CAAP Quarterly Report

Date of Report: *April 10, 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*

Prepared by: *Missouri University of Science and Technology (Missouri S&T)*

Contact Information: *Genda Chen, Ph.D., P.E., F.ASCE, Email: gchen@mst.edu, Phone: (573) 341-4462*

For quarterly period ending: *March 31, 2016*

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.

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.

During this reporting quarter, **carbide paper, pure sodium chloride, and epoxy** were purchased for this project using credit card.

(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 Thermal properties

Two commercially-available silicate glass slurries (with clay and borax) (product ID 15R-972 and 16R-001) and one silicate glass powder (product ID GP2118) from PEMCO were used to prepare coatings for steel samples. These porcelain enamels are vitreous coatings that are bonded to substrate metal by fusing glass frits at a temperature of 800-850°C. The glasses are designed to deliver a smooth, hard, chemically resistant, and durable coating.

Prior to coating steel samples, the thermal properties of glasses 15R-972, 16R-001 and GP2118 such as glass-transition temperature (T_g), softening temperature (T_s), and coefficient of thermal expansion (CTE) were determined using the Orton automatic recording dilatometer (model 1500). Three samples of each coating were prepared and tested to understand the variation of test data. (Since the CTE values of two samples of Glass 16R-001are quite close, the third sample is not prepared.) Table 1 to Table 3 show the general thermal properties of the three glass coatings.

Table 1 Thermal property of glass 15R-972

Glass ID	T_g (℃)	T_s (℃)	CTE (200 to 400℃) (ppm/℃)
15R-972(1)	552	616	13.0
15R-972(2)	551	610	12.1
15R-972(3)	553	611	13.8
Average	552	612	13
STDEV	0.8	2.6	0.7

Table 2 Thermal property of glass 16R-001

Glass ID	T_g (℃)	T_s (℃)	CTE (200 to 400℃) (ppm/℃)
16R-001(1)	537	602	11.3
16R-001(2)	542	611	10.9
Average	540	607	11.1
STDEV	2	4.5	0.2

Table 3 Thermal property of glass GP2118

Glass ID	T_g (℃)	T_s (℃)	CTE (200 to 400℃) (ppm/℃)
GP2118-1	549	612	12.2
GP2118-2	554	620	11.9
GP2118-3	552	616	13.0
Average	552	616	12.3
STDEV	2	3	0.5

Figure 1 shows the thermal elongation in the longitudinal direction of the three glass coatings and steel as a function of temperature. The steel has a measured CTE of 19.7 ppm/℃ while the glass coatings 15R-972, 16R001, and GP2118 have a measured CTE of 13.0±0.7 ppm/℃, 11.1±0.2 ppm/℃, and12.3 ±0.5 ppm/℃, respectively. The CTE of the steel remains constant over a temperature range of 100 to 600℃, while the CTE of the glass coatings is constant only over a range of 200 to 400℃.

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, 16R-001 and GP2118 have similar thermal properties with $540 \leq T_g \leq 552$°C and $607 \leq T_s \leq 616$°C.

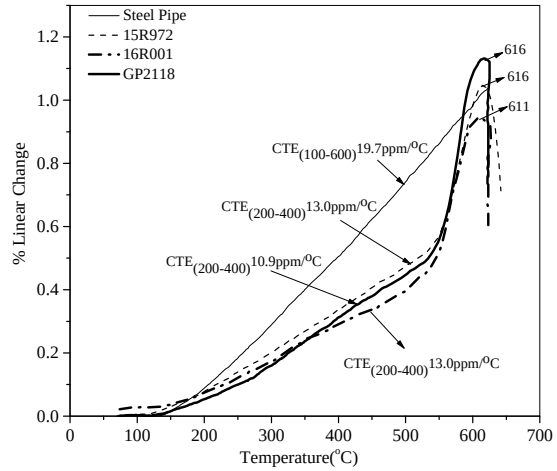


Figure 1 Thermal properties of three coatings and steel measured with Orton dilatometer

1b Particle size analysis

The particle size distribution of enamel materials is outsourced to Mo-Sci in Rolla, MO. LS particle size analyzer is used for particle size analysis. Table 4 shows the particle volume percentage for different diameter ranges. The particle distributions of the three enamels are presented in Figure 2.

Table 4 Particle distributions of three types of coating powder by volume percentage

Diameter (μm)	15R-972 Volume (%)	16R-001 Volume (%)	GP2118 Volume (%)
1.385	0.000	0.001	0.000
1.520	0.001	0.013	0.000
1.669	0.015	0.076	0.005
1.832	0.088	0.227	0.031
2.011	0.256	0.463	0.100
2.207	0.521	0.748	0.219
2.423	0.833	1.064	0.367
2.660	1.171	1.400	0.532
2.920	1.513	1.739	0.707
3.205	1.835	2.056	0.880
3.519	2.124	2.332	1.052
3.863	2.341	2.531	1.200
4.241	2.486	2.659	1.333
4.655	2.546	2.704	1.440
5.110	2.558	2.701	1.539
5.610	2.543	2.667	1.633
6.158	2.534	2.632	1.734
6.760	2.547	2.613	1.856
7.421	2.569	2.597	1.985
8.147	2.614	2.599	2.142
8.943	2.632	2.577	2.282
9.818	2.674	2.576	2.448
10.78	2.698	2.562	2.594
11.83	2.781	2.605	2.779
12.99	2.884	2.672	2.959
14.26	3.026	2.774	3.147

