UNIVERSITI TEKNIKAL MALAYSIA MELAKA

CHARACTERISTICS OF HYDROXYAPATITE SOL-GEL COATING OF STAINLESS STEEL IN HANK'S SOLUTION AND SODIUM CHLORIDE MEDIUM

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

by

SITI WANNORHASSLIZA BINTI WAN HASSAN
B050910032
900619036028

FACULTY OF MANUFACTURING ENGINEERING
2013

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

TAJUK: **Characteristics of Hydroxyapatite Sol-Gel Coating of Stainless Steel in Hank's Solution and Sodium Chloride Medium.**

SESI PENGAJIAN: **2012/13 Semester 2**

Saya **SITI WANNORHASSLIZA BINTI WAN HASSAN**

mengaku membenarkan Laporan PSM ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

1. Laporan PSM adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
3. Perpustakaan dibenarkan membuat salinan laporan PSM ini sebagai bahan pertukaran antara institusi pengajian tinggi.
4. **Sila tandakan (✓)

☐	SULIT	(Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysia sebagaimana yang termaktub dalam AKTA RAHSIA RASMI 1972)
☐	TERHAD	(Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan)
√	TIDAK TERHAD	

Disahkan oleh:

Alamat Tetap:
2215, Lorong Permai 2,
Taman Desa Permai, Bukit Kuang,
24000 Kemaman, Terengganu.

Cop Rasmi:

Tarikh: ____________________

Tarikh: ____________________

** Jika Laporan PSM ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh laporan PSM ini perlu dikelaskan sebagai SULIT atau TERHAD.

APPROVAL

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

…………………………

(Dr Zulkifli bin Mohd Rosli)

DECLARATION

I hereby, declared this report entitled "Characteristics of Hydroxyapatite Sol-Gel Coating of Stainless Steel in Hank's Solution and Sodium Chloride Medium" is the result of my own research except as cited in references.

Signature : ……………………………..

Author's Name : SITI WANNORHASSLIZA BINTI WAN HASSAN

Date : ……………………………..

ABSTRAK

Tujuan kajian ini adalah untuk membuat perbandingan ciri-ciri keluli yang telah dilapisi saduran hydroxyapatite melalui proses saduran celupan sol-gel di dalam dua larutan yg berbeza iaitu larutan hank dan larutan natrium klorida dengan isipadu dan mol yang sama. Selain itu, kadar kakisan sampel keluli yang belum disadur dan telah disadur yang direndam di dalam larutan hank dan larutan natrium klorida dinilai untuk memperakui sampel yang mempunyai ketahanan kakisan yang lebih baik kerana ini adalah tujuan utama proses saduran dilakukan ke atas substrat logam seperti keluli. Sampel plat keluli yang telah diukur disadur dengan mengawal beberapa parameter ketika proses saduran dijalankan seperti kelajuan pengeluaran, bilangan lapisan saduran dan masa rendaman. Pembentukan lapisan saduran, permukaan morfologi dan keratan lintang lapisan saduran substrat bersama-sama dengan kadar kakisan substrat yang tidak disadur dan telah disadur dikaji dengan menggunakan kaedah polarisasi potentiodinamik, Mikroskop Imbasan Electron (SEM) dan Tenaga Dispersif Sinar-X (EDX). Keseluruhan projek ini, perbandingan dilakukan antara keluli yang tidak bersalut dan bersalut dan juga perandingan dilakukan antara ciri-ciri keluli yang telah disalut yang direndam di dalam larutan hank dan larutan natrium klorida. Keputusan menunjukkan substrat yang telah disadur dengan hydroxyapatite mempuyai ketahanan kakisan lebih tinggi di dalam larutan hank berbanding dengan di dalam larutan natrium klorida.

ABSTRACT

The purpose of this study is to compare the characteristics of hydroxyapatite sol-gel coating of stainless steel in two different solutions named hank's solution and sodium chloride solution media with the same mol and volume used. Besides that, the corrosion rate of uncoated and coated stainless steel samples immersed in hank's solution and sodium chloride solution are evaluated in order to declare the better one in revealing corrosion resistance properties as this is the main point of coating process is done on the metallic substrate like stainless steel. The measured stainless steel plates are coated by controlling few parameters during the process such as withdrawal speed, number of coating layer and immersion time. The formation of coating layer, the surface morphology and cross-sectional study on the substrate along with the corrosion rate of both uncoated and coated substrate are investigated by Potentiodynamic Polarization method, Scanning Electron Microscope (SEM), and Energy Dispersive X-Ray (EDX). Throughout this project, the comparison is done between uncoated and coated stainless steel as well as the characteristics of coated stainless steel in two different solutions which are hank's solution and sodium chloride solution. The results show hydroxyapatite sol gel coating samples possess better corrosion polarization resistance in hank's solution compared to in sodium chloride due to wider passive region.

