CAAP Quarterly Report

Date of Report: *January 17, 2016*

Contract Number: DTPH5615HCAP10

Prepared for: *USDOT Pipeline and Hazardous Materials Safety Administration (PHMSA)*

Project Title: *Chemically Bonded Porcelain Enamel Coated Pipe for Corrosion Protection and Flow Efficiency*

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For quarterly period ending: *December 31, 2015*

Business and Activity Section

(a) Generated Commitments – Dr. Genda Chen directed the entire project and coordinated various project activities. Mr. Michael L. Koenigstein from Roesch Inc. oversaw the coating process of enamel on steel parts and provided feedback on research results from the material point of view. PEMCO provided commercial and special enamel materials for this application.

Mr. Fan Liang, a Ph.D. candidate in civil engineering at Missouri S&T, was on board since the beginning of this project, responsible for various material characterization and corrosion tests. Dr. Fujian Tang, a recently Ph.D. graduate under the supervision of Dr. Genda Chen participated in this project and provided input and guidance to Mr. Fan Liang for enamel coating characterization and corrosion tests. Dr. Signo Reis joined Roesch Inc. (our industrial partner on this project) in October, 2015, soon after the beginning date of this project. Since then, Dr. Reis has been contributing to this project in enamel coating process from industrial and material point of view.

The kick-off project meeting was held at Roesch Inc. on October 16 so that all participants (Drs. Chen, Tang, and Reis, and Mr. Fan Liang) can tour the enamel coating facility under the guide of Mr. Koenigstein. Since then, biweekly meetings have been held to discuss project progresses and plan future tasks.

Several pieces of steel pipe were purchased for this project on November 23, 2015, using credit card. As of today, this purchase was not yet posted on the project account. Enamel materials were supplied by PEMCO free of charge as part of the industrial support to this project.

(b) Status Update of Past Quarter Activities – Detailed updates are provided below by task.

Task 1 Optimization of enamel materials for durability, particle distribution, and thermal compatibility with steel

1a Steel samples

API 5L X65 steel pipe (MRC Global) with an outside diameter of 323.850 mm and a wall thickness of 9.525 mm was used in this study. Small samples (25 mm × 50 mm) were cut from the full-size pipe. The chemical composition and physical properties of the steel pipe are given in Table 1 and Table 2, respectively. In Table 2, CTE stands for Coefficient of Thermal Expansion, which was measured using Orton dilatometer.

Table 1 Chemical composition of steel pipe

Element	C	Mn	P	S	Si	Cu	Ni	Cr	Mo	Al	V	B	Ti	Cb	Fe
Wt.%	0.17	1.15	0.07	0.02	0.26	0.10	0.04	0.07	0.07	0.024	0.02	0.001	0.001	0.001	98.003

Table 2 Physical property of steel pipe

Grade	Yield Strength (ksi)	Ultimate Strength (ksi)	Yield/Ultimate Strength Ratio	Elongation %	CTE (ppm/℃)
API 5L X65	70	80	0.875	38.2	21.4

1b Enamel coatings

Two commercially-available boron-alumina silicate glass slurries (with clay and borax) from PEMCO (product ID 15R-972 and 15R-973) were used to prepare coatings for steel samples. These porcelain enamels (also known as vitreous enamel) are specially formulated, highly durable glass that can be permanently fused to metal at temperatures ranging from 800°C to 850°C. The metal may be steel or cast iron. The glasses 15R-972 and 15R-973 are designed to deliver a smooth, hard, chemically resistant, and durable coating.

Prior to coating steel samples, the thermal properties of glasses 15R-972 and 15R-973 such as glass transformation temperature (T_g), softening temperature (T_s), and CTE were determined by Orton automatic recording dilatometer (model 1500). Three samples of each coating were prepared and tested to ensure the repeatability of test data. Table 3 and Table 4 show the general properties of the two glass coatings.

