ELECTRODEPOSITION OF COPPER CHROMIUM (Cu-Cr) ALLOY ON Fe SUBSTRATE IN DEEP EUTECTIC SOLVENT

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

by

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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 requirements for the degree of Bachelor of Manufacturing Engineering (Hons.). The members of the supervisory committee are as follow:

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(Dr. Muhammad Zaimi bin Zainal Abidin)

ABSTRAK

Elektrodeposisi dari aloi Cu-Cr pada substrak Fe telah disiasat dengan menggunakan pelarut eutektik (DES). Pelarut eutektik yang mendalam adalah komnibasi dua mikel iaitu choline chloride dan ethylene gliserol dengan nisbah 1: 2M masing-masing. Walau bagaimanapun, pemendapan Cu mudah terbentuk dalam elektrolit tetapi sukar untuk Cr kerana Cr ions mudah membentuk oksida apabila ia dihubungkan dengan air. Tujuan kerja ini adalah untuk membangunkan pelarut eutektik dalam yang stabil (DES) yang mengandungi ion Cu (II) dan Cr (III). Selain itu, tujuan utama kajian ini adalah untuk mengkaji lapisan aloi Cu-Cr pada substrat Fe. 99.5% besi tulen digunakan sebagai substrat dan substrat digilap sehingga permukaan mirip cermin untuk mengelakkan salutan tidak sekata. Substrat dipasang oleh lapisan resin epoksi untuk mengelakkan salutan hadir di bahagian belakang substrat. Oleh itu, struktur kristalografi, morfologi, komposisi kimia dan sifat-sifat mekanik filem-filem yang disimpan Cu-Cr dianalisis dengan cara X-ray Diffraction (XRD), Mikroskop elektron imbasan (SEM), profilometer penguji kekerasan mikro Vickers untuk kekasaran permukaan. Kesan parameter penyaduran seperti masa penyaduran dan ketumpatan arus pada struktur, struktur dan morfologi permukaan disiasat. Lebih-lebih lagi, pengukuran elektrokimia dilakukan dalam 3.5wt% larutan natrium klorida (NaCl) untuk menguji kelakuan kakisan substrate Fe bersalut. Hasilnya menunjukkan bahawa parameter terbaik kajian ini dengan kekerasan tertinggi dan permukaan yang baik ialah 125mA/dm² pada 15minutes. Kekerasan dan kekasaran permukaan substrat ialah 174.3 HV dan 0.65μm.

ABSTRACT

Electrodeposition from Cu-Cr alloy on Fe substrate has been investigated by using deep eutectic solution (DES). Deep eutectic solvent is comnibation of two micture which is choline chloride and ethylene glycerol with ratio 1:2M respectively. Nevertheless, Cu deposition is easily formed in aqueous bath but it is hard for Cr due to Cr ions easier form oxide when it contact with water. The aim of this work is to develop a stable deep eutectic solvent (DES) containing Cu (II) and Cr (III) ions. Also, the main purpose of this research is to study the coating of Cu-Cr alloy on Fe substrate. 99.5% of pure iron was used as the substrate and the substrate is polished until mirror-like surface to prevent uneven coating. The substrate is mounted by a layer of epoxy resin to prevent the coating present at the backside of substrate. Thus, the crystallographic structures, morphology, chemical composition and mechanical properties of Cu–Cr deposited films are analyzed by means of X-ray Diffraction (XRD), Scanning electron microscopy (SEM), Micro Vickers hardness tester profilometer for surface roughness. The effects of plating parameters such as plating time and current density on composition, structure and surface morphology was investigated. Moreover, electrochemical measurement was done in 3.5wt% of sodium chloride (NaCl) solution to test the corrosion behaviour of coated Fe substrate. The result shows that the best parameter of this study with highest hardness and good surface is 125mA/dm^2 at 15minutes. The hardness and surface roughness of the substrate is 174.3 HV and 0.65μm respectively.

DEDICATION

I would like dedicate this work to
my beloved family
my honourable supervisor and lecturers
my dearest friends
for giving me moral support, cooperation, encouragement and also understandings
Thank you so much

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I would like to express my deepest appreciation to all who supported me in this final year project. A special gratitude I give to my industry supervisor, Dr. Muhammad Zaimi bin Zainal Abidin, whose contribution in advising, giving motivation, guiding me to complete and provide useful critiques throughout this project. His willingness to give his time so generously has been very much appreciated.

