

Influence of CO₂ Gas in the Electro-Carburisation Process of Mild Steel

Nancy Julius Siambun, Daniel A. Jewell, and George Z. Chen

Abstract—In the attempt to develop a clean process, study has been conducted to determine the effect of absorbed CO₂ gas as source of carbon to the case hardening of treated mild steels in the recently developed process of electro-carburisation in non-toxic molten carbonate salts. The concentration of CO₂ in the process was varied by purging a CO₂ and N₂ mixture at flow rate ratios of CO₂:N₂ of 200:0, 150:50, 100:100, 50:150 and 0:200 mL.min⁻¹. Electro-carburisation was performed in two types of molten salt electrolyte i.e. a mixed anion Na₂CO₃-NaCl (mole ratio 4:1) and a pure carbonate Li₂CO₃-K₂CO₃ (1:1 mole ratio). A voltage of 2.5V was applied between mild steel cathode and an inert SnO₂ anode at a carburisation temperature of 800°C for 60 minutes. The results show that greater CO₂ concentration produced samples with greater surface hardness and thicker case-hardening, attributed to the increased availability of the electro-active carbon source which was generated by absorption of CO₂. Electro-carburisation in Li₂CO₃-K₂CO₃ and Na₂CO₃-NaCl gave surface hardness of 1075 ± 25 HV, however Li₂CO₃-K₂CO₃ (540μ thickness) gave thicker case depth compared to Na₂CO₃-NaCl (500μ thickness). This is thought to be due to the amount of carbon deposited in each salt; 12.70 wt.% C in Li₂CO₃-K₂CO₃ and 11.10 wt.% C in Na₂CO₃-NaCl.

Index Terms—Molten salt, carburisation, carbonate salt, surface hardness, case depth.

I. INTRODUCTION

A substantial and sustainable reduction of CO₂ emission from burning fossil fuels is necessary to limit global climate change [1]. However achieving this goal is very challenging due to the demand and necessity of such activity. Wide researches have focus on ways to mitigate the CO₂ gas emission, such as Carbon Capture and Storage (CCS) [2] and Carbon Capture and Conversion (CCC) [3].

A recent breakthrough in molten salt carburisation has been reported by the authors involving an electrolytic process, termed electro-carburisation offer another alternative in utilising the released or stored CO₂ gas as source of carbon for carburisation process. Carburisation process has evolved from the use of solid enrichment sources to the incorporation

of liquid-, gas-, vacuum-, and plasma-based methods. It is known that, carbon atoms diffuse inward from the surface of the metal to increase the carbon content in the near surface region and thus the surface hardness [4]. Different carburisation methods have advantages and disadvantages and are chosen based on the size, quality, and quantity of parts that need to be carburised. For example, whilst carburisation using a solid enrichment source is suitable for large parts, this process is slow, messy, and can only deal with a very small number of parts per day. In addition, the case depth is difficult to control accurately. Conversely, the gas-, vacuum-, and plasma-based carburisation methods provide a good quality of case hardening, but involve complex processes, comparatively high cost, and difficulty in carburising large parts. The case hardening attained from a liquid enrichment source, such as in molten salt carburisation, is comparable to that from gas carburisation, and additionally provides low distortion, considerable flexibility and uniformity of control, and the ability to deal effectively with moderate quantities of parts (100s per day).

The molten salt electro-carburisation process that was developed by authors, whose mechanism has been described in detail elsewhere [5], [6] had successfully carburised mild steel in CO₂ gas environment, and eliminate the usage of toxic cyanide molten salt in the conventional molten salt carburisation process [7], with no formation of thick crust on salt surface and the need for continuous addition of replenishment material as source of carbon that was reported by other researchers to replace the toxic cyanide by carbonate salt [8]-[10]. The influence of CO₂ gas concentration to metal case hardening however was not studied and reported. In this research, the CO₂ gas was continuously purged into the electrolysis cell chamber at constant flow rate of 200 mL.min⁻¹ to provide a CO₂ gas environment which served as a source of carbon for carburisation. The purity of the CO₂ gas environment was varied by mixing the gas with nitrogen (N₂) gas, but maintaining the overall constant flow rate at 200 mL.min⁻¹. The effect of CO₂:N₂ gas environments in electro-carburisation process to the surface hardness and case-hardening was studied and reported in this article.

