SCHOOL OF MATERIALS AND MINERAL RESOURCES ENGINEERING UNIVERSITI SAINS MALAYSIA

BIOACTIVE SURFACE MODIFICATION OF TI-NB ALLOY BY ALKALINE TREATMENT IN POTASSIUM HYDROXIDE SOLUTION

By

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DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled “Bioactive Surface Modification of Ti-Nb Alloy by Alkaline Treatment in Potassium Hydroxide Solution”. I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or university.

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LIST OF SYMBOLS

C	Celsius
G	Gram
ml	Millilitre
M	Molarity
Θ	Theta

LIST OF ABBREVIATION

BCC	Body centred cubic
C	Carbon
Co	Cobalt
Cr	Chromium
Cu	Copper
EDX	Energy Dispersive Electron Probe X-ray Analyzer
Fe	Iron
GPa	Gigapascal
H	Hydrogen
HCP	Hexagonal close pack
$HTiO_3$¯nH_2O	Titanate Hydrate
K	Potassium
KOH	Potassium Hydroxide
M	Molar
Mn	Manganese
MPa	Megapascal
N	Nitrogen

Na	Sodium
NaOH	Sodium Hydroxide
Nb	Niobium
Ni	Nickel
O	Oxygen
OH	Hydroxyl Group
SEM	Scanning electron microscope
Si	Silicon
Sn	Tin
Ti	Titanium
TiO_2	Titanium Dioxide
XRD	X-ray diffraction
Zr	Zirconium
β	Beta
α	Alpha

PENGUBAHSUAIAN BIOAKTIF PERMUKAAN ALOI TI-NB OLEH RAWATAN ALKALI DALAM KALIUM HIDROXIDE

ABSTRAK

Rawatan beralkali terhadap struktur β Ti aloi implant yang serasi dan tidak bertoksik didalam kalium hidroxide untuk mengubahsuai bioaktiviti aloi telah dikaji. Ti-40wt%Nb telah dihasilkan menggunakan kaedah metalurgi serbuk dengan mengisar campuran Ti-Nb, mengenakan tekanan pada 550MPa dan disinter pada suhu 1200°C selama 2 jam. Rawatan beralkali terhadap aloi ini telah dikaji dalam cecair akueus kalium hidroxide (KOH) pada suhu 60°C selama 24 jam pada kepekatan yang berbeza (iaitu 0.5M dan 5.0M). Aloi beralkali menjalani proses penyepuhlindapan pada suhu 600°C selama 2 jam untuk mengkaji kesan daripada rawatan haba terhadap aloi beralkali. Kesan tindak balas aloi yang dirawat menggunakan alkali dibandingkan dengan Ti dan Nb tulen. X-ray analisis pembelauan menunjukkan kehadiran lapisan amorf alkali titanate hydrogel seperti titanium terhidrat, kalium hydrogen titanium oksida dan niobium oksida untuk rawatan 0.5M. Untuk 5.0M, fasa baru di kesan iaitu titanium oksida dan dikalium dititanium oksida. Analisis FTIR menunjukkan dua band penting OH pada 3223 cm^{-1} dan 1633 cm^{-1} di permukaan Ti-40Nb selepas rawatan alkali dan selepas penyepuhlindapan. Pemerhatian dibawah pengibasan elektron mikroskop dan analisis unsur menunjukkan pemendapan lapisan titanate dengan kehadiran komposisi tinggi kalium dan oksigen. Kajian bioaktiviti di dalam cecair Hanks selama 24 jam mendapati unsur Ca dan P wujud di permukaan aloi yang dirawat pada kepekatan yang berbeza selepas rawatan alkali dan penyepuhlindapan. Penemuan ini mencadangkan bahawa aloi baru β Ti dengan 40wt% Nb sesuai untuk rawatan alkali di dalam cecair KOH untuk membentuk lapisan kalium titanate yang penting untuk pemendapan mineral apatite.

