Electrochemistry of CO$_2$ corrosion of mild steel: Effect of CO$_2$ on iron dissolution reaction

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ABSTRACT

The kinetics of the iron dissolution reaction was investigated in CO$_2$-saturated acidic sodium chloride solutions. While the effect of CO$_2$ on the iron dissolution reaction during corrosion process has received little attention in the existing literature, the experimental polarization curves obtained in the present study on X65 mild steel suggest that CO$_2$ and/or its related carbonate species are directly involved in the metal dissolution reaction. The presence of CO$_2$ was found to significantly influence the kinetics and the mechanism of the iron dissolution reaction at partial pressures as low as 1 bar. The higher corrosion rates observed in CO$_2$-saturated brines as compared to strong acid solutions at the same pH could possibly be explained by the effect of CO$_2$ on the kinetics of the anodic reaction.

1. Introduction

Iron oxidation, as the dominant anodic reaction, is a key element in corrosion of mild steel in acidic solutions. With the great interest in better understanding of the corrosion process, the investigation of the mechanisms and kinetics of this reaction became the subject of numerous studies over the last half a century [1–13]. Following the commonly accepted classification introduced by El Miligy et al. [4], the iron dissolution in mildly acidic solutions is believed to occur in four different ranges, based on the observed electrochemical behavior at various potentials and pH values. As demonstrated in Fig. 1, in the order from more negative towards more positive potentials, these were entitled as: *active dissolution*, *transition*, *pre-passivation*, and *passive*.

There are two classic mechanisms proposed for iron dissolution in acidic solutions in the literature: the "catalytic mechanism" and the "consecutive mechanism". These two mechanisms are associated with two distinct electrochemical behaviors observed in the active dissolution range. The catalytic mechanism, first proposed by Heusler et al. [14], is based on the experimental Tafel slope of 30 mV and second order dependence on hydroxide ion (OH$^-$) concentration. On the other hand, the consecutive mechanism proposed by Bockris et al. [9], was formulated to explain the observed Tafel slope of 40 mV and a first order dependence on OH$^-$ ion concentration. These two significantly different reaction kinetics are believed to be caused by the surface activity and microstructure of the metal substrate [2,10,12,13]. In more recent years there seems to be a consensus that these two mechanisms are in fact occurring in parallel. That can be seen in the so called

"branching mechanism" proposed by Drazic [10], which includes two parallel dissolution pathways: one resulting in a 40 mV Tafel slope and a first order pH dependence, and the other with a 30 mV Tafel slope and a second order pH dependence. The author suggested that the change of the mechanism between these two scenarios occurs with variation in the adsorption energy of the intermediate species that are affected by the substrate microstructure or the environmental conditions. It was also noted that the observed reaction orders vs. pH ranging between 1 and 2 occur when both mechanisms are in play simultaneously. A mechanism including parallel pathways was also proposed in the more comprehensive work of Keddam et al. [6,7]. The authors proposed a mechanism including 7 elementary steps, resulting into 3 parallel dissolution pathways that incorporated the elementary steps for both the consecutive and the catalytic mechanisms.

The anodic polarization curves obtained for mild steel dissolution in acidic CO$_2$-saturated aqueous environments have frequently been reported to have a 40 mV Tafel slope and a first order dependence on hydroxide ion concentration [15–19]. The literature, as it relates to CO$_2$ corrosion, appears to be settled on the simplistic consecutive mechanism proposed by Bockris et al. [9], which has been frequently used to describe the anodic currents without considering any effect of dissolved CO$_2$ or the related carbonate species present in the solution [15,17,20–23].

