Tribological Properties Analysis on Nitrided

AISI 316L Stainless Steel

by

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SEPTEMBER 2011

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
Mechanical Engineering Programme
Universiti Teknologi PETRONAS
in partial fulfilment of the requirement for the
BACHELOR OF ENGINEERING (Hons)
(MECHANICAL ENGINEERING)

Approved by,

(AP Dr.Patthi Bin Hussain)

UNIVERSITI TEKNOLOGI PETRONAS
TRONOH, PERAK
September 2011

CERTIFICATION OF ORIGINALITY

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

ABDUL RAHMAN BIN MOHD ZAFRULLAH

ACKNOWLEDGEMENT

I would like to take the opportunity to acknowledge and thank everyone that has given me all the supports and guidance throughout the whole period of completing the final year project. Firstly, many thanks to the university and the Final Year Project coordinators that have coordinated and made the necessary arrangements, especially in terms of the logistics, for this study.

I must also acknowledge the endless help and support received from my supervisor, AP. Dr.Patthi Hussain throughout the whole period of completing the final year project. His guidance and advices are most appreciated. Apart from that, many thanks to Mr Askar Triwiyanto, postgraduate student that help me from the beginning of the project until its completion. He guided me not only in theory, but with practically demonstration as well as how to deal with technical and non-technical problems. He had much bring me a great strength to complete this project successfully.

Finally many thanks to my fellow colleagues for their help and ideas throughout the completion of this study. Thank you all.

ABSTRACT

AISI 316L stainless steel have been treated by surface hardening technique, which is low temperature nitriding gas treatment in horizontal tube furnace. The environment gas for this treatment is ammonia, which become the source of nitrogen gas that purged into the furnace where the steel is located. The temperature of this treatment is at 450 °C with the variable of 2, 5 and 8 hours nitriding time. The purpose of this investigation is to improve the wear resistance and surface hardness of the stainless steel as the element of tribological properties analysis. Theoretically, the low temperature treatment is to avoid the formation of nitride and carbide precipitation because it can reduce the corrosion resistance of stainless steel. Pin-on disc wear test at dry sliding condition become the standard test to analyze the wear resistance of this nitrided steel according to the ASTM G99-95a standard. The further analysis on tribological properties of nitrided steel are conducting microhardness assessment by Vickers hardness test on the surface of treated steel to identify the improvement of surface hardness by the formation of nitrided layer. This investigation also strongly support by conducting nitrided layer morphology by utilizing Field Emission Scanning Electron Microscope (FESEM) instrument which is to measure the thickness of nitrided layer. Surface morphology of worn region by FESEM also investigated to understand the wear mechanism characteristics of nitrided steel. The results showed that the nitrided layer become more thicker through the extending of time treatments and relatively improved the surface hardness. The nitrided layer characteristic that support the protection oxide layer and resist the wear mechanism is confirmed where the wear resistance improved from the result of coefficient of friction by the pin-on disc wear test with the value obtained become lower as the time of treatment is extended. Surface morphology by FESEM investigation on worn region after wear test for untreated and nitrided steels indicates how the wear mechanism influenced the improvement of wear resistance. Untreated samples experienced severe wear while nitrided steel presented only slight abrasion with shallow and narrower wear track.

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CHAPTER 1

INTRODUCTION

1.1 Background of Study

Tribology is the scope of study that relates with the design, mechanism of friction, lubrication and wear. Most of mechanical system performances deal with surface interactions and contacts. This intimate contact will generally lead to wear (Ashby, 2005). Therefore, mechanical design becomes an important factor to experience this phenomenon. When selecting materials, the aim is to minimize wear for particular operation's equipment.

AISI 316L stainless steel is a material that widely used in the engineering stream because of its characteristic with high resistance oxidation and corrosion resistance. However, there are some operation in industry application that bring this steel exposing wear mechanism for instances, in automotive parts like camshafts, cam followers, chemical and oil and gas industries. The introduction of nitriding treatment as the surface hardening technique is recognized where the treatment could improve the wear resistance and surface hardness of the steel during the operation.

In order to understand the wear resistance and surface hardness improvement, the tribological properties of nitrided 316L become the main analysis. The major element involves in the analysis is the wear resistance where this can be practically investigated by dry sliding condition wear test. The test method could be used is pin-on-disc wear test according to ASTM G99-95a and the result of wear resistance in term of coefficient of friction and wear rate from the test become the medium of analysis.

One of the variables that can be applied in the nitriding treatment is the time variables. Difference time of treatment may result in difference of wear resistance under the dry sliding condition wear test. Moreover, the tribological properties on nitrided steel with difference time of treatment can further be analyzed by microhardness assessment, nitrided layer morphology and surface morphology of worn region. Thus, all the analysis obtained may bring the best understanding on how the tribological properties of nitrided steel be influenced by the nitriding treatment.

1.2 Problem Statement

Crankshaft bearing of an automobile is an example where this type of rotating or reciprocating machinery experiences the mechanism of friction, lubricating and wear between materials. AISI 316L stainless steel is one of the materials designed for the crankshaft bearing parts. In order to minimize the friction and wear during operation, nitriding treatment on the steel parts becomes the surface hardening technique to bring better performance.

1.3 Objective

- To improve wear resistance and surface hardness of AISI 316L stainless steel by nitriding treatment as the surface hardening technique

1.4 Scope of Study

Scope of study of this project is mainly in the form of laboratory experiments. Basically, the purpose of the experiments is to analyze the tribological properties of nitrided AISI 316L stainless steel by improving the wear resistance and surface hardness using low temperature gas nitriding treatment with difference in time of treatment. The wear resistance will be analyzed under dry sliding condition wear test. The test method will be used is wear testing with pin-on-disc apparatus where the studies refer to the standard of ASTM G99-95a. Surface hardness, nitrided layer morphology and surface morphology of worn region characteristics also become part of the study to strengthen the tribological properties analysis.

1.5 Relevancy of Project

From this project, nitriding treatment as a surface hardening technique may bring the significance as a scientific reference to heat treatment industries in improving their process design. This project may provide a wider application to the austenitic stainless steel users because of significant improvement in wear resistance and surface hardness.

1.6 Feasibility of Project

This project is analyzed to be feasible where the laboratory equipments are 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 Introduction

In industry, wear happened under the aspect of working environment. For example, rotating and reciprocating parts, sliding, impact load and temperature. The result of wear may lead to loss of dimension from plastic deformation by the sliding surfaces (Surface Engineering, 2011). In operational equipment's part, for example stainless steel material, there are oxide film and lubricant that prevent from the contact of two solid surfaces. However, the breakdown of oxide film under mechanical loading for instance could bring to the contact of solid surfaces at the region (Ashby, 2005). This situation is where the solid surfaces experience wear.

AISI 316L stainless steel become the chosen material in the engineering industry because of its good ductility, weldability and better corrosion resistance. Due to the inherent austenitic structure, this material is poor to the effect of wear and low hardness, which result in poor tribological properties (Haruman et al., 2006). Wear on the steel surface could lead to the failure of the operational parts and to the operational equipments. Therefore, one of the approaches to improve wear resistance and surface hardness of the steel is by nitriding treatment where its offer the benefits of high dimensional stability (Subbiah et al., 2005).

Nowadays, nitriding treatment on austenitic stainless steel by low temperature nitriding (LTN) become important studies and widely used in the industrial applications (Li et al, 2003). By this surface hardening technique, the parts that involved with wear phenomenon are expected to withstand for a longer operation period (NSK Group, 2011). Wear resistance improvement produced by low-temperature nitriding can satisfy the high demand for austenitic stainless steels in many applications. For example, the operational parts are including spindles, ball valves, and butterfly valves for marine environments, safety valves for the chemical industry, and ferrules for couplings in severe environments. Other examples are hip joints, knee joints, and other orthopedic implants (Bell et al, 2002).

2.2 An Overview of Austenitic Stainless Steel

Austenitic stainless steel is a group of stainless steel, which is an alloy material principally, contains iron (Fe) and minimum of chromium (Cr) and nickel (Ni). Due to the present of minimum chromium compositions 10.5%, this material becomes the better corrosion resistance (International Stainless Steel Forum, 2011). The existing of chromium will form the passive layer which is known as chromium oxide (Cr_2O_3) when reacts with oxygen in the environment. This protection layer brings the steel surface prevented from corrosion mechanism to the environment (Fontana, 1987). Moreover, by adding the molybdenum (Mo), it can increase corrosion resistance in particularly to the pitting corrosion (International Stainless Steel Forum, 2011).

Nickel with minimum of 8% in the austenitic stainless steel composition can stabilize the austenitic structure of iron where the steels become non-magnetic and less brittle at low temperature (Callister, 2007). 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) (International Molybdenum Association, 2011). 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 (Dyson, 2011)

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 (TATA Steel, 2011). 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.

