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