EFFECT OF QUENCHING AND POST HEAT-TREATMENT PROCESS TO THE MICROSTRUCTURE AND PHYSICAL PROPERTIES OF ELECTRO-CARBURISED LOW CARBON STEEL

SOW CHAN ON

UNIVERSITI MALAYSIA SABAH

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SOW CHAN ON

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I hereby declare that the material in this thesis is my own except for quotations, excepts, equations, summaries, and references, which have been duly acknowledged.

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ABSTRACT

Limited studies were performed pertaining to the post-heat treatment on low carbon steel that is carburised by electro-carburisation process. In this study, the electro-carburisation (EC) was carried out using the electrolyte mixture of sodium carbonate (Na_2CO_3) and sodium chloride (NaCl) with molar ratio of 4:1. The process was done under the temperature of 800 °C with constant voltage of 4.0 V for three hours in the carbon dioxide, CO_2 gas environment. After electro-carburised, the sample was quenched in either water or oil. Post-heat treatment such as tempering, or annealing was carried out to relieve the residual stresses. After the heat treatment processes, Vickers hardness machine was used to measure the hardness profile from surface (0 μm) towards the core. It was found that the peak hardness of all samples was located at the 100 μm from the surface. The peak hardness of the EC/water quenched sample (WQ, 702 $\pm$ 5 HV) was higher than the EC/oil quenched sample (OQ, 571 $\pm$ 3 HV). This is due to the high cooling rate of water compared to oil. The high cooling rate of water caused the carbon had inadequate time to escape or react with oxygen, but the carbon was trapped to form the martensite. Apart from that, the peak hardness of the EC/post-heat-treated sample which is tempered (WQ-T, 623 $\pm$ 9 HV; OQ-T, 495 $\pm$ 4 HV) or annealed samples (WQ-A, 571 $\pm$ 3 HV; OQ-A, 367 $\pm$ 6 HV) was lower than the EC/quenched samples (WQ and OQ) due to the internal stress was relieved and the rearrangement of structure that lowered the distortion of the steel. Changes in the microstructure such as martensite, tempered martensite and ferrite were observed by optical microscope which the formation of martensite increased the hardness of the sample. While the metal phase was examined by using X-ray Diffraction (XRD). XRD analysis showed that EC/post-heat-treated sample consisted of iron oxide which are the magnetite and hematite. Full width at half maximum (FWHM) values at the XRD peak (between 44.4° to 44.7°) showed that EC/post-heat-treated samples had lower FWHM value than EC/quenched sample, which indicated that the stress was relieved in both EC/tempered and EC/annealed sample. Elemental composition of the samples was determined by Energy Dispersive X-ray (EDX). The EDX analysis showed the carbon content on the outer surface of as-received sample, AR (2.79 wt. %); WQ (5.24 wt. %), WQ-T (5.16 wt. %), WQ-A (2.94 wt. %); OQ (4.73 wt. %), OQ-T (2.78 wt. %) and OQ-A (2.63 wt. %), respectively. Relatively, the carbon content of WQ was higher than OQ sample, whereas the EC/tempered samples had high carbon content compared to the annealed samples, but lower than the EC/quenched samples. The formation of martensite depends on the carbon content of the steel as more martensite was formed with high carbon content, this matched with high carbon content had higher hardness. The oxygen content on EC/post-heat-treated samples was more concentrated compared to other samples which provided the formation of iron oxide on its surface due to occurrence of oxidation and decarburisation. Oxidation and decarburisation happened during cooling process of tempering and annealing. High temperature during the cooling process caused the reaction between oxygen and ferrite. In short, electro-carburisation process increased the carbon content and improved the hardness of the samples. In quenching process, the water quenching provides better hardness than the oil quenching, while tempering or annealing process relieved the internal stresses that led to hardness reduction.