Diameter (μm)	15R-972 Volume (%)	16R-001 Volume (%)	GP2118 Volume (%)
15.65	3.178	2.884	3.327
17.18	3.261	2.927	3.433
18.86	3.331	2.955	3.533
20.71	3.315	2.910	3.558
22.73	3.353	2.925	3.647
24.95	3.405	2.968	3.752
27.39	3.505	3.071	3.913
30.07	3.599	3.179	4.063
33.01	3.577	3.186	4.089
36.24	3.429	3.079	3.976
39.78	3.103	2.810	3.666
43.67	2.681	2.462	3.252
47.94	2.232	2.105	2.806
52.62	1.824	1.806	2.408
57.77	1.512	1.613	2.122
63.42	1.281	1.501	1.930
69.61	1.115	1.440	1.813
76.42	0.983	1.377	1.724
83.89	0.860	1.276	1.628
92.09	0.744	1.132	1.519
101.10	0.628	0.951	1.385
110.98	0.509	0.749	1.213
121.83	0.379	0.539	0.986
133.74	0.243	0.338	0.707
146.81	0.119	0.165	0.396
161.17	0.038	0.055	0.155
176.92	0.006	0.009	0.030
194.22	0.000	0.001	0.002

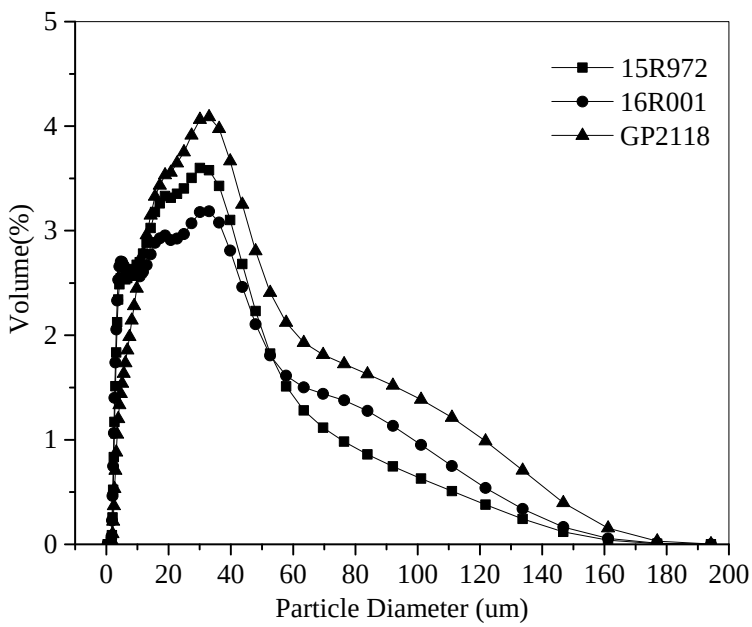


Figure 2 Particle distribution curve

With the same application thickness, reducing particle sizes can reduce the heat (temperature-time) required to soften the particles or mass of particles. The exposed surface increases with the decrease of particle size. As the exposed surface increases, the amount of heat (temperature-time) required to soften

the particles decreases. It can be seen from Table 5 that the average particle diameter, the median particle diameter, and the mode of particle diameter of powder GP2118 are larger than those of enamel powders 15R-972 and 16R-001. Therefore, Glass powder GP2118 has a larger particle size than that of the other two particles. So we use 10 minutes during the firing process for good surface with enamel powder GP2118 instead of 3.5 minutes with enamel 15R-972 and 16R-001, which will be introduced in the following steel sample coating.

As shown in Table 5, the standard deviation of the particle diameter of powder GP2118 is also the largest. The 10%, 50%, and 90% passage diameters (D_{10}, D_{50}, and D_{90}) show that the particle diameter of powder GP2118 is the largest among the three types of powders. This means that the particle size of enamel powder GP2118 is more uniformly distributed over the entire range of particle sizes; the voids among large particles will be filled by relatively small particles, which may result in a much smoother coating surface on steel samples.

Table 5 Volume statistics

	Mean (μm)	Median (μm)	Mode (μm)	S.D. (μm)	$d_{10}(\mu m)$	$d_{50}(\mu m)$	$d_{90}(\mu m)$
GP2118	32.82	23.45	34.58	30.18	5.809	23.45	75.47
15R-972	24.34	16.72	31.50	23.89	4.128	16.72	53.31
16R-001	25.57	15.87	34.58	26.86	3.845	15.87	61.78

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

2a Steel sample coating

Both wet process of enamel slurry and electrostatic process of enamel powder are used to coat steel coupon samples. Wet process requires little capital expenses and provides an acceptable finish for industrial applications particularly when the part to be coated is small. Electrostatic process of enamel powder is most promising to coating large pipelines in field conditions. Electrostatic process also gains several advantages over wet process. Electrostatic process directly using powder instead of slurry not only saves the energy to make slurry and reduce the time and cost, but can also solve the uneven thickness problem associated with gravity effect for coating large pieces. Besides, wet process involves water to make slurry and may lead to more chemical reactions between steel and water and generate more hydrogen gas and air bubbles. Theoretically the coating with electrostatic process has less air bubbles than the coating with wet process.

