EFFECT OF CRYOROLLING ON THE MICROSTRUCTURES, MECHANICAL PROPERTIES AND CORROSION BEHAVIOUR OF LOW CARBON STEEL USING MARTENSITE STARTING MICROSTRUCTURE

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UNIVERSITI SAINS MALAYSIA

2020

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UNIVERSITI SAINS MALAYSIA

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By

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Dissertation submitted in fulfilment of
the requirements for the degree of Master of Science
Materials Engineering
Universiti Sains Malaysia

SEPTEMBER 2020

DECLARATION

I hereby declare that I have conducted, completed the research work and written the dissertation entitled "**Effect of cryorolling on the microstructures, mechanical properties and corrosion behaviour of low carbon steel using martensite starting microstructure**". 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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ACKNOWLEDGMENT

In the name of Allah, the Most Gracious, Most Merciful, Praise Almighty for the blessed and peace upon Prophet Muhammad S.A.W.

Alhamdulillah, praise to Allah the Almighty for the strength and His blessing in completing this project. I would like to suppress my most gratitude to Allah s.w.t for giving me health and inspiration to complete this thesis as scheduled. I would also like to express my gratitude to University Science Malaysia and School of Materials and Mineral Resources Engineering for providing me with sufficient equipment. I would like to give a great sincere appreciation to my supervisor, Dr. Anasyida Abu Seman, for her excellent supervision and constant support. Her continuous help of constructive comments, suggestions and advises throughout the research and experimental works have contributed to the success of this project. Next, I want to thank deeply to Mrs. Siti Aminah Zakaria, a PhD student who is helping me to learn and understand more about my research project. Without their right direction, it is impossible for me to complete my master program and this thesis. My grattitude also goes to all the School of Materials and Mineral Resources staffs and technicians, who have directly or indirectly involved in my research, in particular Mr. Azam, Mr. Mokhtar and Mr. Azrul. The research could not have been successful without their enthusiastic participation and feedback. Special thanks to Jabatan Perkhimatan Awan (JPA) for giving me the opportunity and financial support to pursue my education. Lastly, I would like to express my very profound gratitude to my parents, lecturers and friends for their endless love, prayers and continuous encouragement through the process of researching and writing this thesis. Without their encouragement, it would be nearly impossible for me to finish this project. Thank you.

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LIST OF SYMBOLS

%	percentage
%C	percentage of carbon
%EL	elongation percentage
wt.%	weight percentage
°C	degree celcius
D	spacing between the layers of atoms
HV	hardness Vickers
K	kelvin
kg	kilogram
g	gram
min	minute
MPa	mega pascal
cm	centimeter
mm	milimeter
µm	micrometer
nm	nanometer
α	alpha ferrite
γ	austenite
λ	wavelength of ray
θ	angle between incident rays and surface of the crystal
T_m	melting temperature
A/cm^2	current per centimetre square

LIST OF ABBREVIATIONS

AHSS	advance high strength steel
ARB	accumulative roll bonding
ASTM	American society for testing materials and minerals
BCC	body center cubic
BCT	body centered tetragonal
C	carbon
CE	counter electrode
CG	coarse grain
Cr	chromium
CR	cryorolling
Cu	copper
DP	dual phase
ECAP	equal channel angular pressing
EL	elongation
F	ferrite
Fe	ferrous
FCC	face center cubic
FG	fine grained
FWHM	full width at half maximum
HPT	high pressure torsion
HSS	high strength steel
ICDD	international centre of diffraction data

IF	interstitial free steel
LCS	low carbon steel
M	martensite
Mn	manganese
NaCl	sodium chloride
Ni	nickel
OCP	open circuit potential
OES	optical emission spectrometer
OM	optical microscope
P	phosphorus
RE	reference electrode
S	sulfur
SCE	standard calomel electrode
SPD	server plastic deformation
Ti	titanium
TMP	thermomechanically controlled process
TS	tensile strength
TWIP	twining induced plasticity steel
UFG	ultrafine-grained
UTS	ultimate tensile strength
WE	working electrode
XRD	X-ray diffraction
YS	yield strength

KESAN GELEKAN KRIO TERHADAP MIKROSTRUKTUR, SIFAT-SIFAT MEKANIKAL DAN KELAKUAN KAKISAN KELULI KARBON RENDAH MENGGUNAKAN MARTENSITESEBAGAI PERMULAAN MIKROSTRUKTUR

