SCHOOL OF MATERIAL AND MINERAL RESOURCES ENGINEERING

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THE EFFECT OF THICKNESS REDUCTION ON MICROSTRUCTURE AND MECHANICAL PROPERTIESOF COLD ROLLED AND CRYOROLLED LOW CARBON STEEL

BY

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DECLARATION

I hereby declare that I have conducted, complete the research work and written the dissertation entitled "**The Effect of Thickness Reduction on Microstructure and Mechanical Properties of Cold Rolled and Cryorolled Low Carbon Steel**". I also declare that it has not been previously submitted for the award of any degree or diploma or other similar title of this for any other examining body or University.

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LIST OF ABBREVIATIONS

UFG	Ultrafined Grain
SPD	Severe Plastic Deformation
ASTM	American Society for Testing and Materials
XRD	X-Ray Diffraction
OES	Optical Emission Spectroscopy
AISI	American Iron and Steel Institute
MMC	Metal Matrix Composite
ECAP	Equal Channel Angular Pressing
HPT	High-Pressure Torsion
ARB	Accumulative Roll Bonding
FL-ARB	Four-Layer Roll Bonding
UTS	Ultimate Tensile Strength
YS	Yield Strength
UTM	Universal Testing Machine
EBSD	Electron Backscatter Diffraction
FESEM	Field Emission scanning electron microscope
SEM	Scanning electron microscope

LIST OF SYMBOLS

%	Percentage
K	Kelvin
°C	Degree Celcius
°F	Degree Fahrenheit
wt	Weight

KESAN PENGURANGAN KETEBALAN TERHADAP MIKROSTRUKTUR DAN SIFAT MEKANIKAL KELULI KARBON RENDAH GELEKAN SEJUK DAN KRIO

ABSTRAK

Keluli karbon rendah mempunyai kegunaan yang luas dalam sektor perindustrian. Kekuatannya yang rendah menjadi halangan untuk mengembangkan potensinya dalam kegunaan perindustrian. Sejak kebelakangan ini, struktur bijin kecil telah dikaji dengan meluas kerana kebolehan menghasilkan kekuatan mekanikal yang amat tinggi. Kajian ini mengkaji mengenai mikrostruktur dan sifat-sifat mekanikal gelekan sejuk dan gelekan krio yang digelek dengan pengurangan ketebalan yang berbeza. Pertama sekali, keluli karbon rendah disepuhlindap pada suhu 550 °C selama 60 minit. Untuk gelekan krio sampel dicelup di dalam cecair nitrogen selama 10 minit sebelum digelek. Kedua-dua proses gelekan sejuk dan dan gelekan krio digelek pada pengurangan ketebalan yang berbeza; 50%, 70%, 80% dan 90%. Mikrostruktur, saiz kristal, mikro terikan, ketumpatan terkehel dan sifat-sifat mekanikal untuk kedua-dua proses dalam pengurangan ketebalan yang berbeza telah dikaji melalui mikroskop pengimbas electron, pembelauan sinar-X, ujian mikrokekerasan Vickers and ujian tegangan. Kesemua sampel menunjukan perubahan bentuk bijian apabila bijian memanjang sepanjang arah gelekan. Keluli karbon rendah gelekan krio menunjukan kesan saiz bijian yang paling kecil dengan purata saiz 8.06 µm pada pengurangan ketebalan 90%. Pearlit menunjukan pecahan pada pengurangan ketebalan 70% untuk kedua-dua jenis gelekan. Kandungan cementit bertambah apabila pengurangan ketebalan bertambah. Keluli karbon rendah gelekan krio mempunyai saiz kristal yang paling kecil, mikro terikan dan ketumpatan kehelan yang tnggi untuk kesemua pengurangan ketebalan berbanding keluli karbon rendah gelekan sejuk. Kekuatan ketegangan, kekuatan alah bentuk dan kekerasan menunujukan peningkatan manakala kemuluran menunjukan pengurangan apabila pengurangan ketebalan gelekan bertambah dan gelekan krio pada pengurangan ketebalan 90% menunjukan kekuatan tegangan, kekuatan alah dan kekerasan yang paling tinggi dengan nilai 828.35 MPa, 810 MPa dan 208.5 Hv.

