UNIVERSITI TEKNIKAL MALAYSIA MELAKA

STUDY THE PROPERTIES OF MgAZ91D AFTER THE GAS NITRIDING PROCESS

This report submitted in accordance with requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Engineering Technology Manufacturing (Product Design) (Hons.)

by

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APPROVAL

This report is submitted to the Faculty of Engineering Technology of UTeM as a partial fulfillment of the requirements for the degree of Bachelor of Engineering Technology Manufacturing (Product Design) with Honours. The member of supervisory is as follow:

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PN. YUSLIZA BINTI YUSUF

(Project Supervisor)

ABSTRAK

Dalam kajian ini, teknik gas nitriding adalah sebagai satu alternatif proses rawatan permukaan untuk meningkatkan sifat-sifat permukaan bagi MgAZ91D. Dalam proses gas nitriding, suhu dan masa merupakan dua parameter utama yang akan memberi kesan kepada sifat-sifat MgAZ91D nitrat kerana parameter ini memberi sumbangan besar kepada sifat-sifat kekerasan permukaan. Walau bagaimanapun, kebanyakan kerja yang dilaporkan terhad kepada penilaian MgAZ91D nitrat dan kemasan permukaan yang diperolehi. Oleh itu, kajian ini dijalankan untuk mengkaji pengaruh parameter ini kepada sifat-sifat permukaan MgAZ91D nitrat dan juga untuk mengenal pasti suhu dan masa untuk proses gas nitriding MgAZ91D yang paling sesuai. Suhu proses nitriding yang dicadangkan dalam kajian ini ialah 350^{o}C dan 450^{o}C dengan masa adalah masing-masing 2 jam, 4 jam dan 6 jam. Selepas proses gas nitriding, analisis sifat-sifat permukaan seperti analisis mikrostruktur, analisis kekasaran permukaan dan ujian kekerasan dijalankan untuk membandingkan perbezaan di antara permukaan sampel rujukan dan permukaan sampel nitrat. Analisis mikrostruktur dilakukan dengan menggunakan mikroskop optik dan teknik SEM untuk melihat perubahan mikrostruktur sampel, ujian kekasaran dijalankan untuk mengukur tekstur yang terbentuk di atas permukaan sampel nitrat dan ujian kekerasan dilakukan dengan menggunakan ujian kekerasan Vickers untuk menentukan nilai kekerasan permukaan sampel. Hasil kajian menunjukkan bahawa sifat-sifat permukaan MgAZ91D adalah lebih baik selepas melalui proses gas nitriding. Kekasaran dan nilai kekerasan permukaan MgAZ91D nitrat juga meningkat selepas proses nitriding. Selain itu, kekuatan MgAZ91D didapati meningkat kerana saiz yang mikrostruktur meningkat.

ABSTRACT

In this study, gas nitriding is another alternative technique of surface treatment process in order to improve the surface properties of MgAZ91D. In the gas nitriding process, temperature and time are two main parameters that will affect the properties of nitrided MgAZ91D due to its major contribution on the surface hardness properties. However, most reported work is limited to the evaluation of nitrided MgAZ91D and its surface finish obtained. Therefore, this study is conducted to investigate the influence of these parameters on the surface properties of the nitrided MgAZ91D and also to identify the most suitable gas nitriding process temperature and time. The proposed nitriding process temperatures in this study are 350^{o}C and 450^{o}C with the process time of 2 hours, 4 hours and 6 hours respectively. After the gas nitriding process, the surface property analysis, such as microstructure analysis, surface roughness analysis and hardness test is conducted to compare the difference between the reference sample surface and the nitrided sample's surface. The microstructure analysis performed by using the optical microscope and SEM techniques to observe the microstructure changes of the samples, roughness test conducted to measure the texture formed on the surface of the nitride samples and the hardness test performed using Vickers hardness tester to define the surface hardness property of the samples. The results show that the surface properties of MgAZ91D are improved after the gas nitriding process. The surface roughness and hardness values of the nitrided MgAZ91D are observed increased after the nitriding process. Moreover, the strength of MgAZ91D also increased due to the grain size of microstructure increased.

DEDICATION

Every challenging work needs self-efforts as well as guidance of elders, especially those who were close to our heart.

My humble effort I dedicate to my sweet and lovely

family members.

Whose affection, encouragement and prayers of day and night make me able to get such success and honour,

Project supervisor, Puan Yusliza Binti Yusuf.

Along with all hardworking and respected

lecturers and friends.

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TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF EQUATIONS

LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

Mg	–	Magnesium
Al	–	Aluminum
Zn	–	Zinc
Mn	–	Manganese
RE	–	Rare Earth
Zr	–	Zirconium
NaCN	–	Sodium Cyanide
Na_2CO_3	–	Sodium Carbonate
NaCNO	–	Sodium Cyanate
KCN	–	Potassium Cyanide
K_2CO_3	–	Potassium Carbonate
KCNO	–	Potassium Cyanate
KCL	–	Potassium Chloride
SCFH	–	Standard Cubic Feet per Hour
L/min	–	Litre per Minutes

CHAPTER 1
INTRODUCTION

1.1 Background of Study

Magnesium was categorized as the eighth large quantities element in nature. It is an alkaline earth metal with the lightest weight which only 1.7 g/cm^3 (Fleming, 2012). Since the discovered of the magnesium, it was studied to form magnesium alloys to improve the properties of it in order to fulfill the requirement of industries development. There are variety types of magnesium alloys and the common alloys included AZ series (Mg-Al-Zn), AM series (Mg-Al-Mn), AE series (Mg-Al-RE), EZ series (Mg-RE-Zn), ZK series (Mg-Zn-Zr), and WE series (Mg-RE-Zr) (Yang et al. 2008). Magnesium and its alloys were used in various applications, such as aerospace, automotive, electronic, and etc (Yang et al. 2008). This is because magnesium and its alloys have lower density, high specific strength, good castability and machinability, high thermal and electrical conductivity and also recycle potential properties (Zhong et al. 2012).

MgAZ91D is AZ series alloy which consists of 9.2% of aluminum, 0.71% of zinc, 0.3% manganese, 0.03% silicon, 0.001% copper, 0.003% iron, and the balance is magnesium (Asano and Yoneda, 2008). It is considered as the high-purity alloy which has excellent combination of mechanical properties, corrosion resistance, and castability (Nithin and Venkit, 2015). In addition, Bag and Zhou (2001) also stated that MgAZ91D is the highest strength alloys which provide a high damping capacity and good machinability among other alloys. This magnesium alloy is commonly used in automotive industries application due to its good mechanical properties. However, there

are some limitations in MgAZ91D such as limited cold workability and toughness, limited of strength and deformation during increased of temperature, and also high degree of shrinkage have restricted the uses of MgAZ91D in some applications (Zhong et al. 2012).

Based on these disadvantages of the MgAZ91D, studies have been carried out to improve the surface energy level, hardness, and corrosion resistance of the metal and found that surface treatment is an alternative promising method (Zhong et al. 2012). One of the study states that the effective treatment which is able to improve the hardness, wear and corrosion resistance and fatigue life without any heat treatment required is the nitriding process (MeiYang, 2012).

Nitriding process is thermochemical treatment which introduced the nitrogen into the surface of metal (Czerwinski, 2012). In this process, metal is just took place in free cooling action, so the metal will not faced the shrinkage and deformation problems. There are three main techniques in nitriding process e.g. salt bath, plasma and gas nitriding. According to the studies, gas nitriding is the preferred nitriding process because it able to nitride and harden the surface of the metal homogenously, produce nice surface finish part and perform the reaction to reach the deeper layer (Martins, 2014).

In the gas nitriding process, there have variety of process parameters need to be controlled to produce high quality nitrided metal such as furnace temperature, gas flow, gas activity control, time and process control. According to Pye (2003), choosing the suitable process temperature and time are the most important factors to ensure the total surface area of the metal has been nitrided in higher quality and the properties of the metal will not damaged. Thus, this project will focused on these two parameters to determine the most suitable temperature and time for the gas nitriding in order to get the optimum nitrided layer with superior mechanical and physical properties.

1.2 Problem Statement of Study

Although MgAZ91D has good mechanical properties but it will just occur at room temperature condition (WeiYang, 2013). The yield strength, tensile strength and hardness of MgAZ91D will decrease, and increase its ductility property during its in condition with temperature higher than 120^{o}C (Dynacast, 2016). Other than that, MgAZ91D also has a poor creep resistance (Mordike and Ebert, 2001). Due to these limitations of the MgAZ91D, a few of studies have been done by the researchers and found that surface treatment able to increase the surface energy level, avoid corrosion, and improve hardness property of this alloy (Fleming, 2012).

Other than that, MeiYang (2012) also states that nitriding process is an effective treatment in order to improve the hardness, wear and corrosion resistance and fatigue life without any heat treatment required. Therefore, gas nitriding is the focused techniques as another alternative of surface treatment process in this study. In the gas nitriding process, temperature and time are two main parameters that affect the properties of nitrided MgAZ91D due to the major contribution on the surface hardness properties.

However, previous studies are limited to the evaluation of properties of MgAZ91D after gas nitriding process (Martins, 2014). Hence, it is important to carry out a study to evaluate the suitable process temperature and time during gas nitriding process and study the influence of these parameters on the surface properties of nitrided MgAZ91D. Therefore, a systematic study consist the evaluation for the performance of nitrided MgAZ91D prepared under different process temperature and time is conducted to have better understanding about the effect of this various process parameters to the layer of nitrided MgAZ91D obtained.

1.3 Objective of Study

The objectives for this study are:

i) To compare the surface properties (hardness, roughness, and microstructure) of nitrided MgAZ91D for different temperature and time in compared to reference sample.

ii) To analyze the suitable process temperature and time for gas nitriding process of MgAZ91D.

1.4 Scope of Study

The material used in this project is MgAZ91D. This material is used as the samples to perform gas nitriding. The process temperature and time are the focused parameters in this study to analyze the most suitable temperature and time during gas nitriding process for MgAZ91D. Other related information will refer from other journals according to the study which is approach with this project. Besides that, MgAZ91D will conducted the surface properties analyses such as microstructure analysis, surface roughness analysis and hardness test to compare the difference of the surface properties before and after the gas nitriding process.

CHAPTER 2
LITERATURE REVIEW

2.1 Magnesium and Its Alloy

Magnesium is one of the alkaline earth metals and classifies as the eighth large quantities of element which found in the nature. Normally magnesium is in impurity condition during it was found and consists of the mineral elements magnesite and dolomite. In years 1755, Joseph Black was the first person who aware that magnesium was an element at Edinburgh. He differentiated that the heating process for carbonate rocks, magnesite or even limestone will not only producing the element lime, magnesia also will produced through the process. After the year, many studies about magnesium were discovered such as heated the magnesia with charcoal will produced metallic magnesium but in impurity condition which was invented by Anton Rupprecht in 1792 while Humphry Davy is the person who successful to produce pure magnesium metal but only with a little amount in 1808.

Due to the demand of industries development, magnesium is been studied to form the magnesium alloys to increase the uses and the properties of the magnesium. Magnesium alloys are form by alloying the magnesium with other nonferrous metals, for examples, aluminum, zinc, cerium, silver, thorium, yttrium and zirconium. Cast alloys and wrought alloys are the major groups of magnesium alloys, which been distinguished in term of their processes. Cast magnesium alloys is usually fabricate by shaped castings while wrought magnesium alloys are manufacture by pressing, rolling, forging and stamping (Fleming, 2012).

Since the discovered of the magnesium and magnesium alloys, it was used in various fields and became an important metal from year to year. Therefore, it is important to understand the properties of magnesium and its alloy to investigate how to improve it and make it more widely to use in different fields.

2.1.1 Magnesium and Its Alloy Properties

Magnesium is a moderate hard metal which with silver-white color and will produce bright white flame during burning process. It is the lightest structural metal in the world because the density of it only 1.7 g/cm^3 (Fleming, 2012). Although magnesium is a light metal, but it is strong enough in its structure to prevent it from deformation because the crystalline structure of magnesium is HCP (Hexagonal Closed Packed). Besides that, it can be shaped or bent to a thin sheet without cracking under certain pressure and temperature range. Magnesium is considers as low melting point, 651^{o}C and boiling point, 1100^{o}C metal.

Besides that, magnesium has high chemical reactivity during heating or other chemical reaction because the atomic number of magnesium is 12 and it consists of 2 valence electrons, so it will react easily with other elements to make it more stable. Examples of the compounds will cause chemical reaction with magnesium are oxygen, water, chloride and others. These compounds may cause the metal become tarnish, rust or spoil. Hence, magnesium becomes poor in corrosion resistance (Zhong et al. 2012). The summary of physical and chemical properties for magnesium is shown in the Table 2.1 and 2.2 respectively (Linda, 2016).