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ABSTRAK

KESAN PROSES PELINDAPKEJUTAN DAN RAWATAN PASCAPANAS TERHADAP MIKROSTRUKTUR DAN SIFAT FIZIKAL KELULI BERKARBON RENDAH SELEPAS MENJALANI ELEKTROKARBURASI

Kajian terhad telah dilakukan tentang rawatan selepas haba terhadap keluli karbon rendah yang dikarburkan melalui proses elektrokarburisasi. Dalam kajian ini, elektro-karburisasi (EC) telah dijalankan dengan menggunakan campuran elektrolit natrium karbonat (Na_2CO_3) dan natrium klorida (NaCl) dengan nisbah molar 4:1. Proses ini dilakukan pada suhu 800 °C dengan voltan malar 4.0 V selama tiga jam di dalam persekitaran gas karbon dioksida, CO_2. Selepas elektro-karburisaiti, sampel dilindapkan sama ada dalam air atau minyak. Rawatan pascapanas iaitu pembajaan, atau penyepuhlindapan telah dijalankan untuk melegakan tegasan baki. Selepas proses rawatan haba telah dilakukan, mesin kekerasan Vickers digunakan untuk mengukur profil kekerasan dari permukaan (0 µm) ke arah teras. Didapati bahawa kekerasan puncak semua sampel terletak pada 100 µm dari permukaan. Kekerasan puncak EC/sampel terlindap air (WQ, 702 ± 5 HV) adalah lebih tinggi daripada EC/sampel terlindap minyak (OQ, 571 ± 3 HV). Ini disebabkan oleh kadar pendinginan air lebih tinggi berbanding dengan minyak. Kadar pendinginan air yang tinggi menyebabkan karbon tidak mempunyai masa yang cukup untuk melepaskan atau bertindak balas dengan oksigen, malah karbon terperangkap untuk membentuk jadi martensit. Selain daripada itu, kekerasan puncak EC/sampel yang dirawat dengan selepas haba iaitu terbaja (WQ-T, 623 ± 9 HV ; OQ-T, 495 ± 4 HV) atau tersepuhlindap (WQ-A, 571 ± 3 HV ; OQ-A, 367 ± 6 HV) adalah lebih rendah daripada EC/sampel yang dilindapkan (WQ dan OQ). Hal ini disebabkan oleh tegasan dalaman telah dilegakan dan penyusunan semula struktur dapat merendahkan herotan keluli. Perubahan dalam struktur mikro seperti martensit, martensit terbaja dan ferit telah diperhatikan oleh mikroskop optik yang mana pembentukan martensit meningkatkan kekerasan sampel. Manakala fasa logam pula diperiksa dengan menggunakan Belauan sinar-X (XRD). XRD analisis mendapati EC/sampel yang dirawat pascapanas mempunyai oksida besi iaitu magnetit dan hematit disebabkan oleh pengoksidaan and penyahkarbonan. Pengoksidaan dan penyahkarbonan berlaku semasa proses pendinginan pada pembajaan dan penyepuhlindapan. Suhu tinggi semasa proses pendinginan menyebabkan tindak balas antara oksigen dan unsur pengoksidaan tinggi keluli iaitu ferit. Lebar penuh pada nilai separuh maksimum (FWHM) daripada puncak XRD (julat dari 44.4° pada 44.7°) menunjukkan bahawa sampel EC/post-heat-treat mempunyai nilai FWHM yang lebih rendah berbanding dengan sampel EC/dipadamkan, yang menunjukkan bahawa tegasan telah dilegakan dalam kedua-dua sampel yang dibaja dan disepuhlindap. Komposisi unsur sampel ditentukan oleh 'Energy Dispersive X-ray' (EDX). Analisis EDX menunjukkan kandungan karbon pada permukaan luar sampel yang seperti diterima, AR (2.79 wt. %) ; WQ (5.24 wt. %), WQ-T (5.16 wt. %), WQ-A (2.94 wt. %) ; OQ (4.73 wt. %), OQ-T (2.78 wt. %) dan OQ-A (2.63 wt. %) dengan masing-masing. Secara relatifnya, kandungan karbon WQ adalah lebih tinggi daripada sampel OQ, manakala EC/sampel yang terbaja mempunyai kandungan karbon yang tinggi berbanding dengan EC/sampel yang disepuhlindap, tetapi lebih rendah daripada sampel yang dilindapkan. Pembentukan martensit bergantung pada kandungan karbon disebabkan bahawa lebih banyak martensit terbentuk dengan keluli yang mengandungi karbon yang tinggi, ini dipadankan dengan kandungan karbon tinggi mempunyai kekerasan yang lebih tinggi. Kandungan oksigen pada EC/sampel yang dirawat pascapanas adalah lebih