THE EFFECT OF THICKNESS REDUCTION ON MICROSTRUCTURE AND MECHANICAL PROPERTIES OF COLD WORK AND CRYOROLLED LOW CARBON STEEL

ABSTRACT

The low carbon steel has very extensive application in the industrial field. Its limited strength becomes the obstacle to develop its potential applications. In recent years, ultrafine grained (UFG) structural materials have been studied aggressively, because they are expected to provide superior mechanical properties. The present work investigated the microstructure and mechanical properties of cold rolled and cryorolled low carbon steel at different thickness reduction. Firstly, low carbon steel was heat treated at 550 ºC for 60 minutes. For cryorolling, sample was dipped in the liquid nitrogen for 10 minutes before rolled. Then both cold rolled and cryrolled samples were rolled at different thickness reduction; 50%, 70%, 80% and 90%. Microstructure, crystallite size, strain, dislocation density and mechanical properties for both cold rolled and cryorolled low carbon steel at different thickness reduction were investigated using scanning electron microscope (SEM), X-ray Diffraction (XRD), Vickermicrohardness and tensile test. All the samples showed severely deformed grains which were elongated along the rolling direction. Cryorolled low carbon steel showed the finest grain size compared to the cold rolled low carbon steel with average grain size of 8.06 µm at 90% reduction. The pearlite started to break at 70% thickness reduction for both cold and cryorolled low carbon steel. Amount of cementite increases with increasing thickness reduction. Cryorolled low carbon steel have finer crystallite size, higher micro strain and higher dislocation density for all reduction compared to cold rolled low carbon steel. Tensile strength, yield strength and hardness shows increasing trend while ductility decrease with increasing thickness reduction and cryorolled at 90% thickness reduction showed the highest tensile strength, yield strength and hardness with value of 828.35 MPa, 810 MPa and 208.5 Hv.

CHAPTER 1

INTRODUCTION

1.1 RESEARCH BACKGROUND

Metal industries are keep arising in term of demand. Metal with high properties and low cost are extremely studied and being developed. Low carbon steel which is known as low cost material which is widely used in industrial application such as screw machines parts which includes shafts, spindles and rods. Using low carbon steel is a good platform in producing product with high profit. Low carbon steel has lower tensile strength compared to others steels due to the lower carbon contents but it has good ductility properties. Increasing the mechanical strength of low carbon steel will highly benefits to the industrial application which contain the combination of tensile strength and ductility.

The low carbon steels are unable to be harden through heat treatment due to the low carbon content. Quenching of low carbon steel does not produce martensite needle-like structure which can increase the strength and hardness. This is due to the low carbon content for the carbon to trap in the structure. Strengthening of low carbon normally accomplish by work hardnening. Rolling is one of the methods of work hardening which can strengthening the low carbon steels by producing ultra-fine grained (UFG) microstructure. Reducing the grain size can increase the mechanical strength. Thepromising mechanical properties on rolling were interpreted in terms of characteristics of the microstructure: grain refinement, increased dislocation density, and a high fraction of high angle grain boundaries.

According to the Gopi et al.(2012), ultrafine grain (UFG) structured materials can be achieved by severe plastic deformation (SPD) techniques. However, majority of the SPD techniques require large plastic deformation and complicated procedures along with restricted geometries. The process of rolling overcomes the difficulty in producing sheet and lengthy products. However,during the rolling, dynamic recovery is prone to occur in the deformed microstructure, which reduces the strengthening effect. To circumvent these detrimental outcomes, the cryorolling, rolling under the circumstance of liquid nitrogen, is further developed(Xiong et al., 2017). Cryorolling (CR) is a unique mechanical deformation process at cryogenic temperatures by which high strength and ductility combinations can be achieved.

During the cryorolling process, suppression ofdynamic recovery and increase in dislocation density apart from grain refinement lead to significant increase in strength and hardness when compared to conventional room temperature rolling(Satish et al., 2017). An experiment was conducted by Gopi et al. (2012) on effect of rolling temperature on microstructure and mechanical properties of Al-Mg-Si alloy reveal that finer grain structures produce in liquid nitrogen rolling.The improved mechanical properties of the liquid nitrogen rolled sample is attributed to the combined effect of grain refinement, suppression of dynamic recovery and accumulation of higher dislocation density during cryorolling.

1.2 PROBLEM STATEMENTS

Ultra-fined grain structures have been studied and developed for many years. The main reason is to achieve higher mechanical properties. Many researchers have investigated the formation of UFG grain in low carbon steel by several SPD process. There are many factors can contribute to formation of UFG grain low carbon steel such as starting structure, heat treatment and thickness reduction. For example work done by Ueji et al.,(2004) was focusing on the structure which is martensite on the formation of ultrafinedgrain of low carbon steel usingthermomechanical process. In addition, (Karmakaret al.2013) also have investigated the effect of starting microstructure and heating rate on cold-rolled low carbon steel. Another work by Mostafaei and Kazeminezhad (2016) was carried out to evaluate the effect of ultra-rapid annealing of severely deformed low carbon steel on microstructure and mechanical properties.Xiong et al. (2017) had studied the annealing effect on the mechanical properties of Fe-25Cr-20Ni steel.The formation of ultrafine grain (UFG) by cryorolling on low carbon steel are not emphasized.