Table 2. 1: Physical Properties of Magnesium

Physical properties of Magnesium	
Color	Silvery-white metal
Phase	Solid
Crystalline structure	Hexagonal
Ductility	It can be beaten into extremely thin sheets
Malleability	Capable of being shaped or bent
Luster	Exhibits a shine or glow
Hardness	Relatively soft
Melting point	651^{o}C (1 200^{o}F)
Boiling point	1 100^{o}C (2 000^{o}F)

Table 2. 2: Chemical Properties of Magnesium

Chemical properties of Magnesium	
Chemical formula	Mg
Compounds	Oxide, hydroxide, chloride, carbonate and sulfate. Also Epsom salts (magnesium sulfate heptahydrate) and Milk of Magnesia (magnesium hydroxide)
Flammability	Burns in air with a bright white light
Reactivity	Upon heating, magnesium reacts with halogens to yield halides
Alloys	Magnesium alloys are light, but very strong
Oxidation	It combines with oxygen at room temperature to form a thin skin of magnesium oxide

Besides that, magnesium has low elastic modulus, this will make the magnesium decrease in size during solidification, and also restrict in cold work and toughness. The mechanical properties for magnesium are it has high electrical and thermal conductivity, recycle potential, high specific strength, good castability and good weldability (Mordike and Ebert, 2001). The excellent properties of magnesium have caught the attention of people and intended to utilize it. Therefore, the limitations of pure magnesium which restrict the application of it in industries become the concern factor and some research had done to investigate how to improve it.

According to the researches, found that producing of magnesium alloy will stabilize the magnesium and some of the properties also can be improved based on the alloyed metals added. Aluminum is one of the alloyed metals which commonly used. By decreasing the impurity of magnesium and increasing the proportion of aluminum, it will form a more homogeneous microstructure to make the magnesium become more stable and hard. Hence, the hardness, strength and castability of the magnesium will increase after adding aluminum but the ductility of magnesium will decrease (Hu et al. 2012). Zinc is another common used alloyed metal which is able to increase the alloy fluidity in casting. While others alloyed metals like cerium, yttrium, zirconium and etc also commonly used to alloy with magnesium, in order to improve the properties of magnesium. Table 2.3 shows that some of the example for common alloying elements and their effects of addition (International Magnesium Association, 2016).

Table 2. 3: Effect of the Alloying Element

Alloying Element	Effect of Addition
Aluminum	• Most widely used due to numerous favorable effects • Increases hardness, strength and castability while only increasing density minimally

	• Average alloy contains about 2-9 weight percent of aluminum and can be heat treated with > 6 weight percent • Increased amount of aluminum decreases the ductility of the alloy
Cerium	• Improves corrosion resistance, Increases plastic deformation capability, magnesium elongation, and work hardening rates • Reduces yield strength
Yttrium	• Enhances high temperature strength and creep performance when combined with other rare earth metals
Zinc	• Second most commonly used alloying metal with magnesium • Increases the alloys' fluidity in casting • When added to magnesium alloys with nickel and iron impurities, it can improve corrosion resistance • Additions of 2 weight percent or greater tend to be prone to hot cracking
Zirconium	• Refines grain size in sand and gravity castings (not combined with aluminum)
Rare Earth Metals	• Increase in high temperature creep and corrosion resistance and strength • Allows lower casting porosity and weld cracking in processing

Adding of alloyed elements will not affect the mechanical properties of magnesium. Thus, magnesium and its alloys always are the light constructional metals with high specific strength, good castability, good weldability, better mechanical properties, high electrical and thermal conductivity and recyclable. In

addition, the corrosion resistance and creep properties will improve by using high purity magnesium and added with suitable alloyed elements.

There are variety types of magnesium alloy, and the American Society for Testing Materials has designed the codes to differentiate the alloyed element in magnesium alloys. Table 2.4 shows the code letter for represent elements in magnesium alloy. In the coding, there have one or two letter represent the major alloyed elements and follow by numbers to indicate the mass percentage for the content of elements. The mass percentage value always will round up to a nearest integer (Fleming, 2012). For example, AZ91 magnesium alloy indicates that the alloy Magnesium with 9% of Aluminum mass and 1% of Zinc mass.

Table 2. 4: Code Represent the Elements in Magnesium Alloy

Code Letter	Alloying Element
A	Aluminum
C	Copper
D	Cadmium
E	Rare Earth
F	Iron
G	Magnesium
K	Zirconium
L	Lithium
M	Manganese
N	Nickel
P	Lead
Q	Silver
R	Chromium
S	Silicon
T	Tin
W	Yttrium
Z	Zinc

Magnesium alloys can be produced with most of the conventional casting methods, like sand, permanent and semi-permanent mold and shell, investment and die casting (Fleming, 2012). Recently, the industries demand on die cast alloys is developing quickly. In 1997, die cast Mg AZ91D has took over 81% of all casting techniques (Mordike and Ebert, 2001). Therefore, this study will focus on this material, Mg AZ91D alloy because it is widely used in aerospace and automotive industries applications. It is important to study about the properties of this alloy and how to improve the limitation of this magnesium alloy.

2.1.2 MgAZ91D Properties and Its Application

MgAZ91D alloy is Mg-Al based alloy. It consists of 9.2% of aluminum, 0.71% of zinc, 0.3% manganese, 0.03% silicon, 0.001% copper, 0.003% iron, and the balance is magnesium. Table 2.5 shows the chemical composition of MgAZ91D alloy (Asano and Yoneda, 2008).

Table 2. 5: Chemical Composition of MgAZ91D Alloy

Al	Zn	Mn	Si	Cu	Fe	Mg
9.20	0.71	0.30	0.03	0.001	0.003	Bal.

As previously mentioned, adding the alloyed elements of aluminum able to increase the strength, castability and corrosion resistance of magnesium, thus, MgAZ91D alloy has a good combination of mechanical properties and manufacturability. MgAZ91D alloy is a light magnesium alloy with low density, 1.81 g/cm^3. Moreover, MgAZ91D alloy consists of high thermal and electrical conductivity and the melting point is around 533^{o}C. Other detail about the physical properties e.g. the thermal and electrical conductivity, coefficient of thermal expansion, and also the process to produce this type of alloy are shown in the Table 2.6 (Dynacast, 2016).

Table 2. 6: Physical Properties of MgAZ91 Alloy

Material	Magnesium
Alloy	AZ91D
Density	1.81 g/cm^3
Melting point (Average +/- 50)	533 ^{o}C
Thermal Conductivity	72.3 W/mK
Coefficient of Thermal Expansion	25.2 µm/mK
Electrical Conductivity	12.2 %IACS
Process	Hot Chamber Die Casting

Besides, MgAZ91D alloy is one of the highest strength alloys which provide a high damping capacity, good die castability and machinability (Bag and Zhou, 2001). The details about mechanical properties for this alloy are shown in the Table 2.7 (Dynacast, 2016).

Table 2. 7: Mechanical Properties of MgAZ91 Alloy

Material	Magnesium
Alloy	AZ91D
Tensile Strength	230 MPa
Yield Strength	160 MPa
Impact Strength	3 J
Shear Strength	140 MPa
Hardness	63 HB (Brinell)
Elongation	3 % in 50mm

The equilibrium phase diagrams of Mg-Al-Zn alloy system are shown in Figure 2.1 and Figure 2.2. The phase diagram in Figure 2.1 showed that when contained 12.7 wt% of magnesium at 437^{o}C, Aluminun has a maximum solubility. Moreover, γ ($Mg_{17}Al_{12}$) phase has the melting point of 462^{o}C. The phase of β (Mg_2Al_3) is formed at the temperature 450^{o}C with 38 wt% to 40wt% of aluminum. From the Figure 2.2, it can observe that total content of Al and Zn in Mg alloys should less than 10 wt% for obtaining good castability and weldability properties (WeiYang, 2013).

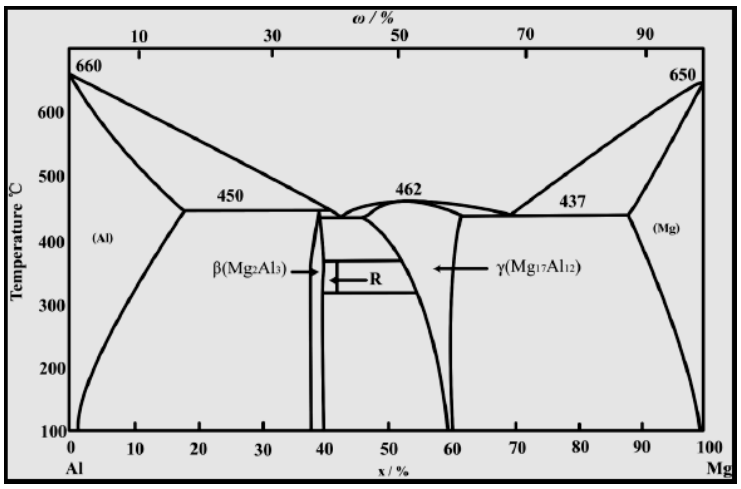


Figure 2. 1: Phase Diagram of Mg-Al-Zn Alloy System

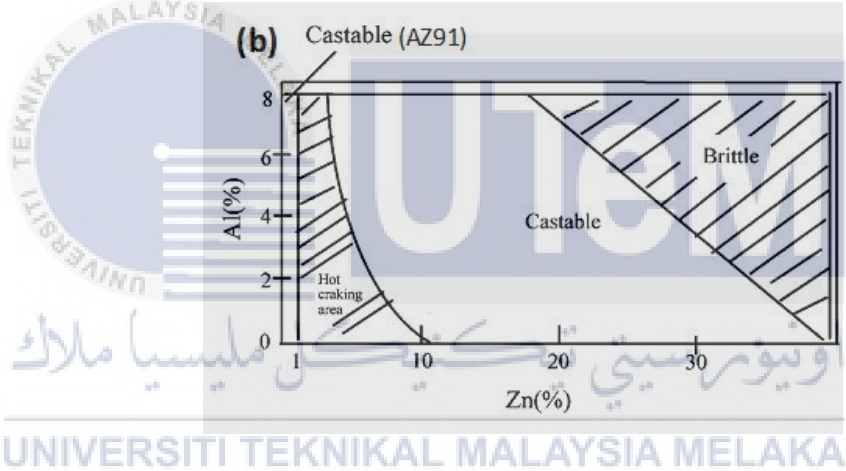


Figure 2. 2: Phase Diagram of Mg-Al-Zn Alloy System

MgAZ91D is usually used for pressure die cast alloy which common use in automotive industries. There have some advantages of using die cast magnesium alloy, such as high productivity, high accuracy and precision, high quality, good cast structure, and also thin wall and complex structure. In comparison with aluminum, MgAZ91D alloy is much better than aluminum because it has higher casting rate, higher wear resistance, lower heat content, better machinability, and lower cost (Mordike and Ebert, 2001).

2.2 Disadvantages of MgAZ91D Alloy

MgAZ91D is a die cast alloy that widely used due to its good mechanical properties, high castability and good corrosion resistance but these properties will just occur at room temperature condition (WeiYang, 2013). When the MgAZ91D alloy in the temperature which higher than 120^{o}C, the tensile strength, yield strength and hardness of magnesium alloy will decrease, and its ductility property will increase (Dynacast, 2016). Other than that, there are several disadvantages of pressure die cast Mg alloy such as poor creep resistance and poor mechanical properties (Mordike and Ebert, 2001). Zhong and co-workers (2012) has performed the MgAZ91D alloy corrosion resistance study and found that in salt solution condition, this type of alloy will undergo the corrosion process within 2 hours. According to Zeng et al. (2006), there are two factors that cause the MgAZ91D alloy has the poor corrosion resistance properties. First, the alloy will become not protective when undergo the oxidation process and second factor is the impurities will cause the galvanic corrosion occur.

Based on the limitation of the MgAZ91D alloy, a few studies have been done to find out the most suitable method to improve the corrosion resistance properties. Surface treatment is one of the alternative methods which able to increase the surface energy level, improve hardness, avoid corrosion and decoration. There are many types of surface treatment including painting, coating, heat treatment, electroplating, oxidizing, carbonizing, nitriding, and etc (Fleming, 2012). According to Hoche et al. (2011), found that producing of magnesium nitride is able to enhance the corrosion resistance of this alloy. Furthermore, Mittemeijer (2013) also states that nitriding process is a most capable and effective method of surface treatment. Therefore, in this study will focus on the nitriding process in order to produce the magnesium nitride as another alternative of surface treatment process.

2.3 Nitriding of MgAZ91D

Nitriding is one of the major thermochemical treatments where the nitrogen substituted into the surface of metal while it is in ferrite state (Czerwinski, 2012). This process was invented in years 1900 and continues widely used in various industries application. In nitriding process, there have no quenching actions or rapid cooling actions required and the metal just takes place in free cooling action. Hence, the size and the dimension of the material will not changing. The process need to be control to ensure the part can nitrided in high quality. The process control factors are included ensure that the total surface area to be nitride, gas delivery pressure system into the chamber, the parameters of the process is correct and others (Pye, 2003). Besides that, nitriding is the effective treatment in order to improve the hardness, wear resistance, corrosion resistance and fatigue life without any heat treatment required (MeiYang, 2012).

Through the nitriding process, nitrogen atoms will substitute into the surface of MgAZ91D. Then, magnesium nitride layer will be produced on the surface of MgAZ91D. There are two layer will be formed on the surface of the metal which are compound layer and underlying diffusion zone. The example of the formation of compound layer and diffusion zone was show in the Figure 2.3. The diffusion zone is responsible for the enhancement of fatigue endurance while the compound layer is responsible to improve the wear and corrosion resistance of metal (MeiYang, 2012).

