Title: Low Alloy Steel Susceptibility to Stress Corrosion Cracking in Hydraulic Fracking Environment

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Definition of Terms 1.

•Stress corrosion cracking is defined as the interaction of a tensile stress and aqueous environment acting on a susceptible metallic surface to initiate and propagate cracks[1].

•Hydraulic Fracking refers to the procedure of creating fractures in rocks and rock formation by injecting fracking fluid at a high pressure in order to allow more oil and gas to flow out of the formation into the well bore[2]

Problem 2.

•Susceptibility of pipeline steel to SCC due to severe operational conditions such as high pressure and presence of aggressive ions in solution

•Difficulty in predicting the ion that causes SCC due to the wide range of chemicals used in hydraulic fracking process

Research Objective 3.

•Relate the chloride ion concentration to the susceptibility of low alloy steel AISI 4340 to localized corrosion.

•Comparative study of the effect of Na_2CO_3 and NaOH as pH adjusting agent on the susceptibility of AISI 4340 to SCC in simulated fracking solution at a pH range of 7 - 10

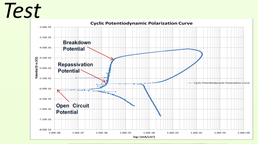
•Surface analysis of the passive film formed on unstressed and fractured AISI 4340 material in the most aggressive environment.

Research Methods 4.

ELECTROCHEMICAL MEASUREMENT

- *Cyclic Potentiodynamic Polarization (CPP) Test*

Provides information on the corrosion behavior of AISI 4340 in different solutions used as electrolyte.



- *Potentiostatic Test*

Compares the corrosion rate of material by measurement the current density at a particular potential in various electrolytes.

STRESS TEST

- *Slow Strain Rate Test*

Stress on the material provides the third factor required for SCC to occur. This test shows the response of AISI 4340 to stress I n the various test environments

- *Static Load Test*

Investigate at what stress level the material is most likely to form cracks.

POST TEST ANALYSIS

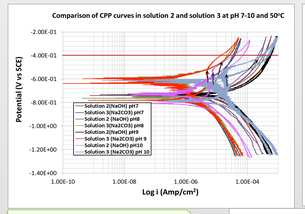
- Scanning Electron Microscope (SEM)

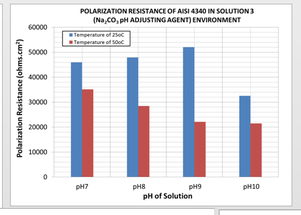
Examine the oxide film and cracked surface of AISI 4340 after test

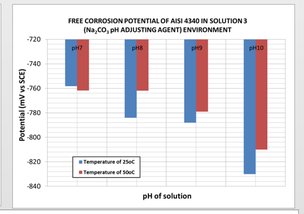
- Auger Electron Spectroscopy (AES)

Analyze area of the sample covered with oxide film and areas around the fracture by detecting all elements presents at levels > 5% except hydrogen[3].

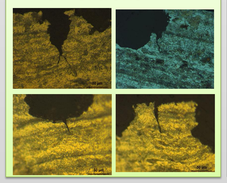
Experimental Results 5.

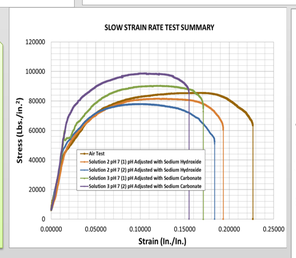


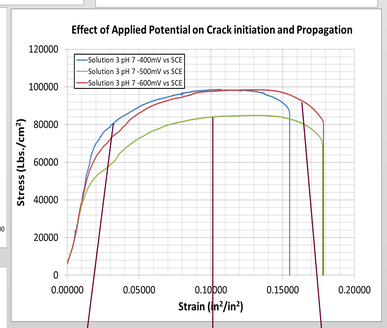


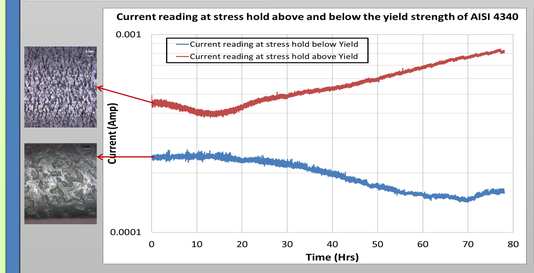


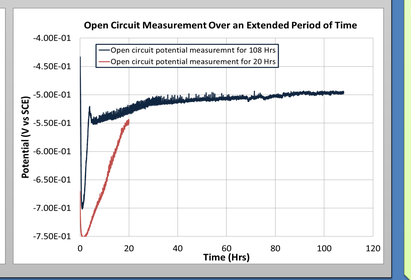
Microstructure of Cracks











Conclusion 8.