II. EXPERIMENTAL

The experimental procedures are described by the authors detail elsewhere [5], [6] and covered briefly as follows; Na₂CO₃, Li₂CO₃, K₂CO₃, and NaCl (purity ≥99.0% purity; Fisher Sci.) were used for preparing two carbonate containing molten salt electrolytes; Na₂CO₃-NaCl (4:1 mole ratio) and Li₂CO₃-K₂CO₃ mixture (1:1 mole ratio). Salt selection and composition is discussed by the author

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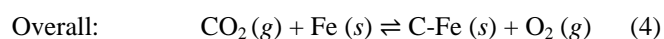
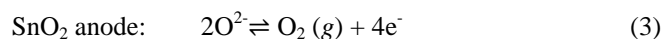
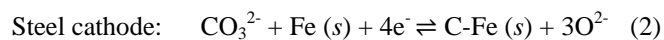
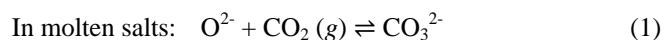
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elsewhere [5], [6]. A mild steel rod (0.1~0.2 wt.% C; 5 mm diam., Unicorn Metals) was made the cathode and an ELR SnO₂ rod (98.5% purity; 10 mm diam.; 300 mm length, Dyson Industries Ltd.) was used as an inert anode [11], [12]. The electro-carburisation process was carried out under a constant voltage of 2.5V applied using an Agilent (E3633A) DC power supply, for 60 minutes. at 800 °C in a stainless steel sealed tube reactor with a continuous purge of CO₂ and N₂ gas mixtures. In this research, studies on electro-carburisation were conducted in CO₂:N₂ gas mixtures at flow rate ratios of 200:0, 150:50, 100:100, 50:150 and 0:200 mL.min⁻¹. After the electro-carburisation process, the treated metal was quenched in water, cleaned, cut to 10 mm length, placed vertically to expose the cross section, and then embedded inside epoxy resin. The hardness profile was determined by the change of Vickers micro-hardness (MHT-1 Matsuzama Seiki Company Ltd.) from the surface towards the core of the sample, using a 1 kg load (*F*) and 10 seconds indentation period. The Vickers Hardness, electrolysis charge and theoretical carbon produced were correlated and compared at various CO₂:N₂ gas mixtures.

III. RESULT AND DISCUSSION

A. Current-Time (*I-t*) Plots of Electro-Carburisation

Carbonate salts are thermally unstable and decompose according to reaction (1) to form metal oxide and CO₂ gas. Elevating the CO₂ partial pressure helps to shift the equilibrium of this reaction to the right, i.e. to formation of carbonate ions, which helps to counters the decomposition. As the available carbonate quantity is fixed for a given input salt mass, the carbon source from the molten salts will eventually be depleted according to reaction (2) if the carbonate ion cannot be regenerated, i.e. unless the process is conducted in a CO₂ environment. This is also observed in a previous study [13] of electrolytic carburisation in an open furnace. Therefore, a continuous supply of CO₂ gas into the system during electrolysis allows reaction (1) to occur and ensures a continuous supply of carbon for the carburisation during the electrolysis process [5], [6].



where C-Fe represents the carbon dissolved in the steel cathode. In this work, the electro-carburisation process was conducted in a sealed tube reactor with a continuous purge of CO₂.

The effect of gas environment on the electro-deposition of carbon was investigated by varying the CO₂ and N₂ gas mixture i.e. by setting the gas regulator outlets of the CO₂ and N₂ gases to 0.2 barg (gauge pressure above the atm. pressure), and controlling the flow rate ratio of CO₂ and N₂ gas using gas rotameters, whilst maintaining the total gas flow rate of 200 mL.min⁻¹. It is interesting to note that the electrolysis curve that represented by the *I-t* curves for samples electro-carburised at lower CO₂ partial pressures in

Na₂CO₃-NaCl (4:1 mole ratio) exhibit an initial current peak Fig. 1(a), which suggests that carburisation begun instantly on application of an appropriate cell voltage. However, this declined rapidly during the first 2–3 minutes of the process due to a rapid reduction in the carbon concentration inside the salts. If the carbon content inside the salts is high enough, for example in molten Li₂CO₃-K₂CO₃ (1:1 mole ratio), carbonate ions would not be depleted as quickly and therefore no current reduction is observed Fig. 1(b).