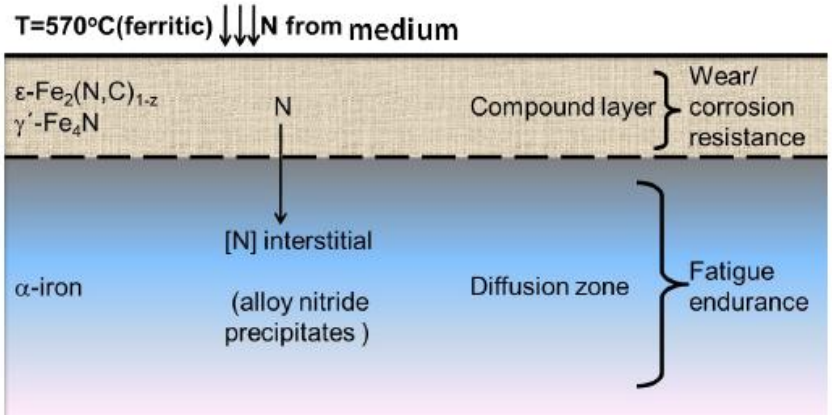


Figure 2. 3: Formation of Nitrided Layer

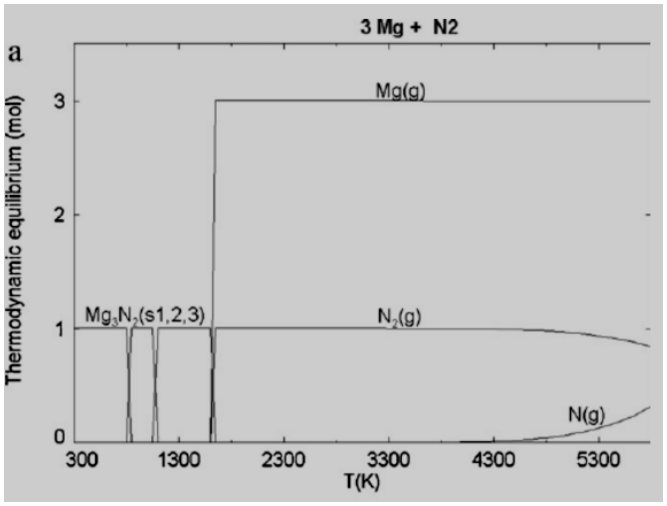


Figure 2. 4: Phase Diagram of Magnesium Nitride

According to Kim et al. (2011), the magnesium nitride will be formed when the temperature between 300 K and 1600 K as shown in Figure 2.4.

There are three main techniques available for nitriding processes which are salt bath, plasma and gas nitriding. Each of the process has its own advantages and disadvantages which will discuss in the following chapter.

2.3.1 Salt Bath Nitriding

Salt bath nitriding is carried out with the fused salt bath that included cyanides or cyanates which invented in years 1940. Common compound used in salt bath consist of 60-70% sodium salts which with 96.5% NaCN, 2.5% Na_2CO_3, 0.5% NaCNO and 30-40% potassium salts which contain 96% KCN, 0.6% K_2CO_3, 0.75% KCNO, 0.5% KCl. This process also can describe as nitro-carburizing process because the molten salt consist the content of carbon and nitrogen. Normally this process is suitable used for steel parts because the elements diffuse into the surface at the same time. The process of salt bath nitriding is carried out around 750-1075 ℉. Figure 2.5 shows the roughly process for the salt bath nitriding. It shows that the salt bath process needs to preheat the furnace in temperature 750^{o}F and start the nitriding process at temperature 1075^{o}F. After the nitriding process, the alloy need to quench in temperature of 750^{o}F and last step is water rinse (Unlimited, 2016).

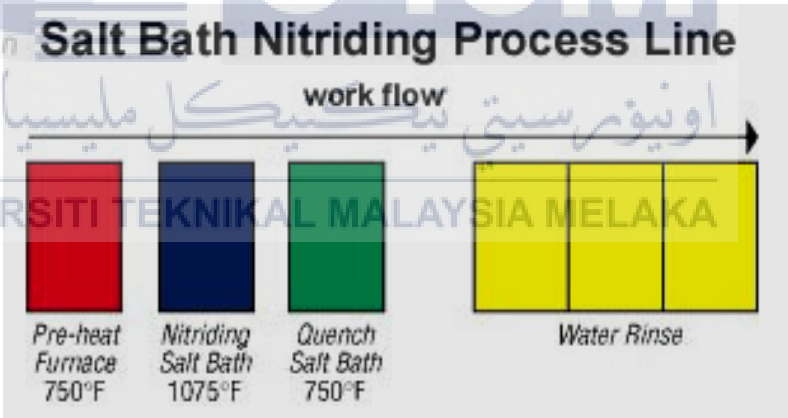


Figure 2. 5: Process Line of Salt Bath Nitriding

(Source: < http://www.houstonunlimitedinc.com/nitriding.aspx > 10/05/16)

The major advantage of using this method is the process has a shorter cycle time (Czerwinski, 2012). Moreover, the process can become faster for recovery and heat up by adding of sulphur or melt pressurizing. This process will produces an aesthetic black finish, and increasing wear and corrosion resistance.

Other than that, the size of the work piece is able to remain constant in the process. Hence, it is possible to produce more parts in the process. This means that it can fulfill the requirement of engineering properties developed during carburizing and carbonitriding (MeiYang, 2012).

Although this method is better in process time, but this technology will produced the toxicity materials and the nitrided surface were defective means it will produce a poor quality of metal surface. The process deal with salt bath nitriding also need a high energy consumption and difficult to control (Czerwinski, 2012). Figure 2.6 shows the example of equipment for salt bath nitriding process. The equipment for this process needs a high investment (Martins, 2014).

Figure 2. 6: Example Equipment for Salt Bath Nitriding

(Source: < http://www.hellotrade.com/om-chem-engineers/salt-bath-furnace.html > 10/05/16)

2.3.2 Plasma Nitriding

In 1932, plasma nitriding or ion nitriding was invented by Wehnheldt and Berghause and it became generally in 1970's. It uses plasma-discharge technology to diffuse the nitrogen on the surface of metal. Plasma is using high electrical energy to motivate the nitrogen ion attack the surface of the alloy and form in the vacuum. Figure 2.7 shows that the view of components during the plasma nitriding process (Czerwinski, 2012)

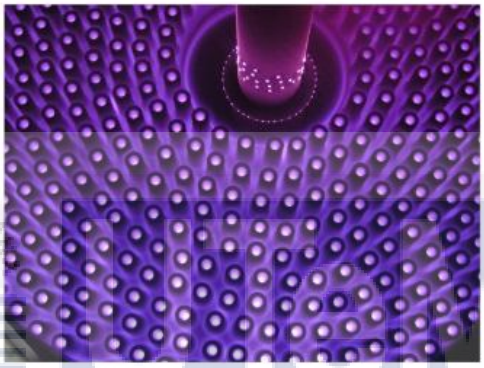


Figure 2.7: View of Components during Nitriding Process

The advantages of the plasma nitriding are it using a lower temperature around 300^{o}C, shorter process time and simple diffusion method to complete the nitriding process. Surface-activation sputtering is the special representative of plasma nitriding. It makes positive ions glow in the discharge to remove the oxide on surface of alloys. Therefore, nitrogen atoms can be diffuse to the metal surface easily. However, conventional direct-current system is the nitrided element supply on the high cathode potential and plasma forms on the surface directly. This may caused the temperature become not constant and overheating, edge effect and surface damage due to arcing (Czerwinski, 2012).

To overcome the limitation of plasma nitriding, active screen plasma nitriding was invented in 1999. Figure 2.8 shows that the concept of active screen plasma nitriding. Active screen plasma nitriding is the improvement of the

previous plasma nitriding system which the nitrided part acted as real cathode and the high cathodic potential is supply to the screen surrounding of the part. Thus, the active screen allows forming of plasma which construct of the ion mixture and start to heating. Then, the electrons and other active nitriding element will flow over the part. Therefore, uniform nitrided layer will form on the complex geometries (Czerwinski, 2012).

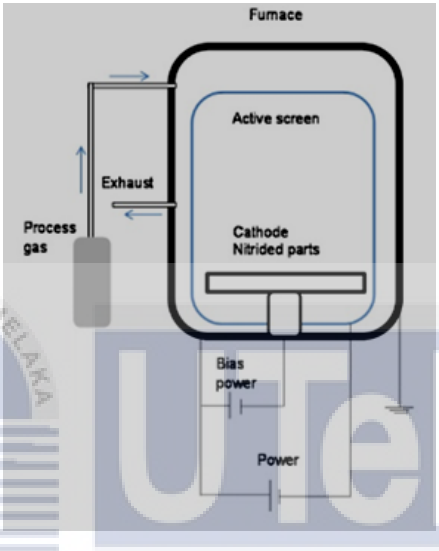


Figure 2.8: Concept of Active Screen Plasma Nitriding

The example equipment for plasma nitriding has shows in the Figure 2.9. The equipment used for plasma nitriding required high investment with high maintenance cost and need a skillful worker.

Figure 2.9: Equipment for Plasma Nitriding

(Source: < http://www.ionitech.net/en/about-us/team.html > 10/05/16)

2.3.3 Gas Nitriding

Gas nitriding is the thermochemical surface treatment process which introduced the nitrogen into the surface of metal by heating the metal until a certain temperature with nitrogen gas or ammonia and within the ferrite and carbide phase region. This process was invented in 1913 but limited in the application because this process has low reliability in production (MeiYang, 2012) but the condition has been improved after the year. The process temperature for gas nitriding generally is around 500^{o}C-580^{o}C (Mittemeijer, 2013) and the metal commonly put in a box furnace or fluidized bed with ammonia or nitrogen gas at atmosphere pressure. The fluidized bed will produce a constant near-ideal temperature through the entire gas-particle volume and fast heating rate (Mittemeijer, 2013). Besides that, gas nitriding consider as the most efficient process compared to other method because it able to uniformly nitride and harden the surface of the metal with complicated shapes (MeiYang, 2012). This advantage is due to the invention of the technique called mixed-gas atmosphere. It is formed from the combination of ammonia and additive gas. As different to conventional gas nitriding, the process is controlled by nitriding potential referring to the furnace atmosphere. Nitriding potential is indicate as the ratio of partial pressures of ammonia and hydrogen. It is the crucial parameter which able to produce more homogenous nitrided surface on the complex geometries part (Mittemeijer, 2013).

Through the gas nitriding process the wear and corrosion resistance will able to improve. There are two factors need to be considered to ensure the process completed perfectly which are choosing a suitable process parameters and controlled the process correctly. The related process parameters included temperature, time, and the nitriding atmosphere (Mittemeijer, 2013). The benefits of using this method are it does not damage the surface of the part and also produce a nice surface finish. Besides, it allows the reaction to reach the deeper

layer and not required a cooling process to minimize the deformation of the part (Martins, 2014).

In this study, gas nitriding was chosen as the process to study due to the various advantages compared to others process. Moreover, gas nitriding is the process widely used today due to the investment cost for equipment of gas nitriding is lower than others. The example of equipment for gas nitriding process is shown in Figure 2.10.

Figure 2. 10: Example Equipment for Gas Nitriding
(Source: < http://www.yibaifurnaces.com/en/product/132.html > 10/05/16)

Figure 2.11 shows the view in atomic molecule state to indicate that how the nitrogen introduced into the surface of the metal. The nitrogen gas or ammonia is diffused into the surface of the metal during the heating process. When the metal is heating with certain temperature, the reactivity of the metal will increased, so the nitrogen gas or ammonia is easily to react with the metal and diffuse the nitrogen atoms into the surface to form the nitrided layer on the surface of metal (Martins, 2014).

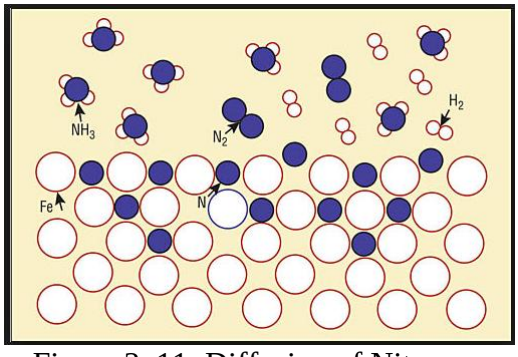


Figure 2. 11: Diffusion of Nitrogen

2.4 Temperature and Time Dependence on Gas Nitriding

Process parameters are the main factors that will affect the formation of the nitrided metal. The process parameters including furnace temperature, gas flow, gas activity control, time and process control. All of these factors help to reduce the deformation of the metal. In these parameters, choosing a suitable time and temperature for the process are the most important factors (Pye, 2003). During the gas nitriding process, suitable temperature and time are required to ensure that the total surface area has been nitrided with high quality and will not damage the properties of the metal. Recent study had mentioned that, the time to form the nitrided metal will decease when using a higher nitriding temperature (Wang and Liu, 2013). Besides that, the surface concentration of nitrogen is increase due to the increasing of temperature (Wu, 2013). Thus, the higher the temperature, the diffusion of nitrogen will become deeper (Hosmani, Kuppusami and Goyal, 2014). The process temperature for gas nitriding generally is carried out at the temperature around 500^{o}C-580^{o}C in a box furnace or fluidized bed in atmosphere pressure. (Mittemeijer, 2013)

Other than that, Cui and co-workers (1999) has conducted the nitridation of Mg at 450^{o}C under normal pressure for 8h and 18h. The experiment is carried out with adding the different additive materials such as $NiCl_2$ and La powder and the experiment result was showed in Figure 2.12. The figure shows that, the yield of Magnesium nitride increase depends on the additive materials added in during the nitriding process.

