature of martensite-finish and martensite-start will decreases to zero [2]. Hence, the stainless steel will be in the form of austenite when cool to ambient temperature.

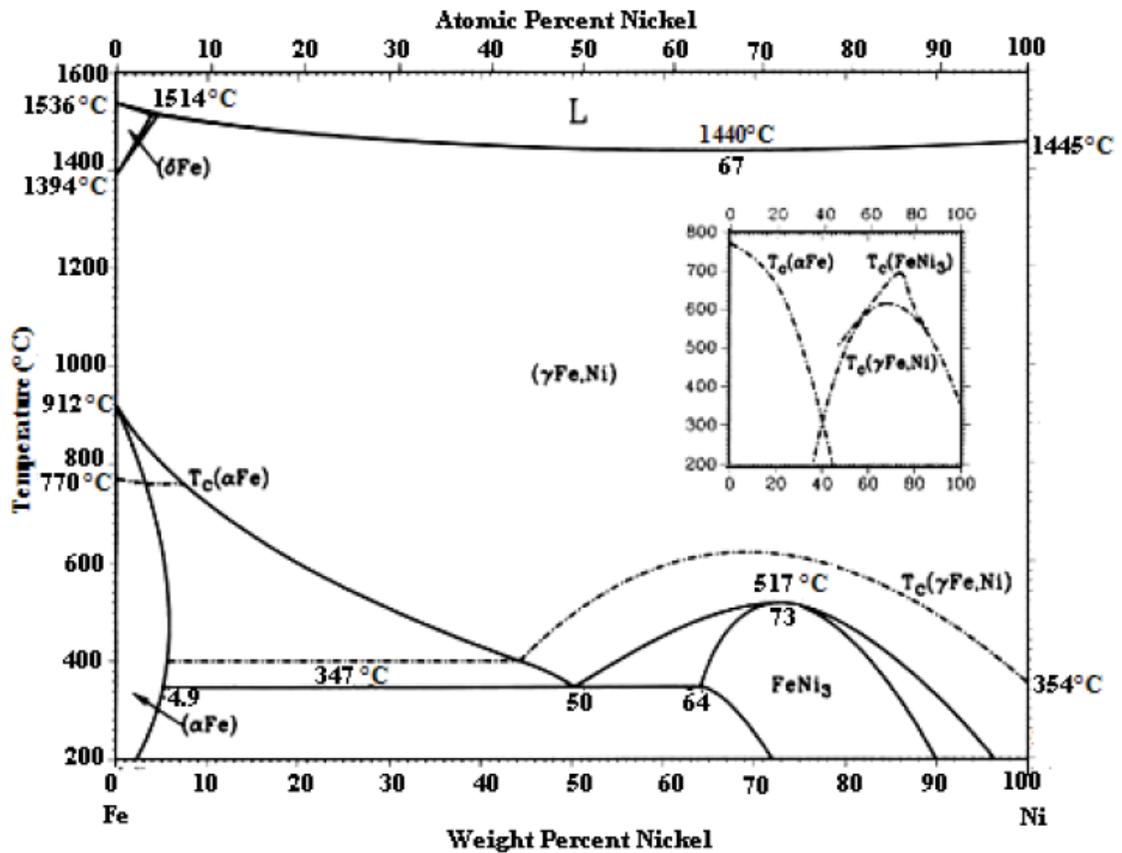


Figure 2.2: Fe-Ni equilibrium diagram [3]

The Table 2.1 below shows the categories of stainless steel according to its microstructure and composition of alloy:

Table 2.1: Categories of stainless steel [4]

| Microstructure | Alloy Composition | Grades |
|---|---|--|
| Martensitic | 11.5–18% Cr, 0.08–0.20% C | Types 403, 410, 414, 416, 416Se, 420, 422, 431, 440A, 440B, and 440C |
| Ferritic | 10.5–30% Cr | Types 405, 409, 429, 430, 430F, 430F–Se, 434, 436, 439, 444, and 446 |
| Austenitic | 16–26% Cr, 0.75–19.0% Mn, 1–40% Ni, 0.03–0.35% C, and sufficient N | 200 and 300 series are groups of this steel |
| Duplex | 18–29% Cr, 2.5–8.5% Ni, and 1–4% Mo, up to 2.5% Mn, up to 2% Si, and up to 0.35% N | Type 329 |
| Precipitation hardening (PH) stainless steel | Austenitic and martensitic with alloy added to form precipitates; Mo, Cu, Al, Ti, Nb, and N | 17-7PH (UNS S17700) and PH15-7Mo (UNS S15700) |

2.2. Austenitic Stainless Steel

Austenitic stainless steel usually composed of the element of Fe, Cr and Ni that have a face-centered cubic (FCC) form due to the presence of some stabilizer element for austenite like nitrogen, manganese and nickel [5]. Compared to the other types of stainless steel, the austenite stainless steel has the highest thermal resistance to strength ratio. Thus, it is normally being applied in the high temperature condition in industry. The austenitic stainless steel also usually being hardened by cold work and has the nonmagnetic characteristic in annealed condition. Moreover, it is also vulnerable to any types of thermally induced embrittlement which is the acute reduction of strength and ductility like sensitization and sigma-phase embrittlement. The Table 2.2 below shows the classification of austenitic stainless steel.

Table 2.2: Classification of Austenitic Stainless Steel [5]

| Groups of Austenitic Stainless Steel | Grades |
|--------------------------------------|--|
| Conventional Austenitic | Types 301, 302, 303, 304, 305, 308, 309, 310, 316 and 317 |
| Low-Carbon Grades | Types 304L, 316L and 317L |
| Stabilized Composition | Types 321, 347 and 348. |
| High Alloyed Austenitic | Types 317LX, JS777, 904L, AL-4X and 254 SMO |
| High-Nitrogen Grades | Types 201,202, 304N, 316N and the Nitronic series of alloy |

The Table 2.3 below shows the compositions for some grades of austenitic stainless steel.

Table 2.3: Composition of Several Austenitic Stainless Steel [6]

| Types | Composition, % | | | | | | |
|-------|----------------|-----------|----------|-----------|-----------|------|--------------|
| | C | Mn | Si | Cr | Ni | S | Other |
| 201 | 0.15 | 5.5-7.5 | 1.0 | 16.0-18.0 | 3.5-5.5 | 0.03 | 0.25N |
| 205 | 0.12-0.25 | 14.0-15.5 | 1.0 | 16.5-18.0 | 1.0-1.75 | 0.03 | 0.32-0.40N |
| 302 | 0.15 | 2.0 | 1.0 | 17.0-19.0 | 8.0-10.0 | 0.03 | - |
| 302Cu | 0.08 | 2.0 | 1.0 | 17.0-19.0 | 8.0-10.0 | 0.03 | 3.0-4.0Cu |
| 316H | 0.04-0.10 | 2.0 | 1.0 | 16.0-18.0 | 10.0-14.0 | 0.03 | 2.0-3.0Mo |
| 316L | 0.03 | 2.0 | 1.0 | 16.0-18.0 | 10.0-14.0 | 0.03 | 2.0-3.0Mo |
| 317 | 0.08 | 2.0 | 1.0 | 18.0-20.0 | 11.0-15.0 | 0.03 | 3.0-4.0Mo |
| 317L | 0.03 | 2.0 | 1.0 | 18.0-20.0 | 11.0-15.0 | 0.03 | 3.0-4.0Mo |
| 330 | 0.08 | 2.0 | 0.75-1.5 | 17.0-20.0 | 34.0-37.0 | 0.03 | - |
| 347 | 0.08 | 2.0 | 1.0 | 17.0-19.0 | 9.0-13.0 | 0.03 | 10x%C min Nb |
| 384 | 0.08 | 2.0 | 1.0 | 15.0-17.0 | 17.0-19.0 | 0.03 | - |

2.3. Nitriding Process

Nitriding is a type of surface treatment which is done thermo chemically in order to increase the strength of the steel [7, 8]. The nitriding process is conducted at a suitable temperature where the nitrogen will be transfer to the steel and diffuse into the steel. A perfect nitriding process will result the formation of nitrite in the steel structure. This can be proof by the formation of white layer comprise of $Fe_2(N,C)_{1-x}$ and Fe_4N phases on the surface of the steel [7,8, 9]. The diffusion zone which is located below the compound layer is the place where the diffusion of nitrogen into the ferrite lattice is occurred. Figure 2.3 below shows the diffusion process of nitrogen into the stainless steel.

