Analysis Cracking Corrosion on Carbon Steel Pipes API 5L-X65 in Solution 7700 ml Aquades, 250 ml Acetic Acid and 50 ml Ammonia with Gas CO_2 and H_2S in Saturation Condition

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Abstract. The oil and gas industry exploration that will generally be followed by corrosive substances including sweet gas (eg H_2S and CO_2), it will result in corrosion event. The corrosion stress cracking will cause the carbon steel pipe to break so that production oil and gas can be stopped. The research aims in this paper is to analyze the corrosion event of carbon steel pipe in laboratory scale on acid environment with the existence of sweet gas H_2O and CO_2 by using three points loading method. This research uses carbon steel pipe API 5L-X65 which stay in condensation environment of 7700 ml aquades, 250 ml acetic acid and 50 ml ammonia, then filled sweet gas CO_2 and H_2S in saturated state. Based on the test results of microstructure and microscope polarized, there is a phenomenon corrosion stress cracking, i.e transgranular stress cracking corrosion and intergranular stress cracking corrosion. The accelerate corrosion that happened at the test sample will be greater if ever greater given deflection for the time of the same presentation. Crack deepness in the test of the test sample will deeper if ever greater given deflection. The cracks in the sample test will deeper if it was given stress σ greater for the same exposure time.

Keywords: stress corrosion cracking, three-point loading, sweet gas, carbon steel pipe.

1. Introduction

Along the pipeline in the oil and gas industry, the event of corrosion phenomena will occur, because of the corrosive substances flowing participate. Initial research was that the test sample was in a salt environment, and also in an acid environment in the absence of ammonia. This study is in acid environments but the presence of ammonia, because ammonia anhydrous condensate (ammonia anhydrous) can cause corrosion of tense cracks in stressed carbon steel. The goal is to analyze the stress corrosion cracking (SCC) events in various environmental conditions.

From other observations indicate that the failure is due to the influence of corrosive media. It is shown that the working voltage is still below the yield stress of the material. The greater the stress, the occurrence of Stress Corrosion Cracking (SCC) faster. The test hardness decreases with the duration of the test specimen submerged in corrosive media.[2]

The corrosion can strike on metals whether under or not. The influence of the load, especially on the metal that experienced the stress will greatly affect the corrosion rate resistance. This corrosion is generally called stress-corrosion cracking. Based on the corrosion research data that has been done on the specimens there are different corrosion rate results from each specimen. The amount of load worn on the specimen gives an effect on the corrosion rate. The test specimen with a larger load resulting in more surface defects in which many surfaces are peeling and forming a hole or a niche, the holes in the specimen cause acceleration of corrosion.[2]

The stress corrosion cracking on the carbon steel pipe in acid and sweet gas solutions.[3] Analysis of corrosion phenomenon of pipe plate carbon steel API 5L-X65 in a solution of 7900 ml of sea water and 100 ml amonial under conditions of CO_2 and H_2S gas saturated at room temperature.[4] Analysis the phenomena corrosion of carbon steel pipe API 5L-X65 with a three-point loading method in the H_2S gas environment under CO_2 saturated conditions in a solution of 7900 ml of sea water and 100 ml of ammonia.[5]

The observation results show that the failure caused by the influence of corrosive media, it is shown that the working voltage is still below the yield stress of the material. The greater the stress, the occurrence of Stress Corrosion Cracking is faster. The hardness of the test decreases with the duration of the test specimen submerged in corrosive media.[1]

Research corrosion rate on carbon steel pipe API 5L-Grade B in the environment of H_2S gas CO_2 saturated condition in NaCl solution[6], corrosion rate on carbon steel pipe API 5L-Grade B in H_2S gas environments and saturated CO_2 conditions in NaCl solution and acetic acid solution[7], study of stress corrosion cracking due to sweet gas (H_2S and CO_2) in carbon steel pipe API 5L-X65 to corrosion rate in acetic acid solution[8], Analysis corrosion rate of Carbon steel pipe API 5L-X65 API with determination three point Method on H_2S Gas Conditions Saturated conditions of CO_2 in Acetic Acid Solutions[9]. Characteristics of carbon steel pipe API 5L-X65 API with three point loading Method in H_2S Gas Conditions Saturated Conditions CO_2 in Acetic Acid Solutions[10], Analysis of the 5L-X65 API Carbon Steel Pipes with Three Point Loading Method on H_2S Gas Conditions Saturated CO_2 in Acetic Acid Solution[11]. In the oil and gas industry in case of crack corrosion cracks will result in fatal, ie pipe rupture that will result in oil and gas production will be stopped.

