Comparative Study on the Mechanical and Physical Properties of Un-nitriding and Nitriding of 430 Ferritic Stainless Steel

by

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

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CERTIFICATION OF APPROVAL

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TRONOH, PERAK
September 2013

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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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(Farhana binti Haris)

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ABSTRACT

This study compares the mechanical and physical properties of un-nitriding and nitriding of 430 ferritic stainless steel. The 430 ferritic stainless steel samples are nitrided via gas nitriding at high temperature of 1200^{o}C for 4 hours in purified nitrogen. Another part of the samples is nitride and quenched at the same time. The two samples were compared to the as received one. Martensite layer can be seen on the surface layer of the nitride samples while the nitride and quenched samples resulted in martensite and austenite phase. The hardness of the nitrided 430 ferritic stainless steel is higher than the as – received ferritic stainless steel. The nitride and quenched stainless steel sample shows a slightly higher in hardness than the nitride only. Thus, the nitriding process enhances the mechanical properties of the 430 ferritic stainless steel.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

ABBREVIATIONS AND NOMENCLATURE

BCC	: Body Centered Cubic
BCT	: Body Centered Tetragonal
EDX	: Energy Dispersive X-ray
FCC	: Face Centered Cubic
FESEM	: Finite Element Scanning Electron Micrograph
OM	: Optical Micrograph

CHAPTER 1

INTRODUCTION

1.1 PROJECT BACKGROUND

Stainless steel was generally invented in 1904 by a French scientist but he did not realize the corrosion properties of the metal. In 1911, a German scientist, Phillip Monnartz published his work on the corrosion resistant. On the following year, Eduard Baurer and Benno Strauss patented austenitic stainless steel in 1912. In 1913, Harry Brearly patented first martensitic stainless steel after he accidentally realized on one of the test sample did not rust while investigating on the ways to overcome rust in gun barrel. Therefore, he named it as ‘rustless steel’, but then the name is changed to a marketable name which is stainless steel [1].

The properties of stainless steel that is resistant to corrosion made it one of the highest demand materials in production and manufacturing industries. Stainless steel is widely used today in manufacturing of automotive bodies, building sculptures and even food packaging.

Ferritic stainless steel which contains 10.5 %– 30 % Chromium is a magnetic type steel which is easy to form and machine. For instance, ferritic stainless steel is used in mufflers, exhaust system and kitchen counters. Some of the application stated above needs high surface hardening in order to improve the wear resistance of the material [2]. Hence, it is important to improve the mechanical properties of the ferritic stainless steel to comply with the needs from industries.

1.2 PROBLEM STATEMENT

Ferritic stainless steel has better engineering properties as compared to the austenitic grades but it lose when it come in terms of corrosion resistance because of the low content of Nickel and Chromium . The magnetic and relatively low cost ferritic stainless steel is mainly used in decorative trim, automation and kitchen counters.

In this study, the as- received 430 ferritic stainless steel is low in strength. Thus, limiting the usage of the metal. Thus, nitriding process will be used in order to enhance the mechanical and physical properties of the 430 stainless steel.

1.3 OBJECTIVES

Based on the problem statement in section 1.2, the objectives to be achieved in this project are:

1. To compare the mechanical and physical properties of un-nitriding and nitriding 430 ferritic stainless steel.
2. To enhance the mechanical properties of the 430 ferritic stainless steel through nitriding process.

1.4 SCOPE OF STUDY

The scope of study is important in order to ensure the project is in the right track. In this research, the study mainly focuses on the experimental work. Firstly, the samples of 430 ferritic stainless steel will be cut to appropriate dimension using wire cut machine of 1.5mm thickness and 13mm radius. Then, the samples will be prepared for nitriding using the tube furnace. One of the samples will be quench and another one will be cooled to room temperature in the furnace. The sample will be observed using the optical microscopy (OM) and scanning electron microscopy (SEM) with Energy-Dispersive X-ray (EDX) Spectroscopy for physical properties. The mechanical testing for the material will only be focused on the hardness test using Vickers (Load = 100g) due to limitation of samples.

CHAPTER 2

LITERATURE REVIEW

Stainless steel is the most suitable material whenever corrosion is a concern in an industry. For instance, stainless steel is used in manufacturing of sterile instrument for food and medical purposes, oil rigs and nuclear reactor. Hence, in this chapter, theories and previous work of stainless steel and nitriding process will be explained.

2.1 Stainless Steel

Referring to Budinski stainless steel can be defines as alloys of iron, chromium of at least 10.5 % and other elements that is resistant to corrosion in any kind of environment. Stainless steel can be divided into three major microstructural components which are austenitic, ferritic and martensitic [3, 4, and 5].

Austenitic stainless steel has a face-centered cubic (FCC) cubic; ferritic has body-centered cubic (BCC) while martensitic has a body-centered tetragonal (BCT) iron [5, 6]. This shows that martensitic has the most hardness as compared to austenitic and ferritic due to the crystal structures. Austenitic stainless steel which contained iron, chromium, carbon and nickel, is the most popular stainless steels used in industries due to its tensile properties and durability.