ABSTRAK

Sifat-sifat keluli berkarbon rendah selalunya disesuaikan mengikut sesuatu aplikasi yang khusus dengan cara memanipulasi mikrostruktur sedia ada. Ciri-ciri mikrostruktur adalah termasuk morfologi, pecahan isipadu martensite dan saiz ira. Ciri-ciri mirkrostruktur tersebut boleh di ubah suai dengan melaras masa rendaman dan suhu dalam zon interkritikal ketika rawatan haba dijalankan. Kajian semasa ini mempunyai matlamat untuk mengkaji kesan interkritikal penyepuhlindapan diikuti dengan gelekan krio terhadap mikrostrutur, sifat-sifat mekanikal dan tingkah laku kakisan keluli berkarbon rendah yg mengandungi karbon sebanyak 0.06%. Keluli berkarbon rendah menjalani interkritikal penyepuhlindapan dengan teknik pelindapkejutan pertengahan dan langkah pelindapkejutan pada suhu 750°C, 800°C, 830°C dan 850°C dengan pelbagai masa rendaman (3, 5, 10 dan 15 minit) dan diikuti oleh gelekan pada suhu kriogenik (gelekan krio) pada pengurangan ketebalan sebanyak 90%. Ciri-ciri mikrostruktur dan sifat mekanik keluli berkarbon rendah yang digelek krio dikaji menggunakan mikroskop optik (OM), pembelauan sinar-X (XRD), mikroskop elektron imbasan (SEM), mikrokekerasan Vicker dan ujian tegangan. Martensite dengan morfologi yang berserabut yang diperoleh dari pelindapkejutan pertengahan mempunyai nilai kekerasan yang lebih baik berbanding martensite dengan blok morfologi yang terhasil dari langkah pelindapkejutan. Pecahan isi padu martensite yang lebih banyak dan saiz mikrostruktur yang lebih halus didapati daripada sampel keluli berkarbon rendah yang menjalani teknik pelindapkejutan pertengahan. Berdasarkan pada nilai kekerasan (404.6 Hv), sampel yang menjalani interkritikal penyepuhlindapan melalui teknik pelindapkejutan pertengahan pada suhu 830°C (5 minit) telah dipilih untuk analisis pengurangan ketebalan yang berbeza (50%, 70% dan 90%). Kekerasan dan kekuatan tegangan menunjukkan peningkatan nilai dengan pengurangan ketebalan dan nilai tertinggi diperolehi pada 90% pengurangan dengan nilai 429.4 Hv dan 1537 MPa, masing-masing. Sampel digelek krio pada 90% pengurangan mempunyai saiz kristalit yang paling kecil (13.70 nm) dan terikan kekisi yang tertinggi (74.6 x 10^{-3}). Rintangan kakisan berkurang dengan pengurangan ketebalan, dan kadar kakisan yang tertinggi didapati pada sampel yang digelek krio pada 90% pengurangan ketebalan dengan nilai 5.968 mm/year.

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ABSTRACT

The properties of low carbon steel are often tailored to suit specific application through the manipulation of microstructure. The microstructural features including morphology, martensite volume fraction and grain size. Such microstructural features can be changed by adjusting the soaking time and temperature within intercritical zone during heat treatment. The present works aims to study the effect of intercritical annealing followed by cryorolling on the microstructure, mechanical properties and corrosion behavior of low carbon steel with 0.06 wt% C. Low carbon steel underwent intercritical annealing through intermediate quenching and step quenching technique at 750°C, 800°C, 830°C and 850°C with various soaking times (3, 5, 10 and 15 minutes) and followed by rolling at cryogenic temperature at 90% reduction. The details microstructural characteristics and mechanical properties of cryorolled low carbon steel were investigated using optical microscope (OM), X-ray diffraction (XRD), scanning electron microscope (SEM), Vicker microhardness and tensile test. A fibrous martensite morphology obtained from the intermediate quenched exhibits much better hardness compared to the blocky martensite morphology produced by step quenching treatment. A higher fraction of martensite volume and a much finer microstructure were obtained in intermediate quenched low carbon steel. Based on the hardness value (404.6 Hv), sample intercritically annealed via intermediate quenching process at 830°C (5 minutes) was chosen for different thickness reduction (50%, 70% and 90%) analysis. Hardness and tensile strength showed an increasing value with increasing percentage reduction, and the highest value obtained at 90% reduction with 429.4 Hv and 1537 MPa, respectively. The cryorolled sample at 90% reduction has the smallest crystallite size (13.70 nm) and highest lattice strain (74.6 x 10^{-3}). Corrosion resistance decreases with thickness reduction, and the highest corrosion rate attained at 90% reduction with 5.968 mm/year.

