

CAAP Quarterly Report

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Prepared for: <Government Agency: U. S. DOT PHMSA >

Project Title: < *Mitigating Pipeline Corrosion Using A Smart Thermal Spraying Coating System* >

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For quarterly period ending: <April 15, 2016>

Business and Activity Section

(a) Generated Commitments

No changes to the existing agreement.

No equipment purchased over this reporting period.

No supplies purchased in this quarter.

(b) Status Update of Past Quarter Activities

Four major studies had been carried out during this quarter.: 1) Important scientific parameters for corrosion mitigation, 2) Material selection of appropriate coating for pipeline corrosion mitigation (Task 2 Subtask 2.2), 3) Fundamental study and numerical analysis for quantitative corrosion assessment using the embedded fiber optic sensors, and 4) Experimental data analysis for quantitative corrosion assessment (Task 3 Subtask 3.1). One journal paper has been accepted by International Journal of Ceramics in December 2015 and scheduled for publication in 2016, one conference proceeding was published in 2016 SPIE NDE/Smart Structures conference on March 2016. Further efforts will be put on material optimization and thickness design for the coating and extending the theoretical and experimental study on quantitative corrosion assessment. The detail progresses, which were completed in this quarter, are presented below:

1) Important scientific parameters for corrosion mitigation)

This report pays special attention to the corrosion mechanism in buried pipelines in order to propose the optimum material that can be applied as a protective coating layer on the outer surface of steel pipes. A comprehensive literature review was required to address the existing challenges in controlling of corrosion mechanisms in buried pipelines. The corrosion rate and cost of the materials and coating process were also considered in the materials selection process.

A. Mechanism of corrosion on buried pipeline

Several types of corrosion can occur in buried steel pipelines including bimetallic corrosion, general corrosion, pitting corrosion, hydrogen embrittlement, and stress corrosion cracking (SCC). The most common corrosion mechanism in buried pipelines is galvanic corrosion (bimetallic corrosion) which requires special attention. Bimetallic corrosion occurs when two metals, with different potentials are in

electrical contact while immersed in an electrically conducting corrosive environment [1, 2]. The basic requirements to initiate bimetallic corrosion are an electrolyte bridging the two metals, electrical connection between the two metals, a sufficient difference in potential between the two metals to provide galvanic current, and a sustained cathodic reaction. In the case bimetallic corrosion, the potential difference is created by the presence of dissimilar metals. Due to the dissimilar natural potentials, a current will flow from the anode (more electronegative) metal to the cathode (more electropositive) which will increase the corrosion on the anode. Since the materials of coating and the pipe could have dissimilar electronegativity, likelihood of the bimetallic corrosion should be considered during materials selection process. The schematic of the bimetallic corrosion is shown in figure 1 [3].

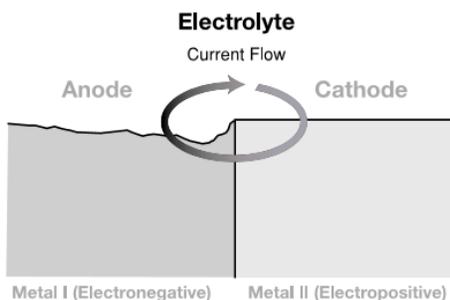


Figure.1. Bimetallic corrosion [3].

In general, the formation and growth of corrosion cells are similar to uncoupled metals, but the rate of attack can drastically increase. An appropriate materials selection can play an important role in maximize the service life of pipelines. In the case of using a coupled material (bimetallic) system, combination of some metals with different electronegativity can alter the total electrical balance of the system resulting in induce of corrosion that would not have occurred in the uncoupled state (e.g. pitting). List of the galvanic series is shown in table 1. Although the ranking in table 1 was derived for seawater, it is very similar for many other near neutral aerated aqueous solutions.

Table. 1. List of galvanic series [1].