Martensitic on the other hand, has lower ductility as compared to austenitic and ferritic stainless steel but they are usually heat treated to a high hardness [7， 8]. Ferritic contained low carbon content (usually $< 0.2\%$) and chromium content is in the range of

10% - 30%. Ferritic stainless steels are relatively resistant to stress corrosion tendency, good ductility and formability [9， 10]. It is usually used in automation industries like mufflers, and kitchen counters like sinks.

2.2 Nitiriding process

Nitriding on the other hand is a heat treatment process which case hardening. The process essentially hardened the outer surface of the materials and left the inside core to be soft. Nitriding process can be divided into three types – gas nitriding, salt bath nitriding and plasma nitriding.

In gas nitriding, the furnace is usually used to heat the metal. When nitrogen is supplied into the furnace it will react with the heated sample, thus creating nitride layer on the surface [11, 12]. In salt bath nitriding, molten salt is used as the nitrogen source and it used the principle of the liberation of nitrogen within the salt for diffusion in steel surface. Other than that, plasma nitriding or also known as ion nitriding, used the discharged plasma from the reaction of two gases to heat the steel surface and to supply nitrogen ions for nitriding [11].

When ferritic stainless steel is nitrided at high temperature in a furnace containing nitrogen – gas nitriding, the nitrogen will permeates into the metal and changing the microstructure of the metal. This will in turn influence mechanical properties of the metal – hardness and wear resistance.

2.3 Phase Transformation

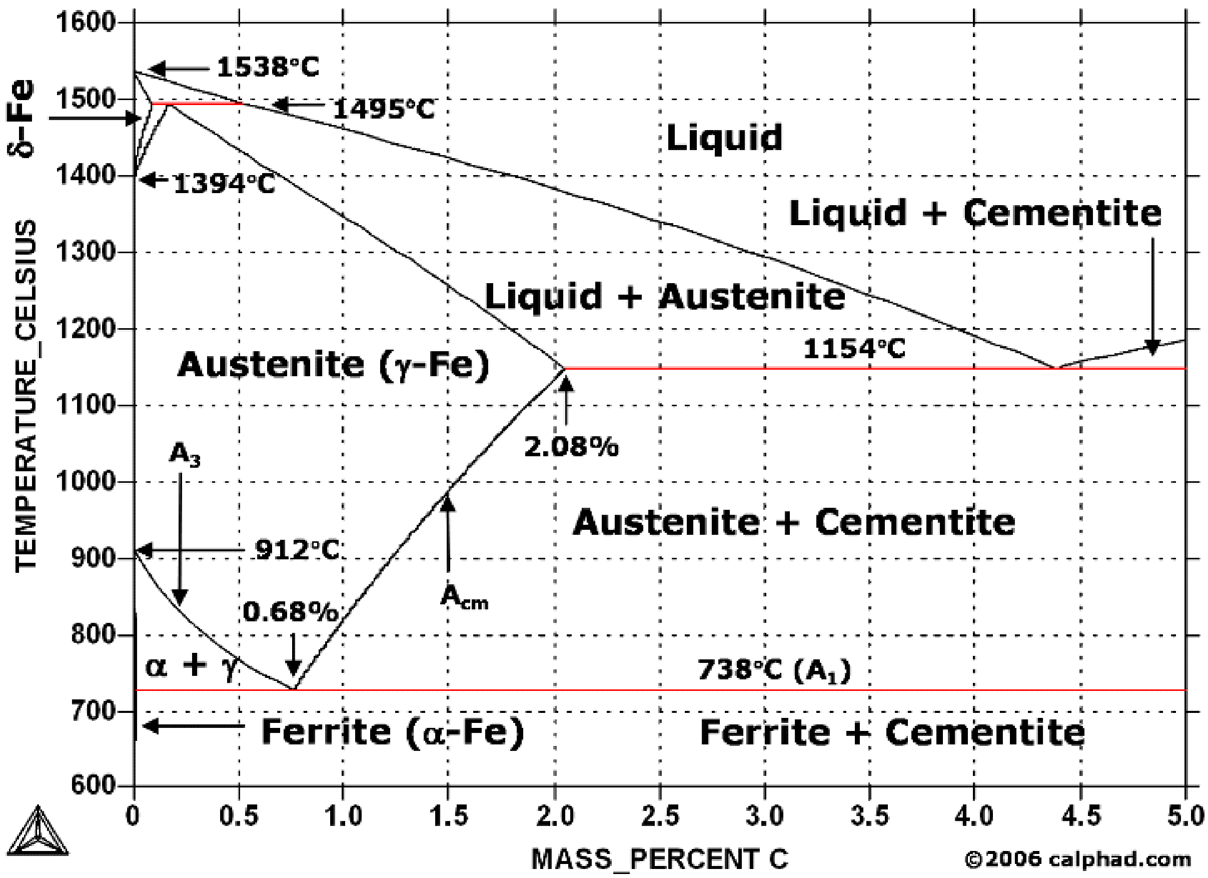


Figure 2.1: Fe-C phase diagram

Phase diagram is important in understanding the transformation of the phase of metal. Different metal relates to different phase diagram based on the chemical composition. For 430 ferritic stainless steel, phase diagram of Iron – Carbon is used as in Figure 2.1. The phase relates to the microstructure of the metal and influences the mechanical properties of the metal itself.

