

CAAP Quarterly Report

Date of Report: *April 9, 2015*

Contract Number: *DTPH5614HCAP05*

Prepared for: *DOT and PHMSA*

Project Title: *Improved Coatings for Pipelines*

Prepared by: *Texas Engineering Experimental Station*

Contact Information: *Dr. H. -J. Sue, 1-979-845-5024, hjsue@tamu.edu*

For quarterly period ending: *March 31, 2015*

Business and Activity Section

1. Generated Commitments -

There have been no additional teaming agreements.

The formulation we have agreed to test has multiple components in addition to the resin and hardener. The manufacturers of most of the additives have agreed to provide free samples. We have received 5-gallon pails of two different epoxy resins (D.E.R. 354 and 383) and a hardener (D.E.H. 615) from Dow Chemical Co. has shipped resin and one of the two hardeners. We have also purchased other components at a cost of ~\$100. Our partners, Shawcor and Dow Chemical Co. have submitted expense reports for this quarter. These are attached at the end of this report.

We are starting to design an automated spraying machine that will allow us to prepare coatings with uniform and predicable thicknesses. We're looking options that range from purchasing a complete system to repairing and modifying a 3D-printer that is available to us in Materials Science & Engineering.

We had a phone meeting with representatives from the DOT, Shawcor, and Dow Chemical on April 2nd, 2015. The notes are included in the Appendix.

2. Status Update of Past Quarter Activities -

Here is the list of plans from the previous quarterly report.

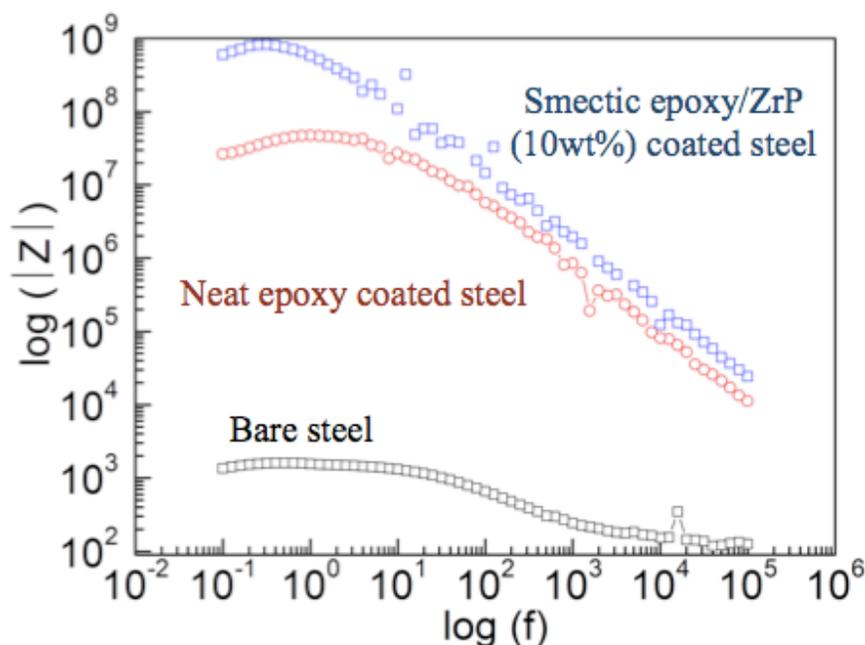
1. Our plans for this quarter were to scaleup our ZrP-epoxy formulation, and coat a portion of the panels.
2. Gather all of the components for the marine coating, locate a mixer capable of adequate mixing, and demonstrate that we can cure a sample with acceptable properties.
3. If the above activities go smoothly, coat our first set of steel panels along with the control panels and ship them to Shawcor.

These were accomplished, although we chose a different route for item #2. Rather than use the complex Dow formulation with 7 components and a complex mixing procedure, we chose to test coated panels provided by Shawcor. Sufficient ZrP-epoxy formulation was prepared to coat 4 panels. Some testing was performed at Texas A&M and Shawcor. We will defer testing the complex filled coating until we gain confidence in our coating procedure.

An acetone suspension of exfoliated ZrP using Jeffamine M1000 surfactant was combined with an epoxy resin (bisphenol F diglycidyl ether) and a curing agent (diethyl toluenediamine). This suspension was used to coat 4 steel panels that were precoated by Shawcor with a standard epoxy coating. A coating thickness of 35 microns was achieved after curing the panels at 200 °C.

One of the panels was tested by electrochemical impedance spectroscopy (EIS) using a saturated calomel electrode as reference. The results are shown in Figure 1 below. A bare steel panel and a panel coated with only the neat epoxy (no ZrP) were used as controls. Dennis Wong (Shawcor) said that the results were promising, but we needed a greater exposure time.

