

Paper No.  
16622



## Effect of H<sub>2</sub>S on CO<sub>2</sub> Corrosion of Mild Steel in HPHT Conditions

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### ABSTRACT

The effect of H<sub>2</sub>S on the aqueous corrosion behavior of mild steel was evaluated at HPHT conditions (supercritical CO<sub>2</sub> pressure) at a total pressure of 12 MPa and a temperature of 160°C. The corrosion rate of steel samples was determined by electrochemical and weight loss measurements. The surface/cross-sectional morphology and the composition of the corrosion product layers were analyzed by using surface analytical techniques (SEM, EDS, and XRD). Results showed that the corrosion rate decreased with time and no significant difference was observed in the presence of 1000 and 2000 ppm of H<sub>2</sub>S at HPHT CO<sub>2</sub> conditions. Surface and cross-sectional analyses revealed that the corrosion process is governed by the formation of FeCO<sub>3</sub> regardless of the presence of H<sub>2</sub>S. Furthermore, the corrosion behavior of mild steel in these conditions did not depend significantly on flow velocity.

**Key words:** Supercritical CO<sub>2</sub>, CO<sub>2</sub> corrosion, carbon steel, H<sub>2</sub>S, high temperature

### INTRODUCTION

The presence of large quantities of CO<sub>2</sub> (from 25% to 89%) in gas fields have imposed increased challenges on the economics of project development and subsequent operations.<sup>1</sup> A major issue in developing those fields is corrosion and materials technology because of aggressive environments (i.e., high CO<sub>2</sub> contents and/or presence of H<sub>2</sub>S) with high pressures and high temperatures (HPHT).<sup>2</sup> Although corrosion resistance alloys (CRAs) have been available as a materials selection option for these severe environments, carbon and low alloy steels are still widely used as tubing materials due to their strength, availability and cost.<sup>3-7</sup>

The increment of temperatures and CO<sub>2</sub> pressure in production wells may lead to CO<sub>2</sub> in supercritical state if the temperature and the pressure are over 31.1°C and 7.38 MPa, respectively.<sup>8</sup> Corrosion issues

in supercritical CO<sub>2</sub> environments relating to carbon capture and storage (CCS), enhanced oil recovery (EOR), and deepwater oil and gas production applications have recently been investigated at relatively low temperatures (< 90°C),<sup>9-13</sup> and it has been understood that aqueous corrosion mechanisms in high pressure CO<sub>2</sub> are similar to those in low pressure CO<sub>2</sub> conditions.<sup>14</sup>

Regardless of the CO<sub>2</sub> pressure, there has been relatively less research on corrosion at high temperatures, above 100°C.<sup>15,16</sup> In the temperature range from 90 ~ 250°C, general corrosion rates of carbon steel were shown to decrease with increasing temperature and were strongly dependent on the formation of magnetite (Fe<sub>3</sub>O<sub>4</sub>) because of the fast kinetics of Fe<sub>3</sub>O<sub>4</sub> formation at high temperatures.<sup>17,18</sup> This indicates that high temperature has a significant effect on the corrosion rate and formation/transformation of the corrosion products. Those effects were also shown in high temperature H<sub>2</sub>S corrosion, yet the understanding of the associated corrosion mechanism is very limited.<sup>19-21</sup>

Until now, there are few studies for aqueous corrosion in high temperature supercritical CO<sub>2</sub> environments with H<sub>2</sub>S related to the downhole conditions. Thus, the objective of the present study was to identify and quantify the key issues that affect the integrity of carbon steel in high temperature supercritical CO<sub>2</sub> in the presence of small amounts of H<sub>2</sub>S.

## EXPERIMENTAL PROCEDURE

The test specimens were machined from UNS K03014<sup>(1)</sup> carbon steel (CS) with two different geometries: cylindrical type with 5 cm<sup>2</sup> exposed area for electrochemical measurements, and rectangular type with a size of 1.27 cm × 1.27 cm × 0.254 cm for surface analysis. The chemical composition of the CS is shown in Table 1. The specimens were ground sequentially with 250, 400 and 600-grit silicon carbide paper, rinsed with deionized (DI) water, cleaned with isopropyl alcohol in an ultrasonic bath for 60 seconds, and then dried.

**Table 1**  
**Chemical compositions of materials used in the present study (wt.%, balance Fe).**

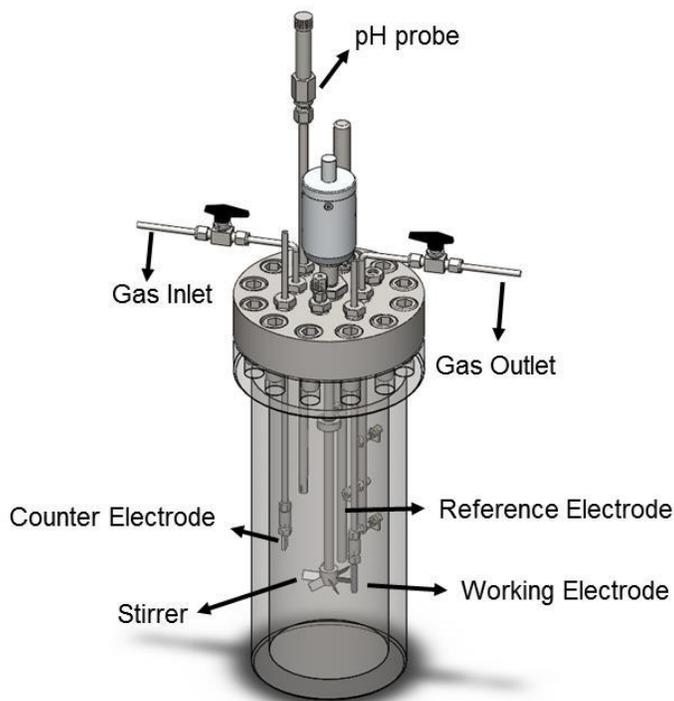
	C	Cr	Mn	P	S	Si	Cu	Ni	Mo	Al
CS	0.065	0.05	1.54	0.013	0.001	0.25	0.04	0.04	0.007	0.041

The corrosion experiments were conducted in a 7.5-liter autoclave (UNS N10276) which contained a CS working electrode, a HPHT Ag/AgCl reference electrode, a platinum-coated niobium counter electrode, and a HPHT ZrO<sub>2</sub>-based pH electrode. A schematic of the experimental setup is shown in Figure 1. The test solution was a 1 wt.% NaCl aqueous electrolyte prepared using DI water. An impeller was used to stir the solution and to generate flow velocities of about 1 m/s and 1.7 m/s (1000 rpm and 1700 rpm, respectively) during the tests.