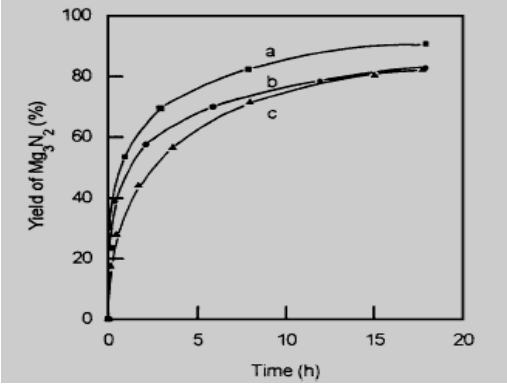


Figure 2. 12 Kinetic Curves of Nitridation of Different Mg Systems. Dopants: (a) NiCl2, (b) La (Lanthanum) powder, (c) none

2.5 Summary of Literature Review

MgAZ91D alloy is the most widely used of die casting alloy which common used in automotive industries and have the new development in aerospace industries. The properties of this alloy such as low density, high thermal and electrical conductivity, high castability, recyclable and other properties have been caught the attention and interest of the people intended to utilize it. But it has the limitation on this alloy, such as low corrosion resistance. Hence, there are various studies related to the properties and application of MgAZ91D alloy. Researchers found that surface treatment to producing the magnesium nitride is able to increase the hardness and the corrosion resistance of the MgAZ91D alloy. In the surface treatment, gas nitriding process is found as the most suitable technique to use in producing the magnesium nitride. The parameters used in the gas nitriding process can be selected by comparing the previous researches and choosing the most suitable parameters which is more similar with this study. The process temperature and time are the focused parameters in this study to investigate how it will affect the process. After the gas nitriding process, the samples used in this study will do the surface properties analyses to compare the difference between the reference and magnesium nitride surface.

CHAPTER 3
METHODOLOGY

3.1 Introduction

The process flow for the gas nitriding process will discuss in this chapter. Work start with samples preparation including grinding, polishing and cleaning process on the surface of the MgAZ91D samples to remove the impurities material on the samples surface and ensure the equivalent condition to all the samples. Then, a sample will choose as the reference sample and perform the surface properties analyses such as microstructure analysis, surface roughness analysis and surface hardness test. The surface roughness of the samples will be tested by using the Mitutoyo SJ-410 Surface Roughness tester. Besides that, the microstructure analysis will be conducted on the surface and cross section area of the sample by using inverted optical microscope and Scanning electron microscope (SEM) and the hardness test will be performed by using the Vickers hardness test machine. Before do the microstructure analysis on the cross section area of the sample, the sample is required to undergo the etching process to reveal the microstructure of the samples.

For the nitrided samples, the nitriding process will carried out at temperatures 350^{o}C and 450^{o}C with 2 hours, 4 hours and 6 hours respectively. Analyses conducted on nitrided samples are microstructure, surface roughness and micro-hardness analysis. The results of surface properties analyses will be compared between the reference sample and the nitrided samples. The summary of the experimental process is shown in the Figure 3.1.

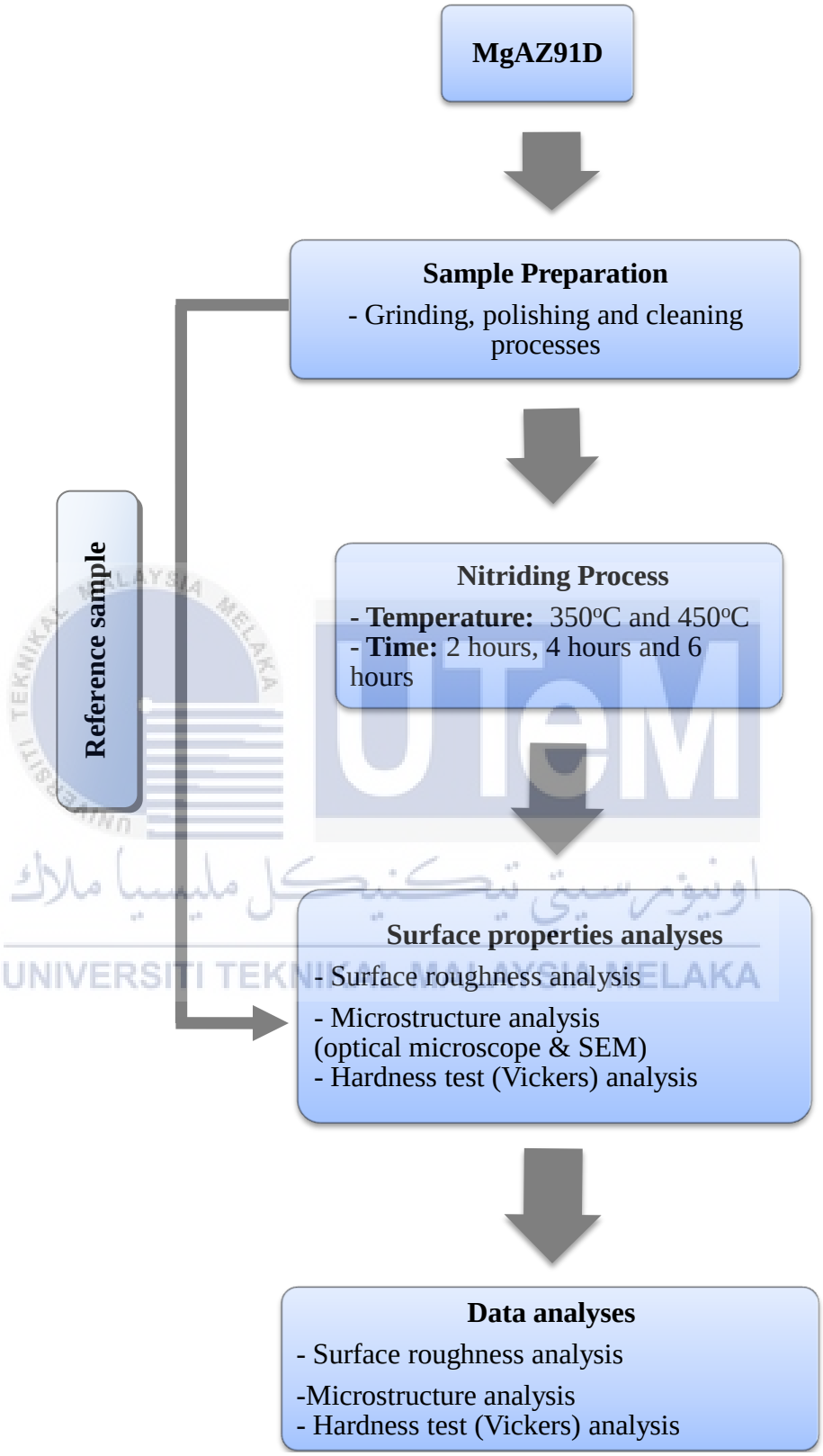


Figure 3. 1: Process Flow of Experiment Process in This Study

3.2 Samples Preparation

The samples used in this study is MgAZ91D (Figure 3.2) with dimension 30 mm x 10 mm x 3 mm in rectangular shape and the chemical composition of MgAZ91D is shown in Table 3.1.

Figure 3. 2: Sample of MgAZ91D

Table 3. 1: Chemical Composition of Mg ZA91D

Material	Chemical	Composition (wt %)
Mg AZ91D	Aluminium	9.20
	Zinc	0.71
	Manganese	0.30
	Silicon	0.03
	Copper	0.001
	Iron	0.003
	Magnesium	Balance

First, the samples will grind and polish by using the metallograpgic polishing machine which shown in Figure 3.3. The grinding process is performed to remove the damage caused during the cutting process. In this study, 800 up to 1200 grit silicon carbide (SiC) abrasive paper is used to grind the samples (Oteyaka et al. 2005) which shown in Figure 3.4. Then, the samples go through the polishing process by using 0.05 µm alumina on the medium nap cloth for 2 minutes. The summary of the grinding and

polishing procedure for MgAZ91D is shown in Table 3.2 as per recommend in (ASM International, 2002). The purposes for polishing the samples are ensured the samples in flat, smooth, scratch free and equivalent roughness condition. After the polishing process, the samples produce mirror like surface finish as shown in Figure 3.5. Besides that, the reaction and chemical bond strength between MgAZ91D and nitrogen atoms will be improved by removing the oxide layer on the surface of the samples through polishing process.

Table 3. 2: Grinding and Polishing Procedure for MgAZ91D

Process	Abrasive / Size	Time, minutes
Grinding	800-grit SiC paper	3
	1200-grit SiC paper	5
Polishing	0.05µm Alumina	2

Figure 3. 3: Metallographic Polishing Machine

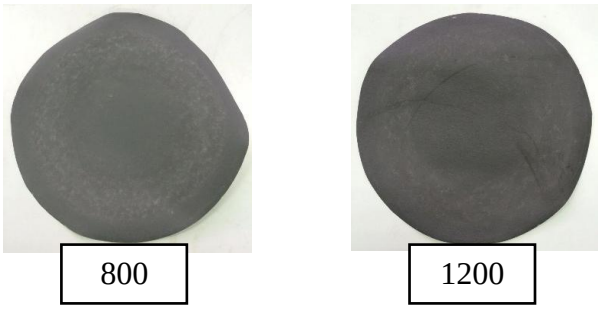


Figure 3. 4: Silicon carbide (SiC) papers with grit size

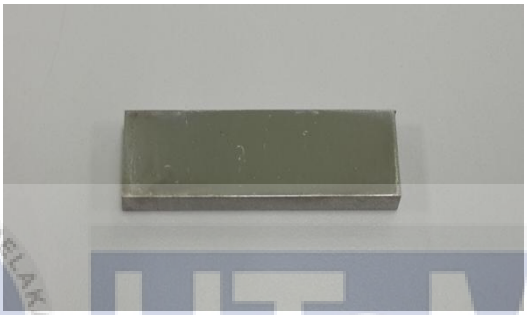


Figure 3. 5: Mirror-like Surface of MgAZ91D

After that, the process will followed by ultrasonic cleaning process. This process is done with solution ethanol for 2 minutes with maximum cleaning temperature 50^{o}C to remove all the unwanted substance and impurities materials on the surface of the samples before proceed to surface properties analyses. Then, the samples are dried in cold air (Qu et al. 2013). The equipment used to conduct the cleaning process is shown in Figure 3.6 which is Branson 8510 ultrasonic bath cleaner.

Figure 3. 6: Ultrasonic Bath Cleaner

3.3 Gas Nitriding Process

The nitiding process will conducted by using Nabertherm Furnace (Figure 3.7). Prior to the nitriding process, the furnace required a preparation to ensure the cleanliness and suitable condition for the nitriding process.

Figure 3. 7: Nabertherm Heat Treatment Furnace

3.3.1 Furnace Preparation

Before started the nitriding process, the furnace needed to prepare properly to ensure there are no other effects will affected the experimental results. First, the furnace will clean manually to remove the dirt or dregs which residual in previous experiment (GmbH, no date). Then, the furnace will calibrated by using thermocouple to verify the actual condition of furnace temperature against the controller (Orlando and Borrini, 2006). The sample is placed on a brick in the box before the box put into the furnace to prevent the samples damaged the furnace (Figure 3.8). After done the preparation, the samples will go through the

nitriding process. During the nitriding process, the oxygen inside the furnace will eliminated by filling the nitrogen gas into the furnace with 5 minutes. Besides that, the waiting time for the heating process was set as 1 hour to ensure the difference of the temperature in the furnace against the controller will not vary greatly.

Figure 3. 8: Sample is placed on a brick in the box of furnace

3.3.2 Parameters of Gas Nitriding

In this study, nitriding process of MgAZ91D samples will perform in different process temperatures and times. The nitriding process temperatures are fixed at 350^{o}C and 450^{o}C with 2 hours, 4 hours and 6 hours respectively. The samples are nitrided at various parameters to investigate the effects of process temperature and time to the microstructure and mechanical properties of the MgAZ91D. Other parameter such as nitrogen gas flow of the furnace needs to ensure that it will provide in uniformly condition and the flow rate is in 5 SCFH which is 2.36 L/min. The summary of nitriding process parameters conducted in this study is shown in the Table 3.3.

Table 3. 3: Gas Nitriding Parameters

Material	Process Temperatures	Process Times	Nitrogen gas flow rate
Mg AZ91D	350°C	2 hours	2.36 L/min
	450°C		
	350°C	4 hours	
	450°C		
	350°C	6 hours	
	450°C		

3.4 Surface Properties Analyses

Surface properties analyses are conducted to investigate the difference of surface properties between the reference sample and nitrided samples. In this study, optical microscope and scanning electron microscope (SEM) will used to perform the microstructure analysis. Other surface properties analyses such as surface roughness and hardness test also will be discussed in following chapter.

3.4.1 Microstructure Analysis (Optical Microscope & SEM)

Optical microscope also known as light microscope. It is uses the visible light and lens system to magnify the small samples image. The lens systems for optical microscope are objective and ocular which provides the different magnification power to enlarge and magnify the image. There are two types of optical microscope which are inverted optical microscope and upright optical microscope. The different between these two microscopes are the optics of the inverted microscope are place under the samples while the optics for upright microscope are place on above the samples (Scheffler and Müller, 2015). In this study, inverted microscope was chosen as the instrument to observe the

microstructure of the samples as shown in Figure 3.9. The light sources and the condenser of the inverted microscope are place on the top of the stage and the objectives and turret of this microscope are below the stage pointing up. This microscope was selected due to it is easy to use and it provides a rapid testing time.