The good properties like improvement in fatigue life, increase wear resistance, high surface hardness and anti-galling properties achieved by the nitriding process make it suitable for case-hardening process of steel [9, 10]. The low temperature used in the nitriding process had resulted to a less deformation and distortion of the heat treated parts [8]. There are various type of nitriding process that can be used for the surface treatment of the steel which are plasma nitriding, salt bath nitriding and gas nitriding.

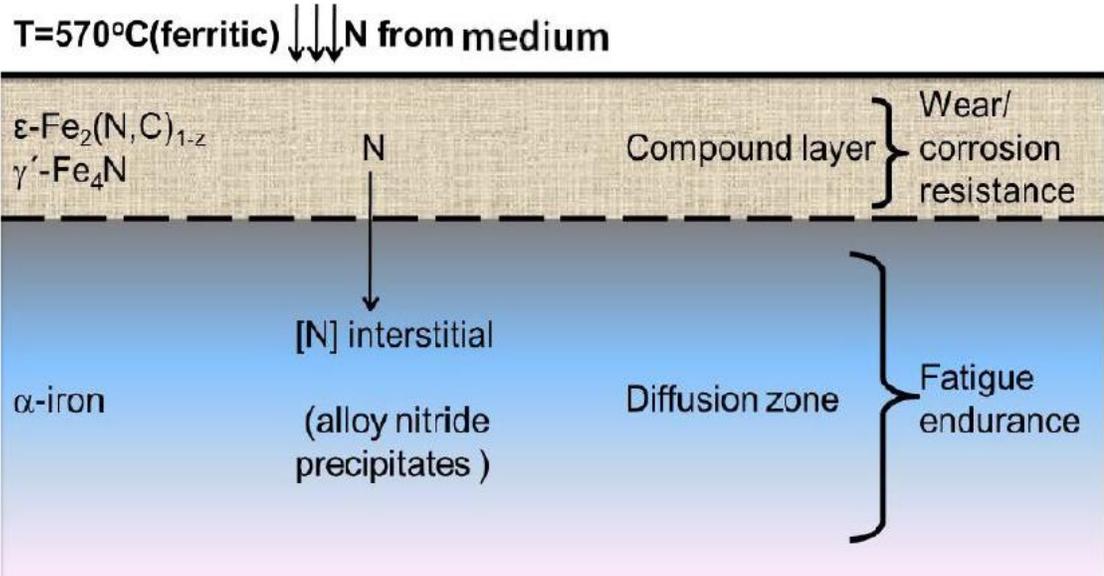


Figure 2.3: Schematic compound layer and diffusion zone of nitride steel[18]

2.4. Gas Nitriding Process

The gas nitriding process is a process where the material which is the austenitic stainless steel in this experiment will be put in an enclosed tube within a nitrogenous atmosphere at a particular temperature. A low-temperature gas nitriding process is where the nitriding process is done at the temperatures ranging from 400°C to 700°C. In this process, the formation of nitrides will occur at the surface of the stainless steel due to the diffusion of nitrogen into the steel. As a result, the steel will become hardened [11]. The drawbacks of this process is the decrease of the ability of the stainless steel against corrosion [12-13]. The gas nitriding process that is done at the temperatures between 1000°C to 1200°C is called solution nitriding which is consider as a latest process to increase the strength of the steel [12]. The solution nitriding process followed by quenching will allow the nitrogen to diffuse interstitially into the steel and cause the increase of strength of the stainless steel. This type of nitriding process is usually apply in the heat treatment process for austenitic [12], ferritic [14], martensitic [13] and duplex [15] stainless steels. Recent studies had shown that the high temperature gas nitriding will cause the nitrogen to diffuse deeper into the stainless steel compared to the low temperature gas nitriding.

Another research had shown that the production of nitrides will occur at the surface of duplex stainless steel when it was subjected to high temperature gas nitriding at 1150°C for 15 hours [16]. Not only that, it was also found that the microstructure of austenitic stainless steel will not be affected by the high temperature gas nitriding [17]. For both steel, it was observed that the increase of pressure of nitrogen will cause a higher absorption of nitrogen. Hence, the size of grain of the steel will increase along with the duration of high temperature gas nitriding when the steel had absorbed all the nitrogen. Moreover, the production of nitrogen pearlite also will happen when the content of nitrogen is more than its solubility in austenite.

CHAPTER 3

METHODOLOGY/PROJECT WORK

3.1. Flowchart

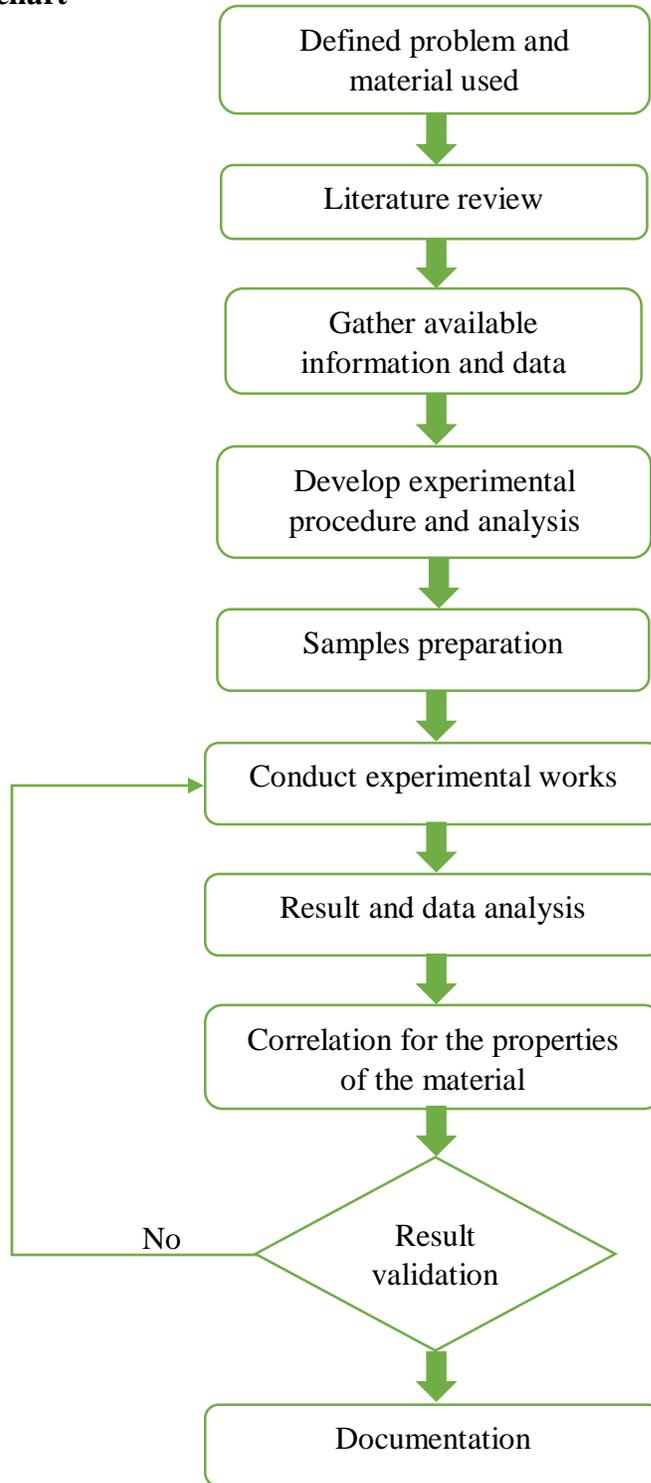


Figure 3.1: Flow Chart for the Progress of FYP

3.2. Project Methodology

3.2.1. Material

The stainless steel that will be use in this project is a type AISI 316L austenitic stainless steel. This type of stainless steel has a better corrosion resistance compared to type 304 stainless steel. The austenitic stainless steel will be divided into 3 samples for different duration of solution nitriding which is 8, 15 and 20 hours.