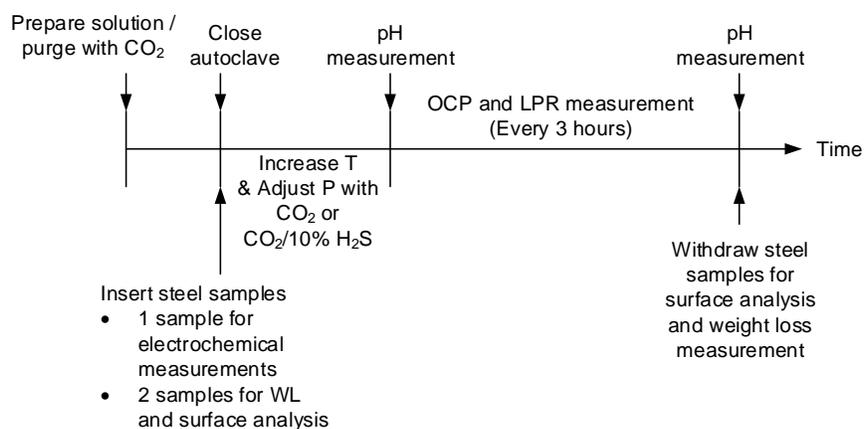
The experimental procedure is outlined in Figure 2. Initially, 6 liters of 1 wt.% NaCl solution was placed in the autoclave and purged with a high flow rate of CO<sub>2</sub> for 1 ~ 3 hours in order to remove dissolved O<sub>2</sub>. The specimens were then placed in the autoclave while purging with CO<sub>2</sub> to avoid air ingress. After closing the autoclave, additional purging with CO<sub>2</sub> or a mixture of CO<sub>2</sub>/10% H<sub>2</sub>S was performed for 1 to 2 hours to ensure the removal of O<sub>2</sub> and the saturation of CO<sub>2</sub> or CO<sub>2</sub>/H<sub>2</sub>S, and then the temperature was increased to the testing temperature. Once the working temperature was reached, the working H<sub>2</sub>S partial pressure was achieved by pressurizing with a mixture of CO<sub>2</sub>/10% H<sub>2</sub>S for the CO<sub>2</sub>/H<sub>2</sub>S conditions. High pressure was then attained by injecting CO<sub>2</sub> with a booster pump.

<sup>(1)</sup> UNS numbers are listed in Metals and Alloys in the Unified Numbering System, published by the Society of Automotive Engineers (SAE International) and cosponsored by ASTM International.

Corrosion rate and corrosion potential of specimens were evaluated with time by linear polarization resistance (LPR) measurements. The corrosion rates were also determined from the weight-loss (WL) method at the end of the test. The details of LPR and WL measurements are shown in previous studies.<sup>22,23</sup> The solution pH was measured at the beginning and end of the experiment by using a commercial HPHT ZrO<sub>2</sub>-based pH electrode and the HPHT Ag/AgCl reference electrode. The electrodes were calibrated prior to each test by the procedure supplied by the manufacturer.<sup>24</sup> After each test, the morphology and compositions of corrosion products were analyzed with scanning electron microscopy (SEM), energy-dispersive X-ray spectroscopy (EDS), and X-ray diffraction (XRD). Table 2 shows the test conditions for the present study.



**Figure 1: Schematic diagram of the autoclave used for the test under HPHT condition.**



**Figure 2: Experimental procedures for evaluating the corrosion behavior in HPHT CO<sub>2</sub> environments with H<sub>2</sub>S.**

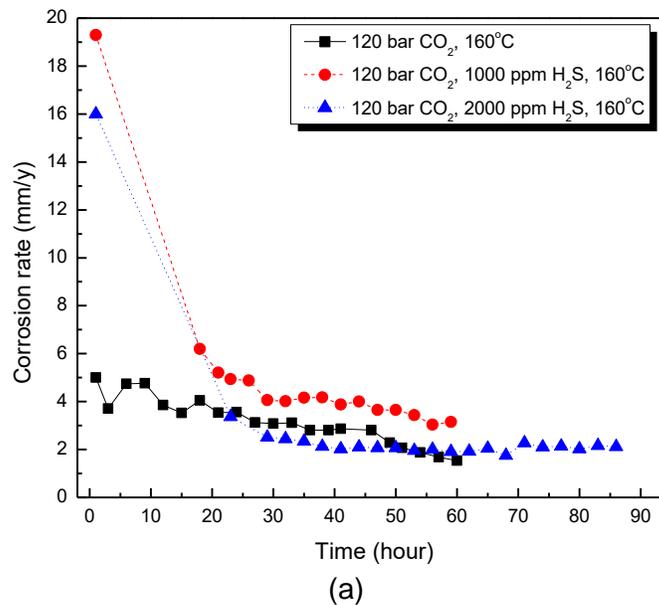
**Table 2**  
**Test conditions for corrosion testing**

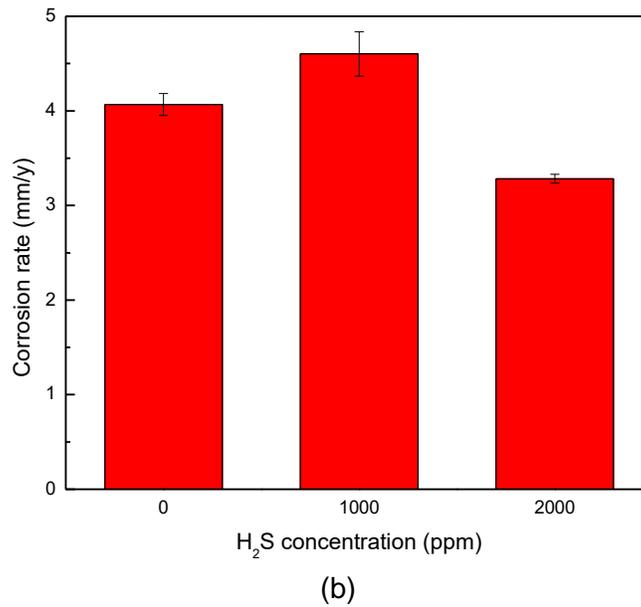
	pCO <sub>2</sub> (MPa)	H <sub>2</sub> S (ppm <sub>v</sub> )	Temperature (°C)	Rotation speed (rpm)
1	12	0	160	1000
2	12	1000	160	1000
3	12	2000	160	1000
4	12	2000	160	1700

## RESULTS AND DISCUSSION

Figure 3 shows LPR and weight loss (WL) corrosion rate data of CS exposed to 1 wt.% NaCl at 12 MPa CO<sub>2</sub> and 160°C with 0, 1000 and 2000 ppm H<sub>2</sub>S. The corrosion rate of CS for the CO<sub>2</sub> condition was about 5 mm/y at the beginning of the experiment and then slightly decreased with time, whereas the corrosion rates of CS with H<sub>2</sub>S (for both H<sub>2</sub>S concentrations) started at much higher values and then decreased to similar values to those of the CO<sub>2</sub> condition after 20 hours. WL corrosion rate shows the same trend as the LPR corrosion rate.