From the results of the research on carbon steel pipe API 5L-X60 that is $17H_1S$ will be susceptible to stress corrosion cracking. The mechanical properties of the degraded carbon steel pipe API 5L-X60 show higher resistance to corrosion cracking than in pipes carbon steel. Based on observations with fractografic bhawa carbon steel pipe due to hydrogen embrittlement process will occur stress corrosion crack, that is caused by hydrogen atoms penetrating into the pipe carbon steel[12].

The high corrosion resistance in the stainless steel carbon pipe (super martensite) is generally used in the oil and gas industry, especially in acidic environments. However there are some that are susceptible to the presence of the hydrogen and the mechanism of the corrosion processes will occur in the presence of H_2S as well as depending on the pH. The diffuses seeping H_2S along the metal structure it will form a sulfide metal pore as it reacts on the metal surface, thereby freeing the hydrogen bonded to the absorbable sulfur and the hydrogen present in the H_2S solution indirectly causing failure[13].

The results in the loss of early mechanical properties especially resistance to brittle fracture, which is based on the engineering calculations at the pipe design stage. At the same time corrosion of tense cracks has been identified as one of the dominant failures in the carbon steel pipes in the humid environments, which would lead to the breakup of high-pressure gas transmission pipes as well as serious economic losses and disasters[12].

Based on the research that has been done, it can be seen that the effect of temperature on the protective properties of poly (TMSPMA) material on the surface of carbon steel by Electrochemical Impedance Spectroscopy (EIS) method in some temperature variations, 25ºC, 45ºC, 60ºC, and 75ºC. From the results of fittings conducted on the EIS measurement results

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obtained information that there is a decrease in the impedance value is often the temperature rise which shows that the resistance of corrosion protection material in critical condition to protect carbon steel decreases.[14]

2. Experimental Section

To make a sample house (holder) of the corrosion test is required a sheet of steel in the form of plates or flat extruded section with rectangular cross section, and in addition can also be used cast iron or iron in the form of a beam as shown in figure 1. The corrosion material sample test API 5L-X65 to be tested is diluted in the form of a thin plate then bent at both ends to obtain a stress, and the strain size can be adjusted by deflection through a screw-driven thread as shown in figure (1.b) to determine the deflection. This study used a 12.5 cm long corrosion test sample, 2 cm wide and 2 mm thickness (figure 2) stored on a three-point specimen holder as illustrated in figure (1.a), figure (1.b) holder shape design [8].

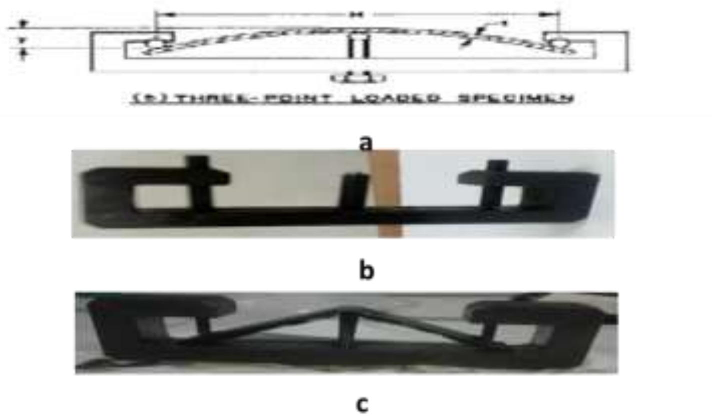


Figure 1. Holder shape and installation of test specimens.

The corrosion test sample research (as in figure 2) stored in the holder (Figure 1.c) filled with 250 ml acetic acid solution, 4700 ml aquades and 50 ml ammonia as shown in figure 3. The population of the corrosion test samples of each time variation consisted of two test samples with 2 deflection variations, and either the weight of the test sample before the corrosion test and the sample weight after corrosion testing were weighed.

