THE EFFECTS OF COLD DEFORMATION ON THE MICROSTRUCTURES AND HARDNESS PROPERTIES OF 316L STAINLESS-STEEL

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A report submitted
In fulfilment of the requirements for the degree of
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2022

DECLARATION

I hereby declare that this project report entitled ‘The Effects of Cold Deformation on the Microstructure and Hardness Properties of 316L Stainless Steel’ is the result of my own works except as cited in the references.

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Date	: 9 JANUARY 2022

APPROVAL

I hereby declare that I have read this project report. In my opinion, this report is sufficient in terms of scope and quality for the award of the degree of Bachelor of Mechanical Engineering (Automotive).

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Date	:	9 JANUARY 2022

DEDICATION

It is with my deepest gratitude and warmest affection that I dedicate this project report

To my beloved parents, family and to everyone who loves me endlessly.

ABSTRACT

This research discusses mainly on cold deformation impacts on the microstructural transformation and the hardness properties of the studied material which is 316L Stainless Steel. A total of five cold rolled samples were prepared accordingly with thickness reduction percentages increased from 0% to 40%. Microstructural modifications of the CR samples were examined under optical microscope image analyser at 10X magnification and the hardness test was carried out by utilising Vickers Hardness Tester equipped with a 0.5kg test load. From the metallurgical test, the results showed that as the thickness reduction percentage (%RT) increased, the content of martensite formation in 316L Stainless Steel also increased and thus, contributing to the enhancement of strength and reduction in ductility of the material. For the polished sample, the Vickers hardness at 0% RT is 165.9 and was increased to the maximum of 337.9 at 40% RT. As for the unpolished sample, the Vickers hardness at 0% RT is 176.2 and was increased to the maximum of 394.7 at 40% RT. In comparison, the micro-hardness reading of the samples (which was taken directly after cold rolling process) before polishing has slightly higher values than after polishing. From the results obtained, it is found that as the cold rolling reduction percentage increased, the content of martensite increased as well as the hardness properties of the studied material.

ABSTRAK

Kajian ini membincangkan tentang kesan-kesan kaedah 'ubah bentuk sejuk' (cold deformation) ke atas perubahan mikrostruktur dan sifat kekerasan bahan yang dikaji iaitu keluli tahan karat 316L. Sejumlah lima sampel telah disediakan secara tertib dengan peratusan pengurangan ketebalan yang ditingkatkan dari 0% sehingga 40%. Perubahan bentuk mikrostruktur telah dikaji dengan menggunakan mikroskop optikal penganalisa gambar dengan kanta 10X dan ujian kekerasan dilakukan dengan menggunapakai mesin ujian kekerasan Vickers dilengkapi dengan 0.5kg beban uji. Menurut apa yang diperolehi daripada ujian metalurgi, keputusan menunjukkan bahawa apabila peratusan pengurangan ketebalan meningkat, kandungan pembentukan martensit di dalam keluli tahan karat 316L juga telah meningkat. Nilai kekerasan Vicker bagi sampel belum digilap meningkat dari 165.9 (0% RT) hingga 337.9 (40% RT) manakala nilai kekerasan sampel yang telah digilap meningkat dari 176.2 (0% RT) hingga ke 394.7 (40% RT). Sebagai perbandingan, hasil bacaan kekerasan sampel (yang diambil secara langsung setelah proses penggolekan sejuk) sebelum digilap didapati mempunyai nilai bacaan yang lebih tinggi sedikit berbanding dengan yang selepas digilap. Berdasarkan keputusan diperolehi, kajian mendapati bahawa apabila peratusan pengurangan ketebalan meningkat, kandungan martensit bahan dan sifat kekerasan bahan juga meningkat.

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LIST OF ABBREVIATIONS

ASS - Austenitic Stainless Steel

ASTM - American Society for Testing and Materials

CR - Cold-Rolled

CNC - Computer Numerical Control

EBSD - Electron Backscattered Diffraction

EDS - Energy Dispersive X-Ray Spectroscopy

HV - Vickers Hardness

RT - Thickness Reduction

SEM - Scanning Electron Microscopy

SFE - Stacking Fault Energy

SS - Stainless Steels

WT - Weight

XRD - X-Ray Diffraction

CHAPTER 1

INTRODUCTION

1.1 Background of Study

Austenitic stainless steels (ASS) are broadly used in medical field, transportation, food, nuclear and the petrochemicals industry. This is due to the fact that austenitic stainless steels are well-known to have high corrosion resistance, high strain hardening, great plasticity and formability. On the other hand, austenitic stainless steel has relatively low yield strength. Thus, the applications of this material in some areas like automotive industry and structural engineering are very limited.

Mechanical properties of the material like the strength of the austenitic stainless steel can be significantly increased in several ways such as cold deformation strengthening, grain size strengthening, bake hardening strengthening and many more. Amongst all these methods, cold deformation strengthening is best believed as a practical way and fits the industrial applications because of the high strain hardening of austenitic stainless steel. It is also considered as the best technique for manufacturing of products that are sizeable and has the benefit of cost-effective (Xiao Li *et al.* 2019).

Cold working; for example cold rolling means a process where metal is strengthened by changing its shape without the presence of heat. The microstructures changes during cold rolling process of austenitic stainless steels may involve; the stacking fault occurrence, deformation bands, ε-martensite, deformation twinning and α'-martensite, the evident grain subdivision and the dislocation multiplication. (Xiao Li *et al.* 2019).

These features will add more complexity of dislocation motion and consequently will cause the steel to be strengthened. The microstructural modification from the austenitic into martensitic can increase the mechanical properties of cold-rolled austenitic stainless steels. Microstructural analysis exhibits that cold deformation causes major microstructural modifications in austenitic stainless steel, especially hardening. (Maria *et al.* 2011).

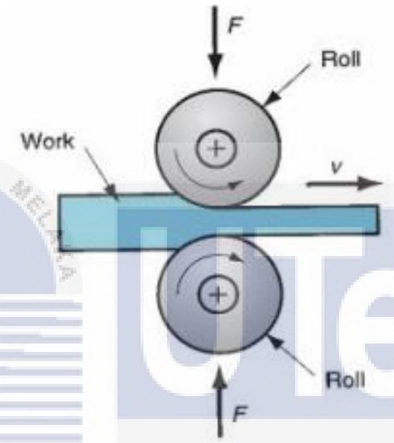

Figure 1.0: Cold rolling process.
(Source: Fundamental of Modern Manufacturing Materials, 4th Ed.)

Cold working has the advantage of producing a smooth surface finish on the product as well as improving the material's strength and hardness properties. Since no heating of the material is required in cold working, it saves money. This process does not only have superior dimension control it also provides better reproducibility and interchangeability of parts.

However, cold working requires a high amount of force to begin and end the work. It also requires high strength and precision of the equipment, large motor power, and high energy consumption.

Table 1.0: Stainless steel groups and their general characteristics.
(Souce: Unifiedalloys.com)

Alloy Group	Magnetic Response	Work Hardening Rate	Corrosion Resistance	Hardenable	Ductility	High Temperature Resistance	Low Temperature Resistance	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 Harden	Medium	Low	Low	High

The microstructure modifications of austenitic stainless steel during cold rolling process is significantly based on stacking fault energy (SFE). The phase alteration in austenitic stainless steels is restrained due to austenite phase stability which is dependent on the Md30 temperatures (temperature where half of martensite changed from austenite after one-third tensile deformation).

Commonly, austenite phase in austenitic stainless steels is an unstable phase where during the plastic deformation phase, martensite is transformed to strain-induced martensite. Martensitic phase normally shows a greater strength and less plasticity. Referring to Deming *et al.* (2018), some austenitic stainless steels for example 316L stainless steels have higher content of alloy elements.

For this project, effects of cold deformation process on the microstructure and hardness properties of material 316L stainless-steels is studied. An optical microscopy will be used to examine the microstructural deformation as the cold rolling reduction increased and Vickers Hardness test will be carried out to obtain the hardness properties. The relationship between microstructure and hardness of the cold deformed steel will be analysed.

1.2 Problem Statements

The hardness and the ultimate tensile strength values in 316L stainless steels are relatively low. Austenitic stainless-steel cannot be hardened with thermal treatment since alloy content does not allow structural transformation. Low yield strength limits the application of austenitic stainless-steel in automotive industry and structural engineering. Cold rolling process is expected to enhance the mechanical properties of 316L stainless steel for broader applications in industry.

1.3 Objectives

1. To examine the microstructure modifications of the cold-deformed steel.
2. To analyse the hardness properties of the cold-deformed steel.
3. To study the relation between microstructure and hardness of the cold deformed steel.

1.4 Scope

This project follows the standards according to ASTM International. The as-received thickness of the test specimen is 2mm at 0% RT and it is increased to a maximum of 40% RT at maximum temperature of room temperature. To examine the microstructures, an optical microscopy with 10X and 20X magnifications is used. To analyse the hardness, Vickers Micro hardness tester is utilised with a 4.905N as the total load force and 10s as the dwell time.

CHAPTER 2

LITERATURE REVIEW

2.1 Definition of Cold Working and Hot Working.

Cold working and hot working are two significant methods and widely used in metallurgy in order to obtain metal products of desired properties. The above processes get their names according to the operating temperatures where they are conducted. Each technique yields a product that is somewhat distinct from the other. Cold rolling and hot rolling are the examples of these two metal strengthening method.

