

TOP OF THE LINE CORROSION IN SOUR ENVIRONMENT – STUDY OF THE CONTROLLING PARAMETERS

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SUMMARY: Under stratified flow and dewing conditions, internal corrosion can occur at the top of horizontal pipelines where continuous injection of corrosion inhibitors does not have a mitigating effect. A considerable amount of research work has already been performed on sweet TLC, and field data are also widely available on this topic. However, several pipe failures attributed to sour TLC have been also reported. The mechanisms controlling the corrosion process in sour environments seem to be quite different from those in sweet environments. This work is focused on identifying the main controlling parameters in sour TLC and listing the remaining factors whose effects are not yet quite clear. Original experimental results obtained in a specially designed “condensing” autoclave are also presented.

Keywords: Top of the line corrosion, H₂S, CO₂, Water condensation rate.

1. INTRODUCTION

Top of the Line Corrosion (TLC) is a corrosion phenomenon driven by water condensation, and specific to the 9 to 2 o'clock position on a pipeline circumference. It is a process exclusively encountered in stratified flow regimes, as standard corrosion inhibitors - typically present in the bottom of the line brine - cannot provide any protection to the top section of the pipe. Corrosion control techniques such as batch treatment or spray pigging are often implemented to mitigate TLC, but these methods are costly and intrusive.

Numerous cases of sweet (CO₂ dominated) TLC field cases have been reported in the literature [1 - 4]. In these cases, areas of pipeline suffering failure or severe corrosion issues could be related to inadequate or damaged thermal insulation. Associated with high fluid temperatures (>50°C), these areas would be subject to local high rates of water condensation. Corrosive gases such as carbon dioxide (CO₂) or organic acid vapors would dissolve in the condensed water and lead to steel dissolution. High rates of water condensation would ensure that dissolved iron would be rapidly transported away from the steel surface, preventing the formation of a protective iron carbonate (FeCO₃) layer. CO₂ dominated TLC has been the focused of an intense research effort over the past ten years, both in terms of experimental [6 - 10] and modelling [11 - 14] work. The water condensation rate, fluid temperature, and CO₂/organic acid content have been identified as the controlling parameters of sweet TLC [10]. Field failures have often been experienced where the fluid is the hottest, leading to strong gradients of temperature between the outside environment and the process fluid, and consequently to high water condensation rates. In addition, the presence of large concentrations of organic acid was found to considerably enhance the rate of corrosion [2]. No TLC issues were experienced at lower temperatures or where WCR was below a critical value. Engineering guidelines have been proposed: critical WCR of 0.25 mL/m²/s in the absence of organic acid and 0.025 mL/m²/s in the presence of organic acid [3]. However, these critical values should be taken with caution as they logically depend on the other field conditions, such as temperature and corrosive gases contents.

Although the basic mechanisms of sweet TLC are fairly well understood, there are still some unanswered questions subject to ongoing research. The impact of hydrocarbon co-condensation on TLC is not well defined [15] although it is assumed that it should be marginal, since the steel surface is hydrophilic. A mechanism of TLC stabilization, where the rate of corrosion would decrease with time and remain a negligible value, has also been put forward [16, 17], but it is not clear whether the observed decrease in corrosion rate is due to a genuine mechanism or changes in production parameters. In addition, a variety of innovative inhibition techniques are being investigated as an alternative to batch treatment (use of volatile inhibitors [18, 19], foam matrix containing inhibitors [20], spreading inhibitors [21] etc.) However, their efficiency is not yet completely proven.

In terms of sour (H₂S dominated) TLC, the mechanisms seem to be more complicated. Sour TLC seems to be a lot less widespread than its sweet counterpart, but it has nonetheless been identified as a cause of pipe failures. Corrosion

mechanisms are not understood, which is logical considering that bottom of the line sour corrosion not very clear either. The main goal of this paper is to compile relevant field and laboratory experience available in the literature, as well as in-house data, to draw some preliminary characteristics of sour TLC and to identify remaining gaps in the understanding.