DEDICATION

To my beloved father, Wan Hassan bin Wan Ab Rahman, my mother, Norma binti Jusoh, siblings and friends, your love is my driving force.

To my supervisor, Dr Zulkifli bin Mohd Rosli, your guidance is enlightenment to me.

Thank you everyone for every guidance, support, help, cooperation, directly or vice versa.

ACKNOWLEDGEMENT

In the name of Allah, the most gracious, the most merciful,

Alhamdulillah, praise to Allah S.W.T. for giving me the chance to complete my Final Year Project from the very beginning until the very end. It was a winding road in completing this project, but due to the help gained by God and people close to me, this burden is lightened. First of all, a million thanks is dedicated to my supervisor, Dr Zulkifli bin Rosli for his guidance, concerns and patients. Through the completion of this project, he has been my major supporter and reference. A countless thanks is also dedicated to both of my parents who has been very supporting and understanding in every single moment since I was born. Without their blessing, I won't be able to make it up to this level of achievement. Special thanks is also delivered to the technicians and staff of Faculty of Manufacturing Engineering, Universiti Teknikal Malaysia Melaka (UTeM), for helping willingly whenever needed at anytime. Last but not least, thank you also to the all my friends for being supportive and for the help given. Thank you to all of you.

TABLE OF CONTENT

LIST OF TABLES

LIST OF FIGURES

LIST OF ABBREVIATIONS, SYMBOLS AND NOMENCLATURE

HA	-	Hydroxyapatite
ASTM	-	American Stansard Test Method
Mg	-	Magnesium
Al	-	Aluminium
Zn	-	Zinc
Zr	-	Zirconium
SiC	-	Silicon Carbide
PECVD	-	Plasma enhanced Chemical Vapour Deposition
$Ca_{10}(PO_4)_6(OH)_2$.	-	Calcium Potassium Hydroxide
MPa	-	Mega Pascal
GPa	-	Giga Pascal
Al_2O_3	-	Aluminium Oxide (Alumina)
NaCl	-	Sodium Chloride
KCl	-	Potassium Chloride
$CaCl_2$	-	Calcium Chloride
I_{corr}	-	Corrosion Rate

Mg-Li	-	Magnesium-Lithium
SEM	-	Scanning Electron Microscope
XRD	-	X-Ray Diffractometry
EDX	-	Energy Dispersive
^{o}C	-	Degree Celsius
$Ca\ (NO_3)_2$	-	Calcium Nitrate
$Ca\ (NO_3)_2.4H_2O$	-	Calcium Nitrate Tetrahydrate
$P(OC_2H_5)_3$	-	Triethyl Phosphate
NH_4OH	-	Ammonium Hydroxide
ρ	-	Density
wt%	-	Weight Percentage

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Kainer and Kaiser (2007) has reported that the current challenge involves the increasing use of light alloys in high technology constructional materials, with the aim of both reducing mass and saving energy. The related typical application areas include vehicle construction, aeronautics and the space sector, along with the mechanical engineering, and electrical industries. Moreover, the increasing concerns on environmental protection and sustainable economic development draw a great deal or attention in reducing greenhouse gas emissions.

Iron and the most common iron alloy, steels, are from a corrosion viewpoint relatively poor materials since they rust in air, corrode in acids and scale in furnace atmospheres. In spite of this, there is a group of iron based alloys. The iron-chromium-nickel alloys known as stainless steels, which do not rust in sea water, are resistant to concentrated acids and which do not scale at temperatures up to 1100ºC. Sedricks (1996) has stated that the usage of stainless steel is small compared with that of carbon steels but exhibits a steady growth in contrast to the constructional steels. Otherwise, stainless steel still have been used in wide applications such as laboratory benches and equipment, coastal architectural paneling, railings and trim, boat fittings, heat exchangers as well as chemical containers including for transport.

