



UNIVERSITI TEKNIKAL MALAYSIA MELAKA

**ELECTROCHEMICAL BEHAVIOUR OF ELECTROLESS
TERNARY NICKEL (Ni-W-P) ALLOY DEPOSIT ON IRON
SUBSTRATE IN 3.5wt% NaCl SOLUTION**

This report submitted in accordance with requirement of the university Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering (Engineering Materials) (Hons.)

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DECLARATION

I hereby, declared this report entitled “Electrochemical Behavior of Electroless Ni-W-P Alloy Deposit on Iron Substrate in 3.5 wt% NaCl Solution” is the results of my own research except as cited in references.

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Date : 23rd June 2014

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfillment to the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Materials) (Hons.). The member of the supervisory is as follow:

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(Dr. Muhammad Zaimi Bin Zainal Abidin)

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ABSTRAK

Aplikasi kejuruteraan untuk tanpa elektrik aloi nikel pemendapan boleh didapati dalam hampir dalam setiap industri. Kepentingan dalam penyaduran tanpa elektrik berasaskan nikel aloi pertigaan telah meningkat kerana kakisan yang sangat baik mereka, memakai, rintangan haba dan elektrik, dan juga keupayaan untuk plat permukaan bukan konduktif menjadikan ini salutan pilihan bagi aplikasi kejuruteraan. Dalam penyiasatan ini, kesan tungsten (W) dalam alkali mandi elektrolit nikel yang telah dikaji dalam mendepositkan aloi Ni-W-P di Fe substrat. Penurunan fosforus (P) kandungan dan peningkatan sedikit dalam saiz bijian telah diperhatikan disebabkan penambahan tungsten dalam deposit Ni-P. Oleh itu, peningkatan kecil dalam rintangan kakisan telah diperhatikan dalam aloi pertigaan. Untuk mencirikan deposit, beberapa analisa akan dijalankan, iaitu morfologi permukaan oleh X-Ray Belauan (XRD), Imbasan Elektron Mikroskop (SEM), Tenaga-X Sebaran Ray-Analisis (EDX) dan Mikroskop optik (OM) teknik digunakan, Elektrokimia Pengukuran (ECM) dan ujian kekerasan.

ABSTRACT

Engineering applications for electroless nickel can be found in virtually in every industry. Interest in electroless plating of nickel-based ternary alloys has increased because their excellent corrosion, wear, thermal and electrical resistance, as well as the ability to plate non-conductive surfaces makes this a coating of choice for many engineering applications. In this investigation, the effect of tungsten (W) in an alkaline electroless nickel baths that has been studied in depositing Ni-W-P alloys on Fe substrate. A decrease in phosphorus (P) content and a marginal increase in grain size have been observed due to the tungsten addition in the Ni-P deposit. Therefore, a marginal improvement in corrosion resistance has been observed in ternary alloy. In order to characterize the deposits, several analysis are will be carried out which are surface morphology by X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy-Dispersive X-Ray Analysis (EDX) and Optical Microscope (OM) techniques are used, Electrochemical Measurement (ECM) and hardness test.

DEDICATION

This report is lovingly dedicated to my respective parents; Mr. Baharom Bin Mohammed and Mrs. Rokiah Binti A Karim, my supervisor; Dr. Muhammad Zaimi Bin Zainal Abidin, my family's members and also to all my friends who have been supported and inspired me to do this project.

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LIST OF ABBREVIATIONS AND NOMENCLATURE

XRD	-	X-Ray Diffraction
SEM	-	Scanning Electron Microscopy
FE-SEM	-	Field-Emission Scanning Electron Microscope
PVD	-	Physical Vapor Deposition
CVD	-	Chemical Vapor Deposition
NaCl	-	Sodium chloride
NiSO ₄ .6H ₂ O	-	Nickel sulphate
NaH ₂ PO ₂ .2H ₂ O	-	Sodium hypophosphite
Na ₃ C ₆ H ₅ O ₇ .2H ₂ O	-	Sodium Citrate
CH ₃ COONH ₄	-	Ammonium Acetate
NaWO ₄ .2H ₂ O	-	Sodium Tungstate
H ₂ O	-	Water
I _{corr}	-	Corrosion current
E _{corr}	-	Corrosion potential

LIST OF SYMBOLS

α	-	Alpha
$^{\circ}\text{C}$	-	Degree Celcius
mm	-	Milimeter
wt%	-	Weight percentage
i_a	-	Anodic
i_c	-	Cathodic
mV	-	Milivolt
A	-	Area
D	-	Density
μm	-	Micrometer

CHAPTER 1

INTRODUCTION

1.1 Background of study

Nowadays, carbon steel is one of the major construction materials used extensively in chemical and applied industries dealing with alkaline, acidic and salt solutions. From the research by Noor and Al-Moubaraki, 2008, the corrosion of carbon steel is a major infrastructure degradation problem in industries, including the chemical, mineral, materials and petrochemical worldwide industries. Corrosion can be defined as the destruction or deterioration of a material because of reaction with its environment.

