

CAAP Quarterly Report

Date of Report: <July 15, 2016>

Contract Number: <DTPH56-15-H-CAAP06>

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: <July 15, 2016>

Business and Activity Section

(a) Generated Commitments

No changes to the existing agreement.

No equipment purchased over this reporting period.

Sample supplies and a few processing tools including sand papers and a hand saw were purchased in this quarter.

(b) Status Update of Past Quarter Activities

Four major studies had been carried out during this quarter: 1) Material optimization of thermal sprayed Al- or Zn-based coating for pipeline corrosion mitigation, 2) Samples preparations for thermal spraying coating (Task 2 Subtask 2.1); 3) Theoretical analysis for quantitative corrosion assessment using the embedded fiber optic sensors, and 4) Experimental data analysis for corrosion rate coefficient (Task 3 Subtask 3.1). Future efforts will be put on characterization of the thermal sprayed coatings using optimized Al- or Zn-based alloy and test the corrosion resistance of the coatings, relate the theoretical analysis of the FBG sensors with experimental data to validate the quantitative corrosion assessment. The detail progresses, which were completed in this quarter, are presented below:

1) Material optimization of thermal sprayed Al- or Zn-based coating for pipeline corrosion mitigation

Last report had reviewed various materials using thermal spraying technique for corrosion mitigation and it was found that Al- and Zn-based coatings are expected to have good corrosion resistance for protecting steel pipelines, in this report, we performed a profound investigation on published works dealing with thermal sprayed Al- and Zn-based coatings deposited on steel substrates. Table 1 summarizes and compares the corrosion performances of thermally sprayed Al- and Zn-based coatings. Effect of addition of other supplemental elements, such as Mg, Si, and RE, on corrosion durability of these alloys in harsh environment was also considered here for an optimal corrosion protection.

Table 1 Corrosion behavior of thermal sprayed Al- and Zn-based coatings on steel at (25°C) [1-39]

Coating material	Coating technique	Corrosive solution	Observations
<i>Al-based</i>			
99% aluminum	Arc spray	3.5% NaCl	Formation of passive layer by increasing the exposure time made of Al ₂ O ₃ and Al(OH) ₃
Al- Al ₂ O ₃	Arc spray+ PEO	3.5% NaCl	Dense coating and excellent corrosion behavior
Al-5%Mg	Arc spray	3.5% NaCl	Better corrosion resistance even that Zn-Al alloys
Al-Zn-Si	HVAS	3.5% NaCl	Self-sealing nature and good cathodic protection
Al-Zn-Si-RE	HVAS and arc spray	3.5% NaCl	5 steps during the corrosion: pitting-dissolution-re-deposition, activation corrosion, cathodic protection, physical barriers formed by corrosion products, and failure of coating
<i>Zn-based</i>			
Zn-15Al	Flame spray	Marine environment, 30 days	Improved cathodic protection, but lower long-term durability compared to pure Al coating
Zn-15Al	HVAS	SRB	Corrosion of surface at first place, formation of ZnS and plugging the pores at second stage
ZnMgAl	Wire arc	5% NaCl	Formation of dense corrosion products, clogging the pores and slowing down the corrosion
Zn-Al-Mg-RE	HVAS	5% NaCl	Better corrosion behavior than Zn-Al-Mg due to the microstructural refinement of coating by adding RE
Zn-Al-Mg-RE-Si	Arc spraying	3.5% NaCl	Self-sealing behavior due to formation of Al ₃ .21Si _{0.47} and Mg ₃ Al ₂ (SiO ₄) ₃