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LIST OF ABBREVIATIONS

CE	-	Counter Electrode
ChCl	-	Choline Chloride
Co-Cr	-	Cobalt Chromium
Cr	-	Chromium
$CrCl_3.6H_2O$	-	Chromium Chloride
Cu	-	Copper
Cu^{2+}	-	Copper (II) ion
Cr^{3+}	-	Chromium (III) ion
$CuCl_2.6H_2O$	-	Copper Chloride
CVD	-	Chemical Vapour Deposition
DCD	-	Direct Current Deposition
DES	-	Deep Eutectic Solvent
E_{corr}	-	Corrosion Potential
EDS	-	Energy Dispersive X-ray Spectroscopy
EG	-	Ethylene Glycol
Fe	-	Iron
Fe-Cr	-	Iron Chromium
GO	-	Graphene Oxide
HBD	-	Hydrogen Bond Donor
I_{corr}	-	Corrosion Current
LVM	-	Linear Voltammetry Measurement
NaCl	-	Sodium Chloride
Ni-Cr	-	Nickel Chromium
PED	-	Pulsed Electrodeposition
PVD	-	Physical Vapour Deposition
Ra	-	Surface Roughness
RE	-	Reference Electrode
SEM	-	Scanning Electron Microscopy
Ti6Al4V	-	Alpha-beta Titanium Oxide

WE	-	Working Electrode
XPS	-	X-ray Photoelectron Spectroscopy
XRD	-	X-Ray Diffraction

LIST OF SYMBOLS

%	-	Percentage
μm	-	Micrometre
A	-	Current
A	-	Area
mm	-	Millimetre
K	-	Kelvin
cm	-	Centimetre
℃	-	Degree Celcius
Mol	-	Mol
wt.%	-	Weight Percentage
s	-	Second
V	-	Volts
d	-	Density
mpy	-	Milli-inches per year

CHAPTER 1

INTRODUCTION

Chromium coating have some advantageous properties which are high wear and corrosion resistance. Coating is a method that used to coat a thin layer on the surface of substrate. The purpose of coating is to improve wear resistance, improve appearance and improve the corrosion resistant of an object. There are few types of coating method such as conversion coting, chemical vapour deposition (CVD), physical vapour deposition (PVD), thermal spraying, electroless plating and electrodeposition. For this study, electrodeposition method is conduct and deep eutectic solvent used as electrolyte. There are certain parameters that influence the nature of deposits produced by electrodepostion for instance current density, bath temperature, electrolyte pH, plating time and concentration of bath.

1.1 Background of Study

Chromium copper alloys that contain 1% of chromium and 99% of copper. Chromium copper alloys are commonly used in industry because of their toughness, durability, good conductivity of heat and electric and resistant to corrosion. Due to the unique behaviour of chromium alloy in which emergence of chromium from solution is occur at high temperature, the chromium copper alloys are classified as age hardenable which implies the mechanical and chemical properties will undergo alternation under circumstances. It can be seen that the corrosion resistance of chromium copper alloys shows superiority compared to pure copper due to the fact that chromium provides a oxide film which retard the erosion of material. Chromium copper is malleable and ductile.

Electrodeposition is a process that deposited a thin layer on the metal at cathode by using electric current. The solution that involves to passing the electric current is called electrolyte. In electrodeposition process, the metal to be deposited is at cathode connected to negative terminal and metal that releases ion is at anode which is connected to positive terminal. Both of the electrodes are placed in an electrolyte that containing salt. A basic mechanism of electrodepostion will be discussed by using copper plating on steel as example. Steel act as cathode that connected to negative terminal whereas copper electrode connected to positive terminal called anode. The electrolyte chosen is copper sulphate solution. Next, the electrodes that connected to power supply will immerse into the electrolyte. Copper sulphate will form ions when the power is switched on and the positively charged Cu^{2+} are attracted to steel electrode and form a thin layer on the surface.

In this study, Fe is being used as substrate and Cu-Cr alloy is used to deposit on Fe substrate by using electrodeposition in DES that contains Choline Chloride and ethylene glycol. Cu (II) and Cr (III) salts are added into the deep eutectic solvents. The surface morphology is studied using the SEM and XRD for the elemental composition analysis. The hardness of coated surface is also measured using micro Vickers Hardness tester. Besides, the corrosion behaviour is tested with 3.5 wt% of NaCl solution and surface roughness is determined by using profilometer in this study.