2.2.1 Grades of Austenitic Stainless Steel

Austenitic stainless steel also in the group of 300 series stainless steel. In this group, 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 (Tverberg, 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% (Roll, 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 (Stainless Steel Processing, Inc, 2011). 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.

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 (Stainless Steel Processing, Inc, 2011).

2.3 Concept of Nitriding

Nitriding is a surface hardening technique by the diffusion of nitrogen into the surface layers and the change of chemical compositions of the steel (Elgun, 1999). 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. (Leppänen et al, 1999, Subbiah et al., 2005)

There are different methods of nitriding process. Salt-bath nitriding is a method with the nitrogen in the cyanide salt as medium for nitriding (Hamdy et al, 2011). 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. For this project, author will utilize the gas nitriding methods, which the equipment facilities are provided in the university laboratory. Ammonia gas will be used to become the medium for denoting nitrogen.

2.4 Gas Nitriding Process

In early 1920's, gas nitriding process had begin to be used in the industry. 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 (Elgun, 1999). This process also can easily form a hardened layer on the surface of material and it is not expensive as those newly developed technologies (Triwiyanto et al, 2009).

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 (Mridha, 2006). 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 (Haruman et al, 2006).

In order to accelerate diffusion on austenitic stainless steel, the steel will be treated at relatively high temperature, about 570 °C (Bell et al, 2002). However, the formation of chromium nitride/carbide could occur during the diffusion at high temperature. As the results, chromium nitride/carbide might be precipitated into the grain boundary and the passive layer which is chromium oxide (Cr_2O_3) will unable to be produced and reduced the corrosion resistance property of stainless steel (Triwiyanto et al, 2009). This phenomenon is known as sensitization effect.

2.5 Low Temperature Nitriding Gas Treatment

The efforts to avoid the sensitization effect relatively with high temperature treatment have done by Zhang and Bell (2002). They have investigated the low temperature nitriding technique where it was found that a nitrided layer is formed in AISI 316L with plasma nitriding technique. The thickness of the layer could be formed up to 20μm at temperature around 400 °C. The characteristics of the layer show that it has very high hardness and excellent wear resistance, as well as very good corrosion resistance.

This gives strong evidence that the nitrogen layer could be produced at low temperature and the term for the nitrogen layer is an expanded austenite (γ_N) (Haruman et al, 2006). Besides that, the nitriding layer formed containing a new type nitrite phase with new phase composition is also known as S-phase (Toshkov et al, 2007). This phase is characterized with its good mechanical properties and acceptable corrosion resistance (Hamdy et al, 2011).

Even though the formation of chromium nitride as strengthening effects is eliminated under the low temperature nitriding, super saturations of the interstitial nitrogen species in austenite matrix become the alternative ways to give the strengthening effects. (Triwiyanto et al, 2009)

In previous research, some variables could be controlled to investigate the effects. For temperature variables, the different in temperature treatment could give the indications on how nitrided stainless steel surface possess the susceptibility to the corrosion attack relates with the sensitivity effect produces by the particular material (Haruman et al, 2006)

Time variables of treatment also could be analyzed in this low temperature nitriding treatment. The case depths of the nitriding layer become the relation with the time variables (Subbiah et al, 2005). There are different case depth could be identified under the nitrided layer morphology. Moreover, the time variables may bring us to have the understanding in the improvement of surface hardness for each different time of treatment. For this project, author will choose the time variables of nitriding treatment to relate with the improvement of wear resistance and surface hardness of nitrided steels.

2.6 Tribological Properties Analysis

Analyzing the tribological properties of nitrided AISI 316L stainless steel with different time of treatment is the main investigation for this project. The analysis could give more understanding on how the nitriding treatment provides the wear resistance and surface hardness improvement of the steel. The analysis involves by conducting wear test, microhardness, nitrided layer morphology and surface morphology of worn region assessment. For the wear test analysis, the pin-on-disc wear test with dry sliding condition could be used to obtain the coefficient of friction of nitrided steels as the results for analyzing the wear resistance. For the microhardness assessment, surface hardness of nitrided steel over the distance from the surface is required in order to present the depth profile of microhardness. Meanwhile, nitrided layer morphology is an approach to measure the thickness of nitrided layer and relates with the extending time of treatment. For surface morphology of worn region, the wear mechanism could be explained and analyzed the characteristic of nitrided steel when experiencing the wear phenomenon.

There are some investigations regarding the tribological properties analysis on nitrided AISI 316L stainless steel with difference time of treatment by several investigators. Haruman et al., (2006) has completed the investigation with low temperature fluidized bed nitriding on AISI 316L at 400°C, 450 °C and 500°C and the time variables for each temperature are 1, 3 and 6 hours. The results obtained bring the significance improvement on surface hardness of ~1350 $HV_{0.5}$ at 500°C with 3 and 6 hours. The achieved value of surface hardness improvement also same for 450°C at 6 hours treatment. For 500°C (3 and 6 hours), the improvement can be explained due to the combined hardening effect by S-phase and formation of chromium nitride (CrN). Whereas, for 450°C (6 hours), the improvement was due to the formation of mono S-phase layer on the surface. Those treatments also performing the low wear rate during the test. The friction coefficient of 450°C (6 hours) sample possessed low coefficient compared to others nitrided sample. Moreover, SEM investigation on the wear track of the 450°C (6 hours) sample revealed that the surface only suffered mild wear although the S-phase layer were smeared by relatively high sliding friction.

Another investigations was done by Triwiyanto et al, (2009) by using the same material termochemical treatment furnace with Haruman et al., (2006) experiments, but the treatments conducted are nitriding, carburizing, nitrocarburizing and hybrid treatments at 450°C (2, 5 and 8 hours) . However, only nitriding treatment results obtained will be highlighted in order to relate with author's project. From the nitrided layer morphology conducted by Field Emission Scanning Electron Microscopy (FESEM), the nitrided specimen has a thickness nitrided layer between 3.26 to 8.35 μm for 450°C (2, 5 and 8 hours).

From the depth profiles of microhardness obtained by the investigation, the surface hardness for 450°C (8 hours) decreasing moderately from 0 to 10 μm which is from 1600 to 1400 $HV_{0.5}$ respectively. This result could relate to the thickness nitrided layer obtained from FESEM and the nitrogen diffusivity characteristic, where the density of atomic nitrogen supersaturated into the surface decreasing moderately through the distance from the surface.

Wear resistance analysis also become the element for this project investigation on tribological properties of nitrided AISI 316L stainless steel. There are several investigations done to analyze the wear resistance of nitrided steel (Podgornik, et al., 1999, Wang et al., 2000 and Y.Li et al., 2011). In theory, coefficient of fraction is the ratio of the force that maintains contact between an object and a surface and the frictional force that resists the motion of the object. When two materials are placed in contact, any attempt to cause one of the materials slide over the other is resisted by frictional force (Ashby, 2005). F_s is the force that will cause sliding to start, related to the force P acting normal to the contact surface. Where μ_s is the coefficient of static friction.

$$F_s = \mu_s \mathrm{P} \tag{1.1}$$

Then, the limiting frictional force will decrease slightly and the coefficient of kinetic friction, μ_k take places.

$$F_k = \mu_k P \tag{1.2}$$

Thus, from the pin-on-disc wear test, the result of coefficient of friction could indicate the wear resistance of nitrided steel and compare with the samples in different time of treatment.

From the wear test also, the worn region also can be investigated through the surface morphology analysis. This is to understand what the mechanism took place during the wear, and how it influence the wear resistance of the nitrided samples. The typical wear mechanism characteristic for austenitic stainless steels sliding against steels are adhesive and abrasive wear mechanism (Y.Li et al, 2011). While for nitriding steel, the investigations done by (C.X.Li et al., and Hashemi et al.,) showed that the characteristics of wear mechanism through the surface morphology analysis were shallow, narrower and superficial wear track, as well as abrasive wear which was dominated on the surface.

Therefore, from the investigation done by several investigators relate with tribological properties analysis, this project could be done followed the previous experimental setup, and result analysis to achieve the standard result for scientific reference and surface improvement application.

CHAPTER 3

METHODOLOGY

3.1 Introduction

In this chapter, the research method that will be used is explained in section 3.2. Sample preparation is discussed in section 3.3 and how the sample being treated with low temperature gas nitriding treatment is presented in section 3.4. In section 3.5, the pin-on-disc wear test is explained accordingly. Characterization techniques, which include wear resistance analysis, microhardness assessment, nitrided layer morphology and surface morphology of worn region, are discussed in section 3.6.

