

DEVELOPMENT AND CHARACTERIZATION OF Ti-5Cu-xNb Alloys FOR BIOMEDICAL APPLICATIONS



Thesis submitted in partial fulfillment for the
Award of Degree

Doctor of Philosophy

By

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This Doctoral Thesis is dedicated to my family,
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TABLE OF CONTENTS

CERTIFICATE	ii
DECLARATION BY THE CANDIDATE	iii
COPYRIGHT TRANSFER CERTIFICATE	iv
ACKNOWLEDGEMENT	v
DEDICATION	vii
TABLE OF CONTENTS	ix
LIST OF FIGURES	xiv
LIST OF TABLES	xviii
LIST OF ABBREVIATIONS & SYMBOLS	xix
PREFACE	xxiii
Chapter 1	1
Introduction	1
Chapter- 2	5
Literature review	5
2.1 Biomaterial	5
2.2 Basic requirement of a biomaterial	6
2.2.1 Biocompatibility	6
2.2.2 Corrosion resistance	6
2.2.3 Mechanical properties	7

2.2.4 Wear resistance	7
2.2.5 Osseo-integration	8
2.2.6 Antibacterial properties	8
2.3 Conventional metallic implant material	9
2.3.1 Stainless steel	9
2.3.2 Cobalt-chromium alloys	10
2.3.3 Titanium and its alloys	10
2.4 Titanium properties and microstructure	12
2.5 Powder metallurgy technique for development of titanium alloys	14
2.6 Peri-implantitis	15
2.7 Corrosion	16
2.7.1 Types of corrosion	16
2.7.2 Corrosion of titanium and its alloys	18
2.8 Tribology	19
2.8.1 Friction	19
2.8.2 Wear	19
2.8.3 Tribology of titanium and its alloys	22
2.9 Effect of alloying Cu and Nb to titanium alloys on microstructure, mechanical properties, corrosion behaviour, wear resistance, antibacterial property, and cytocompatibility	23
2.10 Formulation of the problem and objective of the study	29
Chapter- 3	31

Materials and Methodology	31
3.1. Material selection for the development of ternary titanium alloy	31
3.2. Development of ternary titanium alloy	31
3.3. Phase Evaluation and Microstructural Characterizations	34
3.3.1 X-ray diffraction analysis of hybrid composites	34
3.3.2 Microstructural characterization	35
3.4 Mechanical properties testing	35
3.4.1 Density measurement of sintered alloy	35
3.4.2 Micro-hardness measurements	36
3.4.3 Compression test	37
3.5 Electrochemical corrosion measurements	38
3.6 Wear test of the sintered alloys	40
3.7 Cell culture and antibacterial test	42
3.7.1 Cell Study:	42
3.7.2 Cell proliferation	43
3.7.3 Morphological analysis:	43
3.7.4 Antibacterial test	44
3.7.4.1 Preparation	44
3.7.4.2 Plate count method	44
Chapter-4	46
Results and Discussion	46
MICROSTRUCTURAL, PHYSICAL, AND MECHANICAL BEHAVIOUR OF SINTERED ALLOY	

4.1 Introduction	46
4.2 Phase identification and microstructure characterization	46
4.3 Physical and Mechanical properties	54
Chapter-5	59
Result and Discussions	59
CORROSION AND TRIBOLOGICAL BEHAVIOUR OF SINTERED ALLOY	
5.1 Introduction	59
5.2 Corrosion study	59
5.2.1 Open-circuit potential	59
5.2.2 EIS measurements	61
5.2.3 Potentiodynamic polarization behavior	65
5.2.4 Surface Analysis	68
5.3 Wear and frictional behavior of alloys	71
5.3.1 Coefficient of friction	71
5.3.2 Worn surface width and lateral depth	74
5.3.3 Wear volume and wear rate	77
5.3.4 Worn surface characterization	79
5.3.5 Scanning probe microscope analysis	85
Chapter- 6	88
Result and Discussions	88

ANTIBACTERIAL AND CYTOCOMPATIBILITY BEHAVIOR OF SINTERED ALLOYS	
6.1 Introduction	88
6.2 Antibacterial properties of the sintered alloys	88
6.3 Cytocompatibility assessment of sintered alloys	91
6.3.1 Cell culture and morphological analysis	91
6.3.2 MTT Assay	93
Chapter-7	94
CONCLUSIONS AND FUTURE SCOPE	94
REFERENCES	102

