CAAP Annual Report

Date of Report: *September. 29, 2017*

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Prepared for: *U.S. DOT Pipeline and Hazardous Materials Safety Administration*

Project Title: Development of New Multifunctional Composite Coatings for Preventing and Mitigating Internal Pipeline Corrosion

Prepared by: *North Dakota State University*

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For quarterly period ending: *September. 29, 2017*

Business and Activity Section

(a) Generated Commitments

No changes to the existing agreement

Purchase made for nano-materials over this reporting period

One undergraduate student is hired for participating in this project over this reporting period

(b) Status Update of Past Quarter Activities

The research activities in the annual report include the summary of literature review, experimental tests and data process for new coatings as summarized below.

1. Introduction and the scope of the work

High-performance anti-corrosion coatings have been gaining increasing attention in oil and gas pipeline application in recent years. Abundant oil & gas reserves and petrochemical resources in the U.S. are expected to boost demand for high-performance anticorrosion coatings for prevention and mitigation of pipeline corrosion. The proposed new multifunctional composite coatings are focusing on the enhancement of three most relevant properties to meet the current urgent need.

To developing new high-performance coatings, literature study has been performed in this report. To achieve desirable multi-functionality in the composite coatings, this project will mainly focus in two directions: screen polymers for primer materials and synthesize the Nano-modified coatings. In addition, new nano-modifed composites are expected to have some superior properties which offer the opportunities to fabricate new high-performance composites.

In this report, the experimental study was focused on the following subjects:

- First, the influence of different dispersion methods in nanocomposites.
- Second, the influence of adding nanomaterials into epoxy resin with various concentration.
- Third, in order to find suitable polymer resins, several baseline coatings have been characterized during the polymer screening process.

2. Literature review

A literature review [1-31] covers most of the USDOT and pipeline industral project reports, transportation agency and research publications, various studies for pipeline corrosion mitigation and prevention. The protective coatings for internal corrosion control have a wide variety of types, including single or dual layer fusion bonded epoxy coatings (FBE), coal tar, asphalt enamels, Epoxy Resin lining, Polyurethane/Polypropylene (PE/PP) lining, and other fiber reinforced polymer (FRP) linings. Besides that, more new high-performance coatings have been developed in recent years. In the review, particular attentions are also paid on advanced high-performance coatings, including types of durability, ease in installation, and applicability. Clearly, although high performance anti-corrosion coatings offer promising opportunities, the integration of high performance anti-corrosion coatings into oil and gas pipelines applications still face several challenges. Thus, the information is an essential step toward the effectiveness and functionality of the proposed coating for pipeline corrosion monitoring.

3. Experiment design

This study is to present the work on coating synthesis and characterization for determining the quality of the coatings, including corrosion resistance test, abrasion resistance test and antifouling test.

3.1 Formulation of nano-modified coating

As identified in the previous report, the nanoparticles could be great candidates to develop new high-performance coatings. In this stage, we aim to formulate and characterize nano-modified coatings. Note that conventional epoxy is used as the prime. It is mainly because to select the epoxy instead of other high-performance polymer prime is to gain better understanding of nano materials and their performance in a coating. The another line of the study is overlapped to explore the high-performance polymer primes reinforced with the nano-materials on the basis of these findings and will be docemented in the following reports.

3.1.1 Sample preparations and dispersion

Two dispersion methods have been carried out to find out how to enhance the dispersion and alignment of nanoplatelets in an epoxy matrix. Ultrasonication was used in both methods and solvent was also evaporated before mixing with a curing agent. Fig.1 (a) is showing a sample without ultrasonication. For the samples without evaporation of the solvent before mixing with a curing agent, an example is shown in Fig.1 (b).

Figure 1 Sample without ultrasonication (a) without evaporation of solvent (b)

In the first method, the nanoplatelets were dispersed into a mixture that containing EPON 828 and 20 wt.% of xylenes. Ultrasonication was applied in the dispersion process at 100% amplitude with a duration of 60 mins. Misonic S1805 sonicator with a 3/4" probe was used in this process like shown in **Error! Reference source not found.**.