3.2 Research Methodology

In this study, AISI 316L austenitic stainless steel is used as a material for analysis. This material will undergo low temperature gas nitriding treatment in different treatment time and constant temperature. Period of treatment time varies at 2, 5 and 8 hours. The temperature is set 450 °C. In this experiment, 50% of ammonia gas and 50% of nitrogen gas will diffuse in thermal activated environment. Pin-on- disc wear test will be performed according to the ASTM G99-95a standard. Then, the wear resistance will analyze from the results of coefficient of friction after wear test. Microhardness assessment will be conducted by Vickers hardness test to obtain surface hardness values for each sample with different time of treatment. For nitrided layer morphology and surface morphology of worn region assessment, Field Emission Scanning Electron Microscopy (FESEM) instrument will be utilized.

The flow chart in Figure 1, which is the iterative process of the experiment and the investigation will be described accordingly. Data analysis and final result section are discussed later in the next chapter.

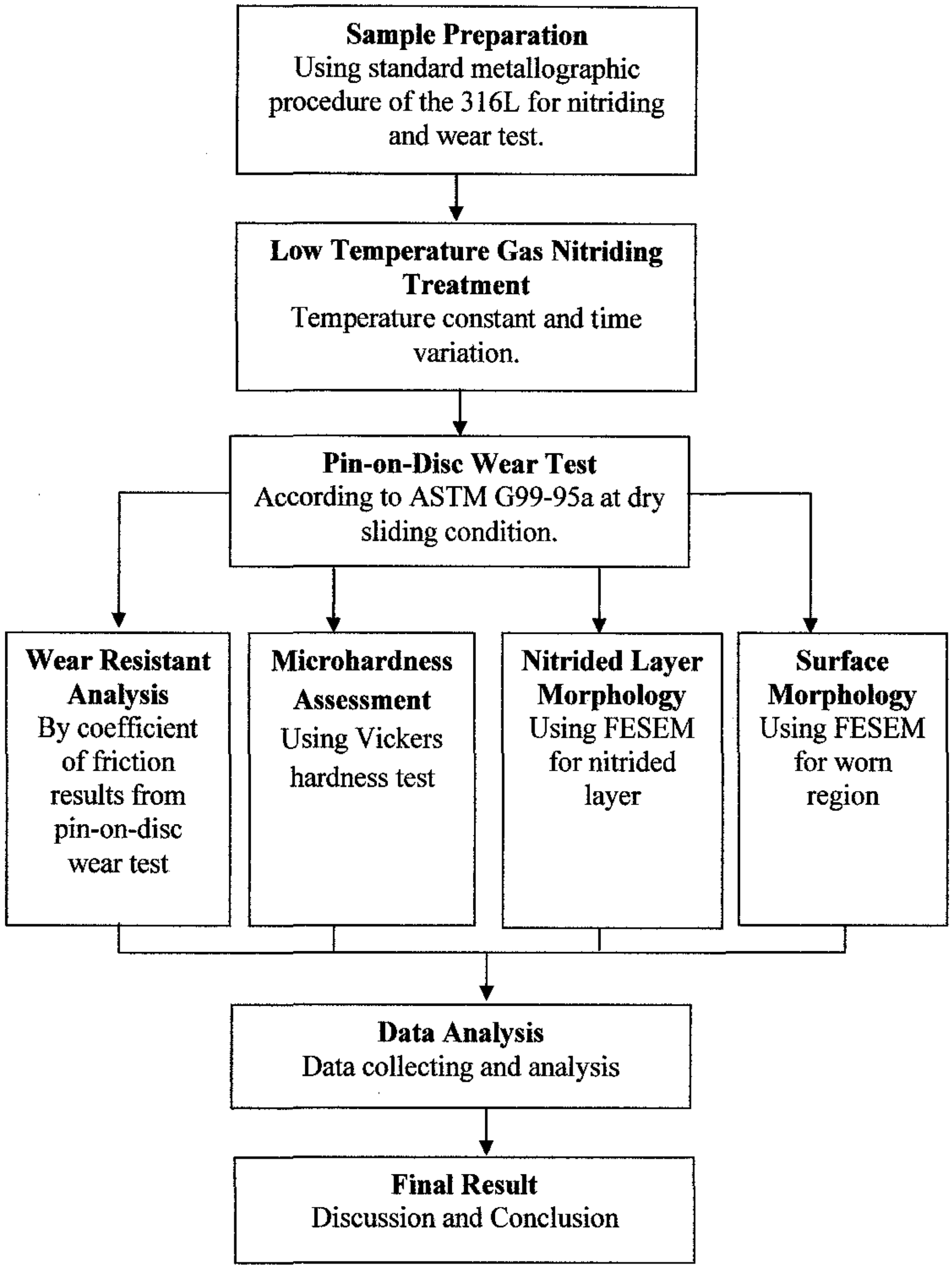

Figure 1: Flowchart of iterative process for experimental work

3.3 Sample Preparation

The material used is AISI 316L stainless steel and the steel supplied in the form of rod with the diameter of 50.3 mm and thickness of 40mm. Abrasive cutter machine is used to cut into ~6mm thickness disc for four samples. The disc samples are altered by conventional lathe into the dimension exactly 50 mm x 6 mm each. The 316L pin dimension required with diameter of 6mm and thickness of 12 mm for four samples. The pin is cut from 50.3 mm x 15 mm 316L rod by EDM wire cut. Those four pins and discs are required for undergo the wear test as be standardized by ASTM G99-95a. Then all the samples surface are ground on 120, 220, 500, 800, 1000, 1200 grit SiC papers, and then polished using 1 μm Al_2O_3 pastes to the mirror finish. Refer to **APPENDIX A.**

3.4 Low Temperature Nitriding Gas Treatment

After sample preparation is completed, all the samples is cleaned using ultrasonic cleaning to remove unwanted contaminants. The samples that will undergo nitriding treatment will be immersed in HCl (2 M) solution for 15 minutes duration to remove the native oxide film that commonly forms on austenitic stainless steel. Table 1 shows the set of samples that be categorized for nitriding treatment and wear test.

Table 1: Sample classification with different treatment.

Sample	Type	Treatment
UN	Pin	Untreated
	Disc	
2N	Pin	2 Hours Nitriding
	Disc	
5N	Pin	5 hours Nitriding
	Disc	
8N	Pin	8 Hours Nitriding
	Disc	

The sample that will undergo nitriding treatment is quickly placed on the quartz (for disc) and alumina boat (for pin) after being immersed and placed in the horizontal tube located in the Carbolite CTF Tube Furnace (Figure 2 (b)). Then the cycle of nitriding is set through the furnace controller. For the segment 1, the target temperature is set to be

450 °C from the room temperature with the rate of 5 °C/min. In segment 1, nitrogen will be purged into the furnace. This is because to maintain the environment inert, where the oxidation of samples and furnace component can be prevented. Moreover, nitrogen purging also prevent potentially explosive mixture when the ammonia begin to purge into the furnace. The segment 2 is set for 2, 5 or 8 hours dwell time (time of variation of treatement) after the temperature of segment 1 is achieved. In segment 2, the ammonia gas will be purged together with nitrogen.