Many studies have been made on effect of strain on the mechanical properties. Increasing strains will achieve higher mechanical strength. For an exampleXionget al., (2017)was focusing the effect of different thickness reduction on the cryorolled AISI 316 stainless steel. The thickness reduction chosen were 30%, 50%, 70% and 90%. The strength and microhardness increase with the increasing cryorolling deformation and 90% thickness reduction shows the highest tensile strength, yield strength and microhardness. Another work by Kumar and Kumar,(2017), was carried out to study the tensile behavior of cryorolled and room temperature rolled of 6082 aluminium alloy. The aluminium alloy were coldrolled and cryorolled at 40%, 70% and 90%. The results showsed that cryorolledaluminium alloy have the higher mechanical strength for all reduction and 90% cryorolled achieve the highest tensile strength and microhardness. In addition,Paghadal et al., (2016) also studied on the cold rolling and cryorolling at different thickness reduction of aluminium alloy. The thickness was reduced for 20%, 40% and 60% at cryogenic temperature and room temperature. 60% thickness reduction shows the highest increase in tensile strength and hardness. From the above mentioned studied thickness reductionalso contributed to improve mechanical strength by formation of UFG grain in several material. Studied on thickness reduction for low carbon steel has been reported by Ueji et al.,(2002)using cold rolling process. The effect of rolling reduction ranging from 25% to 70% on the ultrafine grained structure and mechanical properties of low carbon steel. The tensile strength and yield strength increasing with thickness reduction. 70% shows the highest tensile yield strength. However, research on thickness reduction of low carbon steel by cryorolling still have not been expolored.

Therefore, this study was focused on the effect of different thickness on microstructure and mechanical properties of cryorolled low carbon steel and compared to cold working process.

1.3 RESEARCH OBJECTIVES

The objectives of this research are as follow:

I. To determine the different between cold rolling and cryorolling in terms of microstructure and mechanical properties

II. To investigate the effect of thickness reduction on microstructure and mechanical properties of low carbon steel using cold and cryo rolled.

1.4 SCOPE OF WORK

The research focused on the effect of thickness reduction on the low carbon steel by cold rolling and cryorolling. The material used in this experiment was low carbon steel with initial thickness of 5mm. Optical Emission Spectroscopy (OES) was used to characterize the as received low carbon steel for element percentage while X-Ray Diffraction was used to determine the crystallite size for dislocation density calculation. The manipulated variables in there experiment would be the percentage thickness reduction and also the rolling temperature. The low carbon steel was rolled to 50%, 70%, 80% and 90% thickness reduction for both cold rolling and cryorolling. Hardness test was conducted using Vickers Micro hardness and the average value were recorded. Meanwhile, tensile test was conducted usingInstron Universal Testing Machine. For microstructural observation, all the samples were mounted, ground, polished and etched before analyzed. The etchant used was 2% Nital solution with 25 seconds of etching time. Scanning electron microscope (SEM) was used to study the fracture morphology of low carbon steel.

CHAPTER 2

LITERATURE REVIEW

2.1 INTRODUCTION

Metal is a highly demand material for engineering applications purpose because of it’s good mechanical, electrical and magnetic properties. The required properties for specific application are keep studied and produced. The search for materials with higher strength to weight ratio is one of main topics in metal research and industry. Part of this research is motivated by the Hall-Petch relationship which says that the strength of a material is inversely related to square root of the grain size. Among the metal, steels are basic and the most important materials in human civilization. Improving mechanical properties of steels to achieve excellent strength has always been attention in metallurgy research.

2.2 LOW CARBON STEEL

Steels are iron-carbon alloys that may contain appreciable concentration of other alloying elements. The mechanical properties are sensitive to the content of carbon, which is normally less than 1.0 wt%. Some of the more common steels are classified according to carbon concentration; low, medium, and high-carbon types. Low carbon steels which are produced in the greatest quantities contain about less than 0.25 wt% C. As mentioned Neal Litherland (2018) on the properties of low carbon steel, steel should have less than 0.3 percent carbon to be considered as low carbon steel. Microstructures of low carbon steels consists of ferrite and pearlite constituents. As a consequence, these alloys are relatively soft and weak but have outstanding ductility and toughness. In addition, they are machinable, weldable, and of all steels, are the least expensive to produce. Low carbon steel usually used straight from the forming process, either in hot or cool forming. This is due to the workable and easiest to form property of low carbon steel. Due to the low carbon content of low carbon content steel, it is among the best weldability of any metal. Increasing carbon content makes the steel increase in hardness. However, as the metal gets harder with more carbon contain, they are more prone to cracking when welded. The low carbon steel does not face this problem due to the low carbon content. In term of formability, low carbon steel is easier to form into certain shapes through methods such as pouring, pressing and molding. The ability of low carbon steel to be turned into a number of different forms makes it highly versatile material. Low carbon steel is highly used for case hardened machine parts, chains, rivets, stampings, nails, wire and pipes. Other applications of low carbon steels include automobile body components, structural shapes, and sheets that are used in pipelines, buildings, bridges and tin cans (William et.al, 2015).