Electro Positive	Graphite
	Platinum
	Gold
	High Alloy Stainless Steels (Super Austenitic) (Super Duplex)
	Titanium
	Nickel Chrome (625; C-276)
	Molybdenum Alloys
	Low alloy stainless steels (eg 316) (PASSIVE)
	Alloy 400/Alloy K-500
	Silver
	Nickel Aluminium Bronze
	Copper nickel (70/30; 90/10)
	Gunmetals/Tin Bronzes
	Brasses
	Tin
	Lead
	Austenitic Cast Iron
	Low alloy stainless steels (eg 316) (ACTIVE)
	Cast Iron
	Carbon Steel
Aluminium alloys	
Zinc	
Magnesium	
Electro Negative	

As shown in the table, some materials like Stainless steel (e.g. 316) have a thin protective layer (passive state) which maintains while the corrosion rate is very low. Figure 2 shows two different types of coatings that can be selected for steel pipes for providing anodic and cathodic protections [4]. In the first case no discontinuity in the coating can be tolerated, while it has no importance with cathodic coating, as

illustrated in Figure 2. The coating material and its microstructure play an important role in this type of corrosion. Anodic coatings such as austenitic stainless steels, aluminum bronze, nickel-base alloys, super-alloys MCrAlY materials, cermets (metal matrix re-enforced with WC), Cr₂C₃, and Ni or Co based composites are used against corrosion, often when it is associated with wear. However, such coatings, presenting no galvanic protection, will never protect the substrate if connected porosities and oxide networks exist, which is the case in most of thermal-sprayed coatings [5]. Therefore, the substrate protection requires using a protective bond coat or producing dense coatings, or sealing them.

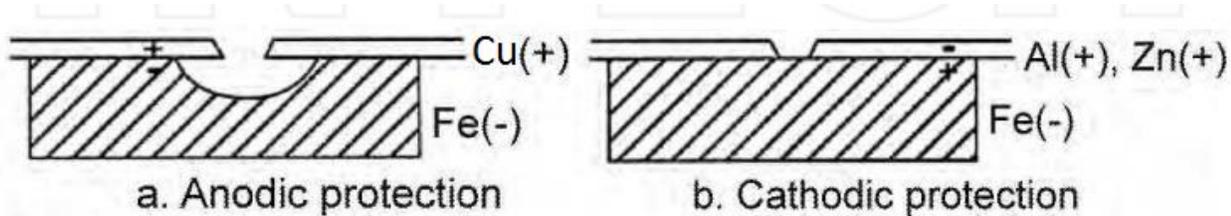


Figure 2. Examples of protective coatings; a) Anodic (no discontinuity possible in the coating), and b) Cathodic (discontinuity possible in the coating, resulting in no corrosion of iron) [4].

On the other hand, cathodic coatings acts like sacrificial anode due to their higher electronegativity respect to the steel. Hence, if the coating breaks and steel pipe be exposed to the corrosive environment, steel will act as cathode and remains un-corroded. In the case of coating steel for corrosion protection, it is recommended that the coating material with higher electronegativity state be selected. Thus, in this case coating will act as sacrificial anode and will protect the steel pipe against corrosion. As sacrificial coatings (cathodic behavior relatively to ions, for example Zn or Al on steel): the thicker they are, the longer protection they provide (typical thickness varies between 50 and 500 μm , the most frequent one being around 230 μm) [4].

A general idea of the qualitative risk of bimetallic corrosion can be gained by putting these effects together into a form as shown in Figure 3. According to Figure 3, alloys that are coupled to metals with more electropositive potentials are the ones, which suffer bimetallic corrosion. In other words, to avoid motivation of the bimetallic corrosion of a metal, the couple with higher electronegativity should be selected. As it can be seen in Figure 3, coupling the steel with Zinc, Aluminum, and Magnesium will not increase the additional corrosion in steel. Using copper might slightly increase the chance of the additional corrosion in steel as its electronegativity is slightly lower than steel. However, copper proved to be much Nobel metal with higher stability in corrosive area once it is used alone (uncoupled).