Figure 1: Electrochemical Impedance Spectroscopy (EIS)



Concurrently, a 28-day cathodic disbondment test was conducted at Shawcor. After 28 days at 65 °C, a disbondment of 10.1 mm resulted for the ZrP-epoxy panel, and 17.3 mm for the control panel. Dennis said that the results were “excellent” but that we will need to test more panels for the results to be conclusive. *We will follow up with a meeting (or email exchange) to discuss the details.*

In our follow-up meeting (see Appendix), Dennis said that our cure temperature (200 °C) is too high for practical application. Although we may be able to cure the existing formulation at a lower temperature, *we agreed to develop a new formulation that cures at 100 °C (ideally).* This will be a challenge, but we will run some tests. Dennis also pointed out (and Dow agreed) that we will eventually need to find a solvent-free formulation.

Figure 2: Cathodic Disbondment



3. Description of any Problems/Challenges -

In our previous report we mentioned the possibility of two problems (first two in the list below).

1. Scaleup of the ZrP nanofiller. So far this has not been a problem but we will need to work on scaleup procedures as we coat an increasing number of panels for testing.
2. Adhesion between the two coating layers. We saw no evidence of failure between layers during our tests.
3. In order to gather reproducible data on our coatings, we need to be able to minimize the thickness variation within one panel and also panel-to-panel. We are investigating the possibility of developing a 'robot-sprayer' to address this problem.

4. Planned Activities for the Next Quarter -

1. Shawcor will provide more panels for us to coat. We will run the EIS tests, and ship samples to back to Shawcor for them to run extended period testing.
2. We will work to develop formulations that can be cured at lower temperatures.
3. We are working on a 'robot-sprayer' that will improve coating thickness variation compared to our current manual process.

Appendix

Meeting notes for DTPH5614HCAP05 (Improved Coating for Pipelines)

02-Apr-2015 at Texas A&M in College Station, TX

Phone Attendees:

- DOT - PHMSA: Jay Prothro
- Dow Chemical Co.: Rajesh Turakhia, Lingyun He, Yong Zhang
- Shawcor Ltd: Dennis Wong
- Texas A&M: Prof. Hung-Jue Sue, Fan Lei, Peng Li, Michael Mullins

Presentations:

- Mullins gave a summary of the contract deadlines: “2015-04-02 DOT pipeline meeting MJM.pdf”
- Peng Li gave a presentation on technical progress: “DOT-Peng Li 2015-04-02.pdf”
- Dennis Wong sent a data slide “TAMU-28 days from D Wong Shawcor.pdf” that was discussed during the Peng Li presentation.

Notes (items in italics are action items):

- Peng described the preparation of the ZrP coating formulation based on bisphenol F diglycidyl ether and diethyl toluenediamine, and the panel coating process. A coating thickness of 35 microns was achieved after curing the panels at 200 °C.
- One of the panels was tested by electrochemical impedance spectroscopy (EIS) using a saturated calomel electrode as reference. A bare steel panel and a panel coated with only the neat epoxy (no ZrP) were used as controls. Dennis Wong (Shawcor) said that the results were promising, but we needed a greater exposure time.
- Dennis discussed the 28-day cathodic disbondment results that were conducted at Shawcor. Dennis said that the results were “excellent” but that we will need to test more panels for the results to be conclusive. *We will follow up with a meeting (or email exchange) to discuss the details.*
- Dennis said that our cure temperature (200 °C) is too high for practical application. Although we may be able to cure the existing formulation at a lower temperature, *we agreed to develop a new formulation that cures at 100 °C (ideally).* This will be a challenge, but we will run some tests.
- Dennis also pointed out (and Dow agreed) that we will eventually need to find a solvent-free formulation.
- We discussed the timing (end of June or early July) and location of the next meeting. *Dennis Wong will investigate the possibility of having it at the Shawcor site in Toronto.*

March 31, 2015

Dr. Hung-Jue Sue
Texas A&M University
College Station, TX 77843-3123
+1 979 845 5024

RE: 2nd quarter industrial support for DOT pipeline project DTPH5614HCAP05

Dear Dr. Sue:

Our 2nd quarter support for the quarter for staff time, expenses, and materials is \$4779. A breakdown of this total is shown below.

Project Activity	Contributed Cost in \$
Staff time for coating formulation, testing, evaluation, meetings	3625
Materials, sample preparation, consulting	
Travel expenses	1154.09
Total	4779.09

Sincerely,



Dennis Wong, PhD, P Eng
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+1 416 744 5807
dwong@shawcor.com



The Dow Chemical Company
2301 Brazosport Blvd.
Freeport, TX 77541-3257
U.S.A.

DATE: 4/7/2015

Dr. Hung-Jue Sue
Texas A&M University
College Station, TX 77843-3123
+1 979 845 5024

RE: 2nd quarter industrial support for DOT pipeline project DTPH5614HCAP05

Dear Dr. Sue:

Our 2nd quarter support for the quarter for staff time, expenses, and materials is \$3835.89. A breakdown of this total is shown below.

Project Activity	Contributed Cost in \$
Staff time for coating formulation, testing, evaluation, meetings	1112.24
Laboratory overhead and admin. services expense	2680.32
Materials, sample preparation, consulting	0.00
Travel expenses	43.33
Total	3835.89

Sincerely,

Lingyun He

PERSONAL AND CONFIDENTIAL



WORLDWIDE PARTNER