As the ferritic stainless steel is heated to a temperature above 750^{o}C, the ferrite phase will transform to austenite phase. When the metal is left to cool slowly at normal environment, the austenitic phase will return to its original phase which is ferrite. Martensite on the other hand, is a phase that is due to the rapid cooling of high temperature metal [6]. The sudden change of temperature hindered the diffusion of the carbon. Thus, martensite phase is formed. In this case, when nitrogen gas permeated into the metal through nitriding, the gas will hindered the diffusion of carbon thus forming martensite phase [13].

Martensite phase is known as the stongest phase as compared to austenite and ferrite phase. This is because martensite has a body centered tetragonal (BCT) which is strong because of the atomic arrangement and less slip in the system. As discussed in section 2.1, the ferritic has BCC crystal structure while austenitic has FCC structure.

CHAPTER 3

METHODOLOGY & PROJECT WORK

3.1 FLOW CHART

Figure 1 below illustrates briefly the methodology of the project.

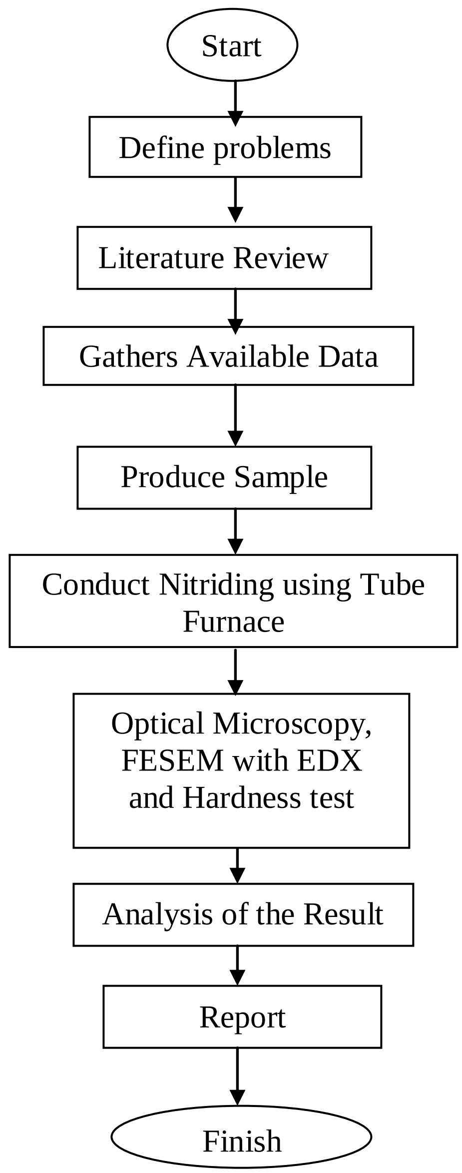


Figure 3.1: Flowchart for Methodology

3.2 Experimental Procedure

3.2.1 Nitriding Process

In this experiment 430 ferritic stainless steel was used. The steels were cut into 6 samples of 1.5mm thickness and 13.0mm diameter –Figure 3.2. Then, the samples are grounded, polished and cleaned with acetone before nitriding. The samples were divided into three parts – as-received, nitriding (quenched) and nitriding (un-quenched).The four samples were put into alumina boat before nitriding.

