

## **CRACKING CORROSION OF LOW CARBON STEEL IN ENVIRONMENT WITH A HIGH CONCENTRATION OF CO<sub>2</sub> AND H<sub>2</sub>S**

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### **ABSTRACT**

Cracking corrosion of API 5CT Grade L80 Type 1 low carbon steel has been studied in a brine solution with H<sub>2</sub>S 12.3 psia and CO<sub>2</sub> 9.4 psia. Testing was performed according to the methodology reference from the NACE TM0177, Bent-beam test method in solution B for stress corrosion cracking and sulfide stress corrosion cracking test and NACE TM0284, immersion test method in solution A for Hydrogen induced cracking test.

The obtained results showed pitting and general corrosion at both temperatures of 24 °C and 82 °C. In case of stress corrosion cracking (SCC) testing at 82 °C, microscopy of the samples tested for 30 days developed pitting corrosion in the surface and cracking starting in the surface of the samples. The cracks, mostly found in the middle of the samples where the maximum bending occurred. General corrosion was also observed in the samples, with significant decrease in the dimensions of the samples after testing (due to general corrosion). However, in case of sulfide stress corrosion (SSC) and hydrogen induced cracking (HIC) tests at room temperature (24-25 °C), no cracking was observed on the sample.

*Keywords:* stress cracking corrosion (SCC, SSC, HIC), CO<sub>2</sub> and H<sub>2</sub>S corrosion, low carbon steel.

### **1. INTRODUCTION**

The presence of carbon dioxide (CO<sub>2</sub>), hydrogen sulfide (H<sub>2</sub>S) and free water can cause severe corrosion problems in oil and gas pipelines. Internal corrosion in wells and pipelines is influenced by temperature, CO<sub>2</sub> and H<sub>2</sub>S content, water chemicals, flow velocity, oil or water wetting and composition and surface condition of the steel.

In oil and gas, the presence of CO<sub>2</sub> and H<sub>2</sub>S dissolving in water create an acid media, which make corrosion of internal pipelines and equipment. When corrosion products are not deposited on the steel surface, very high corrosion rates of several millimeters per year can occur. The corrosion rate can be reduced substantially under conditions where iron carbonate

(FeCO<sub>3</sub>) can precipitate on the steel surface and form a dense and protective corrosion product film. This occurs more easily at high temperature or high pH in the water phase. When H<sub>2</sub>S is present in addition to CO<sub>2</sub>, iron sulfide (FeS) films are formed rather than FeCO<sub>3</sub>, and protective films can be formed at lower temperature, since FeS precipitates much easier than FeCO<sub>3</sub>. Therefore, the presence of H<sub>2</sub>S can make decrease the corrosion rate but may conduit H<sub>2</sub>S cracking corrosion [1]. This process can happen at relatively low temperatures, largely because of atomic hydrogen from wet H<sub>2</sub>S corrosion reactions, which enter the steel and collect at inclusions or impurities within the steel. This happens because the H<sub>2</sub>S prevents the hydrogen recombination reaction that would normally occur, forcing the hydrogen atoms into the metal structure, leading to corrosion and weakness [2-3].

Sulfur stress cracking in wet H<sub>2</sub>S is extremely dangerous and it is a particularly important issue in petroleum industry because petroleum products like natural gas and crude oil often contain a considerable amount of H<sub>2</sub>S. The most common forms of wet H<sub>2</sub>S cracking are hydrogen induced cracking (HIC), stress-oriented hydrogen induced cracking (SOHIC), and Sulfide Stress Cracking (SSC) [4].

Nowadays in Vietnam, it appears more and more oil and gas reservoir with a high reserve but CO<sub>2</sub> and H<sub>2</sub>S content are high. So, the corrosion study in CO<sub>2</sub> and H<sub>2</sub>S is a significant issue to understanding the corrosion mechanism and specially cracking corrosion with a high H<sub>2</sub>S content.