2. REVIEW OF FIELD EXPERIENCE

2.1 Review of field experience

There are only a handful of fairly well-documented sour TLC field failures described in the literature [22 - 26].

The first ever reported TLC case was actually from a sour gas field in France [22]. A serious corrosion issue (Figure 1) was discovered on the top part of a horizontal, buried 6" pipeline transporting a mixture of light hydrocarbons and inhibited water under stratified flow. The gas composition was 9% CO₂ and 15% H₂S and the fluid temperature was between 35°C and 50°C. In these conditions, the water condensation rate should have been extremely low. The corrosion issue was identified as TLC and was mitigated by increasing the gas flow rate and switching from stratified to annular flow regime.



Figure 1 Corrosion in the upper part of the pipe
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Another case was also reported from a field in Canada [23, 24]: a 6" pipe made of API 5LX52 carrying produced gas (5.9 % of CO₂ and 0.3% of H₂S) at an operating pressure of 68 bars. The pipe was split open at the 4 o'clock position (Figure 2) in a straight longitudinal line for the entire length of a pipe section. In the failed section, wall loss was measured up to 64% of the original nominal wall thickness. The corrosion mechanism was identified as TLC due to the condensation of water vapor in presence of acid gases, in the absence of hydrocarbon condensate.

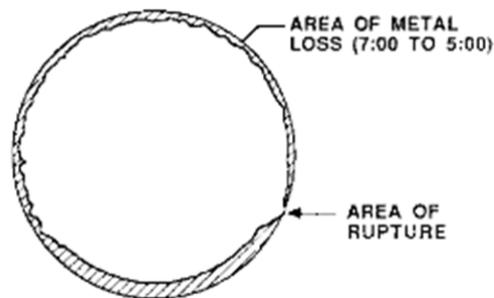


Figure 2: Cross sectional diagram of the pipe failure
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A third case was reported in North Dakota [25], again with low inlet temperatures (below 40°C) and stratified flow regime. The gas contained 2.6 mole % CO₂ and 16.4 mole % H₂S at total pressure around 500 psi. The flow line (10" ID) was pigged every 5 days. The failure occurred on the top of the line, in a segment immediately following an elevation drop.

A more recently reported pipe failure [26] involved a 6" ID onshore pipeline in Canada which occurred also in Canada transporting wet gas at 5.45% CO₂, 2800 ppm H₂S at the time of rupture. The fluid temperature was again quite low (15 °C) which should have led to very small water condensation rates. The line ruptured after 8.5 years of operation with extensive reported wall loss (60 to 85%) on the upper part of the pipe.

It is important to mention that methanol injection was often used in these cases and that it has been determined to be a contributing factor. Due to its high solubility in methanol, oxygen was thought to be introduced in significant enough quantity to impact H₂S corrosion and lead to the formation of sulfur.

Cases where TLC was not detected are also valuable. Two 8" ID wet gas flow lines (few kilometers long) transporting sour gas (although contents are not specified in the publication [3]) with respective inlet and outlet pressures at 116 bars and 107 bars. Inlet temperatures are reported at 63°C and 74°C with corresponding outlet temperatures at 29°C and 41°C. These lines have been in operation for 12 years and ultrasonic scanning showed no sign of TLC.

Bonis [27] listed the main findings of a review of intelligent pigging operations performed on offshore and onshore wet sour gas pipelines. The production conditions ranged from 80 to 40°C inlet temperature, 50 to 20 °C outlet temperature, 2 to 17% H₂S, 3 to 10 % CO₂ and 2-3 to 10 m/s. No indication of serious TLC was ever detected.

A summary of the production parameters of each of the cases briefly described above is shown in Table 1.