Figure 3. 9: Inverted Optical Microscope

Another microstructure analysis is using scanning electron microscope (SEM) as shown in Figure 3.10. Scanning electron microscope is used to observe the microstructure of the samples. It is performed with a photographic print that has been enlarged to generate a high resolution image. Hence, it is allowed to observe the features of the materials in precisely measure. It is uses a focused beam with high energy electrons to create the stimulus on the surface of the samples and the electron beam is inspected in a raster pattern across the surface. Then, the electron detector will detect the emitted electron to display the image of the surface. Besides that, it is also able to perform analysis on the selected location of the sample (Anonymous, 2014). Figure 3.11 shows that the mechanism schematic diagram for SEM (Simpson, 2014).

Figure 3. 10: SEM Zeiss EVO 50

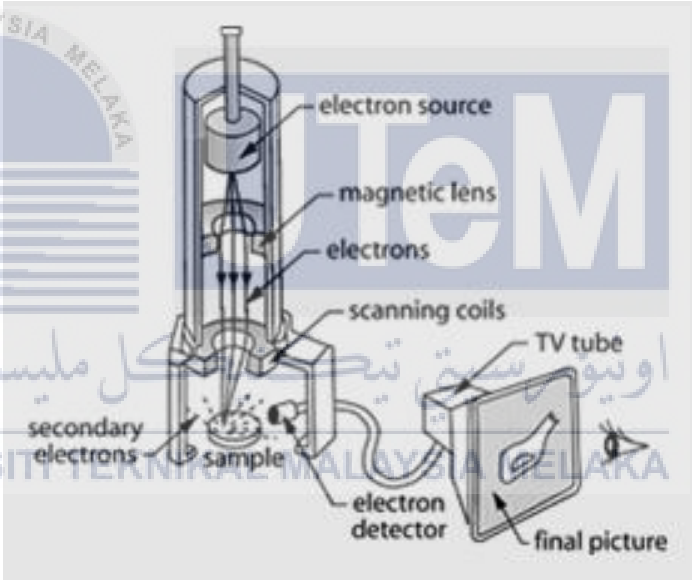


Figure 3. 11: Mechanism Schematic Diagram for SEM

Before start the analysis, etching process is required to perform on samples in order to reveal the microstructure of the samples. This process is used to highlight the features of the samples such as grain boundaries, phase difference and inclusions and also make the micro-structural details more clear and visible under optical microscope (Fiset et al. 2004). The etchant used in this study is Nital reagent and the samples will be etched for 30 to 40 seconds and rinse and dry in cold air. Nital etchant is forms from the mixed of solution nitric

acid and ethanol. Table 3.4 shows the information about the Nital composition (Baker and Hugh, 1999). After the etching process, the samples will immediately clean to remove the dust for microstructure analysis.

Table 3. 4: Composition of Picral Reagent

Etchant	Composition	Concentration	Time
Nital	Nitric Acid	2.5 ml	30 – 40 seconds
	Ethanol	50 ml	

The reference sample and nitrided samples will do the microstructure analysis with optical microscope and SEM and the results are recorded to compare the difference on microstructure between the reference sample and nitrided samples. The microstructure analysis will performed on the surface and cross section area of the samples. To conduct the analysis on the cross section area of the samples, the samples were required to using diamond cut machine to cut the samples into two pieces. Figure 3.12 shows the diamond cut machine used in this study.

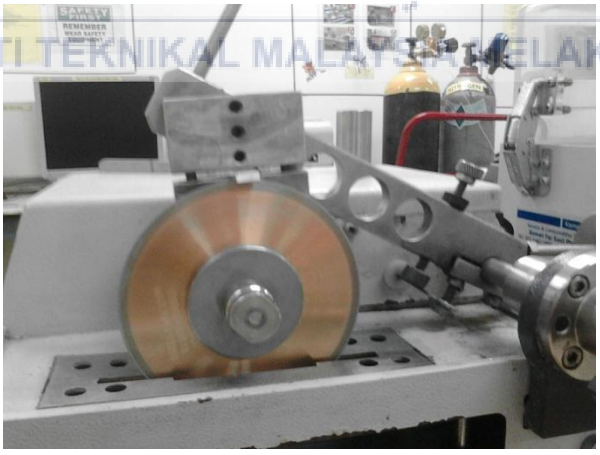


Figure 3. 12: Diamond Cut Machine

3.4.2 Surface Roughness Analysis

Surface roughness is a measurement of surface finish or surface profile where it is indicated the polishing level or the texture forms on the surface of the material. There are some geometrical characteristics to form the roughness surface such as macro-deviations, surface waviness, and micro-irregularities.

The surface roughness can be determined by different methods which divided into three groups which are texture descriptors that used the correlation length to measure, extreme value descriptors that used maximum peak height, maximum valley height or maximum peak to valley height as measurement and statistical descriptors that used the average roughness, root mean square roughness or skewness to determine the surface roughness. Figure 3.13 shows the evaluation of surface roughness (Novin, 2015).

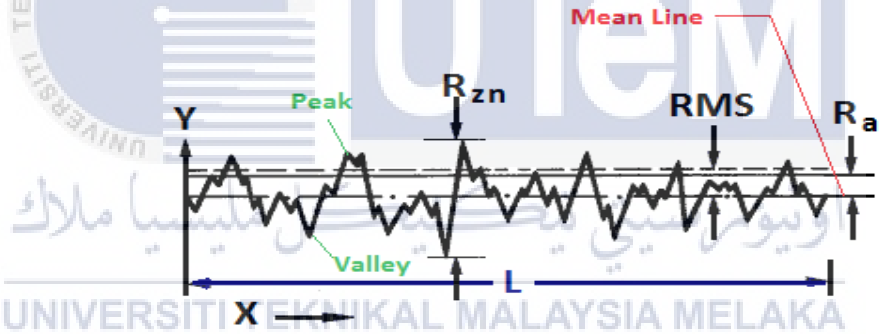


Figure 3. 13: Surface Roughness Measurement

Among the methods, mean roughness R_a is the most effective measurement which commonly used (Xue, no date). Therefore, Arithmetic Mean Value, R_a is used as the method to determine the surface roughness of the samples in this study. R_a is the calculation which calculated the average of the surfaces roughness with measured microscopic peaks and valleys (Novin, 2015). The formula of R_a is shown as below:

$$R_a = \frac{(h1+h2+-----+hn)}{n} \qquad (3.1)$$

Where h_1, h_2, …h_n represent the profile values while n is the number of sample.

In this study, the equipment used for surface roughness test is Mitutoyo SJ-410 as shown in Figure 3.14. The measurement done in this study is 20 times for each of the sample. The directions for surface measurement are usually made along a line as show as Figure 3.15 which with 2 different direction to get the accurate measurements.

Figure 3. 14: Mitutoyo SJ-410 Surface Roughness Machine

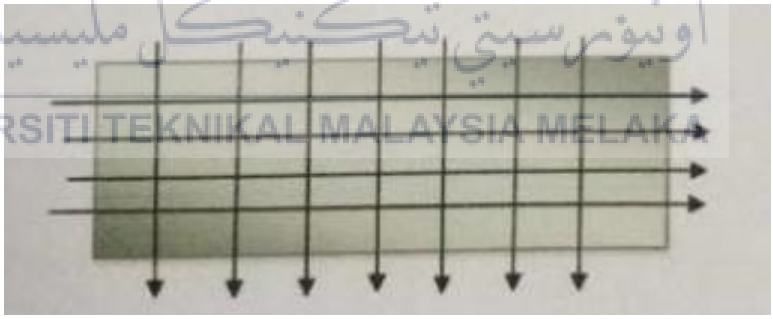


Figure 3. 15: Direction of Surface Measurement

3.4.3 Hardness Test (Vickers)

Hardness property is the characteristic of the material to represent the capability to resist the plastic deformation, bending, scratching or cutting. To conduct a hardness test, indenter is applied on the material and the depth of the indentation is used to define the resistance of material to indenter (Ametek, 2010). The deeper the indentation means the lower the hardness of the material.

In this study, Vickers hardness test is the most suitable test method to measure the hardness of the MgAZ91D due to the sample is moderate hard material. Vickers hardness test usually use for small parts, thin section or soft materials (George and Ryan, 2012). The loads for Vickers hardness test are in the range of light load, typically from 1 gf to maximum 100 kgf (England, no date). Figure 3.16 shows the Vickers hardness tester used to conduct the hardness test in this study.

Figure 3. 16: Vickers Hardness Tester

It is used a diamond indenter which in a square base pyramid shape with an angle 136 degree between the opposite face at vertex to do indentation. Figure 3.17 shows that the indentation of Vickers hardness diamond indenter (Gene, 2015).

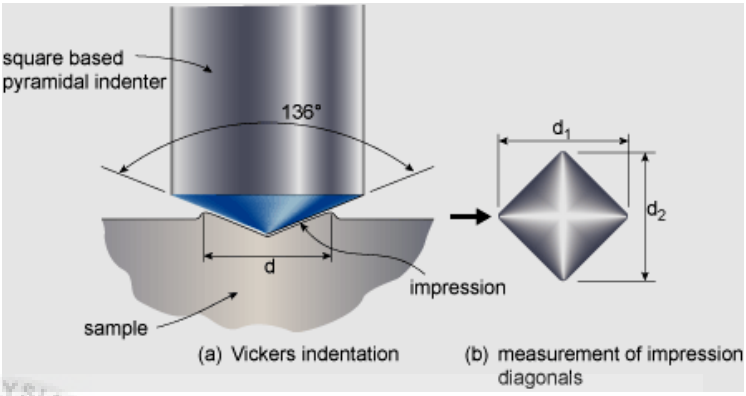


Figure 3. 17: Indentation of Vickers Hardness Diamond Indenter

The time to do indentation is from 2 s to 8 s for initial application and maintained the test force for 10s to 15s (England, no date). After removed the force, the diagonal lengths of the indentation are measured and calculated the arithmetic mean, d. The equation for Vickers hardness number, HV as shown as below:

HV = Constant × Test force / Surface area of indentation

$$HV = \frac{2\,F\,sin\frac{136^\circ}{2}}{d^2} \qquad (3.2)$$

Where F is the load applied and d is the mean of d_1 and d_2 in mm. In this study, the hardness test will conducted with 5 gf load and 15 seconds dwell time. Besides that, 20 times of the hardness test will done on each of the samples to determine the average of the measurement to ensure the hardness value are more accurate.

3.5 Summary of Methodology

As the conclusion, methodology of this study involved the details of gas nitriding process for MgAZ91D with different process temperature and time from sample preparation to analysis process. The samples were gone through the grinding, polishing, cleaning and nitriding process. After done the gas nitriding process, the samples were done the surface properties analyses such as microstructure analysis, surface roughness analysis and hardness test to compare the difference between the samples before and after nitrided to show the improvement of the samples and investigate the optimum nitriding process temperature and time for MgAZ91D.

CHAPTER 4

RESULTS AND DISCUSSIONS

The results obtained in this study were discussed and explained in this chapter. The results included surface roughness analysis, microstructure analysis using optical microscope and scanning electron microscope (SEM) and surface hardness analysis using Vickers hardness tester. These analyses were performed on the reference sample and nitrided samples of MgAZ91D with different nitriding process temperature and time to investigate the effect of different process parameters on the properties of MgAZ91D.

4.1 Surface Roughness Analysis

Surface roughness analysis was carried out to measure the difference of surface roughness in between reference sample and nitrided samples of MgAZ91D by using surface roughness tester. The surface roughness results, R_a value were included each reading of the samples, average and the standard deviation values as shown in the Table 4.1 and the graph of the average surface roughness versus process temperature at different time for nitrided and reference sample conducted is shown in the Figure 4.1. From the table and graph, it shows that the average surface roughness value for reference sample is 0.0331 μm and the values increase gradually with increasing of the nitriding process temperature and time. The sample with nitriding temperature at 350 ℃ and 6 hours shows the highest surface roughness value which is 0.1124 μm.

