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7. PERFORMING ORGANIZATION NAMES AND ADDRESSES
University of Akron
Polsky Building Suite 284

Akron, OH 44325 -2102

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RPPR Final Report
as of 27-Dec-2018

Agency Code:

Proposal Number: 70020MSRIP **Agreement Number: W911NF-17-1-0265**
INVESTIGATOR(S):

Name: Ali Dhinojwala
Email: ali4@uakron.edu
Phone Number: 3309726246
Principal: N

Name: Mark Foster Ph.D.
Email: mfoster@uakron.edu
Phone Number: 3309725323
Principal: Y

Organization: **University of Akron**
Address: Polsky Building Suite 284, Akron, OH 443252102
Country: USA
DUNS Number: 045207552 EIN: 346002924

Report Date: 07-Aug-2018 Date Received: 19-Dec-2018
Final Report for Period Beginning 08-May-2017 and Ending 07-May-2018

Title: Acquisition of Atmospheric Pressure Plasma Jet System: A Practical Approach To Develop Functional Coatings
Begin Performance Period: 08-May-2017 **End Performance Period:** 07-May-2018
Report Term: 0-Other

Submitted By: Mark Foster Email: mfoster@uakron.edu
Phone: (330) 972-5323

Distribution Statement: 1-Approved for public release; distribution is unlimited.

STEM Degrees: 0 **STEM Participants:** 3

Major Goals: The overall objective was to acquire a robotic atmospheric plasma deposition system in support of DoD funded research on plasma polymerized films for use as corrosion protection coatings (FA7000-14-2-20016).

Accomplishments: 1.) Acquired, installed and commissioned a state-of-the-art robotic atmostpheric plasma system from SurfX.
2.) Identified a monomer (precursor), TMS, that yields films when deposited with atmospheric plasma polymerization that have a higher carbon content than atmospheric deposited pp-HMDSO.
3.) Improved the uniformity of the films we deposit with atmospheric plasma deposition by optimization of the movement pattern for the deposition head.
4.) Surveyed how film uniformity and deposition rate vary with sample to head distance.
5.) Verified that the monomer TMCTS, suggested to us by collaborators at ARL, provides a comparatively high deposition rate in atmospheric plasma polymerization deposition.
6.) Shown that an atmospheric plasma deposited film used as a primer coat under PU can substrantially improve corrosion resistance over short times.

Further details in the uploaded report of activities and achievements.

Training Opportunities: PhD graduate students Brenna Rossi and Yang Zhou learned how to properly operate the Surfx Atomflo atmospheric plasma deposition system and educated themselves on the topic of plasma polymerization through literature research for advancing their research projects. They both understand the machine and the principles on which it operates well enough to teach others how to use it.

Undergraduate researcher Reid Bailey learned how to operate the Surfx Atomflo during his short-term summer project as a participant of the REU program at The University of Akron. He is capable of using it properly without direct supervision.

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Results Dissemination: Dissemination of information about this purchase and research has been done by the PI, graduate students, and undergraduate researcher presenting posters and talks at various venues as well as speaking to individuals at conferences.

Prof. Foster presented a research update at the Technical Corrosion Consortium (TCC) Annual Review in August 2017 at the DOD & Allied Nations Corrosion Conference in Birmingham, AL. In November 2017 he attended the Plasma Processing Symposium at the MRS Meeting in Boston where he presented a talk. He spoke to multiple attendees about the acquisition of the SurfX instrument and atmospheric plasma polymerization, in particular
Prof. Elijah Thimsen (Washington University),
Dr. Chi-Chin Wu, U.S. Army Research Laboratory
Mohan Sankaran, Case Western Reserve University
Dr. Frank Papa, Genoa