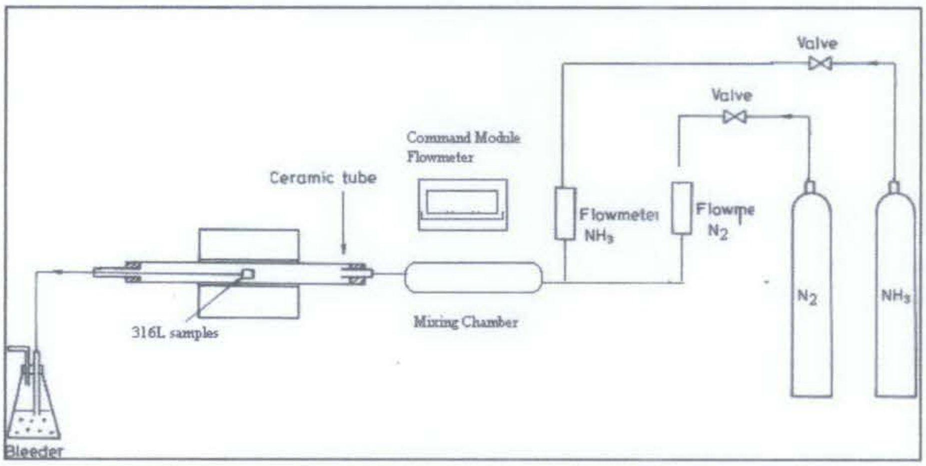

Figure 2 (a): Schematic illustration on nitriding equipment setup

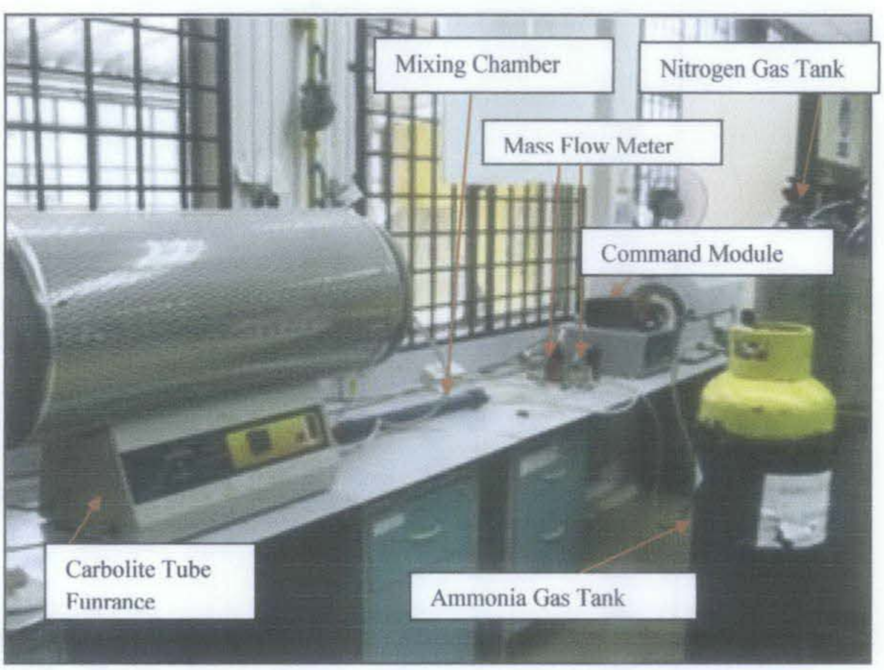

Figure 2 (b): Nitriding equipment setup at laboratory.

The amount of ammonia and nitrogen gas is set to be 50% each. This amount is obtained by setting up the Command Module for flow meter of ammonia and nitrogen gas for 0.3 Standard Liter per Minute (0.3 SLPM) each. Those gases will mix in the mixing chamber before purge together into the tube furnace. Then, segment 3 is set for target temperature 30 °C from 450 °C after complete segment 2 as a cooling cycle. The rate for this segment is 5 °C/min. Refer to **APPENDIX B.**

3.5 Pin-on-Disc Wear Test

Wear test by pin on disc apparatus use to analyze the coefficient of friction of prepared samples according to the standard; ASTM G99-95a. For this project, the wear test is in the dry sliding condition. Ducom TR-701-M6 Multi Specimen Tester machine (Figure 3) is used for pin-on disc wear test. The load values for the test are 17 N with the speed of 75 rpm and sliding distance of 300 m (for 30 minutes time of operation). The temperature is the room temperature and the atmosphere is set to be the laboratory air. Refer to **APPENDIX C.**

Figure 3: (a) Ducom TR-701-M6 Multi Specimen Tester machine and (b) pin and disc holder.

Below are the procedures to perform wear test:

1) Remove all dirt and foreign matter on the samples
2) Insert the disc in the holding device for perpendicular axis.
3) Insert the pin in its holder and perpendicular to the disc.
4) Set the applied load, speed and time of operation (set for sliding distance)

5) Begin the test. The test will stop when the desired number of revolutions is achieved.
6) Remove samples and clean off any loose wear debris.
7) Repeat the test with another sample.

3.6 Characterization Technique

Quantitative and qualitative characterization of the wear resistance, microhardness assessment, nitrided layer morphology and surface morphology are performed by using a wide range of instrument.

3.6.1 Wear Resistance Analysis

Pin-on-disc wear test is conducted for untreated and nitrided 2, 5, and 8 hours treatment samples. The result of coefficient of friction vs. time operation is provided with the software Winducom 2006. Analysis on the result obtained could give the understanding the improvement of wear resistance by nitriding treatment on AISI 316L stainless steel.

3.6.2 Microhardness Assessment

The hardness of particular material sample could be obtained by Vickers microhardness test technique as follow the standard requirement test. (Instron, 2011)

- ASTM E384 – micro force ranges – 10g to 1kg
- ASTM E92 – macro force ranges - 1kg to 100kg
- ISO 6507-1,2,3 – micro and macro ranges

The Vickers hardness test method consists of indenting the test material with a diamond indenter, in the form of a pyramid with a square base and an angle of 136 degrees between opposite faces subjected to a test force of between 1g and 100kg. The full load is normally applied for 10 to 15 seconds (material.co.uk, 2011). The advantages of the Vickers hardness test are that extremely accurate readings can be taken, and just one type of indenter is used for all types of metals and surface treatments. The Vickers method is capable of testing the softest and hardest of materials, under varying loads. (Indentec, 2011)

For this project, microhardness assessment by Vickers hardness test is performed to examine the results surface hardness for each different treatment. The instrument used is Model HV-1000A Micro Hardness Tester (Figure 4) with 10gf load and 15 s dwell time. The surface hardness of nitrided steel will be tested from the edge nitrogen diffusion surface in the range from 0 to 50 μm depth. This is to obtained depth profile of microhardness. According to the investigation by (Triwiyanto et al., 2009), the surface hardness for 450°C (8 hours) decreasing moderately from 0 to 10 μm which is from 1600 to 1400 $HV_{0.5}$. The results of this investigation guide author to apply the same technique in order to relate the thickness of nitrided layer and the surface hardness improvement

Figure 4: Vickers hardness test instrument; Model HV-1000A Micro Hardness Tester

3.6.2 Nitrided Layer Morphology

Field emission scanning electron microscope (FESEM) can provide the wide range of information and versatile which often the preferred starting tool for analytical microscopy. The FESEM instrument used is Carl Zeiss AG - SUPRA 55VP (Figure 5). FESEM enables high resolution (1nm at magnification of 650,000 X) and low beam voltage imaging, in addition to high-resolution characterization. In FESEM, a field-emission cathode in the electron gun of a scanning electron microscope provides narrower probing beams at low as well as high electron energy, resulting in both improved spatial resolution and minimized sample charging and damage.

In this project, nitrided layer morphology for the samples with 2, 5 and 8 hours treatment is analyzed using FESEM for several magnifications such as 50 X, 2200 X and 6000 X The thickness of expected formation of nitrided layer for each samples will be measured using FESEM on the images. Then, the enlarge images of these samples will be recorded.

Figure 5: FESEM instrument; Carl Zeiss AG - SUPRA 55VP

Before perform this analysis, the samples need to be prepared by metallographic technique where the cross-section of nitrided sample is required. In this technique, the nitrided samples will be sectioned and cut for area of interest and for easier handling. This is done by abrasive cutter machine. Then mounting will be done to protect the sample edge and maintains the integrity of the materials surface features Mounting also can improve handling of irregular shaped materials. Mounting is done by hot compression mounting with thermosets-phenolics as the mounting compound. Then, the surface will be be ground and polish into mirror finish. All the samples surface are ground on 120, 220, 500, 800, 1000, 1200 grit SiC papers, and then polished using 1 μm Al_2O_3 pastes to the mirror finish. Etching is required to make the surface of interest visible during the assessment. For the samples of AISI 316L, the samples will be etched in Marble's solution (4g $CuSO_4$ + 20 ml HCl + 20 ml distilled water). Refer to **APPENDIX D.**

3.6.4 Surface Morphology

Surface morphology is an element that can be provided by FESEM instrument (Figure 5). In this project, the surface morphology of worn region for untreated and nitrided samples will be analyzed after wear test. The analysis also using several magnifications from 50 X to 500 X. Wear mechanism characteristic could be analyzed by the image recorded using surface morphology of FESEM.