Table 4. 1: Surface roughness value, Ra of reference sample and nitrided samples of MgAZ91D

No. of readings (n)	Surface roughness value, Ra (μm)						
	Reference sample	350 ℃ 2 hours	350 ℃ 4 hours	350 ℃ 6 hours	450 ℃ 2 hours	450 ℃ 4 hours	450 ℃ 6 hours
1	0.040	0.067	0.052	0.093	0.089	0.102	0.096
2	0.033	0.044	0.062	0.117	0.108	0.095	0.088
3	0.028	0.071	0.081	0.119	0.116	0.123	0.100
4	0.043	0.070	0.06	0.111	0.107	0.104	0.106
5	0.023	0.059	0.065	0.115	0.113	0.102	0.109
6	0.031	0.053	0.069	0.104	0.099	0.087	0.097
7	0.037	0.074	0.053	0.094	0.085	0.113	0.101
8	0.036	0.068	0.054	0.089	0.115	0.084	0.110
9	0.037	0.070	0.099	0.101	0.090	0.152	0.109
10	0.031	0.057	0.074	0.107	0.088	0.090	0.100
11	0.024	0.066	0.095	0.123	0.106	0.104	0.101
12	0.028	0.075	0.067	0.131	0.119	0.102	0.109
13	0.021	0.056	0.076	0.135	0.109	0.095	0.116
14	0.034	0.075	0.061	0.125	0.116	0.103	0.108
15	0.028	0.078	0.093	0.112	0.103	0.092	0.118
16	0.036	0.071	0.063	0.097	0.088	0.103	0.120
17	0.031	0.080	0.061	0.116	0.113	0.124	0.103
18	0.036	0.074	0.062	0.123	0.117	0.123	0.118
19	0.044	0.072	0.072	0.095	0.103	0.110	0.127
20	0.041	0.071	0.073	0.140	0.100	0.105	0.124
Average	0.0331	0.0676	0.0696	0.1124	0.1042	0.1057	0.1080
Standard deviation	0.006512	0.009214	0.013613	0.014727	0.011134	0.015719	0.010136

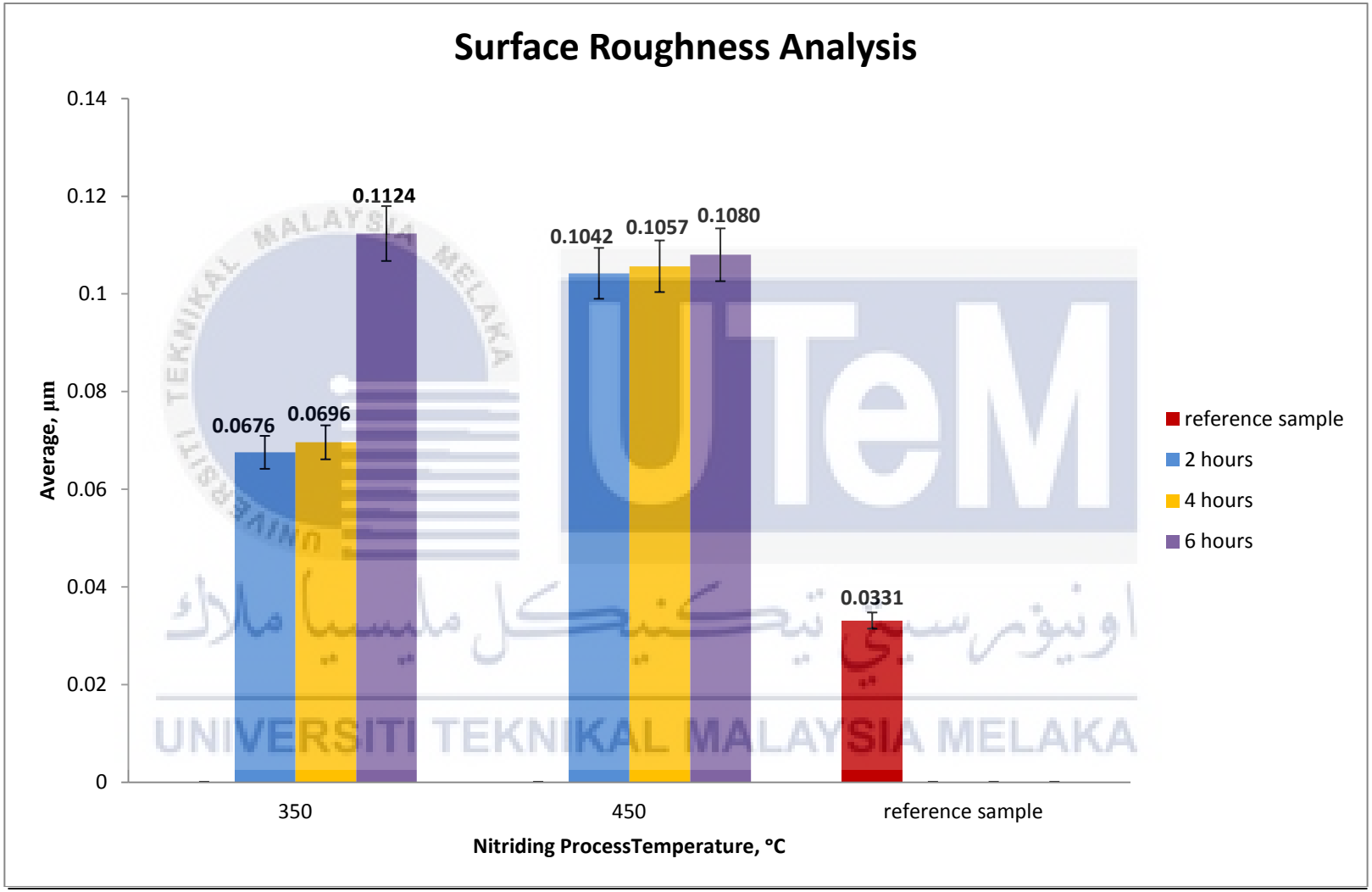


Figure 4. 1: Average surface roughness variation versus nitriding process temperature of MgAZ91D

Based on the graph shows that the surface roughness for the samples after the nitriding process are became rougher. The roughness of the nitrided samples with 350 ℃ for 2 hours and 4 hours increase gradually which from 0.0676 µm to 0.0696 µm. However, nitriding process at 6 hours shows that the surface roughness value increased sharply to 0.1124 µm which is the highest surface roughness value among all the samples. Mean while the surface roughness values for the nitrided sample at process temperature 450 ℃ have shows not much different although the process time increase. The value has increase from 0.1042 µm to 0.1080 µm. It is shows that the surface roughness of the samples performed at nitriding process temperature of 450 ℃ or higher may keep in a range only. The surface roughness values will not change significantly although the process time increased. This is because the diffusion level of the nitrogen gas into the MgAZ91D had reached to the maximum stage during the nitriding process temperature achieved 450 ℃ and most of the area for MgAZ91D have the reaction with nitrogen gas (WeiYang, 2013). Therefore, it is shows that the reactions between the samples and nitrogen gas have decreased, the appearance of the samples were not much different, and the surface roughness did not changed significantly after this temperature. On the other side, the surface roughness value for the samples with nitriding temperature 350 ℃ at will increase in a high amount if the process time increased due to the samples had the high reactivity with the nitrogen gaseous at this temperature (WeiYang, 2013).

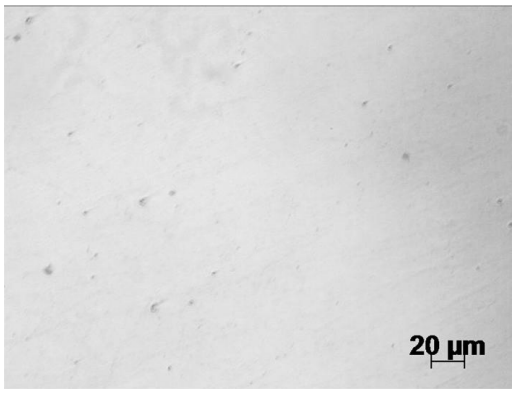
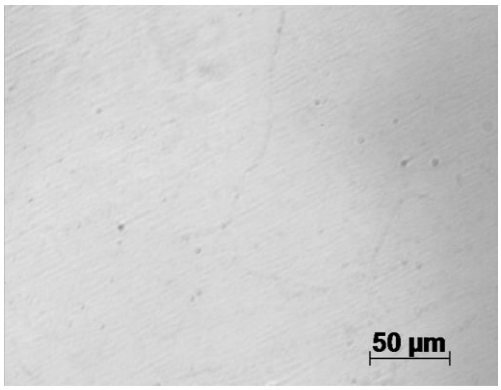
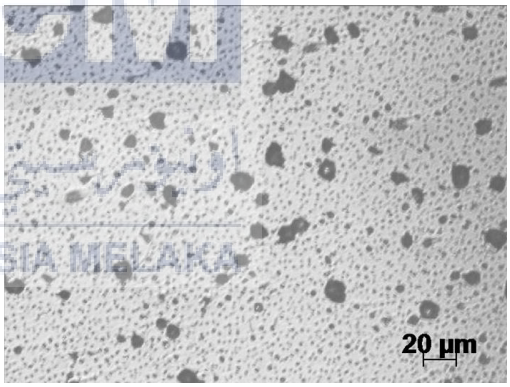
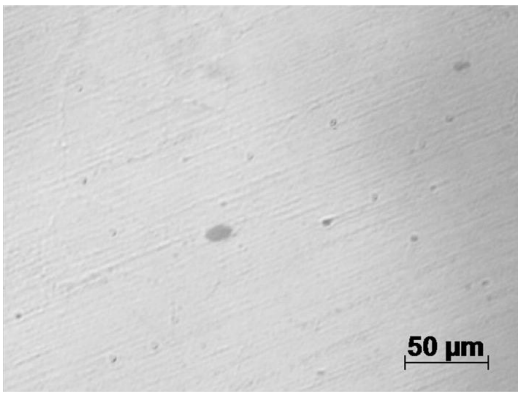
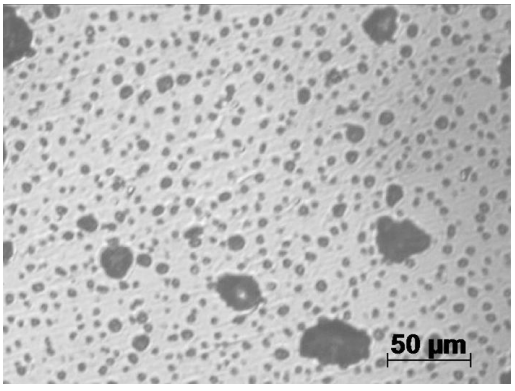
4.2 Microstructure Analysis using Optical Microscope and Scanning Electron Microscope

Microstructure analysis was conducted on the surface and cross section area of the reference sample and nitrided samples by using the optical microscope and scanning electron microscope (SEM) to study and analyze the microstructure of the samples.

4.2.1 Surface Microstructure

The results of surface microstructure for reference sample and nitrided samples is observed using optical microscope shown in the Table 4.2 with using 20x and 50x magnification and Table 4.3 shows the results of surface microstructure observed utilizing the Scanning Electron Microscope with the incident beam energy of 10kV and 4850x magnification.

Table 4. 2: Surface microstructure of reference sample and nitrided samples of MgAZ91D analyzed using optical microscope