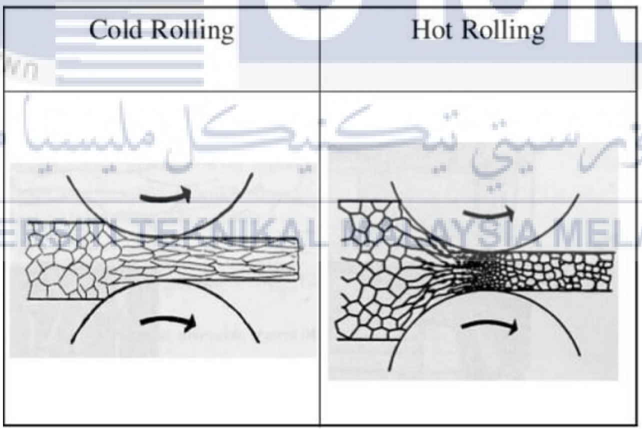

Figure 2.0: Cold rolling vs hot rolling.
(Source: LearnMech.com, 2022)

Cold working refers to a process of strengthening metals by plastic deformation which is done at temperatures below the recrystallization temperature of the metals. The majority of cold working processes are carried out at room temperature. Cold working works best with metals such as steel, copper, and also aluminium. Cold working reduces ductility.

Internal and residual stresses are generated in the metal during cold working (Madhu, 2018). The most popular technique of work hardening is known as cold rolling.

Hot working defines the process of permanent deformations of metals above their recrystallization temperature without causing strain hardening. Hot working is typically done at high temperatures. Hot working is can increase metal ductility and has the ability to reduce yield strength thus making it easier to work with metal. In hot working, the metal deforms and recovers at the same time. Residual stresses are not presented during the hot working process. (Madhu, 2018)

Recent writing on LearnMech.com (Sachin, 2022) described that hot rolling involves heating huge chunks of metal, such as slabs or steel billets, over their recrystallization temperature. An example for hot rolling is hot forging. The metal is subsequently compressed between rollers, resulting in thinner cross sections. The cross sections are thinner compared to all those made by the same number of stages of cold rolling techniques. Hot rolling additionally shrinks the average grain size of the metal while maintaining an equiaxed microstructure.

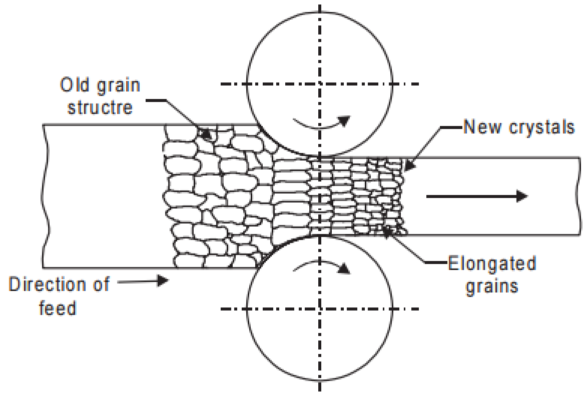

Figure 2.1: Grain transformation during hot forging.
(Source: Inspection-for-industry.com)

2.1.1 Cold Working: Cold Rolling Process

Cold working is a metal forming process in which the metal is deformed under its recrystallization temperature. In the vast majority of cases, cold working of metal is carried out at room temperature. Recrystallization temperature means the lowest temperature where plastically deformed metals creates new grains within the specified period. Majority of metals have a recrystallization temperature of 0.3 to 0.5 of their melting point. (López et al, 2012).

Cold rolling is a technique of metal forming in where metal is forced through a set of one or more rolling mills to achieve desired thickness or a decrease in thickness as the length increases. It is a hardening process that takes place below the metal's recrystallization temperature or, in most cases, at room temperature. After a metal has been hot rolled, it is usually cold rolled to produce nice finishing. It improves the stiffness and the tensile strength of the metal as well. (Kumar, 2021)

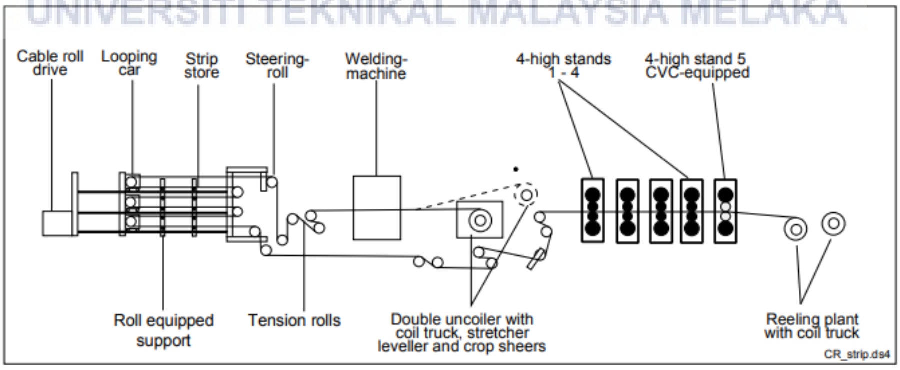

Figure 2.2: Diagrammatic view of a continuous cold rolling mill.
(Source: Jochum et al., 1999)

2.2 The Relationship between Cold Working and Deformation.

An example method of cold work is cold rolling where the process includes the metal being inserted in between a pair of rollers to reduce the thickness or to achieve uniform thickness of the metal. When it is passed through the rollers and is compressed, the metal grains will experience structural change known as deformation. As a metal goes through this process, permanent defects in the metal structure will appear and change their shape or crystalline makeup. These defects cause the reduction of the movement of the crystals within the metal. Hence, the metal becomes resistant to further deformation.

Discussion by Engineering Notes (Surupa, 2021) states that cold working provokes the distortions of crystal grains and inclusions in accordance with the metal flow. Slip is principally on primary glide planes in the initial phases of plastic deformation, and the dislocations will form coplanar arrays. Cross slip will occur as deformation develops. The cold-worked structure creates high dislocation density regions, which quickly develop into networks. At low deformation, the grain size will decrease with strain but quickly attains a fixed size.

2.2.1 Effect of Cold Deformation on the Formation of Austenite

At the present moment, the automotive industry is setting on a trend towards lightening vehicles in order to comply with the new regulations of exhaust emission control. This goal creates a large-scale challenge to steel manufactures all around the world. The trials and tribulations lies in manufacturing new materials (alloys) with the ideal strength, ductility and cost valuation for the multiple parts and components. (Goune *et al*., 2006).

Here, manipulation of the steel's microstructure on the smallest possible scale are likely to be the key to success. In this circumstances, the development of the inter-lamellar distance is an essential answer to the demand to weight reduction in the automotive industry. The cold deformation will affect both parameters: the inter-lamellar distance Dp and the spacing of pearlite bands Db. (Goune *et al*., 2006) A treatment allows for the consideration of the effect of deformation on Dp and Db. In point of fact, it is founded that:

$$Dp = Dp^{o} \exp\left(\frac{-\varepsilon p}{2}\right)$$

$$Db = Db^{o} \exp(-\varepsilon b)$$

Where εp and εf are the local deformations into pearlite and ferrite, respectively. The microstructures shown in Figures 2.3 demonstrate the effects of cold rolling on the pearlite band spacing as well as the inter-lamellar distance. As studied by Goune *et al.* (2006), the greater the cold reduction, the smaller the pearlite band spacing and inter-lamellar distance.

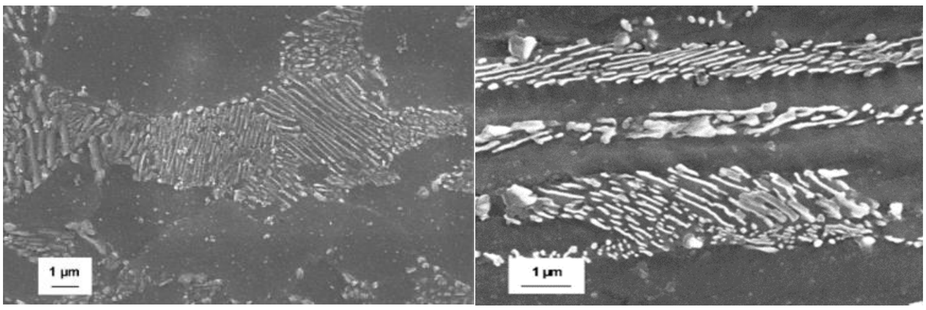

Figure 2.3: Initial microstructures for a cold reduction of 0% and 65%.
(Source: Revue de Métallurgie 103 (10), 2006.)

2.3 Residual Stress after Cold Rolling Process.

Cold-rolling processes are extensively applied in various manufacturing. Rolling-induced residual stresses have a major impact on the material's behaviour. Therefore, knowing this state is essential. The term residual stresses means the stresses that remain inside a structure once all forces applied have been eliminated. Tensile residual stress (positive) pulls the material away, whereas compressive residual stress (negative) pushes it together.

Residual stresses evolve when a material reaches equilibrium due to plastic deformation created by mechanical loads or thermal loads applied, as well as phase changes. Residual stresses form in metal and stay until eliminated by additional heat treatment. If residual stresses are eliminated by reheating well below crystallisation temperature, there is no noticeable change in the physical properties of the grain structure. Additional heating into the recrystallize range removes the influence of cold working and returns the metal to its original form.

Unwanted residual stress can cause stress corrosion cracking, distortions, and component premature failures. Heat treatment, controlled cooling, and localised heating are the approaches that can be used to minimize residual stresses that are possibly harmful. Even small deviations in residual stress can have a large impact on a component's life, hence this stress must be controlled to assure safety and to prevent failure. The initial residual-stress state effects on the final state after cold rolling can be examined by simulations of finite-element analysis (FEA) and X-ray diffraction (XRD).

2.4 Stainless Steel

Generally, stainless steel is one wide range of steel alloy with some chromium composition protecting the metal against corrosiveness and possible damage. The composition of chromium proportion in the alloy and the compositions of other metal elements, differs depending on the type of stainless steel used. Stainless steel is mainly made of iron, Fe and carbon just like steels. Somehow, stainless steel also consists of a minimum 10.5% chromium (Bergsen.com, 2021), a metallic element which provides the finish of the metal and superior resistance to corrosion.