3.7 Hardware and Tools

3.7.1 Raw materials:

- AISI 316L stainless steel rod bar

3.7.2 Tools:

i. Wire cut EDM
ii. Abrasive cutter machine
iii. Conventional lathe machine
iv. Grind/polishing machine
v. Silica carbide abrasive grit paper
vi. Polisher and Aluminum oxide/diamond paste
vii. Etching agent (Marble)
viii. Ultrasonic cleaning bath
ix. HCl (2 M) solution
x. Horizontal Tube Furnace, Flow meter, Command Module
xi. Ammonia and nitrogen gas tank
xii. Mixing chamber
xiii. Pin-on disc wear test machine
xiv. Water abrasive cutter machine
xv. Vickers Microhardness Testing Machine
xvi. Optical Microscope (OM)
xvii. Scanning Electron Microscope (SEM)

3.8 Gantt Chart

Table 2: Gantt chart

Activity	FYP1				FYP2			
	JUNE	JULY	AUG	SEPT	SEPT	OCT	NOV	DEC
Early stage documentation		■						
Preliminary research and literature review		■						
Sample Preparation			■					
Low Temperature Gas Nitriding Treatment				■	■			
Pin-on Disc Wear Test						■		
Wear Resistance Analysis						■		
Nitrided Layer Morphology						■		
Surface Morphology							■	
Microhardness Assessment							■	
Analysis and discussion of results							■	
End of project and report								■

■ Progress of Work

3.9 Key Milestones

Table 3: Key milestones

Key Milestone	August				September				October				November				December			
	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4	1	2	3	4
Sample Preparation	12/8/2011																			
LTN Gas Treatment					27/9/2011															
Pin on-Disc Wear Test									5/10/2011											
Wear Resistance Analysis									8/10/2011											
Nitrided Layer Morphology									28/10/2011											
Surface Morphology													11/11/2011							
Microhardness Assessment													18/11/2011							
Analysis and discussion of results													25/11/2011							
End of project and report																	23/12/2011			

Number of Week — Milestones

Progress of Work — Deadline

CHAPTER 4

RESULTS AND DISCUSSION

4.1 Wear Resistance Analysis Results

The results coefficient of friction after wear test is obtained in this project, which is used to analyze the wear resistance. Coefficient of fraction is the ratio of the force that maintains contact between an object and a surface and the frictional force that resists the motion of the object. From the pin-on-disc wear test with dry condition, the coefficient of friction results were obtained for each different sample. The samples are 2 hours (2N), 5 hours (5N) and 8 hours (8N) nitrided 316L stainless steel as well as untreated 316L stainless steel (UN).

As shown in Figure 6 which is the comparison results of coefficient of friction vs. time profile, after 0.50 hours (30 minutes) wear test with applied load of 17 N and 300 m sliding distance, UN sample yield the highest coefficient of friction, which averagely ranging from 1.4-2.0. Then, the coefficient of friction followed by the sample of 2N with 0.8-1.4, 5N with 0.6-0.8 and 8 hours 0.3-0.5 nitrided steel. Theoretically, lowest coefficient of friction gives highest wear resistance during wear mechanism on the surface.

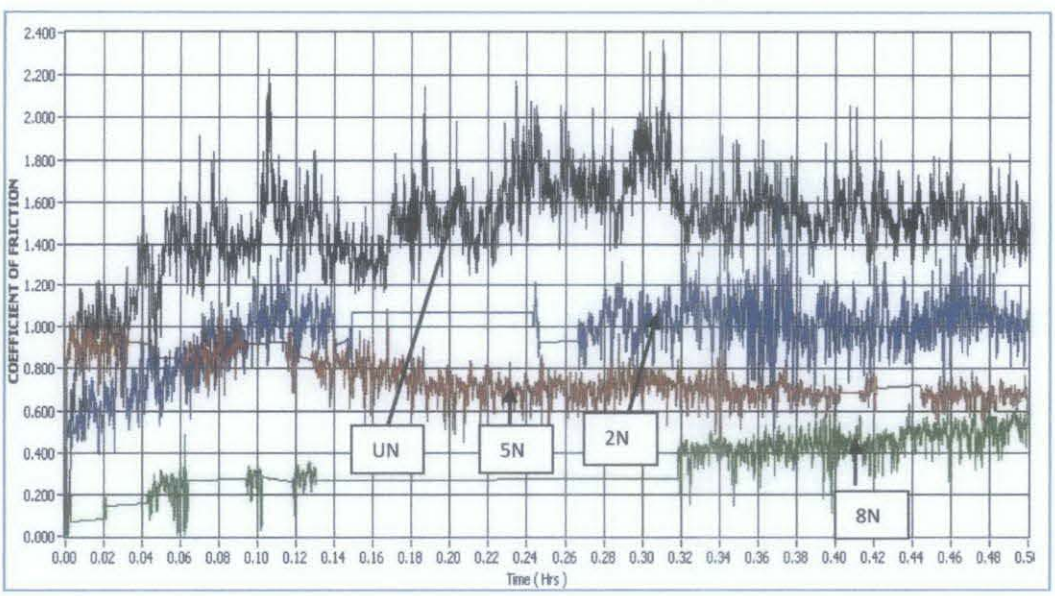

Figure 6: Coefficient of Friction vs. Time profile for UN, 2N, 5N and 8N samples.

These results indicate that the wear was severely occurred on the sample of UN with high frictional force and low wear resistance. Meanwhile, the presence of nitrided layer on the samples of 2N, 5N and 8N could be explained that the formation of layer provide a convenient support to the protective layer of oxides, which may introduce an increasing of wear resistance (Corujeira, 2009). According to investigation by (Subbiah et al., 2005) formation of nitrided layer on steels is increased in thickness as the time of nitriding treatment is extended. This is why the sample of 8N achieved high wear resistance compared to 2N and 5N samples. The surface morphology of worn region after wear test is discussed in the next section. The characteristic appear can be related with wear mechanism that took place on the nitrided samples and how these characters influenced the coefficient of friction and wear resistance for each sample.

4.2 Microhardness Assessment Results

Vickers hardness tests were performed on the surface of nitrided samples (2N, 5N and 8N) using Model HV-1000A Micro Hardness Tester with 10gf load and 15 s dwell time. According to the results obtained, which is presented on the Figure 11, the value of surface hardness obtained for each sample is compared through the distance from the surface.

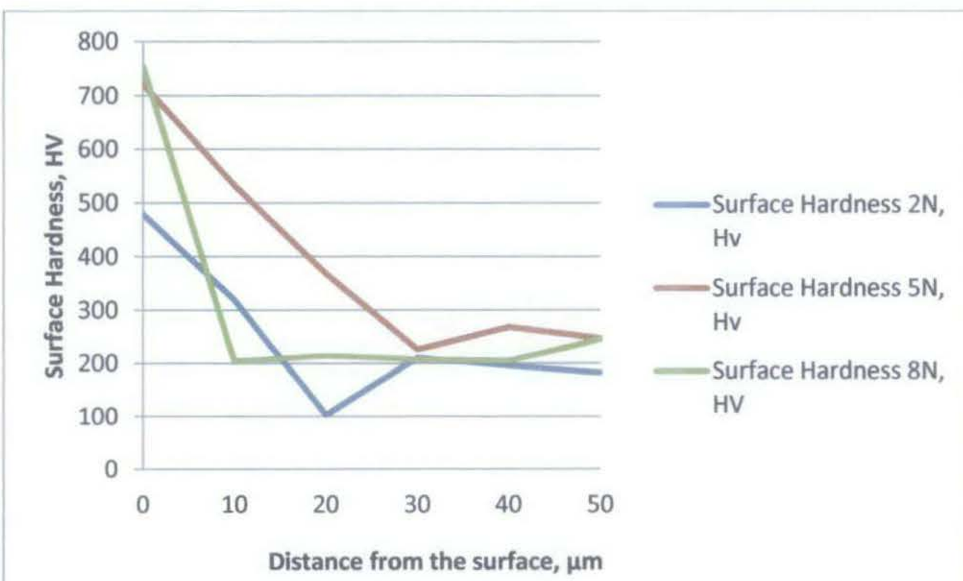

Figure 7: Depth profiles of Microhardness

Surface hardness of the nitrided steel is significantly improved from 2 hours to 8 hours nitriding treatment. 2N samples formed nitrided layer with maximum hardness of about 480 $HV_{0.01}$ Hv, which is much lower than the hardness of 720 to 754 $HV_{0.01}$ for other two nitrided samples. The increasing of surface hardness can be explained due to the extending of time treatment where nitrogen atoms diffused in the surface become more density in the nitrided layer. Theoretically, formation of nitrided layer due to precipitation-free diffusion layer by nitrogen supersaturated, which is normally, knows as S-phase (Toshkov et al, 2007). This supersaturation of nitrogen in the austenite will cause the expansion of the lattice of the substrate austenite (Triwiyanto et al, 2009). Thus, a new type nitrite phase (S-phase) as a nitrided layer provides extremely high surface hardness.

4.3 Nitrided Layer Morphology

From Figure 8 to 10, thickness of nitrided layer formed on the 2N, 5N and 8N samples were measured by Field Emission Scanning Electron Microscopy (FESEM) instrument at 1000 X to 5000 X magnifications. 8N samples formed highest thickness of nitrided layer with 12.84 μm, followed by 5N with 7.93 μm and 2N with 1.34 μm.