LIST OF FIGURES

Fig. No.	Captions	Page No.
Fig. 2.1.	Wear between the hip implant and human bone	8
Fig. 2.2.	Elastic modulus of alloy in comparison to human bone	12
Fig. 2.3.	An investigation of the effect of alloying elements on the phase diagrams of titanium alloys	13
Fig. 2.4.	Peri-implantitis in dental implant (a) healthy implant, (b) peri-implant mucositis, and (c) Peri-implantitis	16
Fig. 2.5.	Mechanisms of wear in general: adhesive wear; abrasive wear; fatigue wear; corrosive wear	20
Fig. 3.1.	The schematic diagram of the ball milling process	32
Fig. 3.2.	The schematic diagram of compaction of powder in die.	32
Fig. 3.3.	The process flow chart of the development of ternary titanium alloy	33
Fig. 3.4	The pictorial image of the alloy (a) after sintering and (b) after cloth polishing	33
Fig. 3.5	(a) indentation marked by indenter and (b) measurements of diagonal dimension	37
Fig. 3.6	Pictorial image of the INSTRON universal testing machine during the compression test	38
Fig. 3.7	Image of the CCORRTEST CS 2350 Potentiostat test setup during testing	39
Fig. 3.8	Image of the Tribometer used for wear study	41
Fig. 3.9	Schematic of tribological study in reciprocating linear motion of samples in SBF.	42
Fig. 4.1	XRD pattern of S1, S2, S3, and S4 alloy powder after ball milling.	47
Fig. 4.2	XRD pattern of (a) S1, (b) S2, (c) S3, (d) S4, and (e) volume fraction of formed phases after sintering.	48
Fig. 4.3	Optical microscopy image of sintered alloy (a) S1, (b) S2, (c) S3, (d) and S4	50
Fig. 4.4	The scanning electron microscopy image of sintered alloy, (a) S1, (b) S2, (c) S3, and (d) S4.	51

Fig. 4.5.	The HR-SEM image (a), EDS map scanning image of Ti (b) and Cu (c), weight and atomic percent of element (d), and EDS point scanning pattern (e) of sintered alloy S1	52
Fig. 4.6.	The HR-SEM image (a), EDS map scanning image of Ti (b), Nb (c), Cu (d), and EDS scanning pattern (e) of sintered alloy S2.	53
Fig. 4.7.	The HR-SEM image (a), EDS map scanning image of Ti (b), Nb (c), and Cu (d), EDS point scanning pattern (e) of sintered alloy S3.	53
Fig. 4.8.	The HR-SEM image (a), EDS map scanning image of Ti (b), Nb (c), and Cu (d), EDS point scanning pattern (e) of sintered alloy S4.	54
Fig. 4.9	Comparison of theoretical and sintered density of the alloys	56
Fig. 4.10.	Vickers micro-hardness of samples	56
Fig. 4.11.	Compressive strength value of samples observed after compression test.	57
Fig. 4.12	Variation of Engineering stress with Engineering strain of alloys S1, S2, S3, and S4.	58
Fig. 5.1	Open-circuit potential test result of the sintered alloys after 3600s	60
Fig. 5.2	The Bode plot obtained from the EIS test	64
Fig. 5.3	The Nyquist plot obtained from the EIS test	64
Fig. 5.4	Proposed equivalent circuit diagram of the EIS test after fitting	65
Fig. 5.5	Potentiodynamic polarization curve of sintered alloys	66
Fig. 5.6	SEM image of the corroded sample (a) Ti-5Cu, (b) Ti-5Cu-5Nb, (c) Ti-5Cu-10Nb, and (d) Ti-5Cu-15Nb sintered alloys	69
Fig. 5.7	XPS spectra of the sintered alloy after the potentiodynamic polarization test	70
Fig. 5.8	XPS spectra of the element (a) Oxygen, (b) Titanium, (c) Copper, and (d) niobium.	70
Fig. 5.9	Coefficient of friction vs. running cycles graph of samples (a) Ti-5Cu, (b) Ti-5Cu-5Nb, (c) Ti-5Cu-10Nb, and (d) Ti-5Cu-15Nb at 10 N, 15 N, and 20 N loads, respectively.	73
Fig. 5.10	The average friction coefficient of sintered alloys running after 3600 cycles at 10 N, 15 N, and 20 N load, respectively.	73