Figure 2. The plate samples test corrosion API 5L-X65 were initially cracked.

Figure 3. Chamber corrosion test. (a container tube white is H_2S gas and container tube black is CO_2 gas) at room temperature.

Tensile test is a mechanical stress-strain test that aims to determine the strength of the material to the tensile force. In the test that the test material is pulled to break and usually the focus is the maximum ability of the material in withstanding the tensile load, and the maximum tensile strength is generally called "Ultimate Tensile Strength (UTS)". The change in length in the curve is called the strain technique (ε), which is defined as the change in length that occurs due to static changes (ΔL) to the initial stem length (L_0). The stress generated in this process is called the stress engineering (σ), which is defined as the loading value that occurs (F) on an initial cross-section (A_0) as shown in figure 4. The stress normal due to static pressure can be determined based on the following equation:

$$\sigma = F/Ao \qquad (1)$$

with :
σ = Stress normal due to static pull load (N / mm^2), F = Tensile load (N), Ao = the initial cross-sectional area of the specimen (mm2) as shown in figure 3.

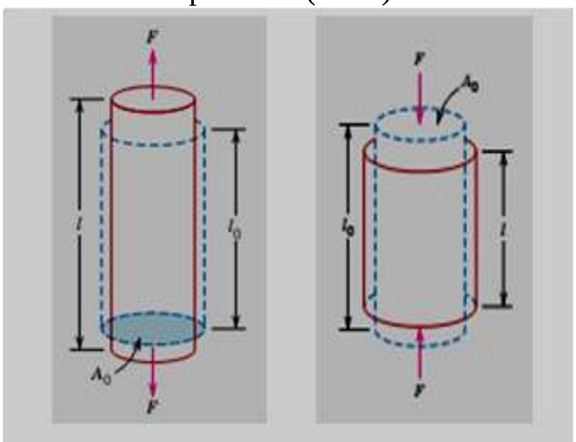


Figure 4 . The basic principle of stress.(Callister, D. William 2007).

The strain due to the static tensile load can be determined by the following equation:

$$\varepsilon = \Delta L/L \qquad (2)$$

with :
ΔL = L - L_0, **ε =** Strain due to static tensile load**,** L = Change in specimen length due to tensile load (mm), Lo = The length of the initial specimen (mm).

The flat-shaped specimens have dimensions of width, length, and thickness of specimens are usually specified, and the buffer of the test sample specimen in both ends is retained and then buckled / pressed with a screw (fitted with a sphere) where the prop of the pusher is centered as shown in figure. (1.a) and (1.b). The sample specimen dimension used can be modified according to need, but its proportion is estimated proportionally. The elastic stress calculation as in the following equation:

$$\sigma = 6\, \varepsilon\, t\, y/H^2 \qquad (3)$$

with :
σ = Maximum tensile stress (N / m2).
ε = modulus of elasticity (N / m2).
t = specimen thickness (mm)
y = maximum deflection (mm).
H = distance between external buffer (mm) and small deflection (y / H less than 0.1) as shown in figure (1.a). Flat chip specimens with 25-51 mm (1-2 inch), 127-254 mm long (5-10 inches) and thickness of material test specimens used as shown in figure 2.

The data obtained based on the weight before and after the corrosion tested from the test sample, ie to calculate the corrosion rate. Then the test sample was tested using microstructure, which is to see the structural pattern of carbon steel plates. This research needs to be developed using the ultrasonic method to determine the length of the crack, which is based on the reflection of the waves that hit the surface of the plate carbon steel.

The sample plate carbon steel API 5L-X65 API is inserted into the chamber corrosion test chamber, then filled in CO_2 gas until saturated and then loaded H_2S gas for 10 minutes. The data calculation corrosion rate of the test sample in the chamber filled with H_2S gas for 10 minutes with a variation of exposure time 336 hour, 672 hour, 1008 hour and 1344 hour with a deflection of 0.5 cm, 1.0 cm 1.5 cm as in table 1 and data calculation stress σ as in table 2. The table 3 shows the graph data of the rate of corrosion against the Crack deepness, and table 4 data graph of the depth of crack between the stress σ as in table 1.

