

**THE ROLLING EFFECT IN MECHANICAL PROPERTIES OF 316L
STAINLESS STEEL FOR IMPLANT MATERIALS**

LIE KEN NIE

UNIVERSITI TEKNIKAL MALAYSIA MELAKA

SUPERVISOR DECLARATION

“I hereby declare that I have read this thesis and in my opinion this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering (Structure & Materials).”

Signature:

Supervisor: DR. HADY EFENDY

Date:

**THE ROLLING EFFECT IN MECHANICAL PROPERTIES OF 316L
STAINLESS STEEL FOR IMPLANT MATERIALS**

LIE KEN NIE

**This report is submitted in fulfillment of the requirements for the award
Bachelor of Mechanical Engineering (Structure & Materials)**

**Fakulti Kejuruteraan Mekanikal
Universiti Teknikal Malaysia Melaka**

JUNE 2013

DECLARATION

“i hereby declare that the work in this report is my own except for summaries and quotations which have been duly acknowledged.”

Signature:

Author: LIE KEN NIE

Date:

This thesis is dedicated especially to my loving parents who have supported me throughout the course of this project. They have constantly given me moral support and encouragement which has given me the strength and motivation to complete this project. I also dedicate this project to friends and family whom have advised and taught me that all things should be done with perseverance and persistence until the target is achieved. I would like to thank them again for their love and never ending support that they have given to me in order to achieve my goals.

ACKNOWLEDGEMENT

First of all, I would like to sincerely thank my supervisor, Dr. Hady Efendy for giving me the opportunity to be under his supervision. I would also like to express appreciation for his precious time spent in guiding and assisting me throughout the entire final year project. Besides that, all the encouragement and advises that were given to me whether technically or morally really enabled me to understand and finish this final year project according to schedule.

Secondly I would also like to acknowledge and thank Mr. Wan Mohd Farid Bin Wan Mohamad for his assistance and advice in understanding the objective of my project. I would also like to acknowledge the panel for this project, Mr. Mohd Basri bin Ali and Dr. Mohd Ahadlin bin Mohd Daud for their comments and willingness to provide their feedback regarding this project. Their comments and feedback have enabled me to improve in areas in which were lacking any significant elements. A special thanks also to the management staff of the laboratory, especially the technicians involved for their cooperation and assistance whenever an experiment was conducted in the laboratory.

Finally, I would like to thank my friends and family for all their love and support throughout the entire final year project. I wouldn't have able to complete this project without their support and encouragement.

ABSTRAK

Keluli tahan karat austenit gred 316L adalah bahan yang paling popular digunakan sebagai bahan *implant* oleh kerana tingkah lakunya yang tahan karat, kebolehbentukan yang baik dan keserasian biology besi. Bagaimanapun, bawah proses permesinan, sifat-sifat mekanikal keluli tahan karat 316L ini mungkin terjejas. Di dalam projek ini, sifat-sifat mekanikal keluli tahan karat 316L itu akan dianalisis melalui pelbagai ujian. Sembilan spesimen telah disediakan dengan 8 spesimen yang akan melalui proses gelek sejuk dengan peratusan 0 hingga 70% *cold work*. Kesan daripada proses gelek sejuk ke atas keluli tahan karat 316L akan dianalisis untuk kesemua Sembilan spesimen tersebut. Ujian *U-bend* telah dijalankan untuk memperolehi data *compressive stress* dan *compressive strain* untuk setiap spesimen. Kekerasan setiap spesimen ditentukan melalui ujian mikrokekerasan pada bahagian atas, tepi dan sebelah bawah spesimen. Pencirian dan analisis mikrostruktur keluli tahan karat austenit 316L sebelum dan selepas rawatan *cold roll* telah dijalankan. Ujian *U-bend* menunjukkan bahawa dengan peningkatan dalam peratusan pengurangan ketebalan untuk 0 hingga 23 peratus, *compressive strength* juga bertambah dan adalah tertinggi untuk spesimen 23 peratus pengurangan ketebalan. Ini diikuti oleh trend penurunan untuk 30 hingga 70 peratus pengurangan ketebalan. Daripada ujian mikrokekerasan, nilai kekerasan untuk permukaan atas spesimen *U-bend* adalah lebih tinggi berbanding dengan permukaan bawah spesimen. Melalui pemerhatian mikroskopik, boleh diperhatikan bahawa deformasi sejuk menyebabkan perubahan dalam mikrostruktur seperti pemanjangan dan mampatan *grains* yang membawa kepada pengerasan permukaan. Pengubahsuaian mikrostruktur ini menyumbang kepada peningkatan dalam sifat mekanikal yang megesahkan aplikasinya dalam bidang perubatan terutamanya sebagai bahan implan.

