UNIVERSITI TEKNIKAL MALAYSIA MELAKA

ELECTROCHEMICAL BEHAVIOR OF NICKEL ALUMINIDE BASED ALLOY DEPOSITED USING DEEP EUTECTIC SOLVENT

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

by

MUHAMMAD ZULHILMI BIN NAZERI

B051610109

950520-08-5845

FACULTY OF MANUFACTURING ENGINEERING

2020

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

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Tajuk: **ELECTROCHEMICAL BEHAVIOR OF NICKEL ALUMINIDE BASED ALLOY DEPOSITED USING DEEP EUTECTIC SOLVENT**

Sesi Pengajian: **2019/2020 Semester 2**

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Alamat Tetap:
743, Taman Tun Sambanthan
31100 Sungai Siput (U)
Perak
Tarikh: 7/9/2020

Cop Rasmi: MUHAMMAD ZAINI BIN ZAINAL ABIDIN
Pensyarah Kanan
Fakulti Kejuruteraan Pembuatan
Universiti Teknikal Malaysia Melaka
Hang Tuah Jaya
76100 Durian Tunggal, Melaka

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I hereby, declared this report entitled "Electrochemical Behavior of Nickel Aluminide Based Alloy Deposited Using Deep Eutectic Solvent" is the result of my own research except as cited in references.

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Author's Name : MUHAMMAD ZULHILMI BIN NAZERI

Date : 7 SEPTEMBER 2020

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 (Hons). The member of the supervisory committee is as follow:

.....

(Dr. Muhammad Zaimi Bin Zainal Abidin)

DR. MUHAMMAD ZAIMI BIN ZAINAL ABIDIN
Penyarah Kanan
Fakulti Kejuruteraan Pembuatan
Universiti Teknikal Malaysia Melaka
Hang Tuah Jaya
76100 Durian Tunggal, Melaka

ABSTRAK

Ujikaji penggunaan pelarut eutektik dalam sebagai larutan elektrolit dalam elektro-pemendapan aloi berasaskan Ni_3Al pada substrat Fe dilakukan kerana pembentukan aloi binari dalam pelarut eutektik masih belum dikaji sepenuhnya. Tujuan kajian ini adalah untuk membentuk lapisan aloi Ni_3Al di atas substrat Fe dengan menggunakan teknik elektro-pemendapan dalam pelarut eutektik mendalam sebagi fungsi ketumpatan arus. Pertama, substrat digilap menggunakan kertas pasir 1000 grit. Seterusnya, substrat dibersihkan, masing-masing dibilas menggunakan etanol dan air suling. Kemudian, substrat disalut oleh resin epoksi. Kemudian, 3 sampel di sadur secara elektrod dengan ketumpatan arus 0.010, 0.013 dan 0.015Acm^2 dengan masa penyaduran selama 1800 saat. Campuran larutan terdiri daripada 0.1 M $NiCl_2$ 0.1 M $AlCl_3$ dicampurkan secara sebati dengan larutan ChCI: EG dengan nisbah molar 1: 2. Untuk analisis, Mikroskop Imbasan Elektron (SEM) dan X-ray Diffraction (XRD) digunakan untuk menentukan morfologi permukaan dan komposisi elemen substrat bersalut. Dari analisis OM, sampel diperhatikan di permukaan dan dianggap Ni_3Al pada substrat. Dari keputusan XRD, puncak Ni dapat dikesan bersama-sama substrat Fe tetapi elemen Al adalah kurang, Kemudian, kekerasan dan kekasaran permukaan sampel bersalut masing-masing ditentukan oleh penguji kekerasan Mikro Vickers dan Profilometer. Kekasaran permukaan adalah 0.45 μm hingga 1.82 μm dan kekerasan adalah dari 900 N/mm^2 sehingga 1060 N/mm^2. Terakhir, pengukuran elektrokimia dilakukan dalam 3.5 wt. % NaCl untuk menyiasat tingkah laku kakisan sampel yang dilapisi. Nilai saduran Icorr ialah 0.13 mA manakala Ecorr ialah 0.92 V. Keadaan hakisan untuk lapisan substrat adalah lebih rendah potensi kakisan berbanding dengan substrat Fe yang tidak dilapisi.