## **2. EXPERIMENTAL CONDITIONS**

To conduct the evaluation of the susceptibility for corrosion and cracking of a steel casing pipe in a downhole environment, the specimens were prepared from the cutout casing pipe made from API 5CT L80 Type 1. The samples were grinded to 600 grit finish before testing.

To perform the SCC and SSC tests in brine solutions saturated with H<sub>2</sub>S and CO<sub>2</sub>, samples were loaded using isolating pins/washers to isolate the loading hardware from the test specimens. Suitable corrosion resistant allow, hardware was used to stress the test samples. Samples were loaded to the desired stress level by affixing a strain gauge to the apex of the sample and stressing the sample to the strain at which the stress level was achieved on a stress-strain curve for the material performed in order to obtain this information. No compensation was made for to account for loading the samples at room temperature followed by testing at elevated temperature (82 °C) as significant changes in material properties at this temperature were not expected. The stress used to load the specimen was 80 % of tensile strength of the steel.

All samples for a specified environment was exposed simultaneously in a single test vessel, maintaining a test solution to exposed coupon surface area ratio of  $30 \pm 10$  mL/cm<sup>2</sup>.

Test was run for 720 hours, as per NACE TM0177 [5] in autoclave. The condition of the test for SCC and SSC is as below: The sulfur cracking corrosion test was carried out in of 5 wt% NaCl, 0.4 wt% Sodium Acetate, 0.23 wt% Acetic Acid, at 82 °C for SCC and 24 °C for SSC and at total pressure of 141 bara with H<sub>2</sub>S 12.3 psia and CO<sub>2</sub> 9.4 psia. Duration of test is 30 days.

After test, samples were analyzed for the presence of pitting and/or cracking using the stereomicroscope at 10x magnification by Carl Zeiss optical microscopy. Samples were cleaned prior to inspection using hot, soapy water and chemical cleaning.

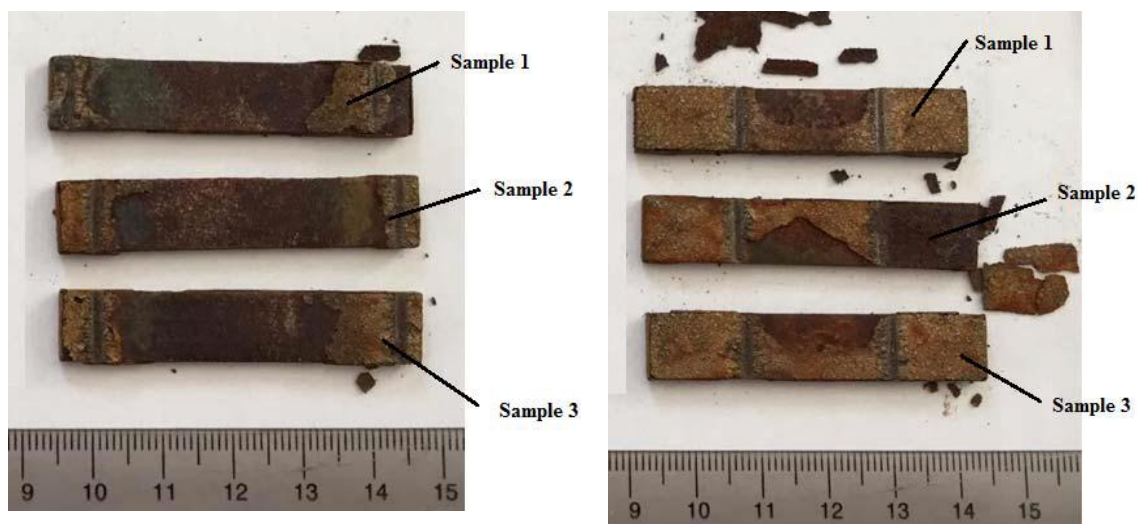
To perform HIC test, samples were sectioned from a casing pipe of size indicated in NACE TM0284 [6], then ground finished with 320 grit paper, degreased, rinsed and stored in

desiccator within 24 hours before test. The samples were loaded in the autoclave and then immersed in a well-prepared solution saturated with H<sub>2</sub>S. The duration of test is 96 hours after finishing 1 hour of H<sub>2</sub>S saturation. After testing, test specimens were sectioned following NACE TM0284, then polished metallographically so that cracks could be distinguished from small inclusions or other discontinuities. Cracks would be measured crack length and thickness under magnification up to 100x scale by Carl Zeiss optical microscopy.