Notwithstanding, the potential difference is not the sufficient factor to predict the risk of bimetallic corrosion, and it provides no information on the kinetic of bimetallic corrosion. There are some crucial factors including area ratio, temperature, flow rate, composition of the electrolyte, etc., which define if the bimetallic corrosion is possible for any combined metallic system [6]. For instance, it has been reported that in the case of magnesium and its alloys, even though they have higher electronegativity state compared to steel, there is a possibility of a significant amount of hydrogen evolution at the cathode in neutral solutions. This resulted in hydrogen embrittlement and disbanding of the coating and failure of the pipe [7].

Metal in Contact \ Metal being considered (a)	Magnesium	Zinc	Aluminium alloys	Carbon Steel	Cast Iron	Lead	Tin	Austenitic Cast Iron	Brasses	Gunmetals / Tin Bronzes	Copper Nickel Alloys	Nickel Aluminium Bronze	Alloy 400/K-500	Low alloy stainless steel	Nickel-chrome moly alloys	Titanium	High Alloy Stainless Steel	Graphite
Magnesium	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Zinc	Dark	Dark	Light	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Aluminium alloys	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Carbon Steel	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Cast iron	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Lead	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Tin	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Austenitic Cast Iron	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Brasses	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Gunmetals / Tin Bronzes	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Copper Nickel Alloys	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Nickel Aluminium Bronze	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Alloy 400/K-500	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Low alloy stainless steel	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Nickel-chrome moly alloys	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Titanium	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
High Alloy Stainless Steel	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark
Graphite	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark	Dark

Additional corrosion unlikely

Additional corrosion may occur

Additional corrosion possible

Figure 3. Risk of additional corrosion from bimetallic contact in neutral aqueous electrolytes. The degree of corrosion refers to the metal on axis.

B. Hydrogen embrittlement

Locally excessive cathodic protection potentials known as “overprotection” is possible to occur in a pipe with a large population of external coating defects [8-12]. Overprotection in the CP system is generally contribute to two possible damages which are cathodic disbonding and hydrogen-related damages to the pipeline body due to the production of hydroxide at the cathode and the release of hydrogen. The actual generation of hydrogen gas takes place only at fairly high-negative potentials but, under rare conditions, it could literally strip off a coating in a matter of hours whilst hydrogen production may cause hydrogen embrittlement in the carbon steel [13].

The important parameter that is required to be considered in materials selection is the change of potential due to exposure to some environments, which might introduce new problems including hydrogen embrittlement [13]. The effect of PH is even far-reaching and depends upon the composition of both metals forming the couple. For instance, by using magnesium and its alloys, considerable amount of hydrogen evolution at the cathode in neutral solution might cause failure. Hence, extra care is required for selecting a suitable material to avoid severe hydrogen evolution. One of the methods of preventing SCC on pipeline structures include minimizing the operation temperature and controlling the CP levels to values more negative than -850mV CSE [1]. The other method is selecting a material with lower possibility of hydrogen embrittlement. Metallic thin films can hinder hydrogen penetration into the metallic substrate by virtue of their low solubility, diffusivity, or surface effects involving adsorption of hydrogen or combination of these mechanism [14]. Pressouyer [14] compiled permeability data for hydrogen through a number of metals and compared these value to those for hydrogen through low alloy austenitic steels. Much of these data were originally published by Tison et al. The summary of their results, in decreasing order of hydrogen permeability, are listed as follow: Nickel (Ni), Platinum (Pt), Copper (Cu), Molybdenum (Mo), Aluminum (Al), Silver (Ag), Gold (Au), and Tungsten (W). Except for

Nickel, all the mentioned metals had less hydrogen permeability compared to steel at temperature less than 300°C. According to ISO 4964 [15], the risk of hydrogen induced cracking mainly exists with a local tensile strength >1200 N/mm², a hardness >34 HRC or a surface hardness >340 HV. Notches, present in the microstructure of the surface, or material inhomogeneity increase the risk of damage.