ABSTRACT

Austenitic stainless steel grade 316L is a popular material that is being used as implant materials due to their corrosion resistance behaviour, good formability and biocompatibility of metals. However, under machining processes, the mechanical properties of stainless steel 316L may be affected. In this project, the mechanical properties of stainless steel grade 316L were analysed through various tests. Nine specimens were prepared with 8 of the specimens undergoing the cold roll process with 0 to 70% cold work percentage. The effect of cold rolling process on stainless steel grade 316L was analysed for all nine specimens. The U-bend test was conducted to obtain the compressive stress and compressive strain data of each specimen. The hardness of each specimen was determined by conducting the microhardness test on top, side and bottom area of the specimen. The microstructure characterization and analysis of the austenitic stainless steel 316L before and after cold roll treatment was conducted. The U-bend test showed that with increasing percent reduction in thickness for 0 to 23 percent, the compressive strength increases and is highest for the 23 percent reduction in thickness specimen. This is then followed by a decreasing trend for the 30 to 70 percent reduction in thickness specimens. From the microhardness test, the hardness values of the top surface of the U-bend specimens are higher compared to the bottom surface of the specimens. Through the microscopic observation, it was observed that cold deformation causes changes in microstructure such as elongation and compression of grains which lead to hardening of the surface. This microstructural modification contributes to the increase in the mechanical properties which justifies its application in the medical field as implant materials.

TABLE OF CONTENTS

CHAPTER	CONTENT	PAGE
	DECLARATION	ii
	DEDICATION	iii
	ACKNOWLEDGEMENT	iv
	ABSTRAK	v
	ABSTRACT	vi
	TABLE OF CONTENT	vii
	LIST OF TABLES	x
	LIST OF FIGURES	xi
	LIST OF SYMBOLS	xiii
	LIST OF APPENDICES	xiv
CHAPTER 1	INTRODUCTION	1
	1.0 Introduction	1
	1.1 Objective	5
	1.2 Scope	6
	1.3 Problem Statement	6
CHAPTER 2	LITERATURE REVIEW	7
	2.0 Stainless Steels As Implant Materials	7
	2.1 Rolling Process	10
	2.1.1 Introduction	10
	2.1.2 Mechanism of Rolling	11

	2.1.3	Type of Rolling Mill Used	12
	2.1.4	Cold Rolling	13
	2.2	Effects of Cold Rolling on the Microstructure and Mechanical Properties Of AISI 301LN Metastable Austenitic Stainless Steels	15
	2.3	Cold Deformation Effect on the 301LN and Microstructures and Mechanical Properties of AISI 316L Stainless Steels	16
	2.4	Characteristics of Mechanical Properties and Microstructure for 316L Austenitic Stainless Steels	17
CHAPTER 3		METHODOLOGY	18
	3.0	Introduction	18
	3.1	Gathering Information	19
	3.2	Problem Identification	20
	3.3	Preparation Of Materials	20
	3.3.1	Stainless Steel 316L	21
	3.4	Rolling Process	21
	3.5	U-bend Test	22
	3.5.1	Specimen Preparation	22
	3.5.2	U-bent Testing	24
	3.6	Microhardness Test	26
	3.6.1	Specimen Preparation	26
	3.6.2	Microhardness Testing	27
	3.7	Microscopic Observation	29
	3.7.1	Metallographic Specimen Preparation	29
	3.7.2	Microstructural Characterization	30
	3.8	Analysis Of Data	30
CHAPTER 4		RESULTS	31
	4.1	U-bend Test Results	31
	4.2	Microhardness Test	35