Aluminum-based Coatings: Aluminum is one of the most commonly used metals for corrosion protection due to its anodic nature that eliminate the need for formation of flawless coating. In fact, aluminum coating acts as sacrificial protection to the steel substrate and forms passive layer made of Al₂O₃ and Al(OH)₃ which protect both coating and substrate from further corrosion [1-19]. It has been reported that arc sprayed Al coating showed better corrosion performance than flame sprayed since the aluminum oxide/hydroxide persisted for longer sputtering time on the lamella (coating layers) than that of flame sprayed [1]. Increase in the thickness of the arc sprayed Al coating deposited on steel could increase its durability in sea water [2]. Passive nature of alumina (Al₂O₃) made it one of the most interesting and common-used coating materials to protect steel from pitting corrosion and provide more uniform corrosion [3-14]. The addition of Al₂O₃, up to 30%, to Al powders helps improving the coating deposition and bonding strength between Al and Al₂O₃ [3]. Magnesium is also considered a common element to be added to Al-based alloys to enhance the long-term corrosion resistance as well as mechanical properties such as hardness [5-7]. It has been shown from previous studies that the weight loss of arc sprayed Al-Mg alloy on a mild steel in sea water, at room temperature after 90 days, was significantly less than that of pure Al coating [5]. Arc sprayed Al-5%Mg shown to successfully protect steel after 14,000 h exposure to salt water, even better than Zn-35%Sn and Zn-27%Al (by mass fraction) [6]. Zinc is another important element added to Al-based coating due to its high sacrificial nature providing cathodic protection especially to steel substrate [8]. Al-Zn alloys, even without sealing, exposed in marine and industrial environment, provide very high galvanic corrosion protection to steel substrate [9]. Al-Zn-Si-based alloys are known as self-sealing material in corrosive environment due to their capability to produce components to clog the pores and prohibit further penetration of corrosive

solution in to the coating [8-12]. Al-Zn-Si-RE (where RE is Rare Earth) coatings, among Al-Zn based alloys shown excellent corrosion resistance performance [10]. It has been reported that the corrosion mechanism of this alloy is very similar to Zn-15%Al, in which five distinct stages were observed: pitting-dissolution-re-deposition, activation corrosion, cathodic protection, physical barriers formed by corrosion products, and the failure of the coating. The RE in the coating does not affect the phase compositions of the corrosion products, but it can improve the formation of fine, continuous and uniform corrosion product layer, which acts as stable self-sealing process.

Zinc-based Coating: Thermally sprayed zinc-based alloys have also been widely used to protect steel constructions due to its high stability in the sea water and also lower electronegativity respect to steel which makes it acts as sacrificial anode [20-39]. However, if the coating is exposed to high humidity or mediums containing aggressive species such as chloride or sulfate ions, the Zn will dissolve in the solution and result in localized corrosion [20]. This problem has not been observed in Al-based coatings [20]. Twin wire arc is one of the most common thermal spray technique used to deposit Zn [20]. It has been shown that corrosion resistance of pure Al coating deposited by wire arc spray technique was higher than that of pure Zn coating [20-23]. Zn-15%Al showed the higher corrosion resistance than both Zn and Al coating since it possesses combination of two protection mechanisms: (1) creating a stable oxide, like Al coating, and (2) acting as sacrificial anode, similar to Zn coating [20-25]. Different percentages of Al content also could significantly effect on the corrosion resistance of Zn-Al-based alloys. It is reported that the porosity of arc sprayed Zn-30%Al coatings was higher than that of Zn-15%Al. Thus, 85%-15% ratio of Zn and Al showed better corrosion resistance in short-term immersion in corrosive medium such as 3.5%NaCl [23]. However, the long-term corrosion durability of pure arc sprayed aluminum coating was still higher than those of both pure Zn- and Zn-15%Al coatings deposited with the same technique [20, 23, 28, 29]. Magnesium can also be added to Zn alloy to work as the sacrificial anode. The addition of magnesium to Zn-Al-based alloy could significantly enhance the corrosion due to self-sealing nature of Zn-Al-Mg alloys [33-36]. This was attributed to formation of intermetallic phases such as $MgZn_2$ and $MgZn_{11}$ and, in some cases, $Zn_5(OH)_8C_{12}.H_2O$ [33], which plug the pores and protect the substrate against corrosive electrolyte. That makes Zn-Al-Mg coating showing even better corrosion resistance than that of pure Zn and Zn-Al coatings [33, 34]. It has been reported that the time for red rust to appear in the neutral salt spray test of Zn-Al-Mg coating is 4-20 times higher than that of pure Zn coatings [34]. Zn-Al-Mg-RE alloys are also commonly used as corrosion protection coating due to their self-sealing nature. They possessed better corrosion resistance than Zn-Al-Mg coating which is believed is due to the formation of denser, more compact, and uniform corrosion products as result of coating refinement by addition of RE elements [37-39]. Zn-Al-Mg-RE-Si coating is yet shown superior corrosion resistance than Zn-Al-Mg-RE due to presence of glass-like state phase attributed to $Mg_3Al_2(SiO_4)_3$ formed on the external surface of Zn-Al-Mg-RE-Si coating which acted as self-sealing barrier, depressed the porosity of coating, and hindered reaching the electrolyte to the substrate.