Prior to coating, all steel samples were steel blasted for 1 min, and cleansed with a commercially available cleansing solvent to remove oil and contamination. For the wet process, the glass slurry was applied manually to steel samples using a spray gun as specified in Table 6. After that, the samples were heated at 150 °C for 10 mins to drive off moisture, then fired at 843°C for 3 min, and finally cooled to room temperature. For the electrostatic process, the glass powder was applied automatically to steel samples using a spray gun as specified in Table 6 and the samples are fired at different temperatures, 788℃, 816℃, and 843℃ for 10 minutes.

Since different types of enamel have varying thermal properties, particle size distribution, and chemical compositions, mixes of the three enamel slurries, 15R-972, 15R973, and 16R-001 are used to coat the steel coupon samples in the wet process. Table 7 shows the mix design of the three enamel coatings, 15R-972, 15R973, and 16R-001 in the wet process.

Table 6 Coating parameters

Coating application parameters	Wet by Hand Spaying	Electrostatic-Dry Automatic spraying
Chain speed (m/min)	12FPM	12FPM
Voltage (KV)	-	80KV
Atomisation pressure	3.5	0.65
Slip troughput per guns	0.6	0.35
Number of guns	1	12
Distance gun to work piece (cm)	40	50
Transfer efficiency (%)	40	60-80

Table 7 Mix design of three enamel coatings

Volume,% / No.	15R-973	15R-972	16R-001
#1	100	0	0
#2	0	100	0
#3	0	0	100
#4	75	25	0
#5	50	50	0
#6	25	75	0
#7	0	75	25
#8	0	50	50
#9	0	25	75

Figure 3 shows the different mix designs of enamel coatings using the wet process.

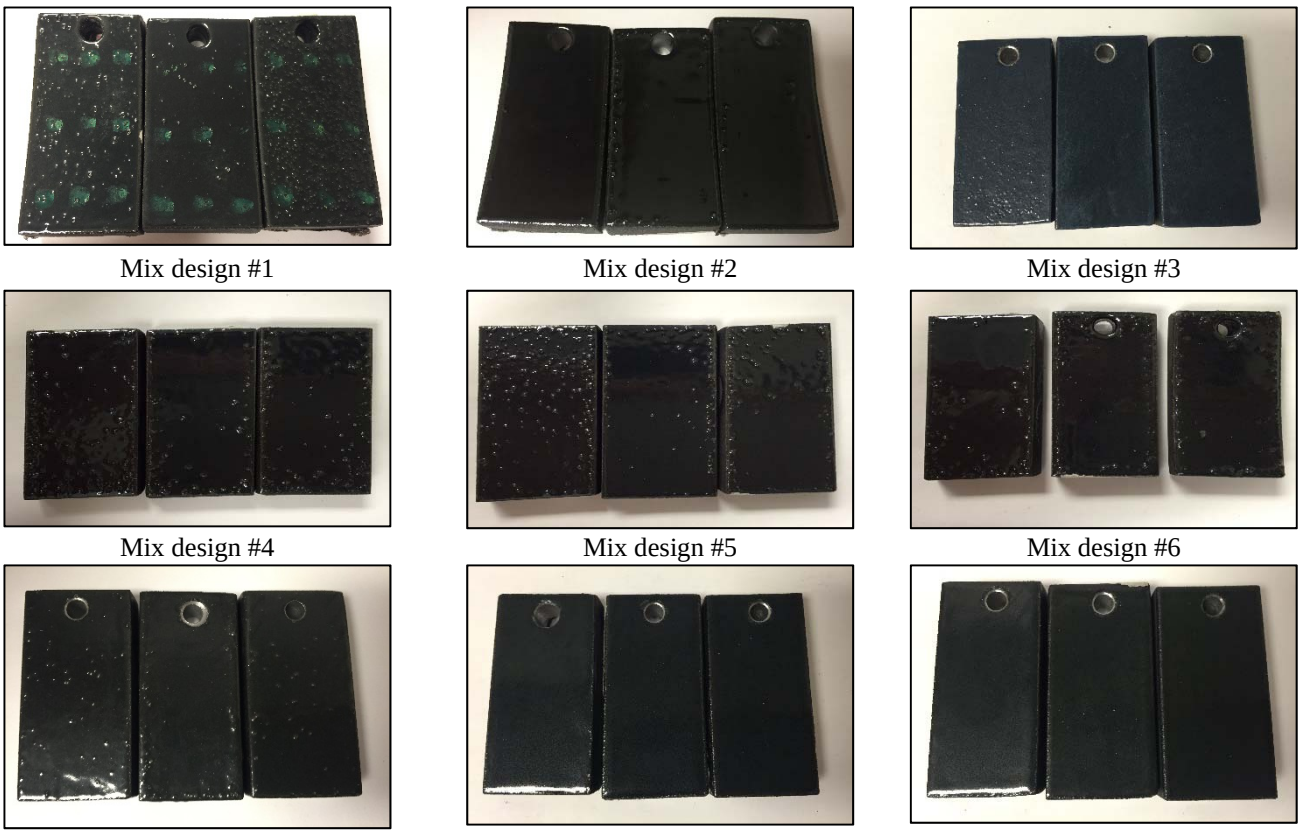


Figure 3 Coupon steel samples (25 mm × 50 mm) coated with the wet process

From Figure 3, we can see that the surfaces of coupons coated with enamel 15R-973 and its mixture with 15R-972 have many air bubbles. Therefore, we decided not to mix these three enamel slurries

together and these coatings will also not be considered in the following coating thickness and roughness analysis and corrosion test. Samples coated with enamel 15R-972 have some air bubbles on the edge of the surface and samples coated with a mix of enamel 15R-972 (75%) and 16R-001(25%) have small air bubbles on the surface. But the other two mixtures of enamel 15R-972 and 16R-001 can give a smooth surface without air bubbles and pin holes.