pekat berbanding sampel lain. Hal ini mengakibatkan pembentukan oksida besi pada permukaan sampel disebabkan oleh berlakunya pengoksidaan dan penyahkarbonan. Pengoksidaan dan penyahkarbonan berlaku semasa proses penyejukan pembajaan dan penyepuhlindapan. Suhu yang tinggi semasa proses pendinginan menyebabkan tindak balas antara oksigen dan ferit. Ringkasnya, proses elektro-karburisasi meningkatkan kandungan karbon dan meningkatkan kekerasan sampel. Dalam proses pelindapkejutan, pelindapkejutan air memberikan kekerasan yang lebih baik daripada pelindapkejutan minyak, manakala proses pembajaan atau penyepuhlindapan melegakan tegasan dalaman yang membawa kepada pengurangan kekerasan.

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LIST OF SYMBOLS

α-iron	-	Ferrite
γ-iron	-	Austenite
λ	-	Wavelength of the Emitted Photon
2θ	-	Angle between Incident Beam and Crystallographic Reflecting Plane
CO_2	-	Carbon dioxide
EL	-	Elongation
Fe-Fe_3C	-	Iron-iron carbide
K_2CO_3	-	Potassium Carbonate
Li_2CO_3	-	Lithium Carbonate
NaCl	-	Sodium Chloride
Na_2CO_3	-	Sodium Carbonate
Mo_2C	-	Molybdenum Carbide
O_2	-	Oxygen

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LIST OF ABBREVIATIONS

AISI	-	American Iron and Steel Institute
AR	-	As received
BCC	-	Body Centred Cubic
B.H.N	-	Brinell Hardness Number
CSP	-	Compact Strip Production
EC	-	Electro-carburised
EDX	-	Energy Dispersive X-ray
FCC	-	Face Centred Cubic
FWHM	-	Full Width of High Maximum
ICDD	-	International Centre of Diffraction Data
JCPDS	-	Joint Committee on Powder Diffraction Standards
OQ	-	Electro-carburised and Oil quenched sample only
OQ-A	-	Electro-carburised - Oil quenched and annealed
OQ-T	-	Electro-carburised - Oil quenched and tempered
PWHT	-	Post Weld Heat Treatment
SAE	-	Society of Automotive Engineers
SEM	-	Scanning Electron Microscope
SLM	-	Selective Laser Melting
SMAW	-	Shielded Metal-Arc Welding
TBW	-	Temper Bead Welding
WQ	-	Electro-carburised and Water quenched only
WQ-A	-	Electro-carburised - Water quenched and annealed
WQ-T	-	Electro-carburised - Water quenched and tempered
XRD	-	X-ray Diffraction

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CHAPTER 1

INTRODUCTION

1.1 Overview

This chapter discussed the research background related to electro-carburisation process on low carbon steel. Problem statement regarding to the induced internal stress due to quenching process had been studied in this chapter. In addition, research objectives, scope of work and thesis content had been discussed.

