

**EFFECT OF COLD TREATMENT  
ON THE MECHANICAL PROPERTIES  
OF 316L STAINLESS STEEL**

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## **DECLARATION**

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

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## ABSTRAK

*Austenitic stainless steel 316L* merupakan salah satu keluli yang biasa digunakan dalam pelbagai aplikasi. Ini disebabkan oleh sifatnya yang tahan kepada kakisan berserta dengan kelebihannya yang lain. Walaubagaimanapun, sifat *yield strength* yang rendah akan menyebabkan berlakunya kehausan dan kelusuhan. Dalam projek ini, kesan *cold rolling* ke atas sifat mekanik *austenitic stainless steel 316L* telah dikaji. Hasil daripada penyelidikan sebelumnya menunjukkan *austenitic stainless steel 316L* mempunyai stacking fault energy yang rendah, yang mana *austenite* akan melalui transformasi ke fasa  $\alpha'$ -*martensite* sekiranya cold rolling dilakukan. Transformasi ini dipercayai akan membawa kesan pengerasan kepada bahan tersebut. *Cold rolling* dipilih dalam projek ini kerana ia adalah salah satu pilihan yang digemari dalam aplikasi industri. Dalam projek ini, bahan dengan ketebalan asal 2mm dibentuk kepada ketebalan yang berbeza dengan menggunakan proses *cold rolling*. Hasil daripada proses tersebut, peratusan *cold work* 10% hingga 69% dapat diperolehi. Pemerhatian mikrostruktur telah dilakukan ke atas specimen dengan menggunakan mikroskop optik dengan pembesaran sehingga 1000 kali ganda. Kemudian, sifat mekanikal bahan tersebut diuji dengan menggunakan ujian tegangan mengikut ASTM E8M. Apabila pengurangan ketebalan meningkat, *tensile strength* dan *yield strength* menunjukkan peningkatan trend sehingga tepu. Jumlah peningkatan dalam *yield strength* dicatat ialah 300%, dari 250MPa hingga 1000MPa. Di sisi lain, *tensile strength* mengalami peningkatan kira-kira 100%. Walau bagaimanapun, *toughness* dan *ductility* menunjukkan penurunan yang ketara selepas *cold work*. Kemuluran bahan akhirnya jatuh kepada hanya 2% selepas sejuk digulung kepada 69%. Modulus Young menunjukkan penurunan sedikit pada awal rolling sejuk dan sampai tepu selepas pengurangan ketebalan. Spesimen dengan peratusan *cold work* yang lebih tinggi telah menunjukkan perubahan bentuk yang lebih tinggi dalam mikrostruktur dan mendorong fasa  $\alpha$ -*martensit* ditunjukkan dalam mikrostruktur

## ABSTRACT

Austenitic stainless steel 316L is one of the most commonly used stainless steel in various applications, mainly due to its great corrosion resistance and other benefits. However, its drawback is its low value in yield strength which will lead to further wear and tear concern. In this research, the effect of cold rolling on the mechanical properties of the austenitic stainless steel 316L was studied. The previous research outcomes show that austenitic stainless steel 316L has low stacking fault energy, which indicating the austenite will go through transformation into  $\alpha'$ -martensite, when being deformed by various method. This transformation is believed will lead to material hardening. Since cold rolling is one of the preferred methods in industry application, this research used cold rolling as cold work method to refine the material. In the research, the material with original thickness 2mm was cold rolled into different thickness, resulting the cold working of 10% to 69%. Microstructure observation was done on the cold rolled specimens by using optical microscope with magnification up to 1000x. The material was then tested its mechanical properties using tensile testing according to ASTM E8M. Analysis was done on the microstructure observation result to see if there is any change in microstructure behavior as referred to other researches. As the reduction of thickness increased, the yield strength and tensile strength showed increasing trends up to its saturation. The total increase in yield strength was recorded to be 300%, from 250MPa to 1000MPa. In the other hand, the tensile strength had increase for approximately 100%. However, the ductility and toughness showed a significant drop after cold working. Ductility of the material eventually dropped to only 2% after cold rolled up to 69%. The Young's modulus showed slight decrease during the beginning of the cold rolling and reach saturation after certain amount of thickness reduction. The specimen with higher cold work percentage had shown higher deformation in the microstructure and induced the  $\alpha$ -martensite phase shown in the microstructure