The glass powder GP2118 is applied in the electrostatic process. Figure 4 shows the coated samples with enamel GP2118 applied with the electrostatic process at three different firing temperatures: 1450°F, 1500°F, and 1550°F, respectively. The samples with firing temperatures 1500°F and 1550°F give a smooth surface but the three samples with firing temperature have numerous pin holes, which may be due to insufficient heat and low temperature.

GP2118-1450F GP2118-1500F GP2118-1550F

Figure 4 Coupon steel samples (25 mm × 50 mm) coated in the electrostatic process

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. The measured coating thickness average and standard deviation values at the nine points are summarized in Table 8. Overall, the thicknesses of enamel coating 15R-972 is less than 200 μm. The thickness of enamel coating 16R-001 has coating thickness of $236\pm16\mu m$, which is relatively larger than that of coating 15R-972. Basically, with more coating16R-001in the mix ratio, the coating becomes thicker. For the electrostatic process, the thickness increases with the increasing firing temperature from $320\pm19\mu m$ to $340\pm9\mu m$.

Table 8 Coating thickness measured by MINITEST 6008

Coating Num.	Thickness (μm)
#2 15R-972	193 ± 16
#3 16R-001	236 ± 16
#7 15R-972(75%) + 16R-001(25%)	203 ± 10
#8 15R-972(50%) + 16R-001(50%)	280 ± 12
#9 15R-972(25%) + 16R-001(75%)	240 ± 17
#10 GP-2118-1450	320 ± 19
#11 GP-2118-1500	327 ± 9
#12 GP-2118-1550	340 ± 23

At each of the nine sample points, four topographic lines as represented in Figure 5 could be drawn using the microscope, which amounts a total of 36 roughness values taken from each sample. The cut-off value of 0.08mm is chosen for all the test with3D Hirox microscope. The measured coating roughness of different samples are presented from Table 9 to Table 11.

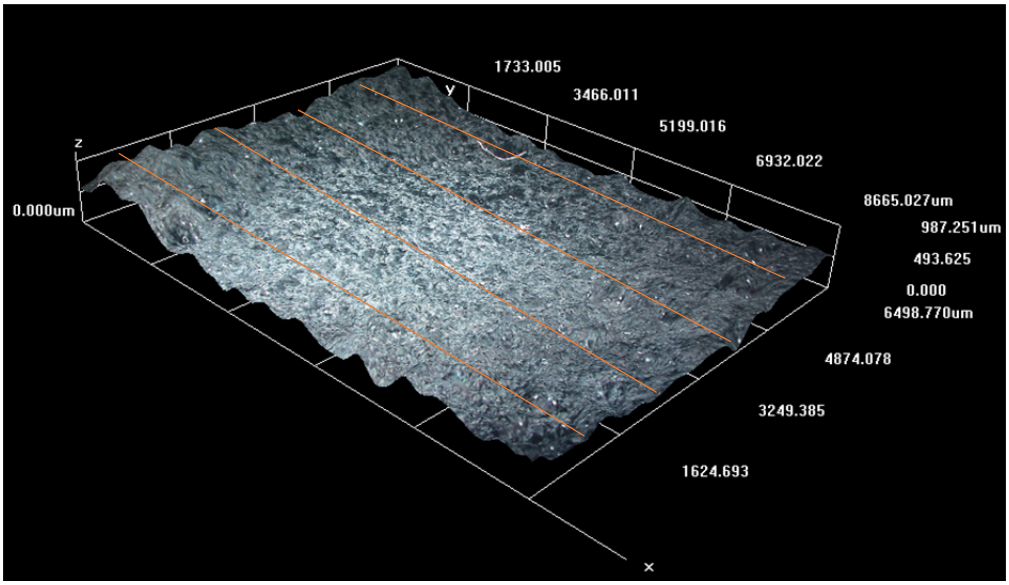


Figure 5 3D view of a single point using the Hirox microscope.