Table 3 Thermal property of glass 15R-972

Glass ID	Density (g/cm^3)	T_g (℃)	T_s (℃)	CTE (200 to 400℃) (ppm/℃)
15R-972(1)	2.71	563	608	11.8
15R-972(2)	2.71	562	608	12.6
15R-972(3)	2.70	560	610	12.1
Average	2.71	562	609	12.2
STDEV	0.01	2	1	0.4

Table 4 Thermal property of glass 15R-973

Glass ID	Density (g/cm^3)	T_g (°C)	T_s (°C)	CTE (200 to 400°C) (ppm/°C)
15R-972(1)	2.71	549	616	12.9
15R-972(2)	2.72	550	611	13.8
15R-972(3)	2.70	550	612	13.0
Average	2.71	550	613	13.2
STDEV	0.01	1	3	0.5

Figure 1 shows the thermal elongation as a function of temperature for two glass coating compositions and steel. The steel has a measured CTE of 21.4 ppm/℃ while the glass coatings 15R-972 and 15R-973 have a measured CTE of 12.2±0.4 ppm/℃ and 13.2±0.5 ppm/℃, respectively. The CTE difference between the steel and glass coatings will generate compressive stress on the coatings during cooling, which is desirable. The glasses 15R-972 and 15R-973 have similar thermal properties with $550 \leq T_g \leq 562$°C and $609 \leq T_s \leq 613$°C.

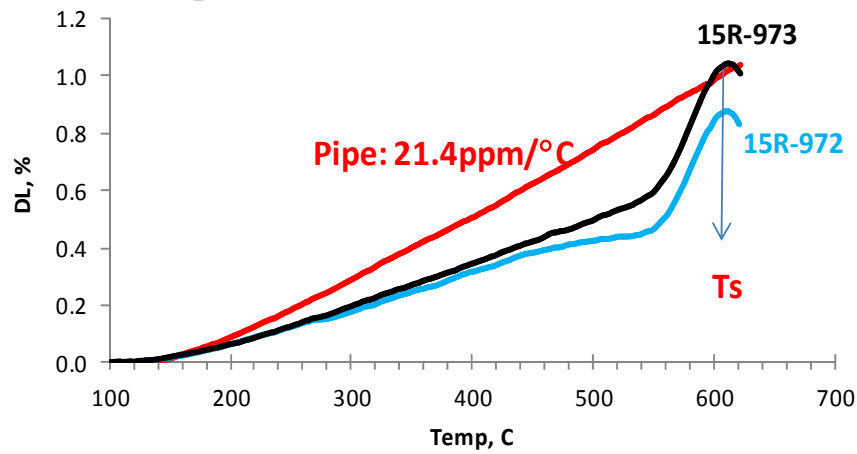

Figure 1 Thermal property of two coatings and steel measured with Orton dilatometer.

Task 2 Enameling process for coating uniformity, surface roughness, and efficiency without adverse effect on steel properties

2a Steel sample coating

Prior to coating, all steel samples were steel blasted for 1 min, and cleansed with a commercially available cleansing solvent. The glass slurry was applied manually to steel samples using a spray gun as specified in Table 5. After that, the samples were heated at 150 °C for 10 mins to drive off moisture, then fired at 840°C for 3 min, 9.4 min, and 39 min, and finally cooled to room temperature. Three representative coated samples are shown in Figure 2 at various feet per minute (FPM). By visual inspection, the sample at 12.5 FPM appears to have less air bubbles except the area around edges.

Table 5 Coating parameters

Parameters	Values
Chain speed (m/min)	1.5 (2 times)
Voltage (KV)	-
Atomisation pressure	3.5
Slip troughput per gun (l/min)	0.6
Number of guns	1
Gun-to-sample distance	30
Transfer efficiency	40