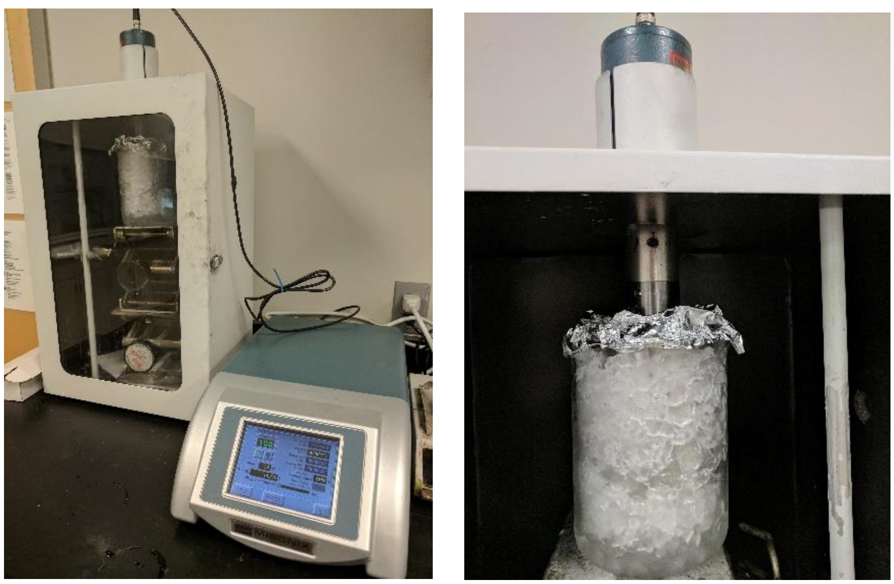


Figure 2 Ultrasonicate nanoplatelets with ice bath

High-speed disk (HSD) dispersers (high-speed impellers, high-intensity mixers) was used in the second method to break down the particles by providing shear stress during the dispersion process, as shown in ***Error! Reference source not found.***. All test samples were illustrated in Fig. 4.

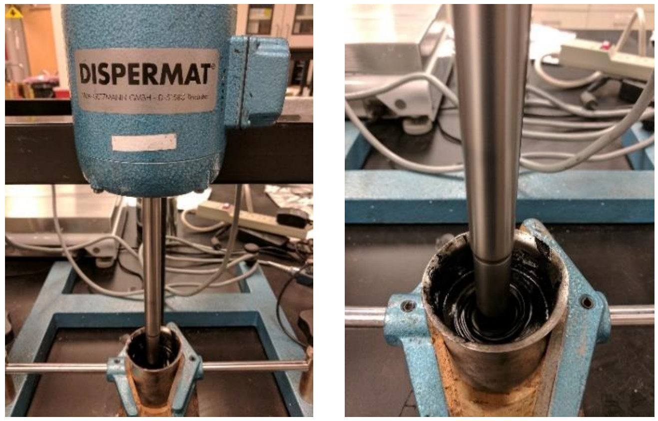


Figure 3 High-speed disk (HSD) dispersers

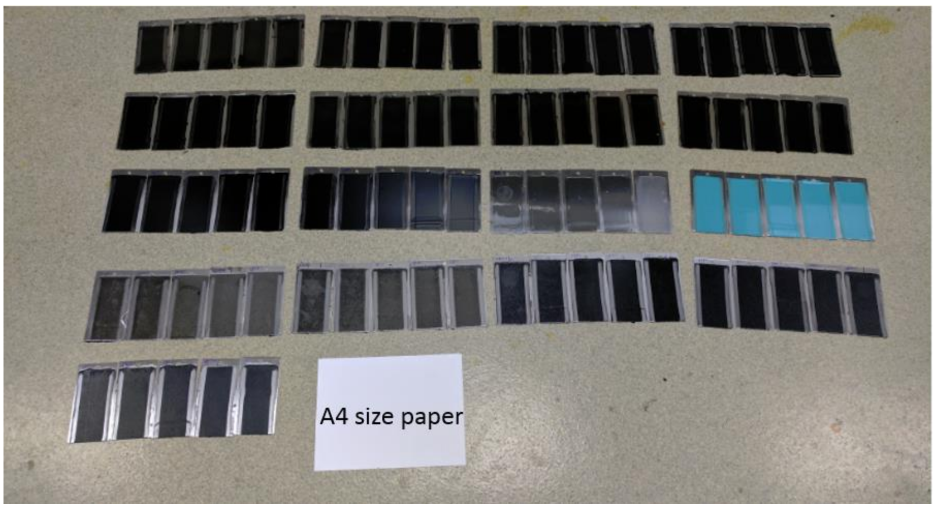


Figure 4 Coated test panels

3.2 Characterization of nano-modified coating

For a better understanding coating performance, the corrosion resistance behaviors of the coating were evaluated at various periods of time using EIS tests under Salt Fog test (ASTM B117). Potentiostatic EIS test was planned to be performed on each sample before the salt fog test, 24 hrs., 200 hrs. and 500 hrs. after the salt fog test.

3.2.1. EIS test

As discussed in the second quarterly report, EIS test is used to evaluate the interface properties between a substrate and conductive electrolyte solution which the applications can be coating performance evaluation.

3.2.2. Salt Fog Corrosion Test

The ASTM B117, Salt Fog test, is an accelerated corrosion test which the samples are exposed to a corrosive environment in a fog chamber. The samples are required to be maintained in a salt fog test environment during the test, and the salt solution should be 5% NaCl solution with a PH ranged in 6.5 to 7.2. The temperature should be maintained at 35°C (95°F).

Figure 5 Scribing coated sample with engraving machine

The test specimens should be suitably cleaned before placing into the chamber. Base on the ASTM B1654, to determine the development of corrosion from an abraded area in the coating, two scribed lines shall be made through the coating like showing in Fig. 5. To protect the uncoated metallic surface in the panel, we have used 3M™ Polyester Tape 8992L to cover these areas and the back of the panel. Sufficient pressure should be being applied by thumb and wood stick (Fig. 6) to make sure the tape is well-glued to the panel. Otherwise, the salt solution will be able to get into the uncoated area during the test. For the area without scribed lines, EIS test can be applied to evaluate the performance of the coating under the corrosive environment (Fig. 7).

