PROPERTIES AND OPTIMIZATION OF TiN COATING ON Ti-51at. %Ni, Ti-27at. %Nb AND Ti-25at. %Ta ALLOYS USING MAGNETRON SPUTTERING FOR BIOMEDICAL APPLICATIONS

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DEDICATION

This thesis is dedicated to Allah Almighty, my Creator, my strong pillar, my source of inspiration, wisdom, knowledge, and comprehension. He has been the source of my strength throughout this programme, and on His wings have I soared. I also dedicate this work to my parents for their unceasing motivational support, as well as to my wives, children, brothers, sisters and friends for their prayers, support, and encouragement.

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ABSTRACT

Ti-based alloys belong to the categories of metals that are being used extensively in biomedical implants because they possess unique properties, such as high strength, corrosion resistance and biocompatibility. Among many Ti-based alloys, Ti-Ni alloys have been widely applied for biomedical applications. However, it has been discovered that Nickel is a toxic element that can cause hypersensitivity on human body. Hence, the need to develop Ni-free Ti-based alloys for biomedical applications is of paramount importance. β type Ti alloys, such as Ti-27at.%Nb and Ti-25at.%Ta consisting of non-toxic elements are some of the strong candidates for the replacement of Ti-Ni alloys. The aim of this research was to modify the Ti-51at.%Ni, Ti-27at.%Nb and Ti-25at.%Ta alloys by applying TiN coating on the surface of the alloys to improve their corrosion resistance, wear resistance, surface hardness and biocompatibility. Titanium Nitride (TiN) was selected as the coating material deposited on the substrates through physical vapour deposition magnetron sputtering method with varying deposition parameters namely, temperature, power, bias voltage and Nitrogen flow rate. The Taguchi method of parameter optimization through design of experiment was adopted and Taguchi orthogonal array standard 9-run matrix $L9(3^4)$ was applied to reduce the number of experimental runs to only 9 experiments. Microstructural and phase variation of the coated and uncoated Ti-51at.%Ni, Ti-27at.%Nb, and Ti-25at.%Ta alloys was determined using scanning electron microscope (SEM), energy dispersive spectrometer and x-ray diffractometer. The surface hardness (344 ± 12.5 HV, 325 ± 26.5 HV and 359 ± 7.9 HV) adhesion strength, (2999 ± 149.5mN, 2110 ± 100mN, 2145 ± 12.3mN) and coating thickness (1.171µm, 1.92µm, 1.78µm) were measured using micro hardness and scratch adhesion test equipment, respectively. Corrosion properties were evaluated using both electrochemical and immersion tests in simulated body fluids (SBF). Antibacterial test was performed on the coated samples using agar disc diffusion technique with *Escherichia coli* bacteria. Based on microstructural characterisation, all the alloys showed typical features and morphologies of Ti-51at.%Ni, Ti-27at.%Nb, and Ti-25at.%Ta. The coating materials deposited on the alloys were found to be composed of TiN coatings. The coatings showed improvement of surface hardness: 88.8%, 30%, 35.5%, and adhesion strength: 10.7%, 30.6% and 15.9% for all coated alloys of Ti-51at.%Ni, Ti-27at.%Nb, and Ti-25at.%Ta, respectively. The results of the bio-corrosion test showed that both the coated and uncoated alloys had excellent corrosion resistance after 28 days of immersion in SBF solution at a constant temperature of 37^oC. Similarly, the electrochemical test conducted at 37^oC in SBF solution, showed that the uncoated and coated samples had high resistance towards corrosion. The antibacterial test results indicated that the uncoated alloys exhibited sign of the presence of antibacterial activities with small inhibition zones formed around them. However, no inhibition zones were observed in the coated alloys due to the presence of the deposited TiN coatings that acted as a physical barrier between the alloys and their surroundings. Nevertheless, the TiN coated Ti-based alloys have tremendous potential as materials for biomedical applications.