3.2.2. Gas Nitriding Process

The Figure 3.2 shows the setup of the gas nitriding process. The procedure of experiment are as below:

- 1) The austenitic stainless steel is nitride with gas nitriding treatment.
- 2) The process will be conducted in Alumina horizontal furnace tube.
- 3) The gas flow meter is attached between the gas cylinder and furnace tube in order to control the flow rate and to maintain a static flow rate during the process.
- 4) In order to increase the gas pressure flown in the furnace, the gas outlet tube from the furnace tube is connected to the Drechsel bottle.
- 5) The material is put inside at the center of the furnace tube which is located in between the heating element.
- 6) The process is conducted at 1200°C for 8 hours.
- 7) Steps 2 to 6 is repeated for a different duration of 15 and 20 hours.

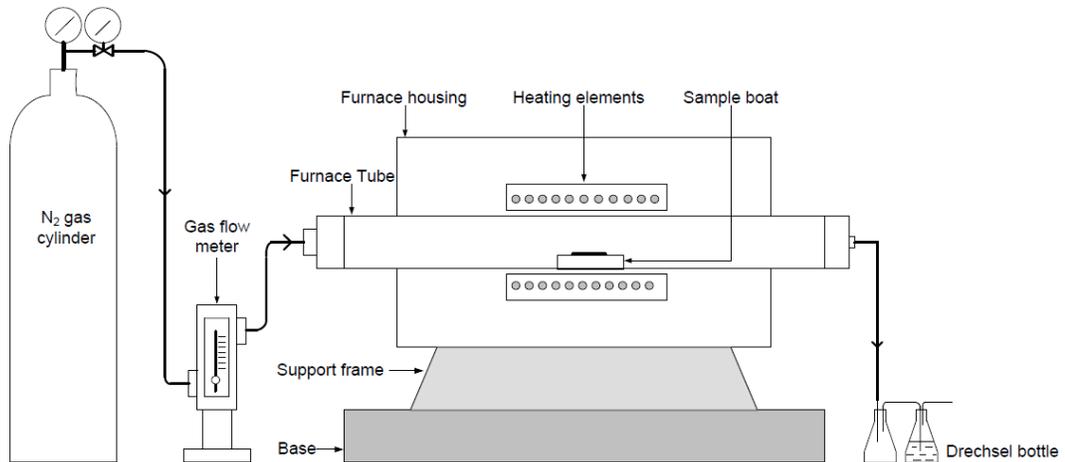


Figure 3.2: Gas Nitriding equipment setting[7]

3.2.3. Metallography Process

a. Sample Preparation

Procedure for sample preparation:

- 1) Using abrasive cutter, the material is cut to desired shape.
- 2) Using the Automatic Mounting Press and Buehler Phenolic Black Powder, the material is hot mounted to make it easier to handle.
- 3) Using rotating disc of 400, 800 and 1200 grit silicon carbide paper and lubrication with water, the material is grinded. To ensure the material is flat during the grinding process, a small amount of pressure is applied at the center of the mounted material.
- 4) Using rotating discs covered with soft cloth impregnated with micro-particles of diamond, the sample is polished.

b. Etchant Preparation and Etching Process

According to the research that had been done, the most suitable etchant for AISI 316L austenitic stainless steel is Viella's Etchant. The Procedure to prepare the Viella's Etchant and the etching process are as below:

- 1) 0.2g of picric acid is weighted using METTLER TOLEDO Electronic Balance and put inside small beaker.
- 2) 1ml of hydrochloric acid is measured and put inside the same beaker as the picric acid.
- 3) The mixed materials is stir until the picric acid is totally dissolved in the hydrochloric acid solution.
- 4) 10ml of ethanol is measured and put inside the same beaker and the solution is stir for a few second.
- 5) The sample is dip into the Viella's Etchant for about 10 second and then washed with water.
- 6) The sample is dried using drying machine.

c. Optical Microscope

The optical microscope that is used in this paper is LEICA Optical Microscope as shown in Figure 3.3. The procedure for using the optical microscope are as below:

- 1) The optical microscope and the computer are switched ON.
- 2) The etched sample is put on the adjustable table and observed using the microscope.
- 3) The image of any interested area within the etched sample is captured at the desired magnification by using the microscope.
- 4) The captured image is saved in the computer.
- 5) Steps 2 to 4 is repeated for the other 2 samples.



Figure 3.3: LEICA Optical Microscope

d. Scanning Electron Microscope (SEM) and EDS

The scanning electron microscope that is used in the study is Phenom ProX desktop SEM. The procedure of the process are as below:

- 1) The SEM and the computer are switched ON.
- 2) The etched sample is put in a holder and put inside the SEM.
- 3) The image of designated area on the etched sample is captured and 2 spot is defined to use the EDS to observe the chemical composition of the spots.
- 4) The image and data observed by the SEM and EDS is saved in the computer.
- 5) Steps 2 to 4 is repeated for another 2 samples.

3.2.4. Hardness Test



Figure 3.4: LECO Microhardness Tester LM247AT

The Vickers hardness test is used in this project by using the LECO Microhardness Tester LM247AT as shown in the Figure 3.4 above. The indenter used in this process is a square-based pyramid form of diamond. The procedure for the hardness test are:

- 1) The power supply is switched on and the indenter is moved forward position close to the operator.
- 2) The sample is placed and raised until the indenter tip touching the sample surface.
- 3) The turning of the hand wheel clockwise will cause the sample to touch the indenter.
- 4) The microscope is used to verify the contact point is reached.
- 5) A right test force is used to press the indenter on the sample.
- 6) A dwell time for 10-15 second is needed to maintain the force before the indenter is removed from the sample.
- 7) The hardness reading of the sample is recorded.
- 8) Turning the hand wheel in the direction of counter clockwise will release the sample.
- 9) All the procedure above is repeated to get another reading at different spot.

3.3. Key Project Milestone

The key milestone of this final year project are as stated in the Table 3.1 below:

Table 3.1: Key Milestone of the FYP

| No | Key Milestone | Weeks | FYP |
|-----|--|-------|-------|
| 1. | Submission of Extended Proposal | 6 | FYP 1 |
| 2. | Proposal Defense | 9 | |
| 3. | Acquire the Stainless Steels | 13 | |
| 4. | Submission of Interim Report | 14 | |
| 5. | Nitriding Process of Stainless Steel | 7 | FYP 2 |
| 6. | Submission of Progress Report | 7 | |
| 7. | Mechanical and Chemical Tests of Samples | 9 | |
| 8. | Pre-SEDEX | 10 | |
| 9. | Submission of Draft Report | 11 | |
| 10. | Submission of Dissertation (Softbound) | 12 | |
| 11. | Submission of Technical Paper | 12 | |
| 12. | Oral Presentation | 13 | |
| 13. | Submission of Project Dissertation (Hardbound) | 15 | |

3.4. Gantt Chart

The Table 3.2 below shows the overall gantt chart for the Final Year Project:

Table 3.2: Gantt Chart of Timeline for FYP

| Project Work | Weeks | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
|--|------------------------------|----------|----------|----------|----------|---------------|----------|---------------|----------|----------|----------|----------|---------------|----|------------------------------|----------|----------|----------|----------|----------|---------------|----------|---------------|---------------|---------------|---------------|----|---------------|----|--|
| | Final Year Project 1 (FYP 1) | | | | | | | | | | | | | | Final Year Project 2 (FYP 2) | | | | | | | | | | | | | | | |
| | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 1 | 2 | 3 | 4 | 5 | 6 | 7 | 8 | 9 | 10 | 11 | 12 | 13 | 14 | 15 | |
| Selection of Topic | Progress | Progress | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Early Research Work on Literature | | | Progress | Progress | Progress | Progress | Progress | Progress | Progress | | | | | | | | | | | | | | | | | | | | | |
| Submission of Extended Proposal | | | | | | Key Milestone | | | | | | | | | | | | | | | | | | | | | | | | |
| Proposal Defense | | | | | | | | Key Milestone | | | | | | | | | | | | | | | | | | | | | | |
| Research on Test Can Be Apply | | | | | | | | Progress | Progress | Progress | Progress | Progress | Progress | | | | | | | | | | | | | | | | | |
| Acquire the Stainless Steels | | | | | | | | | | | | | Key Milestone | | | | | | | | | | | | | | | | | |
| Interim Report Submission | | | | | | | | | | | | | Key Milestone | | | | | | | | | | | | | | | | | |
| Sample Preparation | | | | | | | | | | | | | | | Progress | Progress | Progress | Progress | | | | | | | | | | | | |
| Nitriding Process of Stainless Steel | | | | | | | | | | | | | | | | | | | Progress | Progress | Key Milestone | | | | | | | | | |
| Submission of Progress Report | | | | | | | | | | | | | | | | | | | | | Key Milestone | | | | | | | | | |
| Mechanical and Chemical Test of Sample | | | | | | | | | | | | | | | | | | | | | Progress | Progress | Key Milestone | | | | | | | |
| Full Result Preparation | | | | | | | | | | | | | | | | | | | | | | Progress | Progress | Progress | | | | | | |
| Pre-SEDEX | | | | | | | | | | | | | | | | | | | | | | | Key Milestone | | | | | | | |
| Submission of Draft Report | | | | | | | | | | | | | | | | | | | | | | | | Key Milestone | | | | | | |
| Submission of Dissertation (SoftBound) | | | | | | | | | | | | | | | | | | | | | | | | | Key Milestone | | | | | |
| Submission of Technical Paper | | | | | | | | | | | | | | | | | | | | | | | | | Key Milestone | | | | | |
| Oral Presentation | | | | | | | | | | | | | | | | | | | | | | | | | | Key Milestone | | | | |
| Submission of Project Dissertation | | | | | | | | | | | | | | | | | | | | | | | | | | | | Key Milestone | | |