3. Result and Discussion

3.1. Result

Table 1. Data calculation of corrosion rate to sample test.

No.	M initial (gr)	M final (gr)	PH initial	PH final	Length (cm)	Wide (cm)	Thick (cm)	Deflection (y) cm	Exposure Time (Jam)	Corrosion Rate (mmpy)
A1	53.28	50.78	4	4	13.1	2.42	2.4	0.5	336	2.615746361
B1	52.6	49.78	4	4	13.1	2.42	2.4	1	336	2.950561895
C1	53.54	50.54	4	4	13.1	2.42	2.4	1.5	336	3.138895633
A2	53.11	46.25	4	4	13.1	2.42	2.4	0.5	672	3.588804007
B2	53.05	45.35	4	4	13.1	2.42	2.4	1	672	4.028249396
C2	53.95	45.85	4	4	13.1	2.42	2.4	1.5	672	4.237509105
A3	53.74	48.78	4	4	13.1	2.42	2.4	0.5	1008	1.72988026

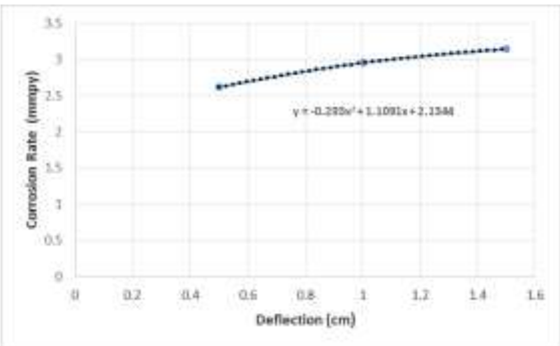
B3	53.13	47.84	4	4	13.1	2.42	2.4	1	1008	1.8449731
C3	53.19	47.74	4	4	13.1	2.42	2.4	1.5	1008	1.900775689
A4	52.32	36.15	4	5	13.1	2.42	2.4	1.5	1344	4.229661865
B4	53.25	36.99	4	5	13.1	2.42	2.4	1	1344	4.253203583
C4	52.52	35.88	4	5	13.1	2.42	2.4	0.5	1344	4.352601944

Table 2. Data stress calculation σ.

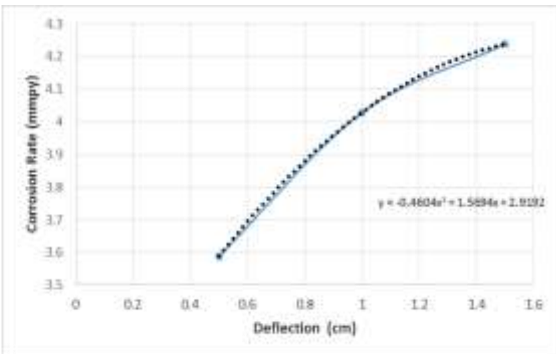
No	Modulus Elascity E (N/m^2)	t (m)	y (m)	H (m)	H^2 (m^2)	Stress σ (N/m^2)
1	2.05E+11	0.0022	0.005	0.0946	0.008949	1.51E+09
2	2.05E+11	0.0022	0.01	0.0946	0.008949	3.02E+09
3	2.05E+11	0.0022	0.015	0.0946	0.008949	4.54E+09

Table 3. Graphical data of corrosion rate on deflection.

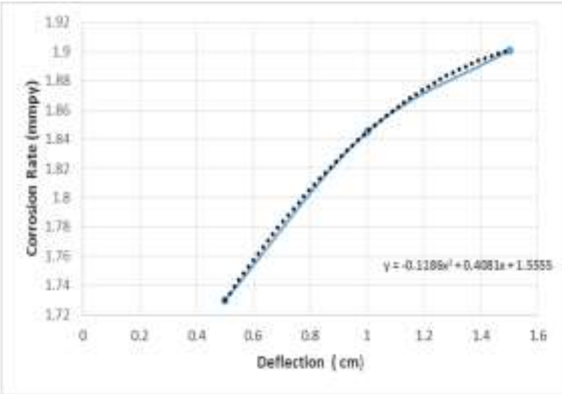
No.	Deflection (cm)	Explosure Time (jam)	PH Initial	PH Final	Corrosion Rate (mmpy)
1	0.5	336	4	4	2.615746361
2	1	336	4	4	2.950561895
3	1.5	336	4	4	3.138895633
1	0.5	672	4	4	3.588804007
2	1	672	4	4	4.028249396
3	1.5	672	4	4	4.237509105
1	0.5	1008	4	4	1.72988026
2	1	1008	4	4	1.8449731
3	1.5	1008	4	4	1.900775689
1	0.5	1344	4	5	4.229661865
2	1	1344	4	5	4.253203583
3	1.5	1344	4	5	4.352601944