Table 9 Coating roughness with enamel 15R-972 and 16R-001

	15R-972(μm)			16R-001(μm)		
Num.	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
1	0.8	0.82	0.69	0.36	0.51	0.68
2	0.8	0.69	0.69	0.44	0.41	0.63
3	0.8	0.67	0.64	0.41	0.47	0.66
4	0.77	0.73	0.7	0.45	0.47	0.65
5	0.71	0.63	0.56	0.48	0.52	0.76
6	0.63	0.52	0.55	0.52	0.52	0.86
7	0.71	0.58	0.46	0.48	0.55	0.77
8	0.72	0.57	0.62	0.5	0.59	0.91
9	0.66	0.67	0.54	0.45	0.65	0.62
10	0.58	0.64	0.61	0.4	0.64	0.52
11	0.62	0.59	0.52	0.44	0.6	0.56
12	0.73	0.63	0.59	0.5	0.68	0.59
13	0.78	0.66	0.58	0.44	0.52	0.55
14	0.74	0.65	0.64	0.38	0.42	0.56
15	0.83	0.73	0.6	0.43	0.49	0.54
16	0.82	0.7	0.6	0.45	0.52	0.58
17	0.68	0.78	0.6	0.57	0.45	0.58
18	0.69	0.79	0.64	0.61	0.51	0.6
19	0.64	0.83	0.78	0.65	0.48	0.52
20	0.66	0.86	0.75	0.72	0.52	0.58
21	0.77	0.73	0.52	0.8	0.42	0.63
22	0.8	0.72	0.57	0.7	0.47	0.61
23	0.7	0.63	0.59	0.74	0.46	0.52
24	0.81	0.81	0.65	0.76	0.45	0.61
25	0.72	0.58	0.89	0.51	0.52	0.73
26	0.64	0.54	0.85	0.51	0.52	0.7
27	0.63	0.54	0.91	0.45	0.51	0.63
28	0.7	0.56	0.9	0.57	0.59	0.77
29	0.91	0.75	0.77	0.51	0.69	0.62
30	0.91	0.78	0.87	0.45	0.66	0.57
31	0.9	0.83	0.85	0.5	0.71	0.65
32	0.84	0.66	0.82	0.42	0.67	0.61
33	0.97	0.64	0.88	0.51	0.79	0.49
34	0.79	0.7	0.74	0.59	0.73	0.56

	15R-972(μm)			16R-001(μm)		
Num.	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
35	0.89	0.61	0.85	0.54	0.74	0.56
36	0.86	0.66	0.84	0.55	0.74	0.51
Average	0.76	0.68	0.69	0.52	0.56	0.62
STDEV	0.09	0.09	0.13	0.11	0.10	0.09

Table 10 Coating roughness with mix design of enamel 15R-972 and 16R-001

	#7 15R-972(75%) + 16R-001(25%) (μm)			#8 15R-972(50%) + 16R-001(50%) (μm)			15R-972(25%) + 16R-001(75%) (μm)		
Num.	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
1	0.78	0.71	0.66	0.54	0.96	0.81	0.63	0.76	0.72
2	0.77	0.66	0.58	0.55	0.7	0.81	0.6	0.73	0.67
3	0.75	0.65	0.61	0.64	0.81	0.74	0.55	0.71	0.8
4	0.68	0.64	0.57	0.62	0.86	0.83	0.61	0.66	0.75
5	0.68	0.66	0.66	0.63	0.65	0.7	0.51	0.65	0.5
6	0.8	0.61	0.57	0.6	0.69	0.61	0.51	0.59	0.63
7	0.71	0.65	0.66	0.58	0.76	0.59	0.49	0.61	0.51
8	0.8	0.68	0.57	0.51	0.84	0.74	0.58	0.66	0.51
9	0.83	0.9	0.7	0.6	0.81	0.59	0.59	0.55	0.58
10	0.68	0.8	0.66	0.63	0.74	0.69	0.51	0.63	0.59
11	0.7	0.75	0.72	0.58	0.81	0.65	0.49	0.58	0.46
12	0.77	0.86	0.65	0.55	0.77	0.68	0.5	0.6	0.56
13	1	0.63	0.82	0.82	0.77	0.87	0.65	0.76	0.78
14	0.84	0.58	0.85	0.74	0.71	0.84	0.62	0.74	0.74
15	0.7	0.69	0.89	0.69	0.76	0.83	0.66	0.69	0.68
16	0.84	0.65	0.91	0.68	0.75	0.94	0.56	0.67	0.72
17	1.12	0.78	0.8	0.71	0.72	0.67	0.54	0.57	0.55
18	0.8	0.65	0.64	0.69	0.7	0.66	0.49	0.59	0.57
19	0.74	0.71	0.73	0.78	0.79	0.68	0.58	0.6	0.61
20	1.06	0.71	0.73	0.88	0.73	0.68	0.57	0.62	0.7
21	0.82	0.95	0.69	0.81	0.75	0.64	0.62	0.67	0.65
22	0.81	0.72	0.75	0.68	0.75	0.8	0.66	0.57	0.57
23	0.89	0.9	0.76	0.71	0.71	0.64	0.61	0.6	0.58
24	0.81	0.83	0.66	0.71	0.77	0.75	0.56	0.7	0.62
25	0.98	0.83	0.61	0.94	1.17	0.72	0.66	0.65	0.67
26	1.01	0.83	0.72	0.98	1.05	0.59	0.58	0.69	0.68
27	0.89	0.82	0.69	1.01	1.05	0.7	0.6	0.68	0.63
28	1.02	0.79	0.78	0.85	1	0.64	0.54	0.69	0.7
29	0.85	0.76	0.82	0.75	0.95	0.7	0.74	0.62	0.59
30	0.74	0.76	0.8	0.7	0.9	0.67	0.81	0.63	0.55
31	0.69	0.72	0.89	0.71	0.8	0.6	0.79	0.65	0.6
32	0.79	0.67	0.85	0.74	0.9	0.7	0.8	0.68	0.54
33	1.05	1.04	0.97	0.76	0.68	0.72	0.53	0.8	0.72
34	0.81	0.98	0.76	0.69	0.74	0.69	0.56	0.68	0.67
35	0.73	0.89	0.77	0.78	0.72	0.77	0.56	0.64	0.75
36	0.9	0.98	0.91	0.79	0.68	0.76	0.6	0.67	0.77
Average	0.83	0.76	0.73	0.71	0.80	0.71	0.60	0.66	0.64
STDEV	0.12	0.12	0.11	0.12	0.12	0.08	0.08	0.06	0.09