CHAPTER 4

RESULTS AND DISCUSSION

4.1. Microstructure

Figure 4.1 below shows the optical micrograph of as-received sample of AISI 316L taken in this study. The image of this as-received sample was taken using optical microscope with the use of Marble's Etchant. Based on the Figure 4.1 below, it shows that the as-received sample had undergone the cold working due to the rolling effect on its surface. The microstructure of the as-received sample contains some recrystallization of twins and have a small grain size.

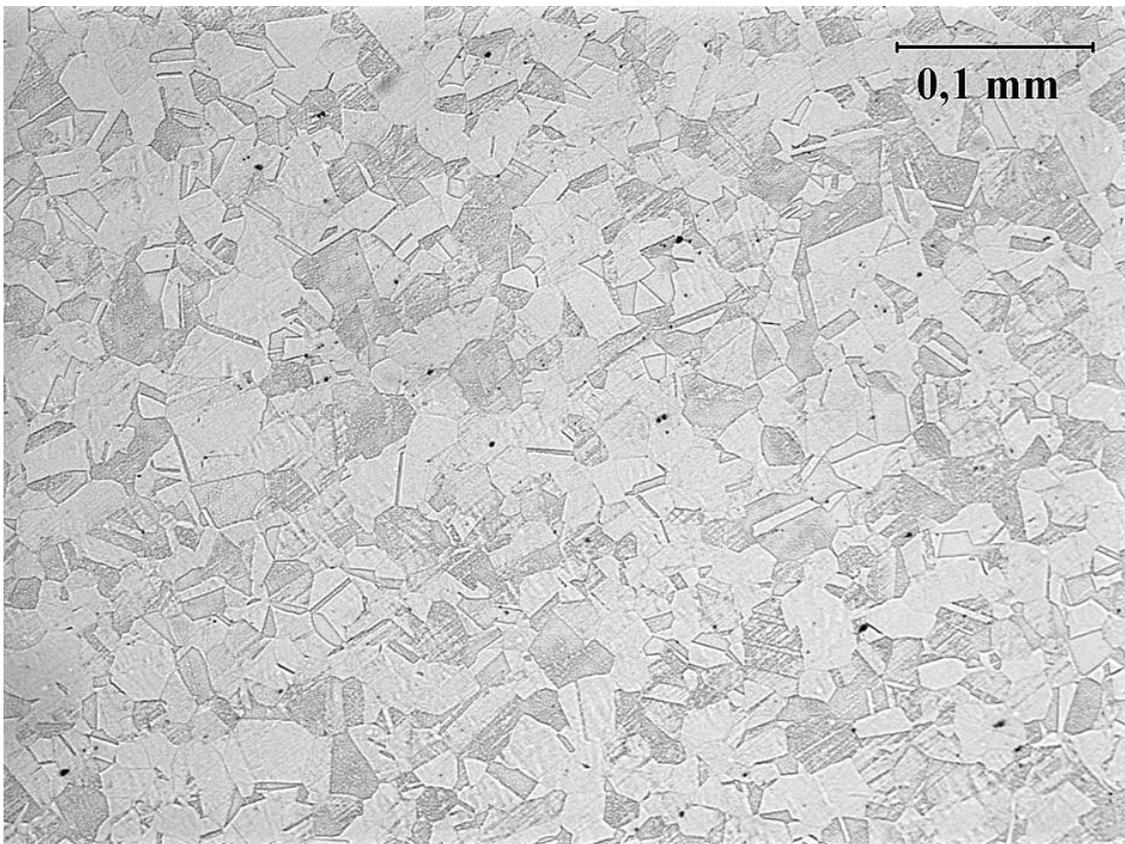


Figure 4.1: Optical Micrograph of as-received AISI 316L

All the 3 samples of AISI 316L were undergone the solution nitriding for different duration of 8, 15 and 20 hours. All the samples were let to cool naturally which is by air cooling. The nitrided samples were then polished, etched with Viella's Etchant to increase the visibility of its microstructures and observed by LECA Optical Microscope.

The Figures 4.2, 4.3 and 4.4 below shows that all the samples had experienced the formation of twins on its microstructures. These formations of twins are considered as normal for the low stacking fault energy metals that have face-centered-cubic structures like austenitic stainless steel. It is observed that the formation of twins in this paper were consist of two types which are transgranular twin and suspended twin. This two types of twins is clearly shown in the Figures 4.2, 4.3 and 4.4 below. The transgranular twins is made up from four parts which are two sides are in coherent twin planes and the both ends are the grain boundary. For the suspended twins, also have four parts, the incoherent twin plane is at the head, the coherent twin planes are at both sides and the grain boundary is at the end.