BIOACTIVE SURFACE MODIFICATION OF Ti-Nb ALLOY BY ALKALINE TREATMENT IN POTASSIUM HYDROXIDE SOLUTION

ABSTRACT

Alkaline treatment of biocompatible and non-toxic new β structure Ti alloy implant in potassium hydroxide for modifying bioactivity of the alloy was investigated. Ti-40wt%Nb was fabricated by powder metallurgy method by milling of Ti-Nb mixture, pressing at 550MPa and -sintering at 1200°C for 2 hours. Alkaline treatment of the alloy was studied in the potassium hydroxide (KOH) aqueous solution at 60°C for 24 hours at different concentration of (i.e., 0.5 and 5M). The effect of post heat was investigating by annealed alkaline treated alloy at 600°C for 2 hours. The responsive behavior of the alloy alkaline treated alloy was compared to pure Ti and Nb. X-ray diffraction analysis showed the presence of amorphous alkaline titanate hydrogel layer such as titanium hydrate, potassium hydrogen titanium oxide, and niobium oxide for 0.5M treatment. For 5M, new phases are noticed (potassium titanium oxide and dipotassium dititanium oxide). The FTIR analysis show two significant band of OH at 3223 cm^{-1} and 1633 cm^{-1} on the Ti-40wt%Nb surface after alkaline and post heat treatment. Morphology observation under scanning electron microscope and elemental analysis indicate the deposition of titanate layer with the presence of high composition of potassium and oxygen. Bioactivity study in Hanks solution for 1 day discovered the trace of Ca and P elements on alloy surfaces treated in different concentration after alkaline as well as annealing treatment. These findings suggest that the new β-Ti alloy with 40wt% Nb were feasible for alkaline treatment in KOH solution forming potassium based titanate layer that were prerequisite for bone mineral apatite deposition.

CHAPTER 1

INTRODUCTION

1.1 Introduction

Titanium alloy is commonly used as implant material application because of their biocompatibility and the existence of natural oxide layer that can improve its corrosion resistance. The commercially used Ti alloy such as Ti-6Al-4V (grade 5) alloy with Young modulus 110 GPa are widely used in hard tissue replacement, dental implants and joint (Viteri and Fuentes, 2012). Lai *et al.*, (2015) reported on cortical bone Young modulus is in the range 17 to 20 GPa. Whereas for pure Ti metal is 120 GPa. In order to be used in the implant, the artificial bone must have Young modulus almost in the similar range as cortical bone. However, the Ti-6Al-4V alloy have high young modulus that result in the stress shielding effect thus not suitable to be used for bone replacement. One way to solve this problem is by introducing β-type structure to the Ti alloy. β-type structure Ti alloy with β-stabilizer like vanadium and niobium has decreased the great differences of Young modulus in bone and replacement implant. For example, Ahmad et al, (2018) reported that Ti-40wt% Nb alloy showed Young Modulus of 26 GPa that is in range with bone's as well as high compressive strength (2123 MPa). Elastic modulus in the range of the bone has been proposed can reduce the stress shielding effect.

However, the properties for a new type of alloy such as biocompatibility may differ with other type of Ti alloy. In order to make sure the new alloy is compatible to be an implant material that can support the healing of the broken bone, surface modification should be done to make sure it able to form a bonding with the bone part that can assist healing. Therefor, biomaterial fields mainly focus on developing new alloy that have specialized mechanical properties which similar with human bone. While biocompatibility of the biomaterial is closely related to their corrosion behavior in biological environment.

Biocompatibility of the new biomaterial can be enhanced by surface modification techniques enhancing their surface bioactivity. The surface modification technique used to improve the properties of titanium-based surfaces such as physical and chemical vapor deposition, thermal and chemical oxidation, thermal spraying, ion implantation, bio ceramic or biopolymer coating deposition and alkaline treatment. Among this surface modification technique alkaline treatment has received attention including surface modification of Ti alloys (Da Silva *et al.*, 2019).