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## LIST OF ABBREVIATIONS

ASTM	-	American Society for Testing and Materials
PSM	-	Project Sarjana Muda
UTM	-	Universal Testing Machine

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## **CHAPTER 1**

### **INTRODUCTION**

#### **1.1 BACKGROUND**

Stainless steel is essentially a low carbon steel which contains chromium at approximately 10 percent by weight. It is the addition of chromium that gives the steel its special stainless, corrosion resisting properties. The chromium content of the steel allows the formation of a rough, adherent, invisible, corrosion resisting chromium oxide film on the steel surface. If it is damaged mechanically or chemically, this film is self-healing, providing that oxygen, even in very small amounts, is present. The corrosion resistance and other useful properties of the steel are enhanced by increased chromium content and the addition of other elements such as molybdenum, nickel and nitrogen. Many unique values provided by stainless steel make it a powerful candidate in materials selection. Engineers often underestimate or overlook these values because of what is viewed as the higher initial cost of stainless steel. However, over the total life of a project, stainless is usually the best value option to choose.

Stainless steel has several significant and common benefits in applications, namely corrosion resistance, fire & heat resistance and strength-to-weight advantage. Stainless steel has greatly enhanced corrosion resistance compared to other type of

metals. Lower alloyed grades resist corrosion in atmospheric and pure water environments, while high-alloyed grades can resist corrosion in most acids, alkaline solutions, and chlorine bearing environments, properties which are utilized in process plants. It has high heat and high resistance due to high chromium and nickel-alloyed grades resist scaling and retain strength at high temperatures. Stainless steel has a moderate high strength to weight ratio. The work-hardening property of austenitic grades, that results in a significant strengthening of the material from cold-working alone, and the high strength duplex grades, allow reduced material thickness over conventional grades.

Stainless steel is defined as a ferrous alloy with a minimum of 10% chromium content. The name originates from the fact that stainless steel does not stain, corrode or rust as easily as ordinary steel. This material is also called corrosion resistant steel when it is not detailed exactly to its alloy type and grade, particularly in the aviation industry. Stainless steels have higher resistance to oxidation (rust) and corrosion in many natural and man-made environments. Other than that, there is a very special feature in stainless steel. The chromium forms a passivation layer of chromium (III) oxide ( $\text{Cr}_2\text{O}_3$ ) when exposed to oxygen. The layer is too thin to be visible, meaning the metal stays shiny. It is impervious to water and air, protecting the metal beneath. Also, when the surface is scratched this layer quickly reforms. This phenomenon is called passivation by materials scientists, and is seen in other metals, such as aluminium. When stainless steel parts such as nuts and bolts are forced together, the oxide layer can be scraped off causing the parts to weld together. When disassembled, the welded material may be torn and pitted, an effect that is known as galling.

There are different types of stainless steels: when nickel is added, for instance, the austenite structure of iron is stabilized. This crystal structure makes such steels non-magnetic and less brittle at low temperatures. For higher hardness and strength, carbon is added. When subjected to adequate heat treatment these steels are used as razor blades, cutlery, tools etc. Significant quantities of manganese have been used in

many stainless steel compositions. Manganese preserves an austenitic structure in the steel as nickel, but at a lower cost.

Stainless steels are classified by their crystalline structure. Austenitic stainless steel will be focus in this project thus will be further discussed. Austenitic stainless steels comprise over 70% of total stainless steel production. They contain a maximum of 0.15% carbon, a minimum of 16% chromium and sufficient nickel and/or manganese to retain an austenitic structure at all temperatures from the cryogenic region to the melting point of the alloy. A typical composition is 18% chromium and 10% nickel, commonly known as 18/10 stainless is often used in flatware.