Low carbon steel cannot be strengthened by heat treatment. It can be strengthened through cold working. The low carbon material is relatively soft and weak but it has high ductility and toughness. Table 2.1 shows the chemical compositions of different types of low carbon steel. The carbon percentage increases in order of 1010, 1018, 1020 and 1022 carbon steels. All the steels contain carbon percentage lower than 0.25% which satisfied the low carbon steel’s carbon content.

Table 2. 1: Chemical compositions of low carbon steel (Coburn-Myers, 2018)

Alloy	UNS Designation	C (max)	Mn (max)	P (max)	S (max)	Si	Cr	Ni	Mo	Other Element
1010	G10100	0.08-0.13%	0.3-0.6%	0.04%	0.05%	-	-	-	-	-
1018	G10180	0.14-0.20%	0.6-0.9%	0.04	0.05%	-	-	-	-	-
1020	G10200	0.17-0.23%	0.3-0.6%	0.04	0.05%	-	-	-	-	-
1022	G10220	0.17-0.23	0.7-1.0%	0.04	0.05%	-	-	-	-	-

Table 2.2 shows different types of low carbon steel with different carbon content percentage principal design with its applications. From Figure 2, strength of low carbon steel 1010 is the lowest while 1022 is the highest due to the higher carbon content compared to the others.

Table 2. 2: Low carbon steel's principal design features and applications (Coburn-Myers, 2018).

Alloy	UNS Designation	Principal Design Features	Application
1010	G10100	1010 is a plain carbon steel with a nominal 0.1% carbon content. It is a relatively low strength steel, but it may be quenched and tempered for increased strength.	Used for applications such as cold headed fasteners and bolts.
1018	G10180	1018 is among the most available grades in the world. Despite its unimpressive mechanical properties, the alloy is easily formed, machined, welded, and fabricated.	Often employed in high volume screw machine parts applications, such as shafts, spindlesand an incredibly wide variety of component parts.
1020	G10200	1020 is a commonly used plain carbon steel. It has a nominal carbon content of 0.20% with approximately 0.50% manganese. It has a good combination of strength and ductility.	Used for simple structural applications such as cold formed fasteners and bolts.
1022	G10220	1022 has a slightly higher carbon and manganese content plain carbon steel than 1020. It is used for its somewhat greater strength while still having good ductility.	Used for moderate strength structural applications such as cold formed fasteners and bolts.

From Table 2.3, 1022 has the highest tensile strength, yield strength and hardness compared to the others. The elongation of 1022 carbon steel is low due to the low in ductility. Carbon steel 1010 has the lowest tensile strength, yield strength and hardness compared to the other types of carbon steels. From the properties shown in the Figure, 1020 carbon steel has the best combination of strength and ductility because it has high tensile strength and high ductility.

Table 2. 3 Mechanical properties of low carbon steel (Coburn-Myers, 2018).

Alloy	UNS Designation	Typical Mechanical Properties (ASTM E8)				
		Tensile (ksi)	Yield (ksi)	Elongation	Reduction of Area (%)	Brinell Hardness
1010	G10100	53	44	20	40	105
1018	G10180	64	54	15	40	126
1020	G10200	64	54	24	54	126
1022	G10220	69	58	15	40	137

According to Azom, (2012), AISI 1018 mild/low carbon steel has a good weldability properties. It have a good combination of toughness, ductility and strength. The AISI 1018 hot rolled provided a higher mechanical properties, machining characteristics and Brinell hardness.(Analysis, 2018) state the same properties which low carbon steel have a good balance of strength, ductility, toughness, excellent weldability, good formability and machinability. Low carbon steel AISI 1018 is easy to cold form, bend and braze. The magnesium content in AISI 1018 contribute to a higher strength and hardness compared to the other carbon steels(DB&S Steel, 2018).

According to (Coburn-Myers,2018), low carbon steel is relatively soft and week, but has ductility and toughness. Compared to medium and high carbon steel, low carbon steel have a lower tensile strength and hardness. Although it has lower strength, the total elongation is much more lower compared to other carbon steels which means it has a better ductility.

2.3 STRENGTHENING MECHANISM

Various strengthening mechanisms have been applied, such as grain refining, transformation strengthening, and work hardening. The work hardening was normally applied to strengthen the structural bulk material. The bulk properties of the steel are modified using the cold deformation, which involves a plastic deformation of the material. The common cold deformation process used are drawing, extrusion, rolling and etc. In cold deformation, the tensile strength and hardness of the steel increase, while ductility decrease. Rolling is a process which reducing the thickness or changing the cross-section of a long work piece by compressive force applied through a set of rollers. It is developed in late 1500s. It is often carried out at elevated temperature first (hot rolling) to change coarse-grained, brittle and porous ingot structures to wrought structures with finer grain sizes and enhanced properties (Dieter, 1988).