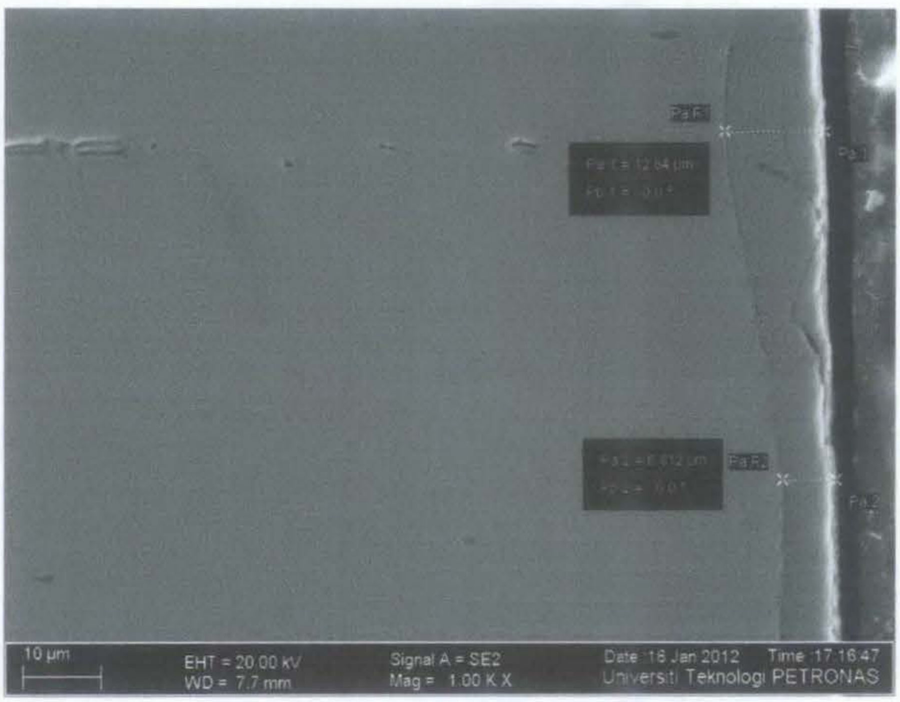

Figure 8: Nitrided layer morphology of 8N samples at 1000 X magnifications

Figure 8 also showed that nitrided layer formed on 8N samples was not uniform. There is thicker and thinner layer formed, which was recorded by FESEM from the cross-sectional samples. This might be due to the characteristic of gas nitriding technique. Theoretically, during the ammonia purged into the furnace, the gas that in contact with tempered steel will disassociate into nitrogen and diffuse into the surface (Mridha, 2006). However, irregular diffusion also can be occurred due to the gas that accumulating near to the surface instead of accelerating directly onto the surface for diffusion.

For samples 2N and 5N, the nitrided layer formed were more uniform. These could be referred in Figure 9 and 10. Nitrided layer become more thicker as the time of treatment is extending from 2N to 8N. These results support the discussion in the section 4.1 regarding the wear resistance analysis results, where the wear resistance improved as the time of treatment is extending. The result of coefficient of friction shown in Figure 6 previously describe on the improvement of wear resistance.

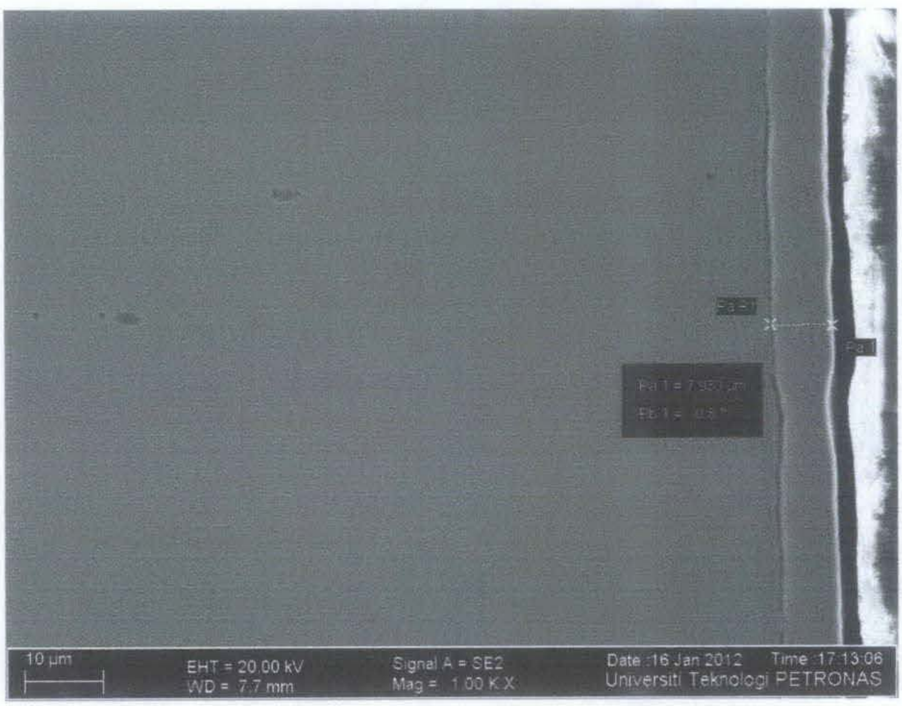

Figure 9: Nitrided layer morphology of 5N samples at 1000 X magnifications

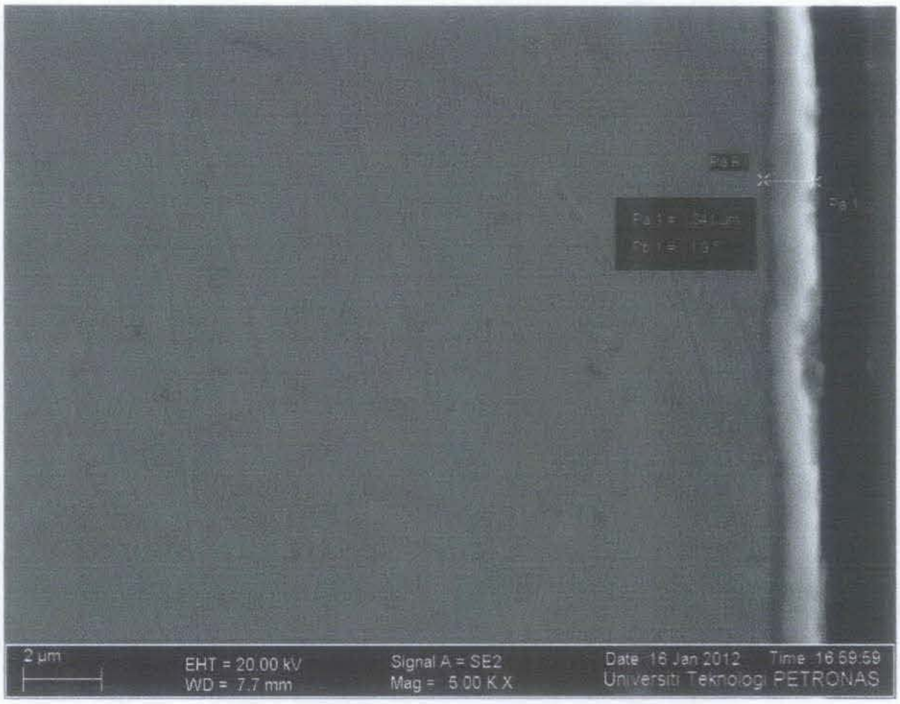

Figure 10: Nitrided layer morphology of 2N samples at 5000 X magnifications

4.4 Surface Morphology Results

Surface morphology of worn region after wear test is investigated by FESEM. Different samples provides different characteristic. For UN sample, the large plastic deformation is obviously observed on the worn region, which is shown in Figure 11 (a) and 11 (b).

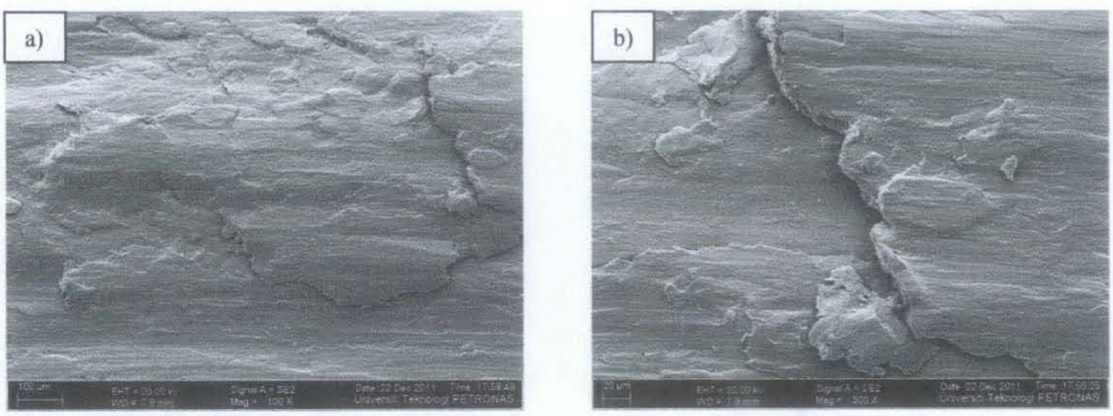

Figure 11: Surface morphology of worn region for UN sample.
(a) 100 X magnification and (b) 300 X magnification

The morphology of the worn region for UN sample also showed the deep plow with plate-like wear debris. The deep plow is explains the wear severely occurred on the sample by abrasive mechanism, while the plate-like wear debris are due to the adhesion wear mechanism, where the material or wear debris (particles) transferred on the surface and would then be plastically deformed and compacted by the rubbing action between the slider and the disc. (C.X.Li et al, 2003) This is the typical wear mechanism characteristic for austenitic stainless steels sliding against steels, where the adhesive and abrasive wear mechanism occurred on the surface (Y.Li et al, 2011). The protective oxide layer is continually disrupted to allow intimate contact and adhesion. At the same time, some loose particles may become embedded in one of the surfaces, and produce deformation and abrasion on the opposite surface.