Table 1.1: Comparison of composition and mechanical properties of austenitic, duplex and ferritic grade of stainless steel. ( N.R.Baddoo )

Group of stainless steel	Grade		Composition (EN 10088)			0.2% proof strength <sup>3</sup> (N/mm <sup>2</sup> )	Ductility <sup>4</sup> (%)
	EN	AISI/ASTM	Cr	Ni	Mo		
Austenitic	1.4301	304	17.5–19.5	8.0–10.5	-	230	45
	1.4401	316	16.5–18.5	10.0–13/0	2.0–2.5	240	40
	1.4162 <sup>b</sup>	S32101	21.0	1.5	0.3	530	30
Duplex	1.4362	S32304	22.0–24.0	3.5–5.5	0.1–0.5	450	20
	1.4462	S32205/S31803	21.0–23.0	4.5–6.5	2.5–3.5	500	20
	1.4510	439	16.0–18.0	-	-	240	23
Ferritic	1.4509	441	17.5–18.5	-	-	250	18
	1.4521	444	17.0–20.0	-	1.8–2.5	320	20

## 1.2 PROBLEM STATEMENT

The material used in this project is 316L Austenitic Stainless Steel. This grade of stainless steels show great value in industries, however, its main drawback is its low tensile strength. The low tensile strength of the stainless steel may lead to tribological concern such as wear and plastic deformation when applied to high stress. Thus, several studies had been done on the objectives of improving mechanical properties of the material.

### 1.3 OBJECTIVE

The objective of this project is:

- (1) To conduct mechanical testing and microscopic observation on the 316L ASS
- (2) To compare the properties of stainless steel which subjected to different percentage of cold work
- (3) To analyse the mechanical properties of cold rolled 316L ASS

### 1.4 SCOPE

The scope of this project involved the very basics steps which was started from the study of the stainless steels, literature review on the cold roll mechanism and the microstructure evolution through cold rolling, suitable mechanical testing to be conducted . Moreover, the scope of this research also included

- (1) The analyze the cold rolled austenitic stainless steel 316L
- (2) To review the cold rolling research by other researchers
- (3) To study the known cold rolling microstructure texture evolution
- (4) To review the most related theoretical analysis of cold rolled stainless steel
- (5) To conduct mechanical testing to obtain the properties for the material
- (6) To conduct microstructure observation using suitable metallographic technique
- (7) To analyze the result from the microscopic observation and mechanical testing

## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 STAINLESS STEEL OVERVIEW

According to Fontana MG (Fontana,1986), stainless steels firstly appeared when scientists were trying to develop a state of passivity for ferrous alloys; a state that would not visibly show any oxidization, as was initially produced by Faraday. Streicher MA (Streicher,2000) stated that Harry Bearley's experiment was done in 1912, which was the addition of 12.5% Chromium to Iron, initiated commercial production of stainless steels. This material had a martensitic microstructure matrix. Later, following the works of Guillet and Giesen, Monnartz developed the Fe-Cr-Ni (iron-chromium-nickel) steel, giving origin to stainless steels of the austenitic matrix, universally known as 18% Cr-8%Ni, according to Davis Jr. (Davis Jr, 1996).

According to Davis Jr. (Davis Jr, 1996), there are five major classes of stainless steel: ferritic, martensitic, duplex, hardened by precipitation and austenitic. The austenitic stainless steel is the focus of this project. The austenitic stainless steel is commonly used in application of chemical production, food industries, nuclear industries and medical implants, due to their combination of good conformability, mechanical resistance and resistance to general corrosion. 316L austenitic stainless

steel is different from other grades of this series due to the addition of Mo (~2.5wt.%). This addition of Mo is known to improve both corrosion resistance and creep behavior. Mo in solid solution acts as a favorable element in reducing dislocation mobility. However, its relatively low yield strength (~200MPa) is a drawback of this material's applications. There has been a growing interest in developing the austenitic stainless steel with high strength.

In the recent research (A.Belyakov et al 2004), among a number of proposed methods for developing nano-grained structure in stainless steels, severe plastic deformation is the most attractive technique enabling the fabrication of commercially sizeable products. According to recent studies, the total strain is the most important property for the development of nano structure in metallic materials, whereas processing methods do not play a crucial role in the microstructural evolution .