Optimized Thermal Sprayed Coating Material: Taking all the above reviews into consideration, the Zn-Al-Mg-RE or Al-Zn-Si-RE alloys can be the candidate for an optimal corrosion protection of steel, due to their cathodic protection and self-sealing nature that could significantly reduce the rate of corrosion. As could be seen in the Table 1, the most commonly used thermal spray method for deposition of Al- and Zn-based coatings is Arc spray technique. In fact, ignitable nature of Al- and Zn-based alloys at high temperature provided by thermal spray techniques such as HVOF and Plasma spraying makes using these specific techniques almost impossible for spraying these alloys. Thus, arc spray method has been selected as an alternative to be used instead of HVOF for spraying the coatings. However, the availability of raw material in the form of wire which required for wire arc spraying is questionable. The possibility of acquire the wire with closest composition to the optimal material will be examined in future.

Arc Spray Technique: Electric arc spray, which also is known as twin-wire arc, arc spray, or wire arc spray, was commercially acceptant in early 1960s [40]. Unlike the other thermal spray techniques, feedstock particles of which indirectly get heated by using heated gas jet, in electric arc spray process

direct current (DC) is used to strike between two consumable electrode wires to effect direct melting. As shown in Fig. 1, an electric arc is formed in the gap between the wire tips as the two wires are continuously fed together. It is worth mentioning that thermal efficiency of electric arc spray process is significantly higher than that of other thermal spray processes. There is a high velocity air jet located behind the intersection of the wires which shears away the molten particles and projects it toward the substrate. The velocity of in-flight particles in arc spray technique is ranged within 0.8-1.8 m/min which is much lower than HVOF (over 8 m/min) technique and consequently less harsh for the substrate [40]. Electric arc spray also transfers less heat to the substrate per unit of material sprayed compared to any other conventional thermal spray methods because there is no flame or plasma jet used in this technique. Materials to be used for arc spray should be electrical conductive in the form of wire. Using cord wires, however, expanded the range of the materials including cermets and amorphous materials. Cored wires, in fact, are made of a tubular metallic sheath with powder in the core which is usually consisted of carbide or amorphous alloys.

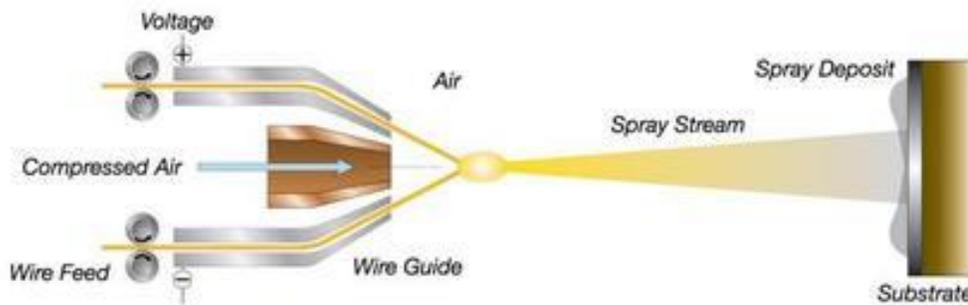


Fig. 1. Schematic of arc spray technique.