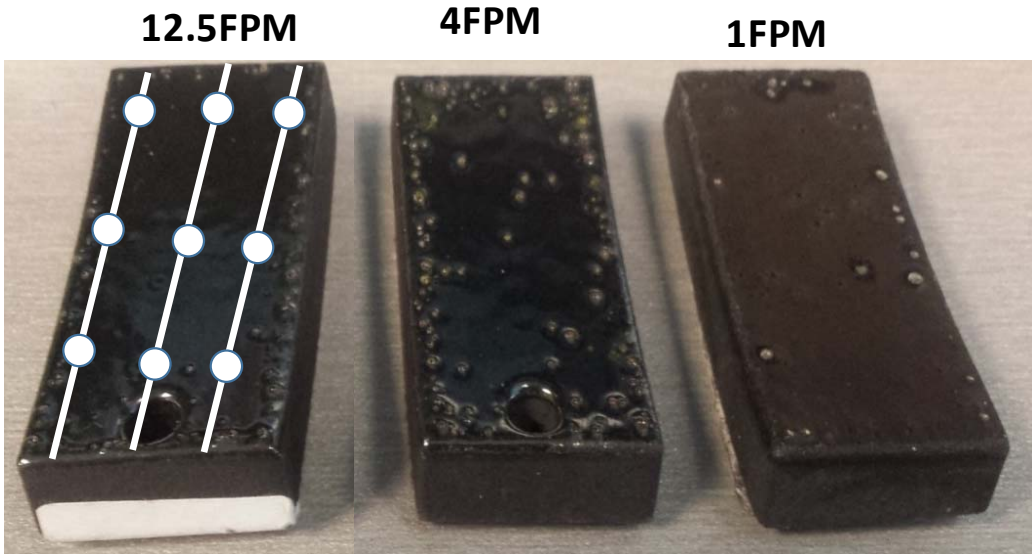

Figure 2 Coupon steel samples (25 mm × 50 mm) coated with glass 15R-972 and fired at 840°C for 3 min.

2b Coating thickness and roughness

The coating thickness and roughness were measured by MiniTest 6008 and 3D Hirox microscope, respectively. Nine test points were selected along three evenly-spaced parallel lines drawn on the surface of a sample, each having three points at equal distance as schematically represented on the left sample in Figure 2. The measured coating thicknesses at the nine points of three samples are summarized in Table 6. Overall, the thicknesses of enamel coatings are 310±12 μm, 232±6 μm, and 180±13 μm for 1 FPM, 4 FPM, and 12.5 FPM samples, respectively.

Table 6 Coating thickness of three samples measured by MINITEST 6008.

Point Number	1 FPM (39 min)	4 FPM (9.4 min)	12.5 FPM (3.0 min)
1	325	235	180
2	298	233	178
3	303	225	180
4	325	243	190
5	300	233	155
6	310	230	170
7	308	225	198
8	325	243	195
9	298	230	175
Average (μm)	310	233	180
Standard Deviation (μm)	12	6	13

At each of the nine sample points, four topographic lines as represented in Figure 3 could be drawn using the microscope, which amounts a total of 36 roughness values taken from each sample. Since the sample at 1 FPM was relatively too thick and its coating surface is relatively too rough for pipe application, only the other two samples were measured for their surface roughness. The measured coating roughness of 4 FPM and 12.5 FPM samples are presented in Table 7. Overall, the coating roughness values of the two samples are 2.7±0.5 μm and 2.2±0.7 μm, respectively.