Figure 2. 1: Rolling process(Dieter, 1988)

The metal plate is roll within two or more rollers at the normal temperature. The force applied by the roller makes the thinning of metal. The grain size was reduced and makes the metal higher in strength. Various studies were carried by many researchers on cold rolling of low carbon steel. A study on the effect of thickness reduction by cold rolling on mechanical properties of low carbon steel was conducted by (Ueji et al., 2004). In their studied, they able to produce ultrafined grain structure of martensite and increased the mechanical properties of the plain low carbon steel at different rolling reduction ranging from 25 to 70% reduction.

The cold rolling able to produce ultrafined grain by heating the low carbon steel at the inter-critical of low carbon steel on 80% cold rolled samples have been made. The starting microstructures are namely ferrite-pearlite, ferrite-blocky martensite and ferrite-fibrous martensite.Due to cold rolling process, more uniformly distribution of martensite island, ferrite-fibrous martensite gave an excellent combination of strength, ductility and strain hardening ability (Karmakaret al.2013). The effect of different annealing temperature (350 $^{\circ}$C to 650 $^{\circ}$C) of 80% cold rolled low carbon steel on the microstructure and mechanical properties was investigated by Karmakar et al. (2013). Increasing of annealing time reduce the strength but ductility increases. In addition to the processes mentioned before, there are other processes that modify the steel's properties by changing its internal structure; this is called, severe plastic deformation (SPD).

2.4 SEVERE PLASTIC DEFORMATION (SPD)

Severe plastic deformation is defined as intensive plastic straining under high imposed pressure. It has received increasing attention due to extremely fine grained structure and very high strength. The advantage of SPD is the development of materials having good machinability, forgeability and formability at potentially low processing cost. Severe Plastic Deformation (SPD) processes to produce ultrafine-grained materials have intensively been studied. Ultrafine grained materials produced by severe plastic deformation processing usually have dislocation densities, non-equilibrium grain boundaries and other structural features associated with plastic strain. Material possesses an ultrafine grained structure with predominantly high angle grain boundaries and these grain boundaries contain a high density of dislocation indicating their non-equilibrium structure.

A lot of research have shown that ultrafine grain (UFG) can be introduced in wide range of materials from pure metals to alloy, intermetallic compound and metal matrix composite(MMC). Majority of research in severe plastic deformation were carried out by equal channel angular pressing (ECAP) process.

Materials produced by SPD techniques usually have grain sizes in the range of 100–1000nm. However, they have subgrain structures, such as subgrains, dislocation cells and X-ray coherent diffraction domains (crystallites), which are often smaller than 100 nm. Therefore, they can be called nanostructured materials (Zhu et al. 2004). Ultrafined grain are expected to enhance mechanical properties such as high strength, super-plasticity and low ductile-brittle transition. The materials possess unusual and extraordinary mechanical and physical properties high strength, high toughness at freezing temperature and high-strain rate superplastic behaviour(Bergwerf, 2007).The common SPD processes used for bulk materials include equal-channel angular pressing (ECAP), high-pressure torsion (HPT), and accumulative roll bonding (ARB).

Equal channel angular pressing (ECAP)is one of the processing tool for introducing the grain refinement to the sub-micrometer level in variety types of metals. In ECAP the sample is in the form of rod or bar which is then pressed through a die within a channel that is bent internally through an abrupt angle. The results by experiment shows that after single ECAP pass exhibit the microstructure consisting of bands of elongated sub-grains and these bands lie oriented parallel to the primary slip system. The high dislocation density is introduced in early stage of straining which leads to formation of inter-granular structure consisting of cells with thick cell walls and low angle misorientation. The thickness of the wall decrease as strain increase by recovery through dislocation annihilation. This will leads to an excess dislocation of only one sign on each boundary and formation of ultrafine grain separated by high-angle non-equilibrium boundaries (Langdon, 2007).

Compared to ECAP, high-pressure torsion (HPT) processing leads to smaller grain which makes it attractive. The HPT sample is in the form of a thin disk which is placed between two massive anvils. The sample subjected to a pressure, P and then torsionally strained through rotation either the lower or upper anvil (Huang and Langdon, 2013). From the HPT process done on Cu-Zn alloy, after ¼ turn, coarse grain are seen in the center of the disk but a clear grain refinement can be observed near the edge of the disks. After 10 turns, both the center and edge of the disk shows high grain refinement. HPT processing shows the potential for achieving excellent grain refinement (Sabbaghianrad et al., 2014).

Accumulative roll-bonding (ARB) has the ability to produce continuous ultrafine grain sheets in large quantities compared to ECAP and HPT. However, ARB process also has its own weakness which is difficulty to control the bonding quality between two layers after 50% reduction ration in each pass. A good quality of interface bonding requires a reduction ratio more than 70%. From study by (Sabbaghianrad et al., 2014), they successfully produced ultrafine grain of pure aluminium sheets using ‘four-layer accumulative roll bonding’ (FL-ARB) techniques. The results shows the ability of this technique to produce ultrafine grain at room temperature with good interface bonding strength.

