Style Guidelines for Symposium Papers

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Inhibition of Carbon Steel Stress Corrosion Cracking in Fuel Grade Ethanol by Chemical Addition or Oxygen Control: A Feasibility Evaluation

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ABSTRACT

Carbon steel is susceptible to stress corrosion cracking (SCC) in fuel grade ethanol (FGE). The SCC can be mitigated by either adding chemical inhibitors or removing oxygen. The present work studied the performance of inhibitors under flowing condition that simulated pipe flow using crack growth rate tests. Oxygen scavenger performance was also evaluated by slow strain rate (SSR) tests and crack growth test. Large-scale flow loop test was also performed to evaluate the scavenger performance. The results suggest that the inhibitors that demonstrated effective in mitigating SCC in SSR also performed well under flowing condition. The feasibility of SCC mitigation using these two methods was evaluated based on the experimental results and economic considerations. Although oxygen control was demonstrated to be effective in the laboratory tests, removing oxygen from large volume of FGE may not be a viable option to mitigate SCC. Thus, inhibitor addition may be a more reasonable option to mitigate carbon SCC in FGE considering inhibitor application is a well-established operation practice in pipeline operation in combating general corrosion.

Key words: stress corrosion cracking, SCC, corrosion, biofuel, ethanol, mitigation

INTRODUCTION

Stress corrosion cracking (SCC) has been observed in carbon steel tanks and piping in contact with fuel grade ethanol (FGE) in user terminals, storage tanks, and loading/unloading racks.[1] Detailed laboratory studies,[2] sponsored by American Petroleum Institute (API),[1] Renewable Fuel Association (RFA), Pipeline Research Council International (PRCI),[2] and Pipeline and

[1] American Petroleum Institute (API), 1220 L St., N.W., Washington, DC 20005-4070.
[2] Pipeline Research Council International (PRCI), 3141 Fairview Park Drive, Suite 525, Falls Church, Virginia 22042.

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Recommended Subsequent Page Top Margin: 13 mm (1/2 in.)

EXPERIMENTAL PROCEDURE

Samples of a Ni-based (UNS N06601) industrial alloy were prepared by cutting 15×8 mm^2 coupons from a 0.5 mm thick sheet, and subjecting these to a set sequence of grinding and polishing steps. The alloy samples were first ground up to P2400 grid (10 µm) in SiC papers and finally polished with 1 µm diamond dust to give a mirror finish, and then ultrasonically cleaned in hexane (99%) before the experiment. Oxidation and carbon monoxide (CO) exposure tests were conducted in a laboratory experimental setup with a vertical steel tube enclosed in a furnace. The alloy samples were hung inside the steel tube with the internal wall plated with gold in order to mitigate the effect of metal dusting on the reactor wall.

After raising the temperature by 10 °C/min in pure oxygen (100% O_2) or diluted oxygen (0.5% O_2 in Ar) the alloy coupons were dwelled for 6 h at either 540 °C, 760 °C, or 980 °C followed by purging and cooling under Ar until room temperature. The resulting oxidized samples were either unloaded for characterization or again raised to 550 °C in Ar atmosphere and kept for 20 min to stabilize the temperature. The carbon formation was thereafter investigated under a high carbon activity (a_C>>1) gas mixture: 20 h at 550 °C in 10% CO in Ar. After exposure, the samples were cooled in Ar and unloaded at ambient conditions. The total gas flow rate was 100 Nml/min and the total pressure was 1 atm (1.01×10^5 Pa) in all experiments.

The resulting oxide layers and carbon deposits were investigated by means of optical microscopy, scanning electron microscopy (SEM) and depth profile analysis by Auger electron spectroscopy under Ar ion sputtering. Cross-sections of selected samples were also prepared and subjected to combined SEM and energy-dispersive X-ray spectrometry (EDS). The mass change by carbon build-up during CO exposure experiments were studied by means of thermo-gravimetric analysis (TGA) in a conventional microbalance setup using 10% CO in N_2 gas mixture and otherwise similar conditions.

Finally, the bulk composition of the fresh alloy sample was checked by electron probe micro analysis (EPMA) via wavelength-dispersive X-ray spectroscopy (Table 1), and found to be in agreement with the specifications of this industrial alloy.[22,23]

Table 1
Bulk Composition of the As-Received Alloy

Composition	Elements present (%)						
	Ni	Cr	Fe	Al	Mn	O	Ti
Average mass%	60.65	22.71	13.38	1.28	0.60	0.14	0.31
Average atomic%	57.65	24.31	13.33	2.64	0.60	0.24	0.36

RESULTS AND DISCUSSION

Optical micrographs of pre-oxidized alloy samples after CO exposure are shown in Figure 1. These samples were all polished before pre-oxidation as described earlier. Optical imaging of polished samples before oxidation and CO exposure could not be obtained due to their mirror-like finish, so an image of an as-received, unexposed alloy specimen (Figure 1[a]) was included for comparison. All the CO-exposed samples manifest presence of solid carbon on the surface, although less apparent from optical micrographs in the case of samples that underwent pre-oxidation at the highest temperature (Figure 1 [d] and [g]).

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******* Definition of a Trade Name *******

The name given by a manufacturer or merchant to a product, process, or service to distinguish it as made or sold by the concern which may or may not be used or protected as a trademark. Trade name also refers to any name under which the concern does business (e.g., company name, association, organization, etc.)." This definition includes company names in addition to product, process or software names, URL (Web) addresses, and does not exclude names that are not necessarily copyrighted or have a trademark.