	4.3	Microscopic Observation	37
CHAPTER 5		DISCUSSION	45
	5.1	Effect of U-Bend on The Compressive Stress and Compressive Strain of Stainless Steel 316L	45
	5.2	Effect Of Cold Work on The Maximum Compressive Load and Maximum Compressive Stress of Stainless Steel 316L	46
	5.3	Comparison Between Tensile Test and U-Bend Compression Test for Stainless Steel 316L	47
	5.4	Effect of Bending on The Microhardness of Stainless Steel 316L	48
	5.5	Effect of Bending on The Microstructure of Stainless Steel 316L	49
CHAPTER 6		CONCLUSION	52
		REFERENCES	54
		BIBLIOGRAPHY	58
		APPENDIX	60

LIST OF TABLES

TABLE	TITLE	PAGE
1.1	Comparative Properties of the Stainless Steel Alloy Families	4
1.2	Composition of 316L Stainless Steel	5
2.1	Functional Attributes of Materials in Medical Devices	9
3.1	Typical U-bend Specimen Dimension	23
4.1	Table of Maximum Compressive Load and Maximum Compressive Stress	33
4.2	Microhardness of Top and Bottom Surface of U-bend Specimen	35

LIST OF FIGURES

FIGURE	TITLE	PAGE
2.1	Rolling Process	10
2.2	Rolling Mechanism	11
2.3	Two-high Mill	12
2.4	Effect of Cold Working and Hot Working on the Microstructure of Metals	13
3.1	Flow Chart of Methodology	19
3.2	Dimensions of Specimen Used in U-bend Test	22
3.3	Method of Stressing U-Bend Specimens-Single-Stage Pressing	24
3.4	Position of Reading Taken on Specimen: (a) Top Surface and (b) Bottom Surface	28
3.5	Mitutoyo Hardness Testing Machine	28
4.1	Graph of Compressive Stress against Compressive Strain	32
4.2	Graph of Average Maximum Force against Percent Reduction in Thickness	34
4.3	Graph of Average Maximum Stress against Percent Reduction in Thickness	34
4.4	Graph of Microhardness against Percent Reduction in Thickness	36

- 4.5 Microscopic Observation of Top Surface with Magnification of 200x 37-38
for U-bend Specimens with Thickness: (a) 2.0mm (b) 1.8mm (c)
1.6mm (d) 1.55mm (e) 1.4mm (f) 1.2mm (g) 1.0mm (h) 0.8mm (i)
0.6mm
- 4.6 Microscopic Observation of Top Surface with Magnification of 500x 39-40
for U-bend Specimens with Thickness: (a) 2.0mm (b) 1.8mm (c)
1.6mm (d) 1.55mm (e) 1.4mm (f) 1.2mm (g) 1.0mm (h) 0.8mm (i)
0.6mm
- 4.7 Microscopic Observation of Bottom Surface with Magnification of 41-42
50x for U-bend Specimens with Thickness: (a) 2.0mm (b) 1.8mm (c)
1.6mm (d) 1.55mm (e) 1.4mm (f) 1.2mm (g) 1.0mm (h) 0.8mm (i)
0.6mm
- 4.8 Microscopic Observation of Bottom Surface with Magnification of 42-44
100x for U-bend Specimens with Thickness: (a) 2.0mm (b) 1.8mm
(c) 1.6mm (d) 1.55mm (e) 1.4mm (f) 1.2mm (g) 1.0mm (h) 0.8mm
(i) 0.6mm
- 5.1 Microscopic Observation with Magnification of 1394.9x and 50-51
1407.4x for Cold-rolled Specimens with Thickness: (a) 2.0mm (b)
1.8mm (c) 1.6mm (d) 1.55mm (e) 1.4mm (f) 1.0mm (g) 0.8mm (h)
0.6mm