Table 3 lists the solution pH values at the beginning and the end of each test. For all three conditions, the solution pH slightly increased indicating a small change in the bulk water chemistry due to the release of Fe<sup>2+</sup>.



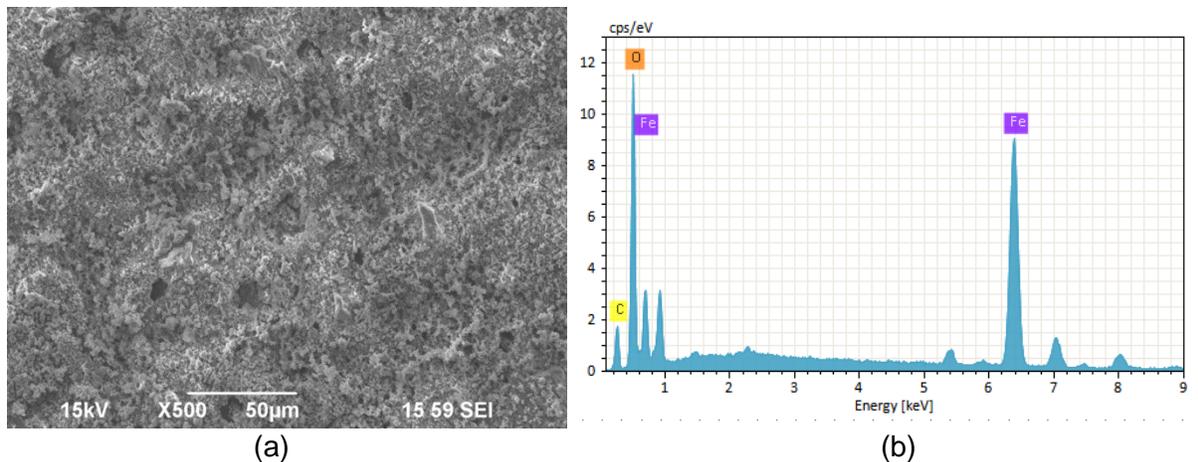


**Figure 3: Comparison of corrosion rate with different H<sub>2</sub>S concentrations at 12 MPa CO<sub>2</sub> and 160°C: (a) LPR corrosion rate, (b) WL corrosion rate.**

**Table 3  
Measured initial and final solution pH at 12 MPa CO<sub>2</sub> and 160°C.**

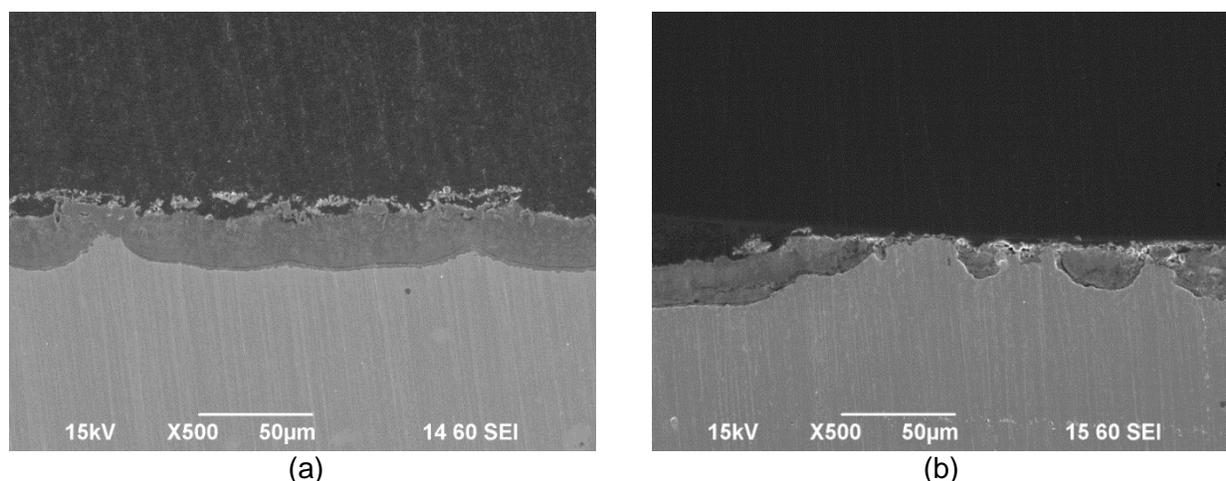
	Initial pH	Final pH
0 ppm H <sub>2</sub> S	4.3	4.8
1000 ppm H <sub>2</sub> S	4.2	4.7
2000 ppm H <sub>2</sub> S	4.4	4.8

Figure 4 shows the surface morphology and chemical analysis of the corroded sample after 60 hours of exposure to the CO<sub>2</sub> condition. A uniform corrosion product was found covering the exposed surface. EDS analysis shows that the corrosion product mainly consists of iron, carbon, and oxygen.



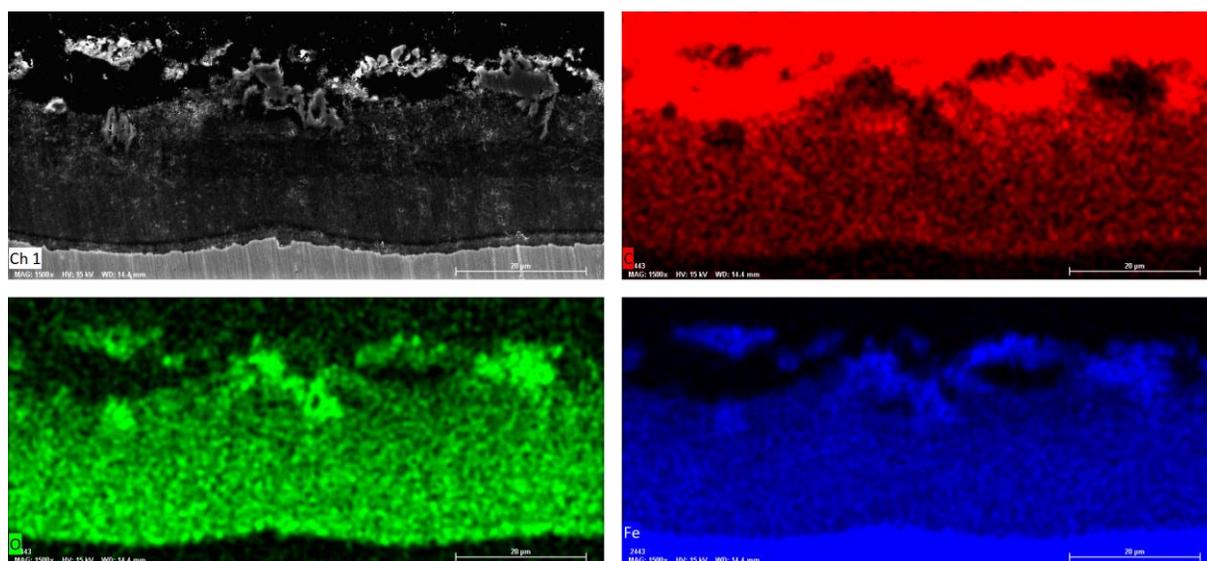
**Figure 4: SEM surface view (a) and EDS analysis (b) of the corrosion product formed at 12 MPa and 160°C.**