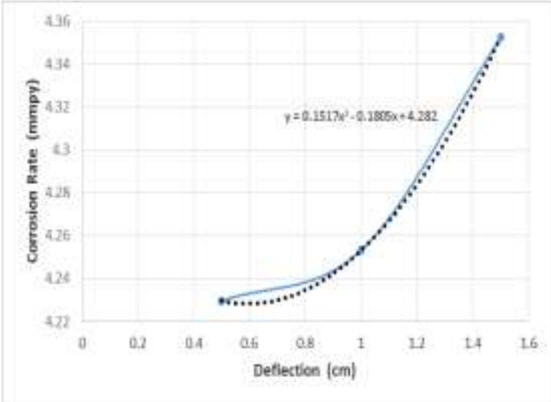
a. Explosure Time 336 hour



b. Explosure Time 672 hour



c. Explosure Time 1008 hour



d. Explosure Time 1344 hour

Figure 5 shows the graph of the corrosion rate between the deflection variations, (a) explosure time 336 hour, (b) explosure time 672 hour, (c) explosure time 1008 hour and (d) explosure time 1344 hour.

Table 4. Graphical data of stress between crack deepness

No	Stress σ (N/m^2)	Crack deepness (µm)
A1	1511873740	47.79
B1	3023747480	90.41
C1	4535621220	109.97
A2	1511873740	110.33
B2	3023747480	61.63
C2	4535621220	91.36
A3	1511873740	112.56
B3	3023747480	127.34
C3	4535621220	94.88
A4	1511873740	125.1
B4	3023747480	144.65
C4	4535621220	150.03

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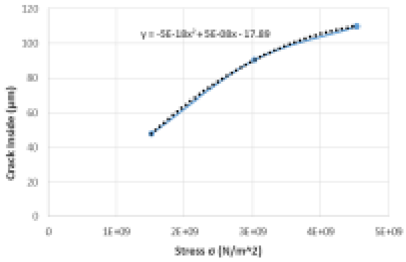
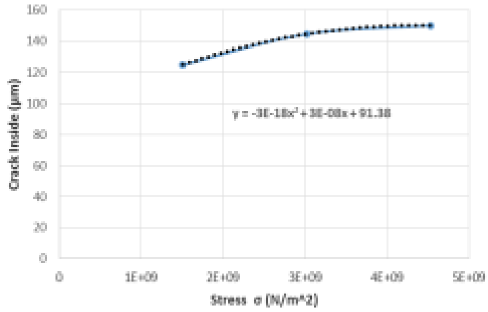
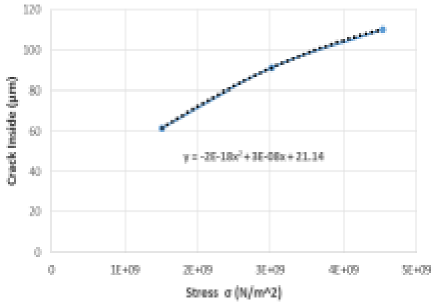


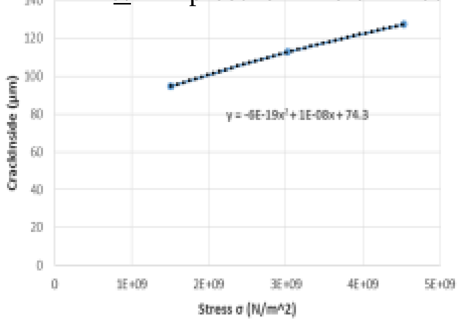
a. Explosure Time 336 hour



b. Explosure Time 672 hour



c. Explosure Time 1008 hour



d. Explosure Time 1344 hour

Figure 6 shows the graph crack deepness of stress variations (a) explosure tima 336 hour, (b) explosure time 672 hour, (c) explosure time 1008 hour and (d) explosure time 1344 hour.

