CAAP Quarterly Report

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Contract Number: DTPH56-15-H-CAP01

Prepared for: *DOT, PHMSA, and Partners*

Project Title: *Corrosion Under Insulation (CUI): Innovative Solutions to Cold Climate Corrosion Challenges*

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For quarterly period ending: *September 30, 2016*

Business and Activity Section

(a) Generated Commitments - No changes in agreements with partners or cofounders. Itemized purchases as follows:

- Foam crevice cells - $435.00/each, $2,175.00 total
  - Five (5) foam crevice glass cells ordered to conduct the crevice cell experiments.
- Thermocouples - $34.00/each, $204.00 total
  - Six (6) thermocouples for temperature control of the foam crevice cell experiments.
- Flexible heaters - $34.50/each, $414.00 total
  - Twelve (12) flexible heaters to heat and maintain the temperature of the foam crevice cell experiments.
- Electric float switch - $21.00/each, $105.00 total
  - Five (5) electric float switches to be connected in series with a peristaltic pumps to maintain the fluid levels in the crevice cells during the experiment.
- Rubber o-rings - $15.95 total
  - Small rubber o-rings are used to maintain the position of the coupons in the 1-L cells on the glass rod. Medium rubber o-rings are used to seal the liquid around the metal in the foam crevice cell.
- Insulation cutting knife - $10.04
  - Insulation cutting knife used to piece out the legacy insulation and create foam slurry mixture.
- Individual air pumps - $12.77/each, $76.62 total
  - Six (6) individual air pumps to continuously supply oxygen to the 1-L cell experiments.
- Super glue and Pyrex “casserole” dish - $9.48
  - Additional Pyrex dishware and super glue to place over temperature controller and protect from moisture condensation or leaks.

(b) Status Update of Past Quarter Activities - All tasks reported below funded by PHMSA.

- Foam slurry solution prepared.
  - Approximately ten (10) liters of foam slurry mixture were prepared in accordance with ASTM C871 – Standard Test Methods for Chemical Analysis of Thermal Insulation Materials for Leachable Chloride, Fluoride, Silicate, and Sodium Ions. The pH of the solution was measured to be approximately 6.90.

- Crevice cell apparatus design, construction, and testing.
  - The Foam Crevice Cell apparatus design has been finalized for fluid level and temperature control. The crevice cell was plugged on both sides with foam pieces which were backed by solid epoxy. One of the pieces had a small hole drilled through it to allow the thermocouple temperature controller to measure the temperature of the solution. This hole was sealed with a flexible silicon sealant. See Figure 1.

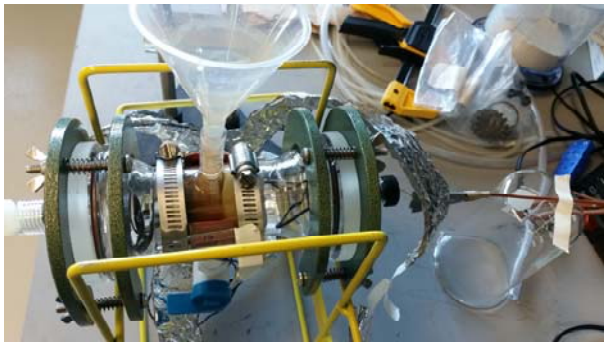

Figure 1: Finalized crevice cell design.

- Crevice cell parts ordered.
  - After finalizing the design, more parts were ordered to conduct the tests shown in the experimental matrix in a timely manner.

- Experiments conducted with 1-liter cells.
  - Iterations of the 1-liter cell experiments were conducted and are still in progress. See Table 1 and highlighted portions. Green highlight signifies finished tests, yellow indicates in progress, and no highlight shows tests yet to be conducted.