Based on the experimental results, the following conclusion have been drawn;

- Irrespective of the pH adjusting agent the oxide film formed on AISI 4340 in solution at high pH is dependent on the Cl^- concentration in solution. The substitutional adsorption of H_2O by Cl^- on the oxide film is given by[4];

$$Cl^-_{(sol)} + H_2O_{(ads)} \leftrightarrow Cl^-_{(ads)} + H_2O_{(sol)} \quad (1)$$

- The passive film formed by AISI 4340 in solutions with NaOH pH adjusting agent is less protective than that formed in solution with Na_2CO_3 pH adjusting agent.

- SCC occurs on AISI 4340 material in simulated hydraulic fracking solution with Na_2CO_3 pH adjusting agent at potential equivalent to the open circuit potential of the material in the environment.

- SCC occurs on AISI 4340 at stress level above the yield strength of the material which shows that the test material only exhibits marginal susceptibility to SCC in hydraulic fracking environment.

- SCC susceptibility of AISI 4340 is highest in simulated hydraulic fracking fluids with Na_2CO_3 pH adjusted especially at a pH of 7 due to high concentration of H atom which is produced at the cathode. The anodic, cathodic and net ionic equation for this process is as follows[5];

Anodic Reaction:

$$Fe_{(s)} + HCO_3^-{}_{(aq)} \rightarrow FeCO_3{}_{(aq)} + H^+{}_{(aq)} + 2e^- \quad (2)$$

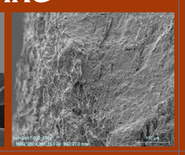
Cathodic Reaction:

$$2H^+{}_{(aq)} + 2e^- \rightarrow 2H_{(g)} \quad (3)$$

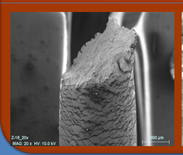
Net Ionic Equation:

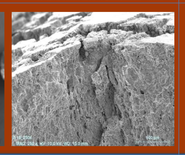
$$Fe_{(s)} + HCO_3^-{}_{(aq)} + H^+{}_{(aq)} \rightarrow FeCO_3{}_{(aq)} + 2H_{(g)} \quad (4)$$

SEM Image of Sample After Testing 6.



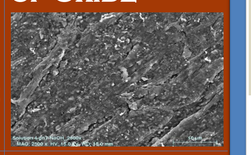
Solution with NaOH pH Adjusting Agent (pH 7)

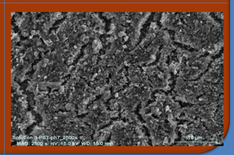




Solution with Na_2CO_3 pH Adjusting Agent (pH 7)

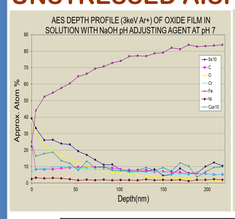
SEM Image of Oxide 6.

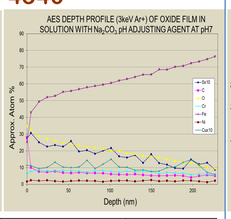




Auger Electron Spectroscopy Results 7.

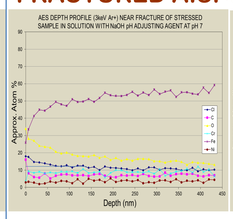
UNSTRESSED AISI 4340

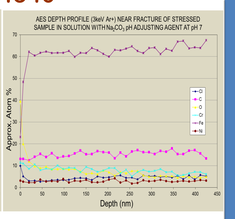




Sample	Oxide thickness (nm)	% O at 140 nm
Solution 2 (NaOH), pH 7, Test 1	30.	4.2
Solution 2(NaOH), pH 7, Test 2	70.	11.1
Solution 3(Na_2CO_3), pH 7, Test 1	140.	17.1
Solution 3(Na_2CO_3), pH 7, Test 2	140.	18.0

FRACTURED AISI 4340





Sample	% Cl at 140 nm
Solution 2 (NaOH), pH 7, Test 1	12
Solution 2(NaOH), pH 7, Test 2	7
Solution 3(Na_2CO_3), pH 7, Test 1	<3
Solution 3(Na_2CO_3), pH 7, Test 2	4

Cited Literature 9.

1. Van Boven, G., Chen, W., Rogge, R., "The role of residual stress in neutral pH stress corrosion cracking of pipeline steel part 1: pitting and cracking occurrence," *Material science*, (2006), 55 (1) , pp. 29-42.
2. DeFosse, G., McCorriston, L., "Process and process line for the preparation of hydraulic fracturing fluid" (2010) US patent number 20100132949A1.
3. Hollas, J., M., "Modern spectroscopy 4th edition," John Wiley & Sons, Ltd (2004)
4. Petek, A., Dolecek, V., "Localized dissolution kinetics of low carbon steel," *Acta Chim. Slov.* 54 (2007) 725-729
5. Alves, V., A., Brett, C., M., A., "Characteristics of passive films formed on mild steels in bicarbonate solution by EIS," *Electromica Acta* 47 (2002) 2081-2091.