An in-depth analysis on the possible effect of dissolved CO$_2$ and/or other carbonate species on the iron oxidation reaction in acidic solutions is not available in the literature, to the best of our knowledge. Nevertheless, a significant effect of carbonate species on the rate of iron
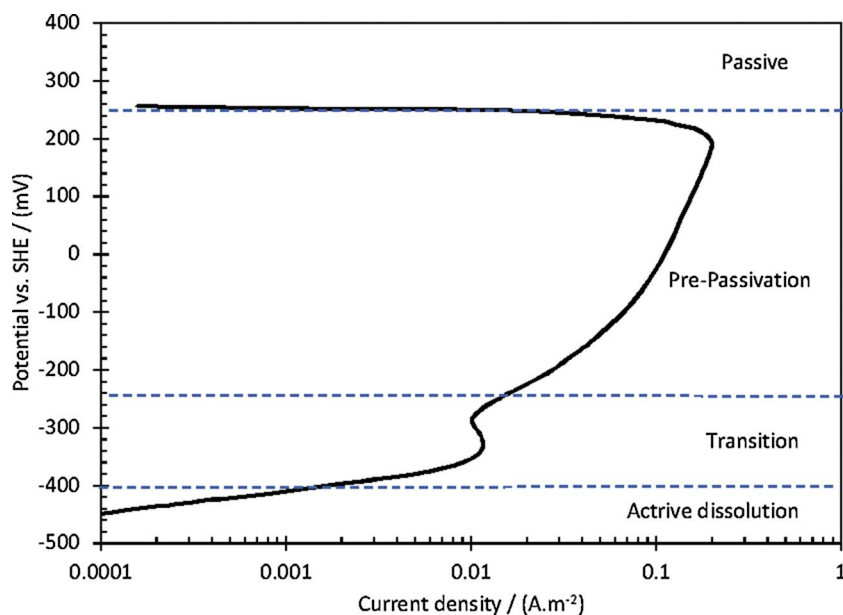
Fig. 1. Anodic polarization curve of iron in 0.5 M Na_2SO_4 solution at pH 5 and 298 K. With the scan rate of 6.6 mV s^{-1} and rotating disk electrode at 69 rps. Data taken from El Miligy et al. [4].

dissolution has been mentioned in a few different studies over the years [5,18,24–27]. The rate of anodic dissolution of iron in alkaline solutions was shown to increase in the presence of bicarbonate ions in several studies [25–27]. It the alkaline pH range, the anodic polarization curves showed that the iron dissolution reaction was in the passivation and *trans*-passivation range. There is a general agreement that the increasing rate of anodic dissolution is a result of chemical reaction of bicarbonate ion with the passive layer (e.g. $Fe(OH)_2$ and Fe_2O_3) to form various more soluble iron-complex species [25–27]. Thereby, the increased rate of the anodic reaction was associated with the chemical deterioration of the passive layer by the bicarbonate ion.

The effect of CO_2 on the iron dissolution reaction was also studied by Linter and Burstein [24] at pH 4 that is more relevant to the typical conditions of CO_2 corrosion in acidic aqueous environments. It was reported that the rate of iron dissolution from 0.5 Cr alloyed steel increased significantly in the transition and pre-passivation range, while the active dissolution range remained unaffected. The authors associated this effect to destabilization of the passive layer through a chemical attack and complex formation by CO_2 or its related carbonate species, similar to what was proposed in previous works [25–27].

In a study by Nesic et al. [5], the anodic polarization curves were used to discuss the effect of CO_2 in a relatively narrow potential range (~100 mV) above the corrosion potential. Experiments were performed in acidic perchlorate solutions (pH 2 to pH 6), and partial pressure of CO_2 limited to the 0–1 bar range. The proposed mechanism by Nesic et al. suggests that CO_2 is actively engaged in the electrochemistry of iron dissolution by directly adsorbing onto the metal surface and forming a chemical ligand that replaced the ferrous hydroxide intermediate species. The authors suggest that the presence of CO_2 did not affect the observed Tafel slopes, whereas the exchange current densities were increased with a linear proportionality to pCO$_2$ up to 1 bar. Furthermore, it was noted that as pCO$_2$ → 1 bar, the effect of CO_2 reaches its maximum and the rate of anodic reaction was not further increased with increasing pCO$_2$. In a more recent study, Almedia et al. [28] revisited this subject by investigating the corrosion of X65 mild steel at pCO$_2$ = 0, 1, and 30 bar. Based on electrochemical impedance measurements at corrosion potential, the authors concluded that CO_2 does not directly act on the metal surface and it has no significant effect on the iron dissolution reaction, in contrast to what was proposed by Nesic et al. [5]. Nevertheless, considering that this mechanistic argument is developed merely based on measurements at the open circuit potential and only at pH 4, the reported mechanistic behavior may not be readily

generalized to the great range of conditions typically encountered in CO_2 corrosion.