Materials Investigated

Tests were conducted with two types of steel each in its own type of coupon. Cylindrical coupons made from UNS G10180 mild steel were used in the initial testing. The trends observed from the experiments with the cylinder or rod coupons were confirmed by conducting a separate series of tests with UNS G10500 steel in the form of flat coupons.

UNS G10180 Steel Coupons: 75 mm (3 in) long, 6.35 mm (0.25 in) diameter, threaded rod coupons of UNS G10180 steel were furnished with glass bead blasted finish. The rods had an effective surface area of 1580.6 mm² (2.45 in²).

UNS G10500 Steel Coupons: UNS G10500 steel flat coupons were furnished as 75 mm (3 in) long, 12 mm (0.50 in) wide rectangular plates that were 1.6 mm (0.0625 in) thick. With correction for the 6.35 mm (0.25 in) mounting hole and the rounded corners, the flat coupons had a surface area of 2,154 mm² (3.34 in²).

Characterization of Soils to be Investigated

SEM and EDX Analysis.

Specimens were examined using an FEI Nova NanoSEM 630† field emission SEM. This device has low-vacuum capabilities making it ideal for examining nonconductive materials such as soils without special sample preparation or metallic coating. Imaging was performed at an accelerating voltage of 18 kV using a backscattered electron detector.

XRD Analysis.

A PANalytical X'Pert Pro X-Ray Diffractometer† (XRD) equipped with a cobalt tube provided phase characterization of the material by examining the sample in reflection sample mode. Each sample was ground in a porcelain mortar and pestle until the sample passed through the number 325 sieve (0.044 mm). Analysis was performed on a reverse-pack powder sample.

Determination of Soil pH.

Soil pH was determined using a 1:1 soil suspension in distilled water. The pH determination followed the colorimetric strip technique discussed and validated for field agricultural use.[15]

Soil Treatments

Two soils, the Vicksburg Loess and the Keamuku Andisol, were used in the corrosion testing. The coupons were placed in the soil-filled test containers, and 50 ml of each of the to each of the containers. The weight of soil required to produce the volu corrosion surface of the coupon varied with the density of the soil. Each te Loess contained approximately 200 g of soil. Each test container of the Ke approximately 115 g of soil. Where sufficient soil was available, three identica

† Trade name.

[1] Unified Numbering System for Metals and Alloys (UNS). UNS numbers are listed in Metals & Alloys in the Unified Numbering System, 10th ed. (Warrendale, PA: SAE International and West Conshohocken, PA: ASTM International, 2004).

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The damage state parameter was calculated for each of the three sensor nodes included in the 172 data sets according to Equation 4. An average damage state parameter was then calculated by averaging the three sensor node damage state values. The average damage state parameter was correlated to the coating defect area (R^2 = 0.87) (Figure 23). The data was best fit using a three parameter sigmoidal function, although a logarithmic fit is given in the figure. This empirical fit, using readily measured impedance data, is another approach to in-situ coating characterization that can be used to more simply assess coating damage.

$$\eta_f = \frac{Log(Z_s) - Log(Z_*)}{Log(Z_s) - Log(Z_f)} \quad (4)$$

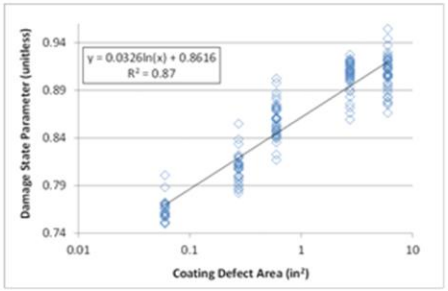

Figure 23: Plot of average damage state parameter relative to the coating defect area for 172 data sets. Logarithmic fit is given; however, a sigmoidal function is more appropriate and statistically significant fit.

CONCLUSIONS

It has been demonstrated that two electrode impedance measurement techniques using simple sensing electrodes can be used to predict coating defect size and relative location. The sensor measurements can be used in combination with artificial neural network algorithms to achieve an automated coating damage prediction. Other methods for accommodating changing tank conditions using data normalization and regression modeling with dimensionless damage state parameters are strategies that may also support coating condition assessment.

Future Work

Although voltage was demonstrated to be dependent on coating defect area, the initial ANN work has focus on using electrochemical impedance over a range of frequencies to characterize the coating condition. It is expected that these and other inputs such as phase, solution conductivity, and temperature may all be useful in determining coating condition and level of cathodic protection.

ACKNOWLEDGEMENTS

This material is based upon work supported by the Naval Sea Systems Command (NAVSEA) under Contract No N00167-11-P-0430. Any opinions, findings and conclusions or recommendations expressed in this material are those of the author(s) and do not necessarily reflect the views of the Naval Sea Systems Command.

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1. NACE SP0390 (formerly RP0390) (latest revision), "Maintenance and Rehabilitation Considerations for Corrosion Control of Atmospherically Exposed Existing Steel-Reinforced Concrete Structures" (Houston, TX: NACE).

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15. NACE SP0408 (latest revision), "Cathodic Protection of Reinforcing Steel in Buried or Submerged Structures" (Houston, TX: NACE).

16. NACE/ASTM G193-11a (latest version), "Standard Terminology and Acronyms Relating to Corrosion" (Houston, TX: NACE).

[1] American Concrete Institute (ACI), 38800 Country Club Dr., Farmington Hills, MI 48331.
[2] ASTM International (ASTM), 100 Barr Harbor Dr., West Conshohocken, PA 19428-2959

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