ABSTRAK

Aloi berasaskan Ti tergolong dalam kategori logam yang digunakan secara meluas dalam implan bioperubatan kerana mempunyai sifat unik seperti kekuatan tinggi, ketahanan kakisan dan keserasian bio. Di antara banyak aloi berasaskan Ti, aloi Ti-Ni telah digunakan secara meluas untuk aplikasi bioperubatan. Namun, telah diketahui bahawa Nikel adalah unsur toksik yang boleh menyebabkan hipersensitiviti pada tubuh manusia. Oleh itu, keperluan untuk membangunkan aloi berasaskan Ti tanpa Ni untuk aplikasi bioperubatan sangat penting. Aloi Ti jenis β, seperti Ti-27at% Nb dan Ti-25at. % Ta yang terdiri daripada unsur-unsur tidak beracun adalah beberapa calon kuat untuk penggantian aloi Ti-Ni. Tujuan penyelidikan ini adalah untuk mengubah aloi Ti-51at.%Ni, Ti-27at.%Nb dan Ti-25at.%Ta dengan menggunakan salutan filem nipis di permukaan aloi untuk meningkatkan kekuatan lekatannya, ketahanan kakisan, kekerasan permukaan dan keserasian bio. Titanium Nitride (TiN) dipilih sebagai bahan salutan dimendap pada substrat melalui kaedah magnetron terpercik pemendapan wap fizikal dengan parameter pemendapan yang berbeza iaitu, suhu, kuasa, voltan bias dan kadar aliran Nitrogen. Kaedah Taguchi pengoptimuman parameter melalui reka bentuk eksperimen digunakan dan matriks piawaian 9-larian Taguchi L9 (3^4) diterapkan untuk mengurangkan jumlah larian eksperimen kepada 9 sahaja. Variasi struktur mikro aloi bersalut dan tidak bersalut ditentukan menggunakan mikroskop elektron imbasan, spektrometer penyebaran tenaga dan difraktometer sinar-x. Kekerasan permukaan (344 ± 12.5 HV, 325 ± 26.5 HV dan 359 ± 7.9 HV), kekuatan lekatan (2999 ± 149.5mN, 2110 ± 100mN, 2145 ± 12.3mN) dan ketebalan lapisan (1.171µm, 1.92µm, 1.78µm) diukur masing-masing menggunakan peralatan ujian mikro dan lekatan. Sifat kakisan dinilai menggunakan ujian elektrokimia dan rendaman dalam simulasi cecair badan (SBF). Ujian keserasian bio telah dilakukan pada aloi berasaskan Ti yang dilapisi menggunakan teknik penyebaran cakera agar dengan bakteria *Escherichia coli*. Berdasarkan pencirian mikrostruktur, semua aloi menunjukkan ciri tipikal dan morfologi aloi berasaskan Ti. Bahan salutan dimendapkan pada aloi didapati terdiri daripada lapisan TiN. Bahan salutan menunjukkan penambahbaikan kekerasan permukaan: 88.8%, 30%, 35.5%, dan kekuatan lekatan: 10.7%, 30.6% and 15.9% bagi semua aloi tersalut masing-masing untuk Ti-51at.% Ni, Ti-27at.%Nb, dan Ti-25at.%Ta. Hasil ujian bio-kakisan menunjukkan bahawa kedua-dua aloi yang disalut dan tidak disalut mempunyai ketahanan kakisan yang amat baik setelah 28 hari rendaman dalam larutan SBF pada suhu tetap 37°C. Begitu juga, ujian elektrokimia yang dijalankan pada suhu 37°C dalam larutan SBF, menunjukkan bahawa sampel disalut dan tanpa salutan mempunyai ketahanan yang tinggi terhadap kakisan. Hasil ujian antibakteria menunjukkan bahawa aloi yang tidak disalut menunjukkan tanda-tanda adanya aktiviti antibakteria dengan zon penghambatan kecil yang terbentuk di sekitarnya, namun, tidak ada zon penghambatan yang diperhatikan pada aloi bersalut kerana terdapat lapisan TiN yang bertindak sebagai penghalang fizikal antara aloi dan persekitarannya. Walau bagaimanapun aloi berasaskan Ti tersalut TiN mempunyai potensi yang sangat besar sebagai bahan untuk aplikasi bioperubatan.