According to S. Allain et al (S.Allain, 2004), a number of techniques including high pressure torsion, equal channel angular pressing, and cyclic extrusion compression have been developed to obtain the desired mechanical properties of the austenitic stainless steel. However, they are plagued by two main drawbacks. Firstly, forming machine with large load capacities and expensive dies are required. Second, the productivity of such techniques is relatively low and the amount of material produced is very limited. Moreover, these processes are thought to be inappropriate for practical applications, especially for large-sized structural material such as sheets. Thus, conventional cold rolling is considered to be the most appropriate process for industrial interest.

In S. Allain et al (S. Allain,2004)'s latest research, various metal and alloys can be characterized by different kinetics of grain refinement during severe deformation. The grain refinement in austenitic stainless steel is accelerated by multiple mechanical twinning or strain-induced phase transformation, leading to fast development of nano structures at relatively small strains. Various studies results showed that both the twinning and the martensitic transformation depend on the

stacking fault energy (SFE). Twinning has been reported to occur in steels with an SFE above  $18 \text{ mJ/m}^2$ , while the formation of martensite requires an SFE below approximately  $18 \text{ mJ/m}^2$ .

In the case of non-ferrous alloys, there is a lot of work on the effect of SFE on the development of texture and microstructure. However, in comparison, not so much work has been reported on steels. Most austenitic steels, such as austenitic stainless steel, high manganese Hadfield steels and high manganese twinning induced plasticity steel (TWIP) have low to moderate SFE and thus they tend to form extended stacking faults, deformation twins and planar dislocation structures.

It is known that applying stress to austenitic stainless steels produces a variety of deformation microstructures, depending on material and testing conditions. Tangled random dislocations, dislocation pile-ups, defect-cleared dislocation channels, stacking faults, twins, and martensite particles were found in the deformation microstructure. Low SFE materials tend to exhibit a more banded, linear array of dislocation and stacking fault. All these features are related to the dissociation of perfect dislocation into two Shockley partial dislocations, so called leading and trailing partials. The separation of the two partials leaves a stacking fault between them and inhibits cross-slip from forming random dislocation structure, in the research of R.W.Hertzberg (R.W.Hertzberg,1989).

In the same research paper, it stated that among the material properties of austenitic stainless steel, the low stacking fault energy is believed to be the most responsible for changes in deformation microstructure in response to external condition. Many authors have studied the influence of alloys elements on SFE of Austenitic Stainless Steel. They have calculated empirically the SFE of 316L ASS from its chemical compositions, and the result is about  $64 \text{ mJ/m}^2$ . The value of SFE indicates that twinning is expected to occur during cold rolled deformation. Twinning is generally considered as a deformation mechanism that is activated at high strain rates which the critical resolved shear stress for dislocation slip is high. Thus in 316L ASS, deformation is expected to improve mechanical properties by

development of dislocation substructure, formation of deformation induced martensite and different slip-twin activities.

Padilha and Rios (Padilha,2012) found that 316L stainless steel contains thermodynamically metastable austenite at room temperature, which can be transformed to martensite ( strain-induced martensite, SIM) by deformation below the  $M_d$  temperature. The austenite phase in Austenitic Stainless Steel is normally not a stable phase. During the early stages of deformation, shear bands consisting of stacking faults, mechanical twins and  $\epsilon$ -martensite (hcp, non-ferromagnetic) form on the austenite phase. The  $\alpha'$ -martensite (bcc, ferromagnetic) nucleates at the intersections of the shear bands. When the deformation level increases, the  $\alpha'$ -martensite grows by consuming  $\epsilon$ -martensite and austenite phases.

Two important factors control the amount of  $\alpha'$  and  $\epsilon$ -martensite :  $M_d$  temperature and stacking fault energy (SFE) of the Austenitic Stainless Steel. The  $M_d$  temperature which is the temperature where 50% of martensite will form at 30% true strain, had been suggested by Angel (Angel,1954). Nohara et al. (Nohara,1977) modified Angel's equation and also considered the grain size into the equation:

$$M_d (^{\circ}\text{C})= 551 - 13.7(\% \text{Cr}) - 29(\% [\text{Ni}+\text{Cu}]) - 8.1(\% \text{Mn}) - 18.5(\% \text{Mo}) - 9.2(\% \text{Si}) - 68(\% \text{Nb}) - 462 (\% [\text{C}+\text{N}]) - 1.42(\text{GS}-8) \quad (1)$$

Where GS is ASTM grain size number.