The salt fog chamber and tested samples are shown in Fig. 8. After exposure, the surface of the coated sample should be cleaned by following ASTM B1654. All the corrosion products should be removed along scribe lines by either scraping, knife, paint stripper, air blow-off or power washer. Then the sample can be evaluated based on the defected areas by following the rating method from B1654.

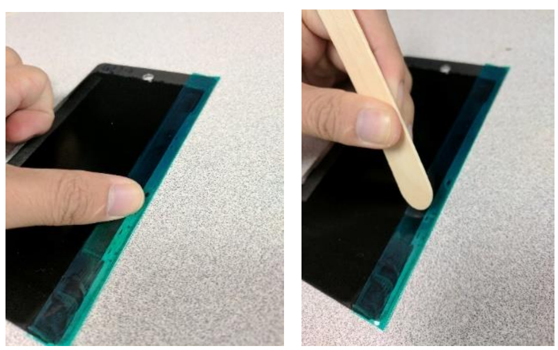


Figure 6 Prepared test sample for Salt Fog test

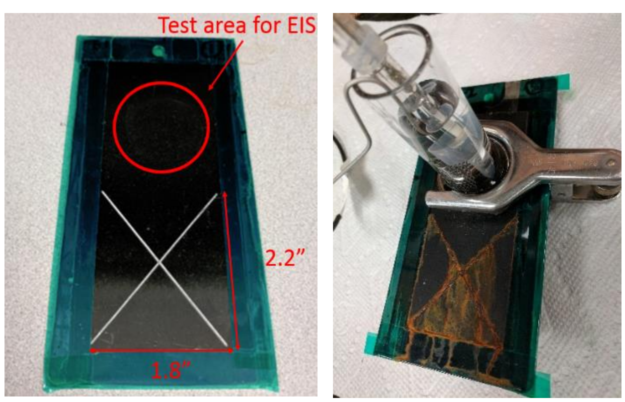


Figure 7 Incorporating EIS test on a prepared sample

Figure 8 Salt Fog Chamber with tested samples

3.3 Mechanical characterization of the coating

Various mechanical tests of the coating are also used to characterize their properties and gain a better understanding of their performance under simulated scenarios.

3.3.1. Adhesive bonding strength: tensile button testing

The adhesive bonds of the coating to metal were characterized by tensile button testing. The purpose of this test is to evaluate the tensile bond strength of nano-reinforced epoxy. The test specimens were prepared to study the influences of incorporating different nanoparticles into polymer matrix on the adhesive bonding strength.

The sample preparation procedures are described in Figure 9, three dollies were glued to each specimen, and the pull-off strength was measured during the experiment. Scotch weld 460 (3M, Co.) was used to the glue to dollies, and the test area was abraded with sand paper (100 grit or finer) to enhance the bonding strength between dollies and tested coatings. The test was performed 24 hours after the dollies were applied. The test area was isolated with other parts of the coating by die cutting. Adhesion tester was used to apply tension load normal to the test surface. The pull-off strength (adhesion) was measured when the dolly was detached by the tension load.

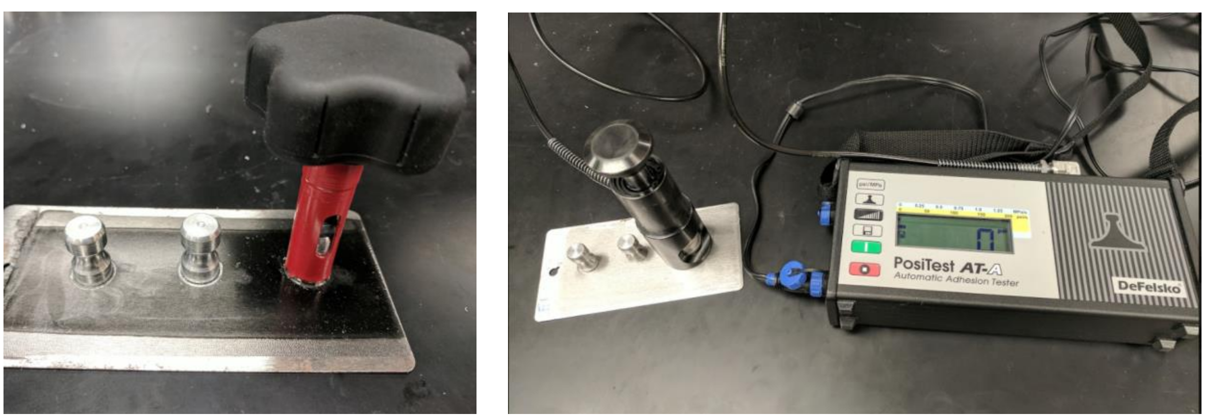


Figure 9 Adhesion test machine

3.3.2. Tensile Properties: dog bone tensile test

The determination of the tensile properties of the new coating was done by the dogbone tensile test (ASTM D638). The test was performed by Shimadzu's EZ-X tester (Figure 10) with a testing speed of 1mm/min. The hardened dog bone specimens were clamped at two ends by the grips during the test. Tensile strength was applied to elongate the specimens during the test until the specimens were broke in the narrow cross-sectional test section. Maximum tensile strength and Young's modulus were determined for each specimen.