Figure 5 shows cross-sectional SEM images of the corroded sample exposed to the CO<sub>2</sub> condition. It can be seen that most of the surface was covered by a thick corrosion product layer (~ 30 μm). However, there are several areas where the corrosion product layer is thinner (~ 5 μm, Figure 5 (b)).



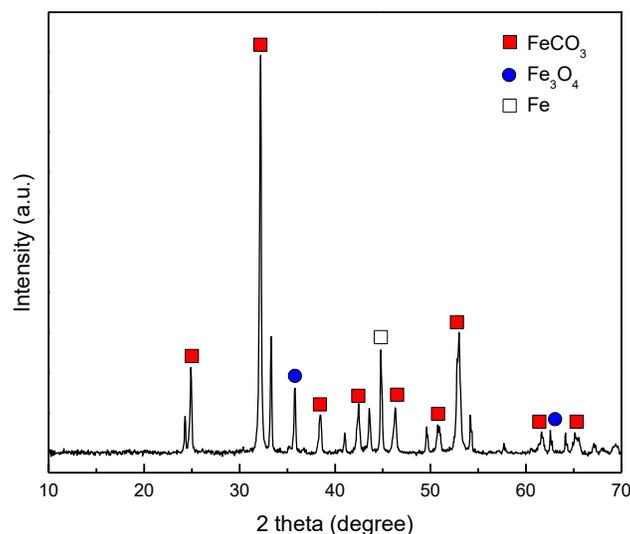
**Figure 5: SEM cross-sectional view of the corroded sample at 12 MPa CO<sub>2</sub> and 160°C.**

In order to identify the compositions of the corrosion product layer and their distribution, cross-sectional EDS mapping analysis was conducted (Figure 6). It shows that the thick compact layer is rich in iron (blue mapping), carbon (red mapping), and oxygen (green mapping). Scattered particles are found above the thick layer, which contains higher relative oxygen content than the thick layer.



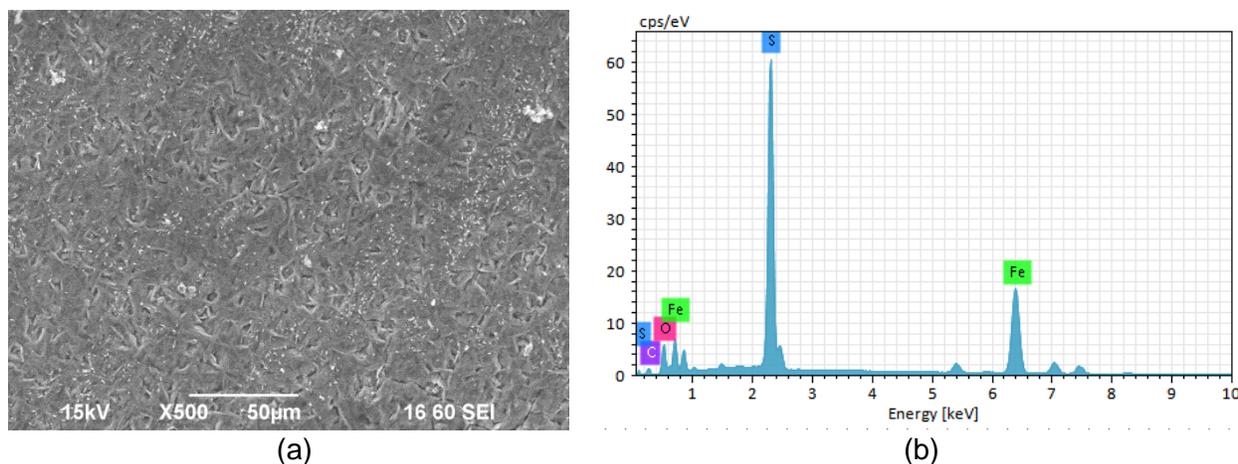
**Figure 6: SEM and EDS elemental mapping images of the cross-section of the corrosion product layer produced after 60 hours of exposure in 12 MPa CO<sub>2</sub> and 160°C.**

Figure 7 shows the XRD pattern of the corrosion product layer formed at 12 MPa CO<sub>2</sub> and 160°C. The layer formed in this condition showed dominant FeCO<sub>3</sub> diffraction patterns with some Fe<sub>3</sub>O<sub>4</sub>. Based on the EDS mapping (Figure 6) and XRD results (Figure 7), it is identified that the thick layer is FeCO<sub>3</sub> and that some Fe<sub>3</sub>O<sub>4</sub> is present possibly on top of the FeCO<sub>3</sub> layer. This indicates that FeCO<sub>3</sub> is a more favored corrosion product than Fe<sub>3</sub>O<sub>4</sub> under the current experimental condition (12 MPa CO<sub>2</sub> and 160°C). The observations also suggest that the relatively high corrosion rate (> 1 mm/y) is attributed to the formation of only FeCO<sub>3</sub>, which is less protective than Fe<sub>3</sub>O<sub>4</sub>.<sup>17,18,25</sup>



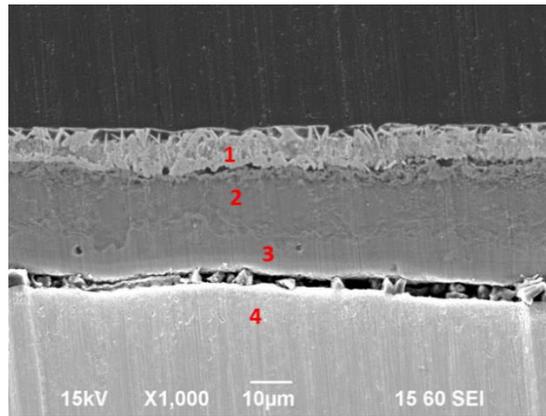
**Figure 7: XRD surface analysis of the CS sample exposed to 12 MPa CO<sub>2</sub> at 160°C.**

Figure 8 shows the surface morphology and chemical analysis of the corrosion product formed at 12 MPa CO<sub>2</sub> and 160°C with 1000 ppm H<sub>2</sub>S. A uniform corrosion product was found covering the exposed surface. In this case, EDS analysis shows that the corrosion product mainly consists of iron and sulfur.