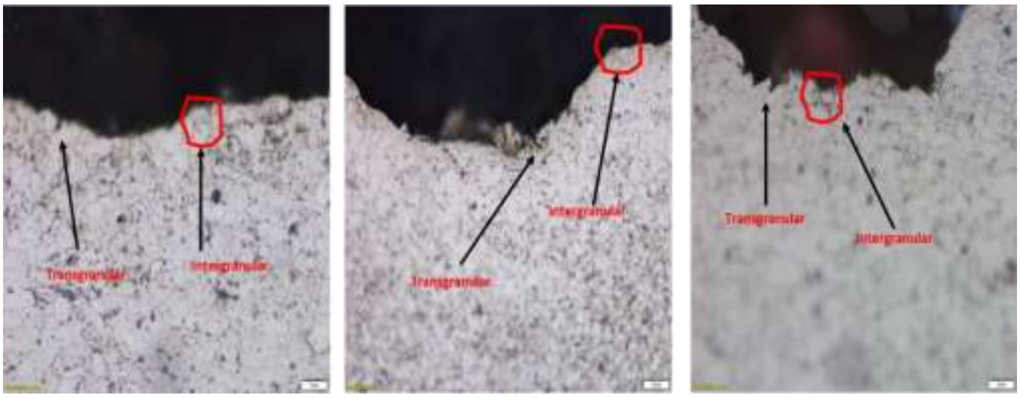


Figure 7 shows the results microstructure test in the stress corrosion cracking transgranular and intergranular

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Figure 8 shows the microstructure results in crack deepness for exposure time same. The graph of corrosion rate of between crack deepness based on table 3 is shown in figure 5. Graph of the depth of the crack between deflection y based on the table 4 is shown in figure 8.

3.2. Discussion.

Based on table 1 and table 3, the corrosion rate will increase with greater stress σ because the deflection y increases for the same exposure time as shown in figure 5. The result of graph as shown in figure 5 forms the following curve **$y = -0.293x^2 + 1.1091x + 2.1344$** for exposure time 336 hour, **$y= -0.4604x^2 + 1.5694x + 2.9192$** for exposure time 672 hour, **$y= -0.1186x^2 + 0.4081 x + 1.5555$** for exposure time 1008 hour and **$y = 0.1517x^2 - 0.1805x + 4.282$** for exposure time 1344 hour.

And based on table 4 that the depth is increasing with stress σ the greater for the same exposure time as shown in figure 6. The result of the graph as shown in figure 6 forms the following curve **$y = -5E\text{-}18x^2 + 5E\text{-}08x\text{-}17.89$** for exposure time 336 hour, **$y = -2E\text{-}18x^2 + 3E\text{-}08x + 21,14$** for exposure time 672 hour, **$y = -6E\text{-}19x^2 + 1E\text{-}08x + 74.3$** for exposure time 1008 hour and **$y = -3E\text{-}18x^2 + 3E\text{-}08x + 91.38$** for exposure time 1344 hour. Based on microstructures as shown in figure 7 there is the stress corrosion cracking transgranular and the stress corrosion cracking intergranular. And based on figure 8, the depth of crack will deepen for the same time with the deflection variation.

4. Conclusion

The research on the corrosion test of the plate carbon steel API 5LX65 lies in the environment of 7700 ml aquades solution, 250 ml acetic acid, 50 ml ammonia filled with saturated CO_2 gas and filled H_2S gas every two days for 10 minutes indicates that corrosion phenomena occurs.

The corrosion occurs is the stress corrosion cracking transgranular and the stress corrosion crackin intergranular due to the sweet gas (CO_2 and H_2S gas) based on figure 7 and ammonia solution which is an element of ammonia anhydrous condensate (anhydrous ammonia). Crack deepness deeper with equal exposure time and variation stress σ based on figure 8.

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