### 3. RESULTS AND DISCUSSION

#### 3.1. Stress Corrosion Cracking Test (SCC)

Figure 1 presents the morphology of the samples after SCC testing in autoclave at 82°C for 30 days. Corrosion products and decreased dimension of samples can be visibly observed showing that corrosion occurs.



*Figure 1.* The front (left) and the back (right) of the samples after SCC testing with corrosion products and decreased dimension.

In order to determine crack corrosion, the samples can be observed also on the cross sections of sample by optical microscopy. Figure 2 shows four photomicrographs of the cross sections of sample after testing at 82 °C for 30 days.

Microscopy of the samples days developed pitting corrosion in the surface and cracking starting in the surface of the samples (observed at 10x and 63x magnification). Large cracks, mostly found in the middle of the samples where the maximum bending occurred, were as deep as 0.5 mm in the side of the sample representing the outside diameter (OD) of the tube.

The top images and bottom left shows the middle of the sample whereas the right-side image shows one of the sides of the sample. Note that the bottom of the sample resembles the side of the test sample subjected to the compression stresses resembling the internal diameter (ID). General corrosion was also observed in the samples, with detached corrosion products from the surfaces of the samples and significant decreased in the dimensions of the samples after testing (due to general corrosion). In this case, the corrosion products are easily detached in most of the samples. However, in some areas where the corrosion products adhere to the

surface in both sides of the samples (OD and ID), there was a significant difference in the corrosion layer. The corrosion layers measured in several locations of the internal diameter (ID) of samples 1 varied from 335  $\mu\text{m}$  to 399  $\mu\text{m}$ , similarly, the corrosion layers measured in several locations of the OD sample 1 varied from 147  $\mu\text{m}$  to 188  $\mu\text{m}$ . In most of the areas of both samples 1 and 2, the total layer of corrosion products (adding both sides of the sample) varied from 272  $\mu\text{m}$  to 671  $\mu\text{m}$ . Based on results for sample 1, it is very likely that while the outside diameter (OD) (the area of the sample with higher stresses) was more susceptible to cracking. In contrast, the internal diameter (ID) was more susceptible to general corrosion.