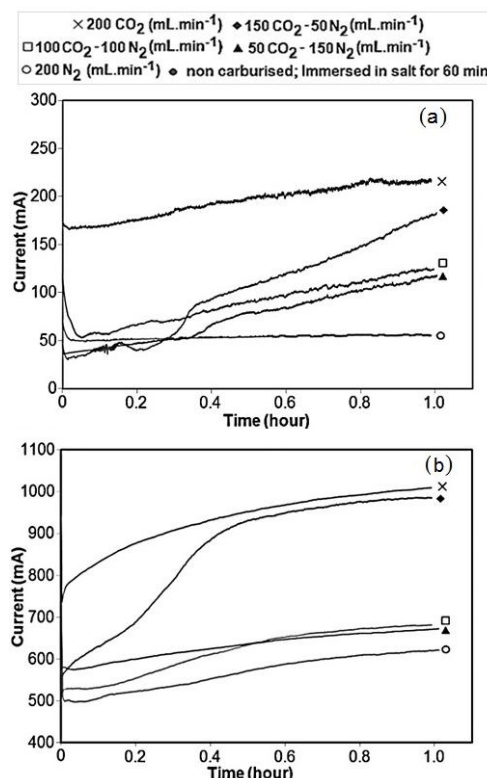


Fig. 1. *I-t* plots for electro-carburisation process in molten (a) Na₂CO₃-NaCl and (b) Li₂CO₃-K₂CO₃ at 800°C, 2.5V, and 60 minutes carburisation, under CO₂: N₂ gas ratio of 200:0, 150:50, 100:100, 50:150 and 0:200 mL.min⁻¹.

B. Surface Hardness and Case Hardening of Electro-Carburised Steel

Case hardening is caused by the penetration of carbon atoms into the crystal lattice of iron during the electro-carburisation process, resulting in internal straining and therefore increasing metal hardness. To assess this effect, Vickers micro-hardness measurements were carried out from surface towards the core on the cross section of the mild steel rod samples to determine the depth of carbon penetration.

The plots of Vickers micro-hardness versus distance from the surface, for samples electro-carburised under various ratios of CO₂ to N₂, and in molten Na₂CO₃-NaCl (4:1 mole ratio) and Li₂CO₃-K₂CO₃ (1:1 mole ratio) are shown in Fig. 2(a) and Fig. 2(b) respectively. These plots display a number of features that are worthy of discussion. In general, all the samples electro-carburised in Na₂CO₃-NaCl and Li₂CO₃-K₂CO₃ molten salt under various CO₂ and N₂ gas mixtures exhibit greater hardness near the surface than in the core region. In the surface region, there was a general trend of increasing hardness with higher CO₂ partial pressure. However, in the core region, all electro-carburised samples had almost the same hardness, which can be interpreted by the carbon atoms being unable to diffuse to the core in the 60

minutes electrolysis period. Comparison of Fig. 2(a) and Fig. 2(b) supported the conclusion that a pure carbonate salt i.e. $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$ mixture is in general more effective than the $\text{Na}_2\text{CO}_3\text{-NaCl}$ mixture for the electro-carburisation process. This is thought to be due to the availability of oxide ions for carbon (CO_2) absorption and the comparative abundance the carbon source for the carburisation process that occurred according to reaction (2). Although electro-carburisation still occurs in pure N_2 gas environments, the surface hardness is lower than samples electro-carburised in a pure CO_2 . Over a long period of carburisation, it is likely that the total carbon content inside the melt becomes depleted by both reactions (1) and (3).