Table 1. Summary of sour TLC experience in the field

Location	Lacq / France [22]	Canada / Crossfield [23, 24]	N. Dakota / USA [25]	Alberta / Canada [26]	Offshore and onshore [27]
Gas temperature	35°C to 50°C	40°C	36.7°C	15°C	80 to 20°C
Total pressure	117 bars	68 bars	34 bars	62 bars	Not indicated
CO ₂ content	9 %	5.9 %	2.6 %	5.4 to 13.4%	3 to 10 %
H ₂ S content	15 %	0.3%	16.4%	2800 to 5300 ppm	2 to 17%
Gas velocity	3.4 m/s	3 to 3.5 m/s	0.3 to 0.8 m/s	<0.1 m/s	2-3 to 10 m/s
Flow regime	Stratified	Stratified	Stratified	Stratified	Not indicated
TLC damage	27% WL in 1.5 years	Pipe split with 64% WL on upper part	TLC pipe failure	TLC pipe failure	No TLC
Possible other contributing factors	Glycol, Methanol	Preferential weld corrosion	Methanol	Methanol	Not indicated

2.2 Common observations

These pipe failures are clearly due to corrosion by water condensation in stratified flow and can be qualified as TLC, although the mechanisms are quite different as compared with sweet TLC. It is difficult to draw any definitive conclusion, but some attempts can be made to highlight the common characteristics [28]. A direct comparison with sweet TLC, which is better understood, also carries a definitive value.

- Field occurrence of sour TLC does not seem to be as common as sweet TLC, but still leads to pipe failure,
- Contrary to sweet TLC, where most of the cases are offshore (leading to large temperature gradients), most of the sour TLC cases are located onshore, sometimes with buried pipelines.
- The temperature difference between the produced fluid and the outside environment does not seem to be a driver for corrosion. Although condensed water is obviously a factor, the rate of condensation seems inconsequential. This constitutes an important difference compared to sweet TLC.
 - Most sour TLC cases are experienced at low gas velocity. Since all of the lines were inhibited, droplet transport could play a role at higher velocity even if the flow regime is still stratified.
 - All of the reported sour TLC cases happened at relatively low temperatures, which suggests that the severity of the corrosion attack could depend strongly on the balance of iron dissolution and scale formation kinetics.
 - It is possible that the type and protectiveness of the iron sulphide film formed at the steel interface is a key parameter controlling sour TLC.
 - As mentioned earlier, the injection of methanol and consequent introduction of oxygen is also a common factor in most of the cases and should not be ruled out.
 - Although the corrosion is clearly localized to the upper part of the pipe (the inhibited bottom of the line is usually reported as clean), the failure mechanism is often characterised as general wall thinning instead of pitting corrosion.
 - The H₂S/CO₂ ratio does not seem to be critical as long as the environment is H₂S dominated.

3. REVIEW OF LABORATORY UNDERSTANDING OF SOUR TLC

Sour TLC has been the subject of focused research only recently. It is fair to say that even "standard" (bottom of the line) sour corrosion is not really understood [29], making any attempt to understand sour TLC rather confusing.

Camacho [30] presented a series of short term (2 to 4 days long) experiments carried out in a 4"ID flow loop. The tests were performed at 3 bars total pressure, at 70°C and at a gas velocity of 5m/s ensuring stratified flow. The condensation was kept constant at 0.25 mL/m²/s. The presence of small quantities of H₂S (up to 0.13 bar) led to a strong decrease in the general corrosion rate compared to its pure CO₂ counterpart. The corrosion was strictly uniform and did not seem to be greatly affected by the range CO₂/H₂S ratio tested. An FeS film was always found to be the predominant corrosion product scale present on the carbon steel surface. As is customary in top of the line corrosion testing, the short duration of the experiment made it difficult to extrapolate laboratory corrosion rates to field reality.