The CO_2 corrosion of mild steel is now considered a standard topic in corrosion studies. Numerous studies has been conducted to address various aspects of this process, as reviewed in detail elsewhere [29–35]. Nevertheless, the mechanistic investigations on this subject have been generally focused on the cathodic reactions of this system, whereas the effect of CO_2 on the anodic iron dissolution reaction in acidic solutions was not investigated in much depth. The present study attempts to determine if CO_2 or its related carbonate species have any significant influence on the kinetics and the mechanism of the iron dissolution reaction from mild steel in mildly acidic pH range. In the present study, the kinetics of the iron dissolution reaction was investigated through steady state polarization curves obtained at pH 4 and pH 5, and CO_2 partial pressures from 0 to 5 bar.

2. Material and methods

The experiments were conducted in the thin channel flow cell described elsewhere [36–41]. The test section was modified for electrochemical measurements by introducing a saturated Ag/AgCl reference electrode, facing the working electrode (3 mm apart). A 0.1 M NaCl supporting electrolyte solution (110 L), used throughout this study, was initially purged for ~ 3 h, with N_2 or CO_2 gas depending on the experimental conditions, while the outlet gas was monitored with an oxygen sensor (Orbisphere 410). Maximum dissolved oxygen content before initiating the experiment was 3 ppb (typically ~1 ppb). For experiments at elevated pCO$_2$, this deoxygenation step was followed by pressurizing the system to 5 bar for at least 3 h (or until a steady pH was obtained). As the last step, the pH was adjusted to the target value by gradual addition of deoxygenated HCl or NaOH solutions into the system. The temperature was controlled (± 0.5 °C) by means of electric heaters placed in the tank and the test section (used for experiments at 30 °C) as well as a heat exchanger (used for experiments at 10 °C) connected to a chiller (Air-3000 FLUID CHILLERS Inc.). The pump output was fixed throughout the experiments such that a 12.7 ms^{-1} flow velocity was obtained inside the thin channel test section.

The disk shaped API 5L X65 mild steel working electrode (chemical composition reported previously [42]) was built in house, similar to those described in an earlier study [41]. Prior to each experiment, the electrodes were abraded with a 600 grit silicon carbide paper and rinsed and sonicated in isopropanol for 5 min. After inserting the
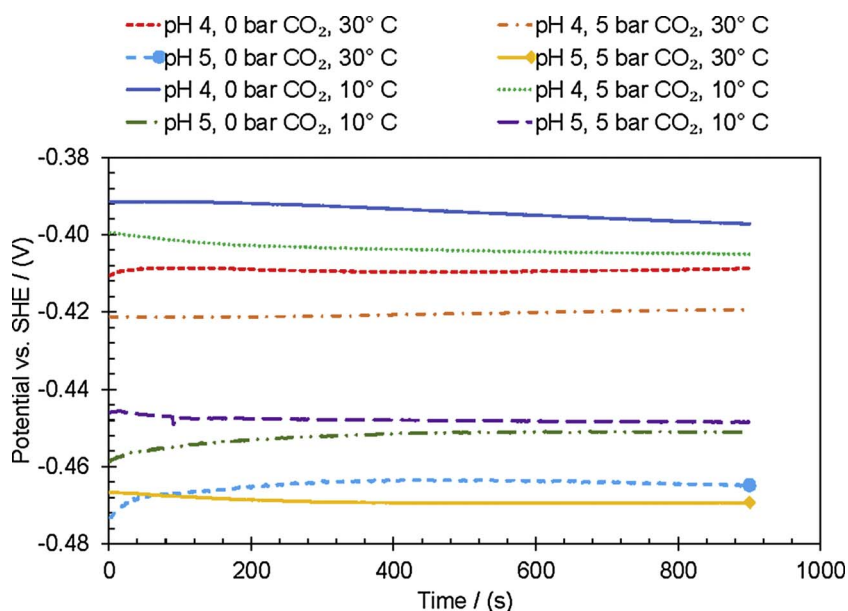
Fig. 2. The open circuit potentials measured immediately after the exposing the electrode to the solution.

working electrodes into the test section, the test section was purged using dry N_2 or CO_2 gas.