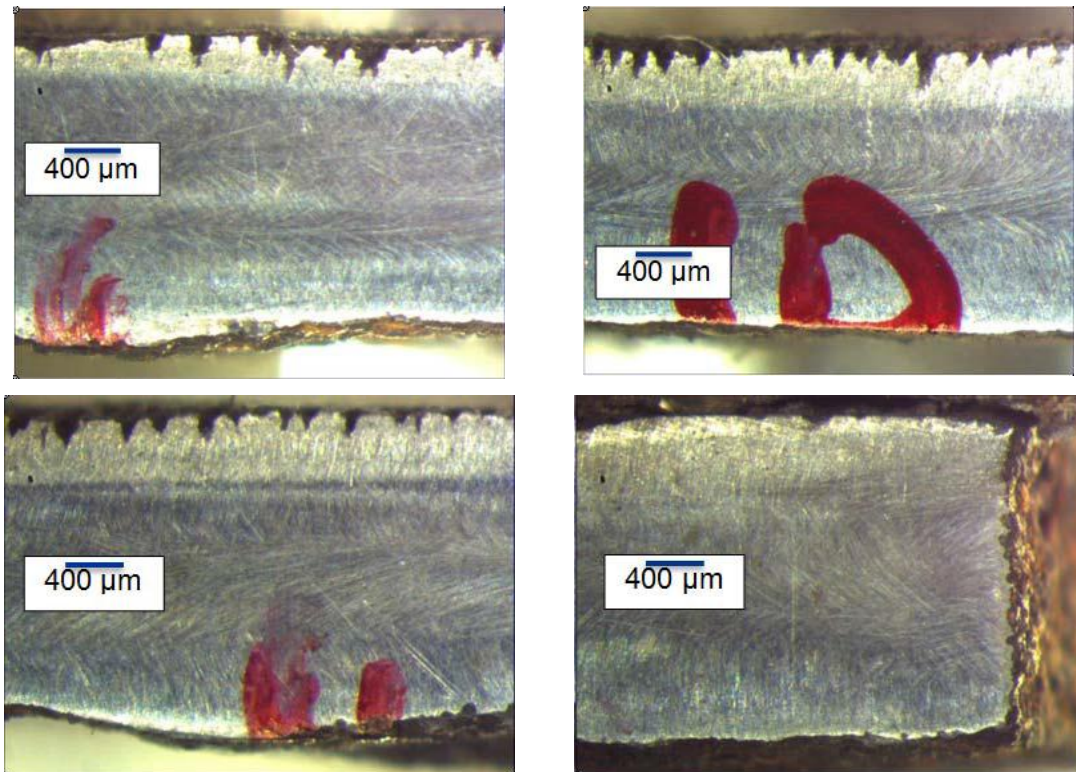


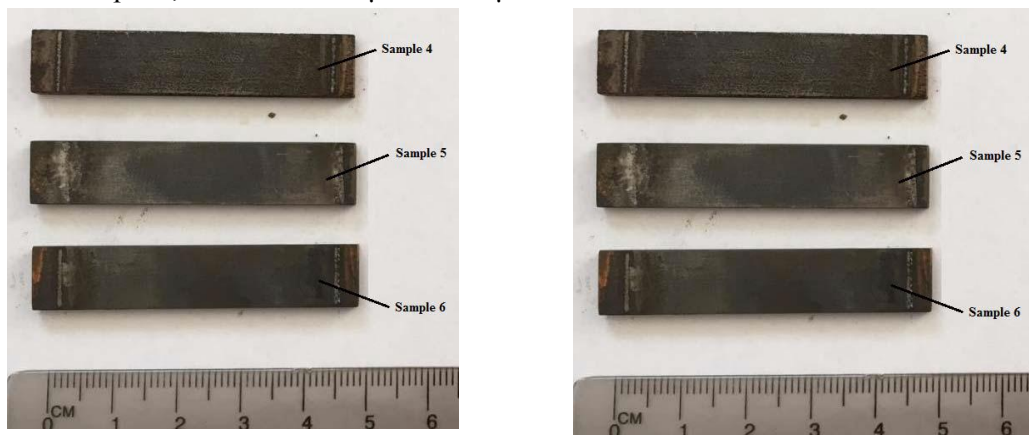
Figure 2. Photomicrographs of sample 1 tested at 82 °C for 30 days.

### 3.2. Sulfide Stress Corrosion Cracking Test (SSC)

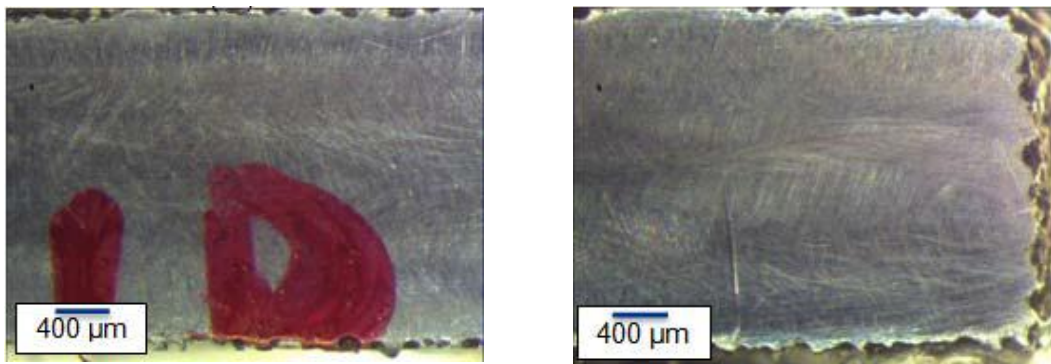
Condition SSC test is similar to SCC test, except the testing temperature of 24°C. Fig. 3 shows the front and the back of the samples after SSC testing.