Brenna Rossi presented a poster titled “Organic Atmospheric Plasma Polymerized Coatings” at the Technical Corrosion Consortium (TCC) Annual Review at the University of Southern Mississippi on August 1, 2018 and at a polymer science departmental poster session at The University of Akron at August 24, 2018. The poster is uploaded elsewhere. She has given periodic research updates to the Foster research group at the University of Akron since June 2018. Individuals she has spoken with about her research using the Surfx Atomflo 500 include but are not limited to: Andres Bujanda, Derek Demaree, and Jim Hirvonen from the Army Research Lab; Christine Sanders from the Navy Research Lab; graduate students from the University of Southern Mississippi (USM) Polymer Science and Engineering program; Prof. James Rawlins at USM; and Tianbo Liu and Darrell Reneker from the University of Akron.
Yang Zhou presented a poster titled “Plasma Polymerized Coatings for Improved Corrosion Resistance” at the same TCC Annual Review as was mentioned above. His doctoral dissertation titled “Plasma Polymerized Coatings for Improved Corrosion Resistance” was recently accepted and approved. Both the poster and the dissertation are uploaded elsewhere.
Reid Bailey participated in the Research Experience for Undergraduates (REU) program at The University of Akron which had him working with Brenna Rossi. He gave periodic progress reports to the program coordinators and spoke about his research with the other students in the program. He presented a poster titled “Uniform Film Deposition with Atmospheric Pressure Plasma Jet for Corrosion Resistance” at the North Ohio Undergraduate Research Symposium (NOURS) at Kent State University on August 2, 2018. His poster is uploaded elsewhere.

Honors and Awards: Prof. Foster has been named a recipient of Fellowship in the American Physical Society. He was notified in September 2018.

Protocol Activity Status:

Technology Transfer: Brenna Rossi and Reid Bailey worked directly with Andres Bujanda and a similar Surfx Atomflo plasma generator at the Army Research Lab July 17-19, 2018. Ideas about how to more efficiently use the Surfx Atomflo were exchanged and coatings were deposited.

PARTICIPANTS:

Participant Type: PD/PI
Participant: Mark David Foster
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Brenna Rossi
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:

International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Graduate Student (research assistant)
Participant: Yang Zhou
Person Months Worked: 1.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

Participant Type: Undergraduate Student
Participant: Reid Bailey
Person Months Worked: 2.00 **Funding Support:**
Project Contribution:
International Collaboration:
International Travel:
National Academy Member: N
Other Collaborators:

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Publication Type: Conference Paper or Presentation **Publication Status:** 0-Other
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Date Received: 09-Oct-2018 Conference Date: 31-Jul-2018 Date Published:
Conference Location: Hattiesburg, MS
Paper Title: Organic Plasma Polymerized Coatings
Authors: Brenna Rossi, Yang Zhou, Reid Bailey, Andres Bujanda, Ali Dhinojwala, Mark D Foster
Acknowledged Federal Support: **Y**

Publication Type: Conference Paper or Presentation **Publication Status:** 0-Other
Conference Name: Northeast Ohio Undergraduate Research Symposium
Date Received: 09-Oct-2018 Conference Date: 02-Aug-2018 Date Published:
Conference Location: Case Western Reserve University , Cleveland, OH
Paper Title: Uniform Film Deposition With Atmospheric Pressure Plasma Jet For Corrosion Resistance
Authors: Reid Bailey, Brenna Rossi, Mark Foster
Acknowledged Federal Support: **Y**

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Date Received: 09-Oct-2018 Conference Date: 02-Aug-2018 Date Published:
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Paper Title: Uniform Film Deposition With Atmospheric Pressure Plasma Jet For Corrosion Resistance
Authors: Reid Bailey, Brenna Rossi,
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RPPR Final Report
as of 27-Dec-2018

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Date Received: Conference Date: 30-Jul-2018 Date Published: 30-Jul-2018
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Paper Title: Plasma Polymerized Coatings for Improved Corrosion Resistance
Authors: Yang Zhou, Qixin Zhou, Ali Dhinojwala, Mark D. Foster
Acknowledged Federal Support: **Y**

DISSERTATIONS:

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Authors: Yang, Zhou
Acknowledged Federal Support: **Y**