After allowing the solution into the test section, the open circuit potential (OCP) was monitored for 15 min or until a steady value was reached (maximum ± 2 mV change over 5 min). The typical behavior of the measured open circuit potentials at various conditions is shown in Fig. 2. The anodic polarization curves reported in the present study were obtained by sweeping the potentials from OCP towards the more positive potentials using staircase voltammetry at 0.5 mV s^{-1} scan rate and 1 s^{-1} sampling period. It was shown previously [5] that the change of scan rate from 0.1 mV s^{-1} to 1 mV s^{-1} does not change the polarization behavior of the iron dissolution reaction at similar conditions, suggesting that the steady state condition is reached at such scan rates. Furthermore, as discussed in the following section, the experimental Tafel slopes observed in the present study agreed well with those reported previously on pure iron electrodes [10,12,43]. This agreement was taken as an additional assurance that the polarization curves were at the steady state. The reported polarization curves were corrected for ohmic drop with the solution resistance obtained by electrochemical impedance spectroscopy (EIS) measurements (DC potential 0 mV vs. OCP, AC potential 5 mV, frequency range 100 kHz to 0.2 Hz at 10 points.dec^{-1}) performed 15 min after polarization measurements, when a steady state OCP was established.

3. Results and discussion

The steady state anodic polarization curves obtained in N_2 purged solutions are shown in Fig. 3, in order to demonstrate the effect of temperature and pH. The polarization curves obtained at 30 °C only show the transition and the pre-passivation range, whereas those obtained at 10 °C also show the active dissolution range. The absence of the active dissolution range in the polarization curves obtained at 30 °C was due to the interference from the cathodic hydrogen evolution reaction. At that condition, the active dissolution range at low anodic current densities, was covered by the cathodic currents. As shown in Fig. 3, the decreased rate of the anodic reactions at the lower temperature (10 °C), allows the active dissolution range to be observed clearly, by shifting the anodic curves towards more positive potentials and minimizing the interference from the cathodic currents.

The Tafel lines in Figs. 3 and 4 are plotted using the expression: $i = i_o \times 10^{E/b}$, based on Tafel equation, in order to estimate the apparent Tafel slope and reaction orders. As shown in Fig. 1, the active

dissolution range has an apparent Tafel slope of 28 mV, which agrees well with the theoretical value of $1/2 \times 2.303RT/F$. The comparison of the results at 10 °C at different pH values in the active dissolution range shows an apparent reaction order of approximately 1.6. The characteristic parameters (Tafel slopes and reaction orders) obtained at these conditions were found to be in agreement with those previously reported in the literature for pure polycrystalline iron substrates [10,12,43]. The deviation of the reaction order from the expected value of 2 has been reported previously, as discussed in the review by Drazic et al. [10] and may suggest the existence of parallel dissolution reaction mechanisms. The pre-passivation range, with a 120 mV Tafel slope ($2 \times 2.303RT/F$) was observed for both sets of polarization curves at 10 °C and 30 °C, similar to what was reported previously [10,44]. The i_o (exchange current density) at the 120 mV Tafel slope range at various solution compositions and temperatures are plotted in Fig. 5. In the experiments with $pCO_2 = 0$, no significant pH dependence was observed in this range at 30 °C when considering the experimental error. At 10 °C a slight pH dependence with a reaction order of ~ -0.15 vs. $[OH^-]$ was obtained.

The effect of pCO_2 on the anodic polarization curves is shown in Fig. 2. Here again only the transition and the pre-passivation range were observed at 30 °C as shown in Figs. 2.A and B. In this range however, as shown in Fig. 5, the anodic currents were significantly increased by increasing pCO_2, where apparent reaction orders of 0.34 and 0.61 vs. pCO_2 were obtained at pH 4 and pH 5, respectively.