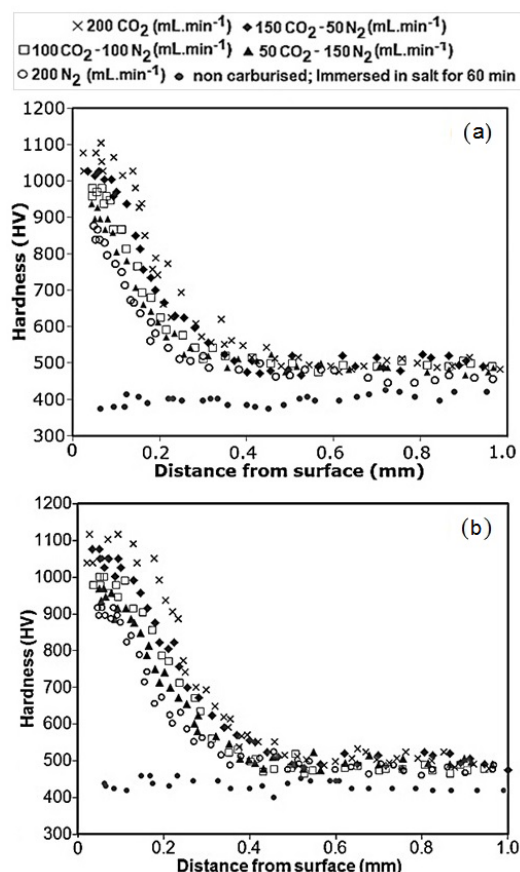


Fig. 2. Vickers micro-hardness vs. distance from the surface measured on the cross section of mild steel that was electro-carburised in molten (a) $\text{Na}_2\text{CO}_3\text{-NaCl}$ and (b) $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$ at 800°C , 2.5V, and 60 minutes carburisation, under $\text{CO}_2\text{:N}_2$ gas ratio of 200:0, 150:50, 100:100, 50:150 and 0:200 mL.min^{-1} . Data were from 3 parallel measurements for each sample

It is also notable that the hardness profile of samples electro-carburised in $\text{Na}_2\text{CO}_3\text{-NaCl}$ and $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$ at higher CO_2 partial pressures (flow rate 200:0 and 150:50 mL.min^{-1}), display a high hardness plateau near the surface with the average Vickers hardness value of 1075 ± 25 HV. This pattern of a high hardness plateau near the surface is also displayed by samples electro-carburised at lower CO_2 partial pressure (flow rate 100:100 mL.min^{-1}) but at higher carbonate content ($\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$). The high Vickers hardness obtained in these cases can be attributed to the ready availability of carbon atoms from the salt with high carbonate ion content, either as a function of CO_2 absorption, or initially present in the salt. The correlation of Vickers hardness profile with $\text{CO}_2\text{:N}_2$ partial pressure and $I\text{-}t$ curve are discussed below.

C. Correlation of Vickers Hardness and Current-Time ($I\text{-}t$) Curve

The hardness profiles observed in the samples (Fig. 2) are strongly correlated with the measured $I\text{-}t$ curves (Fig. 1); the higher the electrolysis current, the larger the reduction charges, the more carbon produced, and therefore the greater the hardness. The $I\text{-}t$ plots and the hardness profiles provide quantitative evidence for reaction (2) which can occur more readily at higher carbonate ion activity, which occurs with higher CO_2 partial pressure that facilitating reaction (1) and in the molten salt bath with higher initial carbonate concentration. Assuming the current passed through the cathode was solely used to produce carbon, the mass of carbon (m_{c-Q}) could be calculated using (5), where Q the total charge obtained from integration of the electrolysis current ($\int I dt$), m_c the molar mass of carbon ($12.011 \text{ g mol}^{-1}$), n number of electron ($4e^-$) in reaction (2), and F the Faraday constant (96485 C mol^{-1}). The m_{c-Q} calculated using (5) from the electrolysis charge is listed in Table I.

$$m_{c-Q} = \frac{Q m_c}{n F} \quad (5)$$

$$C(\text{wt.}\%) = \frac{m_c}{m_c + m_{Fe}} \times 100\% \quad (6)$$

TABLE I: RESULT ANALYSIS FROM MILD STEEL ELECTRO-CARBURISATION IN MOLTEN SALTS AT 800°C FOR 60 MINUTES

Ratio of $\text{CO}_2\text{:N}_2$ flow rate	200:0	150:50	100:100	50:150	0:200
Thickness of hardened case (T_c) measured from hardness profile (unit: mm)					
$\text{Na}_2\text{CO}_3\text{-NaCl}$	0.50	0.47	0.45	0.41	0.35
$\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$	0.54	0.50	0.48	0.47	0.44
Iron content (m_{Fe}) in hardened case (T_c) derived from hardness profile (unit: mg)					
$\text{Na}_2\text{CO}_3\text{-NaCl}$	28.4	27.9	27.4	24.6	24.6
$\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$	81.6	81.6	78.5	78.5	78.5
Carbon content (m_{c-Q}) in hardened case (T_c) derived from electrolysis charge					
$\text{Na}_2\text{CO}_3\text{-NaCl}$	(0.28)	0.17	0.16	0.15	0.12
(mg)	[11.10]	[7.70]	[7.40]	[7.20]	[7.00]
[wt.% C]					
$\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$	10.40	9.50	7.30	6.90	6.30
(mg)	[12.70]	[12.50]	[10.2]	[9.80]	[9.60]
[wt.% C]					

If all the carbon produced by electrolysis penetrated into the mild steel to a depth of T_c , the average carbon content in mass percentage, wt.%, could be obtained using (6), where m_{Fe} is the mass of iron calculated from T_c and the density of iron (7.874 g cm^{-3}). The average carbon content in mass percentage (wt.%) is listed in Table I.