Figure 10 Shimadzu's EZ-X tester with specimen at NDSU

3.3.3. Abrasion test methods

The accelerated test (ASTM G6) is used for determining the relative abrasion resistance of steel pipe coating. The test apparatus (from RAE Coatings Lab) for this test in the revolving drum. The drum, which containing nine coated pipe sample, will fill with a slurry of coarse abrasive and water during the test. Once the drum starts to rotate by the motor, the wet coarse abrasive will impact the coated surface will damage the coating. Even this test is specifically designed for the pipe coating. The coated surfaces are the exterior surface of the pipelines instead on the internal surface of the pipe. The mechanism of falling abrasive (ASTM D968) and Taber Abraser (ASTM D4060) methods are very similar as they both apply abrasion on a coated panel. Both test methods require the coating is applied at a uniform thickness on a rigid panel.

3.3.4. Fouling resistance test methods

There are several well-developed experimental apparatuses have been designed to study fouling in the lab. Even the fouling mechanism for the crude oil is much different and complicated compared with the fouling in transmission pipes; the test method might be modified in the future if necessary. Two test methods are discussed in this report: are hot wire method, closed liquid system microbomb. For the proposed new multifunctional composite coatings, like discussed earlier, the three main objectives are anti-corrosion, anti-fouling, and abrasion resistance. All these three properties must be tested for both baseline coating (existing commercial coatings) and developed nanomaterial-based composites. In this chapter, the methodology of characterizing baseline coating and nanomaterial-based composites will be discussed. The coated samples has been prepared om Q-panel standard steel panels. Falling Abrasive method has been used to the abrasion resistance as shown in Figure 11. For corrosion resistance, Potentiostatic EIS test has been performed to evaluate the protective coatings. Hydrophobicity can be used to determine the fouling resistance indirectly as the droplet contact angle shows the ability of a liquid to maintain contact with the coated surface.

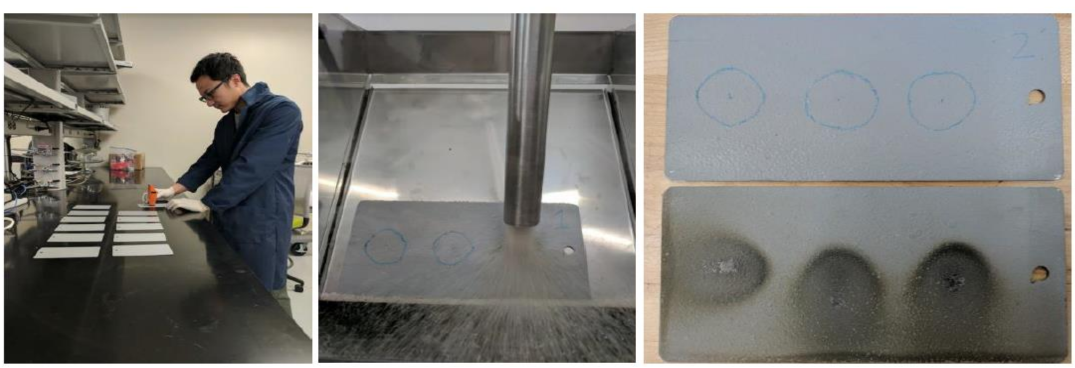


Figure 11 Falling abrasive test

4. Results:

4.1 Nano-reinfored composite coatings

4.1.1. Effects of the nanoparticle dosage on the coating performance

The impedance curves of graphene/epoxy composites which mixed by the second method are shown in Figs. 11 and 13. The nano-modified coatings exhibit higher impedance modulus over control samples at all time. After 200 hours of Salt Fog test, unlike the pure epoxy samples, most of the nano-modified coated samples only have slightly change.