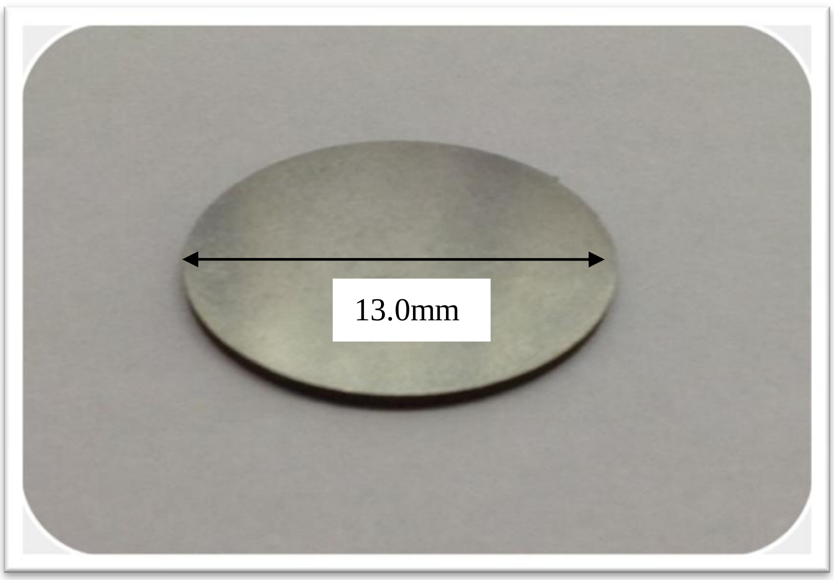


Figure 3.2: Disc-like sample

The nitriding process was carried out in a horizontal tube furnace which attached to a purified nitrogen (N_2) tank. A gas flow meter is attached between the tank and tube furnace to control the flow rate of the gas which is 1000cm^3/min. The other end of the tube furnace was attached to the Drechsel bottle to allow the excess air flown out as in Figure 3.3.

The samples in alumina boat were placed inside the tube furnace and were heated for 4 hours at 1200^{o}C at an increment of 3^{o}C/min. Two samples were quenched immediately in water at high temperature while the remaining was left to cool to room temperature inside the chamber.

Figure 3.3: Tube Furnace

3.2.2 Microstructural Characterization

All the samples were observed using optical micrograph (OM) and field emission scanning electron micrograph (FESEM) to investigate the microstructure of the samples as in figure. The samples were prepared before examining under the microscopy as in Figure 3.4.

Abrasive disc cutter was used to obtain the cross-section of the samples. Then, the samples were ground using emery paper and polished with Alumina paste. After that, the samples were etched with Kalling's reagent – 5g of Copper Chloride, 100ml of Hydrochloric acid and 100ml of ethanol, for about two minutes to reveal the microstructure [14].

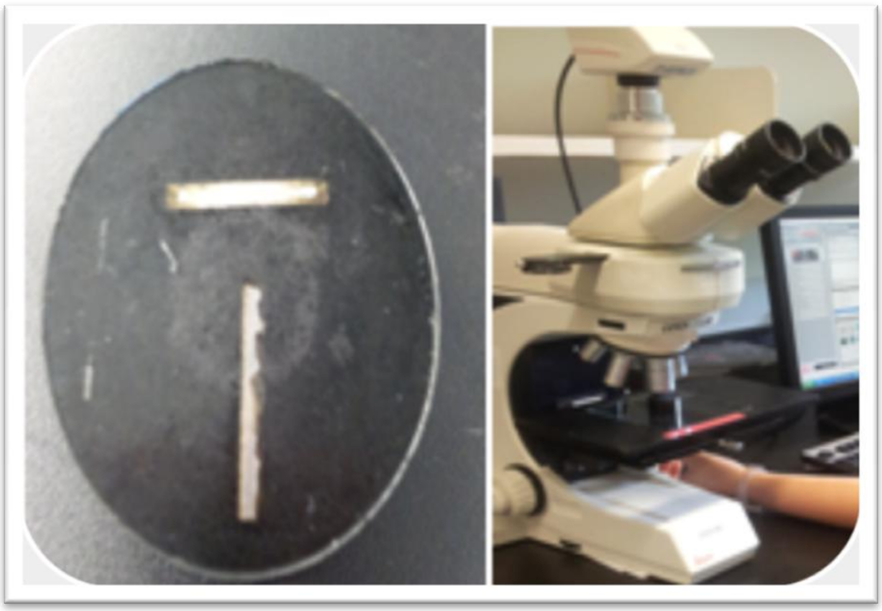


Figure 3.4: Mounted samples and Optical Micrograph

3.2.3 Elemental Analysis

The elemental was done to investigate the presence of Nitrogen in the metal via finite element scanning electron microscopy (FESEM) with energy dispersive x-ray (EDX) spectroscopy. A spot analysis was done on the metal near to the surface of the metal.

3.2.5 Hardness Test

Hardness test were done on the cross sectioned of the samples to investigate the improvement of the hardness of the samples. Micro Hardness Vickers of 100g was applied to the samples – HV100. The indentation was approximately taken at every 0.2mm of the samples.

3.3 Project Work

3.3.1 Key Milestones

The Key Milestones can be refereed on the table below:

Table 3.1: Key Milestones

Detail	Week
1. Submission of Extended Proposal	Week 6
2. Submission of Interim Draft Report	Week 13
3. Submission of Interim Report	Week 14
4. Submission of Progress Report	FYP 2 – week 7
5. Pre - SEDEX	FYP 2 – week 10
6. Submission of Draft Report	FYP 2 – week 11
7. Submission of Dissertation (Softbound)	FYP 2 – week 12
8. Submission of Technical Paper	FYP 2 – week 12
9. Oral Presentation	FYP 2 – week 13
10. Submission of Project Dissertation (Hard Bound)	FYP 2 – week 15