CHAPTER ONE

INTRODUCTION

1.1 Background Research

Low carbon steel is widely used in most manufacturing and production industries due to excellent combination properties such as flexible, weldable, deformable and fracture resistance (Krauss 2015). It was used as structural components and beams for buildings, bridges, pipelines, and car bodies. Furthermore, low carbon steel is also used to produce machine parts which are not exposed to high mechanical strength, such as shafts, gears, pins, and standard screws (Selcuk et al., 2003). Low carbon steel has limited strength which limits its application in modern manufacturing. Over the years, extensive work has been done in metals research and industry to find material with high strength to weight ratio that satisfy advanced technologies, particularly automotive industry.

The number of developers in the automotive industry has increased significantly, resulting in heavy competition and new technologies. Nowadays, the desired properties of steel in the automotive industry is mostly based on ultra-high strength to keep passengers in the safety zone and maximize fuel consumption by weight. Low carbon steel is therefore required to improve its properties. On average, 900 kg of steel is used in every vehicle, with 40% of mild still used as the body structure such as panels, doors, and trunk closures, which required high strength to absorb energy in the area of the crash zone. On the other hand, 23% of cast iron used for the engine block and machinable carbon steel for wear resistance gears. A further 12% of rolled high-strength steel strip was used in the suspension and the remainder is found in the fuel tanks, braking systems and wheels (Hovorun et al., 2017). However, the high strength steel (HSS) and advance high strength steel (AHSS) have been replacing mild steel for the last decade in the automobile bodies regardless that these two materials are high in cost. The typical, recently introduced vehicle contains about 30% of HSS and AHSS.

In these demands, it is essential to improve the long-last handling capabilities and the safety factor of low carbon steel. Various ways have been established to create excellent properties as traditional low-strength steel did not meet the requirements of modern manufacturing and production industry. The surface properties of low carbon steel can be enhanced through surface hardening. This include several method that involve carburizing, carbonitriding or boronizing (Izciler and Tabur 2006), which enhances the formation of a surface layer that improves properties such as corrosion resistance, wear, and friction. In these techniques, however, only surface of the material is treated. Alternatively, the modification of bulk properties of carbon steel can be carried out using a drawing process which reduces the section of a rod and involve plastic deformation of the material (Atienza et al., 2005). Even though this method improves the hardness and tensile strength but the impact resistance and ductility were decreased. Other strengthening methods commonly used in the metals processing industry to enhance mechanical properties are solid solution strengthening, strengthening by introducing a second phase, precipitation strengthening and grain refinement. Among the techniques, the grain refinement is considered most effective because it can improve both the strength and the fracture resistance.

It is well known that ultrafine-grained (UFG) shows excellent balance between strength, toughness and ductility. Recently, advanced severe plastic deformation (SPD) is an effective method for grain refinement of metals and alloys. Under this method, the particle size of materials can be reduced to 100-1000nm and structure such as sub grains, crystallites and dislocation are reduced to 1-100nm (Zhu and Langdon, 2004). There are several different types of SPD techniques which are accumulative roll bonding (ARB), equal-channel angular pressing (ECAP), mechanical milling (MM), high pressure torsion (HPT) and cryorolling. Cryorolling is the simplest method to form ultra-fine grained (UFG) structures in bulk metals and alloys and it employ a relatively low accumulated strain with less force to deform the materials as compared with other SPD techniques (Yuan et al., 2018).