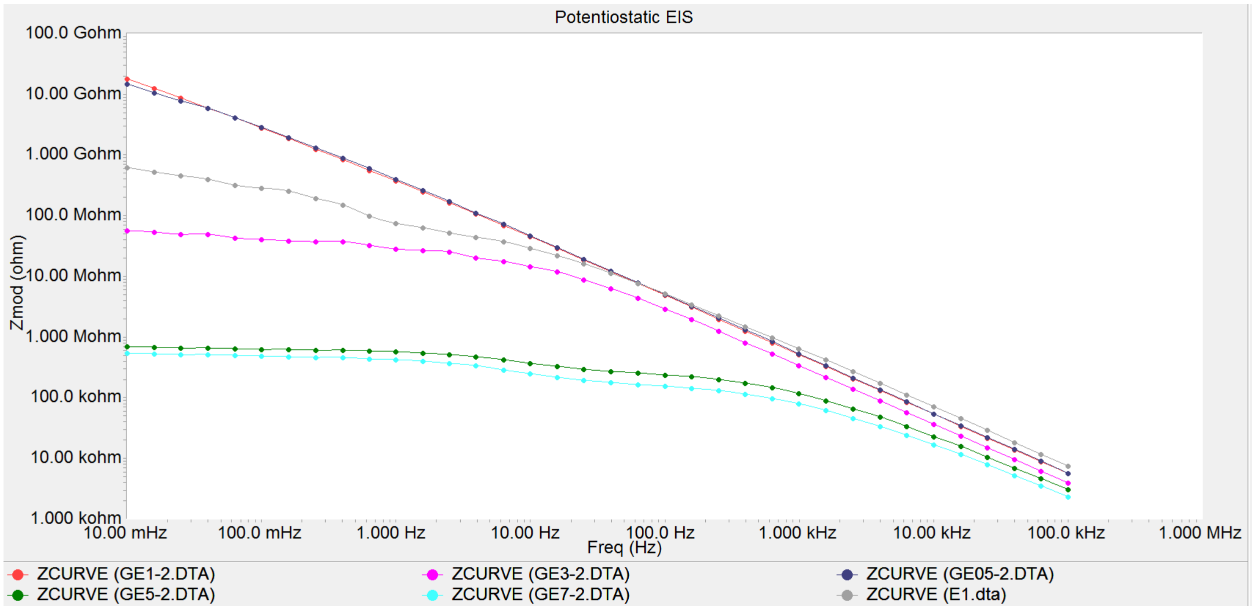


Figure 12 Initial impedance modulus for the coating (dispersion method 2)

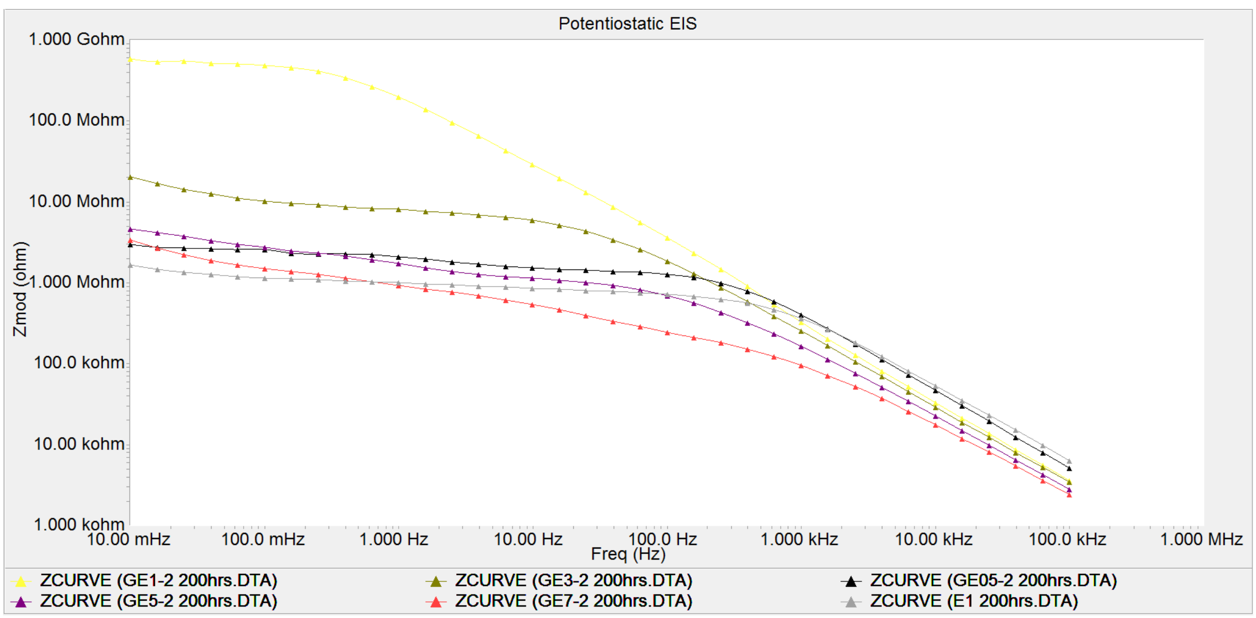


Figure 13 Impedance modulus for the coating after 200 hours Salt Fog test (dispersion method 2)

4.1.2. Effects of the dispersion methods on the coating performance

In addition, it can be easily observed that the dispersion methods can significantly affect the performance of the coating. Fig. 14 shows the Zmod values were mixed by the method 1 and method 2. The plots show that the sample that mixed by the method 2 has significantly higher corrosion resistance than the sample that mixed by the method 1. The results indicate the method 2 (with HSD disperser) can disperse nanoparticles more effectively compare with method 1.