Stainless steel is perfect for long-term applications facing elements, humidity and chemicals that are likely to be corrosive. The risk of contamination is much lower. Hence, its application is ideal for sensitive industries, like food preparation and pharmaceutical products. Stainless steels are frequently chosen for their corrosion resistance, but they are also used in construction. Strength, high-temperature resistance, ductility, and toughness are thus vital mechanical properties. The differentiation in the mechanical properties of different stainless steels can probably be seen clearly in the stress-train curves in Figure 2.4 below:

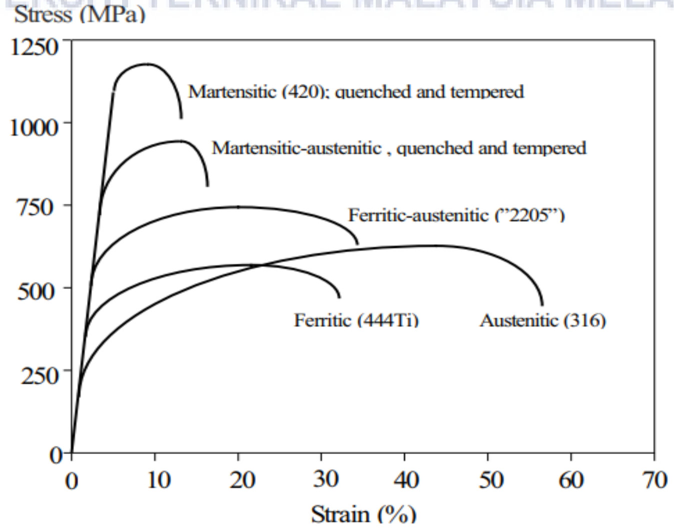

Figure 2.4: Stress-strain curves for some stainless steels.

(Source: www.outokumpu.com)

The high tensile strengths and yield strengths but low ductility of the martensitic steels is obvious, as is the low yield strength and excellent ductility of the austenitic grades. Ferritic-austenitic and ferritic steels both lie somewhere between these two extremes (Leffler, 2020). Stainless steel can be categorized in six groups which are martensitic, martensitic-austenitic, precipitation hardening, ferritic, ferritic-austenitic (duplex) and austenitic.

Table 2.0: Composition ranges for different stainless steel categories.
(Source: STAINLESS - Stainless Steels and Their Properties by Béla Leffler)

Steel category	Composition (wt%)					Hardenable	Ferro-magnetism
	C	Cr	Ni	Mo	Others		
Martensitic	›0.10 ›0.17	11-14 16-18	0-1 0-2	- 0-2	V	Hardenable	Magnetic
Martensitic-austenitic	‹0.10	12-18	4-6	1-2		Hardenable	Magnetic
Precipitation hardening		15-17 12-17	7-8 4-8	0-2 0-2	Al, Al,Cu,Ti,Nb	Hardenable	Magnetic
Ferritic	‹0.08 ‹0.25	12-19 24-28	0-5 -	‹5 -	Ti	Not hardenable	Magnetic
Ferritic-austenitic (duplex)	‹0.05	18-27	4-7	1-4	N, W	Not hardenable	Magnetic
Austenitic	‹0.08	16-30	8-35	0-7	N,Cu,Ti,Nb	Not hardenable	Non-magnetic

There are traces of different grades of stainless steel. The common grades are 304 and 316. Grade 304 stainless steel is widely applied in daily life such as appliances, cooking ware, tubing job and hardware's with a nickel value of up to 35% (Bergsen.com, 2021). Grade 316 SS has molybdenum, making it a perfect fit for marine equipment, chemicals and pharmaceuticals, manufacturing tools, and kitchen wares. It is thus ideal for external applications in marine life or any usage which are susceptible to chloride exposure.

The leading product form for stainless steels is cold rolled sheet. The other products individually form only one third or less of the total amount of cold rolled sheet. Table 2.1 shows how the use of stainless steel is divided between the various applications.

Table 2.1: Uses of stainless steel in the industry.
(Source: Leffler, 2020)

PRODUCT FORMS		APPLICATION CATEGORIES		
Cold rolled sheet	60 %	**Consumer items**		26 %
Bar and wire	20 %	Washing machines and dishwashers	8 %	
Hot rolled plate	10 %	Pans, cutlery, etc.	9 %	
Tube	6 %	Sinks and kitchen equipment	4 %	
Castings and other	4 %	Other	5 %	
		Industrial equipment		74 %
		Food industry and breweries	25 %	
		Chemical, oil and gas industry	20 %	
		Transport	8 %	
		Energy production	7 %	
		Pulp and paper, textile industry	6 %	
		Building and general construction	5 %	
		Other	5 %	

2.4.1 316L Stainless Steel

316 steel is available in many varieties where type 316L is included. 316 and 316L are austenitic alloys. It means that these types of stainless steel products get corrosion resistance from use of a nonmagnetic solid solution of ferric carbide or carbon in iron in the manufacturing process. The major difference between 316 and 316L stainless steel is the carbon content.

Grade 316 got the highest carbon content of 0.08%, while 316L contains the highest carbon content of just 0.03%. Both grade 316 and 316L stainless steel alloys are marine grade steels, somehow there are several significant differences between them. 316L stainless steel, which has two alloys with different characteristics, has less carbon and molybdenum than 316 steel.

For composition, 316L has a lower percentage of carbon. The carbon amount cannot be more than 0.03% if it is to be qualified as 316L stainless steel. This lowers the risk of carbon precipitation and improves welding option to ensure maximum resistance to corrosion. The specified properties of 316L stainless steel are shown in tables below. Note that these properties are specified for flat rolled product in ASTM A240/A240M. (Bergsen, 2021).

Table 2.2: Composition Specification (%).
(Source: Atlas Steel Grade Data Sheet, 2011)

Grade		C	Mn	Si	P	S	Cr	Mo	Ni	N
316	min.	-	-	-	-	-	16.0	2.00	10.0	-
	max.	0.08	2.0	0.75	0.045	0.030	18.0	3.00	14.0	0.10
316L	min.	-	-	-	-	-	16.0	2.00	10.0	-
	max.	0.030	2.0	0.75	0.045	0.030	18.0	3.00	14.0	0.10
316H	min.	0.04	-	-	-	-	16.0	2.00	10.0	-
	max.	0.10	2.0	0.75	0.045	0.030	18.0	3.00	14.0	-

Table 2.3: Mechanical Properties Specification.
(Source: Atlas Steel Grade Data Sheet, 2011)

Grade	Tensile Strength (MPa) min	Yield Strength 0.2% Proof (MPa) min	Elongation (% in 50mm) min	Hardness	
				Rockwell B (HR B) max	Brinell (HB) max
316	515	205	40	95	217
316L	485	170	40	95	217
316H	515	205	40	95	217
316H also has a requirement for a grain size of ASTM No 7 or coarser.					

Table 2.4: Physical Properties.
(Source: Atlas Steel Grade Data Sheet, 2011)

Grade	Density (kg/m^3)	Elastic Modulus (GPa)	Mean Coefficient of Thermal Expansion			Thermal Conductivity		Specific Heat	Electrical Resistivity
			0-100°C (µm/m/°C)	0-315°C (µm/m/°C)	0-538°C (µm/m/°C)	at 100°C (W/m.K)	at 500°C (W/m.K)	0-100°C (J/kg.K)	(nΩ.m)
316 & 316L/H	8000	193	15.9	16.2	17.5	16.3	21.5	500	740

2.5 Review on Previous Studies (Articles/Journals)

Based on online resources, there are numerous articles that carry out studies on the same field as this paper. Out of 50 articles and journals that have been read, some of them are found to have conducted the similar research which is to study on the effect of cold deformation on the microstructures and hardness properties of materials such as stainless steel and brass. As for this project, the material that is going to be used in the experiment is 316L austenitic stainless steel. Reviewing these articles will give better understanding for the concept and methodology involved in this project.

2.5.1 Article 1:

Effects of Cold Deformations on Microstructure and Mechanical Properties of Austenitic Stainless Steels (ASS).

Briefly, the type of cold deformation process done by this experiment is cold rolling, where it is done at room temperature. The results showed that the content of martensite increased as the cold rolling reduction increased.

In its methodology, for the microstructure observation, the volume fraction of martensite in cold-rolled samples are observed by X-Ray diffraction. The Vickers Hardness of test samples are obtained through measurement of up to twenty points in varies of regions of the samples with micro-hardness tester along with 0.5kg force load. As for the result, microstructures evolutions at 0%, 10%, 30%, 50% 70% and 90% cold-rolled test samples have been examined (Xu *et al*., 2018).

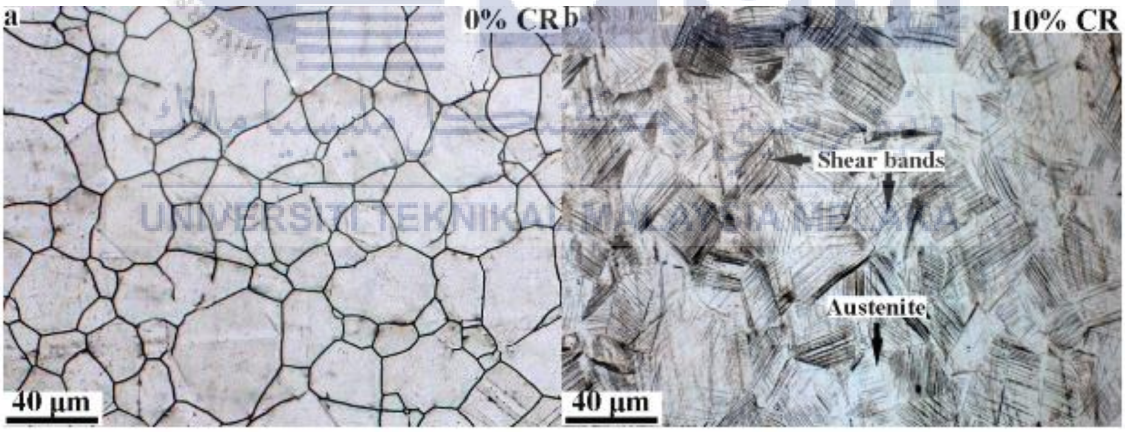

Figure 2.5: Microstructural graphic of initial and 10% CR test samples.
(Source: Xu et al., 2018)

From Figure 2.5, at 0% CR, coarse grained austenite were observed and at 10% CR, many shear bands were discovered. At 30% CR, some strain-induced martensite existed at the shear bands. When CR rised from 10% up to 30%, the structural size of austenite decrease from 7.30µm to 1.30µm. At 50% CR, untransformed austenite was elongated along rolling direction. At CR 70%, martensitic content increased and the martensite segmented the untransformed austenite. At 90% CR, huge blocks of untransformed austenite still exist.