The SFE determined by the composition of the Austenitic Stainless Steel and increases with rising temperature. Schramm and Reed (Schramm,1975) presented the following equation to calculate the SFE of ASSs:

$$\text{SFE}(\text{mJ}/\text{m}^2) = -53 + 0.7(\% \text{Cr}) - 6.2(\% \text{Ni}) - 3.2(\% \text{Mn}) + 9.3(\% \text{Mo}) \quad (2)$$

Strain induced  $\alpha'$ -martensite produced in metastable ASSs during cold rolling led to significant increase in their strength. The formation and the amount of strain induced martensite depends on the austenite stability (chemical composition and initial austenite grain size) and the rolling conditions (the deformation amount and temperature, and rolling speed). According to Murata et al (Murata,1993), when the austenite stability and the deformation temperature are low, the martensite content will be increased. Through research and the authors claim that martensite content will increase the mechanical strength of the material.

## **2.2 PLASTIC DEFORMATION OF METASTABLE AUSTENITIC STAINLESS STEEL**

### **2.2.1 Crystal structure of austenitic stainless steel**

Austenitic stainless steels have FCC microstructure. This is achieved by the combination of chromium and nickel alloying. The most common alloy content is 18% of Cr and 8% Ni. The interstitial atoms, namely carbon and nitrogen, also promote the FCC crystal structure and cause significant solid solution strengthening. The use of carbon for solid solution strengthening is its tendency to form carbides at high temperatures. This may result in the depletion of chromium in the grain boundaries and therefore cause a risk of intergranular corrosion. However, such a risk is smaller for nitrogen. Hence, nitrogen has been used as an alloying element providing austenite stabilization, solid solution strengthening and increased corrosion resistance in various austenitic stainless steel grades. An example is the Austenitic Stainless Steel 316L.

The FCC microstructure of most austenitic stainless steel is not thermodynamically stable at room temperature. Therefore, applied stress or plastic deformation may induce a martensitic phase transformation, by which the metastable austenite phase is transformed to the thermodynamically more stable martensite

phase. Two different martensite phases exist in austenitic stainless steel: hexagonal close packed (HCP)  $\epsilon$ -martensite and body-centred cubic (BCC)  $\alpha'$ -martensite. Due to the relatively low interstitial content, the crystal structure is normally referred to as BCC and not as body centred tetragonal. As below will further discuss the strain induced martensitic transformation have a significant influence on the mechanical properties of austenitic stainless steel.

### 2.2.2 Formation of stacking fault and $\epsilon$ -martensite

From the research by Olson (Olson,1972), Intrinsic stacking faults form in the FCC crystal lattice as a consequence of the dissociation of  $\frac{a}{2} \langle 110 \rangle$  perfect dislocations into two  $\frac{a}{6} \langle 211 \rangle$  partial dislocations. Intrinsic stacking fault is formed between partials and the stacking sequences of the  $\{111\}$  planes are changed from initial ABCABCABC to ABCACABCA. If two intrinsic stacking faults overlap on the successive  $\{111\}$  planes, the resulting stacking sequence will be changed to ABCACBCAB, which has one excess plane with the C stacking. Such a fault is referred to an extrinsic stacking fault.

Due to the low SFE, wide stacking faults are frequently observed in the microstructure of austenitic stainless steels. The width of the stacking fault is limited by the energy stored by the stacking fault. Other than that, the Shockley partial dislocations repel each other due to their mutual interaction, which tends to broaden the fault.

In the case of intrinsic stacking fault, the change in the stacking sequence of the  $\{111\}$  atom planes causes a thin layer of hexagonal close-packed phase with the stacking sequence of CACA. Therefore, even a single stacking fault can be taken as a nucleus of HCP  $\epsilon$ -martensite. As the result, it is difficult to differentiate between single stacking faults, bundles of overlapping stacking faults, and faulted or perfect