ABSTRACT

The experimental study of the utilize of deep eutectic solvent as an electrolytic solution in the electrodeposition of Nickel Aluminide based alloy on Fe substrate is carried out due to the formation of binary alloy in deep eutectic solvent has not fully studied. The purpose of this study is to form a coating of Nickel Aluminide based alloy on Fe substrate by using electrodeposition in deep eutectic solvent as a function of current density. First, the substrate is polished using 1000 grit sandpaper. Next, the substrate degreased, rinsed using ethanol and distilled water respectively. Then, the substrate were mounted by epoxy resin. Later, 3 samples was electrodeposited with current density of 0.010, 0.013 and 0.015A/cm^2 with plating time from 1800 seconds. The plating bath consists of 0.1 M $NiCl_2$ and 0.1 M $AlCl_3$ were mixed homogenously with ChCI: EG solution with molar ratio 1:2. For the analysis, Optical Microscope (OM) and X-ray Diffraction (XRD) were used to determine the surface morphology and element composition of the coated substrate. From OM analysis, the sample were observed on the surface and considered to be Ni_3Al on the substrate. For XRD analysis, the peak of Ni has seen together with Fe element at current density 0.013 A/cm^2. However, the element of Al is less compared to Ni. Then, hardness and surface roughness of the coated samples were determined by Micro Vickers hardness tester and Profilometer respectively. As result, the surface roughness is from 0.45 μm to 1.82 μm and the hardness is from 900 N/mm^2 to 1060 N/mm^2. Lastly, electrochemical measurement were performed in 3.5 wt. % NaCl to investigate the corrosion behaviour of the coated samples. The coating value Icorr is 0.13 mA and Ecorr is 0.92 V. The corrosion behaviour for coating substrate have lower corrosion potential compared to uncoated Fe substrate.

DEDICATION

I would like to dedicate this effort to
my beloved father, Nazeri Bin Hussain
my appreciated mother, Noor Akmar Binti Mohd Shah
my honourable supervisor and lecturer,
for giving me moral support, money, cooperation, encouragement and also understandings
Thank You So Much & Love You All Forever

ACKNOWLEDGEMENT

In the name of ALLAH, the most gracious, the most merciful, with the highest praise to Allah that I manage to complete this report successfully without difficulty. Throughout the venture, I received the help and guidance from many trusted peoples, who deserve my gratitude.

I would like to express my gratitude to Dr. Muhammad Zaimi Bin Zainal Abidin, my supervisor for this project for his mentoring, guidance, encouragement and valuable advice throughout this project. His willingness to spend his time was greatly appreciated.

I would like to give special thanks to my friends and family who gave me encouragement and cooperation mentally while fulfilling this report. Their motivation comment and opinion throughout this report has been much appreciated.

Lastly, I would like to thank everyone who had involve in this project and I would like to apologize if I could not mention personally every person in this report.

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LIST OF ABBREVIATIONS

Cr	-	Chromium
Mo	-	Molybdenum
Zr	-	Zirconium
B	-	Boron
C	-	Carbon
Ti	-	Titanium
W	-	Tungsten
Si	-	Silicon
Ni	-	Nickel
Zn	-	Zinc
Co	-	Cobalt
Ag	-	Silver
Al	-	Aluminium
Fe	-	Iron
AgCl	-	Silver Chloride
KCl	-	Potassium Chloride
HCl	-	Hydrochloric acid
ChCl	-	Choline Chloride
NaCl	-	Sodium Chloride
H2	-	Hydrogen gas
Ni_3Al	-	Nickel Aluminide
Al_2O_3	-	Aluminium Oxide
TiO_2	-	Titanium Oxide
$AlCl_3$	-	Aluminium (III) Chloride
$NiCl_2$	-	Nickel (II) Chloride
SEM	-	Scanning Electron Microscopy
XRD	-	X-ray Diffraction
PVD	-	Physical Vapour Deposition

CVD	-	Chemical Vapour Deposition
HVOF	-	High-velocity Oxy Fuel
DES	-	Deep eutectic solvent
EG	-	Ethylene Glycol
EMIM	-	1-ethyl-3-methylimiazolium chloride
FCC	-	Face centered cubic
R	-	Reducing agent
Me	-	Metal to be deposited
WE	-	Working electrode
CE	-	Counter electrode
RE	-	Reference electrode
Eocp	-	Open-circuit potential
Icorr	-	Corrosion current density
Ecorr	-	Corrosion potential

LIST OF SYMBOLS

%	-	Percentage
Mm	-	Micrometre
A	-	Area
Ampere	-	Current
cm	-	Centermetre
d	-	Density
GPa	-	Giga Pascal
HV	-	Hardness Vicker
K	-	Kelvin
M	-	Mol
Nm	-	Nanometre
℃	-	Degree Celcius
S	-	Second
V	-	Volts
wt.%	-	Weight Percentage
Θ	-	Theta
Q	-	charged passed
I	-	current passed
t	-	the time the current is passed
z	-	change in oxidation state
m	-	mass for oxidized/reduced species
M	-	molar mass for oxidized/reduced species
F	-	faraday constant
n	-	amount of substance oxidized or reduced