C. Cost estimation and corrosion rate

Several factors including galvanic series, hydrogen embrittlement, corrosion rate and cost of the process were considered to propose a suitable material as coating being compatible with the pipeline and the soil environment. Table 2 shows the estimation of the material cost and the experimentally measured corrosion rates in quiet seawater. Melting point of the materials was also taken to the account to estimate the energy (cost) required for thermal spraying each of listed materials.

Table.2. Selected important properties of the Al and Zn alloy coating materials.

Material	Melting point °F	Price USD/kg [19]	Corrosion rate in quiet seawater mm/year [16-18]	Electronegativity
Al-alloy	1060-1240	1.5-1.6	<0.03	1.61
Zinc-alloy	707-917	1.7-1.9	<0.031	1.65

As seen in Table 2 both Zinc and Aluminum alloys expected to be suitable materials to be coupled with steel pipe in order to corrosion protection. Both of these metals are more electronegative with respect to the steel. This makes them as sacrificial anode to steel resulting in its protection. Considering their relative low price range, these two materials can be considered as an economical protective coating. However, the melting point range of the zinc is less than aluminum, which may result in the lower total cost of the deposition process since it may require lower energy for thermal spraying process. Zinc performs better than aluminum in alkaline conditions, while aluminum is better in acidic conditions. The aluminum-zinc coating with high zinc content and the zinc coating possessed the best anti-corrosion and anti-fouling properties [4]. According to Davis (2004) the lifetime of a 255- μ m thick zinc or zinc aluminum coating is about 25 years and it can be extended by 15 years by sealing it with vinyl paint [7]. Besides painting, impregnation with special compositions (epoxy resin, silicon resin, etc.) is also one of common sealer materials that is used currently.

2) Material selection of appropriate coating for pipeline corrosion mitigation

It can be concluded that zinc and aluminum alloys could be considered as very suitable candidates to be used as protective coating for steel pipe due to their good corrosion resistance, low possibility of inducing additional bimetallic corrosion on steel, and low hydrogen permeability. It is speculated that application of these two metals are economically justified due to relatively low cost of materials. Here are some information about the zinc and aluminum alloys.

A. Zinc-alloy:

Galvanized steel has been used for more than 100 years and it is still remains in high demand. Zinc has inherently good corrosion resistance to normally prevailing atmosphere. Addition of approximately 0.1% Al to the zinc, used for galvanizing iron and steel, greatly reduces the formation of a brittle intermediate layer of zinc-iron compound. Adding aluminum to hot-dip galvanizing has shown significant increase in ductility. This also improves the strength with reducing the grain size. Adding 0.01 to 0.06% Mg prevents intergranular corrosion in zinc-aluminum alloys. However, the optimum amount of recommended aluminum to zinc is between 3.5 to 4.3% to achieve the best impact strength and surface finish [7, 20, 21].

B. Aluminum-alloy:

Aluminum perhaps is the most important non-ferrous metal due to its good corrosion resistance along with low density, reasonable high strength, and relative low cost. Aluminum-coated steel requires a more difficult process compared to zinc, but is used in major applications including chain links, roofing panels, and automotive exhaust components. The good corrosion resistance of the aluminum is due to almost instantaneous formation of protective oxide (Al_2O_3) film when the metal is exposed to the air or any other oxidizing medium. When first formed, this film at approximately 0.25nm thickness acts as a protective barrier to oxidation. Aluminum-zinc alloy coated steel provides a combination of galvanic protection and low corrosion rate. Iron, silicon, and copper are the major alloying elements being used in aluminum alloys. However, because copper reduces the corrosion resistance of the aluminum more than the other alloying elements, it usually is added in very small amount (usually less than 0.2%) to improve the strength and fabrication characteristics without appreciably affecting the corrosion resistance. Addition of manganese to aluminum alloy may result in formation of manganese aluminide (MnAl_6) which hold iron impurity in solid solution, thus avoiding the adverse effect on corrosion resistance [7, 20, 21].