Table 1: Experimental Matrix

Test #	Temp. (°F)	Electrolyte Composition	Cell	Test #	Temp. (°F)	Electrolyte Composition	Cell
1	140	Seawater	1L cell	**13**	140	Seawater	Foam crevice cell
2	160	Seawater	1L cell	**14**	160	Seawater	Foam crevice cell
3	180	Seawater	1L cell	**15**	180	Seawater	Foam crevice cell
4	140	Foam slurry[1]	1L cell	**16**	140	Foam slurry[1]	Foam crevice cell
5	160	Foam slurry[1]	1L cell	**17**	160	Foam slurry[1]	Foam crevice cell
6	180	Foam slurry[1]	1L cell	**18**	180	Foam slurry[1]	Foam crevice cell
7	140	SWSNaB[2]	1L cell	**19**	140	SWSNaB[2]	Foam crevice cell
8	160	SWSNaB[2]	1L cell	**20**	160	SWSNaB[2]	Foam crevice cell
9	180	SWSNaB[2]	1L cell	**21**	180	SWSNaB[2]	Foam crevice cell
10	140	FSSNaB[3]	1L cell	**22**	140	FSSNaB[3]	Foam crevice cell
11	160	FSSNaB[3]	1L cell	**23**	160	FSSNaB[3]	Foam crevice cell
12	180	FSSNaB[3]	1L cell	**24**	180	FSSNaB[3]	Foam crevice cell

[1] Foam slurries will be prepared using an adaptation of the method described in ASTM C871. Foam slurries will also be prepared using Seawater.

[2] SWSNaB: Seawater-saturated sodium bentonite

[3] FSSNaB: Foam slurry-saturated sodium bentonite

(c) Description of any Problems/Challenges -

- Additional foam plug molds.
  - During a test of the crevice cell, leaks were detected in the foam plugs. Evaluation revealed that the plugs and rubber O-rings were not forming a tight enough seal to the glass. New molds were 3D printed, the insides were coated with paraffin wax, and the foam was poured. The new foam plugs form a much tighter seal and no leaks have been detected. The new foam plugs were subjected to heat shrinking when the heaters were heating the solution up. This caused leaks to occur.

    - The foam plugs were then backed by a solid epoxy which was used to create the custom LPR probes. These new foam plugs work much better and maintain the tight seal through heating and cooling cycles.
- 1-liter cell experimental design and construction.
    - The oxygen replenishment system initial balancing efforts were difficult due to the changing levels of liquid in the 1-L cells. To fix this issue, individual air pumps were procured to maintain individual oxygen replenishment in each cell.

(d) Planned Activities for the Next Quarter -

- Apparatus design and construction.
    - The additional foam crevice cell devices will be constructed and will be tested in a 15 day exposure experiment.
- Foam Crevice Cell testing.
    - Experiments will be conducted for 15 days of exposure using the crevice cells and the corrosion solutions.
    - Further experiments will be conducted for longer exposure periods if the corrosion rates are not measurable with a reasonable degree of uncertainty.
- Additional 1-Liter Cell testing.
    - An additional experiment will be conducted for 15 days of exposure using 1-Liter cells and the seawater-saturated sodium bentonite solution. The initial seawater-saturated sodium bentonite tests did not show significant differences from the seawater only solution. The sodium bentonite content will be increased to see what effects it has on the corrosion system.
    - The foam slurry-saturated bentonite solution was prepared with a large amount of bentonite and the results will be analyzed once the test is finished to determine if the amount of sodium bentonite had a significant effect on the corrosion system.
- Metal sample preparation continuation if necessary.
    - If necessary, additional metal sample surfaces will be finished per applicable ASTM standards and references.

Injector prototype and testing report

Prepared by: Hayden Maxwell (Undergraduate Research Assistant)

Introduction

For this project, I was tasked with prototyping an injection device to coat the inner surface of a simulated weld-pack with a bentonite powder and isopropanol solution. The bentonite solution was found to be effective at reducing the rate and severity of corrosion at the pipe's surface, and thus an apparatus was needed to apply it under the simulated weld-pack's foam insulation. My part of the project consisted of manufacturing the simulated weld-packs and designing a prototype injector and injection process that could effectively coat the surface of the inner pipe of the weld-pack.

Part I: Simulated Weld-Packs

The simulated weld packs were constructed regularly onsite out of a steel pipe and a pourable, two-part polyurethane foam. The foam was formed in a custom mold. The mold consisted of an inner steel pipe, an outer tube, two plywood end caps with gaskets, and five all-thread bars with nuts and washers. See Figure 2.

