

Wear, Corrosion, and Biocompatibility of Ta-Coated 316L Stainless Steel for Orthopedic Applications



*Thesis submitted in partial fulfillment for the
Award of Degree*

DOCTOR OF PHILOSOPHY

By

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Abbreviations

ASTM	American Society for Testing and Materials
316L SS	Low Carbon Stainless Steel type 316
DC	Direct Current
Ta	Tantalum
SBF	Simulated Body Fluid
CNC	Computer Numerical Controller
EDM	Electrical Discharge Machining
SCCM	Standard cubic centimetres per minute
MFC	Mass Flow Controller
DCMS	Direct Current Magnetron Sputtering System
DOE	Design of Experiment
OM	Optical Microscopy
OES	Optical Emission Spectroscopy
SEM	Scanning Electron Microscopy
EDS	Energy Dispersive X-Ray Spectroscopy
BT-SEM	Bench Top Scanning Electron Microscopy
AFM	Atomic Force Microscopy
XPS	X-ray Photoelectron Spectroscopy
SPM	Scanning Probe Microscopy
ICP-MS	Inductive Coupled Plasma-Mass Spectroscopy
CAM	Contact Angle Measurement
WEDM	Wire Electrical Discharge Machining
PVD	Physical Vapor Deposition
COF	Coefficient of Friction
min	Minute
THA	Total Hip Arthroplasty

OCP	Open Circuit Potential
EIS	Electrochemical Impedance Spectroscopy
PD	Potentiodynamic Polarization
SHE	Standard Hydrogen Electrode
CPE	Constant Phase Element
CPS	Counts Per Second
EEC	Equivalent Electrical Circuit
FBS	Fetal Bovine Serum
PBS	Phosphate-buffered saline
CA	Cell Adhesion
CP	Cell Proliferation
DAPI	4',6-diamidino-2-phenylindole

Symbols

$K_2HPO_4 \cdot 6H_2O$	Di-potassium hydrogen phosphate hexahydrate
$MgCl_2 \cdot 6H_2O$	Magnesium dichloride hexahydrate
$(CH_2OH)_3 \cdot CNH_2$	Tris (Hydroxymethyl) amino methane
NaCl	Sodium Chloride
KCl	Potassium chloride
NaHCO₃	Sodium bicarbonate
Na₂SO₄	Sodium Sulfate
CaCl₂	Calcium chloride
Ta₂O₅	Tantalum oxide
R_q	Root Mean Square roughness
\bar{z}	Arithmetic average height
F_n	Normal Force
F_t	Tangential Force
mN	Micro Newton
μm	Micrometer
Ag/Ag. Cl	Silver Chloride
Pt	Platinum
W	Warburg Impedance
Hz	Hertz
β_a	Anodic Tafel Constant
β_c	Cathodic Tafel Constant
R_p	Polarization Resistance
E_{corr}	Corrosion Potential
i_{corr}	Corrosion Current Density
wt%	Weight Percentage
μL/s	Microliter Per Second

PREFACE

Austenitic stainless steel (Grade 316L) is one of the most widely used biomedical metals. Orthopedic implants, hip implants, plastic surgery devices, and cardiovascular stents represent some major applications. However, despite extensive research and clinical observation spanning decades, medical-grade 316L SS continues to face several unresolved issues that require attention and resolution. In this study, Tantalum (Ta) coating for 15, 30, and 60 minutes (**1.504 μm , 3.809 μm , and 6.083 μm thickness**, respectively) was applied on the surface of 316L stainless steel by DC Magnetron Sputtering, followed by evaluation of wear, corrosion and biocompatibility behavior. The lack of resistance to wear of the biomaterials and release of ions cause implant loosening, which leads to implant failure. As a result, surface modification of such biomaterials (316L SS) is required to ensure implant durability. To improve the wear resistance and durability of the implant, the Ta layer was deposited on 316L SS, and film quality can be enhanced by having excellent control of parameters during deposition. Metallic coating (Tantalum) was applied on the surface of stainless-steel type 316L by DC Magnetron Sputtering, followed by wear behavior and microhardness investigations. Wear studies through pin-on-disc tribometer are used to evaluate tribological properties, i.e., wear rate and friction coefficient behavior following ASTM G99-95a and ASTM G133-95. Wear tests were conducted in Simulated Body Fluid (SBF) under various normal applied load conditions (**10N, 20N, and 40N**) and a sliding distance of 10m. Based on wear test results, the wear rate (**62.50 $\times 10^{-5}$ mm³/Nm**) was found to be very high for bare 316L SS at an applied load of 40N, and the wear rate (**3.75 $\times 10^{-5}$ mm³/Nm**) was found to be low for Ta-coated (60min.) 316L SS at the same load. Wear-resistant and surface hardness of Ta-coated 316L SS were found to increase

with an increase in the thickness of the coating.

