



**A Study on Corrosion Resistance of Sodium  
Metavanadate ( $\text{NaVO}_3$ ) Coated on AZ91D Magnesium  
Alloy by Anodizing Technique**

By

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A thesis submitted in fulfillment of the requirements for the degree of  
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**School of Manufacturing Engineering  
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i

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## LIST OF ABBREVIATIONS

Al	Aluminium
AFM	Atomic Force Microscope
ANOVA	Analysis of Variance
Cl <sup>-</sup>	Chloride ion
DOE	Design of Experiment
EDX	Energy Dispersive X-Ray
Hr	Hour
Mg	Magnesium
MgO	Magnesium Oxide
Mg (OH) <sub>2</sub>	Magnesium Hydroxide
MgV <sub>2</sub> O <sub>6</sub>	Magnesium Vanadium Oxide
Mn	Manganese
MPY	Miles per Year
Min	Minute
NaCl	Sodium Chloride
NaVO <sub>3</sub>	Sodium Metavanadate
RE	Rare Earth
SEM	Scanning Electron Microscope
V	Vanadium
XPS	X-ray Photoelectron Spectroscopy
XRD	X-ray Diffraction
Zn	Zinc

## LIST OF SYMBOLS

$\alpha$	Alpha Phase
$\beta$	Beta Phase
$\mu\text{m}$	Micrometer
$\text{mA}/\text{cm}^2$	Mili Ampere per Centimeter Squared
$\text{mm}/\text{y}$	Millimeter per Year
V	Voltage (V)

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## Kajian Mengenai Rintangan Kakisan Salutan Sodium Metavanadate ( $\text{NaVO}_3$ ) Terhadap Aloi Magnesium AZ91D Melalui Teknik Penganodan

### ABSTRAK

Penganodan atau salutan anodik pengoksidaan telah menarik minat di dalam kajian ini kerana berpotensi untuk memperbaiki rintangan kakisan aloi. Salutan anodik pengoksidaan adalah kaedah lapisan yang dipilih untuk aplikasi yang istimewa seperti rintangan kakisan untuk kerja berat. Kebelakangan ini, sebatian kromat telah digunakan secara meluas untuk menghasilkan salutan bagi melindungi permukaan paling logam seperti aluminium, keluli dan aloi magnesium. Ketoksikan kromat heksavalen (IV) untuk mengawal kakisan adalah perkara penting perlu diambil kira dalam industri logam. Setelah menggabungkan kesan ekonomi akibat kerosakan kakisan, masalah alam sekitar dan kesihatan yang disebabkan oleh kromat heksavalen (IV), ahli-ahli sains mempunyai insentif yang besar untuk membangunkan satu sistem salutan perlindungan yang mesra alam. Tujuan kajian ini dijalankan adalah untuk menentukan rintangan kakisan terhadap aloi magnesium AZ91D dengan menggunakan sodium metavanadate ( $\text{NaVO}_3$ ) sebagai penyelesaian pembentukan salutan. Selain itu, untuk mengkaji mikrostruktur permukaan, ketebalan, kekasaran permukaan dan komposisi kimia bagi salutan dan sumbangannya kepada rintangan kakisan. Kemudian untuk menyiasat sifat dan pencegahan pengaratan aloi magnesium AZ91D. Sodium metavanadate ( $\text{NaVO}_3$ ) digunakan sebagai larutan elektrolit untuk menghasilkan lapisan. Pendekatan perancangan pemfaktoran  $2^k$  digunakan di dalam kajian ini untuk merancang eksperimen. Design Expert 7.0.0 digunakan bagi membentuk sebuah eksperimen. Faktor penting di dalam proses salutan yang dipilih adalah kepekatan sodium metavanadate ( $\text{NaVO}_3$ ) (g/l), masa anodik (min) dan ketumpatan arus ( $\text{mA/cm}^2$ ). Ujian pengaratan dijalankan bagi menentukan kadar pengaratan aloi magnesium AZ91D yang disalut. Keputusan ujian kadar pengaratan akan dianalisa dan dinilai juga menggunakan Design Expert 7.0.0. Teknik pengimejan optik, Mikroskop Elektron Imbasan (SEM) dengan Sebaran Tenaga Keupayaan (EDX), Belauan Sinar X (XRD) dan ujian kekasaran digunakan untuk mengkaji mikrostruktur permukaan, ketebalan salutan, struktur fasa dan kekasaran permukaan. Kesan penyelesaian vanadia, masa rawatan dan ketumpatan arus terhadap prestasi perlindungan kakisan aloi magnesium telah disiasat menggunakan ujian keupayaan pengutuban lengkungan dalam 3.5 % sodium klorida (NaCl). Keputusan menunjukkan bahawa penggunaan 0.1 g/l sodium metavanadate ( $\text{NaVO}_3$ ) pada 10  $\text{mA/cm}^2$  ketumpatan arus selama 5 minit masa rawatan mampu digunakan untuk menghasilkan lapisan bagi memperbaiki ketahanan daya kakisan AZ91D magnesium aloi. Lapisan yang padat dan tebal yang terdiri daripada MgO dan  $\text{MgV}_2\text{O}_6$  di permukaan AZ91D magnesium aloi bertindak sebagai penghalang daripada diserang pengaratan. Lapisan yang terhasil di permukaan AZ91D magnesium aloi juga mampu menahan aktiviti pengaratan apabila mendapati bahawa kurangnya kawasan berkarat, teretusnya suatu keadaan pembaikan di mana terdapat aktiviti penyembuhan secara semulajadi, turunnya ketumpatan arus pengaratan dan naiknya ketahanan polarisasi.