3.3.2 Gantt Chart

Table 3.2: Gantt Chart

		FINAL YEAR PROJECT 1															FINAL YEAR PROJECT 2													
	Week	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
1	Selection of Topics																													
2	Submission of Extended Proposal																													
3	Proposal Defence																													
4	Interim Draft Report Submission																													
5	Interim Report Submission																													
6	Progress Report Submission																													
7	Pre-SEDEX																													
8	Submission of Draft Report																													
9	Submission of Dissertation (soft bound)																													
10	Submission of Technical Paper																													
11	Oral Presentation																													
12	Submission of Project Dissertation (Hard Bound)																													
	Project Work																													
1	Research - Finding Data																													
2	Produce Sample and nitriding																													
3	Testing and Analysing																													

CHAPTER 4

RESULTS AND DISCUSSION

In this chapter, the nitrided samples were compared to the as received samples as well as quenched-nitrided samples. The samples were analysed in terms of the hardness and microstructure formed via the experiment and analysis conducted to study the influence of the penetrating the nitrogen into the metal. Figure 4.1 below shows the surface of metal as-received and after nitriding.

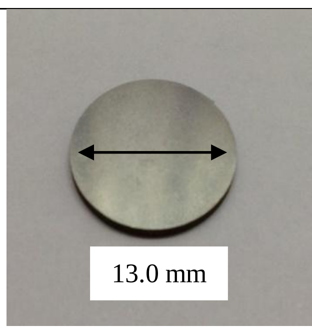
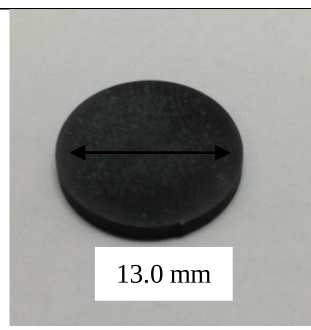
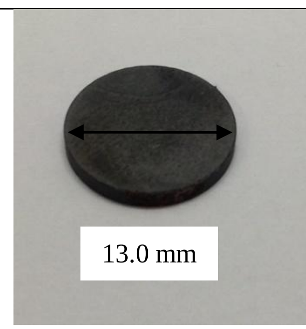
Sample A	**Sample B**	**Sample C**
		
As Received	After Nitriding (unquenched	After Nitriding (Quenched)

Figure 4.1: Surface of the metal as received and after nitriding.

4.1 Microstructure analysis

As discussed in section 3.2, the samples were prepared before proceed to undergone analysis experiment likes micrograph. The metals were analyzed for its microstructure at the cross section by using optical micrograph (OM) and finite element scanning electron micrograph (FESEM).

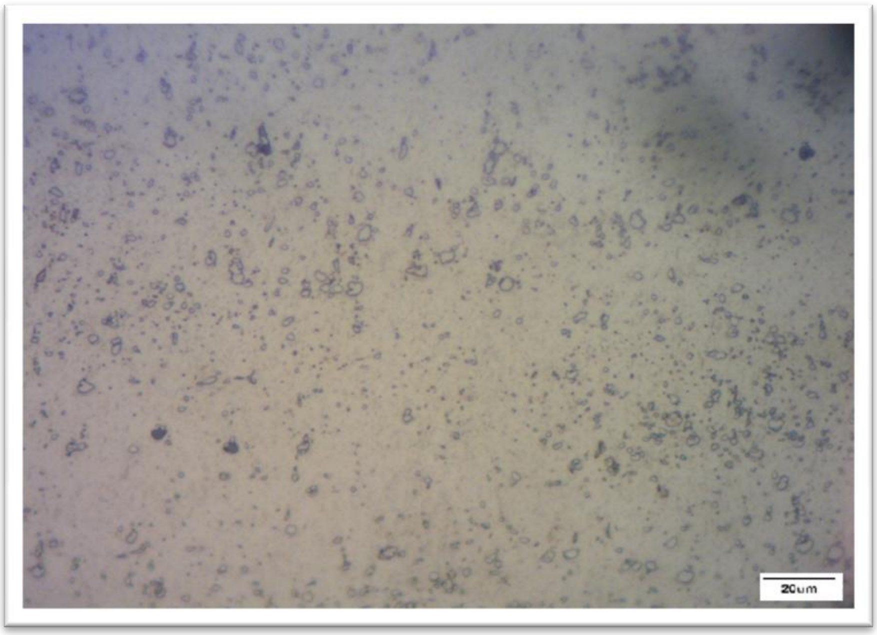


Figure 4.2: Microstructure of Sample A - OM

Figure 4.2 above shows the microstructure of the as-received 430 ferritic stainless steel using optical micrograph (OM). The microstructure shows a typically ferrite phase with the elongation of the grain due to the rolling process.

The strength and hardness of the metal is essentially depends on its microstructure. Based on the Fe-C phase diagram in Figure 2.1, as the ferritic stainless steel is heated approximately above

740°C, the will transform from ferrite to austenite phase as the sample is heated to 1200°C. As the stainless steel is cool to room temperature in the furnace, the martensite phase is formed.