Reference sample		
Mg AZ91D nitriding samples	350˚C	450˚C
2 Hours		
		

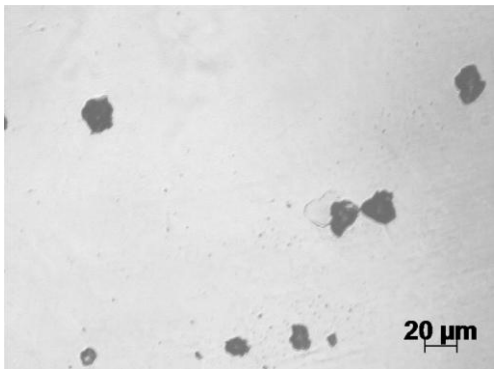
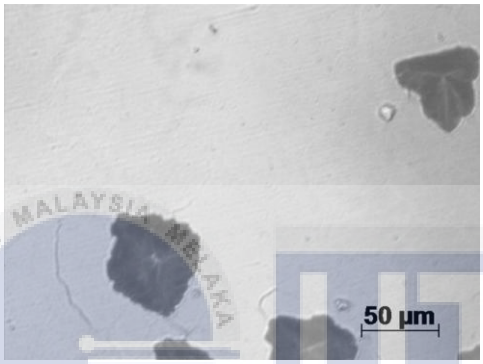
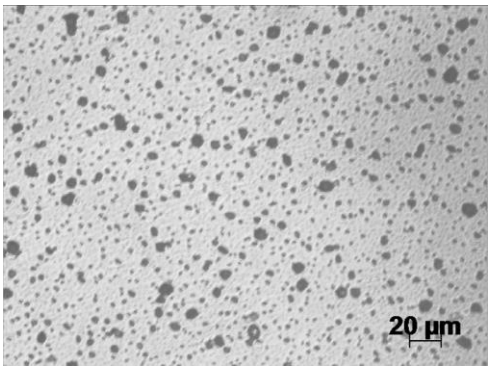
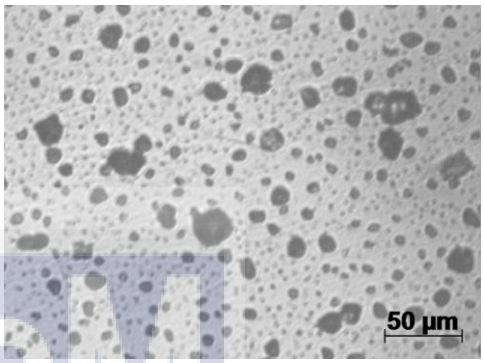
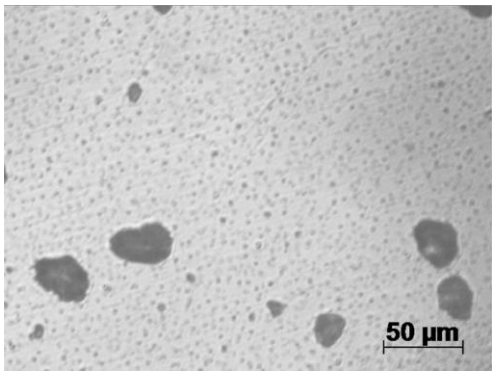
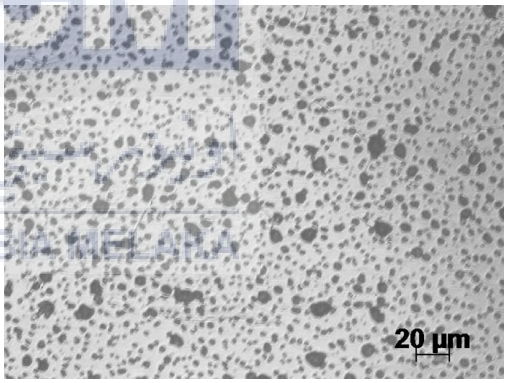
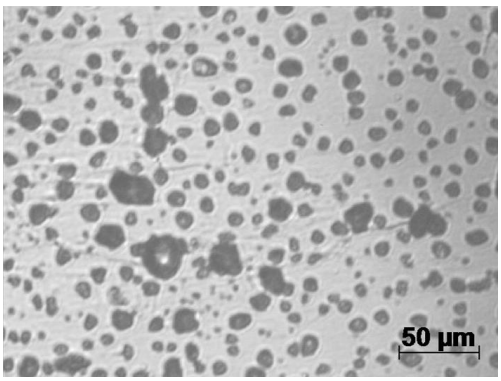
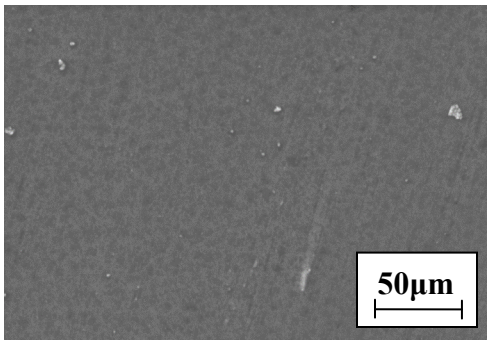
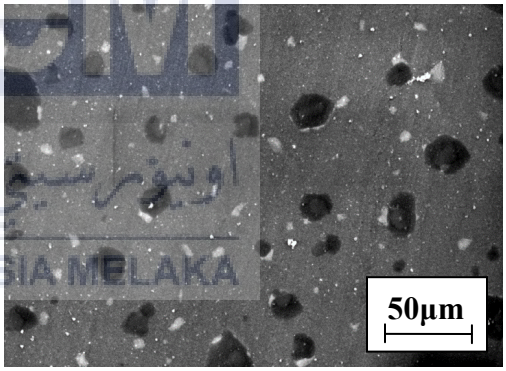
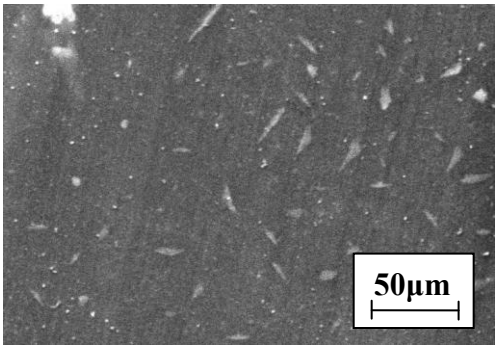
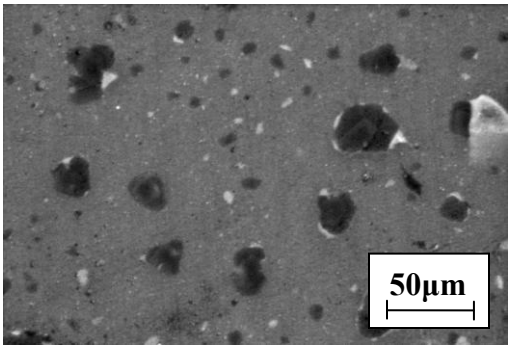
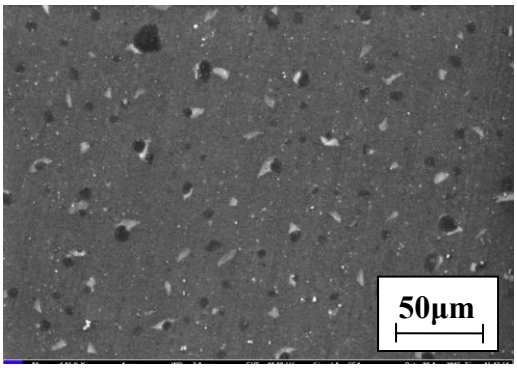
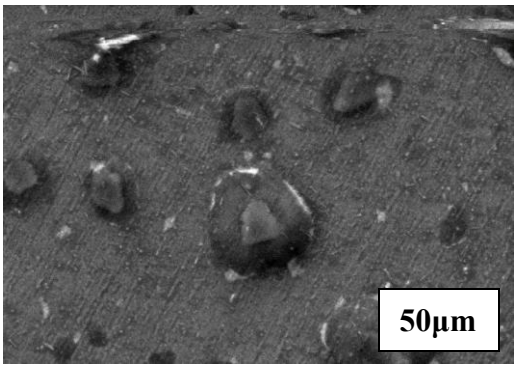
4 Hours	 	 
6 Hours	 	 

Table 4. 3: Surface microstructure of reference sample and nitided samples of MgAZ91D analyzed using scanning electron microscope (SEM)

Reference sample	

Mg AZ91D nitriding samples	350˚C	450˚C
2 Hours		
4 Hours		

6 Hours		

Based on the results of surface microstructure obtained, the surface condition of the reference sample is smooth and no any substance observed on the surface of the sample while there have some particles observed on the surface of the nitrided samples. The particles on the nitrided surface are observed increased by the size and quantity when the nitriding process time and temperature increased. This is due to the nitrogen gaseous easily to diffuse into the MgAZ91D samples in the high process temperature and long process time. The particles observed on the surface of the samples are related to the surface roughness value of the samples (Mohammad and WeiGao, 2008).

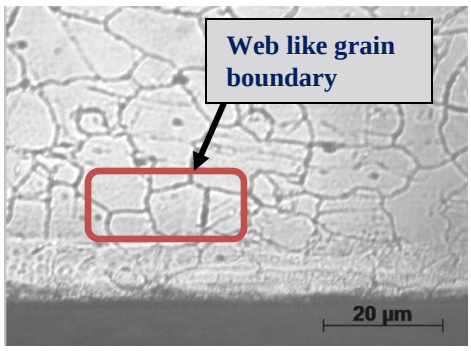
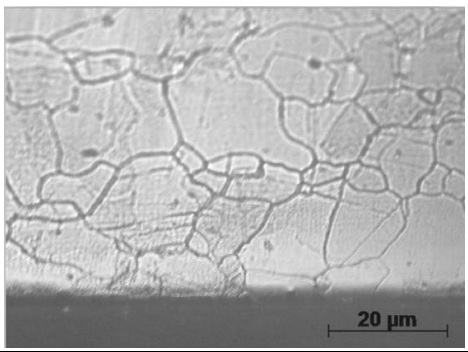
From the analysis show that at the highest the process temperature and time, will produce the highest quantities of particles. Besides that, it also shows that the highest quantities of the particles observed, the rougher the surface of the samples.

4.2.2 Cross Section Microstructure

The cross section microstructure analysis of the reference sample and nitrided samples were conducted by using optical microscope and scanning electron microscope. The results of cross section microstructure analysis for the reference sample and nitrided samples show in the Table 4.4 and 4.5 below. Table 4.4 shows the results of cross section microstructure analysis obtained by using optical microscope and Table 4.5 shows the results of cross section microstructure analysis obtained by using Scanning Electron Microscope with incident beam energy 15kV and 500 x magnification.

Table 4. 4: Cross section microstructure of reference sample and nitided samples of MgAZ91D analyzed using optical microscope

Reference sample	

Mg AZ91D nitriding samples	350˚C	450˚C
2 Hours		

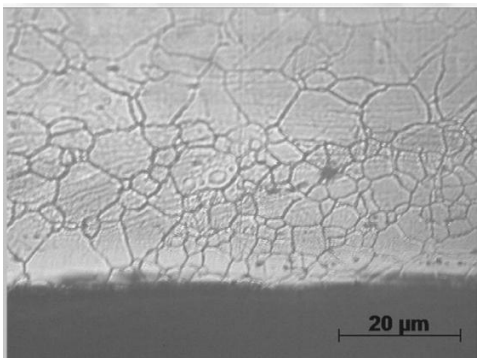
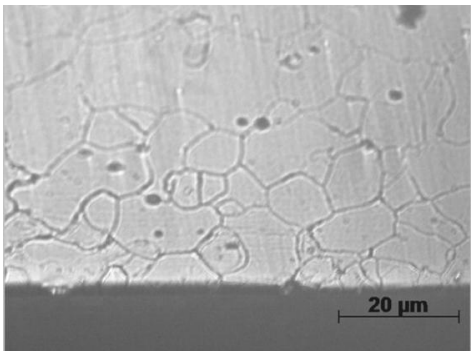
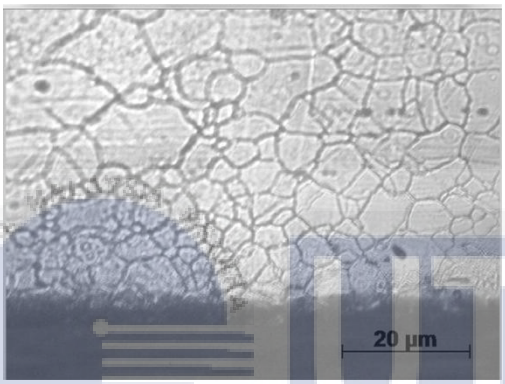
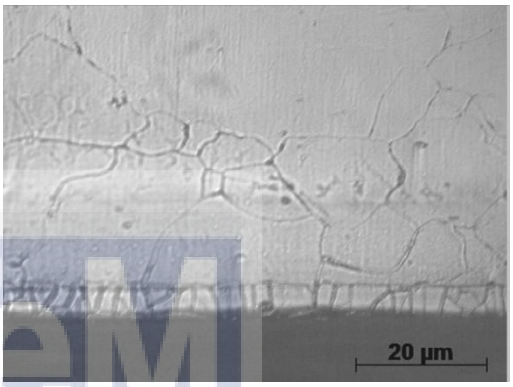
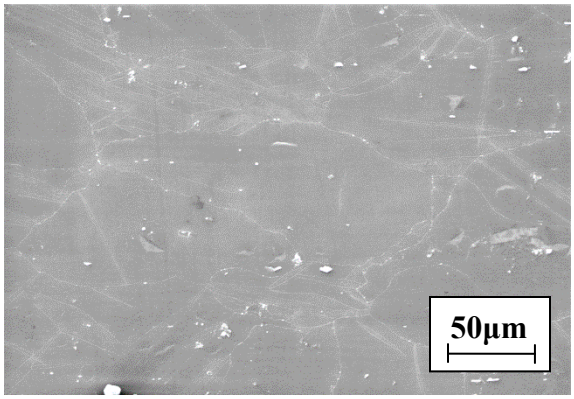
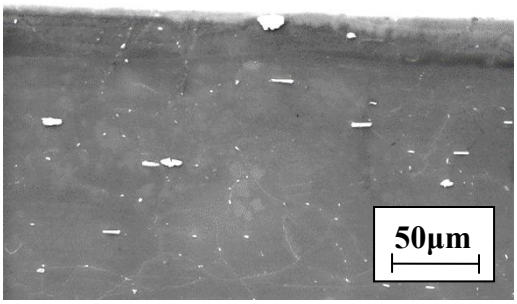
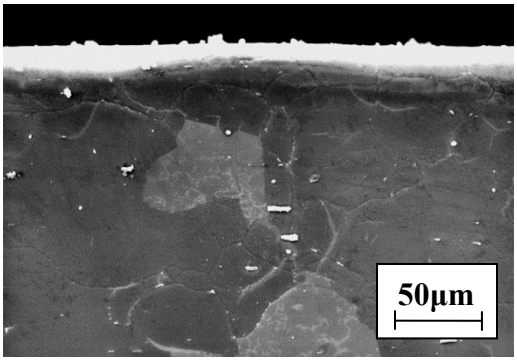
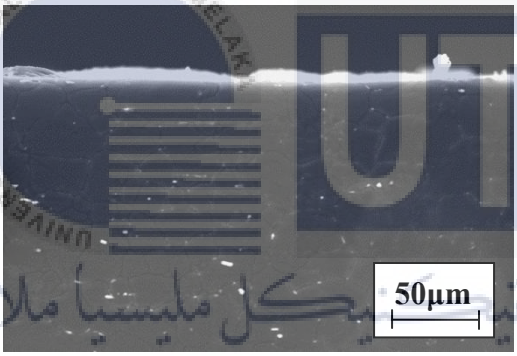
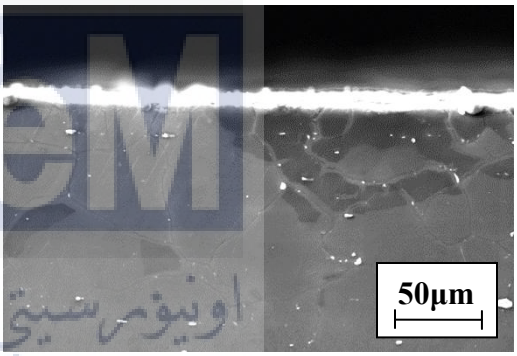
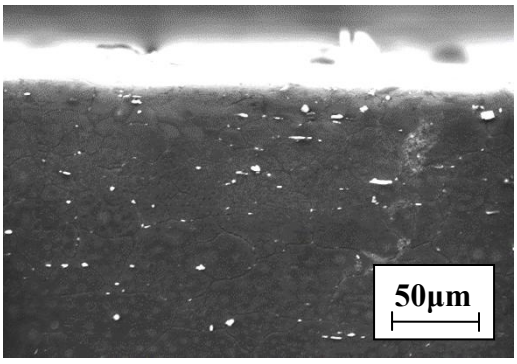
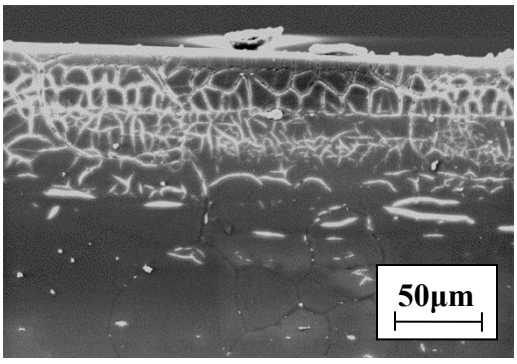
4 Hours		
6 Hours		

Table 4. 5: Cross section microstructure of reference sample and nitided samples of MgAZ91D analyzed using scanning electron microscope

Reference sample	

Mg AZ91D nitriding samples	350˚C	450˚C
2 Hours		
4 Hours		
6 Hours		

According to the of microstructure results, the shape of the microstructure for MgAZ91D alloy is a web like grain boundary. The grains size increase with the increasing of nitriding process temperature and time. This will lead the strength of the samples increases with the increase of grain size (Brush Wellman Inc., 2010). From this analysis showed that the nitriding process has improved the strength property of all these samples. The sample with nitriding process temperature and time 450 ℃ and 6 hours shows the largest grain boundaries size. Other than that, the result obtained also shows that the nitrided layer was formed clearly at process temperature 450 ℃ and 6 hours.