Ta-coated was found to be a higher hardness and lower modulus of elasticity against bare 316L SS. Electrochemical behaviors were investigated by potentiodynamic and electrochemical impedance spectroscopy techniques. Simulated body fluid (SBF) was used as the corrosive medium for the electrochemical measurement. The highest corrosion resistance was obtained for the sample subjected to 60 min. of Ta deposited on 316L SS according to EIS measurements, and the lowest corrosion rate of **0.0047 mm/year** was obtained for the same sample according to the PD measurements. All the measurements were carried out after determining open circuit potential (OCP) at $37^{\circ}\text{C} \pm 1^{\circ}\text{C}$. Ta_{4f} is present as oxidation states in Ta-coated stainless steel 316L, whereas Ta_{4f} is the main species examined along with other elements (C1s, N1s, O1s, Cr2p, Fe2p, Ni2p, and Mo3d). It was also observed that there was strong adhesion between substrate and Ta-coating. After the potentiodynamic studies, results show a significant improvement in corrosion resistance attributed to the strong, stable oxide layer formation.

As 316L stainless steel is likely to fail soon after implantation due to inadequate biological reactions. To fix these issues, Ta coating was deposited on 316L SS to increase biocompatibility and hydrophilicity. Compared to bare 316L SS, Ta-coated for 60 min showed superior characteristics, with the application of **human osteoblast MG 63** cells in cell culture on specimens for **1, 7, and 14 days** of incubation period showed improved biocompatibility for Ta-coated 316L SS with higher cell adherence, significant cell spreading, proliferation, and cell differentiation. Despite the ability to create highly adhering coatings of both aspects, studies on adhesion using a scratch test revealed significantly **better adhesion strength** at coated surfaces. Studies on the surface morphology of Ta-coated and

bare 316L SS were characterized by Optical Microscopy (**OM**), Optical Emission Spectroscopy (**OES**), Scanning Electron Microscopy (**SEM**), Energy Dispersive X-ray Spectroscopy (**EDS**), and Inductive Coupled Plasma-Mass Spectroscopy (**ICP-MS**). Atomic Force Microscopy (**AFM**) was used to characterize the surface roughness, and X-ray Photoelectron Spectroscopy (**XPS**) was used to identify the chemical states of coated and uncoated 316L SS. Considering its enhanced performance in Wear, Corrosion, and Biocompatibility behavior, Ta-coating on 316L SS is therefore recommended as a preferable coating for improving long-term performance and a promising candidate for orthopedic applications.

The current thesis focuses on a systematic study to understand the effect of tantalum (Ta) coating on 316L stainless steel compared to conventionally used stainless steel. The thesis aims to improve the mechanical (Wear behavior), Corrosion, and biocompatibility properties. A brief overview of the chapters is as follows:

Chapter 1 is *Introduction*; this chapter introduces biomaterials used in orthopedic implants, including their types and the increasing focus on metallic implants with surface modification coatings. Addressing bone-related diseases and replacements reflects the commitment to advancing medical technology and improving patient outcomes in developed countries. The goal is to create materials that can provide long-lasting and effective solutions for implants in wear behavior, corrosion, and biocompatibility behavior for individuals with bone-related conditions, ultimately enhancing their quality of life.

Chapter 2 is the *Literature Survey*, which comprises criteria for an ideal bone implant, Current materials used in the implant, Implant Failure, Biological interactions on metallic biomaterial surfaces, Corrosion Assessment of Orthopedic Implants, Mechanical Behavior

of Orthopedic Implants, Biological Evaluation of different types of coated 316L for the orthopedic application, Need for the metallic coatings and significance of Tantalum (Ta).

Chapter 3 is *Experimental Details*; this chapter discusses the details of the current investigations, such as the preparation of the sample (316L Stainless Steel), Wear, Electrochemical Corrosion, and biocompatibility behavior. Various characterization techniques like Optical Microscopy (OM), Scanning Electron Microscopy (SEM), Electron Diffraction X-ray Spectroscopy (EDAX), Atomic Force Microscopy (AFM), Profilometry, Inductive Coupled Plasma-Mass Spectroscopy (ICP-MS), Contact Angle Measurement (CAM), X-ray Photoelectron Spectroscopy (XPS), and Open Circuit Potential (OCP), Electrochemical Impedance Spectroscopy (EIS), Potentiodynamic Polarization also briefly described.

Chapter 4 is about the *Wear behavior of Ta-coated 316L Stainless Steel*, investigating wear and microhardness studies to determine the candidate biomaterial for higher durability without internal implant failure and effective performance (especially Ta-coated 316L SS).

Chapter 5 is the *Electrochemical Corrosion behavior of Ta-coated 316L Stainless Steel*; to check the corrosion behavior in simulated body fluid at 37⁰C of Ta-coated against bare 316L stainless steel.

Chapter 6 is *Biocompatibility behavior of Ta-coated 316L Stainless Steel*; to identify the Biocompatibility (cell adhesion and cell Proliferation), Wettability, and Scratch behavior of Ta-coated 316L Stainless Steel by DC Magnetron Sputtering for orthopedic applications.

Chapter 7 is *Summary and Major Conclusions* summarize the overall summary and the key findings.

Suggestions and Scope for Future work, also discussed after the last chapter, provide

valuable insights for future research. **The references section** lists the citations from Chapters 1-6 of the thesis.

The present study is reported in several publications, International conferences and one book chapter at the end of the thesis.