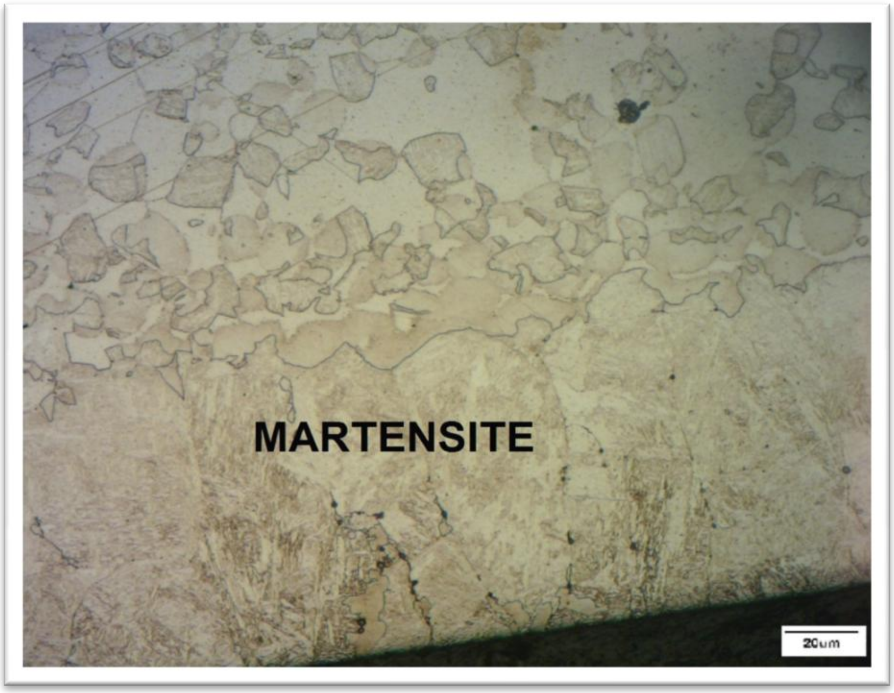


Figure 4.3: Microstructure of Sample B - OM

From Figure 4.3, it shows that there are needle-like shape which is martensite phase formed near the surface of the ferritic stainless steel after 4 hours of heating at 1200°C in nitrogen. The brownish colour revealed on the microstructure shows the nitrogen had penetrated into the metal. The center part of the metal retained some of the ferrite phase.

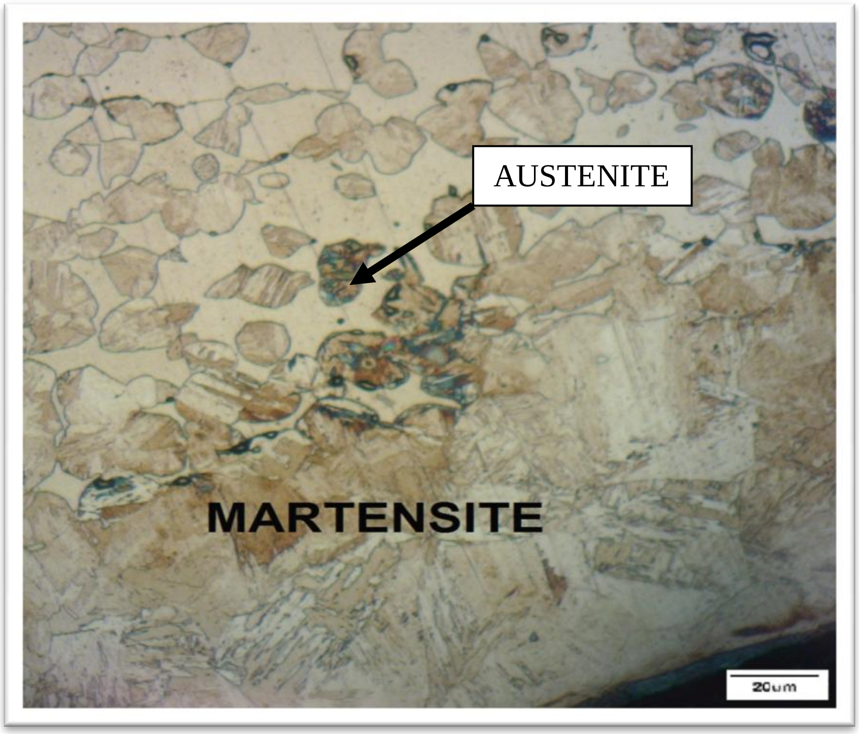


Figure 4.4: Microstructure of Sample C - OM

Figure 4.4 shows the microstructure of sample C which was nitrided and quenched in water. From the figure above, it shows that there was also formation of martensite since it was heated to high temperature in nitrogen. But at certain location the austenitic phase can also be seen in the microstructure above due to the rapid cooling process.