Figure 4 shows photomicrographs of the cross sections of sample after testing at 24 °C for 30 days. The left side image shows the middle of the sample whereas the right-side image shows one of the sides of the sample. Note that the bottom of the sample resembles the side of the test sample subjected to the compression stresses resembling the internal diameter (ID). In general, the microscopy of sample (tested at 24 °C for 30 days) revealed that the samples developed pitting corrosion in the surface. However, no cracking was observed on the sample at 10x and 63x magnification. General corrosion was also observed in the samples (Fig. 5) but no significant decrease in the dimensions of the samples occurred after testing (as compared to the as received sample). The corrosion layers, measured in several locations of the sample, varied

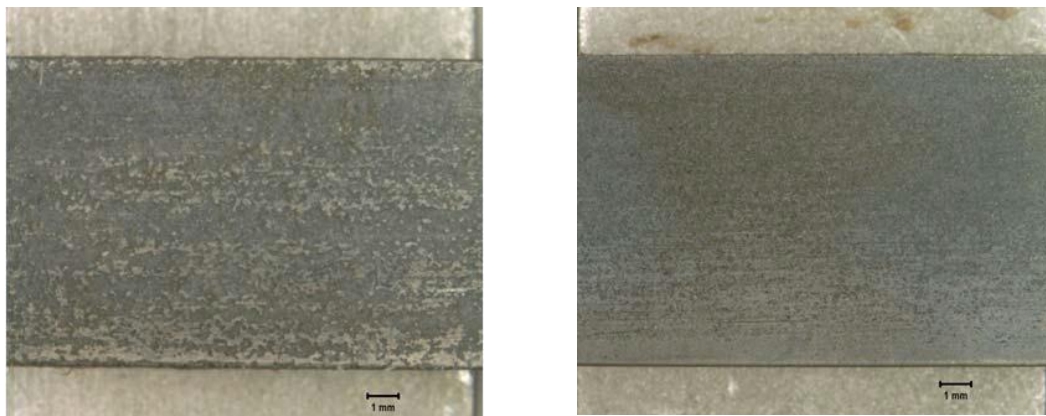
from in thickness from 71  $\mu\text{m}$  to 105  $\mu\text{m}$ . Similarly, the corrosion layers, measured in several locations of sample 5, varied from 63  $\mu\text{m}$  to 105  $\mu\text{m}$ .



*Figure 3. The front (left) and the back (right) of the samples after SSC testing.*



*Figure 4. Photomicrographs of sample 4 tested at 24 °C for 30 days.*



*Figure 5. Corroded surface of sample 4 (a) and 6 (b) after testing at 24 °C for 30 days.*

### **3.3. Hydrogen Induced Cracking Test (HIC)**

Three testing samples were cut (in the axial direction) from the 3.5-inch tube to generate samples for HIC testing following NACE TM0284 in autoclave. For each sample, the following surfaces shall pass visual test after being metallographically polished.

After testing finish, the sample have been cut to observation section surface. All the surfaces of section on testing samples show no detection of any inclusion or other discontinuities (Fig. 6).



Figure 6. Visual test of surface of sections for testing samples in HIC test.

Results of visual test under magnification up to 100x scale are shown in Fig. 7, from that can be concluded that no detection of hydrogen-induced cracks.