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1.2 Background Study

Plain carbon steel is the alloyed version of iron and carbon that consisted of manganese along with other residual element such as silicon or aluminium. The plain carbon steel can be classified into low carbon steel, medium carbon steel, high carbon steel and ultra-high carbon steel which the difference is dependent on the carbon composition percentage of the plain carbon steel. The characteristics of outstanding ductility, machinability, weldable and low-cost of low carbon steel caused the major portion used of it (Islam & Rashed, 2019). Low carbon steels with less than 0.3 wt.% carbon content consisted mostly of ferrite. Ferrite, which is the softest phase of steel, makes low carbon steel have high machinability but low hardness (Evans, 2012).

Carburisation is a heat treatment process to improve the surface hardness and durability of low carbon steel products such as wire spring, carbon steel forging at the same time maintaining toughness and strength of the core. The carburising increases strength and wear resistance by diffusing carbon into the surface of the steel creating a surface hardening and at the same time retaining a substantially lesser hardness in the core. Carburisation temperatures process is generally carried out at high temperature between 800 °C to 950 °C at which austenite is in stable crystal structure with high solubility for carbon (Jon L. Dossett & George E. Totten, 2013). There are various carburising process methods such as pack (Darmo et al., 2021), gas (Izciler & Tabur, 2006), vacuum (Tsuji et al., 1987), plasma (Edenhofer et al., 2001), liquid (Finkel'shtein et al., 1963), and electro-carburising (Siambun *et al.*, 2011). These carburisation methods have their own pros and cons. The methods are chosen based on the size, quality and quantity of parts that need to be carburised (Siambun *et al.*, 2015). Carburisation method such as gas, vacuum and plasma provided good quality of case hardening, but this process involved complex process, expensive and difficulty in carburising large part. The carburisation process such as molten salt carburisation that obtained from liquid enrichment source is comparable to that of gas carburisation. Molten salt carburisation used cyanide bath or known as conventional carburisation provided low distortion, very flexible and uniformly of control and very effective with moderate quantities of parts. However, this method used salt bath that containing toxic cyanide and led to the production of toxic wastes, hence caused the usage of cyanide bath tend to be reduced for surface treatment of the steel (Lantelme et al., 2013; Liew et al., 2016; Siambun et al., 2015).

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In the early stage of molten salt carburisation, the conventional carburisation used a cyanide bath ($Ba(CN)_2$) as the electrolyte under heating for a period of time. However, the production of toxic discharge from this process led to the utilisation of other type of electrolytes. Thus, an alternative method, electro-carburisation (EC), known as molten salt electro-carburisation, was introduced to replace the conventional carburisation. The molten salt electro-carburisation was implemented to increase the carbon content on the surface of the low carbon steel and improve its hardness and wear resistance, and contact fatigue (Lingamanaik & Chen, 2012). Moreover, it was found that the internal strain, induced due to the carbon atoms interstitially present in the crystal lattice of the iron after the electro-carburisation, caused the increment in hardness (Siambun *et al.*, 2011). Furthermore, the improved hardness of electro-carburised low carbon steel showed the reduced severity of adhesive wear and tendency of the worn surface to fracture, resulting in a longer lifespan of low carbon steel (Liew et al., 2017). The rapid cooling process or known as quenching process also improved microhardness, which related to the resistance to slip and dislocation, by altering the primary phases (Abdullah *et al.*, 2020).

Electro-carburisation process had proven to give advantage compared to the conventional molten salt method where the mild steel samples with the use of CO_2 will produce higher surface hardness of the sample (Siambun *et al.*, 2015). Aside from this, the application of electro-carburisation process in the manufacturing industry will contribute to the good side in the production of the steel industry as well as the environment. For example, electro-carburisation process will help in the preservation of the nature by reducing the production of toxic wastes after this process. In other words, electro-carburisation produced less harmful discharge compared to conventional molten salt carburisation.