3) Fundamental study and numerical analysis for quantitative corrosion assessment using the embedded fiber optic sensors

By embedding fiber optic sensors, specifically, fiber Bragg grating (FBG) sensors, inside coatings or adhesives as shown in Figure 4(a), the sensor senses strain in the coating or adhesives induced by expansions from corrosion products as seen in Figure 4(b). Initiation and formation of pitting corrosion results in sudden increase in volume and “lift up” of both the adhesive and the FBG sensor, which may result in delamination and detachment of them from steel plate.

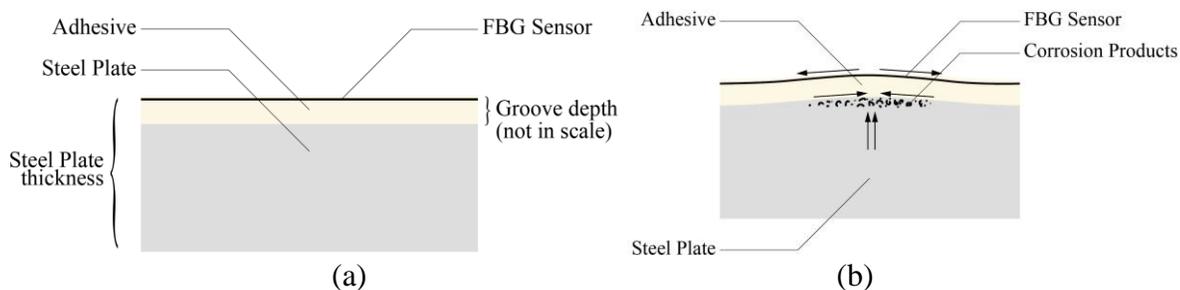


Figure 4. (a) Cross-section of the steel with sensors embedded inside coatings and (b) Lift-up effects of corrosion products.

In the case of pure pitting corrosion the coatings or adhesive act as a simple supported beam as the middle part of it detaches from steel plate while the side part is still attached to steel plate. The lift-up phenomenon can serve as a displacement acted in the middle of the beam (shown in Figure 5).

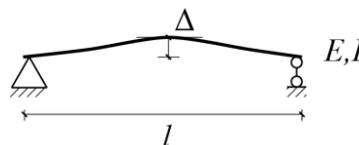


Figure 5. Simplified model of lift-up effects of corrosion products.

In steel, corrosion products have an average of 6 times in volume occupation of the original material. However, studies showed the porosity of corrosion product is around 30% ~ 50%. Thus, if the space around corrosion reaction is limited, part of the pores could be eliminated, the total increase in volume in compacted space scenarios would be less than ones in open space scenarios. The displacement (Δ) in model could be around 1% ~ 5% of total span (l).

On the other hand, uniform corrosion will have more complicated impact on the interaction between steel and coatings or adhesive, resulting in a different boundary condition for the sensor. Thus, the pattern of sensor's reading for pitting corrosion or uniform corrosion inside the coating will be different which could reveal the corrosion pattern through monitoring. Further efforts would be put into study to develop a suitable model for uniform corrosion in next quarter.

To validate the theoretical model for corrosion detection using the embedded optical fiber sensor, a numerical simulation of a simple support beam model was performed using ANSYS. A set of displacement values from 3% to 15% (increased by 3%) of total span was applied. The span of beam is 1/8 inch, as same as the length of pitting corrosion in the accelerated corrosion test. The cross section of beam is 1/16 in width by 1/8 in depth determined by cross section area of the pre-fabricated groove on the steel plate. The Young's modulus of beam is 504 psi at room temperature (80 °F), based on physical property of Durabond 954 adhesive using linear interpolation.