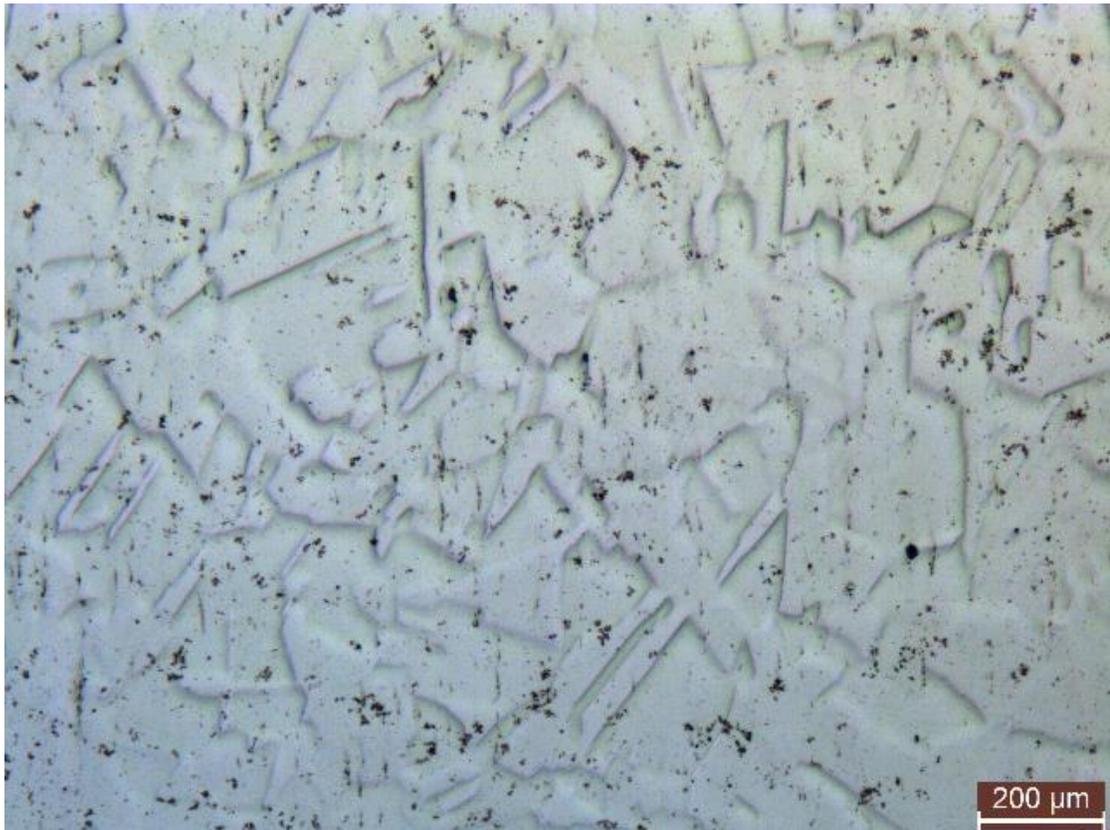


Figure 4.2: Optical Micrograph of Nitrided AISI 316L for 8 Hours



Figure 4.3: Optical Micrograph of Nitrided AISI 316L for 15 Hours

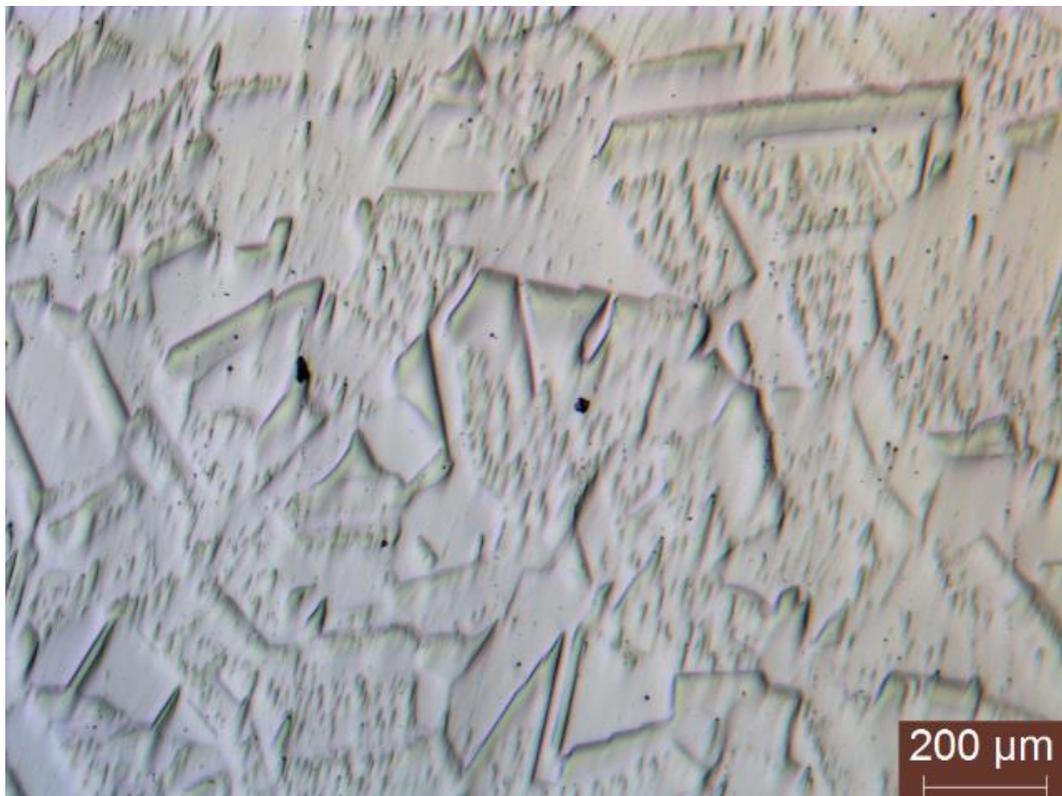


Figure 4.4: Optical Micrograph of Nitrided AISI 316L for 20 Hours

Moreover, it was also observed that the grain size of austenite in the samples is increasing with the increase of duration of solution nitriding due to the effect of slow cooling process. If the samples is quenched after nitriding process, the grain size will become smaller and the toughness will become higher. However, the duration of solution nitriding was not affecting the single phase structure of austenite. Thus, the diffusion of nitrogen into the steel is totally in solid solution austenite leaving no trace of nitrides behind.

All of the nitrided samples had experienced the slow cooling process from the temperatures of about 1200°C. So, the samples was exposed to the sensitization temperature of austenitic stainless steel which is in the range from 450°C to 870°C. At this temperatures, a normal austenitic stainless steel will experience the formation of chromium carbide precipitation on its grain boundaries. However, for all the nitrided samples, it was observed that there was no formation of chromium carbide precipitation on its grain boundaries due to the low carbon content in the AISI 316L.

4.2. Chemical Composition

The nitrided samples of AISI 316L was investigated using the SEM machine. The Energy Dispersive X-ray Spectroscopy (EDS) analysis by the SEM was done to figure out the chemical composition of the nitrided austenitic stainless steel especially for the nitrogen content in the steel. The EDS analysis was done at random spot on the surface of the nitrided austenitic stainless steel. Figure 4.5, 4.6 and 4.7 below shows the result captured from the EDS analysis by SEM at a different duration of solution nitriding.

