



**EFFECT OF CITRATE BATH COMPOSITION AND HEAT  
TREATMENT PROCESS ON THE PROPERTIES OF  
ELECTRODEPOSITED NICKEL-FLY ASH (Ni-FA) COMPOSITE  
COATING ON ALUMINIUM ALLOY 7075 SUBSTRATE**

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

by

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**BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA**

Tajuk: **EFFECT OF CITRATE BATH COMPOSITION AND HEAT TREATMENT PROCESS ON THE PROPERTIES OF ELECTRODEPOSITED NICKEL-FLY ASH (Ni-FA) COMPOSITE COATING ON ALUMINIUM ALLOY 7075 SUBSTRATE**

Sesi Pengajian: **2016/2017 Semester 2**

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## **APPROVAL**

This report is submitted to the Faculty of Manufacturing Engineering of Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirement for Degree of Manufacturing Engineering (Engineering Materials) (Hons). The member of the supervisory committee are as follow:

.....  
**(Dr. Intan Sharhida Binti Othman)**

## **ABSTRAK**

Aluminium 7075 (AA7075) dielectroenapan oleh salutan nikel dan abu melalui pelbagai komposisi natrium sitrat melalui proses rawatan haba yang berbeza. Tujuan kajian ini adalah untuk mengkaji sifat-sifat mekanikal iaitu kekerasan dan sifat kakisan Ni-FA salutan komposit AA7075 berdasarkan pelbagai komposisi natrium sitrat. Selain daripada itu, untuk mengkaji kesan proses penyepuhlindapan ke atas sifat-sifat lapisan Ni-FA komposit Proses elektroenapan menggunakan mandi sitrat yang mengandungi natrium sitrat di 400 °C suhu. Pelbagai natrium sitrat iaitu 30 g / L, 50 g / L, 70 g / L dan proses rawatan 90 g / L dan haba pada 200 °C dan 400 °C. Morfologi permukaan salutan telah diperiksa dengan menggunakan Scanning Electron Microscopy (SEM), fasa lapisan komposit telah dikenal pasti dengan menggunakan X-Ray Diffraction (XRD) dan komposisi zarah abu terbang telah ditentukan dengan menggunakan X-Ray pendarfluor (XRF) . The morfologi dan struktur Ni-FA salutan menjadi lebih padat dan lebih kemas apabila menjalani proses rawatan haba pada 200 °C dan sifat mekanik telah menurun dengan meningkatkan komposisi natrium sitrat..

## **ABSTRACT**

The Nickel- Fly Ash (Ni-FA) coating are electrodeposited on aluminium alloy 7075 (AA7075) via various sodium citrate composition at different heat treatment process. The aim of this study is to examine the mechanical properties which are hardness and corrosion properties of Ni-FA composite coating AA7075 based on the various composition of sodium citrate. Other than that, to examine the effect annealing process to the properties of Ni-FA composite coating The electrodeposition process using a citrate bath that contain sodium citrate at 40<sup>0</sup>C temperature. Various sodium citrate which are 30 g/L, 50 g/L, 70 g/L and 90 g/L and heat treatment process at 200 °C and 400 °C. The surface morphology of coating was examined by using Scanning Electron Microscopy (SEM), the characterization by X-Ray Diffraction (XRD) and the compositional analysis by X-Ray Fluorescence (XRF). The morphologies and structure of Ni-FA coating become more compact and denser when undergo heat treatment process at 200 °C and mechanical properties was decreased by increasing the sodium citrate composition.