PROJECT FINAL TECHNICAL REPORT

PROJECT OVERALL GOAL AND SPECIFIC OBJECTIVES

The overall goal was to acquire a robotic atmospheric plasma deposition system in support of DoD funded research on plasma polymerized films for use as corrosion protection coatings (FA7000-14-2-20016). This work is being pursued in collaboration with Andres Bujanda of the Army Research Laboratory. Plasma enhanced chemical vapor deposition (PECVD) of highly crosslinked polymeric films offers a potential means to avoid the use of environmentally unfriendly chromate pretreatments and solvent-based top coats containing volatile organic compounds (VOC's) found in current corrosion protection systems[1,2]. Furthermore, PECVD will find wider use in DoD applications if the plasma can be generated under atmospheric conditions, circumventing the need for deposition of the coatings under vacuum.

The primary need is to provide a means to produce plasma polymerized coatings at atmospheric pressure which can then be evaluated and characterized as protective coatings. The new instrumentation includes capabilities to generate plasma at varying power levels and gas flow rates, to both treat surfaces and deposit films on them, to deposit films with one or two monomers at a time, and to control the motion of the plasma across the substrate. The PECVD project will develop coatings for optimizing corrosion resistance that enhance the lifetimes of DoD assets such as ships, planes, and other outdoor structures.

ACTIVITIES AND ACCOMPLISHMENTS:

State-of-the-art Instrument Acquired

An AtomFlo 500 was purchased from SurfX Technologies LLC and installed at The University of Akron (UA) on December 7, 2017. The AtomFlo is capable of generating plasma with helium or argon as the plasma gas with the option to mix it with oxygen and/or nitrogen as process gasses at ambient conditions. Additionally, we acquired a monomer delivery system that allows us to precisely control the flow rate and temperature of vapor from one or two liquid monomers for deposition and a robot that can move the plasma head in relation to a substrate in the X, Y, and Z directions. The combination of the programmable plasma head motion and the freedom to deposit in air opens opportunities to both plasma treat (e.g. clean) substrates and uniformly deposit coatings on substrates of various sizes and shapes quickly, easily, and safely. Power level, plasma and process gas flow rates, bubbler flow rates, plasma head to substrate distance, and plasma head movement speed can all be adjusted to optimize coating characteristics such as thickness and composition. This can all now be done in our lab at UA, complementing the capabilities available in the Army Research Lab with Andres Bujanda.

DOD-Funded Research Project Supported

Atmospheric Plasma Polymerization for Improved Corrosion Protection (FA7000-14-2-20016***)***

The objective of the project is to compare the structures and performance of plasma polymerized (pp-) films for improving corrosion protection made with two types of the plasma deposition and to develop the second type (atmospheric) to achieve performance gains similar to those achieved with the first type (vacuum).

Background for comparisons

We have done comparisons between coatings deposted with vacuum plasma and with atmospheric plasma primarily with the plasma deposition of the widely studied hexamethyldisiloxane (HMDSO) monomer (shown in Figure 1). The coating system for testing