Table 11 Coating roughness of samples in the electrostatic process

	GP-2118-1450°F			GP-2118-1500°F			GP-2118-1550°F		
Num.	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
1	0.62	1.02	1.52	1.18	0.73	0.79	0.88	0.73	0.98
2	0.61	0.97	1.49	1.23	0.78	0.65	0.91	0.87	1
3	0.69	1.09	1.6	1.23	0.69	0.89	0.92	0.85	0.82

	GP-2118-1450°F			GP-2118-1500°F			GP-2118-1550°F		
Num.	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3	Sample 1	Sample 2	Sample 3
4	0.62	1.04	1.24	1.3	0.65	0.74	0.95	0.97	1.07
5	0.57	1.08	0.75	0.97	0.73	0.9	0.96	0.62	1.35
6	0.63	0.91	0.78	0.88	0.8	1.03	1.03	0.76	1.1
7	0.67	1.06	0.79	1.14	0.76	1.04	0.95	0.69	1.12
8	0.59	0.97	0.8	1.06	0.69	1.1	1.04	0.67	1.17
9	0.97	1.24	1.41	1.58	0.56	1.3	1.09	0.67	0.78
10	1.08	1.13	1.39	1.42	0.54	1.22	1.26	0.68	0.88
11	1.15	1.18	1.28	1.48	0.59	1.19	0.99	0.82	0.95
12	0.88	1.11	1.44	1.46	0.72	1	1.1	0.87	0.88
13	0.81	0.9	1.44	1.38	0.77	1.11	1.14	1.08	1.12
14	0.72	0.92	1.49	1.17	0.77	1.1	1.06	0.96	1.15
15	0.72	0.88	1.21	1.11	0.79	1.3	1.04	0.94	1.2
16	0.74	0.72	1.19	1.12	0.66	1.33	1.12	0.98	1.2
17	0.79	0.84	1.11	0.69	0.83	0.76	1.73	0.78	0.9
18	0.86	0.67	1.06	0.83	0.94	0.83	1.61	0.8	0.89
19	0.89	0.85	1.25	0.93	0.87	0.91	1.68	0.8	1.02
20	0.96	0.76	1.12	0.75	0.84	0.96	1.79	0.79	0.92
21	0.85	0.79	0.94	1.17	0.63	0.99	1.64	0.79	1.24
22	0.9	0.77	1.02	1	0.65	1.06	1.54	0.7	1.31
23	0.93	0.89	1.06	1.05	0.64	0.87	1.45	0.71	1.22
24	0.84	0.68	1.02	1.25	0.61	0.89	1.33	0.76	1.2
25	0.71	0.75	0.91	1.33	0.5	0.67	1.42	0.75	0.71
26	0.74	0.77	0.89	1.18	0.47	0.8	1.44	0.66	0.84
27	0.86	0.76	0.74	1.15	0.5	0.8	1.48	0.72	0.9
28	0.95	0.78	0.9	1.07	0.63	0.65	1.41	0.71	0.72
29	1.28	0.92	0.83	1.59	0.76	0.97	1.15	0.75	0.92
30	1.39	0.91	0.96	1.76	0.84	0.81	1.38	0.66	0.94
31	1.3	1.09	0.88	1.48	0.86	0.86	1.22	0.74	0.93
32	1.44	1.13	0.8	1.63	0.8	1.04	1.43	0.71	0.91
33	1.26	1.03	0.97	1.02	0.77	0.96	1.54	0.86	0.94
34	1.19	1.17	1.07	0.83	0.79	1.14	1.55	0.78	1.09
35	1.18	1.05	1.04	0.84	0.85	1.03	1.66	0.98	1.06
36	1.25	1.09	1.16	0.97	0.97	0.94	1.88	0.84	1.08
Average	0.91	0.94	1.10	1.17	0.72	0.96	1.30	0.79	1.01
STDEV	0.24	0.16	0.25	0.26	0.12	0.18	0.28	0.11	0.16

It can be seen from Table 12 that the surface roughness with coating 16R-001is lower than that with enamel coating 15R-972. For a mix of 15R-972 and 16R-001, the roughness values decreases with the increase of coating 16R-001 in the mix. Overall, the coating roughness is less than 1 μm for the wet process and the standard deviation is around 0.1 μm. The coating roughness with the electrostatic process is larger than that with the wet process. With the increasing temperature, the roughness goes larger. The roughness value is larger than 1 μm for some samples and the standard deviation is around 0.2 μm.