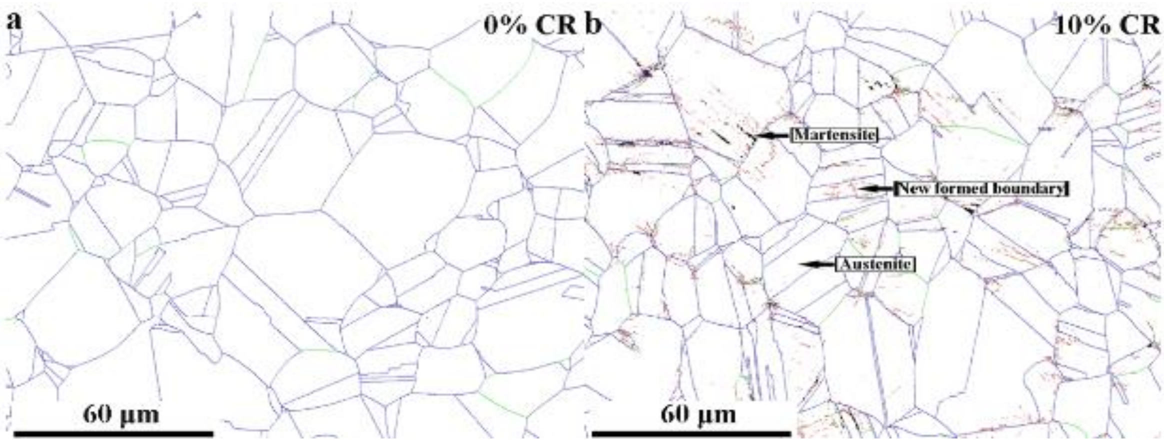

Figure 2.6: EBSD microstructural graphic grain boundary reconstructions map of austenite.
(Source: Xu et al., 2018)

The formed martensite becomes fine martensite mix with the fine austenitic structures. The original (0% CR) and 10% CR test samples are characterized using EBSD and are displayed in Figure 2.6 above. The major structure of the 0% CR test sample are coarse austenitic grain with many annealing twinning. A little amount of strain induced martensite with grains boundaries formed in austenite grains after 10% CR. (Li *et al.*, 2018).

To sum up the result of this journal, as the CR% increases, the austenite structure size decreases. Also, it was found that when the percent of CR increases, martensitic volume fraction increase too. Plus, the material hardness value, tensile and yield strength increase as well but the elongation decreases. (*Source: Metals - Open Access Metallurgy Journal 8(7):522.)*

2.5.2 Article 2:

Effects of Cold Rolling on Microstructure and Mechanical Properties of AISI 304N Stainless-steels.

This second article investigated the effect of cold rolling at room temperature on the microstructure and mechanical properties of a nitrogen-bearing austenitic stainless steel AISI 304N. Cold rolling reductions ranging from 5% to 90% were used in the cold rolling test. In its methodology, cold rolling was done at a room temperature where 6rpm is the speed of rolling. The optical microscopy was used to examine the microstructures, a ferrite scope was used to measure the volume fractions of strain induced '-martensitic, and X-ray diffraction was used to investigate deformation induced transformation. (Li *et al.*, 2019). The microstructures of the specimen are displayed as in sequenced figures below:

Figure 2.7: Optical micrograph of 0% CR AISI 304N specimen.
(Source: Li et al., 2019)

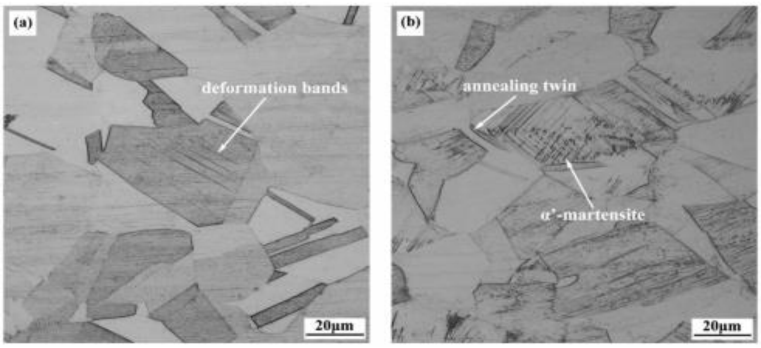

Figure 2.8: Optical micrograph of 5% and 20% CR AISI 304N specimen.
(Source: Li et al., 2019)

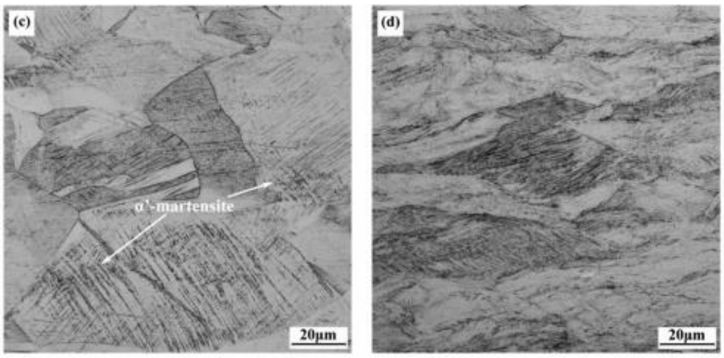

Figure 2.9: Optical micrograph of 30% and 50% CR AISI 304N specimen.
(Source: Li et al., 2019)

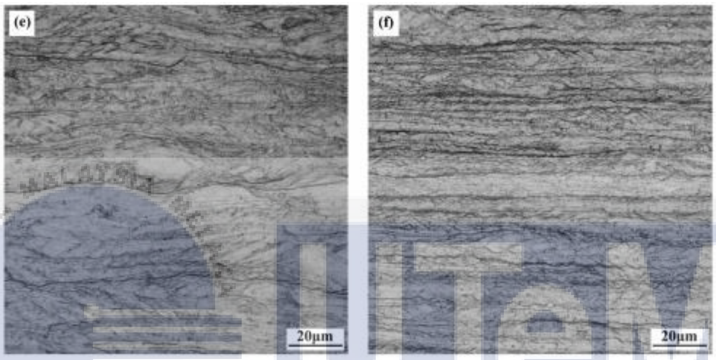

Figure 2.10: Optical micrograph of 70% and 90% CR AISI 304N specimen.
(Source: Li et al., 2019)

The phase of AISI 304N ASS transformed from the single austenite in the solution annealed state to the dual phase of austenite + α'-martensite with the increase of cold rolling reduction. (Li *et al.,* 2019) The overall yield strength for dual phase structure (austenite + α'-martensite) can be calculated using the following equation:

$$\sigma 0.2 = FA\sigma 0.2 \text{ (Austenites)} + FM\sigma 0.2 \text{ (Martensites)}$$

Meanwhile, as the cold rolling percent is increasing, the dislocation density of austenite and α'-martensite increases, grains sizes for both phase decrease. Hall-Petch-type relationship that has been modified can be utilised to evaluate the yields strength for the phases of austenite or martensite. (Li *et al.,* 2019).

$$\sigma 0.2 = \sigma 0 + K\varepsilon D\text{-}0.5 + \alpha Gb\rho 0.5$$

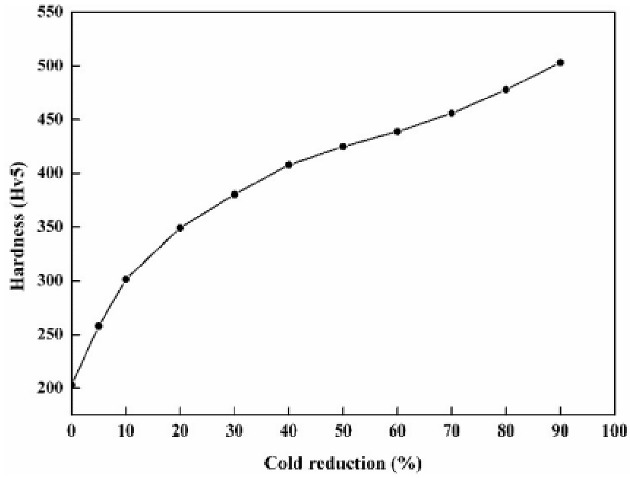

Figure 2.11: Correlation of cold rolling with hardness of AISI 304N.
(Source: Li et al., 2019)

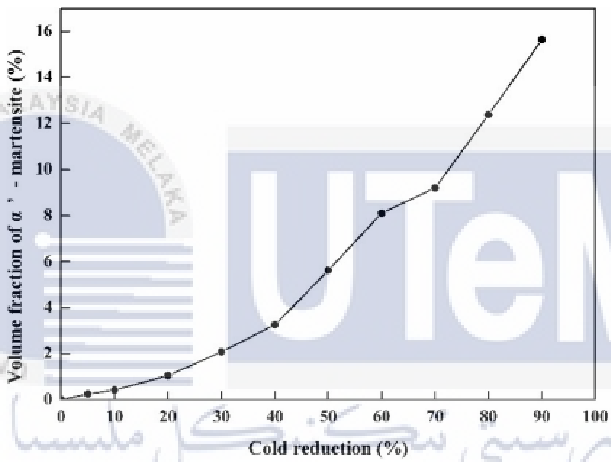

Figure 2.12: Correlation of cold rolling with volume fraction of α'-martensite of AISI 304N
(Source: Li et al., 2019)

Figure 2.11 shows the relation of hardness with the increment of cold rolling percent. As presented in the graph, the mechanical process had significantly hardened the material. From Figure 2.12, the line indicates that as the cold reduction % increased, the volume fraction of α'-martensite increased. For its mechanical properties, at 90% CR, the yield strength increased from 310MPa to 1534MPa, tensile strength increased from 653MPa to 1668MPa and the elongation decreased from 56% to 1.5%. The results proved that cold working increased the strength of the austenitic stainless steel due to the formation of α'-martensite and the lamellar grains in its microstructure. (Li *et al.*, 2019).