CHAPTER 1

INTRODUCTION

A coating is a process that is applied to the surface of certain material, usually called as a substrate. The common reason of this method is to provide protection, improve wear resistance and corrosion resistant as well as useful for decorative purpose. The coating process can be divided into three major step which are surface preparation, paint or curing process and equipment cleaning (Quintino,. 2014). A variety of methods are used in coating application including dip coating, thermal spraying, powder coating, diffusion coating, electrodeposition, galvanizing and conversion coating. In this experimentation, the study focused on one of the method which is electrodeposition. Electrodeposition is a development of a thin film substance on a substrate's conductive surface using a conductive solution containing the material's ionic molecules (Nur et al,. 2011). In addition, deposition on the conductive surface is allowed by submersion of the substratum and counter electrode into the solution and by adding a potential difference to it creating a chemical reaction between anodic and cathodic electrodes (Skreekanth,. 2017). The mathematics equation also applied in electrodeposition technique which is the total quantity of chemical change generated by a current is always proportional total amount of charge passing through the electrolyte. A well design electrodeposition technique will provide several benefits such this technique uses low cost, simple equipment and highly energy efficient.

1.1 Background of Study

Electrodeposition known as a process of depositing the material onto another surface of material or called as electrode by electrolysis technique. Electrodeposition method is utilized to apply a thin layer of material to the surface of the object required to change its exterior properties, such as enhancing corrosion protection, increasing abrasion resistance and improving the surface finish of the object (Liu and Pope, 2000). For plating metals, metal mixtures and also semiconductor, electrodeposition can be used.

The mechanism of electrodeposition is divided into two part of systems which is "electro" and "chemical" parts. The "electro" part of the system includes current source, electrode, anode and cathode, soaked in the "chemical" part of the system which is electrolyte with the circuit by the flow of ions from the electrolyte solution to the electrode. The metal deposition takes place at the cathode when the current flows between the electrodes (El Escoubas, 2003).

Ni_3Al based alloy are mostly used in high temperature properties zone, in an oxidizing and carburizing environment such as coating process for compressor and turbine blades in aircraft engine. Ni_3Al can be manufactured by casting and powder metallurgy. The advantages of NiAl-based intermetallics are a melting point above 1460 °C, which is about 100 to 250 °C higher than the melting point of the conventional Ni-based superalloys. A thermal conductivity about four times that of nickel based alloys. An excellent resistance to oxidation and hot corrosion.

In this study, Ni_3Al is hard to be deposited using electrodeposition in aqueous solvent due to Aluminide oxidation in water. Study by Nor Akmal (2019) shows that Ni_3Al could be deposited in non-aqueous solvent. However, the method is relatively dangerous. Alternatively, Ni_3Al can be deposited in deep eutectic solvent based on previous studies. Ni_3Al based alloy is used to coat on Fe substrate in deep eutectic solvent. Deep eutectic solvents (DESs) is used due to their low cost and high purity. Moreover, DES is a powerful medium for the electrodeposition of metals with controllable microstructures and properties. DES-based electrolytes can also be used to produce alloys coatings (Gomez et al, 2011).

1.2 Problem Statement

Traditional electrodeposition technique uses water as aqueous solution to conduct the movement of ions. Ni_3Al is difficult to deposited in aqueous solution because of Al oxidation in water. Ni_3Al may be deposited in non-aqueous solvent (Nor Akmal,. 2009). However, the method is relatively dangerous. Alternatively, Ni_3Al can be deposited in deep eutectic solvent based on previous studies.

Electrodeposition in aqueous solution for plating or coating processes have certain limitations such as narrow potential window but deep eutectic solvent possesses extensive potential window and better chemical reacted. Besides, Nickel Aluminide deposition in eutectic solvent is not fully utilized and require further study regarding the plating bath composition, plating parameter and coating properties. Moreover, the details of surface roughness, surface morphological properties, composition and hardness of the metal need to be investigate as well.

1.3 Objective

The objective of this study are:

I. To apply the coating of Nickel Aluminide based alloy on Fe substrate by using electrodeposition technique in deep eutectic solvent as a function of current density.

II. To investigate the surface morphological, composition element and hardness of Nickel Aluminide based alloy coating by using Optical Microscope (OM), X-ray Diffraction (XRD), Micro Vickers hardness and surface roughness tester respectively.

III. To identify the corrosion behaviour in 3.5 wt. % NaCl of the coating by using linear voltammetry measurement as a function of current density.