Table 12 Coating roughness summary (μm)

Coating Num.	Roughness(μm)		
	Sample 1	Sample 2	Sample 3
#2 15R-972	0.76±0.09	0.68±0.09	0.69±0.13
#3 16R-001	0.52±0.11	0.56±0.1	0.62±0.09
#7 15R-972(75%) + 16R-001(25%)	0.83±0.12	0.76±0.12	0.73±0.11
#8 15R-972(50%) + 16R-001(50%)	0.71±0.12	0.8±0.12	0.71±0.08
#9 15R-972(25%) + 16R-001(75%)	0.6±0.08	0.66±0.06	0.64±0.09

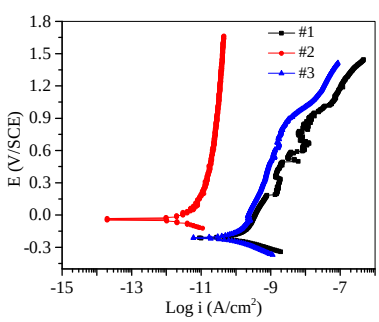
GP-2118-1450	0.91±0.24	0.94±0.16	1.1±0.25
GP-2118-1500	1.17±0.26	0.72±0.12	0.96±0.18
GP-2118-1550	1.3±0.28	0.79±0.11	1.01±0.16

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

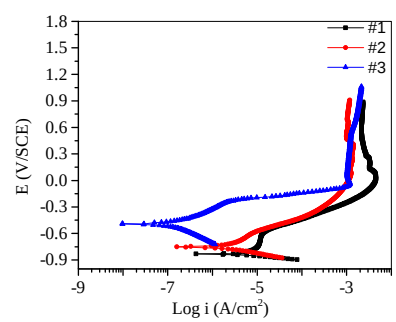
Electrochemical tests were conducted to understand the corrosion process occurring at the interface between a metal and its surrounding electrolyte. They are rapid because an instrument is used to polarize the metal sample to accelerate the corrosion process and make each measurement completed in minutes or hours. The electronic instrument used in these tests is referred to as Potentiostat that controls the potential between a working electrode and a reference electrode and measures the current between the working electrode and a counter electrode. To run an experiment, the potential is changed in a systematic way and the current response is measured. The applied potential will allow a redox reaction to occur and the current will be indicative of the rate of the reaction.

3a Potentiodynamic polarization test

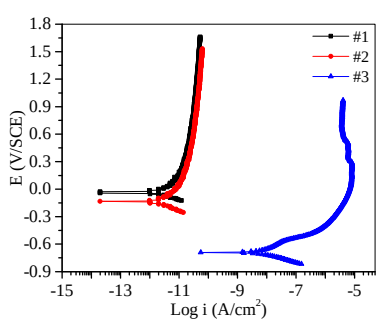
In this experiment, all samples were immersed in 3.5wt.% salt solution consisting of distilled water and purified sodium chloride. Samples were tested at room temperature with a typical three-electrode setup, including a 25.4mm×25.4mm×0.254mm platinum sheet as a counter electrode, saturated calomel electrode (SCE) as a reference electrode, and the steel coupon as a working electrode. All three electrodes were connected to a Gamry, Reference 600 potentiostat/galvanostat/ZRA for data acquisition. Open-circuit potentials (OCP) were recorded after the samples have been immersed in the solution for 1h. After the OCP test, the potentiodynamic polarization test was conducted from E_{OCP} -300mV to E_{OCP} +1500mV with a scanning rate of 5.0 mV/s. Potentiodynamic polarization curves (logarithmic current versus potential) of the enamel coated samples in 3.5wt.% NaCl solution are presented in Figure 6.



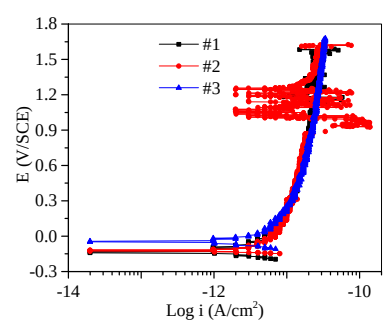
#3 16R-001



#7 15R-972(75%) + 16R-001(25%)



#8 15R-972(50%) + 16R-001(50%)



#9 15R-972(25%) + 16R-001(75%)