LIST OF SYMBOLS

w.t	-	Weight total solution
R _c	-	Rockwell hardness scale
α	-	Angle of contact
I	-	Moment of inersia
Δl	-	Absolute elongation
%CW	-	Percentage cold work
A ₀	-	Original cross sectional area
A _d	-	Area after deformation
γ	-	Gamma phase
L	-	Length
M	-	Distance between two holes on U-bend test specimen
W	-	Width
T	-	Thickness
X	-	Distance from centre of curvature to edge of specimen
Y	-	Distance between edges of specimen after U-bent test
R	-	Radius of bend curvature
β	-	Angle between edge and base of specimen

LIST OF APPENDICES

APPENDIX	TITLE	PAGE
A	Gantt Chart	60
B	Universal Testing Machine Instron	61
C	Grinding and Polishing Machine	61
D	Linear Precision Cutting Machine	62
E	Microhardness Indentation Sample	62
F	U-bend Specimen	62

CHAPTER 1

INTRODUCTION

1.0 INTRODUCTION

Stainless steels were invented by Krupp Stahl in 1912 (Richard J. Choulet, 1997). Stainless steels are iron alloys in which small quantities of carbon are added to its composition. Besides that, stainless steel contains about 10.5% chromium that form thin, transparent and durable chrome protective passive oxide layers that contributes to its corrosion resistance characteristic. The corrosion resistance of steel and the stability of the passive layer increases with increasing chrome content in the alloy. This protective oxide film is self-healing in which regardless of how much of the surface is removed; the steel is still corrosion resistant. In contrast to cases where carbon or low alloy steels are protected from corrosion by metallic coatings such as zinc or cadmium or by organic coatings such as paint (Technical Handbook of Stainless Steels, 2008). The corrosion resistance of stainless steels is also dependant on the metallurgical and processing variables. Although stainless steel maybe costlier than other metals with similar mechanical characteristics yet it is commonly used due to the main validation of its improved corrosion resistance. The presence of chromium (16-28%wt.) and nickel (3.5-32%wt.) near to the small contents of the carbon (usually below 0.1%wt.) assures a stable austenitic structure in the whole range of the temperature (from the temperature of solidus to the room temperature) (W. Ozgowicz & A. Kurc, 2009).

Stainless steels can be mainly categorized into five distinct groups; austenitic, ferritic, martensitic, duplex and precipitation hardening. Austenitic stainless steels are the most common and familiar types of stainless steel (Michael F. McGuire, 2008). Austenitic stainless steels have many advantages such as the ease of its formability in which they can be made soft enough to be easily formed using similar tools that work with carbon steel, however they can also be made incredibly strong as they increase in hardness after going through cold working. At elevated temperatures, austenitic stainless steels do not lose their strength as rapidly as ferritic iron based alloys. The least corrosion-resistant versions of austenitic steels are able to resist the normal corrosive attack of the daily environment experienced by humans, while the most corrosion-resistant grades is able to withstand boiling seawater. While having so many advantages, austenitic steels do have some disadvantages as well. Austenitic stainless steels are less resistant to cyclic oxidation compared to ferritic grades due to their greater thermal expansion coefficient that has the tendency to cause the protective oxide coating to spall. Other than that, they may experience stress corrosion cracking if used under an environment to which they have insufficient corrosion resistance.

Ferritic stainless steels can be considered the simplest and lowest cost grade of stainless steels. They contain sufficient amounts of chromium to overcome their inherent level of carbon impurity and hit the minimum 11% of chromium that gives it the stainless characteristic. The mechanical properties of ferritic stainless steels appear similar to austenitic steels in terms of strength, yet they lack the ductility of austenitic steels and are limited at lower temperatures by brittleness and softness at high temperatures. The corrosion resistance of ferritic stainless steels are hindered by their inability to utilize nitrogen. Ferritic stainless steels are essentially free from stress-corrosion cracking as they are below the threshold hardness for hydrogen embrittlement in body-centred cubic ferrous alloys.