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LIST OF ABBREVIATIONS

AFM	-	Atomic force microscopy
APC	-	Amorphous calcium phosphate
ASTM	-	American Society for Testing and Materials
BCC	-	Body Centred Cubic
BCP	-	Biphasic calcium phosphates
CAE	-	Cathodic arc evaporation
CFU	-	Colony Forming Units
CPE	-	Constant phase element
CVD	-	Chemical Vapour Deposition
DC	-	Direct current
D-GUN	-	Detonation gun
DOE	-	Design of Experiment
DSC	-	Differential Scanning Calorimeter
EBSD	-	Electron backscatter diffraction
EDM	-	Electro-Discharged Machining
EDS	-	Energy Dispersive Spectroscopy
EDX	-	Energy Dispersive X-ray
EEC	-	Electrical equivalent circuit
EIS	-	Electrochemical impedance spectroscopy
FCC	-	Face Centred Cubic
FESEM	-	Field Emission Scanning Electron Microscope
FWHM	-	Full width at half maximum
GIXRD	-	Grazing-incidence X-ray diffraction
GPa	-	Gigapascal
HA	-	Hydroxyapatite
HCA	-	Hydroxyl carbonated apatite
HCP	-	Hexagonal Closed Pack
HTSMA	-	High-temperature shape memory alloys
ICDD	-	International Centre for Diffraction Data

IPF	-	Inverse pole figure
OA	-	Orthogonal array
OM	-	Optical Microscope
PBS	-	Phosphorous Buffer Solution
PIRAC	-	Powder immersion reaction assisted coating
PVD	-	Physical Vapour Deposition
PVDMS	-	Physical vapour deposition magnetron sputtering
RF	-	Radio frequency
RSM	-	Response surface methodology
S/N	-	Signal to noise ratio
SBF	-	Simulated body fluid
SE	-	Super-elasticity
SEM	-	Scanning Electron Microscope
SMAs	-	Shape Memory Alloys
SME	-	Shape Memory Effect
TiN	-	Titaniun nitride
TNTZ	-	Titanium-niobium-tantalum-zirconium
Ts	-	Substrate temperature
Vb	-	Bias voltage
XRD	-	X-ray Diffraction

LIST OF SYMBOLS

ε - Strain value

$|Z|$ - Impedance on the Nyquist Plot

A - Area of exposed specimen (cm3)

d - Density of the corroding metal in (g/cm3)

D - Crystallites(grain) size

E.W - Equivalent weight of the corroding metal(g)

Iac - Alternating current(A)

i_{corr} - Corrosion current density ($\mu A/cm^2$)

k - Scherrer constant

n - Number of observations

Sa - Surface area of specimen (mm^2)

Vac - Voltage for alternating current(V)

V_S - Volume of SBF (ml)

y - Observed data

$\bar{y}$ - Average of observed data

$\bar{\gamma}_m$ - Total average S/N ratio value

Z - Impedance(ohm)

β - Full-width at half maximum

βa - Tafel slopes of the anodic reactions

βc - Tafel slopes of the cathodic reactions

λ - Wave length of the x-ray source

φ - Phase angle

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CHAPTER 1

INTRODUCTION

1.1 Background of the Research

The human body inner framework is the skeletal system, which comprised many bones of different physical structure roles. The bone is subjected to deterioration due to social, bodily processes, injury, and disease. A significant illness that is faced by the aged and sometimes the young people is arthritis, it causes impairment to the life of those affected, and it could lead to unbearable pain and immobility (Manivasagam *et al.*, 2010). Apart from people affected by the disease, agile and young people such as sportsmen and women often need replacements of bioimplants due to fracture and excessive strain. The complex problems encountered in bioimplants has been their contact with the biological environment of various physico-chemical nature and interaction with tissue and bone (Manivasagam *et al.*, 2010). It is essential for biomaterial choice to be acceptable to the human body without immunological rejection in the body and to have an excellent response to tissue cells. A biomaterial is an adapted material for medical applications (Tathe *et al.* 2010).