In conventional metallurgy such as rolling, forging and extrusion, small grained structures materials are developed through proper thermo-mechanical processing methods. Nevertheless, these procedures have some drawbacks because separate processing routes are required for each different alloy composition and these method yield materials with grain sizes in the range of ∼1–10μm (Langdon, 2007).

Grain refinement is the major hardening technique that can be adopted to enhance strength in low carbon steel. Several researchers has successfully produced low carbon steel with ultrafined grain using equal channel angular pressing (ECAP), high pressure torsion (HPT), and rolling process.

An ultrafine grained low carbon steel was successfully fabricated by (Wang et al., 2005) using equal channel angular pressing (ECAP) at room temperature after pressed to a maximum of 10 passes. The results shows an elongated substructure and increment of tensile strength to 1200MPa. The ductility of the pressed sample increased after annealing at 773 K for 1 hour.

Effect on microstructure and hardness of AISI 1020 low carbon steel was studied by (Marulanda Cardona et al., 2017). A cylindrical sample with 10mm diameter and 0.8mm length were tested with high-pressure torsional (HPT) and the results were recorded. The crystallite size decrease with increasing number of turns while the microstrain keep increasing. After 5 turns, the ferrite and pearlite were in elongated grain shape. The microstructural refinement can be observed as number of turn increases which makes original grain compressed with increasing torsion applied. The hardness results also shows increment in both center and outer region.

The limitations of SPD processes are it involve great amount of plastic deformation, special procedures, high cost for tooling, complications in design and fabrication of quite small quantities of materials. This limitation has motivated the growing attention in the improvement of other SPD methods such as cryorolling to produce UFG materials which would require less plastic deformation for formation of UFG structure.

2.5 CRYOROLLING

Cryo-rolling is a process which is carried out under cryogenic temperature. Cryogenic temperatures are as being temperature below 120K (-244 $^{\circ}$F, -153 $^{\circ}$C) as defined by Cryogenic Society of America. Cryogenic rolling is used to improve the strength and hardness of the materials. Grain size of the material can be improved or reduced by using the cryo-rolling. Besides improving mechanical strength, cryogenic processing can makes change to the crystal structures of materials. Effect form rolling can result in brakage of microstructure. These changes are to enhance the abrasion resistance and fatigue resistance of materials (Shivkumar et.al., 2016).

Mechanism for grain size refinement can be explained based on a model proposed by (Mishraet al., 2005) as shown in Figure 2.2. During cryorolling process, the dislocation are not randomly distributed. As the metals under rolling, the dislocation will accumulate at dislocation walls and the regions surrounded by these dislocation wall have relatively low density of dislocation. When the grain deform result from rolling process, the grains subdivided together with dislocation wall act as barriers to dislocation movement. As deformation proceeds, the sub-grains progressively change into grain boundaries. These elongated sub-grains plastically deformed causing further break-up and formed equiaxedmicrograins.

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Figure 2. 2: Schematic diagram of microstructural evolution occur during severe plastic deformation. (a) homogeneous distribution of dislocation, (b) elongated cell formation, (c) dislocation obstructed by sub-grain boundaries, (d) destruction of elongated sub-grains and (e) reorientation of sub-grain boundaries and development of UFG structures (Mishra et al., 2005)

Cryo-rolling has the high potential for producing bulk ultra-fine grain materials which can enhanced the material properties. The cryogenic rolling deformation requires less plastic deformation achieving ultra-fine grain(UFG) (Stevens, 2007) compare to other severe plastic deformation(SPD) process. In cryorolling, the material will be dip into the liquid nitrogen (-190 $^{\circ}$C) and hold it a few minutes before doing the rolling process. The main advantages of using cryo-rolling are achieving and ultra-fine grain structure which improve a strength, ductility and micro-structure as well compared to cold rolling process. Moreover, handling process of the material is easier in cryo-rolling compared to hot rolling process. A desirable ductility can be achieved if annealing process is done after cryorolling and less plastic deformation required to form achieve ultra-fine grain compared to severe plastic deformation(SPD) which need large plastic deformation (Shivkumar et.al., 2016).

Several studies have successfully produced ultrafine grained for stainless steel (Xiong et al., 2017), aluminium alloy (Satish et al., 2017), copper alloy (Han et al., 2015) and titanium alloy (Chuvil'deev et al., 2017).

Nano-sized grains microstructure of pure copper were achieved in a study done by (Han et al., 2015) using Equal Channel Angular Pressing (ECAP) process. The average size of the grains were about 320 nm and 300 nm after 4 and 8 passed of ECAP. The result from the grain refinement were increased in tensile strength from 210MPa to 420MPa.

Besides steels, copper alloy and aluminium alloy, research on mechanical properties of ultra-fined grain titanium alloy was done by (Chuvil’deevet al., 2017). The process used by them to achieved ultra-fined grains was by using Equal Channel Angular Pressing (ECAP). The results shows the increasing of tensile strength, micro hardness and good ductility. The tensile strength of course grain increased from 730MPa to 1000-1010MPa for UFG titanium.