However, by referring to Talha et al. (2012), stainless steel is still poor in corrosion resistance properties when it comes to its application in biomedical especially implant because corrosion of metal implants is critical due to its effect to biocompatibility. Hence, the surface treatment of stainless steel is mainly comprised of the following technologies. One of the most common coating processes done on metal such as titanium alloy is known as sol-gel coating. In recent years, upon the wide application of sol-gel conversion coatings, a few reports of sol-gel coatings on stainless steel can be found. Tan et al. (2005) reported the effect of sealing of double-layer silane-based sol-gel coating on anodized film. Apart from that, Supplit et al. (2007) found that after acid pickling and subsequent sol-gel coating, the corrosion rate of AZ31 magnesium alloy for example, is reduced to 1/60 as much as that before treatment. This shows that sol-gel coating has its own competitive value in coating history apart from other process types such as sputtering, ion plating, thermal spraying as well as plasma spraying. After all, sol-gel coating is an electroless metal coating which is more accurately described as conversion coating, because it produces a protective layer or film on the metal surface by means of a chemical reaction.

1.2 Problem Statement

The main disadvantage of stainless steels in biomedical application is that they are prone to corrosion in which they are one of the most electrochemically active metals. Therefore, it can be concluded that stainless steel is degrading very fast time by time and thus, reduce its lifespan or services life. Due to this limitation of stainless steel, a broad range of coating systems is being developed to overcome this weakness for biomedical especially implant application. Compared to uncoated metal surface, the coated one has relatively lower rate of corrosion and hence, gives its surface to be shiner and smoother.

Hydroxyapatite sol-gel coating is a part of conversion coatings in which its applications on stainless steel have been quite widespread but still has an issue in corrosion resistance properties. Hydroxyapatite or HA has attracted much attentions as a material for artificial bones (Hench, 1998), scaffolds for tissue engineering (Ebaretonbofa and Evans, 2002; Li et al., 2002) and chromatographic packing (Kawasaki, 1999) because of its high bioactivity and particular adsorb ability for various ions and organic molecules. Although HA has brought many uses in biomedical implant technologies, its uses in metal coating is still not in an advanced state for stainless steel coating. Therefore, the coating process need to be improved by introducing other additional methods into the processing steps so that, the corrosion resistance of stainless steel can be increased.

To promote the best coating for stainless steel, HA has been chosen because it has good potential in exploiting the biocompatible and bone bonding properties of the ceramic. However, the improvement for this coating should been done to enhance the functionability of the coating by introducing phytic acid as pre-coating material prior to HA coating process. Apart from that, to promote environmental-friendly, sol-gel coating method has been chosen due to its low consumption of temperature during the process as low temperature will reduce loss of volatile components.

1.3 Objectives

1. To compare the characteristics of hydroxyapatite sol-gel coating of stainless steel in hank's solution and sodium chloride medium.
2. To evaluate the corrosion rate of stainless steel in hank's solution and sodium chloride medium.

1.4 Scope

The scope of this project is to examine the characteristics of hydroxyapatite (HA) sol-gel coating of stainless steel in hank’s solution and sodium chloride media. This project will start with the corrosion testing of uncoated stainless steel in both hank’s solution and sodium chloride media followed by material characterization analyses of uncoated stainless steel. This step is taken in order to compare its performance with the coated one. The performance of the HA sol-gel coating stainless steel done with pre-determined precursor, solvent, and catalyst along with controlled parameters chosen will be then tested with the same testing and analyses of the uncoated one. When all data is obtained, the result of the uncoated and coated stainless steel will be compared to see the different performances of both substrate types in term of corrosion behavior. This project will include only one type of corrosion which is general corrosion. HA sol-gel coating stainless steel sample preparation and mechanical testing will not be covered in this project.

1.5 Project Organization

This project is organized into five chapters:

1. Chapter 1 is the introduction of the report. In this chapter, it includes the background, problem statement, objectives and scope of the project.
2. Chapter 2 describes the findings of the project as well as theoretical or methodological review before the experimental works.
3. Chapter 3 explains the methodology used in the experiment including equipments and procedures required for the study.
4. Chapter 4 shows the testing and morphology results together with discussions.
5. Chapter 5 will conclude the whole project and recommend for further study to be done for this related title.

CHAPTER 2

LITERATURE REVIEW

2.1 Introduction

In this chapter, the basic and fundamental information is discussed to ease the project. This chapter will discuss mainly about stainless steel as the substrate and its properties including hydroxyapatite or HA as coating material. Apart from that, this chapter will cover the process route until the application of sol-gel coating. Finally, testing and analysis techniques are also included in this chapter for the purpose of discussing on related techniques available in order to analyze the corrosion rate and surface morphology of the specimens.

2.2 Stainless Steel

Substrate is a primary or underlying material on which other materials such as ink, coating, paint or treatment are applied, or from which other materials are made. In a simple word, substrate is nothing but a material on which a coating is applied. The