In the human body, biomaterial and tissues will be in contact, therefore the material presence should not trigger an unacceptable level of harm to the body. The materials should have mechanical properties such as tensile strength, hardness, and low modulus of elasticity, corrosion resistance, and wear resistance (Manjaiah and Laubscher, 2017). In this respect, the need for collaboration between specialists such as mechanical engineers, material scientists, metallurgists, orthopaedists with track records of experience are of paramount importance in achieving excellent results in the research, development, and execution of the knowledge extracted in practice (Li *et al.*, 2014b). However, developments in the field of biomedical engineering have led to continuous renewed interest in biomaterial requirement to resolve the problems of failed hard tissues such as hip joints, knee joints, dental implants, by using stainless steel, cobalt-based alloys, and titanium alloys, which are known to be primary metallic materials as a suitable replacement for hard tissues (Geetha *et al.*, 2009; (Niinomi, 2003).

In recent years, there has been an increasing interest in titanium and titanium alloys among metallic biomaterials because of their properties of low elastic modulus, corrosion resistance, wear resistance, shape memory behaviour, high specific strength, and excellent biocompatibility (Niinomi, 2002; Temenoff and Mikos, 2008) which makes them suitable metals for biomedical applications. Recent developments in biomedical Ti-alloys focus on their shape memory behaviour. In this regard, Ti-Ni alloys, which fall within the category of shape memory alloys, have been widely used in biomedical applications, among many Ti-based alloys.

At the end of the 1970s, researchers introduced Ti-Ni alloy as a biomedical metal after finding that the alloy possesses unique mechanical properties, excellent corrosion resistance, shape memory properties, and biocompatibility (Kim *et al.*, 2006a; Miyazaki *et al.*, 2006; Pfeifer *et al.*, 2013). Additionally, Ti-Ni alloy has two mechanical properties comparable to those of natural biomaterials such as bone, including the ability to recover a large amount of strain and a low elastic modulus. Ti-Ni alloy can recover up to 8% strain, and this is beneficial since it can substitute bone that naturally recovers a strain by 2% compared to stainless steel, which is just around 0.8%. Ti-Ni alloy elastic modulus could be down to 48 GPa near the dense bone with less than 20 GPa. By contrast, stainless steel elastic modulus can reach up to 193 GPa (Chu *et al.*, 2004). They are ideal as biomaterials, especially for orthopaedic and orthodontic surgery due to the two qualities mentioned earlier.

The more extensive medical applications of Ti-Ni alloys were restricted despite their use as a biomaterial for a long time, some researchers (Xiong *et al.*, 2010) later discovered that these alloys are not entirely biocompatible. There have been reports that Ni is a toxic agent and allergens that can induce hypersensitivity to the human body (Zhang *et al.* 2015). Many researchers (Bernard *et al.*, 2011) have taken serious steps towards finding a solution to Ni ions release from the surface of Ti-Ni. High levels of Ni in Ti-Ni alloys may damage the bone structures because of their toxic effect on structures of the soft tissue compared to Co and V, also widely used in implants, the effects of which are significantly lower (Brojan *et al.*, 2008). Additionally, previous research (Abbass *et al.*, 2018) has shown that toxicity is not the only problem in Ti-Ni alloys biocompatibility and bioactivity. The integrity of the implant-bone interface was found to be a critical problem (Chen *et al.*, 2004).

In line with tissue engineering and biomedical technology, biomaterials are meant to have wear resistance, shape memory effect, or super elasticity and low elastic modulus. Additionally, they are required to get rid of toxic effects that could lead to corrosion, wear, leaching, and avoid the use of Ni in biomedical implants (Hermawan *et al.*, 2011). It is, therefore, crucial to develop safe, Ni-free alloys for biomedical applications.

The attention of researchers (Miyazaki *et al.*, 2006) have been drawn to a newly discovered biomedical β type Ti-based alloys with shape memory and super elastic properties due to their outstanding cold workability. β Ti-based alloys are commonly seen as β disordered body centre cubic (BCC) in stable high-temperature phases and α hexagonal close pack (HCP) low temperatures. Previous studies (Nagase *et al.*, 2010) demonstrated that β-type Ti-based alloys might also be suitable for future medical applications due to their low Young's modulus and ease of cold treatment formation. (Bartáková *et al.*, 2009).