Fig. 5.11	Measurement of the worn surface by profilometer (a, b, and c) of Ti-5Cu, and (d, e, and f) of Ti-5Cu-5Nb at 10N, 15N, and 20N load, respectively	76
Fig. 5.12	Measurement of the worn surface by profilometer (a, b, and c) of Ti-5Cu-10Nb, and (d, e, and f) of Ti-5Cu-15Nb at 10 N, 15 N, and 20 N load.	76
Fig. 5.13	Width and lateral depth of worn surfaces at all three loadings against zirconia ball.	77
Fig. 5.14	The wear rate of all four samples running after 3600 cycles against zirconia ball in SBF.	78
Fig. 5.15	The SEM image and EDS analysis of the worn surface of Ti-5Cu at 10 N (a,d,g), 15 N (b,e,h), and 20 N (c,f, i) load after 3600 cycles.	80
Fig. 5.16	The SEM micrograph and EDS analysis of the worn surface of Ti-5Cu-5Nb at 10 N (a,d,g), 15 N (b,e,h), and 20 N (c,f, i) load after 3600 cycles.	81
Fig. 5.17	The SEM micrograph and EDS analysis of the worn surface of Ti-5Cu-10Nb at 10 N (a,d,g), 15 N (b,e,h), and 20 N (c,f, i) load after 3600 cycles.	82
Fig. 5.18	The SEM micrograph and EDS analysis of the worn surface of Ti-5Cu-15Nb at 10 N (a,d,g), 15 N (b,e,h), and 20 N (c,f, i) load after 3600 cycles.	83
Fig. 5.19	EDS point spectrum analysis of the worn surface of Ti-5Cu-5Nb at 20 N load.	84
Fig. 5.20	EDS point spectrum analysis of the worn surface of Ti-5Cu-10Nb at 15 N load.	85
Fig. 5.21	Surface topography of Ti-5Cu alloy at (a) 10 N, (b) 15 N, and (c) 20 N load of worn surface, and images (d), (e), and (f) are 3D surface topography, respectively.	87
Fig. 5.22	Surface topography of Ti-5Cu-15Nb alloy at (a) 10 N, (b) 15 N, and (c) 20 N load of worn surface, and images (d), (e), (f) are 3D surface topography, respectively.	87

Fig. 6.1	Bacterial colony of <i>E. coli</i> . (a) negative sample, (b) control sample (CP-Ti), (c) S1, (d) S2, (e) S3, and (f) S4.	90
Fig. 6.2.	Bacterial colony of <i>S. aureus</i> . (a) negative sample, (b) control sample (CP-Ti), (c) S1, (d) S2, (e) S3, and (f) S4.	91
Fig. 6.3	The above panel represents the cell culture images of MG-63 human bone osteosarcoma cells on the various steel samples after 3 days of incubation. Blue color: nuclei staining; red color: actin cytoskeleton filaments staining	92
Fig. 6.4	MTT assay of MG-63 animal cells on S1, S2, S3, S4, and control samples on surfaces, the following culture for 3, 5, and 7 days.	93

LIST OF TABLES

Table No.	Captions	Page No.
Table 3.1.	Purity and supplier of the purchased powder	31
Table 3.2.	The nomenclature and composition of sintered alloy	34
Table 3.3.	Chemical composition of the reagents in 1000 mL simulated body fluid	39
Table 4.1.	Average crystalline size (angstrom) and average lattice strain (percentage) of ball-milled powder	48
Table 4.2.	Average crystalline size (angstrom) and average lattice strain (percentage) of sintered alloys.	49
Table 4.3.	The volume fraction of phases in the sintered alloys.	49
Table 4.4.	Micro-hardness (HV), density, porosity, and compressive strength of sintered alloys	55
Table 5.1.	Impedance constraints of the proposed equivalent circuit attained by fitting the experimental result of EIS	65
Table 5.2.	Corrosion current density, corrosion potential, breakdown potential, passive current, and re-passivation current of the sintered alloys	66
Table 5.3.	Parameter and results obtained after wear test of the samples in SBF.	78
Table 5.4.	The summary of surface roughness of worn surface for different loading.	86
Table 6.1.	Results obtained after the antibacterial test.	90