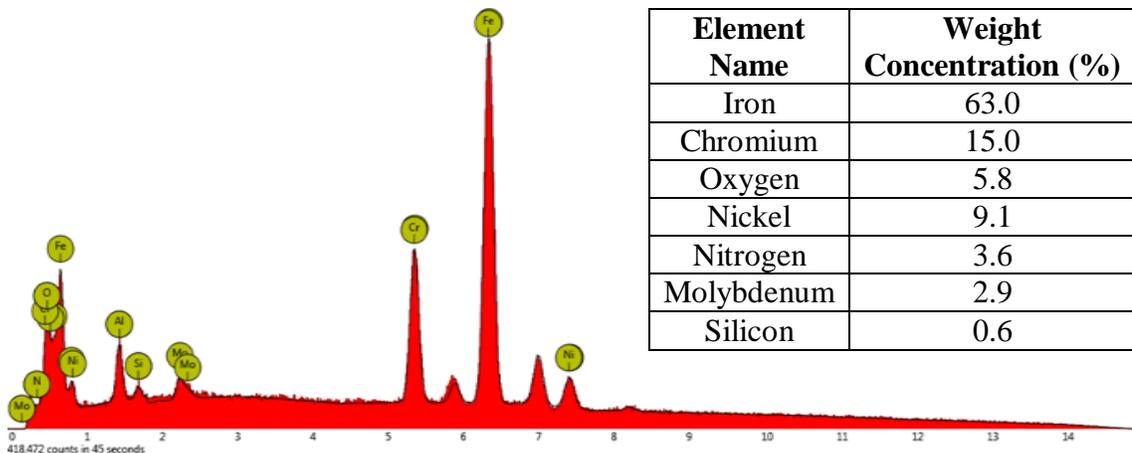


Figure 4.5: EDS analysis by SEM of Nitrided AISI 316L for 8 Hours

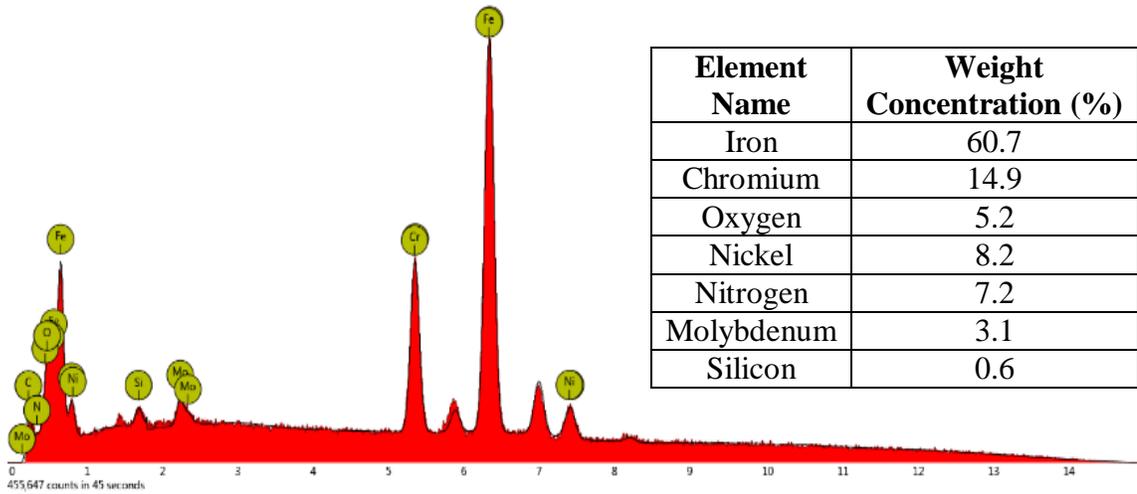


Figure 4.6: EDS analysis by SEM of Nitrided AISI 316L for 15 Hours

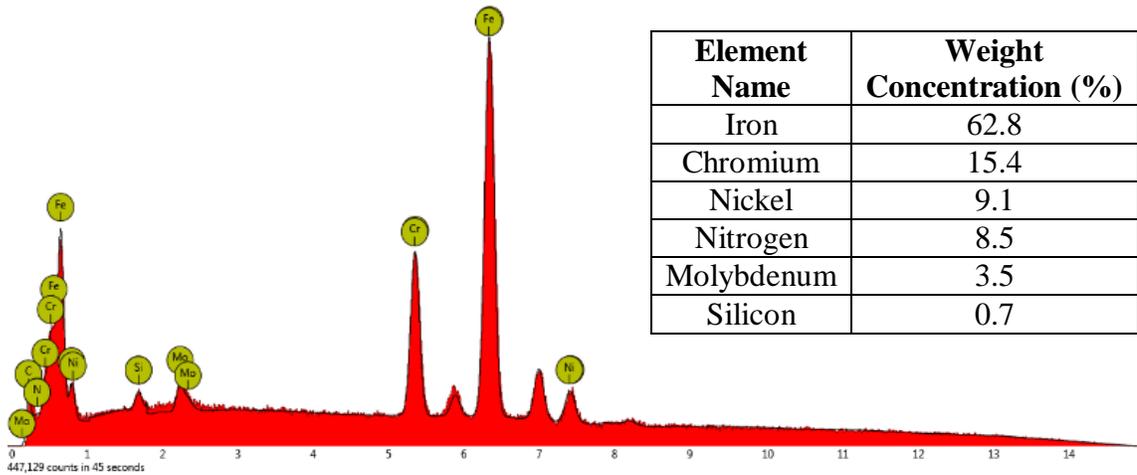


Figure 4.7: EDS analysis by SEM of Nitrided AISI 316L for 20 Hours

From the result of the EDS analysis in the Figure 4.5, 4.6 and 4.7 above, it was clearly shown that the nitrogen content in the steel is increasing with the duration of solution nitriding even though the EDS analysis was executed at random spot on the surface of the steel not at the same spot. The lowest nitrogen content which is about 3.6 wt.% for 8 hours of solution nitriding whereas the highest nitrogen content is about 8.5 wt.% for 20 hours of solution nitriding. Hence, the higher the duration of nitriding will result to the higher diffusion of nitrogen into the steel.

4.3. Micro-hardness Test

The Vickers hardness test was completed by using the LECO Microhardness Tester LM247AT. The Vickers hardness test was conducted at the cross sectional surface of the as-received sample and the nitrided austenitic stainless steel samples. The result obtained from the test was plotted in the charts as shown in the Figure 4.8 and 4.9 below. The error bar in the Figure 4.8 was estimated to be ± 5 HV due to some human error in conducting the hardness test.

Based on the chart in Figure 4.8 below, the hardness of the nitrided austenitic stainless steel is increasing with the increase in the duration of solution nitriding. It was also observed that the hardness is high at the surface of the as-received austenitic stainless steel and start to decrease when approaching the center of the steel due to the cold working of the as-received austenitic stainless steel. For the nitrided AISI 316L, the hardness also is high at the surface and slowly decrease as it approached the center of the nitrided AISI 316L due to the higher concentration of nitrogen on the surface and the diffusion of nitrogen into the steel was not deep enough to reach at the center.

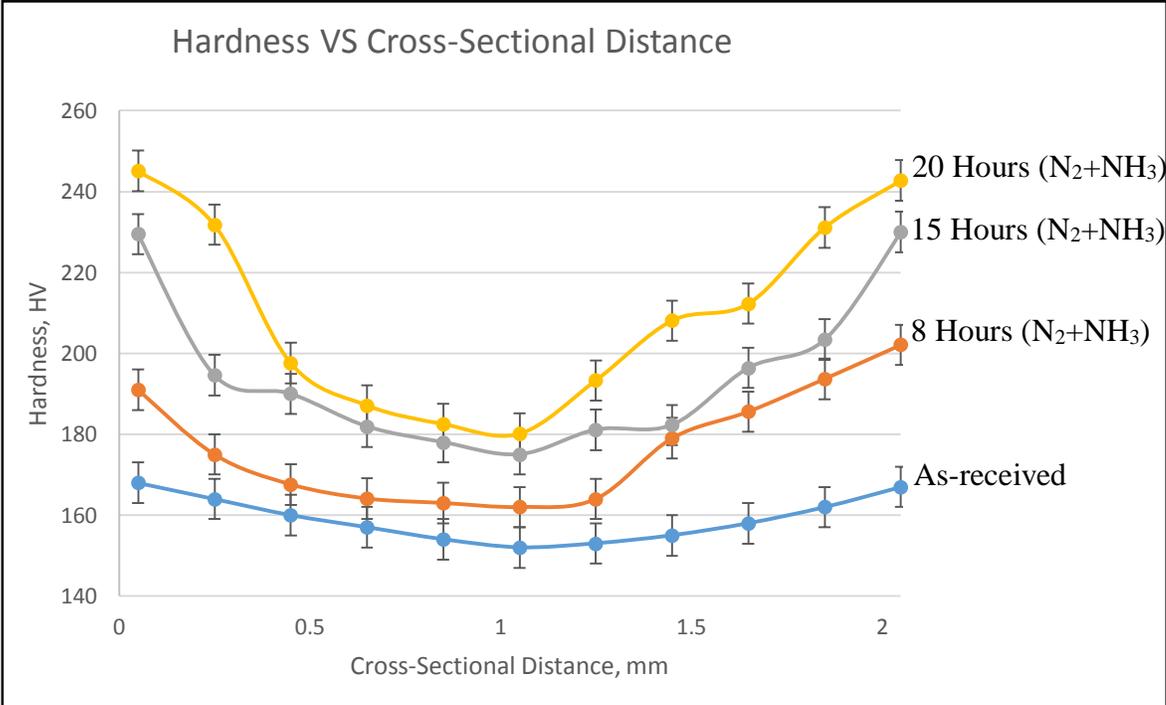


Figure 4.8: Chart of Hardness VS Cross-Sectional Distance for AISI 316L

The chart in Figure 4.9 below shows the effect of the duration of solution nitriding on the surface hardness of the AISI 316L. The surface hardness is expected to increase with the increase of the duration of solution nitriding until about 22 hours before the surface hardness become constant. The highest surface hardness was obtained from the nitrided samples for 20 hours that have about 245.1 HV which is about 70% higher than the hardness of the as-received samples that only have 168 HV. Hence, this proof that the micro-hardness is increasing along with the duration of solution nitriding. Compared to the previous study which used the same austenitic stainless steel AISI 316L but only had the different in the type of nitriding that used plasma nitriding at 450°C for 3 hours, the highest hardness value was found to be 1733 HV [19]. This plasma nitrided samples was agreed to be very effective to increase the strength of the stainless steel. However, the cost of plasma nitriding process which is very high compared to solution nitriding process makes it less economic and not suitable to be apply in any industry.