A widespread study has been conducted on Ti-based alloys consisting of several other non-toxic and β-stabilizing elements such as Nb, Zr, Ta, Sn, Mo, and Pt, which may be appropriate for biomedical applications (Wang *et al.* 2010). Nb and Ta are candidates of choice as alloying elements for biomaterial implants compared to pure Ti due to their biocompatibility and superior electrochemical properties. Besides, Ta has excellent resistance to corrosion with low ion release (Ching *et al.*, 2014). The Studies carried out previously (Xu *et al.*, 2013) revealed Ti-Nb as the most exciting alloy to be developed for biomedical applications because it exhibits shape memory effect, low elastic modulus, and high compatibility. Other than Ti-Nb, Ti-Ta alloys are also an excellent option for future medical applications. Besides being non-toxic and having excellent biocompatibility, Ti-Ta also has a low elastic modulus similar to Ti-Nb alloy, which is essential for implant materials. Bartáková *et al.*, (2009) observed that the Ti-Ta alloy has the best biocompatibility for all of the tested biocompatibility parameters. The properties of these alloys may be enhanced by surface coating with biocompatible materials.

Coatings are usually used to improve the surface properties of a material without negatively impacting the bulk materials. Furthermore, coatings can serve as an effective protective layer to reduce ions release, which contributes to corrosion (Fauchais and Vardelle, 2012). It can enhance hardness while also providing quality surface finishing, lowering friction and wear rates (Chen *et al.*, 2013; Chowdhury *et al.*, 2008). It is important to remember that a coating layer is only functional if it adheres well to the metal substrate and is strong enough to transfer all loads, depending on its original specific purpose. Coatings, on the other hand, have a limitation in terms of adhesion to the substrate, which allows chemical bonds to form between the layers.

In this research work, as cast Ti-51at%Ni, Ti-27at%Nb, and Ti-25at%Ta alloys were coated with TiN. Ti target was used to produce TiN coating by using a physical vapour deposition (PVD) magnetron sputtering system. The effects of the deposition parameters such as power, temperature bias voltage, and nitrogen flow rate on the surface hardness and adhesion strength of the TiN coated alloys for bioimplant implant, such as hip, knee and toe prostheses were investigated. The uncoated and coated samples were subjected to characterization and antibacterial tests to determine which coated alloy is the most suitable for biomedical applications.

1.2 Problem statement

Titanium alloys have various successful applications in surgical intervention and biomedical, aerospace, automobiles, chemical industries, and other vital industries, owing to their high strength, low weight ratio, and exceptional corrosion resistance. In many engineering applications, titanium alloys are replacing heavier, less serviceable, or less cost-effective materials. Titanium alloys properties enable designers to create systems and components that are dependable, cost-effective, and long-lasting.

Ti alloys are known to have low resistance to wear when compared to stainless steel and Co-Cr based alloys, which are used for biomedical applications. Their use as implant materials is therefore restricted as they wear easily when rubbed between themselves or between other metals (Nasab *et al.*, 2010). The use of the alloys in applications for load-bearing surfaces is rejected due to several cases of aseptic loosening resulting from the formation of metallic particulate debris (Bougherara *et al.*, 2010; Clarke *et al.*, 1992). Debris formation in load-bearing applications causes tissue blackening and metallosis as a result of the formation of a low adhering surface oxide layer, which periodically detaches from the surface (Dearnley, 1999). Ti-based alloys as implant materials are therefore limited only to applications where wear resistance properties, e.g., hip implant femoral head, are not required (Nag *et al.*, 2009). The development of Ti-Ni alloys for a more extensive medical use has been restricted despite their relatively long time use as biomaterials; this is because the medical profession is concern about the high nickel content of about 50% in bulk, which is a possible cause of nickel ion release (Li et al., 2014a; Xiong *et al.*, 2010). High levels of Ni in Ti-Ni alloys can damage bone structures because of its toxic effects on soft tissue structure (Brojan *et al.*, 2008). As a result, non-toxic nickel-free alloys with the lowest modulus of elasticity and high biocompatibility, such as Ti-Nb and Ti-Ta, are used to replace Ti-Ni (Xu *et al.*, 2013). Furthermore, coating is extremely important for improving the wear resistance, hardness, and biocompatibility of the Ti-alloys used as implant materials.