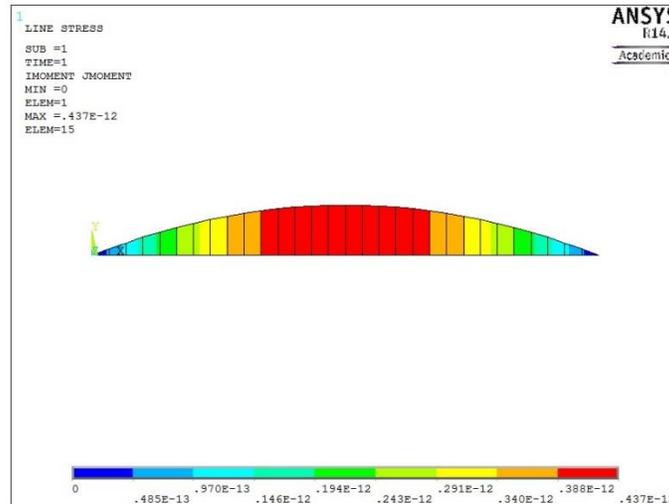


Figure 6. Simulation result of simplified model.

Table 3 shows the simulation results of the average strain in the beam which a FBG sensor monitors along the grating length direction. A linear relationship between the displacement due to increase in volume of corrosion products and strain was found.

Table.3. Average strain value result at the upper edge of beam of simulation

Displacement (of total span)	3%	6%	9%	12%	15%
Strain ($\mu\epsilon$)	18.3	36.5	54.8	73.0	91.3

4) Experimental data analysis for quantitative corrosion assessment

To better understand the corrosion monitoring system and validate the theoretical and numerical hypothesis of the corrosion assessment, two steel plate samples were prepared by embedding two FBG sensors inside Durabond 954 (from Cotronics Corp.) stainless steel based adhesive as filling material to create a space-restricting environment as shown in Figure 7.

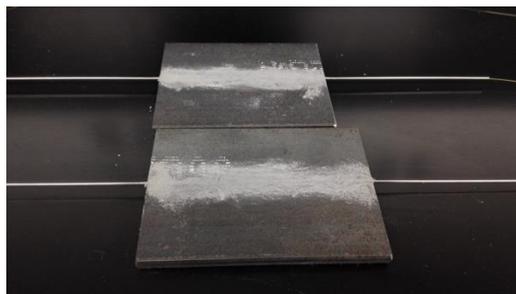


Figure 7. Steel plate sample embedded with FBG sensor.

Accelerated corrosion tests were then performed on the two samples with embedded FBG sensors by emerging them in PVC tubes filled with 3.5wt% sodium chloride (NaCl) solution for 21 days. Figure 8 shows the detail experimental setup. The center wavelength changes of the two samples with embedded sensors had been recorded continuously for the 21 days with a sampling frequency of 10Hz. Besides, a reference sensor for eliminating the temperature effect was located.