Heat treatment process is a technique to alter the mechanical and physical properties of the steel without changing its shape. It controlled the heating and cooling process of the steel by various temperatures, holding time and cooling rate. Heat treatment such as annealing can softening the steel to produce a less brittle by the process of heating the steel to particular temperature with specific duration and then cooling slowly to the room temperature. While another heat treatment is tempering which is used to improve the toughness of the steel. It is typically used to reduce the high brittle or excessive hardness of the steel by heating to specific temperature below critical temperature and cooling in the atmosphere (Khera *et al.*, 2014). The process of reheating the heat-treated steel for heat treatment again is so called post-heat treatment. For instance, the steel is heat-treated by hardening and it is tempered to reduce the brittleness.

In short, the microstructure of low carbon steel depends on its carbon content which is only found to have ferrite and pearlite, provided soft and high ductility characteristics (Islam & Rashed, 2019). Process of electro-carburisation on low carbon steel had increase the carbon content through carbon diffusion and it will be hardened by quenching process. The reason of improved hardness of the steel is due

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to the formation of martensite during quenching process (Kowser & Motalleb, 2015). Meanwhile, residual stresses induced by quenching process might also cause the brittleness of the steel. With the drawback of excessive residual stresses, the steel might suffer cracking and dislocation (Olabi & Hashmi, 1996). Therefore, stress relieving post-heat treatment such as tempering, and annealing are considered to be carried out to relief the stresses.

1.3 Problem Statement

For the past years, there are many studies on electro-carburisation process reported, such as effect of cooling rate to the surface hardness (Siambun *et al.*, 2012), influence of CO_2 gas to the surface hardness and case hardening of treated low carbon steel (Siambun *et al.*, 2015), influence of quenching medium to treated metal (Bahrin *et al.*, 2016), effect of electro-carburisation duration on wear behaviour of low carbon steel (Liew et al., 2016) and effect of electro-carburising temperature and voltage (Jin *et al.,* 2018).

Heat treatments involved the heating of the steel to certain temperature and eventually quenching it to obtain the desired microstructure of steel (Grum *et al.*, 2001). Various cooling rates of different quenching medium (water, oil, etc.) will affect the hardness of steel, as fast cooling rate provides better hardness performance (Kowser & Motalleb, 2015). The effect of quenching after the electro-carburisation process improved the hardness due to the formation of martensite (Bahrin *et al.*, 2016). However, it was found that the high level of residual stresses were induced after the quenching process due to the severe thermal gradient at the surface and core of the work piece (Masoudi *et al.*, 2015). In addition, there are presence of residual macro-stresses after cutting the steel which led to the distortion of the steel (Fu *et al.*, 2018). A high level of residual stress distorts the manufacturing process, which results in cracking and general instability of the steel. It was shown that residual stress leads to the cracking of metal in the corrosive environment. Moreover, a fast cooling process such as quenching will cause quench cracks (Panchal, 2013).

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Therefore, an idea was triggered to apply the post-heat treatments (tempering or annealing) of low carbon steel after electro-carburisation and quenching process to relieve the EC/quenched steel's internal and residual stresses and improve its properties. The mechanical properties such as toughness, hardness and ductility could be improved by the post-heat treatment through relieving internal stresses (Abdullah & Sazali, 2019). The tempering process diminished the brittleness and improved the strength of metal, while the annealing process refined the grain structure, which caused the metals more ductile and softer. In other words, the induced internal stress in low carbon steel sample which underwent electro-carburisation process had motivated this study. There is a need to carry out further studies to investigate the influence of quenching process and post heat treatment process on low carbon steel after the electro-carburisation process. It is essential to investigate the effect of post-heat treatment on relieve of internal stresses on electro-carburised low carbon steel as there is limited number of studies on post-heat treatment on quenched low carbon steel after electro-carburised.

1.4 Objective

In this thesis, the effects of quenching process and post-heat treatment process to the physical properties of electro-carburised low carbon steel will be investigated. The objectives of thesis are as shown below:

i. To evaluate the hardness profile of post heat treated (tempered or annealed) electro-carburised low carbon steel quenched in different quenching mediums.

ii. To examine the microstructure and elemental determination of post-heat treated (tempered or annealed) electro-carburised low carbon steel quenched in different quenching mediums.

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