Nyborg [31] presented a series of experiments in a 2"ID flow loop using carbon steel tubes as corrosion samples. The tests were conducted at 25 °C, with 0.02 bar of H₂S and 10 bars of CO₂ and with 300 ppm of acetic acid. The experiments lasted for more than 30 days at very low condensation rates (lower than 0.006 mL/m²/s). The corrosion rate was very stable during the entire test duration. A porous and fluffy FeS film was found on top of a more protective FeCO₃ layer covering the metal surface. It was proposed that dissolved H₂S acted as a "sink for ferrous ions" by promptly forming an un-protective FeS layer and enabling the corrosion to continue, albeit at a low rate (0.1 mm/year).

Pugh [32] performed experiments in an autoclave equipped with top of the line corrosion capabilities. The aim was to simulate specific field conditions where TLC was observed. The tests were conducted at 25 and 55°C and at condensation rates of 0.002 and 0.1 mL/m²/s respectively. The gas phase was made of 2.4% CO₂ and 1% H₂S and the test was performed over a 6 to 10 weeks period. The results showed that the corrosion rate was higher at a lower temperature and lower condensation rate (25°C and 0.002 mL/m²/s) than at a higher temperature and higher condensation rate (55°C and 0.1 mL/m²/s). In both cases, a mackinawite film formed on the metal surface but had different characteristics depending on the temperature; at 25°C, the film is fluffy, porous, crystalline with 500nm grains and un-protective; at 55°C, the film was denser, crystalline with 10 microns grains and protective. The presence of organic acid increases the general corrosion rate and promotes localized corrosion, especially if the FeS film was protective.

Singer [33] continued Camacho's work [30] and conducted a parametric study in a 4" ID flow loop. The partial pressure of H₂S (up to 0.13 bar) and the acid acetic concentration (up to 1000 ppm) were studied in a series of 21-day experiments. In the presence of H₂S, the presence of acetic acid seemed to affect the integrity of the FeS film and trigger the occurrence of localized corrosion initiation.

4. NEW EXPERIMENTAL WORK IN ORIGINAL TLC AUTOCLAVE

4.1 Experimental setup

The experiments presented in this paper were conducted in a 20L autoclave made of alloy C-276 (UNS N10276). The autoclave is specially manufactured to enable corrosion measurements under condensing conditions. The top lid of the autoclave is equipped with an internal cooling system and the sample holder plate (Figure 3). The steel samples have a diameter of 5.7 cm and are made of API X65 steel. They were coated with an electrical insulator except on the face exposed to the vapour phase. Prior to each test, the samples were polished using 600 grit sand paper and cleaned with isopropanol alcohol. The design of the sample holder enables the study of the effect of the condensation rate in one single test. This was done by "hanging" some of the steel samples in the gas phase but away from the cooled plate. No samples were immersed in the bulk liquid phase.



Figure 3: 20L autoclave setup (left) and details of the sample holder (right)

4.2 Experimental conditions and procedure

The two tests presented in this study were performed at the same time using the specially designed sample holder, which enables 4 samples to be cooled and 4 other samples to be insulated from the cooling system. At the beginning of the test, 8 liters of de-ionized water was introduced in the autoclave and de-oxygenated for two hours before 2.8 ml of pure acetic acid was introduced. The sample holder was then attached to the top lid and the autoclave was sealed, heated to the required

temperature and pressurized with N₂ at 2 bars total pressure. Pure H₂S gas was then bubbled until the total pressure reached a stable reading of 6 bars (i.e. p_{H₂S}= 4 bars). In the same manner, CO₂ was added to reach a partial pressure of 10 bars, and the pressure was increased up to 25 bars with N₂. The concentration of H₂S in the gas phase was measured at the end of the test at 16%, which corresponds to 4.28 bars. The Fe²⁺ concentration in the liquid phase was measured at the end of the test a 9.5 ppm. According to calculation, the pH of the main liquid bulk solution should have remained around 3.4-3.5 during the three weeks of testing. The temperature of the steel sample holder was measured using a thermocouple, and the water condensation was calculated using an in-house heat transfer model.