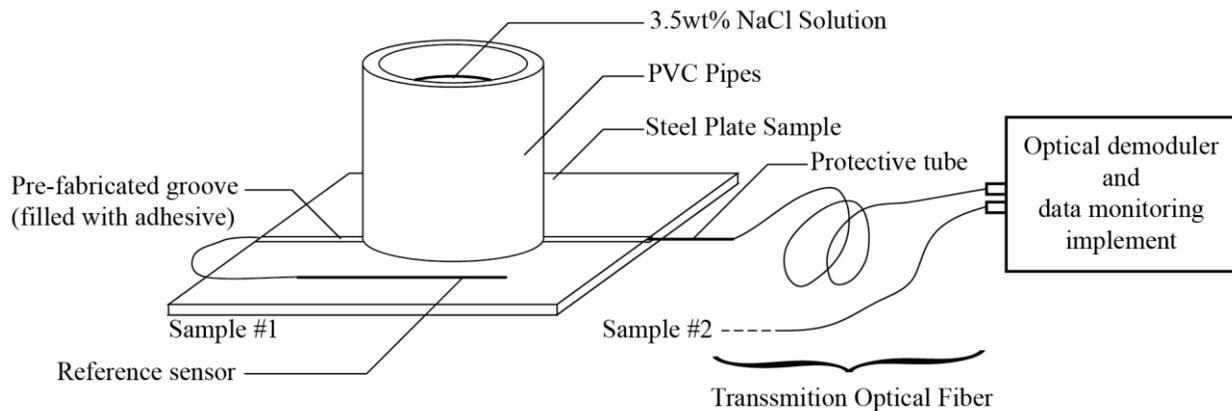


Figure 8. Accelerated corrosion test set-up.

Figures 9 and 10 show the changes in wavelength reading during the 21-day of testing, with two conditions where temperature effects are taken to the account and eliminated, respectively. From Figure 10, it is clearly seen that the two samples have shown two different corrosion signal patterns. For Sample #1, the center wavelength of the FBG sensor increased almost linearly for the first three days. The wavelength of FBG sensor on Sample #1 changed 55 pm (from -5 pm to 50 pm) within first 2 days of experiment. It may indicated a pitting corrosion due to the sudden corrosion occurrence. For Sample #2, the center wavelength of the FBG sensors did not show sudden changes but gradual reductions in wavelengths. Less than 20 pm of center wavelength change was found in Sample #2. It indicates a potential uniform corrosion pattern. Thus, the center wavelengths of the FBG sensor could qualitatively identify corrosion pattern as well as with the growth of corrosion.

Visual inspection of two samples clearly exhibited two different corrosion patterns as shown in Figure 11 and Figure 12. Only one corroded spot has been found in Sample #1, indicating a pitting corrosion pattern, whereas, in Sample #2 a uniform corrosion pattern was detected, validating the qualitative assessment of corrosion pattern from the sensor readings.

In addition, comparison of the sensor's reading for Sample #1 in Figure 10 with the numerical simulation results in Table 3 are in good agreement in the first three days of experimental result. This result could serve as a validation of proposed model. After 3 days, the center wavelength changes starts to fluctuate around at 50 pm as steel plate got further corroded, while the simulation result still showed an increase in strain value (from $54.8 \mu\epsilon$ at 9% of total span to $91.3 \mu\epsilon$ at 15% of total span). This could be caused by inaccurate boundary condition and displacement applied on the model. Further modification on model and propose suitable model for uniform corrosion is planned for the next quarter. Further study will be performed to quantitatively measure the corrosion rate of steel through analyzing the signal changes.

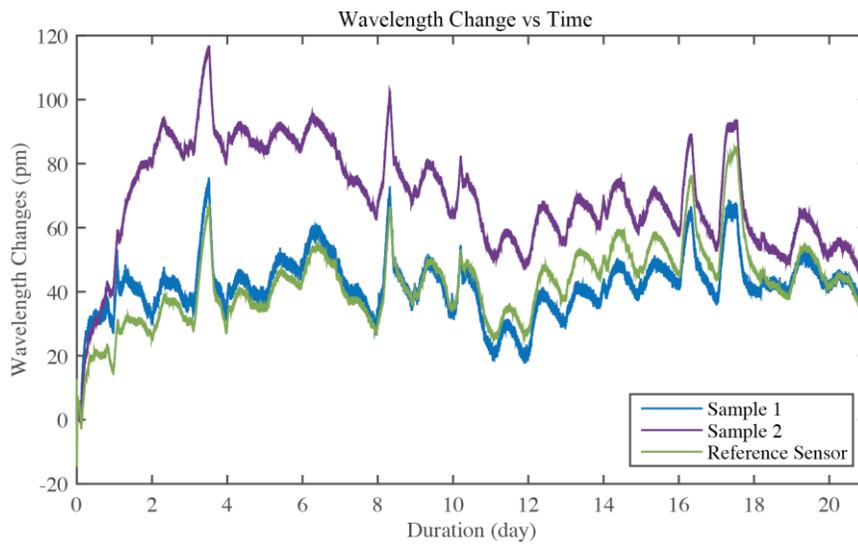


Figure 9. Wavelength change during the 21-day from accelerated corrosion test before eliminating temperature effects.