2) Samples preparations with embedded fiber optic sensors

Two sets of pipe and plate samples have been prepared as shown in Fig. 2. FBG sensors (designations and detailed information of which could be found in the previous reports) are embedded by using the adhesives Durabond 954 and M-Bond 200. In the earlier experiments, both of these adhesives were successfully used for attaching the sensors to the substrates to be coated by HVOF process. Considering lower in-flight particle speed and temperature input associated with arc spray technique, it has been speculated that these adhesives will persist on the sensors during the processes. One plate and pipe samples without embedded sensor were also prepared to be coated by the desired coating materials. These samples will be used for the mechanical, microstructural, and corrosion characterization of the arc sprayed coatings.

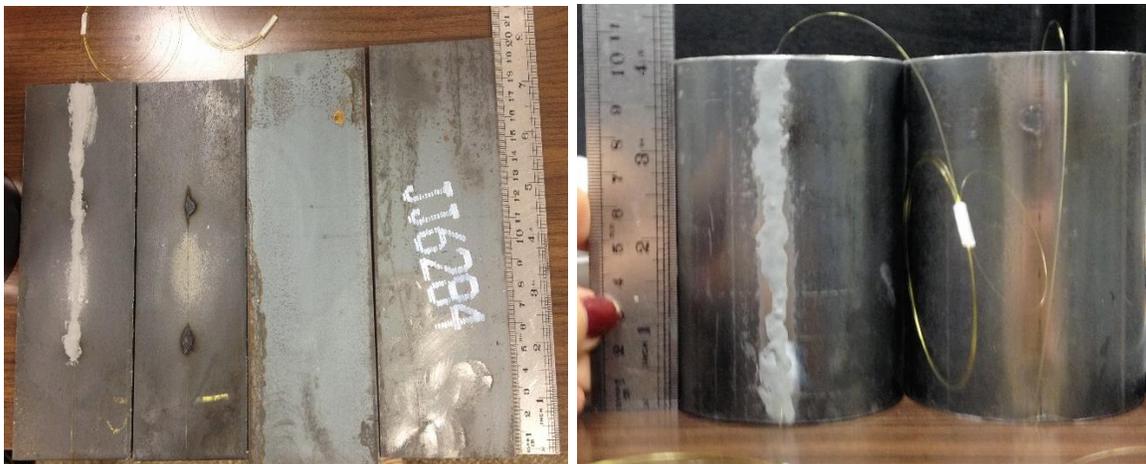


Fig. 2. Schematic of sample preparation for arc spray coating.

3) Theoretical study for quantitative corrosion assessment using the embedded fiber optic sensors

Based on the previous work reported in the last quarter, the simplified model for interaction between corrosion products and adhesive as shown in Fig. 3(a) was further studied in this quarter. Two assumptions were made based on a typical pitting corrosion observed shown in Fig. 4: 1) the corrosion productions of pit corrosion were appeared within a relatively small area comparing to the total span; 2) the corrosion productions mainly grew in vertical direction. With these two assumptions, the displacements in the middle of span caused by pit corrosion can be analyzed using simple-supported beam with a concentrated force F in the middle span as shown in Fig. 3(b).