1.4 Scope

In this study, the electrodeposition of nickel aluminide based alloy coating on Fe substrate in deep eutectic solvent will be tested. Through altering the electrical charge, electrodeposition can be conveniently used to manage the thickness of the layer and can be applied to surfaces with complex form. Since Al cannot be electrodeposited from aqueous solvent, eutectic solvent has been takes place as an electrolyte solution. The parameter of nickel aluminide (NiAl) properties need to be considered such as current density, bath temperature and plating time. The parameter is important to achieve a good result from the lab experiment.

During the experiment, surface morphology need to be investigate by using Optical Microscope (OM) and X-ray diffraction (XRD). Furthermore, the hardness test must be conduct by using Micro Vickers hardness testers. The purpose of hardness testing is to evaluate a material characteristics such as strength, ductility and wear resistant. Surface roughness also need to be analyses by using profilometer. Nevertheless, the investigation of corrosion behavior need to be conduct as well by using electrochemical measurements.

1.5 Organization of Report

This report comprises of five portion. First chapter is describing about the introduction of the study area. This part including background of study, problem statement, objective, scope and also the important of study. Any issue are acknowledged through the environmental and current circumstances.

Second chapter is emphasize on the literature review component. In this chapter, all the content covers about the research topic such as type of coating process, electrodeposition technique, and application of Nickel Aluminide based alloy as well as the past investigations from different journals, articles and books.

Third chapter is focus on the methodology component. This chapter will clarifies every flow of the entire experiment including the parameters by referring to the standard procedure. In addition, all the equipment and machine used will be explained.

Forth chapter is express briefly on the result component. This chapter will be include the characteristics and analysis of the sample after completely deposited in deep eutectic solvent. The result that will be discussed are surface morphology, element composition, hardness, surface roughness and corrosion behavior.

Lastly, fifth chapter is review on the final discussion regarding to the results obtained. The conclusion will finalize the result analysis. Additionally, this chapter also review on the suggestions and recommendations that can be used for further studies.

1.6 Summary

To be summarized, this chapter is highlight about the Nickel Aluminide based alloy deposited onto the substrate in deep eutectic solvent by implement electrodeposition method. The objective is clearly explained in order to achieve the purpose of this study. Besides, the scope of this project has been stated to ensure the experiment study can be conduct in an efficient way. The arrangement of this project plan is important to obtain a good experimental result.

CHAPTER 2

LITERATURE REVIEW

2.1 Coating

A coating is a layer of material embedded in a substrate to raise the corrosion and wear resistance of the surface properties. The coating definition could be interpret as a layer of material, coated naturally or synthetically on the surface of a substrate made of another material, in order to obtain desired decorative or technical properties (Burakowski and Wierzchon, 1999). The coating protects the substrate from deterioration due to electrochemical reaction with environment. There are several factors that influence the selection of coating including environment, lifetime, and material of substrate compatibility, size as well as cost. In industries, every manufactured product need to be coating for protective function. For example, an unappealing object might have to aesthetically enhance to be improved in decorative intention (Audigiè *et* al., 2018). Some materials need to be coated to strengthen the life structure and durability of coat. A coating is a method to improve some properties that related with the material itself such as abrasive resistance, lower frictional resistance, wear resistance, surface roughness, corrosion resistance, chemical resistance and also for decorative purposes. Coating consists of physical and chemical vapor deposition, galvanizing, diffusion coating, electroless deposition, thermal spraying, conversion coating, powder coating, and also electrodepositing method. Every different methods have different benefits and constraints.

2.2 Types of coating method

In a various coating applications with a common aim of protecting a component or substance that revealed to the mechanical or chemical damage, a widely variety of coating methods and materials are available. The processes consists of different parameters in the form of material microstructure, suitability, effectiveness and durability. However, coating methods are beneficial in certain applications according to the preferred features among which corrosion and wear protection are the most essential (Mohammadian and Namdari, 2018).

2.2.1 Vapour Deposition

Vapour deposition relates to any process whereby condensation, chemical reaction or transformation. The materials in a vapour state are compressed to form a solid material. These method are used to modify the properties of the substrates in term of mechanical, electrical, thermal, optical, corrosion resistance and also wear resistance. Processes of vapour deposition usually take place in a vacuum chamber. Furthermore, Vapour Deposition can be utilized in many applications such as aerospace, biomedical instruments, automotive, optics, and firearms. It provides the benefits of versatility when using any natural and synthetic material as a surface layer while the coating layer is extremely hard and resistant to corrosion (Mattox and Andrew, 2010). There are two classification of vapour deposition which are physical vapour deposition and chemical vapour deposition.

2.2.2 Physical Vapour Deposition (PVD)

Physical Vapour Deposition (PVD) is a vacuum coating process to enhance wear and corrosion resistance. There are many type of applications for PVD such as machine tools, decorative, optical enhancement, moulds, dies and blades. For example, machining tools are