

UNIVERSITI TEKNOLOGI MARA

**S-PHASE LAYER BY HYBRID LOW
TEMPERATURE
THERMOCHEMICAL TREATMENT
PROCESS ON 316 LVM STAINLESS
STEEL: CORROSION AND
BIOCOMPATIBILITY TEST**

SITI UMRAH BINTI ZAINAL

Thesis submitted in fulfillment
of the requirements for the degree of
Master of Science
(Mechanical Engineering)

College of Engineering

March 2022

ABSTRACT

The 316 LVM stainless steel has been used in biomedical applications. The present study focuses on the improvement of surface properties of medical grade austenitic stainless steel (ASTM F138). The aim is to develop a homogenous supersaturated hard layer of expanded austenite (S-phase) at the surface of the AISI 316LVM using a low-temperature hybrid thermochemical heat treatment process in a customised mixing chamber of a tube furnace. Hybrid means treating both nitriding and carburizing at the same time. The S-phase layer would improve the surface integrity at the surface by increasing the hardness and improving the wear resistance without impairing the corrosion resistance of the steel. During the thermochemical process, ammonia (NH_3) and methane (CH_4) gases were introduced into the furnace for 6 and 12 hours at 425 °C, 450 °C, 475 °C, and 500 °C with a gas composition of 75% NH_3 , 5% CH_4 , and 20% nitrogen (N_2) and 75% NH_3 , 10% CH_4 , and 15% N_2 . The S-phase characterization was analysed by XRD. The thick bright layer of the S-phase formation was measured at up to 15.9 μm as analysed by SEM. Besides, the surface hardness also significantly increased from 210.9 HV to 1121.0 HV. These results have significant effectiveness in corrosion resistance at 475°C. The $E_{(\text{corr})}$ increased from a value of 364 mV (untreated) to -217 mV. Biocompatibility of treated samples shows enhancement of osteoblast cell proliferation on the S-phase surface, implying the low temperature hybrid thermochemical treatment could improve up to 8 % of cell viability after 24 hours. Finally, the low-temperature hybrid heat treatment process can improve the surface integrity of 316 LVM for use in biomedical implants.

ACKNOWLEDGEMENT

Firstly, I wish to thank God for giving me the opportunity to embark on my Master and for completing this long and challenging journey successfully. My gratitude and thanks go to my supervisor Dr Mohd Shahrizan b Adenan and Assoc Prof Dr Esa Haruman. Also a very special appreciation to Ministry of Education with grant number FRGS/1/2016/TK05/UITM/02/4.

My appreciation goes to the lecturer assistants who provided the facilities and assistance during sampling, En Azrol zulkefli, En Rahimi, En Mahmud and En Suhairil. Special thanks to my colleagues and friends for helping me with this project especially Pn Siti Humairah, Pn Fatin Athirah and a special assistance, Mr. Yuki Hamada from Ube College, Japan.

Finally, this thesis is dedicated to my husband, En Fuad b Abu Bakar, my mother, Pn Sadiyah bt Sheikh Mohd as well as to the loving memory of my very dear late father, Allahyarham Zainal b Md Ali, for the vision and determination to educate me. This piece of victory is dedicated to both of you. Alhamdulillah.

TABLE OF CONTENTS

	Page
CONFIRMATION BY PANEL OF EXAMINERS	ii
AUTHOR'S DECLARATION	iii
ABSTRACT	ivv
ACKNOWLEDGEMENT	v
TABLE OF CONTENTS	vi
LIST OF TABLES	x
LIST OF FIGURES	xi
LIST OF SYMBOLS	xvi
LIST OF ABBREVIATIONS	xvii
LIST OF NOMENCLATURE	xx
CHAPTER ONE: INTRODUCTION	1
1.1 Research Background	1
1.2 Problem Statement	2
1.3 Research Question	3
1.4 Objectives	3
1.5 Scope and Limitation of Study	4
1.6 Significance of Study	4
1.7 Thesis Outlet	5
1.7.1 Introduction	5
1.7.2 Literature Review	5
1.7.3 Eperimental Procedure	5
1.7.4 Result and Discussion	5
1.7.5 Conclusion	6

CHAPTER ONE

INTRODUCTION

1.1 Research Background

316 LVM Austenitic Stainless Steel (ASS) used as bio implant material began in the 1920's because of its good corrosion resistance compared with other alloy materials [1]. ASS was discovered after chemical modification was established to optimise corrosion resistance and provide a free ferrite microstructure. 316 LVM is produced by re-melting vacuum arc,(VAR), whereas secondary melting is produced in a premium step to improve cleanliness. As a result, the alloy became well known, as stated in ASTM F138, which is a medical-grade forging and machining applications for orthopaedic and surgical instruments [2] [3].As the alloy is fitted into the orthopaedic application, a surface modification is needed to develop surface strength [4]. Otherwise, without a surface modification treatment, it will probably cause damage due to the cycle of impacts and then be harmful to the body.

Another alloy, such as titanium and chromium cobalt, is also used as a biomedical material [5]. These alloys are reasonably defined as having better corrosion resistance than 316 LVM, making them best suited for implantation in the body. A possible explanation for the superior performance of these alloys for biomedical applications is the massive chromium elements that make it possible to react quickly with oxygen to form a passive nano-coating chromium oxide, which is very hard and durable, and titanium is very reactive with oxygen to form instantaneously adhesive oxides that can prevent corrosion. Even though 316 LVM ASS has good corrosion resistance, unfortunately, the surface integrity is inadequate for comparison to titanium and chromium cobalt. Thus, the surface of 316 LVM has developed by employing several methods of surface engineering techniques to improve the corrosion resistance, durability, and tribological performance. Mostly, the common methods used in surface engineering processes are conducted by plasma technology [6], physical vapour deposition (PVD) [7], chemical vapour deposition (CVD) [8], salt bath [9], and conventional methods such as fluidized bed furnace and tube furnace [10] [11]. Untreated 316 LVM typically has a low regeneration rate to form a protection layer due to its low chromium content compared to cobalt-chromium. Because of that, the