4.3 Surface Hardness

The surface hardness test of reference sample and nitrided samples of MgAZ91D were conducted by using the Vickers hardness tester to study the effect of the nitriding process with different process temperature and time on surface hardness properties. The hardness test was performed by using 5 grams indentation load and 15 seconds dwell time. The measurement of the hardness test is repeated for 20 times for each of the sample. The results of the surface hardness of reference sample and nitrided samples of MgAZ91D are shows in the Table 4.6. Based on the table, the average surface hardness value for the reference sample is 65.3 HV and the values have increase gradually during the nitriding process temperature and time increase. The highest average surface hardness value is 73.5 HV which increased 12.6% of surface hardness for the sample with the nitriding process at 450 ℃ and 6 hours.

Figure 4.2 shows the graph of the hardness variation versus nitriding process temperature with different process time for reference sample and nitrided samples obtained in this study.

Table 4. 6: Hardness value, HV of reference sample and nitrided samples of MgAZ91D

Hardness value, $HV_{0.005}$ / No. of readings (n)	Reference sample	350 ℃ 2 hours	350 ℃ 4 hours	350 ℃ 6 hours	450 ℃ 2 hours	450 ℃ 4 hours	450 ℃ 6 hours
1	63.9	67.7	63.9	72.6	70.7	81.0	78.0
2	61.3	64.9	67.7	66.6	73.3	80.2	79.5
3	63.3	67.7	63.3	68.3	76.6	68.3	71.3
4	64.4	68.9	70.7	72.0	73.3	67.7	68.9
5	64.9	62.8	73.9	70.1	68.3	68.3	76.6
6	66.0	67.2	63.9	68.9	67.7	72.0	75.3
7	63.9	61.8	66.6	67.2	70.7	70.1	72.6
8	60.3	69.5	70.1	84.1	76.6	79.5	69.5
9	66.0	63.3	62.3	76.6	66.0	67.7	83.3
10	66.0	64.4	66.0	68.9	74.6	84.9	64.9
11	63.9	63.3	67.2	73.9	72.0	77.3	69.5
12	65.5	67.7	72.0	72.0	69.5	63.3	73.3
13	66.0	62.8	66.6	62.8	72.0	76.6	80.2
14	68.3	66.0	70.1	73.3	70.7	75.3	73.3
15	66.6	67.2	61.8	62.3	73.3	78.8	73.9
16	67.7	70.1	63.9	67.7	72.0	73.9	73.3
17	67.7	65.5	66.0	68.3	68.9	76.6	64.9
18	64.9	63.3	65.5	73.3	76.6	68.9	73.3
19	69.5	63.9	66.6	75.3	67.7	67.2	72.6
20	65.5	66.0	67.2	76.6	68.3	70.1	75.3
Average	65.3	65.7	66.8	71.0	71.4	73.4	73.5
Standard deviation	2.2230	2.4670	3.2555	5.0688	3.1650	5.8201	4.7079

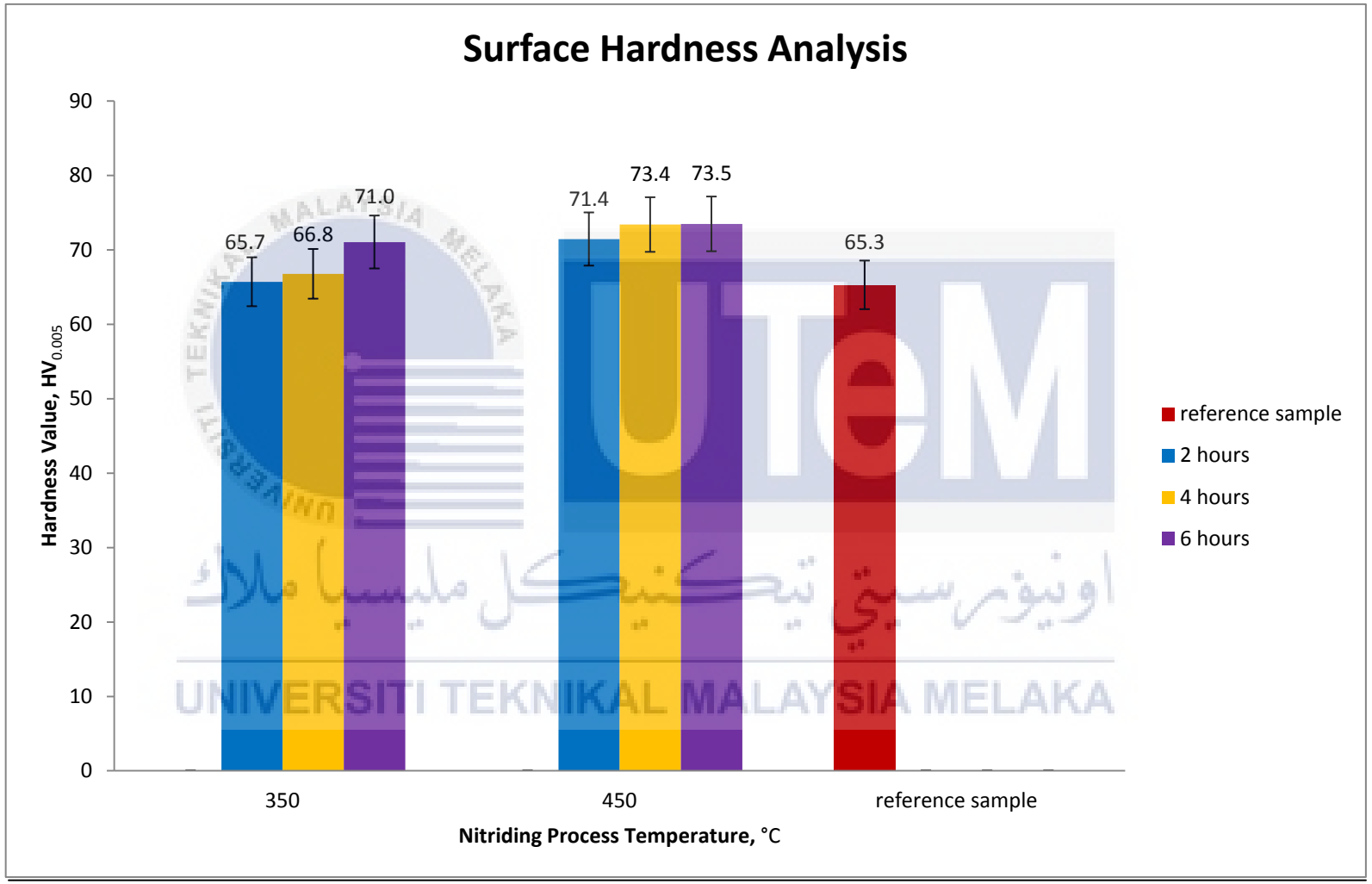


Figure 4. 2: Hardness variation versus nitriding process temperature of MgAZ91D

According to the Figure 4.2, the surface hardness of the nitrided samples had been improved in compared to the reference sample. Fleming (2012) stated that the nitriding process is able to enhance the surface energy level and hardness of the samples. Besides that, the surface concentration of nitrogen will increased due to increasing of temperature (Wu, 2013). From the result, the surface hardness values for the samples with process temperature 450 ℃ were higher than 350 ℃. The surface hardness values for the samples which go through the nitriding process temperature 350 ℃ increased significantly from time to time. The surface hardness value for the sample with nitriding process 350 ℃ and 2 hours is 65.7 HV which only slightly higher than the reference sample. When the process time for nitrided samples at 350 ℃ increased until 6 hours, the surface hardness value increased obviously up to 71.0 HV. On the other side, the surface hardness value for the samples go through the nitriding process temperature 450 ℃ have not much different although the nitriding process time increased. The sample which contained the highest surface hardness value, 73.5 HV is the nitrided sample with process temperature 450 ℃ and time 6 hours but it only increased the surface hardness 0.10 HV compare to the nitrided sample with process temperature 450 ℃ and 4 hours. This showed that the surface hardness of the samples will increased through increasing the nitriding process temperature and time but the surface hardness of the samples will maintain consistently if the nitriding process temperature up to 450 ℃. Although increased the process time, there have no significantly changed on the surface hardness value.

4.4 Summary of Results and Discussions

From the results, it shows that the surface properties of MgAZ91D samples are changed after the nitriding process according to different process temperature and time. By observing surface roughness results, it showed that the surface roughness of the samples are increased during increased the process temperature and time, but the surface roughness will not increased obviously if the process temperature raised to 450 ℃. In addition, the surface roughness results are supported by the results of surface microstructure based on the particles obtained on the surface of the samples increased will increase the roughness of the samples. The microstructure of the samples also will change due to the different process temperature and time. The grain size of the sample increased with increased the nitriding process temperature and time. Other than that, the surface hardness results proved that the nitriding process is able to enhance the surface hardness of the samples. By increasing the process temperature and time will increased the surface hardness of the samples. In a conclusion, the surface properties of the MgAZ91D are able to improve through the gas nitriding process by comparing the results of different analysis for the reference sample with the nitrided samples.

CHAPTER 5

CONCLUSION AND RECOMMENDATION

5.1 Conclusion

The surface properties analyses conducted in this study were proved that the gas nitriding process is able to enhance the surface properties of MgAZ91D by comparing the results of reference sample and nitrided samples. The analyses conducted are included roughness, microstructure and hardness analysis. There are six samples undergoes the gas nitriding process which with 350 ℃ and 450 ℃ for 2, 4 and 6 hours respectively. The results shows that the concentration of the nitrogen gas diffused into the surface of the samples will increased through increasing the nitriding process temperature and time. This is based on the roughness and the hardness of the samples are observed increased during the process temperature and time increased. Besides that, the size of grain boundaries also increased with increasing of the process temperature and time. This proved that the strength property of the samples will increased through the gas nitriding process with higher process temperature and time.

According to the results, the most suitable process temperature and time for gas nitriding process of MgAZ91D is 450 ℃ with 6 hours due to this sample has found with the harden and rougher surface after gas nitriding process. Besides that, this sample has the larger grain boundaries size which shows that the strength of the sample is increased.

5.2 Recommendation

In the future studies, the XRD analysis can be conducted to observe the phases of the samples. By obtaining this analysis, it can prove that the elements consist in the samples and the results will be more accurate. The presence of certain phases may be the reason of improving the surface properties of the samples.

Other than that, the samples preparation process also needs to be caution to get the well surface condition. The surface condition will affect the results of the analyses such as microstructure analysis. If the sample preparation process is not conducted well, the image for the microstructure may not occur. In addition, the etchant reagent used for the samples need to ensure it is suitable. This is because the wrong etchant is selected may destroy the surface of the samples and the grinding and polishing processes need to be repeat to obtain the desired finishing of the surface. This will waste the time and the chemical that use in the sample preparation process.

CHAPTER 6

PROJECT POTENTIAL

In the previous chapter of this study stated that MgAZ91D has poor creep resistance and poor mechanical properties in high temperature condition. Due to the limitations, the application of MgAZ91D in industries is restricted. In this study, gas nitriding process as an alternative of surface treatment process is conducted to enhance the surface properties of MgAZ91D. The gas nitriding process is conducted with various process temperature and time to analyze the suitable process temperature and time. Moreover, this study shows that the surface properties of MgAZ91D have been improved after the gas nitriding process. Thus, this method can be applied to enhance the surface properties of MgAZ91D and expand the uses of it in various applications for the industries such as aerospace and automotive.

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APPENDICES

Project Planning PSM 1

Project Activity	Week														
	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Understanding the title															
Identify the problems															
Research about the relevance articles and journal															
Draft of proposal															
Finalize the proposal															
Do literature review															
Preparation for report introduction															
Preparation for report literature review															
Preparation for report methodology															
Finalize the final report															
Preparation for presentation															
Presentation for PSM 1															
Report submission PSM 1															

Process Planning PSM 2

Project Activity	Semester Break						Week													
	July		August				1	2	3	4	5	6	7	8	9	10	11	12	13	14	15
Preparation of materials																					
Laboratory session																					
(i) Gas nitriding process																					
(ii) Surface roughness analysis																					
(iii)Microstructure analysis																					
(iv)Hardness analysis																					
Preparation for report result and discussion																					
Preparation for report conclusion																					
Finalize the final report																					
Submission of full report																					
Preparation for presentation																					
Final presentation																					