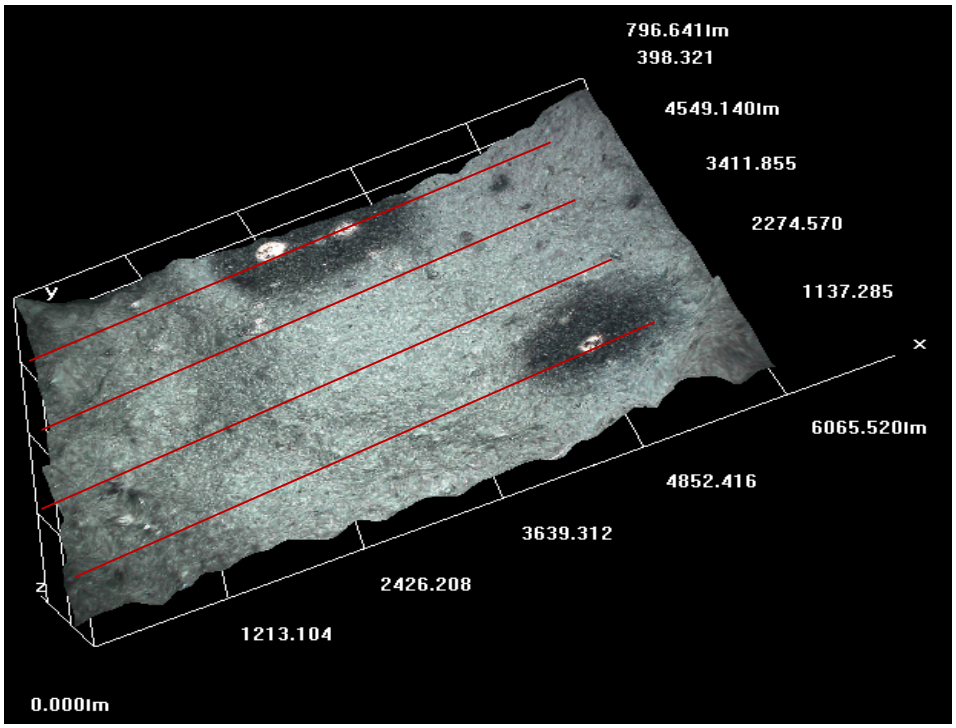

Figure 3 3D view of a single point using the Hirox microscope.

Table 7 Coating roughness of two samples

Point Number	Ra (μm)	
	4 FPM	12.5 FPM
1	2.57	2.37
2	2.09	1.60
3	3.02	1.63
4	4.20	2.56
5	3.92	2.16
6	2.58	1.81
7	2.25	1.43
8	2.29	2.53
9	3.80	3.90
10	2.71	2.87
11	2.91	2.99
12	2.42	2.83
13	2.42	1.91
14	2.22	1.44
15	1.87	1.38
16	2.91	2.24
17	3.22	2.03
18	2.22	1.76
19	1.90	1.54
20	3.23	1.66
21	2.93	2.64
22	2.18	3.21
23	2.65	2.19
24	2.21	2.46
25	3.03	1.76
26	2.06	1.66

27	2.74	1.52
28	2.31	1.37
29	3.12	2.99
30	2.79	3.04
31	2.84	2.61
32	2.08	3.60
33	2.99	1.93
34	2.63	1.85
35	2.40	1.55
36	2.23	1.73
Average (μm)	2.67	2.19
Standard Deviation (μm)	0.54	0.66

Task 3 Characterizations of enamel-coated pipes for microstructure/porosity, chemical adhesion, and corrosion resistance

This task will not start till the 1st quarter in 2016.

Task 4 System performance of in-situ enamel-coated pipelines-stress distribution and SCC

This task will not start till the 2nd quarter in 2017.

(c) Description of any Problems/Challenges - In this quarter, the research team has been waiting for delivery of the third type of enamel from PEMCO in order to optimize the enamel material for pipe application. Towards the end of this quarter, the team received the third type of enamel.

***(d)* Planned Activities for the Next Quarter -** The following activities in various tasks will be executed during the next reporting quarter.

Task 1 Optimization of enamel materials for durability, particle distribution, and thermal compatibility with steel

The particle size distribution of enamel materials will be outsourced to Mo-Sci in Rolla, MO, which is indirectly associated with our industrial partner (Mr. Michael Koenigstein) from Roesch Inc.

Task 2 Enameling process for coating uniformity, surface roughness, and efficiency without adverse effect on steel properties

Enamel slurry and dry power application processes will be compared. The coating uniformity, surface roughness will continue to be measured for various samples.

Task 3 Characterizations of enamel-coated pipes for microstructure/porosity, chemical adhesion, and corrosion resistance

A metric of corrosion tests for three types of enamels and their combination will be developed and executed partially.