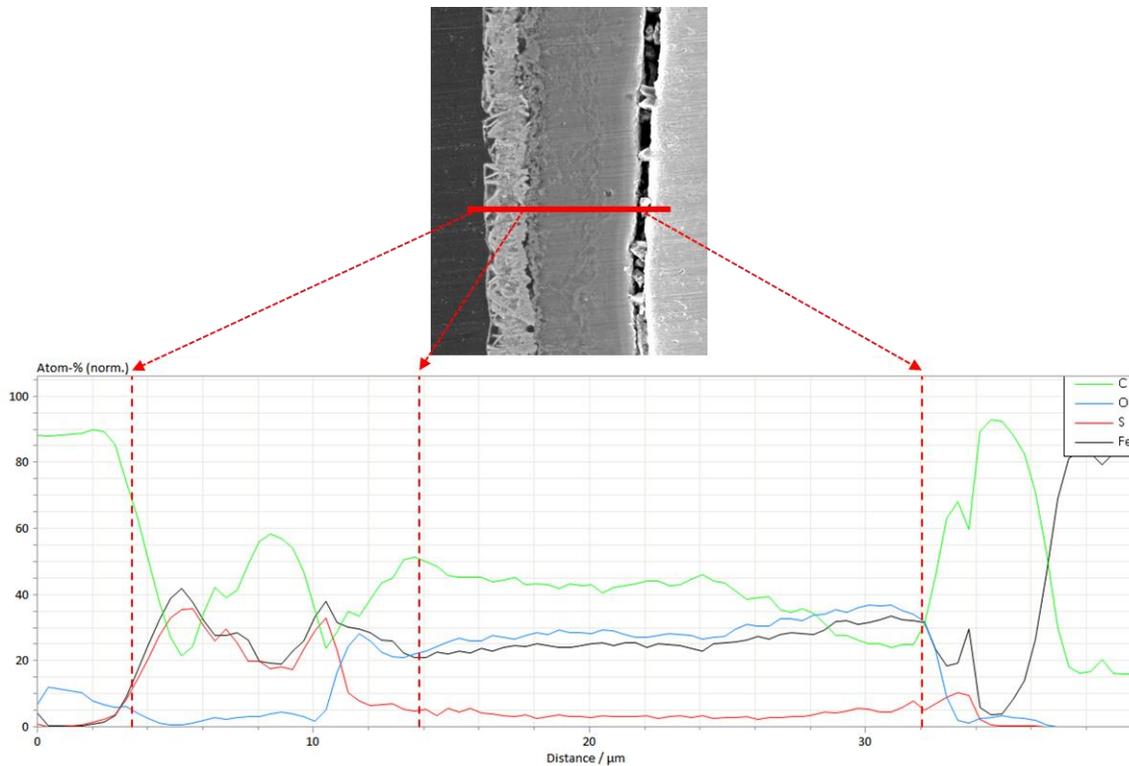
**Figure 8: SEM surface view (a) and EDS analysis (b) of the corrosion product formed at 12 MPa CO<sub>2</sub> and 160°C with 1000 ppm H<sub>2</sub>S.**

Figure 9 represents the cross-sectional morphologies and chemical analysis of the corrosion product formed at 12 MPa CO<sub>2</sub> and 160°C with 1000 ppm H<sub>2</sub>S. It can be seen that it has a two-layer structure; a thin outer Fe and S containing layer and a thick/continuous inner Fe, C and O containing layer. This was also confirmed by the results of the EDS line scanning analysis (Figure 10) and mapping analysis (Figure 11). It is worth noting that there is a gap between the steel surface and the corrosion product, possibly due to the poor adhesion of the inner layer to the substrate. This can consequently reduce the protectiveness of the corrosion product layer and increase the corrosion rate.<sup>23</sup>

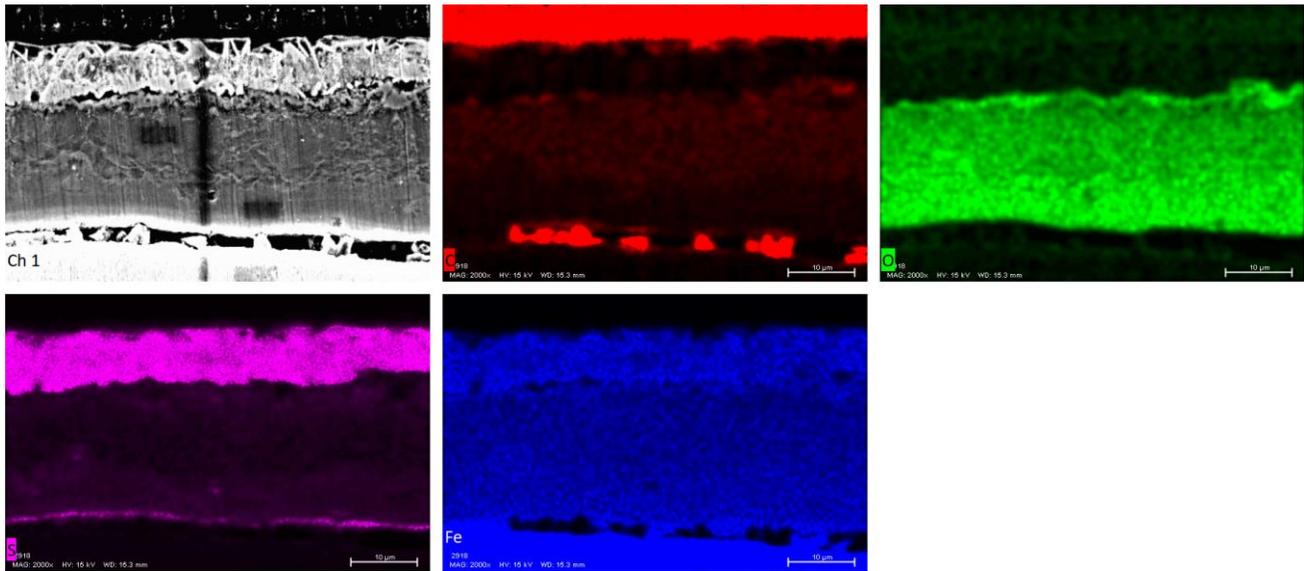


At.%	C	O	S	Fe
1	36.18	1.20	30.39	29.99
2	42.96	28.47	2.49	25.56
3	25.90	36.78	4.49	32.46
4	13.52	-	-	86.48