Duplex stainless steels are the newest and fastest-growing alloy grade of stainless steels. They are called duplex because they consist of ferrite and austenite phases at room temperature. Duplex stainless steels are exceptionally strong, have excellent toughness and corrosion resistance. Besides that, they also exhibit exceptional resistance to stress-corrosion cracking and corrosion fatigue. Their limitations are in their lack of cryogenic toughness and their inability to withstand

temperatures above 300°C without forming embrittling phases. However, between temperatures of -100 and 300°C they are exceptional materials.

Martensitic stainless steels are the most marginally corrosion resistant among all the grades of stainless steel alloys. The requirement that they can be fully austenizable limits the amount of corrosion-resisting chromium and molybdenum that they can contain. Martensitic stainless steels are always susceptible to stress-corrosion cracking when their hardness exceeds Rockwell hardness of R_c 22. These limitations combine to make their excellent properties usable in only mild environments compared to other stainless steels. Their high strength and hardness for their relatively low cost ensures that they are very useful engineering materials.

The precipitation-hardenable grades of stainless steels are a highly specialized family of stainless steels. The existence of this grade of stainless steel comes from the need for a very high-strength material with good corrosion resistance. The precipitation-hardenable stainless steels exploit the low austenite stability possible in the chromium and nickel stainless steels by making the alloys as lean as possible in composition that they are able to transform almost completely to martensite by thermal or mechanical treatment. The advantage that the precipitation-hardenable stainless steels have over the strictly martensitic stainless steels is that they attain great strength with higher toughness and corrosion resistance than can be obtained through the hardening of martensite through carbon addition. In addition to that, they can be fabricated in a relatively soft state and then hardened with very little changes in dimension.

Martensitic and ferritic steels have magnetic characteristics. Martensitic steels have low formability and are usually hardened by heat treatment. Duplex grades stainless steels are tougher than ferritic grade steels and are more resistant to stress corrosion cracking than austenitic steels. Austenitic steels have a face-centered cubic crystal structure which is very tough and ductile whereas martensitic steels crystallize in body-centered cubic structure at low carbon concentrations. Martensitic steels are harder and stronger compared to austenitic steels.

Table 1.1: Comparative Properties of the Stainless Steel Alloy Families

(Reprinted from the Technical Handbook of Stainless Steels, p.10, 2008)

Alloy Group	Magnetic Response (note 1)	Work Hardening Rate	Corrosion Resistance (note2)	Hardenable	Ductility	High Temperature Resistance	Low Temperature Resistance (note 3)	Weldability
Austenitic	Generally No	Very High	High	By Cold Work	Very High	Very High	Very High	Very High
Duplex	Yes	Medium	Very High	No	Medium	Low	Medium	High
Ferritic	Yes	Medium	Medium	No	Medium	High	Low	Low
Martensitic	Yes	Medium	Medium	Quench & Temper	Low	Low	Low	Low
Precipitation Hardening	Yes	Medium	Medium	Age Hardening	Medium	Low	Low	High

Stainless steel grade 316L is made up of a composition of different elements such as carbon, chromium, nickel, molybdenum, etc. Stainless steel grade 316L is a type of austenitic stainless steel which is widely used in many applications in the industry such as surgical and medical tools and implants, chemical and pharmaceutical industry, petroleum refining equipment and textile tubing. This is due to its excellent resistance to corrosion and phosphates, sulphates and other salts, good formability and welding ability. 316L stainless steels have high work hardening rate during deformation and is generally accepted that high work hardening rate is related to stacking fault energy which controls the ease of cross-slip, so that different deformation mechanisms can be activated at the different stages of deformation.