2.6 Research Gap

From the literature review made based on articles and journals related to the title of this project, the study on the hardness of the cold-rolled samples before and after polishing process has not been clarified yet. Due to surface alterations, there should be differences on the hardness results of the samples before and after they undergo processes like grinding, polishing and etching. The grain structures of the tested samples are also expected to give obvious differences before and after surface alterations has been made.

CHAPTER 3

MATERIALS AND METHODOLOGY

3.1 Introduction

In this chapter, the research methodology for studying the effect of cold rolling on the microstructure and hardness properties of 316L SS is explained in more details. Before conducting the experiment, it is best to determine all the variables. In this project, the constant variables are the temperature of surrounding and the initial thickness of samples. The manipulated variable is the cold rolling percentage (CR %) and the responding variables are the microstructural transformation and micro-hardness of the tested samples.

For the sample preparation, the as-received samples will be inspected its dimension such as thickness and length. The initial condition of microstructures and the hardness of 316L austenitic stainless steel will be examined and recorded before they undergo cold rolling process. The mechanical process involved in this project is cold rolling. This process is going to be carried out at room temperature with fixed initial thickness of the testing samples.

In order to investigate the outcomes, microstructural deformation of tested samples and its hardness properties after cold deformation process will be analysed using optical microscopy and Vickers micro hardness tester respectively.

3.2 Project Flowchart

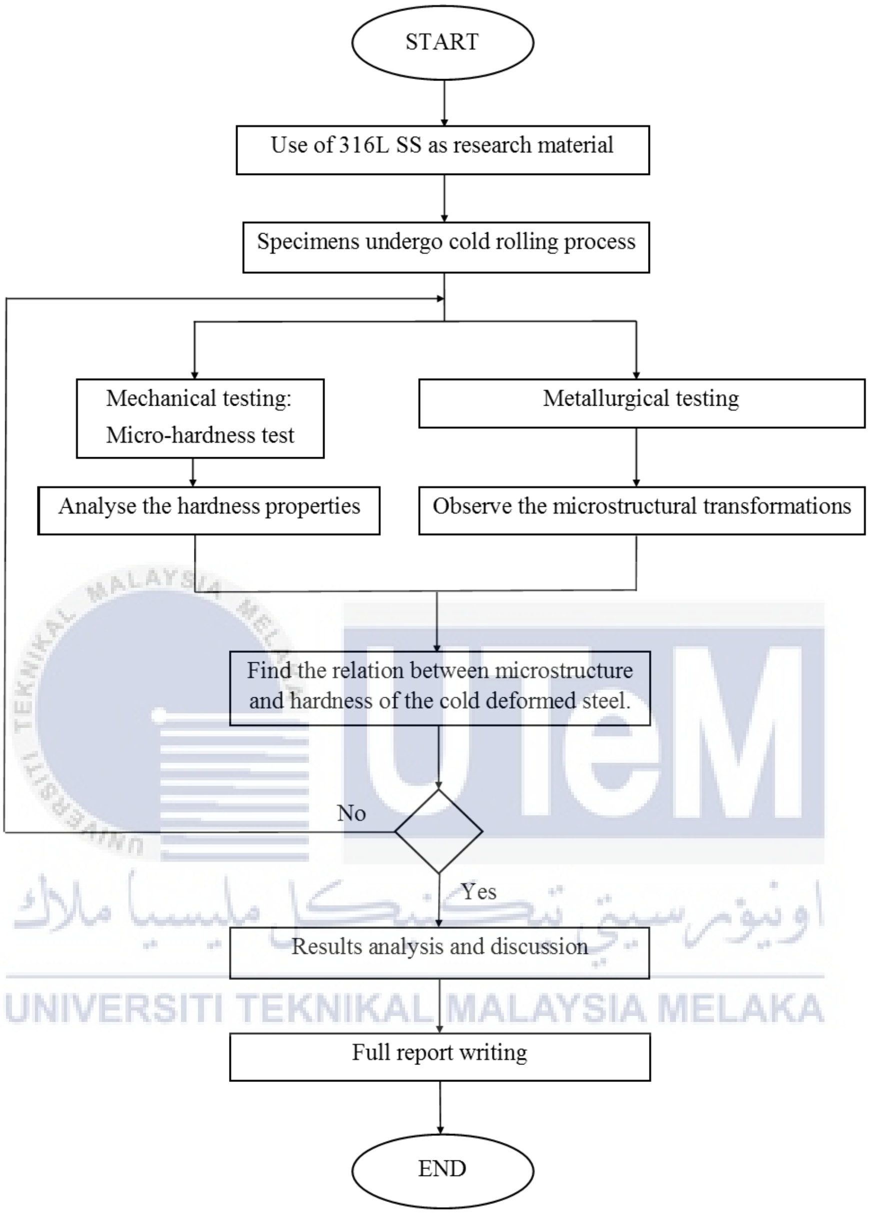

Figure 3.0: Flowchart of project.

The work flow of this project begins with doing literature review on previous studies and after that, this project starts by the using 316L SS as the research material. Next, the test specimen is let to the process of cold rolling. The samples are then underwent a mechanical testing and a metallurgical testing. The results were obtained and discussions were made.

3.3 Material Used for the Research

The material used as the test specimen for the research is 316L stainless steel (316L SS). It comes in the form of sheet-metal rolls with as-received thickness of 2.0mm. The manufacturer of the steel product is POSCO AST Co. Ltd., Korea. It comes in the form of cold rolled coil with an ASTM 316L SS 2B surface finishing. (Mohamad *et al*., 2015)

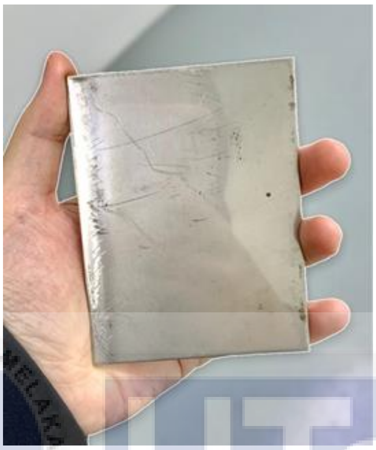

Figure 3.1: 316L Stainless steel sheet.

Table 3.0 below displays the chemical composition (wt %) of 316L SS as the material used in this project. According to the literature review, the main alloying elements, like Cr, Ni, and C, have a slight difference in their austenitic grade. Nevertheless, the elements differs greatly with others austenitic grades. (Mohamad *et al*., 2015)

Table 3.0: Chemical compositions of the 316L as provided by supplier.

316L SS	Chemical composition (wt %)										
	Cr	Ni	C	Mo	Mn	S	P	Si	Cu	N	Fe
	16.61	10.44	0.02	1.048	2.03	0.0022	0.03	0.571	0.28	0.0221	Bal.

3.4 Cold Rolling Process

The only mechanical process that involved in this project is cold-rolling. Cold rolling is chosen as the mechanical process because the material of 316L stainless steel are not able to be hardened by heat treatment. This mechanical process takes place in a room temperature. The model of cold rolling machine used in this process is MDM LS120 Motorized Single Rolling Mill.

In cold rolling, the test specimens were put to pass in between a rolling mill and its plate under gradually increasing force until required thickness reduction is achieved. This mechanical process leads the specimen to experience plastic deformation. The 316L stainless steel sheet is prepped into five smaller pieces of samples. They are then forced to undergo cold rolling by using rolling mill for plate which only have one roller with rolling speed of 4 m/min.

Figure 3.2: Cold rolling machine.

3.4.1 Experimental Procedures for Samples Preparation

The initial (as-received) thickness of the 316L stainless steel plate is 2.0 mm. It was divided into five pieces of samples with dimension of 80.0 mm (length) x 20.0 mm (width). The process will test the specimen by five thickness reduction percent (%RT); 0%, 10%, 20%, 30% and also 40% which means that the cold-rolled specimen thickness are 2.0mm, 1.8mm and 1.6mm, 1.4mm and 1.2mm respectively.

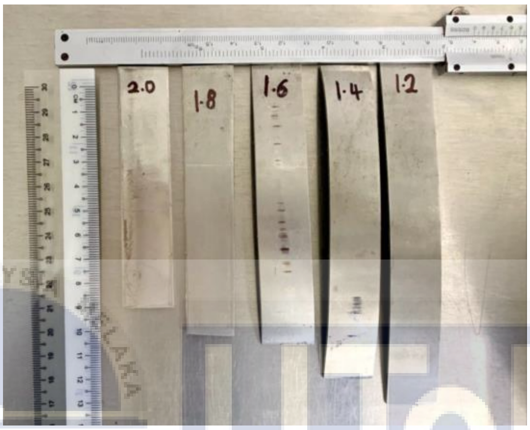

Figure 3.3: Cold-rolled samples of 316L SS.

