UNIVERSITI TEKNOLOGI MARA

DEVELOPMENT OF AISI 316LVM AUSTENITIC STAINLESS STEEL HYBRID S PHASE LAYER FOR MEDICAL APPLICATION

MOHAMMAD FIRDAUS BIN MOHAMMED AZMI

Thesis submitted in fulfillment
of the requirements for the degree of
Doctor of Philosophy
(Mechanical Engineering)

College of Engineering

April 2023

ABSTRACT

Attempts to improve the surface hardness and wear resistance of austenitic stainless steels using surface treatments in the past have resulted in corrosion resistance degradation due to chromium precipitation in the hardened layer. In this study, systematic gas diffusion thermochemical treatments and characterisation were performed on medical grade austenitic stainless steel AISI 316LVM (Sandvik Bioline) in order to establish the optimised treatment conditions (temperature, time and gas composition) which can maximise the austenitic stainless steel performance without sacrificing corrosion resistance. The hybrid S phase layer was systematically characterized by microscopy investigation, microhardness, phase analysis, potentiodynamics, pin on disk test, nanoindentation and nanoscratch according to each testing standards. According to the DOE optimisation results, the ideal treatment parameters for the low temperature hybrid heat treatment were when the temperature was set at 475 °C for 12 hours of holding time. The optimum gas composition was when methane, ammonia and nitrogen simultaneously introduced at 10 %, 80 % and 10 % respectively. The nitrogen and carbon element dissolved in the austenitic lattice forming an interstitial supersaturated solid solution called the hybrid S phase layer. From the microscopy result, up to 13.3 μm hybrid S phase layer thickness was developed. The characterization results found the hybrid S phase layer able to significantly increase the bulk material surface hardness up to 1461 $HV_{0.025}$, the wear resistance where about three times improvement and the corrosion resistance compared to the untreated material. The nano-tribological behaviour of the hybrid S phase layer shows improved the wear resistance coefficient and decreased the coefficient of friction. In addition, it has good cohesion where no evidence of delamination when the samples are nanoscratched vertically up to 50 mN and it is a noteworthy discovery for the biological field. The biocompatibility studies on the hybrid S phase found that they are biocompatible under the cytotoxicity (ISO 10993-5) and cell adhesion (ASTM F813-20) tests conducted. Therefore, the use of hybrid S phase as surface modification process on medical grade austenitic stainless steel might be suitable in biomedical applications.

ACKNOWLEDGEMENT

Alhamdulillah, thank you Allah for giving me this opportunity for my PhD journey. My deepest gratitude to my supervisors Associate Professor Ts. Dr. Juri bin Saedon and Dr. Shahriman Adenan from the School of Mechanical Engineering, College of Enginering, Universiti Teknologi MARA and Professor Dr Esa Haruman from Bakrie University for their invaluable supervision, guidance and encouragement.

My deepest appreciation to Mr. Mohd Azrol Bin Zulkefli, Mr. Mohd Rahimi Bin Abdul Rahman @ Mohd Salleh, Mr. Mahmud Mahat, Nor Azizi Bin Che Soh and Muhammad Suhairi Bin Zainuddin in School of Mechanical Engineering, College of Engineering laboratory for their assistance and help relating to the experimental work and workpiece preparation.

Special thanks to Madam Shirin Ibrahim, Researcher in Industrial Centre of Innovation in Biomedical, SIRIM Industrial Research in Kulim, Kedah for providing tools and facilities for biocompatibility testing as well as support and guidance in the experimental work. My gratitude to Dr Animesh Basak from Adelaide Microscopy Laboratory, Adelaide University for his kind assistance in nanoindentation and nanoscratch experiment.

I would like to thank the Ministry of Higher Education for their financial support throughout my study through “Hadiah Latihan Persekutuan” sponsorship program.

I would like to thank my family for their understanding and support for me to complete this research. Lastly, thanks to my colleagues and friends for their assistance and support.

TABLE OF CONTENTS

Page

CHAPTER ONE
INTRODUCTION

1.1 Research Background

Metallic materials are extensively used in biomedical applications as implants and medical instruments. The use of metallic material as a biomedical implant started in the 1890s when Sherman Vanadium Steel was introduced and to be used as a material for bone fracture plates and screws [1]. However, due to the insufficient strength and corrosion resistance of the Sherman Vanadium steel, 18-8 stainless steel was selected to be the replacement of the biomedical implant material. The selection of 18-8 stainless steel is due to its greater corrosion resistivity and high strength characteristics during that period. Hence, it attracted more clinician to employ stainless steel and become the main interest in biomedical device development for clinical use [2].

Commonly used metallic materials used as biomedical implant are 316L stainless steel, titanium and its alloys and cobalt based alloys [3]. These types of materials played a major role in biomedical applications due to their enhanced mechanical properties and good biocompatibility. The selection process of metallic material for implant rest on its medical application. Material to be used as implant materials must meet the prerequisite of non-toxicity, excellent mechanical properties, high corrosion resistance and biocompatible with living tissue. However, no metallic materials are exempted to corrosion in a radical solution condition in the human body. Thus, the selection of metallic material depends on the evaluation according to material toxicity level and its durability [1].

Stainless steel type 316L (316L SS) are most widely used for implant material due to its low cost compared to titanium and cobalt alloy [4]. 316L stainless steel also has acceptable biocompatibility and good mechanical properties. The use of 316L stainless steel as a temporary implant also was approved by the Food and Drug Association of the United States (US FDA). Medical grade stainless steel type 316L are employed in orthopedic implants including application of intramedullary nail, total hip replacements and bone fracture plates and screws [5].

Although, the chromium element in the stainless steels results in the evolution of thin, chemically stable and passive oxide film (Cr_2O_3) upon the surface, however, the