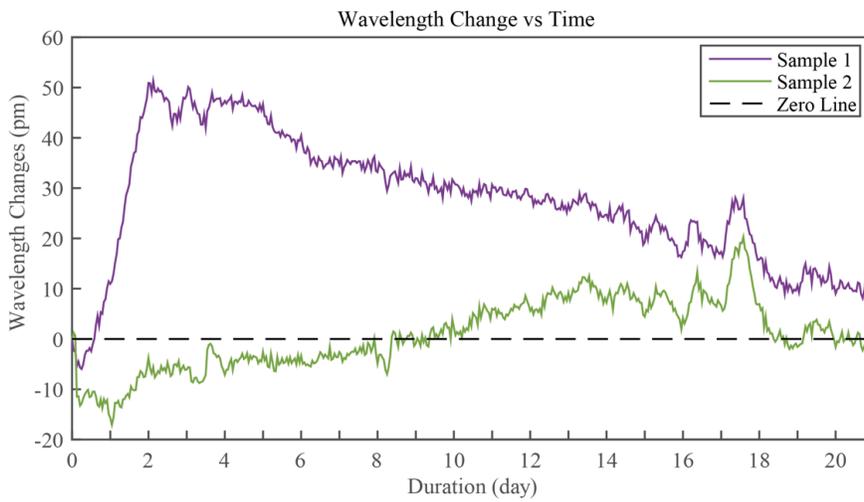


Figure 10. Wavelength change during the 21-day from accelerated corrosion test after eliminating temperature effects.

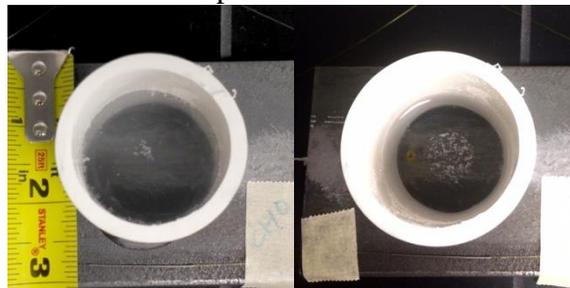


Figure 11. Visual inspection result for accelerated corrosion test of Sample 1 (Left: day 1, right: day 7).

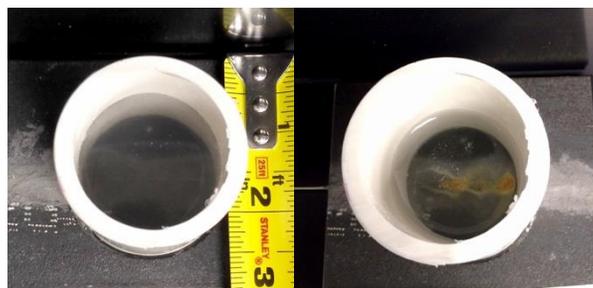


Figure 12. Visual inspection result for accelerated corrosion test of Sample 2 (Left: day 1, right: day 7).

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(c) Description of Problems/Challenges

No challenges were observed in this quarter.

(d) Planned Activities for the Next Quarter

The planned activities for next quarter are listed as below:

- 1) Material optimization and thickness design for the coating (Task 2.2 & 2.3);
- 2) Updating numerical analysis model for pitting corrosion pattern and studying on model for uniform corrosion pattern (Task 3.1);
- 3) Quantitatively measure the corrosion rate through analyzing the sensor reading (Task 3.1).