The rolling process started by inserting the test specimen into the roller blade for better handling in operation. It is positioned in the centre of the rollers and in line with the direction of rolling. The 2.0 mm thick specimen was marked and rolled in one direction only.

Earlier in the process, the specimen is positioned in the centre of rolling mill and the plate to make sure that the rolling load is distributed uniformly to the surface. The thickness of the specimen is then decreases slightly for each one pass by making adjustments to the roller gap. (Mohamad *et al*., 2015)

The procedure is repeated continuously until the specimen reach the required thickness at 40% RT. Oil lubrication was applied continuously to the test specimen and on the set of roller for each rolling pass for the purpose of reducing friction and minimizing the formation of residual stresses.

Once all the samples have been rolled according to their thickness reduction, the middle part of the samples were cut in size of 10mm x 20mm for mounting purpose. The samples are mounted by using phenolic powder mounting compound and the automatic mounting press machine. Then, all the mounted samples were hand grinded manually one by one with six levels of sand papers. Starting with the roughest one which is sand paper 320, followed by 400, 600, 800, 1000, and finally the finest one which is sand paper 1200.

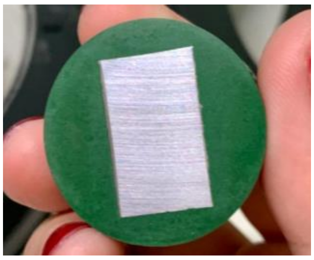

Figure 3.4: Surface of sample after grinding.

After the grinding process, the next step is to polish the surface of the samples. The solution that was used during the polishing is called diamond suspension 0.3µ polycrystalline. A polishing cloth was attached on the polishing plate with sufficient amount of diamond suspensions smothered on it. All the samples were also individually polished by manual instead of putting the samples onto the polishing head due to technical issue.

As the polishing process done, a mirror like surface should appear on the surface of all the polished samples. This indicates that the grinding and the polishing process has refine the surface roughness and the scratches of the samples after they experienced thickness reduction by the cold rolling process. Consequently, each of the samples were etched with oxalic acid solution to reveal the microstructural details.

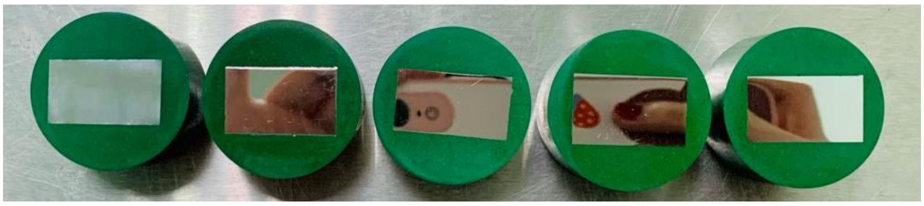

Figure 3.5: Surface of samples after polishing.

3.5 Metallurgical Testing

The metallurgical tests are carried out to analyse the phase transformation of the cold-rolled steel. After the test specimen undergoes a mechanical process, it is advantageous to correlate surface modification with phase present, surface defects, strain hardening effect, and mechanical resistance. These transformations happened to the steel's phase analysis, microstructure, and morphology (Mohamad *et al.*, 2015).

3.5.1 Microstructure Observation

After cold-rolling process, the microstructures of the 316L SS samples were examined by using a digital optical microscopy image analyser Axioskop 2 Mat of brand Zeiss and the software iSolution Lite. Surface modifications were observed on mounted specimens in both non-etched (10% RT and 40% RT) and etched state (0% RT to 40% RT). Microstructure of all the cold rolled samples are analysed on its cross sectional and along its direction of rolling where it is 10X magnification for side and 20X magnification for the surface.

Figure 3.6: Optical microscopy image analyser.

3.6 Mechanical Testing

The purpose of carrying out mechanical testing is to obtain the mechanical properties of the specimen such as tensile strength and the hardness properties. In this project, the tensile strength was not carried out due to time constraint and limitation of raw material. However, the hardness test was done to examine the mechanical resistance to enable the permanent deformation of specimen due to the mechanical process.

3.6.1 Hardness Test

The Vickers hardness test is carried out to determine the hardness of materials, particularly thin parts and small pieces such as the samples in this study. The Vickers Pyramid Number (HV) is the unit for the micro hardness provided by the test. The Vickers hardness of each samples were evaluated by measuring up to 9 points in various locations on the samples' surface (right, middle and left) and the average values were calculated.

The micro-hardness tester used (SHIMADZU Micro Vickers Hardness Tester HMV-G Series) was set with a 0.5kg test load or 4.905N force. The holding time of the indenter is set as 10s to make sure that the hardness tester's loading device is stationary. Thus, the load applied to the material is a static load and not dynamic load (moving) because it will affect the accuracy of the hardness test.

Figure 3.7: Micro Vickers Hardness Tester HMV-G Series.

CHAPTER 4

RESULTS AND DISCUSSIONS

4.1 Microstructural Transformation with Increasing Thickness Reduction % RT.

The grain structures of the original 316L stainless steel sample and the cold rolled samples were analysed by using an optical microscopy. The mechanical properties of the cold-rolled 316L stainless steels are determined by their microstructural deformations. The optical micrographs of the as-received 316L stainless steel sample and 10%, 20%, 30%, and 40% cold rolled samples are presented in Figure 4.0 until Figure 4.4.

As shown in Figure 4.0, the fundamental structure of the initial condition sample (0% RT) for 316L stainless steel were coarse austenite grains with multiple annealing twins. Meanwhile in Figure 4.1, at thickness reduction of 10% which is at the start of the cold deformation, the grain shape remained in equiaxed shape with just a few grains showing deformation bands. The deformation bands can be seen to form in single direction and parallel with one another.

As can be seen in Figure 4.2, once the cold rolling thickness reduction reached 20%, deformation bands were founded in some more grains. According to Xiao Li et al. (2018), during this point, the content of strain-induced martensite has increased as the thickness reduction percentage increased. From observation on the optical micrograph for 30% RT (Figure 4.3), there are formations of some strain-induced martensite at the shear bands. The strain-induced martensite generated upon cold deformation could enhance the strength of the samples significantly (Deming et al., 2018).

The untransformed austenite experienced elongation along the rolling direction as the thickness reduction (%RT) was raised to 40%. With increasing cold rolling reduction, the density of the grain boundary and volume fraction of martensite increased. In general, martensite phase indicates a higher strength but lower plasticity. Jianxin et al. (2018) justified that the austenite phase in stainless steel is an unstable phase and will eventually turn into strain-induced martensite during plastic deformation process.

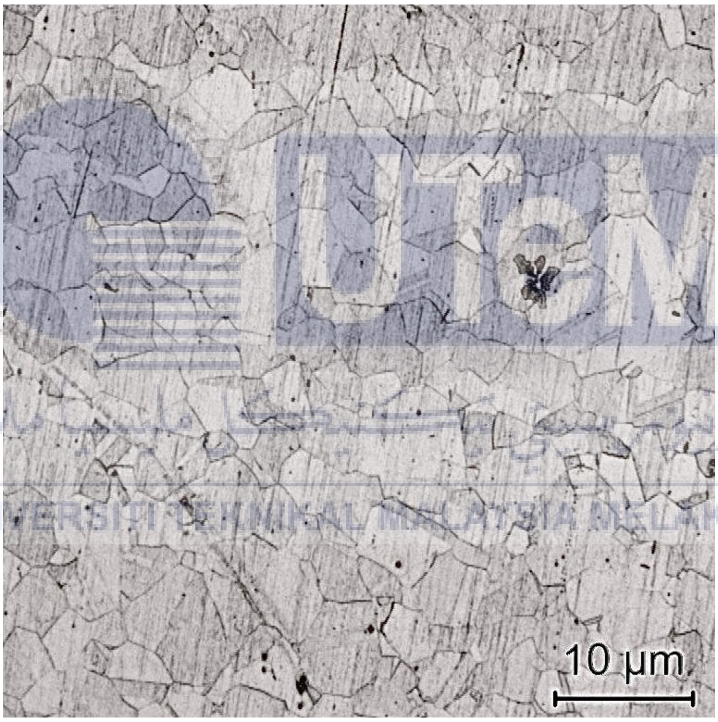

Figure 4.0: Optical micrograph of 0% RT 316L SS sample.

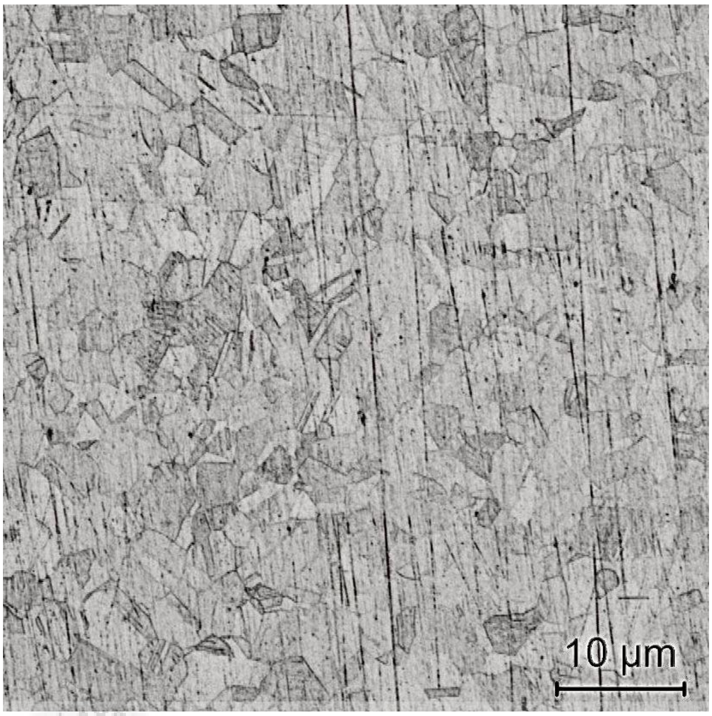

Figure 4.1: Optical micrograph of 10% RT 316L SS sample.