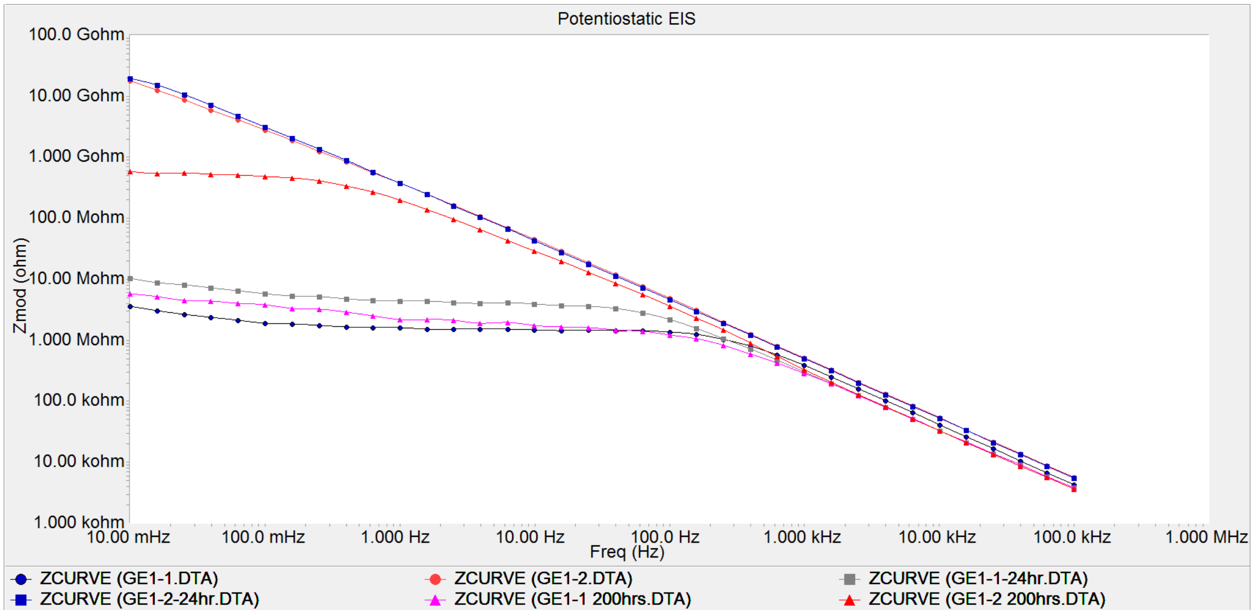


Figure 14 Impedance modulus of the coating with 1 wt.% of graphene under two methods

4.1.2. Effects of the abrasion resistance on the coating performance

The results of the experiments clearly indicate that with the reinforcement of carbon nanotube, the abrasion resistance of CNT/epoxy composites can be significantly increased. The results show a maximum increase with 1 wt.%, which the wear index has been decreased by 41%. In Fig. 15, the new coating can produce the maximum reinforcement in the epoxy matrix, as compared to the reference (without reinforcement).

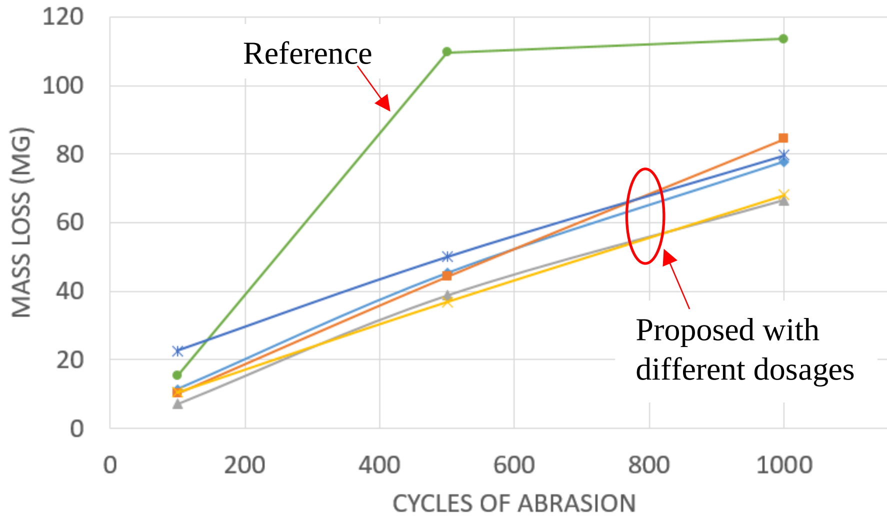


Figure 15 Mass loss of the specimens

Further study of the integration of two different nanoparticles reveal that the further improvement is observed in the coating performance.

4.1.3. Effects of the adhesive bonding strength on the coating performance

The pull-off strength (adhesion) was measured by following ASTM D4541 to evaluate the tensile bond strength of a Nano-reinforced epoxy with various CNT and graphene concentration. The measurements reveal that the adhesion generally decreases by incorporating one nanomaterial into the epoxy adhesive, which could be strengthened by integration with two nanoparticles. Figure 16 shows the thickness of the coating was barely reduced after the pull-off test. This indicates the adhesion between the coating and steel substrates is higher than the bonding strength of the glue between the dolly and the composite.

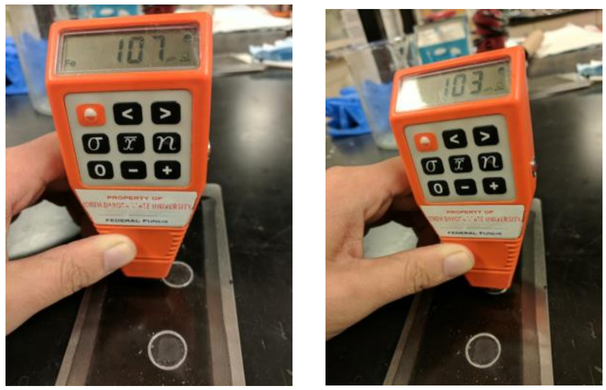


Figure 16 Thickness changes after adhesion test

4.1.4. Effects of the tensile strength on the coating performance

The tensile properties of CNT reinforced epoxy were determined by dog bone tensile test by following ASTM D638. The Nano-composites can be characterized by measuring maximum tensile stress and Young’s modulus in this test. The new coating’s Young’s modulus exhibits the increase in strength by 90%, as compared to the pure epoxy group.