Figure 2. 3: Stress-strain curve for course grain(CG) and UFG of titanium (Chuvil’deevet al., 2017).

A study of tensile behavior of cryo-rolled and room temperature rolled was done on 6082 Al alloy by Kumar and Kumar (2017) . The research was done with 40%, 70% and 90% thickness reduction for cryorolling and cold rolling. The result shows that cryorolling samples have a better mechanical strength compared to the room temperature rolling. The of rolling temperature on the mechanical properties and microstructural evolution towards an Al-Mg-Si alloy was studied by (Gopi et al., 2012). The materials were rolled up with three different temperature which were at room temperature, liquid propanol and liquid nitrogen. From the result, it shows that material which are rolled with liquid nitrogen produced a superior properties with a good ductility compare to the others.

In a paper study on cryorolling treatment for the 316LN austenitic SS on the structural evolution and mechanical properties, the strength and microhardness of 316 LN austenitic SS increases significantly with the increasing cryorolling deformation. However the total percentage elongation reduced with cryorolling process. The cryorolled sample produced nano-grained structure with the generation and interaction of high dislocation density. Figure 2.4 shows the TEM result for this experiment. High dislocation density, deformation twins and deformation-induced martensites can be seen after deformation with small strain as shown in Figure 2.4(a). The dislocation density saturation and quantity of deformation twins increased significantly with the increasing strains. The width of martensite lath decrease from 200 nm (at strain of 30%) to 80 nm (at strain of 70%) as can be seen from Figure 2.4 (b-c)(Xiong et al., 2017). The martensitic grain size were ranging from 30 nm to 50 nm as in Figure 2.4 (d).

Figure 2. 4: TEM Image of 316 Austenitic Stainless Steel After Cryorolling: (a) After 30% Deformation, (b) After 50% Deformation, (c) After 70% Deformation and (d) After 90% Deformation(Xiong et al., 2017)

2.6 MICROSTRUCTURAL AND MECHANICAL PROPERTIES OF COLD ROLLING AND CRYOROLLING

2.6.1 Influence of Cold Rolling and Cryorolling on Microstructure

The microstructure of sample was essential to determine the properties of the sample produced. The pearlitic low carbon steel was found to refine the pearlite matrix and produces long and continuous cementite flakes as shown in Figure 2.4. The refinement of the pearlite matrix will result in decreasing of the inter-lamellar spacing, thus makes dislocation movement is prevented and leads to hardening of the low carbon steel (Kumar et al., 2017).