For 2N sample, the morphology characteristic could be obviously seen in Figure 12 (a) which is 100 X magnification where it appears to be less worn and the shallow plow also built up on the surface. This indicates that abrasive wear also experienced on the sample but not severely compared to untreated sample. Figure 12 (b) with 500 X magnification, the shallow plow and plate-like wear debris built up on the surface clearly seen.

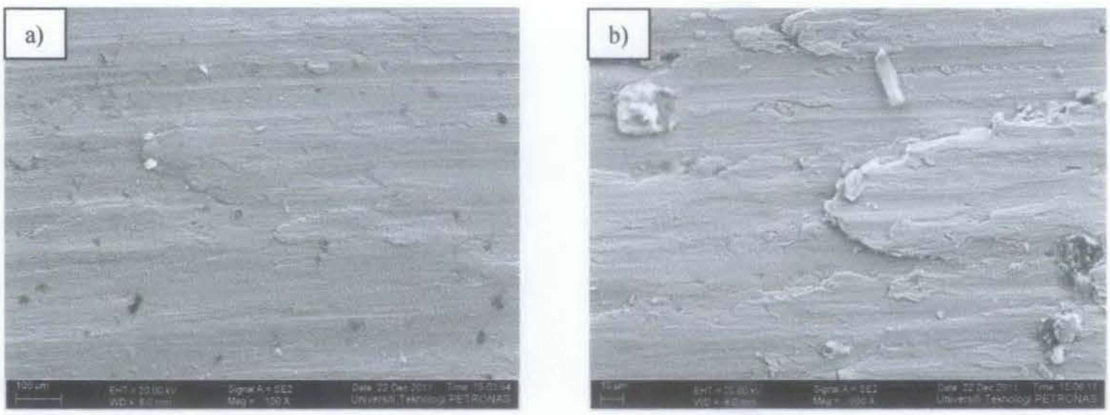

Figure 12: Surface morphology of worn region for 2N sample.
(a) 100 X magnification and (b) 500 X magnification

From the image of FESEM shown on the Figure 10 previously, the thin nitrided layer was formed on the surface after 2 hours nitriding with the improvement of surface hardness, which can be referred in Figure 7 for depth profile of microhardness. Thus, the thin nitrided layer begins to support the protective oxide layer on this sample and resist the wear action (Corujeira, 2009). However, the mechanism of the abrasive and adhesive wear still took place on the sample slightly compared to untreated sample.

In Figure 13 (a), the worn region morphology at 40 X magnification for 5 hours nitrided sample only observable with several surface digging and also with narrower and superficial wear track (Hashemi et al., 2011, C.X.Li et al., 2003). The structure seems that the mechanism occurred is slight abrasive wear. This could be strongly support by analyzing the morphology in 500 X magnification in Figure 13 (b). The spot taken on the surface digging clearly had shown the abrasion of the samples. This is the characteristic of nitrided steel where only abrasive mechanism dominates the wear process (Hashemi et al., 2011, Y.Li et al., 2011 and Wang et al., 2000). The thicker nitrided layer formed on this sample combining with protective oxide layer prevent from intimate contact and adhesion on the surface. This is why only abrasive mechanism took place.

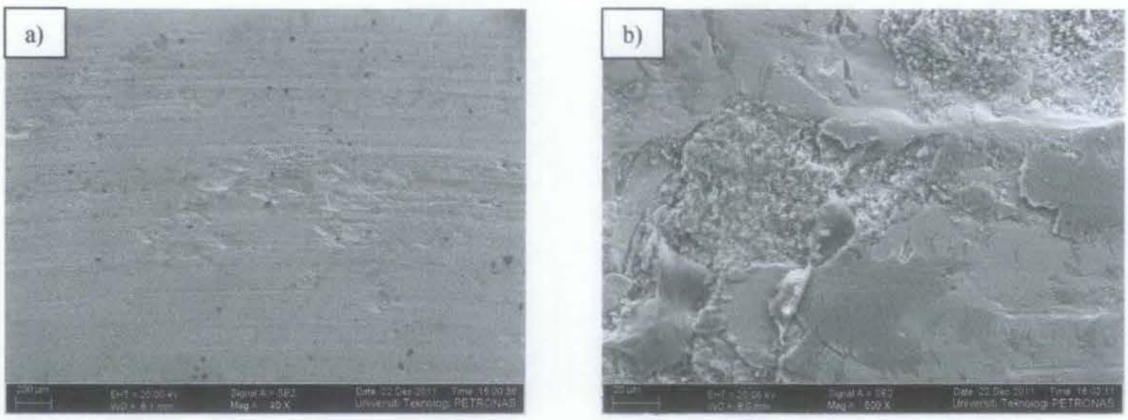

Figure 13: Surface morphology of worn region for 5N sample.
(a) 40 X magnification and (b) 500 X magnification

Supposedly, the wear mechanism characteristic for 8 hours nitrided steel after wear test similar with 5 hours treatment, which is shallow, narrower and experiencing and slight abrasion. However, from the result of wear track obtained on this sample at 200 X and 500 X magnifications in Figure 14 (a) and (b) show that there was some crack occurred on the worn region. This is possibly due to the nitrided layer formed on this sample was not uniform, which can be seen on the Figure 8 previously, in the nitrided layer morphology result. Thus, morphology of this nitrided layer leads to the crack when the slider (pin) slide on the uneven nitrided layer and hit the thicker layer.

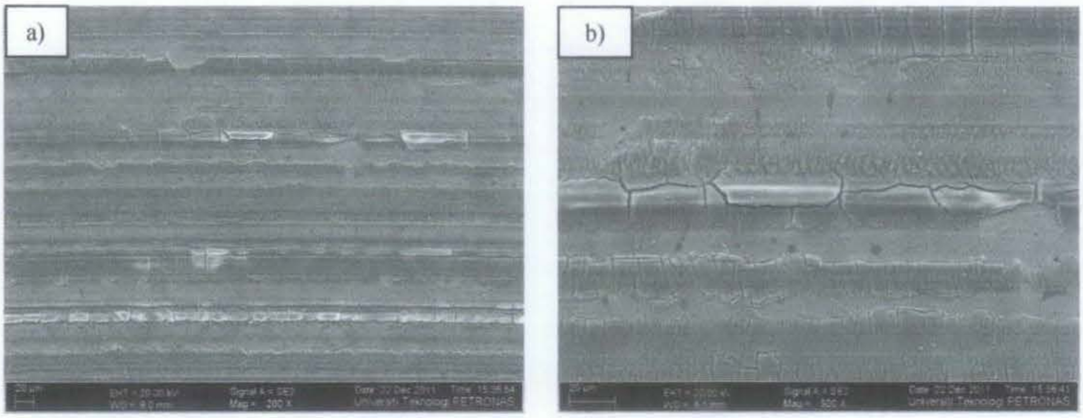

Figure 14: Surface morphology of worn region for 8N sample.
(a) 200 X magnification and (b) 500 X magnification

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

In conclusion, wear resistance and surface hardness of nitrided AISI 316L stainless steel is significantly improved through the low temperature gas nitriding treatment with 2, 5 and 8 hours time variables. 8 hours treatment achieved the lowest value of coefficient of friction result, which was 0.3 and gave high wear resistance. Meanwhile, maximum surface hardness achieved was 754 $HV_{0.01}$ after 8 hours treatment. These improvements are due to the formation of nitrided layer on the treated steel that successfully formed during low temperature nitriding gas treatment. The maximum thickness layer formed was 12.84 μm at 8 hours treatment. Moreover, the formation of nitrided layer also supported the protective oxide layer during the wear mechanism on the surface, where the in contact surface only experienced slight abrasion with shallow and narrower wear track.

5.2 Recommendation

From the results obtained by the tribological properties analysis, there are several variables that can be recommended to give more improvement in the wear resistance and surface hardness through the formation of nitrided layer. The extending time of treatment bring also the increasing of nitrided layer formed on the surface which is relatively high hardness. Thus, further investigation can be made by extending time of treatment above than 8 hours to analyze its inluence in the tribological properties.