# DEDICATION

Only

My beloved father, Azmi bin Elias

My appreciated mother, Zubaidah binti Mahmood

My adored sisters and brothers Norazwarfahmi Fizri, Normimi Zairul Murni, Noralfarizam  
Qkhairam and Noratihah Azrihah

for giving me moral support, money, cooperation, encouragement and also understandings

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## LIST OF ABBREVIATIONS

AA7075	-	Aluminium Alloy 7075
Zn	-	Zinc
Mg	-	Magnesium
Cu	-	Copper
Cr	-	Chromium
Ni	-	Nickel
Co	-	Cobalt
CVC	-	Chemical Vapor Deposition
PVC	-	Physical Vapor Deposition
HVOF	-	Plasma and High Velocity
MMC	-	Metal Matrix Composite
PMC	-	Polymer Matrix Composite
CMC	-	Ceramic Matrix Composite
NiCl <sub>2</sub>	-	Nickel Chloride
NiSO <sub>4</sub>	-	Nickel Sulphate
H <sub>3</sub> BO <sub>3</sub>	-	Boric Acid
Na <sub>3</sub> C <sub>6</sub> H <sub>5</sub> O <sub>7</sub>	-	Sodium Citrate
NaOH	-	Sodium Hydroxide
Ni <sub>3</sub> H <sub>4</sub>	-	Nitric Acid
FA	-	Fly Ash



Ni-FA	-	Nickel-Fly Ash
SEM	-	Scanning Electron Microscope
XRD	-	X-Ray Diffraction
XRF	-	X-Ray Fluorescence
ASTM	-	America Standard Testing Material
SiO <sub>2</sub>	-	Silicon Oxide
Al <sub>2</sub> O <sub>3</sub>	-	Aluminium Oxide
Fe <sub>2</sub> O <sub>3</sub>	-	Iron Oxide
SiC	-	Silicon Carbide

## LIST OF SYMBOLS

g/L	-	Gram Liter
$^{\circ}\text{C}$	-	Degree Celcius
$\mu\text{m}$	-	Micro Meter
wt. %	-	Weight Percentage
$\text{A}/\text{m}^2$	-	Area Meter

# CHAPTER 1

## INTRODUCTION

### 1.1 Background of Study

Aluminum alloy 7075 (AA7075) was broadly utilized as a part of the aircraft application, ranging in complexity and performance requirements from simple components through to essential load bearing structures in air ship and aviation vehicle because of its appealing far reaching properties such as low density, ductility, toughness and resistance to fatigue (Canakci & Varol, 2014; Dayo et al., 2013). AA7075 have a chemical composition roughly of zinc (Zn), magnesium (Mg), copper (Cu) and chromium (Cr). However, this material have a weakness which is it has low corrosion resistance. Therefore, coating system has been introduced to the AA7075 to overcome this problem and to prevent corrosion.

Various techniques of coating can be applied to AA7075 substrate such as electrodeposition, laser beam deposition, ion implantation, chemical vapor deposition (CVD), physical vapor deposition (PVD), plasma and high-velocity fuel (HVOF) (Mohammadi, 2015; Ahmad & Mohamed, 2014). Many recent studies (Mohammadi, 2015; Rie, 2003; Góral et al., 2010) have shown that electrodeposition has been identified as the superior technique and economic method for preparation of composite coatings in a single step without going any secondary treatment and. This technique has various advantages such as uniform deposition, precise control, low energy requirement and high production rate (Ahmad & Mohamed, 2014). Metal matrix composites (MMC) such as alloy based nickel are extensively used in the electrodeposition due to decorative appearance, their properties for an example wear resistance and corrosion resistance in high temperature as protection

coatings in order to improve the corrosion resistance compare to pure metal or alloy. (Pradeep and Senthilvelan, 2014; Góral et al., 2010).

There are many types of bath that used in the electrodeposition such as Watts bath (Nowak et al., 2016), Nickel Sulfamate bath (Pradeep and Senthilvelan, 2014) and Citrate bath (Kamel et al., 2010). A considerable amount of literature has been published about the common bath that used to perform the electrodeposition which is Watts bath and the electrolytic solution commonly containing nickel chloride ( $\text{NiCl}_2$ ), nickel sulphate ( $\text{NiSO}_4$ ), and boric acid ( $\text{H}_3\text{BO}_3$ ) (Alizadeh et al., 2016; Nowak et al., 2016; Góral et al., 2010). According to Šupicová et al., (2006) investigated boric acid act as a surfactant which adsorbs on the surface and interferes with metal nucleation processes. Recently in Japan, there are regulations about the environmental protection that restricting dumping of waste containing boron. Therefore it becomes important to discover other option to replace or reduce the usage of boric acid for nickel electrodeposition (Kamel et al., 2010).