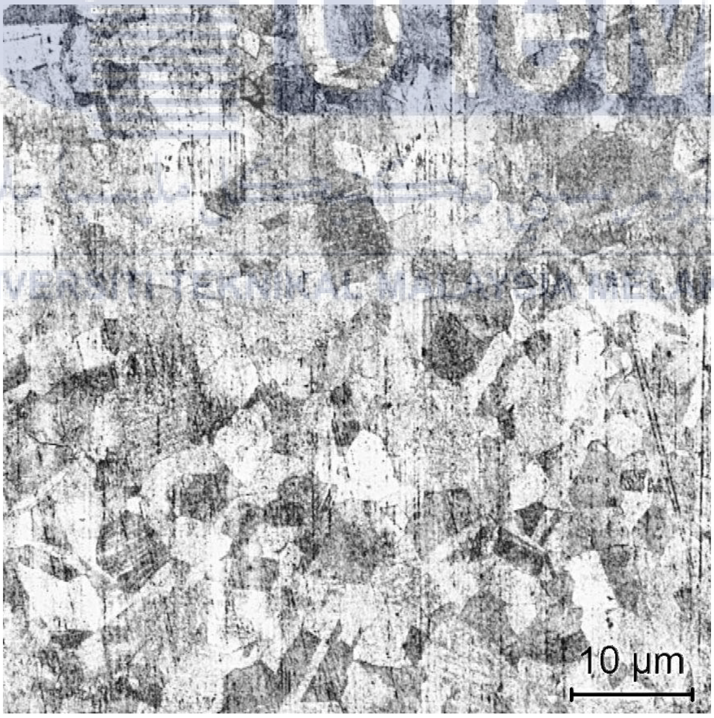

Figure 4.2: Optical micrograph of 20% RT 316L SS sample.

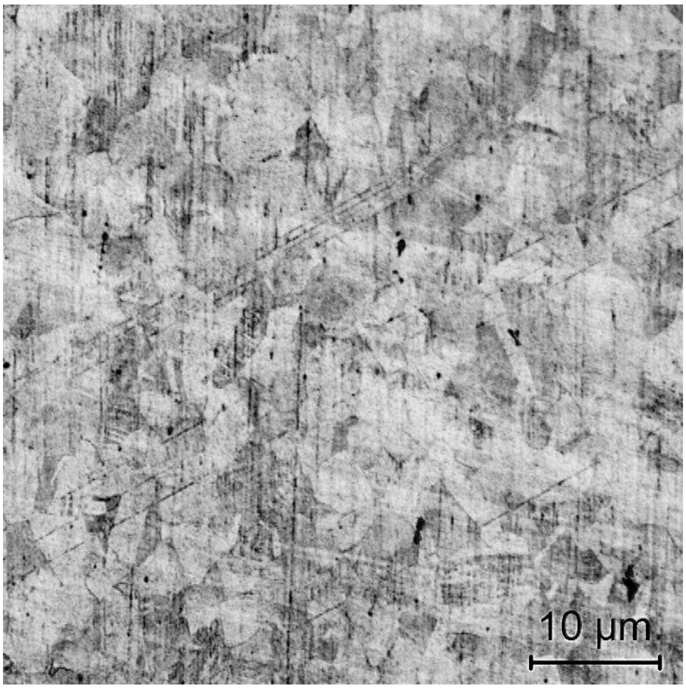

Figure 4.3: Optical micrograph of 30% RT 316L SS sample.

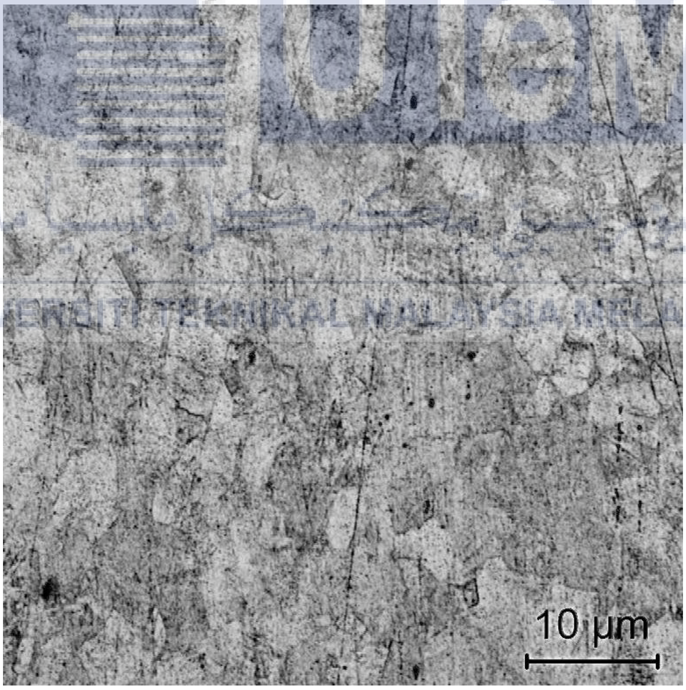

Figure 4.4: Optical micrograph of 40% RT 316L SS sample.

4.2 Observation on Surface Structure for Unpolished Samples

After the cold rolling process was done, 2 unpolished samples which are 10% (thickness of 1.8mm) and 40% (thickness of 1.2mm) were cut into the size of 10mm x 20mm and mounted on a polymer clay. The surface structure and the cross-sectional structure for each of the samples was studied under an optical microscopy. Lens magnification of 20X was used to examine the surface structure along the cold rolling direction. Meanwhile, the cross-sectional was observed with the magnification of 10X. Below are the optical micrographs for the samples examined.

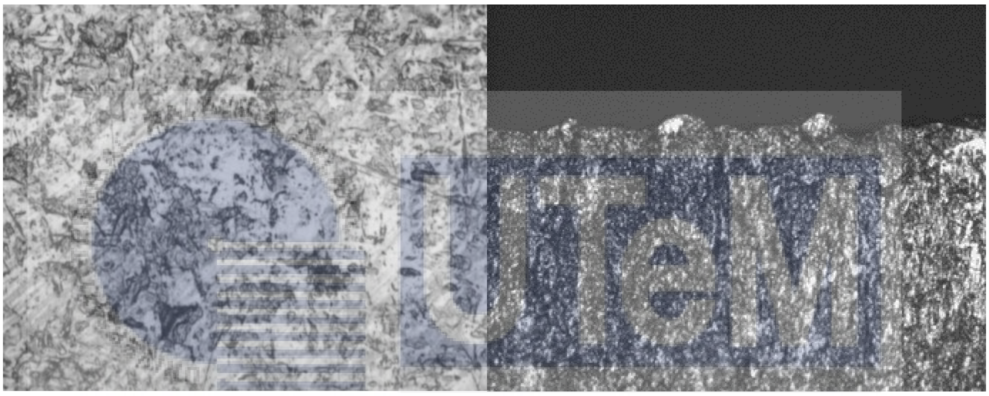

Figure 4.5: Optical micrographs of 10% RT of cold rolled sample (surface and side).

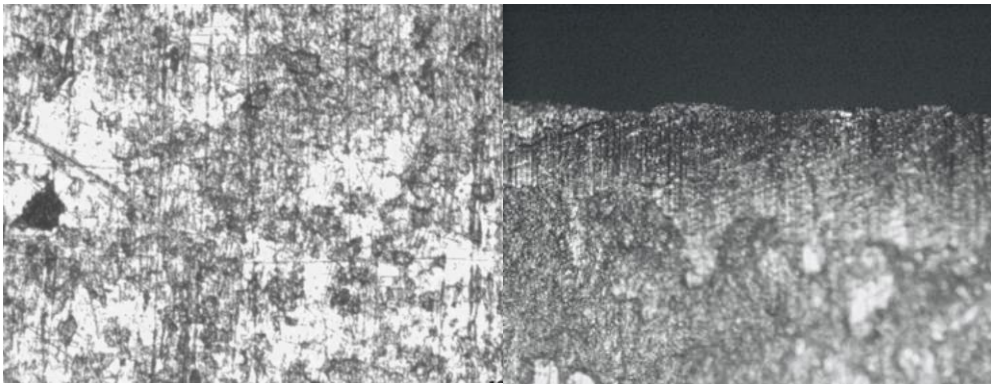

Figure 4.6: Optical micrographs of 40% RT of cold rolled sample (surface and side).

Based on the observation, the original form of the grain structures for the material cannot be seen after being plastically deformed due to the mechanical process it underwent. Apart from that, the boundaries between grains are totally invisible under the maximum magnification. From Figure 4.5 and Figure 4.6, the bold appearance of major scratches and cavities are shown on the surface of the cold-rolled samples. For the cross-sectional, there are appearance of severe surface roughness on the top part where the surface was rolled due to machining effect. These are the factors affecting the micro hardness of the material.

4.3 Vickers Hardness with Increasing Thickness Reduction % RT.

Table 4.0: Vickers hardness before grinding, polishing & etching.

RT (%)	Left Part (HV)			Middle Part (HV)			Right Part (HV)			Average (HV)
0	188.4	182.2	188.7	186.3	174.7	169.0	167.8	164.3	164.3	**176.2**
10	265.3	261.7	261.9	258.4	265.5	258.8	255.0	255.0	255.2	**259.6**
20	295.4	288.0	291.0	307.9	307.5	299.3	311.6	312.1	320.7	**303.7**
30	360.9	365.5	360.1	349.4	345.0	344.6	350.5	345.6	350.0	**352.4**
40	394.5	388.7	401.4	401.2	401.7	401.2	395.8	390.0	377.8	**394.7**

Table 4.1: Vickers hardness after grinding, polishing & etching.