Thus, to prevent the corrosion from occur continuously, the protection method are applied such as alloying, painting, galvanization, and coating. These methods are creating a protective layer for the metal surface that can prevent from corrosion. The corrosion resistance properties can be improved by alloying the substrate with more corrosion resistance metal like nickel (Ni), tungsten (W), zinc (Zn), tin (Sn), and cobalt (Co). For example, tin-plated carbon steel is frequently used for production of beverage cans, food cans, and aerosol cans because of its excellent corrosion resistant properties (Oni et al., 2008)

In this study, coating method has been chosen in order to improve the corrosion resistance of iron substrate. Coating can extend the material use of limitation at upper end of their performance capabilities, by allowing the mechanical properties of the

substrate material to be maintained while protecting them against wear and corrosion by a protective coating (Palaniappa and Seshadri, 2008). The coating method is divided into dry and wet process. There is a lot of type of coating methods available such as Physical Vapour Deposition (PVD), Chemical Vapour Deposition (CVD), painting, electrodeposition, electroless deposition, spraying and chemical conversion. Among of these coating methods, electroless deposition method that has been introduced by Brenner and Riddell is chosen, that do not require any electricity. Electroless nickel coatings are the more popular variant of electroless coating which can enhance corrosion and wear resistance (Prasanta and Suman, 2010).

Electroless deposition is an autocatalytic process where the substrate develops a potential when it is immersed on the various plating bath solution that contains a source of metallic ion, reducing agent (sodium hypophosphite, NaPO_2H_2), complexing agent (sodium citrate), stabilizer and buffers. The chemical deposition of metal from the solution has an electrochemical mechanism which is oxidation and reduction (redox). The loss of electron is presented of oxidation of anodic process while reduction is extinguished by a gain of electrons indicates cathodic action.

Particularly, electroless binary nickel-phosphorus (Ni-P) alloy plating has been used as a functional coating to enhance the corrosion resistance and wear resistance properties of the material. Instead, the coating is deposited on the part's surface by reducing nickel ions to metallic nickel with sodium hypophosphite (NaPO_2H_2). In fact, development of electroless nickel polyalloy deposits can be considered as the most effective method to alter the chemical and physical properties of binary Ni-P alloy deposits. Therefore, tungsten (W) is adding into binary Ni-P alloy to produce ternary Ni-W-P alloy has been introduced by Pearlstein and Weightman in 1963. Codeposition of W in binary electroless Ni-P deposit improves the deposit characteristics such as corrosion resistance, wear resistance, thermal stability and electrical resistance. By the addition of even small amounts of tungsten, the hardness of binary Ni-P matrix greatly increases (Balaraju et al., 2006). Furthermore, due to the tungsten addition in the Ni-P deposit a decrease in phosphorus (P) content and a marginal increase in grain size have been observed. Therefore, a marginal improvement in corrosion resistance has been observed in ternary alloy.

In addition, the surface morphology of electroless ternary Ni-W-P has been improved by varying the concentration of sodium tungstate in the plating process and the pH of the baths (Balaraju et al., 2006). Hence, the deposit structure and surface morphology can be analysed by X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy-Dispersive X-Ray Analysis (EDX), and Electrochemical Measurement (ECM). Hardness test also should be carried out to test the strength of the material, as the example, hardness tester and Vickers Hardness Test. The electrochemical behavior of the alloy coating in 3.5wt% NaCl solution is also investigated using electrochemical measurement method such as Tafel Extrapolation technique and Anodic Polarization Measurement.

In this research, the target is to improve the corrosion resistance and the hardness of iron substrate by using electroless deposition coating method. The binary electroless Ni-P alloy is adding with tungsten as the third element to produce ternary Ni-W-P directly to enhance the properties. The surface morphology and the electrochemical behavior of the ternary alloy is also investigated.

1.2 Problem statement

Iron (Fe) is a type of metal that can undergo corrosion under ambient environment. In order to increase the life of the metal and enhance corrosion resistance, the iron should be coated by a method that can improve the hardness, strength, wear resistance, corrosion resistance and will provide uniform coatings. However, there are a lot of methods are available. The first problem faced in this study is to investigate the suitable method to produce ternary Ni-W-P alloy by using electroless deposition method. By adding W element into the nickel alloy coating, mechanical properties such as hardness as well as corrosion resistance can be enhanced. In this study, the corrosion behaviour of nickel alloy deposit on iron substrate is compared with stainless steel in sodium chloride solution to study the effect of W addition into the nickel alloy matrix. Furthermore, optimum composition of the electroless Ni-W-P alloy coatings for corrosion resistance in sodium chloride solution is need to be studied by varying the plating bath pH during deposition.

1.3 Objectives

The purpose of doing this study is to study the effect of ternary alloy, Ni-W-P deposition onto the Fe substrate composition. The study is carried out that relate with two objectives:

- i. To analyze the effect of plating bath pH on electroless Ni-W-P alloy deposition on Fe substrate composition.
- ii. To investigate the effect of nickel alloy composition from various bath pH on electrochemical behaviour in 3.5wt% salt solution.

1.4 Scope

The focus of this study is to study the electrochemical behaviour of electroless Ni alloy coating onto the iron (Fe) substrate. Further, electroless deposition method is used to coat the substrate by applying Ni alloy deposit on the iron substrate in order to increase the corrosion and wear resistance. The substrate must undergo a few process such as cleaning and degreasing, etching which is degreasing process by acetone (ultrasonic), and rinsing before proceed to the electroless Ni alloy deposition.

Additionally, the coating of the Fe substrate will be done at several plating bath pH condition in order to study the optimum composition of deposition and also the electrochemical behaviour in the salt solution. In addition, the surface morphology of electroless ternary Ni-W-P can be improved by varying the concentration of sodium tungstate in the plating process and the pH of the baths.

The deposit structure and surface morphology of the alloy can be analysed by X-Ray Diffraction (XRD), Scanning Electron Microscopy (SEM), Energy-Dispersive X-Ray Analysis (EDX), and Electrochemical Measurement (ECM). Other than that, hardness test also should be carried out to test the strength of the material using hardness tester and Vickers Hardness Test. The electrochemical behavior of the alloy coating in NaCl solution is also investigated using electrochemical measurement method such as Tafel Extrapolation technique and Anodic Polarization Measurement.