There are many types of inert particles in the composite plating and fly ash (FA) is a one of that particles. Coal that produce from the combustion at high temperature and pressures in the construction sector produces different types of ash and FA is known as ‘fine’ ash fraction that carried upwards with the flue gases from the electro static precipitators (Ismail et al., 2007). The amount of FA discharge by production lines in thermal power plants and construction has been expanding around the world and the amount of FA that disposal in environment become a serious problem. Fly ash is commonly grey in color, abrasive, mostly alkaline and refractory in nature (Ahmaruzzaman, 2010).

## **1.2 Problem Statement**

The disadvantage of the AA7075 is it easily oxidate and this oxidation shows up as white residue and pitting (cavity) and if suitable precautions against electrolysis are not taken, corrode can easily occur. A coating system needed to be used to overcome this problem. Watts bath is a most common electrolysis and boric acid is basic chemical composition to control a bath pH and to create or forming smooth and ductile deposits (Kamel et al., 2010). However, the waste bath that contain boron is harmful.

A survey of the literature review indicates that little work seems to have been done on the electrodeposition of nickel from Citrate bath. Citrate bath is an electrolytic solution that containing a nickel sulphate , nickel chloride and sodium citrate ( $\text{Na}_3\text{C}_6\text{H}_5\text{O}_7$ ) (Kamel et al., 2010). Previous research has shown electrolytes for nickel electrodeposition which contains sodium citrate instead of boric acid as the buffer (Chaoqun, 2015). Chaoqun (2015) investigated that citrate is easily soluble in the nickel plating and can give higher buffer capacity than the boric acid. This study set out to investigate the effect of sodium citrate on the properties of nickel-fly ash (Ni-FA) composite coating.

FA can be considered as one of the largest raw material resources in the world and to be exceedingly contaminating because of their potentially toxic trace element which condensed from the flue gas (Ahmaruzzaman, 2010). According to Ahmaruzzaman (2010) to overcome this problem, the FA should utilization for new products rather than disposed of as landfill because disposal of FA will soon be too costly. FA as inert particles have been utilized on metallic substrates in the metal matrix composite (MMC) composite coatings (Panagopoulos et al, 2011). Nguyen et al, (2013) studied that FA as an additive to improve mechanical and chemical properties of metallic alloy particularly aluminium and inclusion of FA in metal show the high abrasion, wear resistance and low friction coefficient. In this study, FA particles will be added as inert particle and will be co-deposited in nickel matrix.

Heat treatment is an operation including heating at the particular rate, cooling at some predetermined rate and immersion at a temperature for a period time and. In this study, heat treatment will be utilized to acquire a required microstructure to accomplish certain predetermined properties (physical, mechanical and magnetic) on the properties of the electrodeposited nickel

### **1.3 Objectives**

- i. To study the effect of the various sodium citrate composition on hardness properties of electrodeposited Ni-FA composite coating on AA7075 substrate.
- ii. To investigate the influence of various sodium citrate composition on the corrosion properties of electrodeposited Ni-FA composite coating AA7075.
- iii. To examine the effect of annealing temperature on the properties of electrodeposited Ni-FA composite coating on AA7075