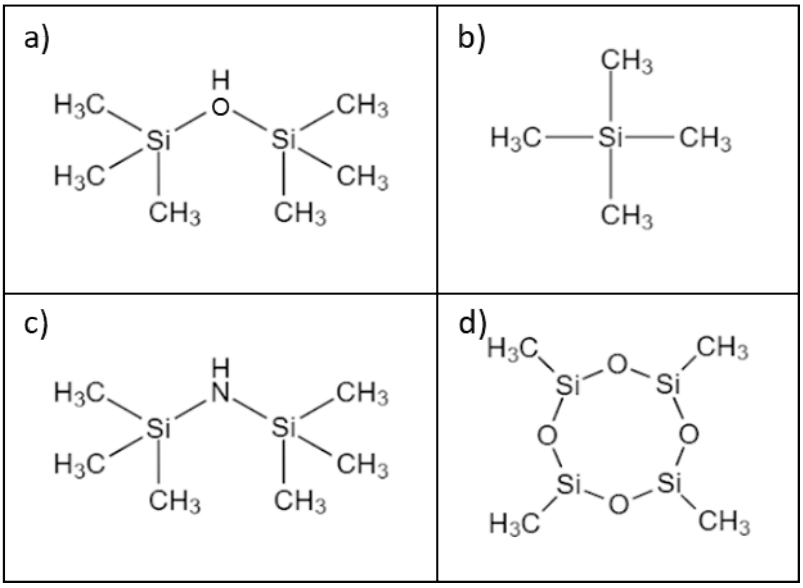

Figure 1. Monomers used in depositing coatings with atmospheric plasma include a) hexamethyldisiloxane (HMDSO), b) tetramethylsilane (TMS), c) hexamethyldisilizane (HMDSN), and d) tetramethylcyclotetrasiloxane (TMCTS).

corrosion resistance diagrammed in Figure 2 was produced by depositing a 40 nm think film of plasma polymerized HMDSO (pp-HMDSO) on aluminum alloy 3003 H14 sheet and a topcoat of polyurethane (PU) was applied to the pp-HMDSO coating. The performance of the pp-films for mitigating corrosion has been quantified in two ways: adhesion to topcoat and electrochemical impedance. The adhesion of a polyurethane (PU) top coat to a vacuum-deposited pp-HMDSO

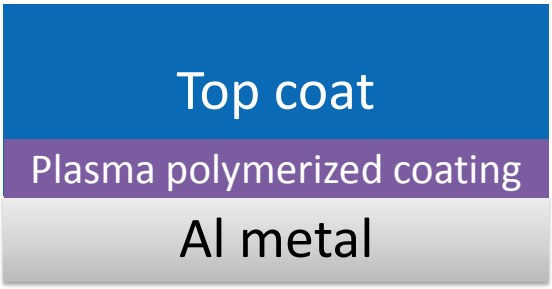

Figure 2. Schematic of the coating system of PU on pp-film on Aluminum[3]

primer coat on Al has been quantified using a PosiTest Pull-off Adhesion Tester in accordance with the ASTM-D4541 standard. For pp-HMDSO films deposited in vacuum, the adhesion of PU to pp-film achieved is 0.5 ± 0.02 MPa.[4]

Short term resistance to corrosion of the two layer PU/pp-HMDSO coating system has been characterized using electrochemical impedance spectroscopy (EIS). The experimental setup was a typical three-electrode EIS setup in 3.5% NaCl solution.[5,6] An O-ring glass cell was attached to the coating surface with a glass tube clamp. The sample functioned as the working electrode (testing area of each coating sample was 14.6 cm^2); the saturated calomel electrode was the reference electrode; and a platinum mesh was the counter electrode. EIS measurements were performed using the Reference 600 Potentiostat by Gamry Instruments. The frequency range scanned was from 10^{-2} to 10^5 Hz with 10 points per decade (spaced logarithmically) using a 15 mV AC perturbation coupled with the open circuit potential. The setup conditions were the same for all samples.[7]

The EIS results after ca. 230 days' exposure to 3.5% salt water show that the coating system resists corrosion better with the pp-HMDSO film underneath the PU top coat. The aluminum alloy coated with only polyurethane (Figure 3) experienced a large drop in impedence—its ability to resist the electrical activity that causes electrochemical reactions resulting in corrosion—within a few days, while the impedance dropped much less for the aluminum alloy with the pp-HMDSO film in between (Figure 4).