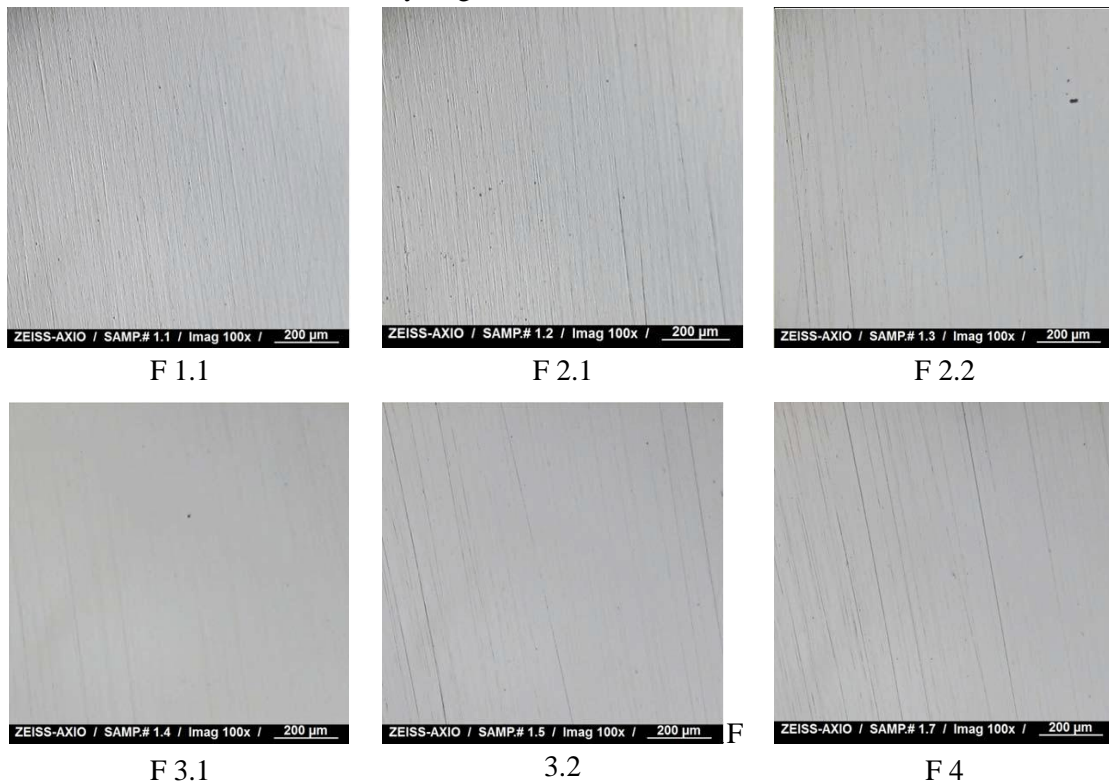


Figure 7. Microscopy under magnification up to 100x scale of sample.

#### 4. CONCLUSIONS

The samples tested under the selected downhole environment in a test solution prepared following NACE TM0177, Bent-Beam Test Method, Solution B (SCC and SSC test) showed pitting and general corrosion at both temperatures (24 °C and 82 °C). For the SCC test at 82 °C, microscopy of the samples developed pitting corrosion in the surface and cracking starting in the surface of the samples (observed at 10x and 63x magnification). The cracks, mostly found in the middle of the samples where the maximum bending occurred, were as deep as 0.5 mm in the side of the sample representing the outside diameter (OD) of the tube. General corrosion was also observed in the samples, with significant decrease in the dimensions of the samples after testing (due to general corrosion). For the SSC test at 24 °C, microscopy of the samples developed pitting corrosion in the surface. However, no cracking was observed on the sample at 10x and 63x magnification. General corrosion was also observed in the samples but no significant decrease in the dimensions of the samples occurred after testing.

The results of HIC test following NACE TM0284 shows that microscopy of the samples tested at 25 °C for 96 hours under developed no detection of hydrogen-induced cracks. All the surfaces of section on testing samples show no detection of any inclusion or other discontinuities.

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