At the end of the test, the gas phase was purged for a few hours with nitrogen before opening the autoclave and removing the steel samples. A liquid sample was then taken for acetic acid analysis which returned a concentration of 290 ppm. The steel samples were dried and weighed. X-ray diffraction (XRD), scanning electron diffraction (SEM) and Electron dispersive spectroscopy (EDS) analyses was performed before the ASTM G1 [34] procedure was followed to remove the corrosion products and determine the corrosion rate by weight loss. Surface profile analysis was then performed to investigate the extent of localized corrosion.

The test conditions are summarized in Table 2:

Table 2. Test conditions

Parameters	Test A	Test B
Total pressure (bar)	28	
Gas temperature (°C)	55	
H ₂ S partial pressure (bar)	4	
Sample temperature (°C)	50	24.4
CO ₂ partial pressure (bar)	10	
CO ₂ /H ₂ S ratio	2.5	
Acetic acid (ppm)	280	
Condensation rate (mL/m ² /s)	0.02	0.12

4.3 Corrosion rate results and surface analysis

The corrosion rate results are shown in Figure 4. Error bars (maximum and minimum values) as well as number of samples are also displayed in the graph. The corrosion rates are quite low in both cases and the water condensation rate seems only to have a slight impact on the overall mass loss.

4.3.1 Test A – WCR=0.02 mL/m²/s

Photographs of the weight loss samples taken immediately after the end of the test are shown in Figure 5.

The coupons all had very similar aspects with a grey layer covering the surface. Some circular-shaped features observed on the sample surface were an indication of the presence of droplets of condensed water staying attached to the metal surface for a long time. This is also an indication that the condensation rate on the samples was low. The corrosion product layer seemed non uniform but no breakdowns could be observed. Very peculiar features could be observed on the SEM all identified to be FeS with the EDS. The variety of the shapes observed infers that potentially different phases of FeS formed on the steel surface. However, the XRD analysis performed on the coupon surface identified the presence of mackinawite. Traces of triolite were also detected (Figure 6).

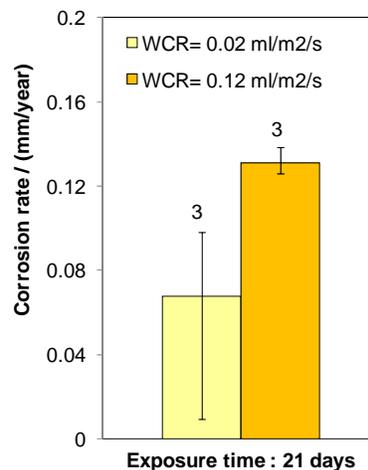


Figure 4: Weight loss corrosion rate at the top of the Line

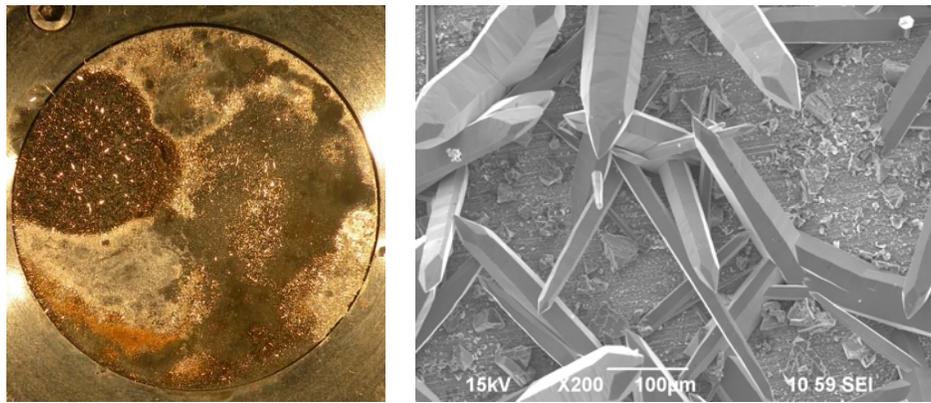


Figure 5: Test A (WCR=0.02 mL/m²/s) - Pictures of the steel sample after the test