RT (%)	Left Part (HV)			Middle Part (HV)			Right Part (HV)			Average (HV)
0	172.8	167.6	171.1	163.6	166.0	166.0	162.3	160.8	162.5	**165.9**
10	223.3	234.3	228.3	225.6	239.7	234.0	213.1	231.4	231.2	**229.0**
20	255.4	245.8	251.8	254.9	257.9	246.1	258.8	245.6	262.3	**253.2**
30	299.0	310.8	299.0	299.0	303.0	295.2	299.9	303.0	302.9	**301.3**
40	339.3	339.7	329.6	334.7	339.9	344.8	339.5	334.1	339.5	**337.9**

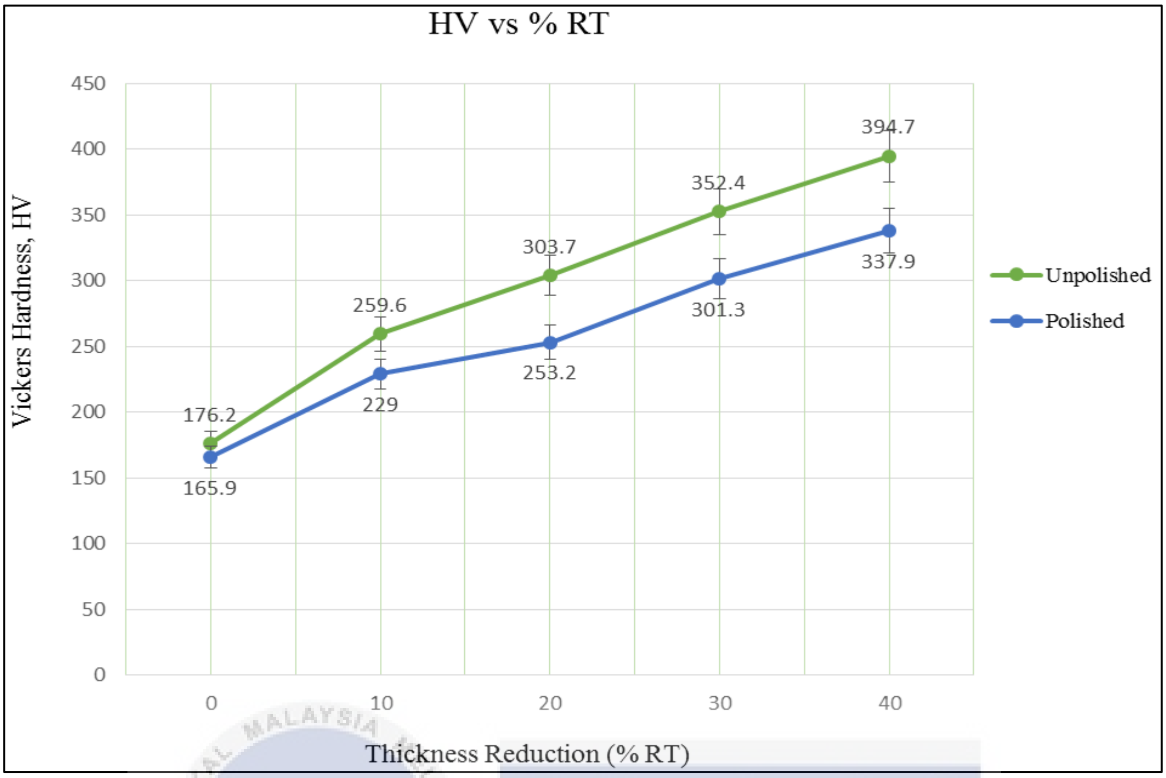

Figure 4.7: Graph of Vickers hardness versus thickness reduction.

Figure 4.7 represents the variation of hardness with cold rolling reduction. From the graph above, both polished and unpolished samples show a steady increasing trend of Vickers hardness after being cold-rolled. Also, it can be seen that the highest value of Vickers Hardness, HV achieved by the polished and unpolished samples of 316L stainless steel in this project are 337.9 and 394.7 respectively.

On average, the percentage increase of the polished samples from one thickness reduction to another is 16%. Even so, when the cold rolling reduction was increased from 10% to 20%, the hardness only increased for around 9.6%. Meanwhile, for the unpolished samples, the highest percentage increase is 32% which occurred in between 0% RT to 10% RT. After that, the graph trend after that shows a constant increase of hardness at 13% on average. A study on material of 316LN ASS by Deming et al. in 2018 agreed that the average Vickers hardness increased as respond to the increasing cold rolling reduction.

It is proven that cold rolling process had significantly increase the hardness of the material for both polished and unpolished samples, as was illustrated in the graph. According to a recent study by Xiao Li et al. (2019), the primary factors for the hardness increment may include the formation of mechanical twins, an increase in dislocation density, and overlapping boundaries due to plastic deformation as discussed in previous section.

Based on the micro hardness test conducted, the Vickers hardness for the unpolished samples depict greater values than the polished samples for each % RT which means they are harder. To justify this finding, the surface roughness of the samples should be considered. This is because, unpolished samples mean that they have higher surface roughness due to cold rolling which caused the surface to have serious crossed scratches hence, strengthen the steel. Plus, inclusions and other elements that exist on the samples' surface which were not removed after the rolling process may also contribute to the increase of Vickers hardness reading for the samples.

4.4 Summary of Findings

Table 4.2: Summary of findings.

Objectives	Findings
To examine the microstructure modifications of the cold-deformed steel.	Permanent defects in the metal structure appeared and changed their crystalline makeup. Formation of martensite from coarse austenite grains and elongated grain structures were observed.
To analyse the hardness properties of the cold-deformed steel.	Both polished and unpolished samples showed a consistent increasing trend of Vickers hardness readings as %RT increased.
To study the relation between microstructure and hardness of the cold deformed steel	The transformation of microstructures as a result of the increasing cold rolling reduction has significantly hardened 316L stainless steel.

4.5 Issues Encountered During Project Execution

Some technical challenges have been encountered while carrying out this project. These problems had indirectly affected the results obtained. For instance, the machine that was used during the cold rolling process is no longer at its best condition of setting. The force pressuring the sample is giving inconsistent thickness reduction with every increment of the rolling pressure scale. Hence, during the execution, many alterations of pressure by the roller were required. The rolling machine is advised to undergo calibration and maintenance periodically in order to provide the optimum function.

Next, there is also an issue with the grinding and polishing machine used. During these two important processes, the tube attached to the polishing head was broken frequently and needed to be change every time. It is the tube that gives the pressure to hold the mounted samples in the machine holder which will press the samples surface onto the rotating disc. This issue lead to instable grinding and polishing because the samples were not firmly pressed on the sand paper or polishing cloth, and sometimes they even detached from the holder. It is suggested for the tube to be upgraded to a higher quality which is not as fragile as the current one.

Another major challenge faced during the execution of this project was during the Vickers hardness testing, where there was floor vibration. The source of vibration was detected from the operating pressure pump that is located in the same lab. It is acknowledged that the Vickers hardness test is highly sensitive to any vibration even a minor one. During the hardness test, the indentation shape was found to be odd where it is supposed to look like a diamond shape (for Vickers indenter tip). To overcome this problem, the hardness test was carried out carefully whenever the pump is not operating to obtain precise result.

CHAPTER 5

CONCLUSIONS AND RECOMMENDATIONS FOR FUTURE RESEARCH

5.1 Conclusions

In this project, cold rolling reduction's impacts on the microstructures and hardness properties of 316L stainless steel were investigated. From the microstructural examination of the cold rolled samples, elongated grain structures and overlapping boundaries were observed after the steel is deformed plastically which results in strain hardening. Moreover, it can be concluded that the transformation from austenite phase into martensite phase increases the strength of the studied material. After being cold-rolled, both polished and unpolished samples showed a consistent increasing trend in Vickers hardness. The unpolished samples indicated higher Vickers hardness because of the unaltered surface finishing after cold rolling process. The average of percentage increase of Vickers hardness (from one % RT to another) for polished samples is 16% while for the unpolished samples is 22.5%. To summarise, it is proven that the transformation of microstructures as a result of the increasing cold rolling reduction percentage has significantly hardened 316L stainless steel.

5.2 Recommendations for Future Research

Since this paper did not cover on the analysis of micro hardness on the cross sectional side of the samples due to Vickers limitation, it is recommended for that area of study to be focused. It is better if the total point of indentations to be increase at more locations on the sample to obtain data variation. Plus, elaboration on the comparison between the results of hardness on the sample’s surface and on the side should also be included in. Utilisation of Nano indenter is recommended for this work.

Another suggestion for future work indicated by this research is to figure out what are the inclusions or specifically other elements that presented after cold rolling process, which contribute in the increase of hardness properties of 316L SS. For example, the lubricant excess and the rolling mill itself. There are possibilities that elements other than the original chemical composition of 316L SS (C, Mn, Si, P, S, Cr, Mo, Ni, and N) could exist on the material as the result from contamination during the mechanical process. These elements can be identified by using Scanning Electron Microscopy (SEM) and Energy Dispersive X-Ray Spectroscopy (EDS).

Additionally, the study on the optimum thickness reduction that could give constant Vickers hardness is also to be included in the proposal for future research. This work might need to increase the percentage of cold rolling reduction for more than 40% and manipulate the variables of Vickers hardness tester such as the holding time and the test force loaded on the sample. This study aims to determine at which thickness reduction (% RT) at some point, will result to the highest and steady hardness on the % RT versus HV graph.

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