Samples electro-carburised in $\text{Na}_2\text{CO}_3\text{-NaCl}$ salt under pure CO_2 gas have 11.1 wt.% C in the hardened case (T_c) according to the derived electrolysis charge. The wt.% C reduces when the partial pressure of CO_2 is reduced. A similar pattern was observed for samples electro-carburised in $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$, but the effect is enhanced by the higher initial concentration of carbonate ions in the pure carbonate salt. Consequently, the wt.% C was higher in $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$ than in $\text{Na}_2\text{CO}_3\text{-NaCl}$ salt at similar CO_2 gas partial pressure. The calculated carbon content of samples electro-carburised at the highest CO_2 partial pressure in both $\text{Na}_2\text{CO}_3\text{-NaCl}$ and

$\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$ salt reached 11.10 wt.% and 12.70 wt.% carbon respectively. This value is too high considering that the highest carbon content allowed to dissolve in mild steel to form Fe_3C at 800°C is 6.69 wt.% C [14].

As described in earlier research [5], [6], a layer of pure carbon can be formed on the surface of the cathode during electrolysis if the rate of deposition is greater than the rate of diffusion into the mild steel. Whilst this layer can help to maintain a constant flux of carbon atoms, the charge consumed in its formation which explains the observed differences in carbon deposited vs. carbon dissolved. The depth of case hardening and surface hardness were directly influenced by the CO_2 partial pressure during the electro-carburisation process. Electrolysis at high $\text{CO}_2\text{:N}_2$ partial pressure produced large amounts of carbon deposition on the surface of the cathode (which is evident from the I - t curve), thus providing a more significant driving force for carbon penetration into the cathode.

Further understanding of the electro-carburisation process at various $\text{CO}_2\text{:N}_2$ partial pressures can be obtained through correlation between the hardness value and the carbon content in the hardened case. The hardness profile in Fig. 2 showed the highest hardness of 1075 ± 25 HV corresponded to 6.69 wt.% C in Fe_3C , whereas the average carbon content in the was 0.1~0.2 wt.% C (302 ± 5 HV). The hardness profile (Fig. 2) reduces gradually towards the core and reaches a constant average hardness value of 515 ± 35 HV. Mild steel samples that were not electro-carburised but immersed in 800°C $\text{Na}_2\text{CO}_3\text{-NaCl}$ and $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$ molten salt for 60 minutes and thereafter quenched, gives flat hardness profile within the 1 mm depth that gives higher average hardness value i.e. 430 ± 30 HV ($\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$) and 400 ± 26 HV ($\text{Na}_2\text{CO}_3\text{-NaCl}$) compared to the untreated mild steel. The hardness increase for both samples is not due to the increase of carbon in the mild steel samples, but the heating and cooling effects. This observation matches well with the literature data for martensite steels, and agrees with the expected quenching effect instead of increase of carbon in the mild steel samples [15].

IV. CONCLUSION

A non-toxic molten salt electro-carburisation process has demonstrated to successfully utilised CO_2 gas for carburisation of mild steel cathode in two types of salt mixtures i.e. $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$ and $\text{Na}_2\text{CO}_3\text{-NaCl}$. The effects of CO_2 partial pressure on the hardness of mild steel samples has demonstrated that the variation of partial pressure of CO_2 by controlling the flow rate ratio of CO_2 to N_2 at 200:0, 150:50, 100:100, 50:150 and 0:200 mL.min^{-1} gave significant effect to the electrolysis I - t curves, and the surface hardness values and the case hardening profile. The experimental findings have shown that electro-carburisation at higher CO_2 partial pressure produced higher surface hardness in the mild steel samples, due to the increased availability of carbonate ions whose production was favoured at high CO_2 partial pressure. In a pure carbonate molten salt, i.e. in $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$, where again more carbonate atoms were available for carburisation, a higher surface hardness was

achieved. Based on the I - t curves, the calculated carbon produced during the electrolysis should have achieved a maximum carbon content of 11.10 wt.% and 12.70 wt.% respectively for samples electro-carburised in $\text{Na}_2\text{CO}_3\text{-NaCl}$ and $\text{Li}_2\text{CO}_3\text{-K}_2\text{CO}_3$ salt mixtures, under pure CO_2 gas. However due to this value exceeding the maximum solubility of carbon into the lattice of mild steel at 800°C , some of the produced carbon may only be present on the surface of the cathode.

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