Alkaline treatment is the chemical treatment in which the alloy of interest are immersed in a known concentration of aqueous sodium hydroxide for given temperature and period time (Ouarhim *et al.*, 2018). has studied the surface modification for Ti-6Al-2Nb-Ta using alkaline treatment with sodium hydroxide solution (NaOH). The surface modification using alkaline treatment produces strong bonding of apatite layer to the substrate and uniform gradient of stress transfer to be used in bone replacement. Many different parameters has been studied using alkaline treatment including soaking time of the alloy, concentration of the alkaline solution and temperature used during alkaline treatment . Du *et al.* (2014) has studied the influence of alkaline treatment to the Ti-6Al-4V and hydroxyapatite coating, whereas Zhao *et al.*, (2015) has studied the effect of concentration of alkaline treatment to the Ti alloy surface and Hanib *et al.*, (2016) has studied the effect of temperature used for alkaline treatment to the Ti-6Al-4V.

Even though many research have investigated modifying Ti and alloys surface by alkaline treatment for improving bioactivity properties, treatment in KOH is still less reported. Investigation on phase formation and the developed microstructure under the variation of treatment variables such as concentration of KOH and post heat treatment, are still lacking. In addition, alkali-heat treatment on new Ti alloy has not been explored and reported to date. Research on simple alkaline treatment of new alloy such as Ti-Nb is necessary to determine the feasibility of Ti-Nb undergo alkali-heat treatment considering it has potential as non-toxic and biocompatible implant alloy.

The present study was aimed to investigate the feasibility of surface modification of a new alloy of β-Ti alloy with Ti-40wt% Nb in KOH solution towards the formation of alkali titanate layer. Phase, surface morphology and chemical composition of the deposited materials was compared to treated pure Ti and Nb in order to understand the behaviour of the new alloy under KOH treatment.

1.2 Problem statement

A new biocompatible and non-toxic β-Ti alloy has been reported to solve the problem of great difference in elastic modulus of human bone and commercial Ti and Ti-Al-V alloy implants. Surface treatment in alkaline solution has been reported to be success on the enhancement of bioactivities commercial pure titanium and titanium alloy with low amount of alloying elements, such as Ti-6Al-2Nb-Ta. Thus, responsive of the Ti-40wt% Nb alloy towards alkaline treatment needs investigation as a comparison to pure Ti and Nb. Alkaline treatment has been done in different kind of alkaline solution. Till date sodium hydroxide (NaOH) received more attention compared to potassium hydroxide (KOH) while KOH has been claimed is more reactive than NaOH. Thus, structure and composition of the deposited material on the Ti-Nb alloy need detail investigation.

Ti metals exposed to strong alkali formed apatite on the surface when soaked in simulated body fluid due to formation of negative charge titanate hydrogel that form when Ti metal immerse in KOH. This is because absorption and accumulation of positively charge K^+ ions in KOH solution to the negative charge titanate hydrogel layer on the Ti metal surface. Concentration of the alkaline solution is one of the important variables in controlling the surface charge. While high concentration hydroxyl (OH^-) ions could excessively attack the TiO_2 passive layer as well as Ti substrate. Due to these possible reactions that occur simultaneously, morphology and type as well as surface properties of deposited material need to be identified under high and low concentration of KOH.

Combination of alkaline treatment and post heat treatment is reported increase biocompatibility and enhance bonding with living bone because alkaline treatment alone formed unstable hydrogel titanate layer with poor strength which could be damaged during preservation or implantation in human body. The post heat treatment would densify the hydrogel formation, which is accompanied with enhanced mechanical integrity, thus bone bonding ability of the titanate gel could be maintained. However, elevated temperature could cause diffusion of atoms in the hydrogel that might results in high crystallinity or phase transformation. New characteristic of the heat treated titanate requires an investigation on the bioactivity properties.