## **A Study on Corrosion Resistance of Sodium Metavanadate (NaVO<sub>3</sub>) Coated on AZ91D Magnesium Alloy by Anodizing Technique**

### **ABSTRACT**

Anodizing also known as anodic oxidation coatings have attracted significant interest in the study because of their potential to improve the corrosion resistance of alloys. Anodic oxidation coatings are usually chosen for special applications heavy duty corrosion resistance is required. Previously, chromate compounds were widely used for producing conversion coatings to protect most metallic surfaces such as aluminum, steel and magnesium alloys. In spite of its toxicity hexavalent chromate (IV) has remain an essential ingredient in the metal finishing industry for corrosion control. But combining the economic impact of corrosion damage, the environmental and health problems caused by hexavalent chromate (IV), and the increasing the regulatory restrictions, scientists have a huge incentive to develop a new generation of environmentally friendly protective coating systems. The objective of this research is to determine the corrosion resistance of AZ91D magnesium alloy utilizing sodium metavanadate (NaVO<sub>3</sub>). Besides, to study on surface microstructure, thickness, surface roughness and chemical composition related to the coating and its contribution to corrosion performance. Then, investigate the corrosion behavior and corrosion protection mechanism of AZ91D magnesium alloy. Sodium metavanadate (NaVO<sub>3</sub>) is used as anelectrolytesolution to form coating in this research. The approach used in this study for design of experiment is 2<sup>k</sup> factorial design. The design of experiment is using Design Expert 7.0.0. The samples are prepared for the surface treatment. Three factors such as concentration of sodium mtavanadate (NaVO<sub>3</sub>) (g/l), anodizing time (min) and current density (mA/cm<sup>2</sup>) are involved in coating process. The corrosion test is carried out for the coated sample to determine the corrosion rate. The result is to be analyzed and the performance is evaluated statistically again using Design Expert 7.0.0. The surface microstructure, thickness, phase structure, surface roughness of the coating were studied by optical microscope, scanning electron microscope (SEM) and energy dispersive X-ray (EDX), X-ray diffractometer (XRD) and roughness test, respectively. The effects of vanadia solution, anodizing time and current density on the corrosion protection performance of magnesium substrate were investigated by Potentiodynamic polarization test in 3.5% Sodium Chloride (NaCl). These results with use 0.1 g/l of NaVO<sub>3</sub> at current density 10 mA/cm<sup>2</sup> for 5 min anodizing time is capable to be used to form coating to improve the corrosion resistance of AZ91D magnesium alloy. AZ91D magnesium alloy coated in 0.1 g/l of NaVO<sub>3</sub> for 5 min at 10 mA/cm<sup>2</sup> provide smooth, compact, thick and composed with MgO and MgV<sub>2</sub>O<sub>6</sub> on the surface of magnesium alloy which act as barrier from corrosion attack. Coated AZ91D magnesium alloy has highest corrosion resistance as reduced in corroded area, produced good self-healing functionality for pitting repairing, increased the corrosion potential, decreased the corrosion current density and increased the polarization resistance.

## CHAPTER 1

### INTRODUCTION

#### 1.1 Background study

Magnesium is well known as the lightest metal with lowest density of  $1.74 \text{ g/cm}^3$  as to be compared to all the engineering metals. It is 35% lighter than aluminum ( $2.7 \text{ g/cm}^3$ ) and four times lighter than steel ( $7.86 \text{ g/cm}^3$ ). Recently, many automotive manufacturing industries are start looking for lighter material than aluminum, zinc and steel (Musfirah and Jaharah, 2012). It is proven that the weight of a car influences fuel consumption. More weight results in increased fuel consumption. High demand for light weight automotive leads to the usage of magnesium has created opportunities to replace aluminum, zinc and plastic components. Electrical industries also start looking for lightest material and high temperature application to substitute plastic with other material which is magnesium.

Magnesium has many advantages that make it best choice in automotive application such as:

- i. Strong and tough.
- ii. Good machine ability.
- iii. Ductility.
- iv. Easy recycling.

Magnesium also has many advantages over plastic for electrical application because:

- i. Plastic parts are often subject to dimensional stability issues, surface deterioration, fit issues due to temperature change and lack of rigidity.
- ii. Magnesium has greater impact resistance and energy absorbing capacity over plastics.

Magnesium in automotive application is suitable for steering wheels, clutch pedal, brake pedal, valve cover and chain housing and gearbox housing. While magnesium for electrical application is suitable for computer casing and camera casing. Commonly for engineering application, magnesium is usually alloyed with one or more elements, which include Aluminum (Al), Manganese (Mn), Rare Earth (RE) metals, lithium, Zinc (Zn) and Zirconium (Zr) (Kulekci, 2007). Magnesium alloy are recognized alternatives to iron and aluminum to reduce the weight of structural materials.

In this project, magnesium with AZ91 will be focused as being the most widely used. This is because cast magnesium alloys dominate 85-90% of all magnesium products (Nazihah, 2013). Parts and components presently manufactured in Malaysia are for aesthetic, structural and functional uses for the electronics, telecommunication and automotive industries (Ministry of Human Resources, 2010). AZ91D magnesium alloy which contains approximately 9% of Al and 1% of Zn is commonly used in automotive industry for having a better corrosion resistance. In recent years, magnesium alloy, previously used only in a limited range of application, have served as automotive parts and cases for portable electronics devices such as notebook and camera. The B80 gearbox housing made of AZ91D which was introduced by VW/Audi in 1996 marked