Cryorolling was originally introduced by Wang et al., (2012) and it involve severe cold rolling process at liquid nitrogen temperature to form UFG structure in bulk metals with an average grain size of less than 1μm. Cryorolling has been carried out on a wide range of aluminium alloys. The modified grain structure of aluminium alloys from the micrometer regime down to the nanometer regime or submicrometric regime have increased its strength and toughness (Nageswara et al., 2013). In the same way, cryorolling also has been successfully employed on interstitial-free (IF) steel (Sharma et al., 2012), TWIP steel (Klimova et al., 2017), austenite stainless steel (Shi et al., 2017, Mallick et al., 2017 , Xiong et al., 2015, Roy et al., 2015) and low carbon steel (Yuan et al., 2018).

The cryorolling technique requires pre-heat treatment to change the initial microstructure or morphology, relieve internal stresses and dissolution of soluble phase before cryorolling. Some method of initial pre-treatment include annealing, quenching, and quenching followed by tempering. The pre-treated sample is then soaked in liquid nitrogen for a period of time before it is rolled between two rollers. The resulting UFG in bulk samples has improved the mechanical and corrosion properties and has enormous potential to replace some high-cost alloy steels. The mechanical and corrosion properties of low carbon steel can be significantly enhanced via cryorolling process due to the effect of grain refinement. The microstructural factor of grain size is therefore considered to be a key factor affecting almost all the properties of the mechanical and corrosion behaviour of polycrystalline metals.

1.2 Problem statement

Cryorolling has been proved to have a significant impact on grain refining compared to traditional room temperature rolling and the initial microstructure prior to cryorolling is a significant factor that will affect the formation of ultrafine-grained structure in steel as well as its final properties. Steel required pre-heat treatment prior to cryogenic deformation in order to form the desired phases. The lack of any pre-heat treatment will lead to inadequate mechanical properties (Aminah et al., 2019). Many researchers have performed a different type of pre-heat treatment prior cryorolling such as Zheng et al., (2016) which investigated the effect of cryorolling on the microstructure and mechanical properties of Fe-36Ni steel. Steel was pre-treated with annealing process at 950°C for 1 hour to form homogenized austenite microstructure. In addition, Roy et al., (2015) have reported the formation of nanostructured or ultrafine-grained austenitic AISI 304L stainless steel (SS) through cryorolling in which the steel first was solution treated at 1100°C for 1 hour. In the meantime, Mallick et al., (2017) also evaluated the effect of cryogenic deformation on 304 austenitic stainless steel but with different solution treatment parameters (1040°C for 40 minutes) prior to cryorolling.

Yuan et al., (2018) were motivated by the above-mentioned study to apply cryorolling on low carbon steel. The steels were first austenitized at 1050°C for 30 minutes, followed by water quenching to obtain martensitic starting microstructure and the effect of rolling reduction on microstructure and mechanical properties was investigated. In addition, Karmakar et al., (2013) investigated the effect of ferrite pearlite and ferrite martensite microstructure on cold rolled low carbon steel (0.1wt%C). Three different heat treatments (furnace cooling, step quenching and intermediate quenching) were employed to produce different initial microstructure prior to cold rolling. This study demonstrated that the ferrite-martensite structure showed the finest grain size (3-6 µm) compared to ferrite-pearlite (9-17 µm). Therefore, it provided the best combination of strength, ductility and strain hardenability. These desirable properties as identified by many researches are due to the existence of special microstructure in which, soft ferrite and hard martensite shows their presence in the form of ferritic matrix with martensite reinforcements (Sunil and Rajanna 2020).

Movahed et al., (2009) have investigated the tensile properties and work hardening behaviour of dual phase (DP) steels in which intercritical heat treatment through intermediate quenching technique was performed at 760°C to 840°C for 20 minutes followed by water quenching, to produce the ferrite-martensite structure. They reported that, work hardening of dual phase steels containing approximately equal amounts of ferrite and martensite phases exhibit optimum mechanical properties in terms of tensile strength, ductility and fracture energy. The initial microstructure specifically affects the formation of UFG in low carbon steel and thus its properties.

Pre-heat treatment is important to modify the initial microstructure prior to deformation. Based on previous studies on steel, an initial microstructure of ferrite+pearlite and ferrite+martensite was used. Ferrite-martensite has been identified as the best initial microstructure to improve the mechanical properties of ultrafine-grained steel. However, at present, there is only one paper reported on the cryorolling of low carbon steel using ferrite-martensite starting microstructure. Two different pre-