### **1.4 Scope of Study**

In this study, the scope is to examine the effect of citrate bath composition and heat treatment process on the properties of Ni-FA composite coating on AA7075 substrate. The properties including the corrosion resistance, hardness and wear resistance. Nickel selected as anode while AA7075 as cathode. Two types of electrolyte prepare which are Citrate bath and Watts bath. The amount of FA will be fixed in both bath which is 50 g/L. Before electrodeposition process, surface pre-heated using grinding and polishing on the AA7075 to remove dirt. The sample will immersed into a two types of solution which is different composition of sodium citrate (30 g/L, 50 g/L, 70 g/L, and 90 g/L) for Citrate bath to find the optimum composition of sodium citrate. The characterization of the sodium citrate composition and coating is carried out using Scanning Electron Microscope (SEM) and X-Ray Diffraction (XRD). The effect of sodium citrate composition on the coating properties is investigated through hardness test and corrosion test.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Composite Coating**

##### **2.1.1 Introduction**

According to Mallick et al., (1997) composite material is the combination of two or more chemically different materials with a different interface between one another. The properties and characteristics from the combination of constituent material are different and one of those constituent forms a phase is called the matrix. The matrix is important to protect and hold the reinforcement from environment and physical damage. The reinforcement in the form of fibers is added into matrix to improve the matrix properties. The effectiveness fiber reinforcement depends on the type, length, volume fraction and orientation of the fibers in the matrix. The reinforcement surface can be chemically treated or coated in order to increase wetting of the reinforcement by the matrix and enhance the interfacial bonding throughout the matrix between reinforcement and the matrix. Composite materials can be category as a metal matrix composite (MMC), polymer matrix composite (PMC) and ceramic matrix composite (CMC) as shown in Figure 2.1. In the PMC, there are several types which is thermoset, thermoplastic and rubber.

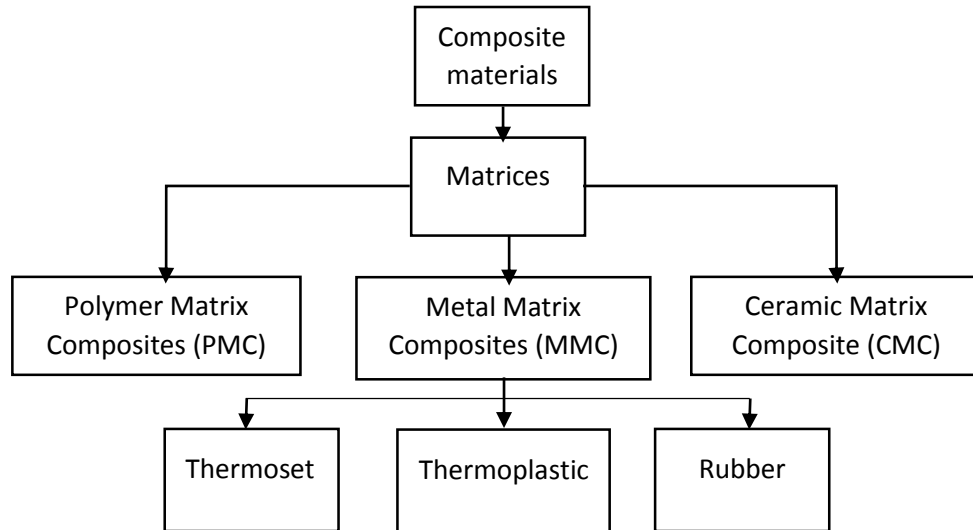


Figure 2.1: Classification of matrices (Mallick, 1997)

### 2.1.2 Types of reinforcements

There are three main types of reinforcement which is continuous fibers, discontinuous (short fibers/whiskers) and particulate as shown in Figure 2.2. Continuous fibers are long strands of fibers that have a small cross sectional area. The fibers will place in the matrix in geometrical pattern and the composite will have anisotropic properties due to single direction of aligned. Anisotropic are very strong in the fiber direction but weak when the fiber perpendicular with the direction. Short fibers usually placed randomly into a matrix and the advantage of this reinforcement will make composite to be more isotropic than continuous fibers. Another type of reinforcement is particle which is contain a ceramic powder or metal that used to reduce friction, increase wear resistance and this reinforcement are extremely cost effective (Mallick et al., 1995).