1.2 Problem Statement

Iron is a shiny, malleable, ductile and bright white metal. It is a widely used as construction, household application and machine manufacturing. However, the lifespan and the properties of iron will affected by corrosion. This can causes enormous economy losses. The surface of iron usually discoloured by corrosion since it contacted with the air and water. Cu-Cr alloys are synthesized by using mechanical alloying because it is difficult to synthesise using conventional method. The properties of Cu-Cr are high strength, heat treatable, high corrosion and wear resistance. Applications of Cu-Cr include ball connector, electrode holder, IC lead frame, switch gears and power contactors. Conventional electrodeposition method uses water as a solvent for cation and anion movement. Cu deposition is easily formed in aqueous bath but it is hard for Cr deposition because of Cr

ions form oxide on the surface of the substrate. Electrodeposition in deep eutectic solvent provides the medium without the presence of hydroxide that will oxide the Cr surface. Nevertheless, the study of electrodeposition of Cu-Cr alloy on Fe substrate in the deep eutectic solvent with choline chloride and ethylene glycol l mixture of ration 1:2 respectively is yet to be developed requires further investigation regarding plating bath composition, plating parameter and its coating various properties. Therefore, the surface morphology, composition, hardness are studied by using SEM, XRD and Vickers hardness tester. The corrosion behaviour is tested with 3.5 wt% of NaCl solution and surface roughness is determined by using profilometer in this study.

1.3 Objective

The objective of this study:

i. To study the surface morphology and composition of Cu-Cr alloy coating on Fe substrate by using electrodeposition process in DESs.
ii. To investigate mechanical properties of the Cu-Cr alloy coating.
iii. To investigate the effect of corrosion properties of Cu-Cr alloy coated on Fe substrate in ChCl/EG based DES bath.

1.4 Scope

The aim of this study is the electrodeposition of Cu-Cr alloy on Fe substrate in DESs. DESs are eutectic mixture of choline chloride (ChCl) as hydrogen bond acceptor and ethylene glycol as hydrogen bond donor. Current density, plating time and bath temperature are studied during electrodeposition of Cu-Cr alloy on Fe substrate. The variant current densities used for this experiment are 25, 50, 100, 125 and 250 mA/dm^2 while the concentration of $CuCl_2$. $6H_2O$ and Chromium Chloride ($CrCl_3.6H_2O$) used is 0.1 mole. The plating time for these studies is 15, 30 and 45 minutes separately. The bath temperature used is 50°. Next, the surface morphology analysis is studied using Scanning Electron Microscopy (SEM) while the X-ray diffraction (XRD) is for elemental

composition analysis. Linear Scanning Voltammetry (LSV) is used as electrochemical measurement method to investigate the corrosion behaviour of coated Fe substrate in 3.5wt% of NaCl solution. The hardness of Cu-Cr alloy coating on Fe substrate is determined by using micro Vickers Hardness tester while for the surface roughness is analysed by profilometer.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction Of Coating

Coating is well known and widely accepted surface finishing method. It is a process to form a thin layer on the surface of substrate. The thin layer on the substrate is to protect the surface of substrate and have a good appearance. It can be the good improvement for surface properties. The thin layer coated on the surface of substrate is to prevent chemical corrosion or atmospheric corrosion such as oxygen and water. The risk of corrosion is low finished with polishes, paints and coatings for decorative since many objects intended for indoor and outdoor use (Kumar *et al*., 2015). Coating is a method used to improve the corrosion resistance and to extend the material used to the maximum capacity of the material.

2.2 Types Of Coating

Most coatings on external surfaces are used to prevent natural corrosion and contamination of the metal. It can also be necessary to protect from accidental spills and splashes. Sometimes, coatings are applied in vessels to resist corrosion resistance. Coating processes can be divided into five categories which are chemical vapour deposition (CVD), physical vapour deposition (PVD), spraying, conversion coating, electroless plating and electroplating.

2.2.1 Conversion coating

Conversion coatings allow metals to be protected using metal and coating solution. Conversion coatings are coatings for metals where surface of part is subjected to an electrochemical process by the coating material which that transforms it into a decorative or protective substance. It enhances surface durability and also protects against corrosion.

2.2.2 Thermal spraying

Thermal spraying is an industrial process of coating that melted material is sprayed on the surface. The heating is usually carried out electrically in form of an arc or plasma. Typically, sprayed thermal coatings are typically used on metal substrates and on certain plastic substrates. This coating process can be used at different thicknesses, usually 100-750 microns and can be more than that. Thermal sprayed coatings significantly enhance and improve the component performance. It has an enormous scope to increase the lifespan of new components or to repair worn or damaged components using techniques tested. Thermal spray consists of four main processes such as electric wire arc, flame spray, plasma spray and high velocity oxy-fuel.

2.2.3 Vapour deposition

Vapour deposition refers to the process in which materials are condensed into a solid material by condensation, chemical reaction, or conversion. All of these processes are used to form a coating layer that change the mechanical, electrical, thermal, optical, resistance to corrosion and wear properties of the substrates. They are also used to create standalone bodies, films, and fibres and to infiltrate fabric into composite materials. Vapour deposition processes usually occur in a vacuum chamber. There are two categories of vapour deposition processes which are Chemical Vapour Deposition (CVD) and Physical Vapour Deposition (PVD).