Image of microstructure using finite element scanning electron micrograph (FESEM) had also been taken at magnification of 1000. Figure 4.5 shows the microstructure of sample B using FESEM. Clear image of martensite formation is obtained. While in Figure 4.6, martensite phase and some part of austenite can be observed.

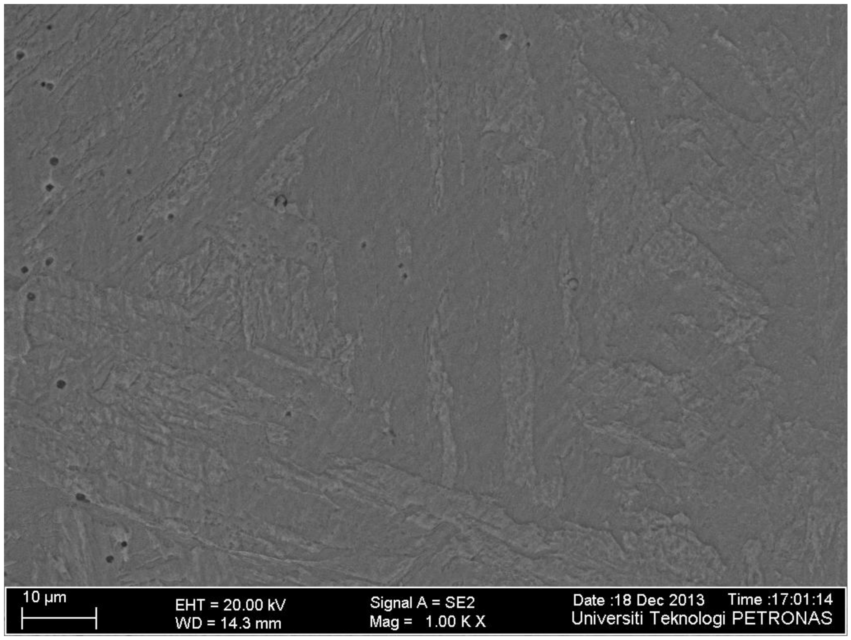


Figure 4.5: Microstructure of Sample B – FESEM

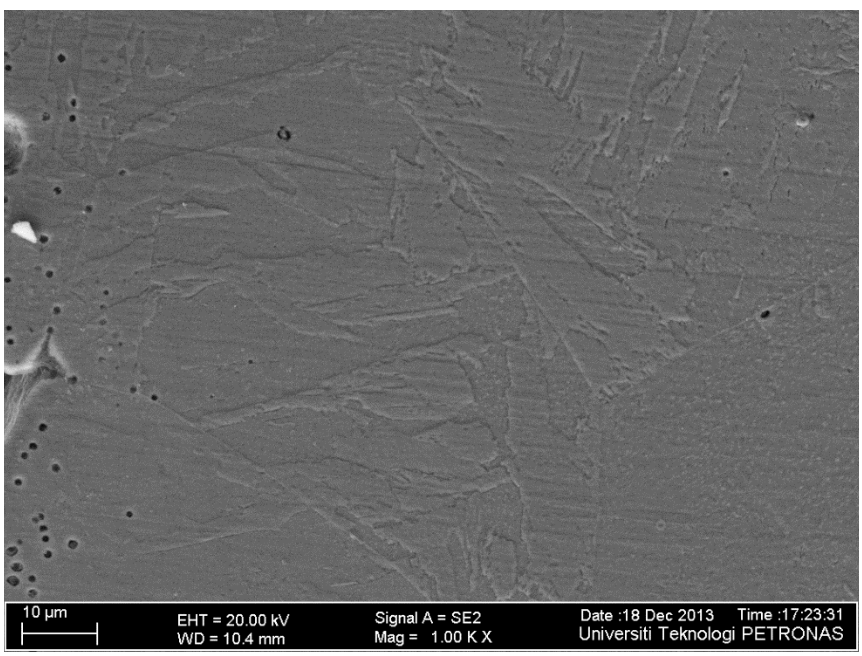


Figure 4.6: Microstructure of Sample C – FESEM

4.2 Elemental Analysis

In this part, elemental analysis was done to verify the penetration of nitrogen into the 430 ferritic stainless steel. Therefore, spot analysis using energy dispersive x-ray (EDX) spectroscopy near the surface was done. Table 5.1 below shows the weight percentage of elements at a point near the surface of the metal for sample B and C.

Table 4.1: Weight Percentage of Elements

Elements	Weight Percentage, %	
	Sample B	Sample C
Carbon,C	4.89	3.82
Nitrogen N	1.65	0.60
Chromium, Cr	16.30	16.55
Iron, Fe	77.16	79.03
Total	100	100

Based on the table above, it is verified that there were presence of nitrogen in the 430 ferritic stainless steel near the surface of the steel. Based on Callister, the martensite occur due to the rapid cooling that restricted the diffusion of carbon. In this case, although sample B is left to cool slowly to room temperature in the furnace, the presence of nitrogen inhibits the diffusion of carbon to the metal [6]. Thus, the part of metal which near to the surface – contain nitrogen, has martensite phase while the center part still remained the ferritic phase.