The anodic polarization curves obtained at 10 °C are shown in Figs. 2.C and D for pH 4 and pH 5, respectively. Here the active dissolution range as well as the transition and pre-passivation range were clearly observed. At the pre-passivation range, a ~ 120 mV Tafel slope and apparent reaction orders of 0.39 and 0.31 vs. pCO_2 was observed at pH 4 and pH 5, respectively. In the presence of CO_2, the Tafel slope at the active dissolution range was slightly decreased from ~ 28 mV to ~ 22 mV. The Tafel slope did not further decrease with the increase of pCO_2 to 5 bar. Furthermore, in the transition range, an additional linear behavior with Tafel slope of ~ 60 mV was observed (most clearly at pH 4 and 5 bar CO_2).

The observed influence of CO_2 on anodic reaction in active dissolution range was found to partially agree with the results reported by Nesic et al. [5], in the sense that the presence of CO_2 resulted in an increased rate of reaction which was not further intensified at $pCO_2 > 1$. However, in the present study the effect was in the form of a slight change of Tafel slope. Although, this effect was quantitatively
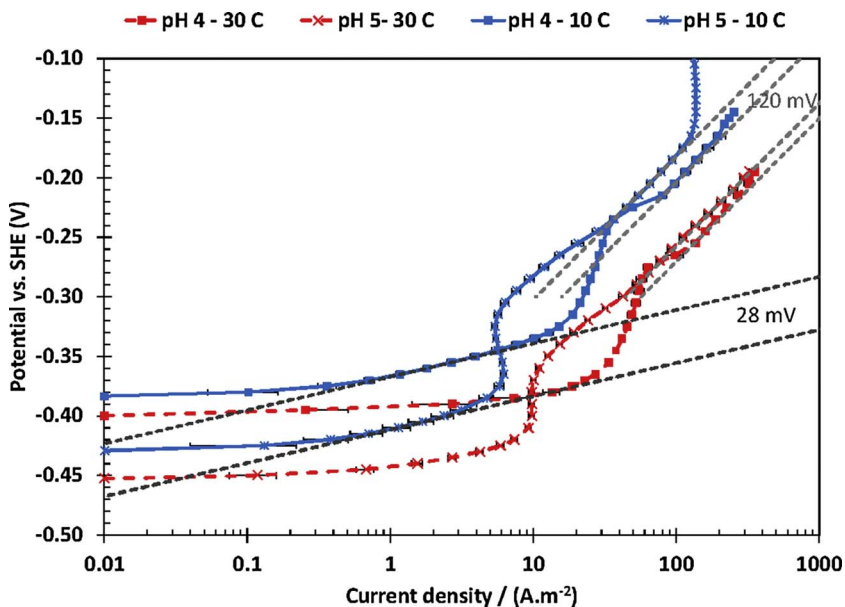
Fig. 3. Anodic polarization curves at 12.7 m s^{-1}, 0.1 M NaCl, 0.5 mV s^{-1} scan rate on an X65 mild steel surface. Error bars represent the minimum and maximum values obtained in at least three repeats.
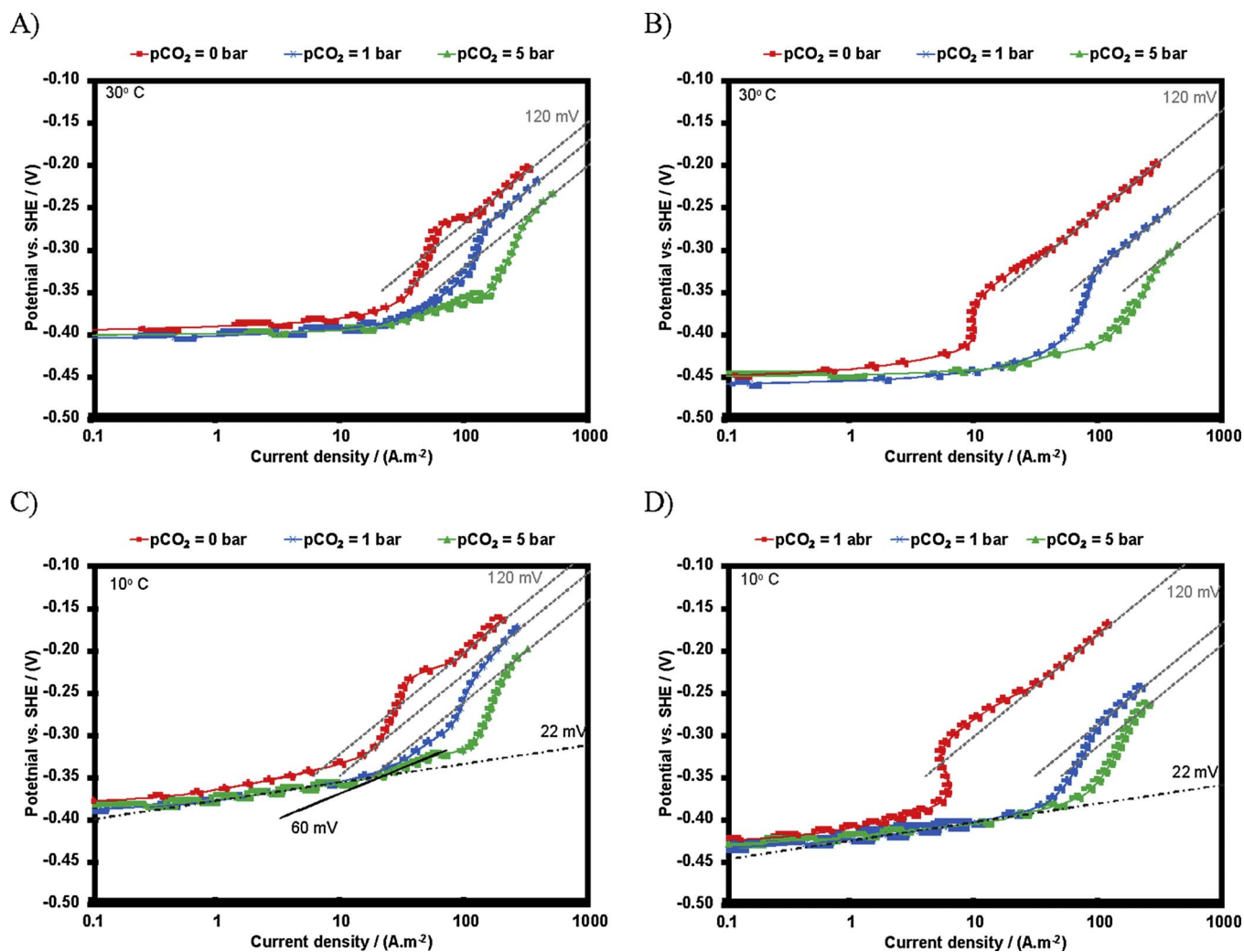