LIST OF ABBREVIATIONS & SYMBOLS

SS 316	:	Low carbon stainless steel
Co-Cr	:	Cobalt-Chromium
Ni	:	Nickel
CP-Ti	:	Commercially pure titanium
HCP	:	Hexagonal closed packed
BCC	:	Body centered cubic
mm	:	Millimeter
XRD	:	X-Ray Diffraction
HR-SEM	:	High- resolution scanning electron microscopy
EDAX	:	Energy Dispersive X-Ray Analysis
XPS	:	X-ray photoelectron spectroscopy
N	:	Newton
OCP	:	Open-circuit potential
EIS	:	Electrochemical impedance spectroscopy
SBF	:	Simulated body fluid
SEM	:	Scanning electron microscopy
SPM	:	Scanning probe microscopy
S. aureus	:	Staphylococcus aureus
E. coli	:	Escherichia coli
GPa	:	Giga Pascal
°C	:	Degree centigrade
Ti	:	Titanium

Nb	:	Niobium
Cu	:	Copper
PM	:	Powder metallurgy
μm	:	Micrometer
BT-XRD	:	Bench top X-Ray Diffraction
RPM	:	Revolution per minute
H	:	Hour
s	:	Second
min	:	Minute
\AA	:	Angstrom
$^{\circ}$:	Degree
Kgf	:	Kilogram force
kN	:	Kilo newton
mL	:	Milliliter
g	:	Gram
cm	:	Centimeter
Hz	:	Hertz
mV	:	Millivolt
FBS	:	Foetal bovine serum
PBS	:	Phosphate buffer saline
pH	:	Potential of Hydrogen
Ω	:	Ohm
μA	:	Micro Ampere
eV	:	Electron volt
nm	:	Nanometer

R_a	:	Average roughness
R_q	:	Root-mean-square roughness
S_a	:	arithmetic mean height
S_q	:	squared mean height
%	:	Percentage

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PREFACE

Metallic implants are frequently utilized in load-bearing applications to repair or replace bone and other hard tissues that have been injured or shattered. The replacement of natural bone with an artificial one made of metallic material is not a novel concept in the field of medical science. Since the 1920s, medical professionals have found the stainless steel 316L to be an appropriate material to utilize as a mimic for bone and tissues. The development of Co-Cr alloys in the latter half of the 1960s led to the establishment of another option for SS 316L. Unfortunately, the long-term application presents a challenge due to the fact that these alloys give out hazardous oxides composed of Cr and Ni. Titanium and its alloy have gained much popularity in medical science due to its outstanding biocompatibility, strong resistance to wear and corrosion, and overall good mechanical strength.

The higher Young's modulus of metallic implant material causes stress shielding, resulting in earlier failure of the implant. Commercially pure titanium (CP-Ti) and Ti-6Al-4V possess lesser young' modulus than Co-Cr and SS 316 L alloys. Pure titanium possesses two crystal structures; at room temperature, it shows a closed-packed hexagonal (*HCP or α -phase*) structure, and at a temperature above 882 °C body-centered cubic (*BCC or β - phase*). These different phases of titanium control the different properties based on the crystal structure. Generally, the alpha phase shows strong resistance to corrosion, and the beta phase gives it suitable mechanical properties to use as an implant. Ti-6Al-4V is an excellent example of a combined $\alpha+\beta$ phase. Various elements, including Zr, Al, Cu, Nb, Mo, and Ta, are utilized depending on the application to stabilize the various phases of titanium. When alloyed with titanium, niobium demonstrates superior cytocompatibility to both surrounding cells and tissue. The metallic endosseous implant in long-life applications also failed by the growth of bacteria around the periphery of the implant. "Peri-implantitis" is the medical term for implant failure

caused by bacteria. The continuous research is going on to develop an antibacterial metallic implant to resolve these problems with a metallic implant.

Many studies have been done on alloying copper to CP-Ti and Ti-6Al-4V to make it antibacterial and prevent the growth of *S. aureus* and *E. coli*. Bacteria around the periphery of the implant. The study shows that the inclusion of 5 wt.% copper is enough to make the titanium alloys antibacterial. Copper also enhances mechanical properties, corrosion, and wear resistance.

Thus, the present works aim to investigate phase formation, microstructure, mechanical behavior, corrosion confrontation, wear behavior, antibacterial ability, and biocompatibility of Nb inclusion to Ti-5Cu binary alloy. The novel Ti-5Cu-(0, 5, 10, and 15) %Nb alloys were successfully developed by the powder metallurgy technique. The different characterization techniques are used to investigate the various physical, chemical, mechanical, and biocompatibility properties of the sintered alloys.