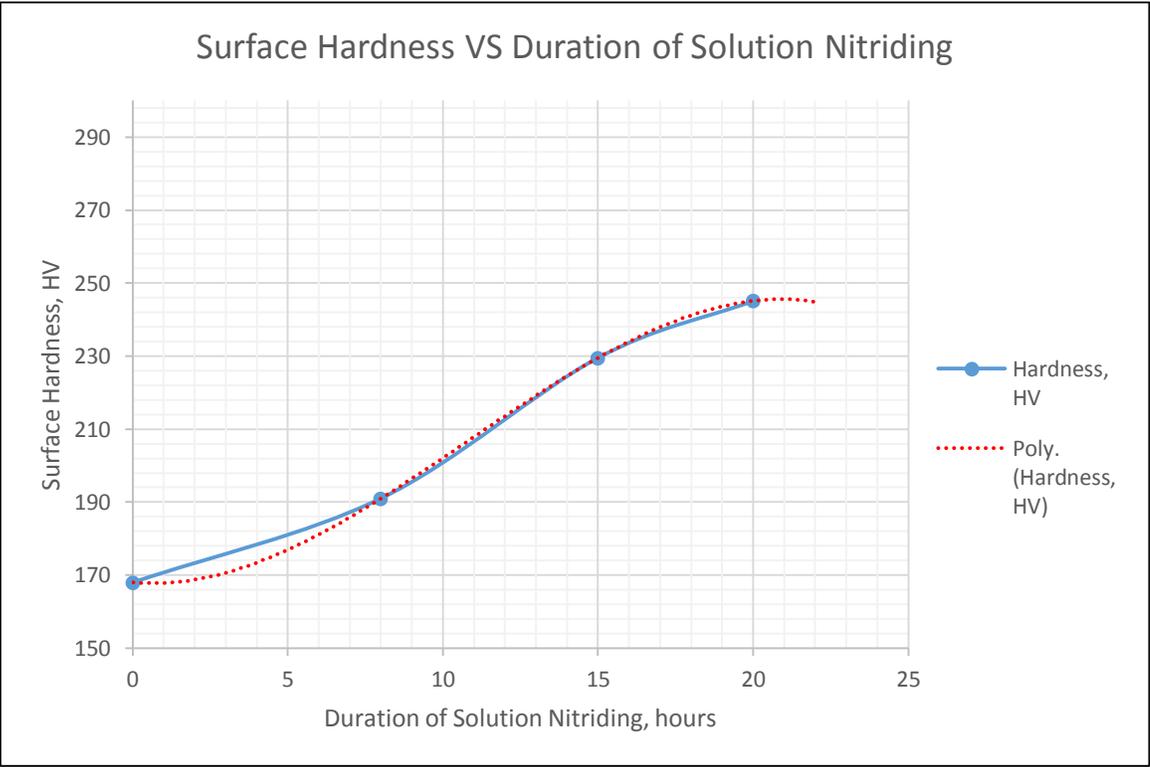


Figure 4.9: Chart of Surface Hardness VS Duration of Nitriding for AISI 316L

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS

5.1. Conclusions

Addition of nitrogen into the steels was indicated by the improvement of hardness on the steel. The highest hardness was acquired from the sample that was nitrided for 20 hours that have 245.1 HV which is 70% higher compared to the hardness value of as-received sample. The duration of solution nitriding had caused in the higher diffusion of nitrogen into the steel. As a result, the hardness of the nitrided AISI 316L was increased and the grain size is expected to decrease. However, due to the effect of the slow cooling process after the solution nitriding process, the grain size of the nitrided AISI 316L is increasing with the duration of nitriding. From this study, it was found that the microstructure for the both as-received and nitrided samples were almost similar that contained the formation of twins and only a bit different in the size of the grains. In conclusion, the increase of duration of solution nitriding had led to the increase of the micro-hardness of the austenitic stainless steel but had no effect on its microstructure.

5.2. Recommendations

There are several suggestions that can be done in order to improve the findings in this paper. Among the suggestions are the XRF machine should be used to analyze the chemical composition in the nitrided AISI 316L in order to get an accurate content of nitrogen that had diffused into the steel. Not only that, an automated Vickers hardness testing machine should be use in measuring the hardness of the austenitic stainless steel so that the human error can be avoided to get an accurate and precise reading. It is also suggested to investigate the phase transformation of the nitrided austenitic stainless steel.

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APPENDICES

Appendix A

Data and graph of as-received sample.

Table A1: Data of Microhardness Test for As-Received Sample

| Cross-Sectional Distance (mm) | Microhardness (HV) |
|-------------------------------|--------------------|
| 0.05 | 168 |
| 0.25 | 164 |
| 0.45 | 160 |
| 0.65 | 157 |
| 0.85 | 154 |
| 1.05 | 152 |
| 1.25 | 153 |
| 1.45 | 155 |
| 1.65 | 158 |
| 1.85 | 162 |
| 2.05 | 167 |

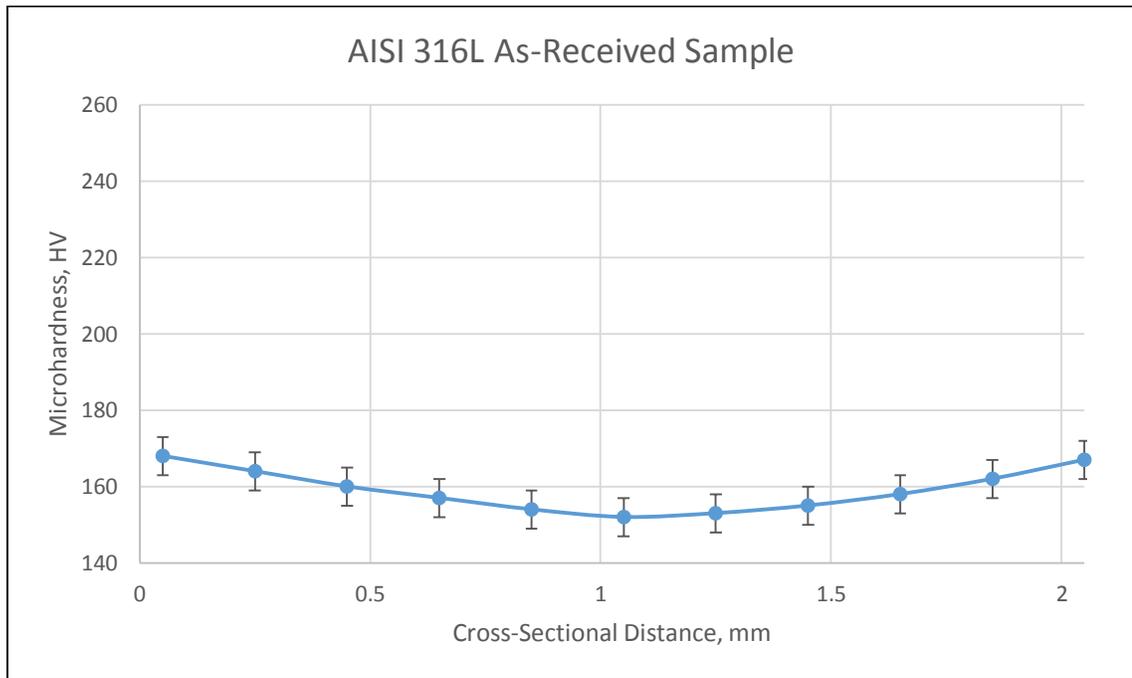


Figure A1: Graph of microhardness vs distance for as-received sample

Appendix B

Data and graph of nitrided sample for 8 Hours.

Table B1: Data of Microhardness Test for 8 Hours Nitrided Sample

| Cross-Sectional Distance (mm) | Microhardness (HV) |
|-------------------------------|--------------------|
| 0.05 | 191 |
| 0.25 | 175 |
| 0.45 | 167.6 |
| 0.65 | 164.1 |
| 0.85 | 163 |
| 1.05 | 162 |
| 1.25 | 164 |
| 1.45 | 179 |
| 1.65 | 185.6 |
| 1.85 | 193.7 |
| 2.05 | 202.1 |