4.3 Hardness Test

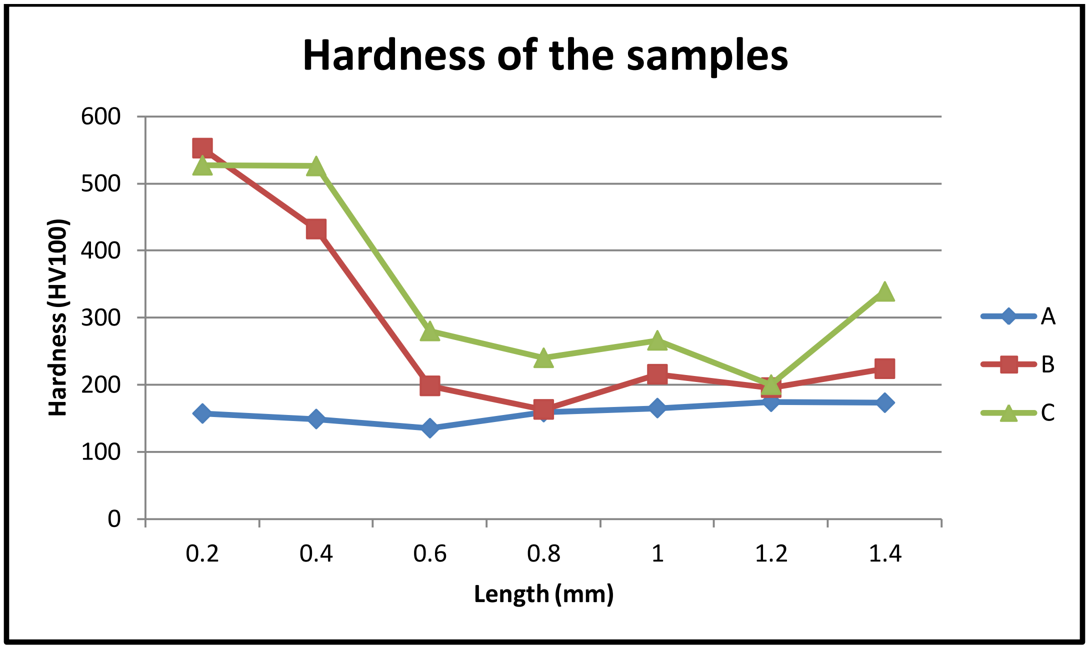


Figure 4.7: Hardness of the samples

Figure 4.7 shows the hardness of the sample A, B and C at every interval of 0.2mm. The applied load of the hardness Vickers is 100g – HV100. The hardness was done across the cross section of the disc-like 430 ferritic stainless steel. From figure above, as the length is moving towards the center of the metal, the hardness decreases and highest hardness lies on the both end of the metal surface.

Based on the graph, it shows that sample A which was as-received sample has the lowest hardness as compared to the nitride samples – sample B and C. Sample C which has been nitride and quenched has slightly higher hardness as compared to sample B.

4.4 Discussions

Based on the result obtained, the first and second objectives of the project are achieved. The mechanical properties of the 430 ferritic stainless steel have been successfully achieved through the process of nitriding. This can be verified based on the microstructure and hardness test result.

The hardness of the nitrided 430 ferritic stainless steel sample is higher than the unnitrided one. This is because the interstitial of the nitrogen into the metal through the nitriding process has changed the atomic arrangement of the metal from body centered cubic (BCC) to body-centered tetragonal (BCT) which was the martensite phase [13]. By referring to section 5.1, the martensite phase can be observed in nitrided samples – sample B and C near to the surface of metal.

Martensite is the hardest and strongest as compared to austenitic and ferritic. This is due to the less slip system which the dislocations move. As the hardness increases, the ductility of the material will decreases due to brittleness. Since, the center part of the metal still retaining the ferritic phase, hence, it will overcome some of the brittle effect to the metal.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

In a conclusion, the hardness property of the nitriding 430 ferritic stainless steel is higher than the as-received sample. The mechanical property of the steel is enhance through the process of nitriding by 30%.

The test shows that the nitrogen permeated into the metal and changes the microstructure at the surface layer of the 430 ferritic stainless steel from ferrite to martensite. The martensite phase formed at the surface layer strengthens the metal. This is due to the atomic arrangement of the crystal structure which is body centered tetragonal (BCT).

The nitriding and quenched ferritic stainless steel sample has higher hardness than the nitriding without quenching sample. The high hardness value of the nitride 430 ferritic stainless steel is due to the diffusion of nitrogen into the steel which caused by the locking slip system. Hence, it is recommended for future studies, the nitriding sample will be compared to the quenched sample so that the effect on the microstructure and hardness can be determined.

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