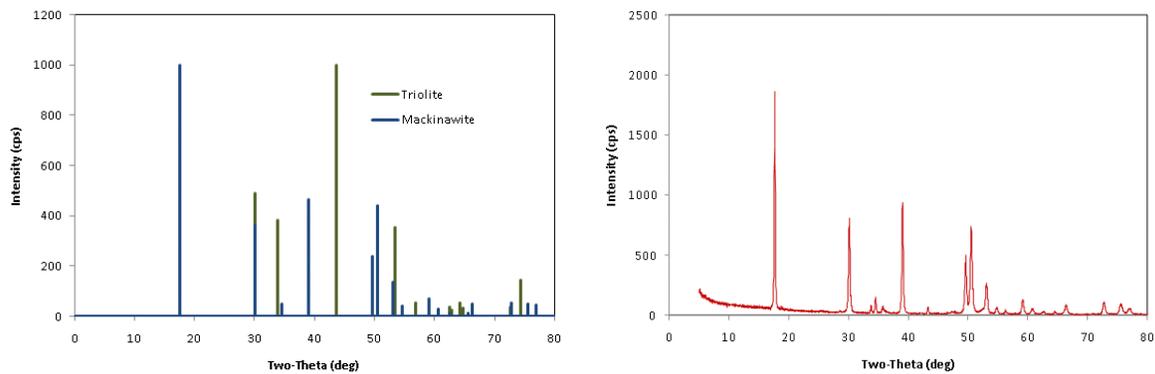


Figure 6: Test A (WCR=0.02 mL/m²/s) – XRD analysis

The cross section analysis is shown in Figure 7. The film seems to be made of two layers lying on top of each other. A first dense and seemingly adherent layer covers the steel surface with a thickness around 10 µm (which is quite more than the average 4 µm wall loss). On top of this first layer, larger features could be found, which corresponds to the crystals observed in the SEM image. Neither layer has any significant difference in terms of chemical composition. The overall roughness of the steel surface does indicate that the corrosion attack was uniform, which was confirmed by the surface profile analysis.

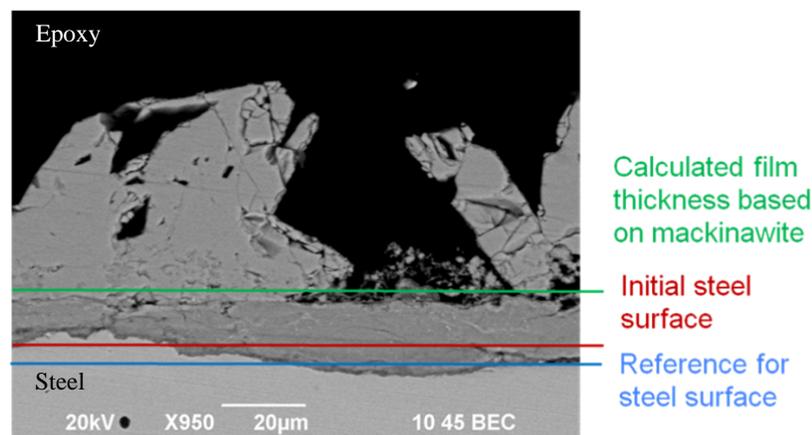


Figure 7: Test A (WCR=0.02 mL/m²/s) – Cross section analysis

4.3.2 Test B – WCR=0.12 mL/m²/s

Photographs of the weight loss samples taken immediately after the end of the test are shown in Figure 8. Compared with test A, no circular-shaped features could be observed on the sample surface. This could be an indication of a greater droplet renewal rate, and consequently a higher condensation rate. No clear indication of breakdowns in the layer could be observed

either. Crystalline features could be observed on the SEM, all identified to be FeS with the EDS. XRD analysis performed on the coupon surface identified the presence of mackinawite.