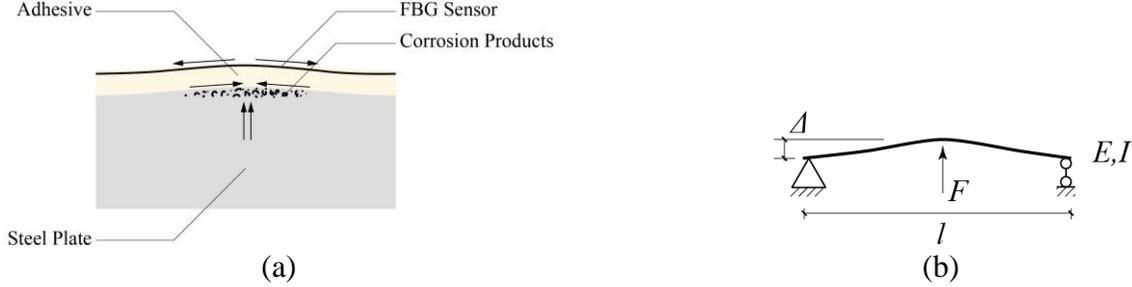


Fig. 3 (a) Simplified model of corrosion assessment system, and (b) Schematic of displacement due to pitting.

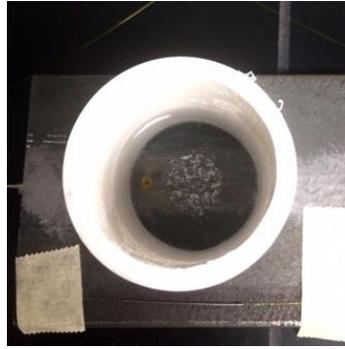


Fig. 4 A typical pitting corrosion (visual inspection of corrosion Sample #1, previously reported).

The strain on the embedded FBG sensor induced by the pit corrosion in the middle of span can then be calculated as:

$$\varepsilon = \frac{\sigma}{E} = \frac{My}{EI} = \frac{ly}{2EI} \cdot F \quad (1)$$

where E is the Young's modulus of adhesive, σ is the stress on the sensor, M is the moment induced by the centered force, I is the moment of inertia of the adhesive section, l is the span of sensor, and y is the vertical distance between the surface and the location of the sensor, which is assumed to be half of the height of cross-section in this analysis. Thus, the displacement in middle span can be calculated as:

$$\Delta = \frac{Fl^3}{48EI} = \frac{l^3}{48EI} \cdot F \quad (2)$$

Let $k_1 = ly/(2EI)$, and $k_2 = l^3/(48EI)$. Then relationship between the strains on FBG sensors and the corrosion induced displacements can be described as a linear relationship as:

$$\varepsilon = k_1 \cdot F = \frac{k_1}{k_2} \cdot \Delta = \frac{24y}{l^2} \cdot \Delta \quad (3)$$

Let $k_3 = \frac{k_1}{k_2} = 24y/(l^2)$, thus, it is clearly that the strains on the FBG sensor is linearly proportional to the corrosion induced displacements in the middle span, which is only related to the location of the embedment of the sensor and the length of the sensor. This result matches the simulation result

performed with ANSYS in the last quarter, which is shown in Table 2. In the simulated simple-supported beam system, it can be obtained in this analysis, $k_3 = k_1/k_2 = 6.1$.

Table 2. Strain value result at the upper edge of beam in simulation.

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

From geometry, it obvious that the total volume change of corrosion products (V) have a linear relationship with the corrosion induced displacement (as volume increased linearly corresponding to the increase in height), which can be described as

$$V = k_4 \cdot \Delta = l\Delta/2 \quad (4)$$

The corrosion rate (CR) is the derivative of the total mass reduction of the steel material, or the mass increase, m , of the corrosion products, which is linearly proportional to the density, D , of the corrosion products and their volume, V , change with respect to time, which can explained as below:

$$CR = \frac{dm}{dt} = D \frac{d(V)}{dt} = k_5 \frac{d(V)}{dt} \quad (5)$$

From equations (3) ~ (5), and let $K = k_4k_5/k_3 = Dl^3/24y$, the relationship between corrosion rate and strains monitored by the FBG sensor can be described as:

$$CR = K \frac{d\epsilon}{dt} = K \cdot A \cdot \frac{d\lambda}{dt} \quad (6)$$

Since $d\epsilon/dt$ is linearly proportional to the slope of the Bragg wavelength change, $d\lambda$, with an coefficient of A , and k_1 , k_2 , and k_3 will stay constant when the simple-supported beam system remains unchanged, the corrosion rate can be calculated directly from the Bragg wavelength change curve slope with properly calibrated scaling factor $K \cdot A$.