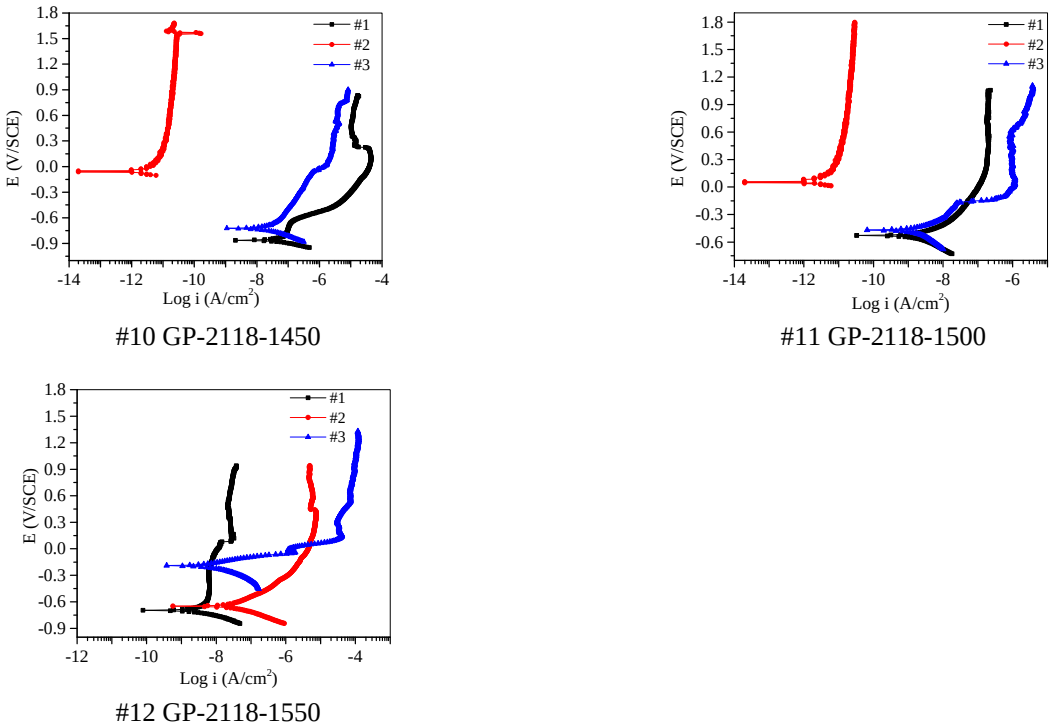


Figure 6 Potentiodynamic polarization curves

An applied voltage that is larger than E_{corr} will accelerate an oxidation (corrosion) reaction and an applied voltage that is less than E_{corr} will accelerate a reduction reaction. The value of either the anodic or cathodic current at E_{corr} is called the corrosion current, I_{corr}. I_{corr} can be used to calculate the corrosion rate of the metal. Corrosion rate r can be defined as the amount of corrosion loss per year in thickness. E_{corr} and I_{corr} can be obtained by extrapolating the linear portions of the logarithmic current versus potential plot back to their intersection. The coordinate of the intersection point corresponds to E_{corr} and I_{corr}.

After analyzing the above potentiodynamic polarization curves, we can get E_{corr}, I_{corr} and r for each sample, which are summarized in Table 13. It can be seen from Table 13 that the corrosion rate of the samples coated with a mix of 15R-972(75%) + 16R-001(25%) is the largest with an average value of 1.705 mils/yr and the corrosion rate of the samples coated with a mix of 15R-972(25%) + 16R-001(75%) is the smallest with an average value of 2.37710^{-6}mils/yr, which is 10^{-6} times smaller than that of samples coated with a mix of 15R-972(75%) + 16R-001(25%). The coupon samples coated in the electrostatic process were found to have higher corrosion rate than that of coupon samples coated in the wet process.

The free potential of low carbon steel is close to -0.7V to -0.6V according to the galvanic series for sea water, which is a list of metal and alloys arranged according to their relative corrosion potential. The average E_{corr} of samples coated with a mix of 15R-972(75%) + 16R-001(25%) is 0.693mV, which means the steel samples coated with this coating corrode. It is likely that this particular coating contains preferential pathways through which the electrolyte can easily penetrate and reach the metal surface and, thus, leading to the activation of the electrochemical reactions on the steel surface.

The potentiodynamic polarization test was conducted from E_{OCP} -300mV to E_{OCP} +1500mV. With an increase of the potential, the anodic current gradually becomes independent of potential at several hundred mV noble to E_{OCP} in a mix design of 15R-972(75%) + 16R-001(25%). The phenomenon is probably related to the formation of corrosion products at the metal/enamel interface and concentration

polarization may happen. This phenomenon is due to the fact that the diffusion of oxygen or hydrogen ion is not fast enough to sustain the kinetically controlled reaction rate and the steel ions cannot leave the steel surface or diffuse out of the corrosion products on the steel surface into the bulk solution.

Table 13 E_{corr} and I_{corr} summary

Num.	$I_{corr}(nA)$	$r(mil/yr)$	$E_{corr}(V)$
15R972 #1	226.000	0.103	-0.774
15R972 #3	0.012	5.63E-06	-0.163
16R-001 #1	0.116	5.31E-05	-0.222
16R-001 #2	0.006	2.79E-06	-0.044
16R-001 #3	0.102	4.67E-05	-0.213
0.25 15R-972&0.75 16R001 #1	0.004	2.05E-06	-0.130
0.25 15R-972&0.75 16R001 #2	0.005	2.13E-06	-0.122
0.25 15R-972&0.75 16R001 #3	0.006	2.95E-06	-0.044
0.5 15R-972&0.5 16R-001 #1	0.006	2.64E-06	-0.037
0.5 15R-972&0.5 16R-001 #2	0.007	3.23E-06	-0.137
0.5 15R-972&0.5 16R-001 #3	4.690	0.002	-0.688
0.75 15R-972 &0.25 16R-001 #1	8510.000	3.892	-0.833
0.75 15R-972 &0.25 16R-001 #2	2430.000	1.111	-0.753
0.75 15R-972 &0.25 16R-001 #3	247.000	0.113	-0.492
GP2118-1450 #1	96.600	0.044	-0.864
GP2118-1450 #2	0.006	2.52E-06	-0.057
GP2118-1450 #3	40.500	0.019	-0.725
GP2118-1500 #1	7.800	0.004	-0.536
GP2118-1500 #2	0.006	2.67E-06	0.061
GP2118-1500 #3	9.480	0.004	-0.478
GP2118-1550 #1	5.170	0.002	-0.694
GP2118-1550 #2	41.900	0.019	-0.649
GP2118-1550 #3	4.990	0.002	-0.182

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 started with corrosion test. Since the samples prepared for corrosion test are inconsistent with respect to the use of epoxy cover, new samples are required and the corrosion test must be redone.

(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 chemical composition of the three enamel powders will be tested.

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 according to coating thickness, coating roughness, coating efficiency and corrosion resistance.

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

A performance metric of corrosion tests for three types of enamels and their combination will continue to be executed. Microstructure/porosity and chemical adhesion will also be tested.