**Figure 9: Cross-sectional SEM and EDS analysis of the corrosion product formed at 12 MPa and 160°C with 1000 ppm H<sub>2</sub>S.**

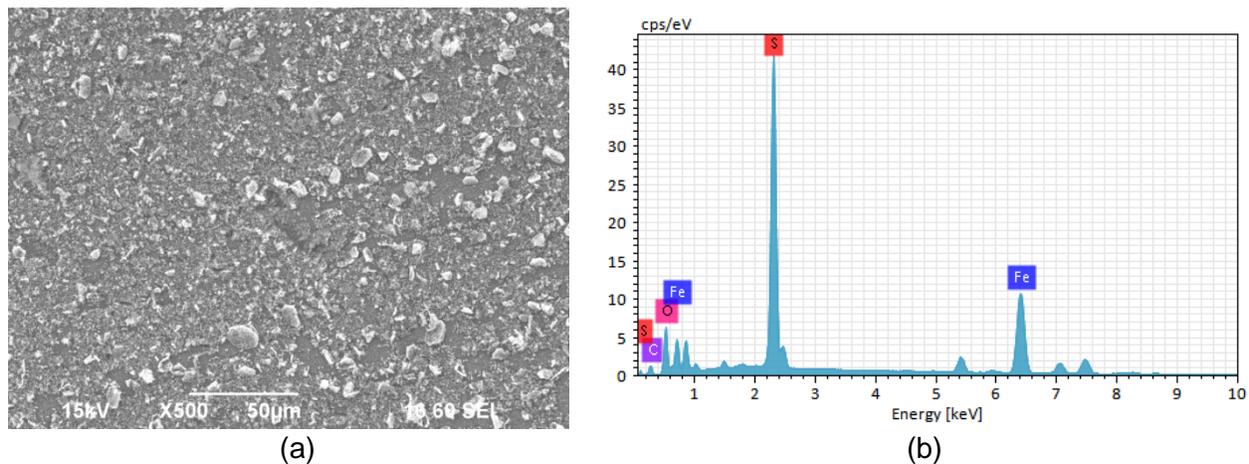


**Figure 10: Cross-sectional EDS line scanning of the corrosion product formed at 12 MPa CO<sub>2</sub> and 160°C with 1000 ppm H<sub>2</sub>S.**



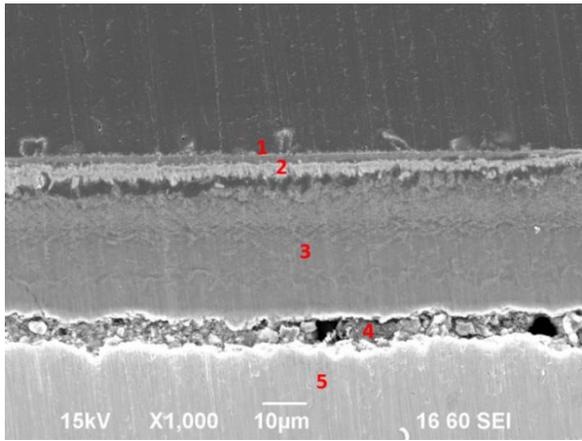
**Figure 11: Cross-sectional SEM and EDS elemental mapping of the corrosion product formed at 12 MPa CO<sub>2</sub> and 160°C with 1000 ppm H<sub>2</sub>S.**

Figure 12 shows the surface morphology and chemical analysis of the corrosion product formed at 12 MPa CO<sub>2</sub> and 160°C with 2000 ppm H<sub>2</sub>S. Similar to the case with 1000 ppm H<sub>2</sub>S, a uniform corrosion product was found covering the exposed surface. EDS analysis shows that the corrosion product mainly consists of iron and sulfur. Small amounts of carbon and oxygen are also detected.



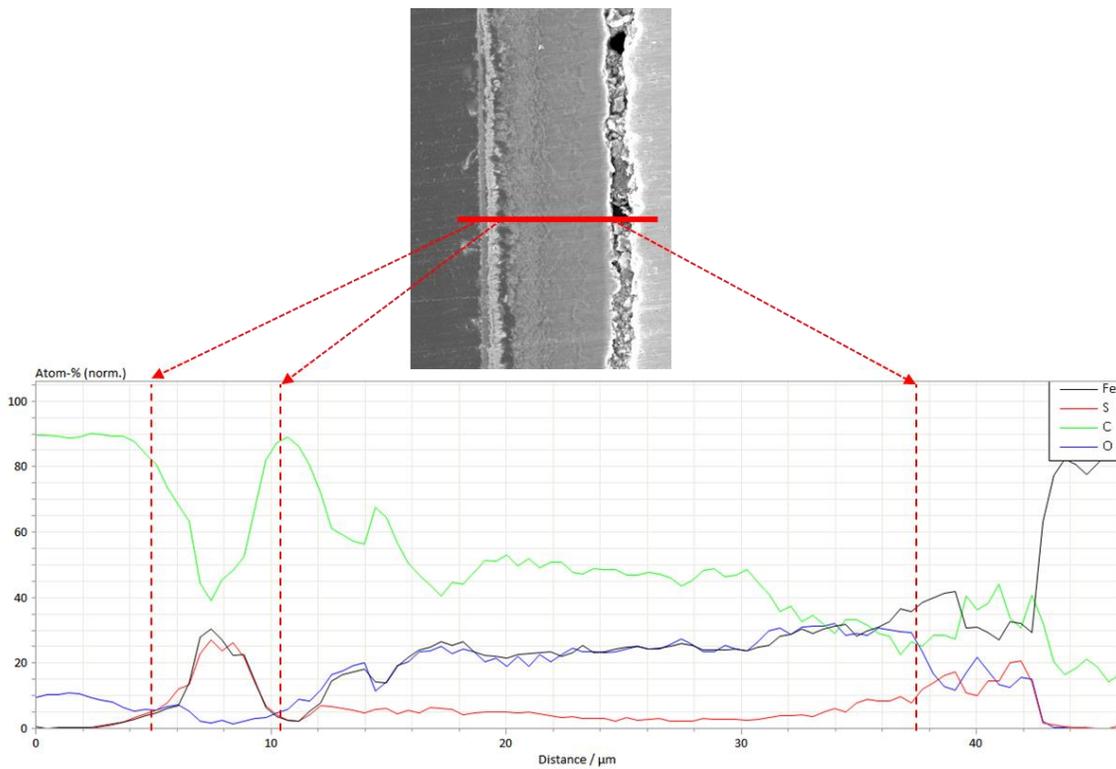
**Figure 12: SEM surface view (a) and EDS analysis (b) of the corrosion product formed at 12 MPa CO<sub>2</sub> and 160°C with 2000 ppm H<sub>2</sub>S.**

Figure 13 to Figure 15 represent the cross-sectional EDS analysis (point, line scanning and mapping) of the corrosion product formed at 12 MPa CO<sub>2</sub> and 160°C with 2000 ppm H<sub>2</sub>S. As in the previous case with 1000 ppm H<sub>2</sub>S, the corrosion product has a two-layer structure; a thin outer Fe and S containing layer and a thick/continuous inner Fe, C and O containing layer. Furthermore, a gap is also found between the inner layer and the metal surface due to the undermining effect of the corrosion process.