Figure 2: This image displays from left to right the mold's outer tube, inner pipe and all-thread rods on the lower end cap, and the upper end cap.

For the inner pipe, a two-foot-long piece of 3 inch, schedule 40, ASTM 53, black pipe was utilized. The outer tube was a generic 12 inch, two-foot-long, cardboard concrete form tube. Lastly, the plywood endcaps were built from 1-inch-thick and sixteen-inch-long plywood squares. Each square was fitted with an 11.5-inch diameter plywood insert to secure the form tube and inner pipe. Two holes were added to one endcap allowing for a vent and pouring hole. Finally, five small holes were drilled in each endcap for the five all-thread rods. The large gaskets were cut from a PVC shower underlay mat. Refer to Figure 1.

Before pouring the foam, the mold was prepared with tape and grease. The wooden endcaps were taped, one with aluminum tape and the other with Gorilla tape. Both tapes were found to be successful at helping prevent bonding of the foam to the plywood, but the Gorilla tape was found to be more workable and also more financially conservative. The edges of the concrete form tubes were also taped with Gorilla tape to help increase the durability of the tube.

After taping, the mold was lubricated to assist with the ease of the mold removal after the foam had been added and cured. Specifically, the end caps and the outer tube's interior were lubricated to help prevent foam bondage. The inner pipe was not lubricated. For lubrication, standard automotive grease was found to work very well in preventing the foam from bonding to the end caps and outer tube. However, automotive grease was quite messy and fairly time intensive to apply. Thus, several spray lubricants were tested. WD-40 Gel Spray was used and found to work adequately. Blaster's Silicon Spray was also used and found to work but less effectively. After lubrication, the mold was assembled and secured with the all-thread rods.

The mold was then filled with foam. Forty-eight fluid ounces of both parts of the foam (for a total of 96 fluid ounces of mixture) were poured into a disposable plastic container and thoroughly mixed for 30 seconds with an electric drill. The mixture was then poured into the mold, and the mold was quickly capped. The mold was filled while standing vertically but laid horizontally while the foam expanded. The mold was rotated while horizontal so that the vent hole was located as far up as possible allowing for the maximum amount of air to be released from the mold. See Figure 3.

Figure 3: This figure shows the fully assembled weld-pack mold with vent located at its highest position.

The vent hole was plugged after the foam began to exit the vent hole. The foam was allowed to cure for about an hour or until the foam was hard before the mold was removed. The endcaps were pulled off by hand. The concrete form was removed by placing two clamps on the edges of the outer tube and then pulling up on the clamps by hand while pushing the inner pipe down with one's foot.

After the foam and pipe core (now referred to as the simulated weld-pack) had been removed, the mold was cleaned of any foam residue, re-lubricated where necessary, and prepped with a new inner pipe.

Part II: The Injector and Injection

The prototype injector was fabricated from basic lumber parts and utilized a large syringe as the actual pumping apparatus. See Figure 4.

Figure 4: This image shows the finalized injector

The prototype featured a long handle to achieve a significant mechanical advantage, a fluid storage tank to reduce the need for refills, and a pressure gauge to monitor the applied force. The fluid storage tank was built from a number 1 plastic (polyethylene terephthalate) bottle as other clear plastics failed via crazing in the presence of concentrated isopropanol. The original prototype featured threaded valves and compression joints in the tube assembly, but these were replaced with ball valves and quick connect joints for ease of use. The injector head was built with a custom fluid sealing device and mounting plate. See Figure 5.

Figure 5: This picture displays the injector head which featured a mounting plate and circular steel cup to prevent backflow of the fluid.

The large plate provided a means to strap the quill to the weld-pack while the circular cup prevented most of the injected fluid from seeping back out the injection hole. The whole piece was connected to the quill by an adjustable compression fitting.

The injection process occurred in several steps. To begin, a hole was bored at the injection site with the quill. The quill was inserted into the foam up to the inner pipe's surface and then removed several times while twisting the quill to achieve a clear a hole as possible. Any foam swallowed by the quill was removed before reinserting the quill. After a hole had been cleared, the quill's effective length was reduced by adjusting the compression fitting so that the quill was between one to two inches away from the inner pipe's surface. The injector head was then fixed to the weld-pack with two ratchet straps as depicted in Figure 6.