4) Experimental data analysis for quantitative corrosion assessment

Fig. 5 shows the changes in Bragg wavelength reading of Sample #1 during the 21-day of testing with temperature effects eliminated. From the figure, the Bragg wavelength change rate in first two days is (with curve fitting method):

$$\frac{d\epsilon}{dt} = 35.19 \text{ pm/day}$$

Uncoated steel generally has an average corrosion rate of 1.5 mill/year. So the scaling factor K would be

$$K = CR \cdot \left(\frac{d\epsilon}{dt}\right)^{-1} = 0.0426$$

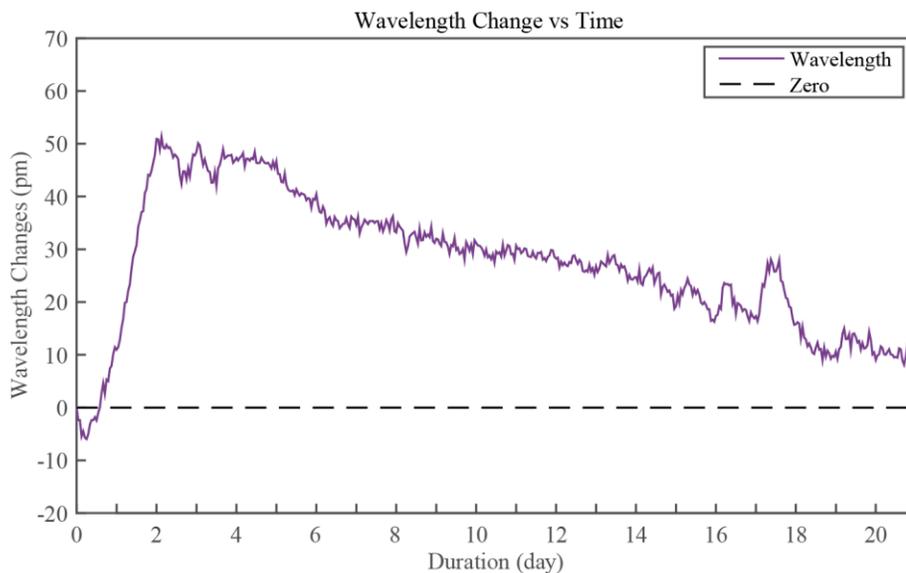


Fig. 5. Wavelength change during the 21-day from accelerated corrosion test after elimination of the temperature effects.

To further prove the calculated scaling factor K, two more samples were prepared and will be tested in the next quarter. The samples were prepared using an identical process as the two tested samples as reported in the previous quarter and shown in Fig. 6. The accelerated corrosion tests will be performed using similar testing procedure as previous one, in order to minimize environmental effects and human errors.

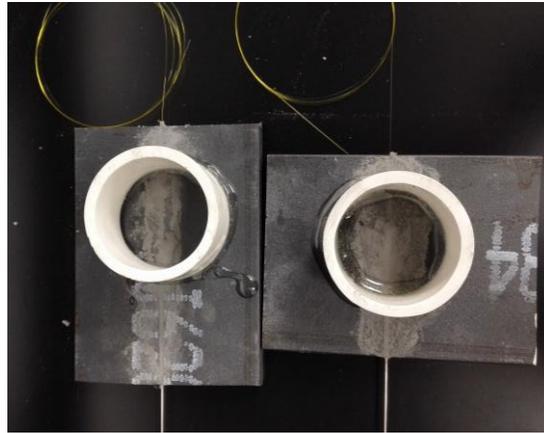


Figure 7 Steel plate samples embedded with FBG sensor

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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) Thermal spray the samples and test the characteristics of the coatings (Task 2.2 & 2.3);
- 2) Validate the theoretic analysis of the corrosion rate with center wavelength change of the embedded FBG sensors through experiments (Task 3.1).