4.2 Polymer screening

In this study, several polymer binders have been characterized by Taber Abraser method (ASTM D4060), Contact Angle Measurement (ASTM D7334), and Electrochemical Impedance Spectroscopy (EIS). With the obtained results, we can observe or hypothesize the abrasion resistance, fouling resistance, and corrosion resistance of the organic coatings, respectively.

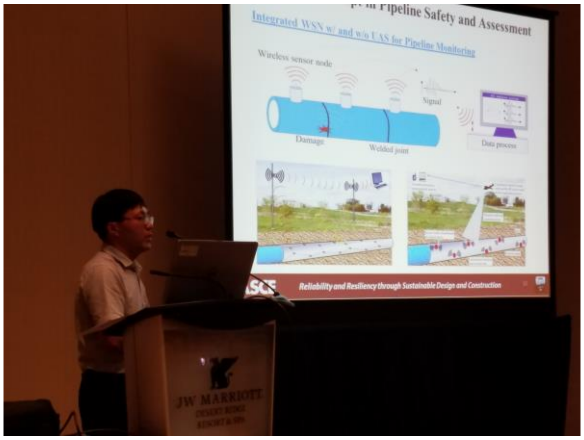
5. Dissemination of project results

5.1. National Conference Presentation

The PI Dr. Lin attended the national conference: American Society of Civil Engineers (ASCE) Pipeline 2017 at Phoenix, AZ, August 6-9 (see Fig. 17a), where he has chaired one conference session and also presented the group study related to this project (in his conference paper and presentation slides with acknowledgment to USDOT CAAP support). The presentation topic was entitled "Embedded wireless passive sensor networks for health monitoring of welded joints in onshore metallic pipelines". This work of the corrosion-induced damage near weldment is currently funded by ND DOC. This on-going project is focusing on applications in sensor systems and potentially leverage our current USDOT CAAP project by providing additionally thorough understanding on the coating performance near/at weldment and measurement techniques from sensor networks. In this trip, Dr. Lin also had a great experience in communication with different pipeline industry, from coating manufactures to pipeline corrosion technologies

5.2. Internationally Invited Talk

The PI Dr. Lin has several internationally invited talks in this summer by four international universities: China University of Mining and Technology, Beijing, China; Nanchang University, Nanchang, China; Nanchang Institute of Technology, Nanchang, China; and Jinggangshan University, Jian, China, for the dissemination of the latest research results through the seminars (see Fig. 17b). These activities not only could contribute to the public awareness of the coatings, oil and gas pipelines, but also educate next-generation civil engineers who will pursue this area in their future career.



(a) ASCE Pipeline conference session chair and presentation at Phonix, US. (Aug., 2017)

(b) Internationally invited talks at Jinggangshan University, China (June, 2017)

Figure 17 Dissemination of the research in the national conference and international invited talk

5.3. Undergraduate student and graduate student mentoring through this project

The PI Dr. Lin is mentoring two Ph.D. students (Xingyu Wang, and Mingli Wang) who are mainly taking charge of this project. The research received supported partially by Mr. Mohsen Azimi who graduated this summer, and visiting PhD student, Hong Pan. We actively involve undergraduate research assistants, including Matthew Pearson (senior student in Civil Engineering), Joe Baneck (senior student in Civil Engineeing) and Ganghyun Hyung (graduated this summer in 2017 and he tentatively plans to continue this work for the M.S. degree in spring 2018).

5.4. High school student outreach program

In this summer, two high school female students, Joelle Sherlock and Desiree Parsons, from North Dakota Governor's Schools program were invited to have five-week to experience the research in our research group. They worked with our graduate students for coating processing, including two different epoxy resin and curing agent pairs mixing, and on how to spray on steel plates, and evaluation of coating performance on environmental chamber using basic weight loss. During these weeks, students gained ideas of the corrosion behaviors of metallic materials and conduct experiments in the lab.

This outreach is be unique for us to disseminate our research and foster the next-generation engineers to gain better understanding of the science and material, and could motivate them to pursue this area in their future career.