Titanium nitride (TiN) is a bioactive coating that improves both the biological and mechano-chemical behaviour of Ti-Ni in body applications among the several available coatings (Piscanec *et al.*, 2004). Besides, TiN is considerably more resistant to wear and corrosion than other compounds such as titanium oxide and titanium carbide that are bio-compatible (Poon *et al.*, 2005; Yeung *et al.*, 2005). TiN was thus chosen as a bio-active coating capable of improving the adhesion strength, hardness and corrosion resistance of Ti-Nb, Ti-Ta, as well as reducing the release of Ni ions from the Ti-Ni surface and improving its adhesion strength, hardness and corrosion resistance (Piscanec *et al.*, 2004; Starosvetsky and Gotman, 2001). The effects of the process parameters (temperature, power, bias voltage and nitrogen flow rate) on the improvement of surface hardness and adhesion strength of TiN coated samples for biomedical implant such as hip, knee and toe prosthesis were considered.

With regard to hip and knee replacement using biomedical implants, the femoral head of hip prosthesis and joints of knee prosthesis require greater adhesion strength of TiN coating as the primary criterion as well as a significant amount of surface hardness to prevent wear and debris formation, whereas the ankle and femoral stem necessitate greater hardness of TiN coating as the primary criterion as well as a significant amount of adhesion strength in the load bearing surfaces (Gobbi et al., 2019; Wang and Zreiqat, 2010). Therefore, optimizations of process parameters, such as temperature, power, bias voltage and nitrogen flow rate to obtain optimum adhesion strength or hardness of the TiN coated alloys for specific biomedical applications.

1.3 Significant of the Research

This study aims to coat TiN on Ti-51at % Ni, Ti-27 at %Nb, Ti-25 at % Ta alloys using the PVDMS method and evaluate the effects of the coating on the properties such as surface hardness, adhesion strength, corrosion resistance, and biocompatibility for biomedical applications. The results obtained through parameter optimization using the Taguchi method showed significant improvement in the properties, which would be beneficial in the production of implants for biomedical applications. However, the findings are expected to eliminate the need for additional surgery with the newly formed implant materials. The TiN coated alloys will improve wear resistance, implant durability and longevity, patient time, cost, risk, and morbidity.

1.4 Objectives of the Research

The objectives of this research are as follows:

(a) To determine the best coating parameters (power, temperature, bias voltage and nitrogen flow rate) for adhesion strength and hardness using the Taguchi method.

(b) To investigate the microstructural variation between the uncoated and coated Ti-51at. %Ni, Ti-27at. %Nb and Ti-25at. %Ta alloys at the selected process parameters using EBSD, FESEM/EDS, XRD AND AFM

(c) To determine the mechanical properties (hardness and scratch adhesion strength) of the coated Ti-51at. %Ni, Ti-27at. %Nb and Ti-25at. %Ta alloys at the selected process parameters

(d) To determine the antibacterial activities and bio-corrosion behaviour of the coated Ti-51at%Ni, Ti-27at. %Nb and Ti-25at. % Ta at the selected process parameters

1.5 Scopes of the Research

The scopes of the research are as follows:

(a) The deposition was carried out using DC magnetron sputtering physical vapour deposition (PVD) method to deposit TiN coating on Ti-51at. %Ni, Ti-27at. %Nb and Ti-25at. %Ta alloys

(b) Initial study on the PVD method by varying the deposition parameters such as bias voltage, temperature, pressure, and gas flow rate in order to determine suitable parameters to obtain the coating.

(c) Optimization process using Taguchi method was applied to get the best coating parameters for better adhesion and hardness.

(d) Coating adhesion, thickness and hardness were investigated using micro scratch testing machines (Micro Material Nanotest, Wrexham, UK), micro hardness tester and field emission scanning electron microscopy/ energy-dispersive X-ray spectroscopy (FESEM/EDS)

(e) Material characterization was performed on the coated and uncoated alloys using optical microscope (OM), field emission scanning electron microscopy/ energy-dispersive X-ray spectroscopy (FESEM/EDS), and X-ray diffraction (XRD).

(f) Bio-corrosion and antibacterial tests were carried out using electrochemical methods (EIS and Tafel plot), immersion and Agar diffusion technique.

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