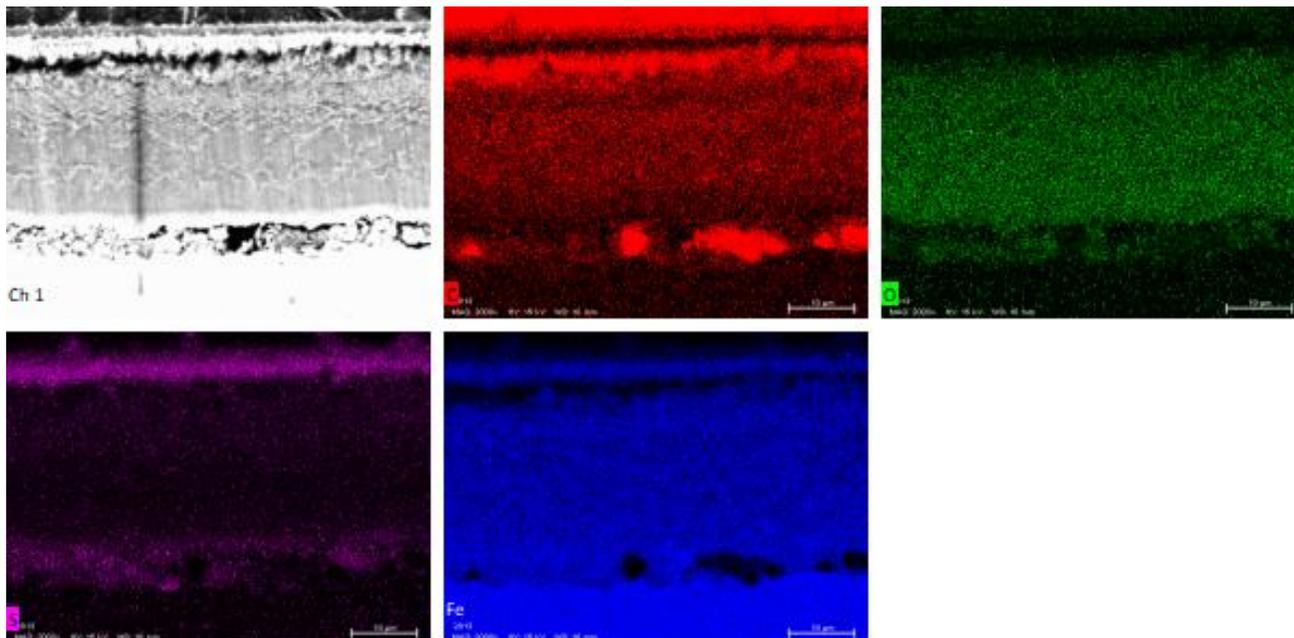


At.%	C	O	S	Fe
1	66.76	6.74	13.04	8.04
2	27.85	0.63	35.81	34.73
3	49.06	21.89	4.70	23.64
4	27.10	10.80	23.44	37.52
5	18.40	-	-	81.60

**Figure 13: Cross-sectional EDS analysis of the corrosion product formed at 12 MPa CO<sub>2</sub> and 160°C.**

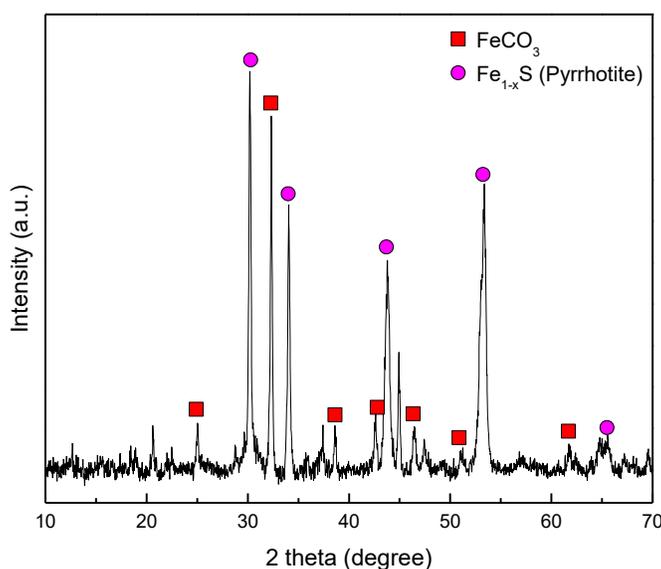


**Figure 14: Cross-sectional EDS line scanning of the corrosion product formed at 120 MPa CO<sub>2</sub> and 160°C with 2000 ppm H<sub>2</sub>S.**



**Figure 15: Cross-sectional SEM and EDS elemental mapping of the corrosion product formed at 12 MPa CO<sub>2</sub> and 160°C with 2000 ppm H<sub>2</sub>S.**

Figure 16 shows the XRD pattern of the corrosion product layer formed at 12 MPa CO<sub>2</sub> and 160°C with 2000 ppm H<sub>2</sub>S. The layer formed in this condition showed FeCO<sub>3</sub> diffraction patterns with pyrrhotite (Fe<sub>1-x</sub>S). This allowed ready identification of the corrosion products formed in CO<sub>2</sub>/H<sub>2</sub>S conditions as Fe<sub>1-x</sub>S for the outer layer and FeCO<sub>3</sub> for the inner layer. It is interesting to note that Fe<sub>3</sub>O<sub>4</sub> was not detected in the corrosion products formed in CO<sub>2</sub>/H<sub>2</sub>S conditions and the predominant corrosion product was FeCO<sub>3</sub>, the same as the CO<sub>2</sub> condition.