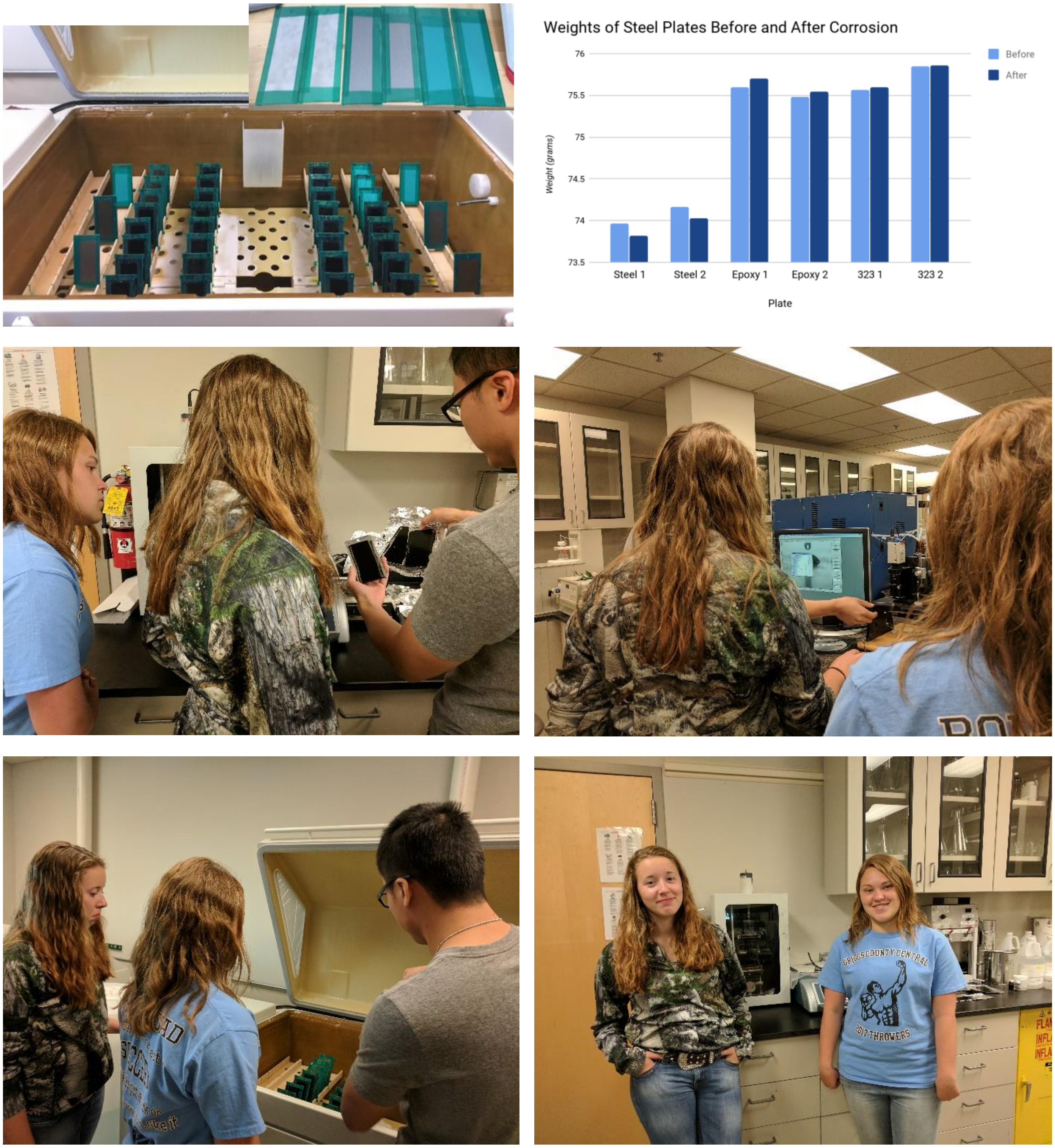


Figure 49 High school students participating in our research from North Dakota Governor's Schools

5.5 Curriculum enrichment.

The PI Dr. Lin is integrating the research progress in the courses CE425/625 "*Bridge Evaluation and Rehabilitation*" and Individual study CE 763 Durability of Civil Structures that are taught by Dr. Lin., which will foster students with knowledge and skills in corrosion mitigation, advanced coatings, and promote students interests in the fields of pipelines.

6. *Summary & conclusion*

In this report, a comprehensive experimental study of new coatings with nanomaterials and the polymer has been performed. Two dispersion methods have been carried out to find out how to enhance the dispersion and alignment of nanomaterials in the matrix.

The influence of incorporating nanomaterials into epoxy on abrasion resistance, fouling resistance, and corrosion resistance of the organic coatings were determined. Also, accelerated corrosion resistance test was performed to study the coating, and dog bone tensile test was used to characterize the tensile properties of the coating.

In the meantime, the methodology for characterize both baseline coatings and new nanocomposite coatings and synthesize new nanocomposite coatings has been discussed. The measurements of serval baseline coatings have been presented in the previous chapter.

The performance of each tested polymer binders is summarized. The number in each column indicates the rank of the polymers in various properties, which indicates their potentials for synthesizing the new high-performance coatings in the future study.

The fabrication of modification surface has also been developed in this study. It has been discussed that the surface tension of hexadecane is very close to the surface tension of petroleum liquids that will be considered to synthesis the new coatings.

7. *Future work*

In the future study, three main aspects will be focused on the next step:

- Experimental study on the influence of nanomaterials into other polymer systems.
- Meanwhile, more baseline coatings will be tested, and the results will be summarized in next quarterly report.
- Development of new modified surface will be continued for fabricating a durable coating system.

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