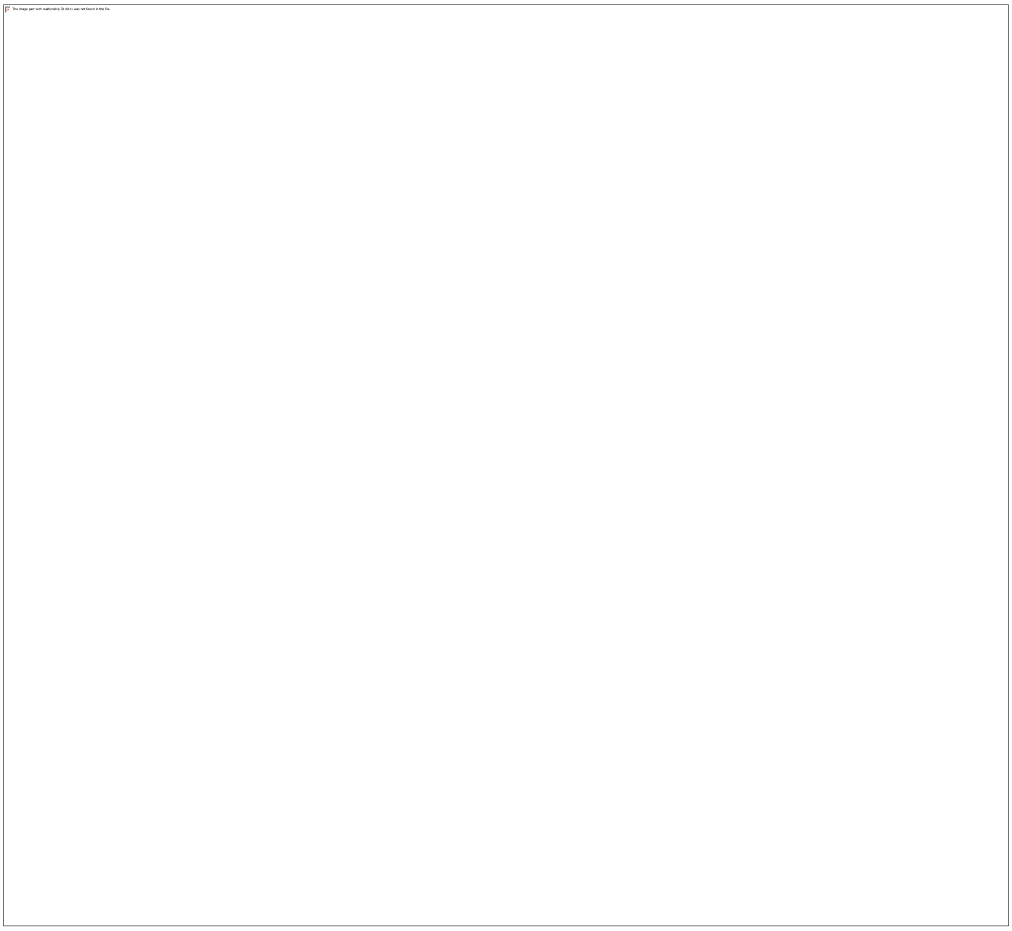

Figure 2. 5: Electron micrograph of low carbon steel brake disc (C50) non-cryotreated and cryotreated(Kumar et al., 2017)

A study was made by (Ye et al., 2014) on producing nanostructured 304 stainless steel by rolling at cryogenic temperature. The EBSD results shows that the average grain size after rolled at cryogenic temperature reduced to about 200nm to400nm compared to the sample rolled at room temperature which result in average grain size of 800nm.

The extent of fineness in the grain size and structure was improved by reducing the rolling temperature. The presence of finer grains in the cryogenic rolled sample is due to the suppression of dynamic recovery during deformation (Gopi et al., 2012).

2.6.2 Influence of Cold Rolling and Cryorolling on Hardness Properties

Hardness is one of the important mechanical properties which a metal must have. Hardness value will determine how much a metal can resist from deformation. A soft metal will have a lower hardness which make it lower in physical properties. Hardness is measured by the depth of indentation. Cryorolling process can increase the hardness value of metals. Several researchers have proved that the presence of ultrafine grain (UFG) by cryorolling method can increase the hardness properties (Paper and Delhi, 2016).

An investigation was conducted to study the mechanical properties between cold rolled and cryorolledaluminium alloy as shown in Figure 2.6. Hardness of the rolled samples was measured using Rockwell Hardness (HR) and the result clearly shown that the hardness value for the cryorolled samples are higher compared to the cold rolled samples in every thickness reductions. The thickness reduction of 60% showed the highest different increase in hardness between cryorolled and cold rolled samples. The hardness value for cryorolled sample after 60% thickness reduction is 49.45 HR compared to cold rolled sample which is 36 HR. This show that rolling in cryogenic temperature gave rise in hardness value (Paper and Delhi, 2016)

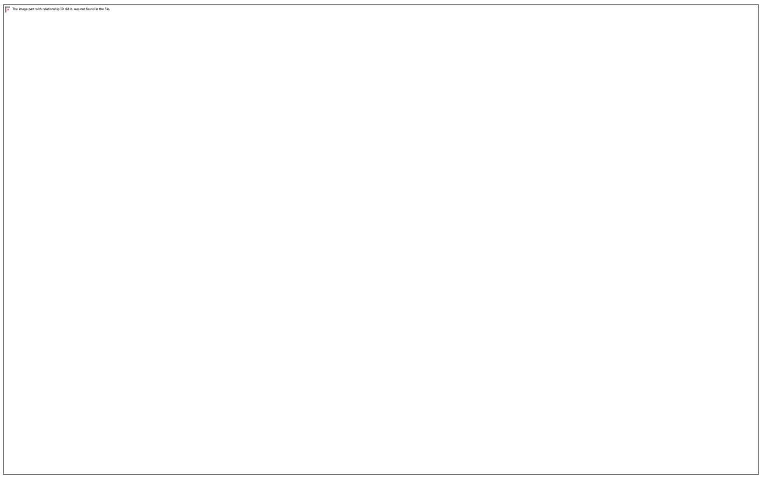

Figure 2.6 Hardness value comparison between cold rolled and cryo rolled in different thickness reduction(Paper and Delhi, 2016)

The increased in the hardness value of cryorolled sample is due to the higher dislocation density and the formation of sub microcrystalline structure. They said that themechanism that dynamic recovery was effectively suppressed during cryorolling which increase the dislocation density. Hardness increased due to the restriction of dislocation mobility and misorientation of grains from the formation of ultrafine grins in the cryorolled sample (Paper and Delhi, 2016).

Dislocation density, grain size and dislocation mobility are the main factors which affect the micro hardness. The amount of deformation can anchor the dislocation effectively avoid its motion. Large amount of defect such as entanglement of dislocation during process increases dislocation density and high work hardening capacity makes the hardness increases. Moreover, the refinement and nano crystallization in microstructure lead to increase in micro hardness. These are the factor that contributed to increase in the micro hardness after cryorolled of AISI 316 stainless steel. The micro hardness increased from 170 Hv to 415Hv at strain of 30% and 528 at strain of 90%. So, rolling at cryogenic temperature will definitely increase the hardness value of metal (Xionget al., 2017).