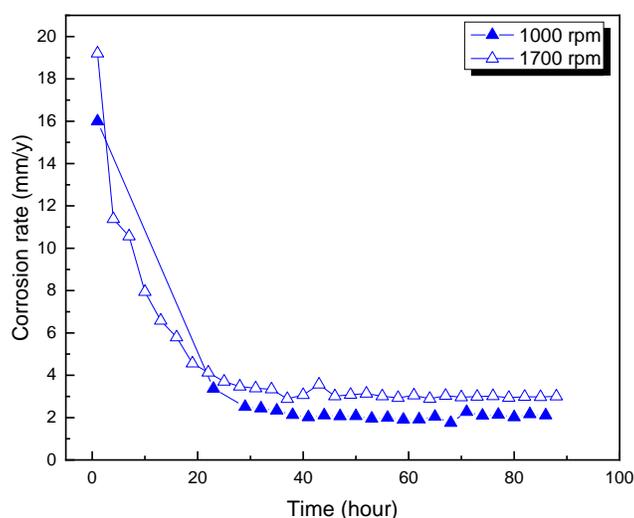


**Figure 16: XRD surface analysis of the CS sample exposed to 2000 ppm H<sub>2</sub>S and 12 MPa CO<sub>2</sub> at 160°C.**

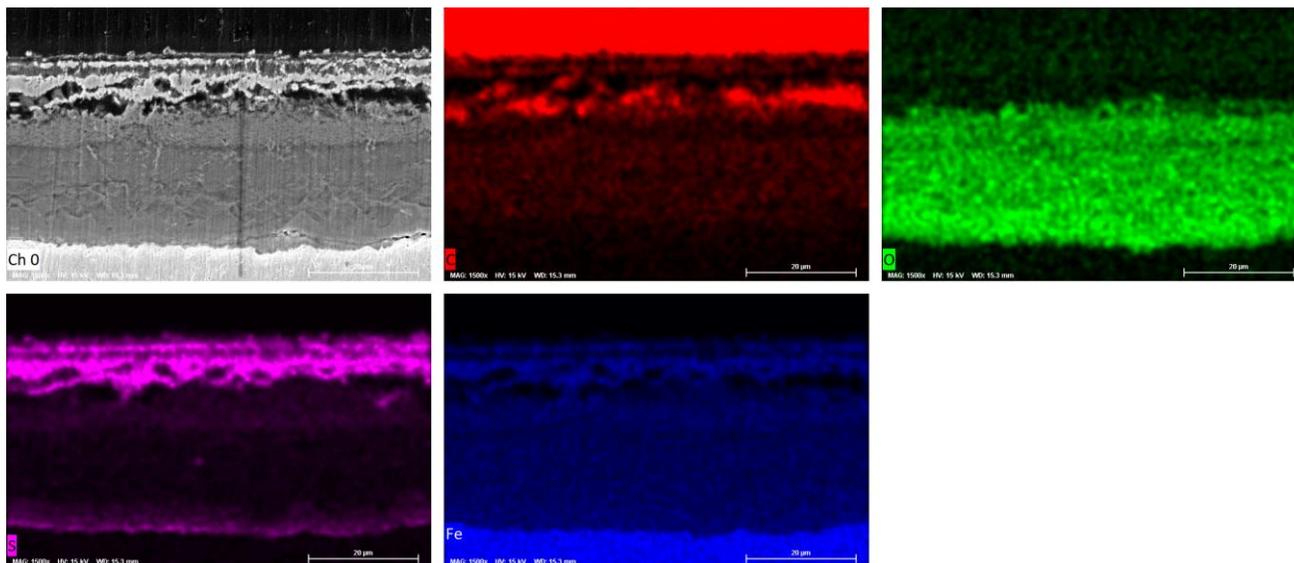
It has been reported that thin and continuous Fe<sub>3</sub>O<sub>4</sub> layer formed at steel surface provides instantaneous corrosion protection at high temperatures (> 120°C).<sup>17,25,26</sup> Although the increase in CO<sub>2</sub> partial pressure makes the formation of FeCO<sub>3</sub> more favorable compare to Fe<sub>3</sub>O<sub>4</sub>, Fe<sub>3</sub>O<sub>4</sub> coexists with FeCO<sub>3</sub> and offers

corrosion protection.<sup>18</sup> However, in the present study, relatively high corrosion rate ( $> 1$  mm/y) was measured with very high  $\text{CO}_2$  partial pressure (12 MPa), and the corrosion behavior was controlled by a thick  $\text{FeCO}_3$  layer. Furthermore, subsequent formation of an outer  $\text{Fe}_{1-x}\text{S}$  layer provided no additional corrosion protection. It can be speculated that increased concentration of  $\text{CO}_2$  species in aqueous environment could affect the thermodynamic stability and kinetics of  $\text{Fe}_3\text{O}_4$  formation. Further work is required to examine the relationship of the  $\text{CO}_2$  partial pressure with the formation of  $\text{Fe}_3\text{O}_4$  at high temperatures.

In order to evaluate the effect of flow velocity on the corrosion behavior of CS at  $160^\circ\text{C}$ , an autoclave test was conducted at higher rotation speed of 1700 rpm. A comparison of the corrosion rates with different rotation speeds (1000 rpm and 1700 rpm) at 12 MPa  $\text{CO}_2$  and  $160^\circ\text{C}$  with 2000 ppm  $\text{H}_2\text{S}$  is shown in Figure 17. For both cases, the corrosion rate decreased with time and slightly higher corrosion rate was measured for the case with 1700 rpm compared with 1000 rpm. The morphology and composition of the corrosion product were almost identical with the 1000 rpm case (Figure 18) indicating that the corrosion behavior of CS in the  $\text{CO}_2/\text{H}_2\text{S}$  condition does not vary within the tested flow velocities. This also implies that the surface water chemistry is supersaturated with respect to  $\text{FeCO}_3$  even in conditions where mass transfer is accelerated by flow.<sup>17,27</sup>



**Figure 17: Comparison of corrosion rates of CS with different rotation speed of impeller at 12 MPa  $\text{CO}_2$  and  $160^\circ\text{C}$  with 2000 ppm  $\text{H}_2\text{S}$ .**



**Figure 18: Cross-sectional SEM and EDS elemental mapping of the corrosion product formed at 12 MPa CO<sub>2</sub> and 160°C with 2000 ppm H<sub>2</sub>S (1700 rpm).**

## CONCLUSIONS

The effect of H<sub>2</sub>S on the aqueous corrosion behavior of CS was evaluated at high pressure (12 MPa) and high temperature (160°C) in supercritical CO<sub>2</sub> conditions. The following conclusions are drawn:

- No significant difference in the corrosion rate in the presence of H<sub>2</sub>S was observed.
- The predominant corrosion product was FeCO<sub>3</sub> for all conditions, indicating that the formation of FeCO<sub>3</sub> is more favorable than that of FeS and Fe<sub>3</sub>O<sub>4</sub> at these conditions.
- No significant effect of flow velocity was observed on the corrosion behavior of CS.

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