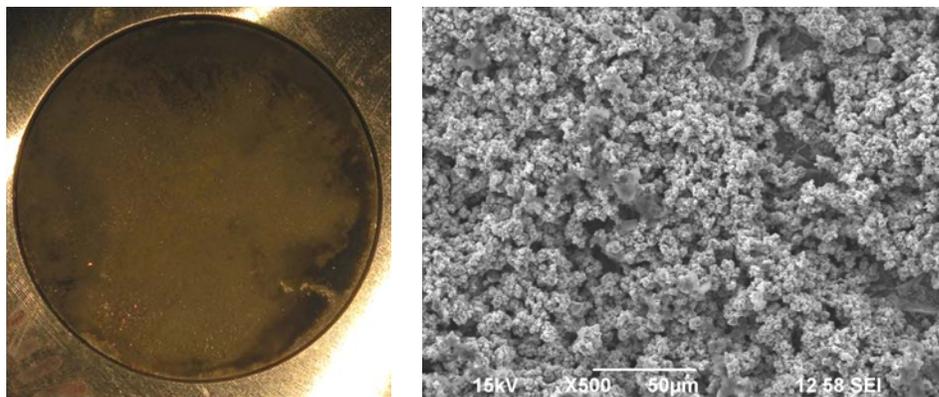


Figure 8: Test B (WCR=0.12 mL/m²/s) - Pictures of the steel sample after the test

The cross section analysis is shown in Figure 9. A thick, very porous layer covers a more dense and adherent film. The porous layer is about 25-30 µm thick, while the dense layer is, on average, only 10 µm thick. By comparison, the wall loss corrosion is 7.5 µm. The small crystals “trapped” in the epoxy matrix correspond to those observed in the SEM images. Once again, there was no chemical composition difference between the two layers, and the corrosion was uniform everywhere on the layer.

The surface profile analysis showed no sign of localized corrosion

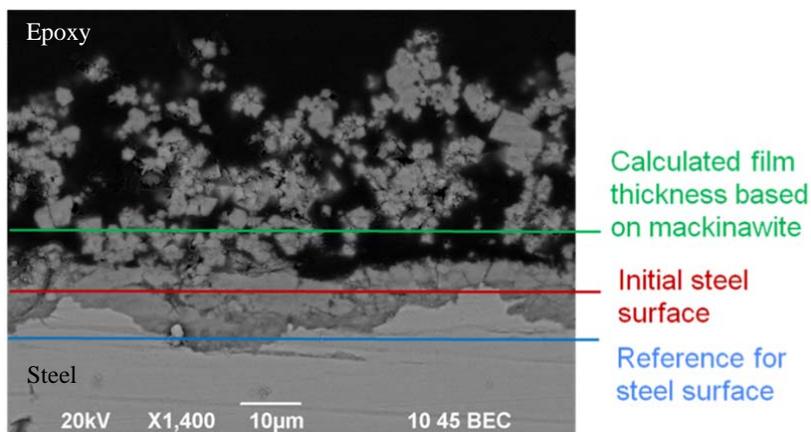


Figure 9: Test B (WCR=0.12 mL/m²/s) – Cross section analysis

4.4 Summary

With a pCO₂/pH₂S ratio of 2.5, the system seems to be clearly sour dominated. The corrosion product formed is Fe_xS_y identified mainly as mackinawite. Large crystals (possibly triolite) are also seen growing on top of a more adherent layer at higher steel temperature (lower condensation rate). No pitting corrosion could be found at an exposure time of 21 days. The water condensation seems to have an effect of the uniform corrosion rate although further tests (not reported here) did not confirm this observation.

5. DISCUSSION

Sour TLC is indeed a complex phenomenon and a lot of work remains to be done in order to better define the mechanisms and to provide tools for prediction in field applications. This part aims at listing some of the more pressing remaining interrogations, in the author’s view, and to try to give some beginning of answer when possible.