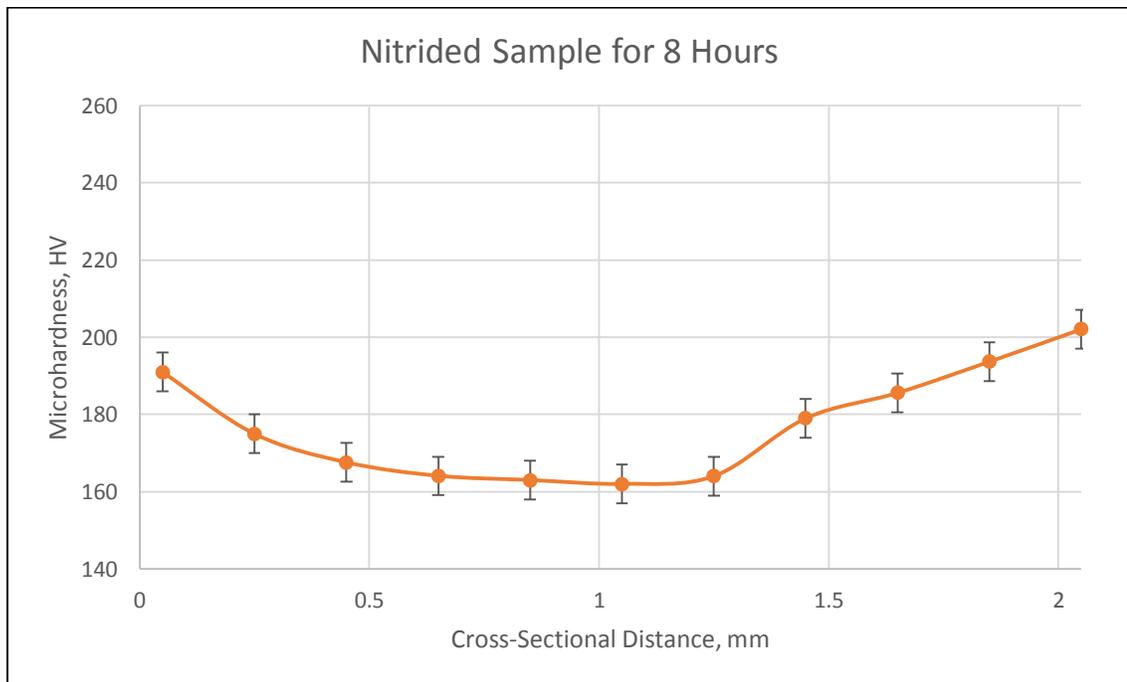


Figure B1: Graph of microhardness vs distance for 8 hours nitrided sample

Appendix C

Data and graph of nitrided sample for 15 Hours.

Table C1: Data of Microhardness Test for 15 Hours Nitrided Sample

| Cross-Sectional Distance (mm) | Microhardness (HV) |
|-------------------------------|--------------------|
| 0.05 | 229.5 |
| 0.25 | 194.6 |
| 0.45 | 190 |
| 0.65 | 181.9 |
| 0.85 | 178 |
| 1.05 | 175 |
| 1.25 | 181.1 |
| 1.45 | 182.3 |
| 1.65 | 196.4 |
| 1.85 | 203.4 |
| 2.05 | 230 |

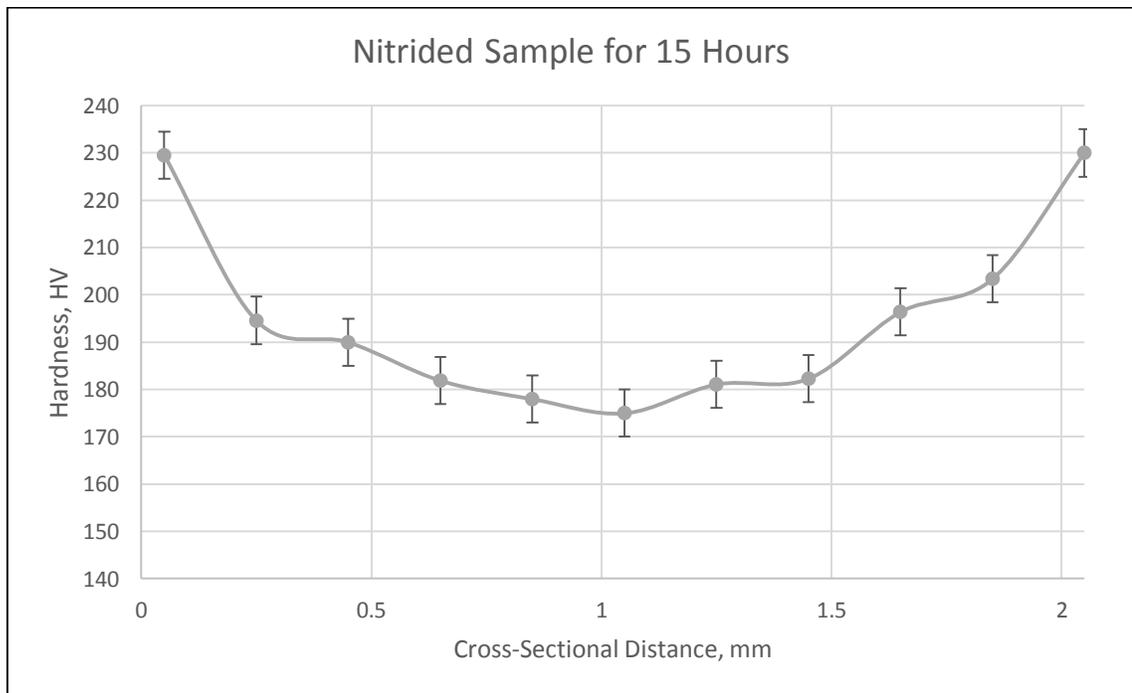


Figure C1: Graph of microhardness vs distance for 15 hours nitrided sample

Appendix D

Data and graph of nitrided sample for 20 Hours.

Table D1: Data of Microhardness Test for 20 Hours Nitrided Sample

| Cross-Sectional Distance (mm) | Microhardness (HV) |
|-------------------------------|--------------------|
| 0.05 | 245.1 |
| 0.25 | 231.8 |
| 0.45 | 197.6 |
| 0.65 | 187.1 |
| 0.85 | 182.5 |
| 1.05 | 180.1 |
| 1.25 | 193.3 |
| 1.45 | 208.1 |
| 1.65 | 212.3 |
| 1.85 | 231.1 |
| 2.05 | 242.8 |

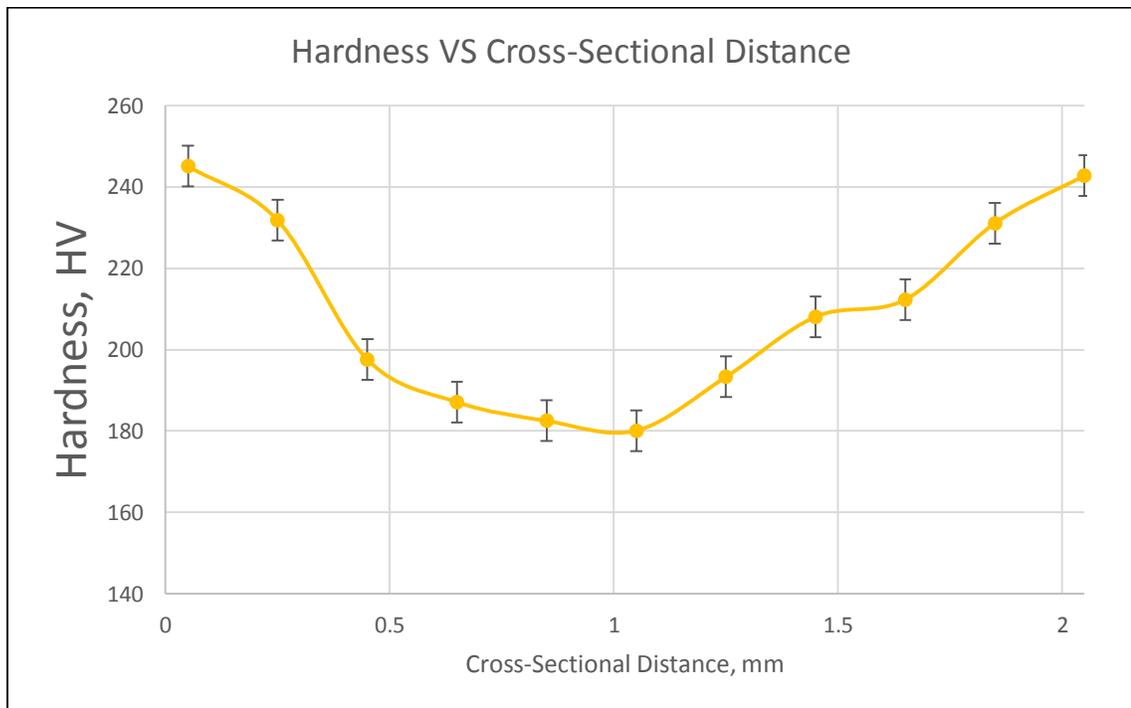


Figure D1: Graph of microhardness vs distance for 20 hours nitrided sample