Another recommendation is, the composition of ammonia gas that purge into the furnace as a source of nitrogen gas can be increased. In this project, 50% ammonia and 50% nitrogen gas only be conducted. Thus, by increasing the ratio of ammonia gas composition, the density of nitrogen diffusion probably can be achieved, where the supersaturated nitrogen as a layer can be more harder.

Nano-indentation hardness test is strongly recommended to obtain the value of hardness along the nitrided layer. This can give the best understanding regarding the relation of increasing of thickness layer with the increasing of surface hardness.

In the scope of wear test, there are some investigation done by researcher previously by using ball pin as the slider on the disc during pin-on disc wear test. The signficance obtained by the investigations, where the ball pin sliding mechanism can give stongly argument in the investigation of wear track. This is because, the wear track size can indicates the wear behaviors for nitrided steel.

Finally, the tribological properties analysis on low temperature nitriding gas treatment have many significance in various aspects. The investigations in many area can lead to more understanding on how the improvement can be achieved. Thus, this analysis can give great impact to the industrial applications.

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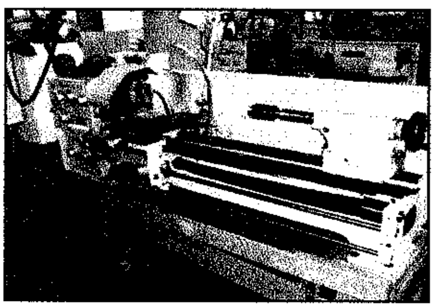
APPENDICES

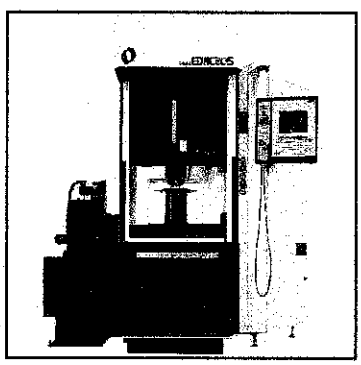
A. Sample Preparation

i) Sectioning and Cutting

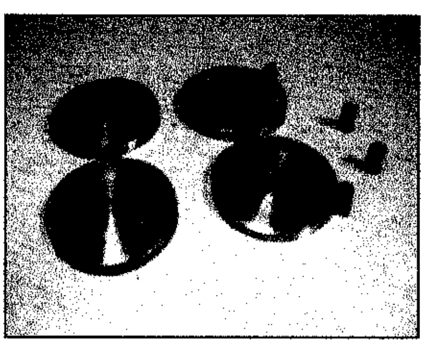
Abrasive Cutter machine



Conventional Lathe machine

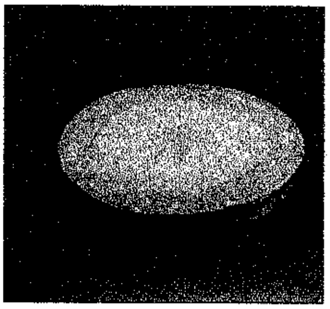


EDM Wire Cut machine

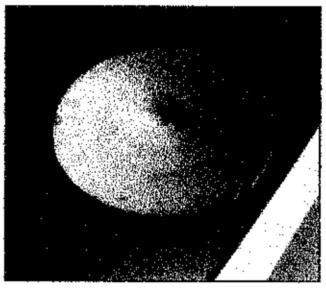


Pin and disc after section and cut

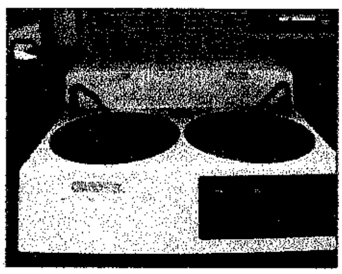
ii) Grinding and Polishing



Silicon Carbide
Abrasive Grit Paper

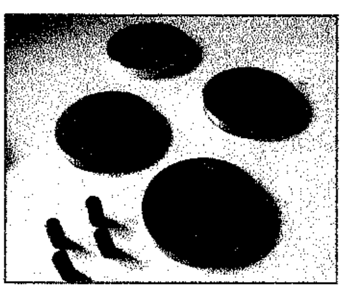


Polishing Paper



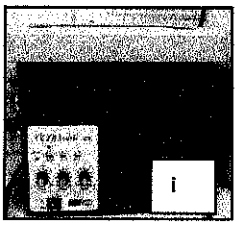
Grind/Polish Machine

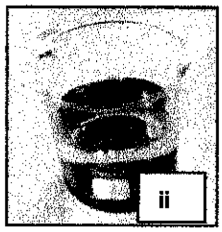
Aluminum
Oxide/Diamond Paste



Pin and disc with mirror
image

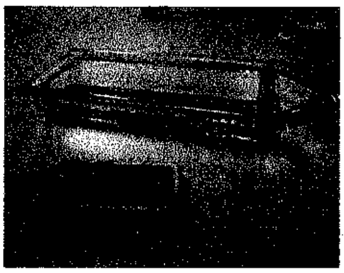
B. Low Temperature Gas Nitriding Treatment Preparation



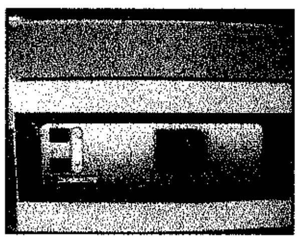


i) Ultrasonic cleaning bath

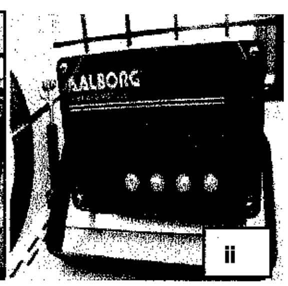
ii) HCl (2 M) solution



Quartz and Alumina Boath

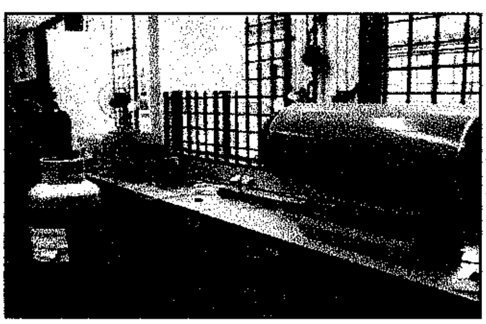


Carbolite CTF tube furnace controller



i) Analog mass flow meters

ii) Microprocessor driven digital Command Modules

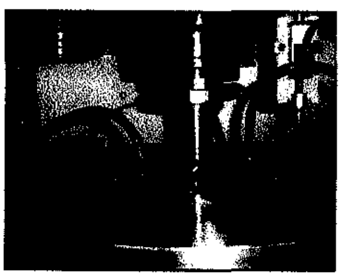


All equipment setup for nitriding treatment

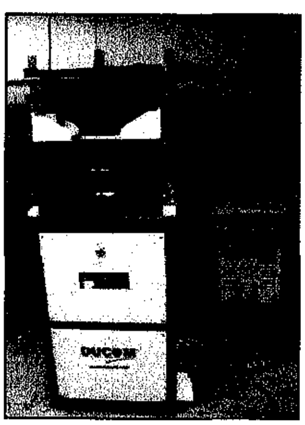
C. Pin-on-Disc Wear Test Preparation

Disc clamped on the disc holder

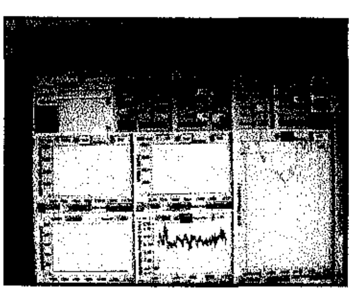
Pin fitted on the pin holder



Pin and disc on the holder fitted into the machine



Ducom TR-701-M6 Multi Specimen Tester machine

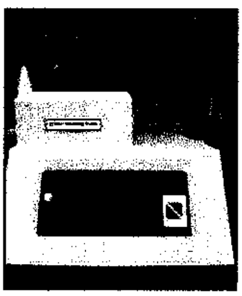


Data input in Winducom 2006 software

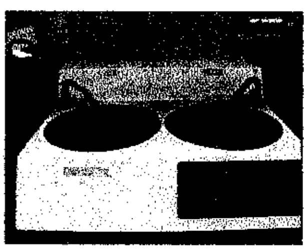
D. **Metallographic Technique (cross-section sample)**

Water Abrasive Cutter for sectioning and cutting

Cross-section samples after sectioning and cutting



Hot Mounting machine for mounting



Grind/Polish machine for grinding and polishing

Cross-section sample with mirror image

Marble solution for stainless steel etching