Figure 6: This image shows the simulated weld-pack prepared for injection.

Finally, the fluid in the storage tank was thoroughly mixed immediately before injection to maximize the amount of bentonite powder suspended in the fluid. The fluid was injected at under 60 psig as higher pressures tended to rupture the foam. Fluid was injected until it was observed seeping out of the pipe/foam interface. This usually occurred after 100-150 mL fluid had been injected. After fluid began to seep out, the injection head was removed, and the foam was removed from the pipe core. The foam was removed by first making two opposite side cuts in the foam along the pipe's axis with a reciprocating saw. See Figure 7. Care was taken not to score the inner pipe during this process. Then the saw's cuts were then brought to the inner pipe's surface by carefully running a large knife down the cut lines.

Figure 7: After the injection, the foam is removed from the inner pipe by first making two axial cuts along the pipe.

Next, the weld-pack was placed vertically on a small elevated base such that the foam rested on the base, and the inner pipe was free floating about an inch above the ground. See Figure 8. In most tests, some edges of the foam required trimming so that the weld-pack would sit level and stable on the base. Finally, a sacrificial board was placed on the top side of the inner pipe, and the board was beaten with a sledge hammer until the inner pipe broke free of the foam. The result was two foam clamshell halves and the separate inner pipe. In most of the tests, there was good surface coverage by the alcohol both radially and longitudinally often exceeding 100 square inches. The distribution of the bentonite powder was difficult to determine as whatever deposits were left behind were not observable visibly. Samples of each test were kept, and it was found that the free expansion density of the foam was about 3 lb/ft^3 while the density using 48 fluid ounces of each part of the foam was about 5 lb/ft^3. Observation under a stereo microscope revealed that the different densities of foam directly affected the foam's cell size as may be expected. The denser foam showed a smaller cell size while the lighter foam had a larger cell size.

Part III: Bentonite Solution

Alcohol was chosen as the medium in which to transport the bentonite powder as it has a high vapor pressure, low toxicity, low cost, and did not accelerate the corrosion process. The advantage of a high vapor pressure pertained to the fact that the alcohol should completely evaporate several days after the injection leaving just the bentonite powder. A problem that was found with the alcohol, though, was its inability to suspend much of the bentonite powder. The lack of solubility lead to clogs in the injector and scant deposits of bentonite on the inner pipe's surface.

To resolve this problem, a surfactant of the company name Triton X-100 was tested as an additive to the solution to help increase the amount of suspended bentonite. While there was some success in the surfactant's ability to increase the miscibility of the bentonite and alcohol, it was ultimately not used for primarily for two reasons. First, a relatively large amount of surfactant was needed to see a significant solubility increase; approximately a 30% by volume concentration of Triton X-100 was needed to obtain a noticeable visual increase in solution cloudiness. Secondly, it was unclear how the surfactant would affect the corrosion process. While not overtly corrosive, it was

unknown how it would act long term in the high temperature and chloride rich environment of an oil pipe's insulation.

Figure 7: The scored weld-pack is placed on an elevated base with a sacrificial board on top.

Active mixing of the fluid storage tank during the injection process was also tested in an effort to maintain the solution's homogeneity. This process achieved minor success as it helped prevent clogging and did somewhat increase the injected solution's cloudiness. To inject with active mixing, though, did require two individuals making the process less feasible.

Conclusion

In this project, an apparatus and process were designed and tested to coat the inner surface of a simulated weld pack with a bentonite solution. A custom hand-driven pump was built and utilized as an injector for the bentonite solution, and practical method for injecting the solution was developed.

While substantial progress was made in both areas, much work is still left to be done. The injector apparatus was formed cheaply and prone to clogging. Additionally, no reliable method was developed to test quantitatively for the presence of bentonite on the inner pipe's surface after injection. Therefore, it is recommended that further research be done to test the effectiveness of the injection process. Specifically, it should be researched how much bentonite is required to effectively reduce the corrosion process and how the injector and bentonite solution can be modified to reliably provide at least the required amount of bentonite. Answers to these questions would be a practical next step in the prevention corrosion under insulation.