Fig. 4. Anodic polarization curves at 12.7 m s^{-1}, 0.1 M NaCl, 0.5 mV s^{-1} scan rate on an X65 mild steel surface. A) pH 4, 30 ℃, B) pH 5, 30 ℃, C) pH 4, 10 ℃, D) pH 5, 10 ℃. Error bars represent the minimum and maximum values obtained in at least three measurements.
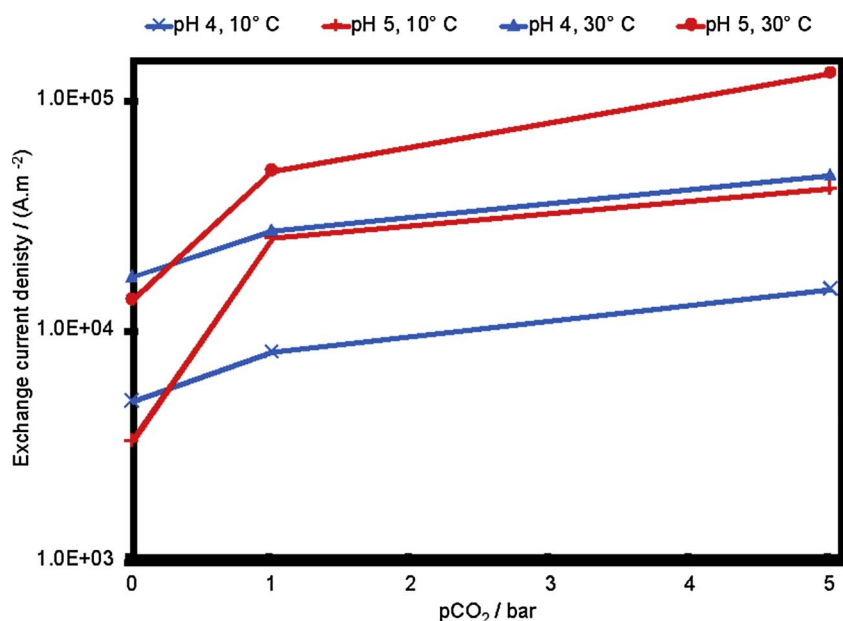
Fig. 5. The effect of pH and pCO_2 on the exchange current density at the pre-passivation (120 mV Tafel slope) range.