1.3 Objectives

1. To study alkaline titanate hydrogel formation effect by KOH alkaline treatment on Ti-40wt%Nb alloys with different concentration of KOH solution.
2. To compare phase, surface morphology and chemical composition of the KOH treated surface Ti-40wt%Nb with pure Ti and Nb.
3. To investigate the effect of heat treatment on surface of alkali treated metals

1.4 Scope of study

In this work, KOH alkaline treatment was conducted on a Ti-40Nb alloy in order to enhance bioactivity of the alloy. The alloy was fabricated using powder metallurgy parameters and composition developed by previous work (Ahmad, 2020 and Ahmad and Hussain, 2018) which reported has potential for implant material. Two concentrations of KOH were selected based on literature i.e., 0.5 and 5M which reported for Ti and Nb while the other variables were kept constant. Heat treatment at 600°C was also performed after the KOH treatment to study the effect of heat on the deposited material on the metal surface. Surface structural changes of the treated and non-treated Ti-40Nb alloys were examined using X-ray diffractometer (XRD) and a scanning electron microscope (SEM) attach with energy-dispersive electron probe X-ray analyzer (EDX).Bioactivity of the alloy was investigated in Hank solution as it is necessary to investigate electrochemical and behavior of the new alloy under the simulated body fluid (Hong *et al.*, 2010).

1.5 Thesis outline

This thesis was organized into five chapter. The first chapter cover the research background, problem statement, objective, and scope of research. In the second chapter, literature review cover on the Ti-40Nb, alkaline treatment and heat-treatment. Chapter 3 explains the material used, experimental procedure, parameter conduct, and the characterization. The result and discussion are discussed in chapter 4. Finally, chapter 5 conclude the research and recommendation for future work.

CHAPTER 2

LITERATURE REVIEW

2.1 Biomaterial for implant

Biomaterial for implant nowadays has become a demand for the biomedical industry because it was a material that can be used in human body which the biocompatibility between the bone and the implant material is essential. Suitable biomaterial for implant material should be biocompatible, bioactive or surface reactive, bioinert, biodegradable, low weight, reasonable cost as well as excellent in mechanical and physical properties.

Based on Table 2.1, the used of biomaterial is depend on their specific properties which suitable for the body part of human body that would be replace or support the part of lost function due to diseases or trauma. The replacement of damage part is to assist healing and improve the performance of the body. Different biomaterial has different properties which would influence the function of the implant part.

Table 2. 1: Uses of biomaterials (Parida et al., 2012)

Problem area infected	Examples of implant part
Replacement of damaged or diseased part	kidney dialysis machine and artificial hip joint
Support in healing	Sutures, screws and bone plates
Recover the function	Intraocular lens and Cardiac pacemaker
Correct functional abnormality	pacemaker of cardiac
To diagnosis	Probes and catheters
For treatment	Catheters, drains

Total hip joint replacement as shown in Figure 2.1 gaining popularity in medical field to improve the quality of patient that suffering arthritis and replacing damaged joint. However, factor that influencing the implant is the type of material used, mechanical properties and the biocompatibility of the material that would affect the life span and performance of the implant material.

The commonly used biomaterial in medical field is metal, ceramics, glass and plastic that were used as an implant, devices, and support. Despite that, although metal alloy implant are frequently used as total hip replacement, metal ions are released due to wear which can create negative effect to the patient body as mentioned by Ghalme *et al.* (2016). In total hip replacement, the femoral head used ceramic because of its high specific strength and toughness which would reduce the risk of fracture according to Affatato et al. (2015). As stated by DiPuccio et al. (2015), the plastic used only for the socket replacement which is Plastic Liner in between acetabular component and femoral head.

Variety of implant material are produced to fulfill the need of various medical application. Biomaterial that has been improved in term of inertness and corrosion resistance which can enhance the cytotoxicity. Cytotoxicity are involving the quality of the material whether it will become toxic to cell that affected the acceptance of the body to material. Implant that is rejected by the body will cause implant failure. Preferred biomaterial should be material that biologically, functionally, and mechanically compatible with bone.