- What constitutes a H₂S dominated environment in TLC?

Since sweet and sour TLC seems to have very different controlling mechanism, it is crucial to be able to predict when the corrosion will be CO₂ or H₂S dominated. Laboratory experience shows that trace amounts of H₂S is enough to suppress CO₂ TLC and make Fe_xS_y the dominant component of the corrosion product layer. The threshold ratio of CO₂/H₂S partial pressure [35] used in the industry to define the transition between CO₂ and H₂S (pCO₂/pH₂S = 500) dominated corrosion is often not validated by these experiments. Recent work [36] performed on the validity of this threshold ratio showed that it

was highly sensitive to the accuracy of the thermodynamic input data and should not be used as an engineering tool. Instead, comprehensive chemistry model including FeCO_3 and FeS precipitation should be considered.

- What is the effect of pCO_2 and pH_2S on sour TLC?

The partial pressure of CO_2 does not have a significant effect on the general TLC as long as the system is H_2S dominated. The content of H_2S itself cannot be easily correlated to the severity of the corrosion attack.

- What kind of corrosion product film form at the metal surface?

In the presence of H_2S , a rapid formation of a FeS film (mainly mackinawite) is always observed. The pH in the condensed water should be considerably lower than in sweet TLC as FeS formation does not require saturation (less Fe^{2+} in solution). The mechanisms of FeS formation or precipitation are not clear and many types of film have been encountered [37]. Metastable mackinawite seems to be always present in laboratory testing and cubic FeS and triolite could also form in acidic environment like condensed water. The phase of the FeS layer that forms on the steel surface may be the key to understand and predict its degree of protectiveness.

- What is the effect of temperature on the protectiveness of the film?

The protectiveness of the FeS film formed on the metal surface does depend strongly on the temperature at which it forms and on the water chemistry. The FeS films seem to offer less protection against corrosion at lower temperature (20-50°C) than at higher temperature.

- Is the water condensation irrelevant?

Since most of the TLC failure observed in the field at very low condensation rates, the WCR should not be a relevant parameter. This is confirmed to some extent by laboratory testing, the protectiveness of the FeS being the main factor affecting sour TLC.

- What is the effect of organic acid on sour TLC?

In the presence of H_2S , the presence of acetic acid seems to affect the integrity of the FeS film. As an additional acid in solution, it does influence the general corrosion rate by affecting the water chemistry and possibly providing an additional cathodic reaction.

- Is sour TLC localized corrosion (pitting) or general wall thinning?

Field experience seems to infer that sour TLC occurs more through general wall thinning than pitting. This seems to be confirmed in large part in laboratory testing although it is too early to make any definitive conclusion.

- Is droplet transport from the bottom to the top of the line an efficient way to inhibit the small amount of condensed water?

Since most of sour TLC occurs at low water condensation rate, droplet transport could be an efficient way (when applicable) to provide some degree of inhibition to the top of the line. The quantity of inhibitor needed to be transported could be relatively small compared to a sweet TLC scenario.

6. CONCLUSIONS

Field and laboratory experience of sour TLC were reviewed in this work. Pipeline failures due to H_2S dominated TLC remains a rare event. Although the conditions leading to reported TLC cases are not completely clear, they have been associated to low temperatures (<50°C), low condensation rates, low gas velocities and possibly some oxygen contamination. Sour TLC is currently the focus of several research efforts where the complexity of H_2S corrosion can be better studied. So far, efforts to determine corrosion mechanisms have not been completely successful although some main characteristics of sour TLC are now identified. Contrary to its sweet counterpart, sour TLC should not be controlled by the water condensation rate but should rather depend on the protectiveness of the iron sulphide layer that forms on the steel surface. The determination of the characteristics and formation process of this film should provide a better insight on the overall mechanism.

7. ACKNOWLEDGMENTS

The author would like to acknowledge the financial support of Saudi Aramco for this research work.

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