small (Only ~6 mV), it could possibly imply a change of mechanism. The 28 mV Tafel slope which correlates well with $1/2 \times 2.303RT/F$, is a characteristic of an electrochemical reaction proceeded by one or more elementary steps, whereas the 22 mV Tafel slope ($2/5 \times 2.303RT/F$) may suggest an electrochemical reaction proceeded by two or more elementary steps.

The significant increase of the anodic reaction rate in the transition and pre-passivation ranges are found to be in agreement with the results reported previously by Linter and Burstein [24]. In that study, the authors suggested that the increased rate of iron dissolution was a result of destabilization of the passive layer ($Fe(OH)_2$ or Fe_2O_3 species) through a chemical attack by bicarbonate ion, similar to that proposed for the alkaline pH range [25–27]. However, such an explanation may not be assumed valid for the conditions of the present study, considering that the formation of a passive layer on a mild steel surface is not thermodynamically favored at the pH and potential range of interest [45]. However, a rather similar chemical interaction of CO_2 or other carbonate species could still be feasible. The previously proposed mechanism of iron dissolution in acidic solutions without CO_2 suggests that the first current maximum observed in the polarization curves is associated with accumulation of the reaction intermediate Fe(I) (i.e. FeOH) on the metal surface [6,7,13]. Keddam et al. suggest that the increased current after the first maximum is a result of a parallel reaction pathway involving Fe(II) intermediate species [6,7,13]. The second current maximum at more positive potentials leading to the surface passivation (not observed in the polarization curves presented above), was associated with a chemical transformation of Fe(II) intermediates to insoluble passivating species (e.g. Fe_2O_3 or $Fe(OH)_2$) [13]. Therefore, the increase of current at the first current maximum could possibly be explained by an interaction of dissolved carbonate species with Fe(I) reaction intermediate, providing an additional chemical desorption pathway for this species that could increase the observed current densities. However, the observation of an additional 60 mV Tafel slope range below the first peak and the continuous increase of the exchange current density at the 120 mV range with increasing pCO_2, could also be a strong indication of electrochemical reactions where carbonate intermediate species are involved.

An inclusive mechanism for the iron dissolution reaction at such conditions requires a comprehensive investigation on the subject, considering the complexity of the reaction. However, the results presented here demonstrate a clear and significant effect of CO_2 on the rate and the mechanism of iron dissolution in acidic environments. The

influence on the transition and pre-passivation range is of particular interest in the typical CO_2 corrosion scenarios. As shown in Fig. 4.B, at pH values of 5, even at 30 °C, the corrosion current is in the transition/pre-passivation range. The polarization curves suggests that the effect of CO_2 on the anodic reaction rate can be as significant as its effect on the cathodic limiting current density.

4. Conclusions

The anodic polarization behavior of API 5L X65 mild steel at various pH and pCO_2 values was discussed based on the experimental data obtained in well-controlled environmental conditions at 30 °C and 10 °C. A significant effect of pCO_2 on the anodic currents was observed throughout the active dissolution, transition and pre-passivation ranges. In the active dissolution range, the presence of CO_2 (1 bar) decreased the Tafel slope, while a further increase of pCO_2 did not magnify this effect. The current densities in the pre-passivation range showed a Tafel slope of ~120 mV and a significant dependence on pCO_2. These observations suggest that CO_2 and/or its related carbonate species were directly involved in the iron dissolution reaction and significantly influenced the anodic currents at CO_2 partial pressures as low as 1 bar.

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