The present thesis has been organized into eight chapters, as summarized below:

Chapter 1: This chapter comprises the introduction of titanium and its alloys as implant material with its specific properties and significant problem. Mechanical, corrosion, wear, antibacterial, and cytocompatibility properties are also discussed, followed by the application of the developed alloys in the present study.

Chapter 2: This chapter of the thesis presents the literature review of the topic related to the present study. The brief details of the history of biomaterials, metallic implants, advantages and disadvantages of metallic implants over ceramic and polymeric implants, the crystal structure of titanium, the effect of alloying different elements on the behavior of titanium alloy, peri-implantitis are reviewed in this chapter. Furthermore, the detailed literature survey of the mechanical behavior, corrosion, wear behavior, cytocompatibility, and antibacterial study of recently developed titanium alloys are discussed. Based on the literature survey, the motivation

of the present study, along with the objective of the study, is given in this chapter.

Chapter 3: This chapter of the thesis contains the details of materials and experimental techniques involved in the development of the novel Ti-5Cu-(0, 5, 10, and 15) %Nb alloys. In the present study, five number of samples of each composition of size ϕ 15 mm \times 5 mm were sintered using a conventional muffle furnace. The green samples of each composition were placed inside the hollow quartz tube of internal diameter 18 mm and sealed using gas flame with maintaining a high vacuum of 10^{-6} torr using a diffusion pump. After sealing, the tube was kept inside the muffle furnace at 900 °C for 1 h with a heating rate of 4 °C/min. After the sintering, the phase analysis of the alloys is done using X-Ray Diffraction (XRD). Also, the microstructure, elemental analysis, and mapping of the element distribution of the alloys are done using High- resolution scanning electron microscopy (HR-SEM) equipped with EDAX. The detailed procedure, methodology, and effect of varying alloying elements on mechanical and corrosion behavior are discussed in this chapter. The analysis of corroded surfaces by SEM and X-ray photoelectron spectroscopy (XPS) techniques is also performed in this chapter. Followed by the reciprocating wear test of the alloys against the zirconia counterpart at 10 N, 15 N, and 20 N in simulated body fluid is thoroughly discussed in this section. The details procedure of cytocompatibility testing of the alloys in MTT MG-63 animal cells and the antibacterial property assessment of the alloys are also discussed in detail in this chapter.

Chapter 4: This chapter deals with the results obtained after phase analysis of both milled powder and sintered alloys, along with the microstructural analysis of the sintered alloys. The volume phase fraction of alpha and beta-titanium, crystalline size, and average lattice strain of different phases are also discussed in detail. The results of elemental analysis and mapping of the element formed after sintering are also discussed in this chapter. Furthermore, the result and discussion about density, microhardness, and compressive strength of the alloys are discussed in detail.

Chapter 5: The potentiodynamic polarisation approach was used to investigate the corrosion behavior of the alloys in order to guarantee the stability of the alloys in their respective chemical environments. In this chapter, an in-depth discussion is done on the findings that were obtained from the open-circuit potential (OCP), electrochemical impedance spectroscopy (EIS), and potentiodynamic polarisation tests performed on the alloys in SBF. This chapter presents the findings and a discussion of the SEM and XPS analyses of the compound that formed on the surface of the alloys as a result of the corrosion test. The results of the reciprocating wear and friction test performed on the alloys in SBF are also discussed in this chapter. The results of friction coefficient, wear volume, depth, and width of the worn track, as well as wear rate obtained at different loadings of the alloys against the zirconia ball, are described in detail. These findings were acquired by rubbing the zirconia ball against the alloys. SEM, EDAX, and SPM are used to conduct the analysis of worn surfaces after the wear test. The results of these analyses are also described in detail in this chapter.

Chapter 6: This chapter contains the result and discussion about the cell culture, proliferation, viability, and antibacterial test of the alloys. The cell culture test is carried out in MG-63 animal cells for 3 days, 5 days, and 7 days to see the growth of cells on the surface of the alloys. The counts of the cell as also reported in this chapter. Also, the results and discussion about the antibacterial test of the alloys carried out in *Staphylococcus aureus* (*S. aureus*) and *Escherichia coli* (*E. coli*) bacteria are discussed in this chapter.

Chapter 7: This chapter contains the significant conclusions of all the tests conducted on the alloys in the present study. The findings in this chapter demonstrate that the currently developed alloys can perform their intended role, which is to simulate the natural load-bearing bone and hard tissues found in the human anatomy. The future scope of the above study is also contained in this chapter.