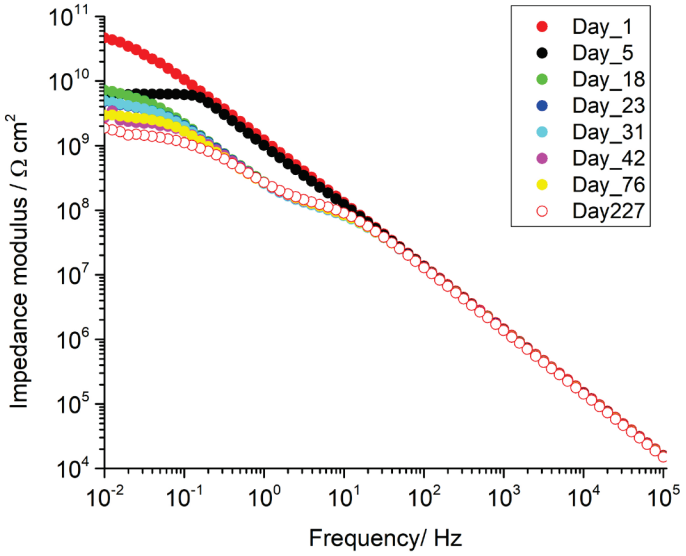

Figure 3. EIS data for sample with PU top coat directly on aluminum alloy 3003 H14 exposed to 3.5% NaCl solution in an EIS cell.[4]

The chemical composition of the coating deposited in vacuum was characterized with X-ray Photoelectron Spectroscopy (XPS). Data in Figure 5 (a) show that when deposited in vacuum the carbon and oxygen contents are 54.2 and 27.3 at. %, respectively.

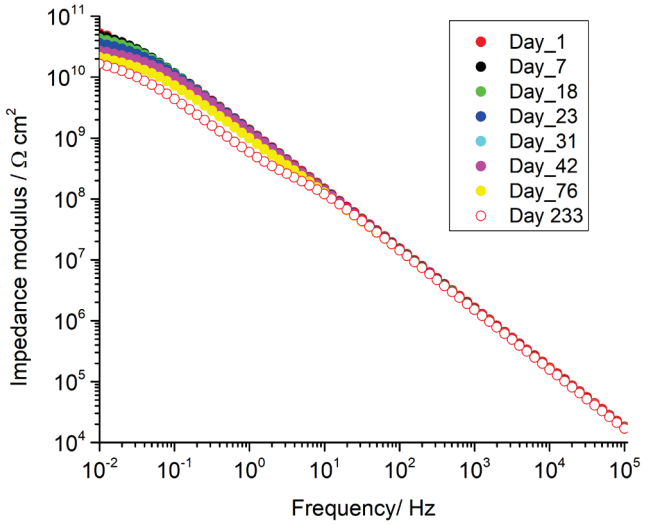

Figure 4. EIS data for the case of a coating system of PU top coat on 40 nm pp-HMDSO film on aluminum alloy 3003 H14 exposed to 3.5% NaCl solution in an EIS cell.[4]

We began making atmospheric plasma polymerized (APP) films in collaboration with the Army Research Lab (ARL) using their Surfx Atomflo apparatus. For sophisticated characterization of the films with X-ray and neutron scattering techniques, very well-defined films of uniform thickness are needed. However, the first films made with APP at ARL were not uniform and thus discovering values of process parameters for which the films would be more uniform was a key first goal with the new APP apparatus from the DURIP grant. Having that capability at UA was important for being able to broadly explore deposition parameters to achieve uniform films as well as to investigate a wide variety of possible monomers to optimize corrosion protection.

Results with new instrumentation:

Higher Carbon Content

One key objective with the new instrumentation was to achieve film deposition with monomers that might yield films with higher carbon contents to render the coatings more hydrophobic, similar to what was achieved with vacuum plasma polymerization. While films with high oxygen contents would be expected to give good adhesion, resistance to incursion of water or electrolyte should be improved by having a somewhat more hydrophobic pp-film.[7] It was found early on that pp-HMDSO films deposited at atmospheric conditions yielded a much lower carbon content, i.e. ~5 at. %, while a vacuum-deposited pp-HMDSO films had a carbon content ~50 at.[4] %. Experiments to raise carbon content in APP films started by depositing films using TMS (Figure 1) as a monomer[8] because the atomic ratios of carbon to oxygen and silicon in that molecule are higher than they are in HMDSO. HMDSN (Figure 1) was also investigated as a monomer in hopes of at least lowering the oxygen content in films while maintaining the ease of deposition demonstrated with HMDSO, which has a structure very similar to that of HMDSN.

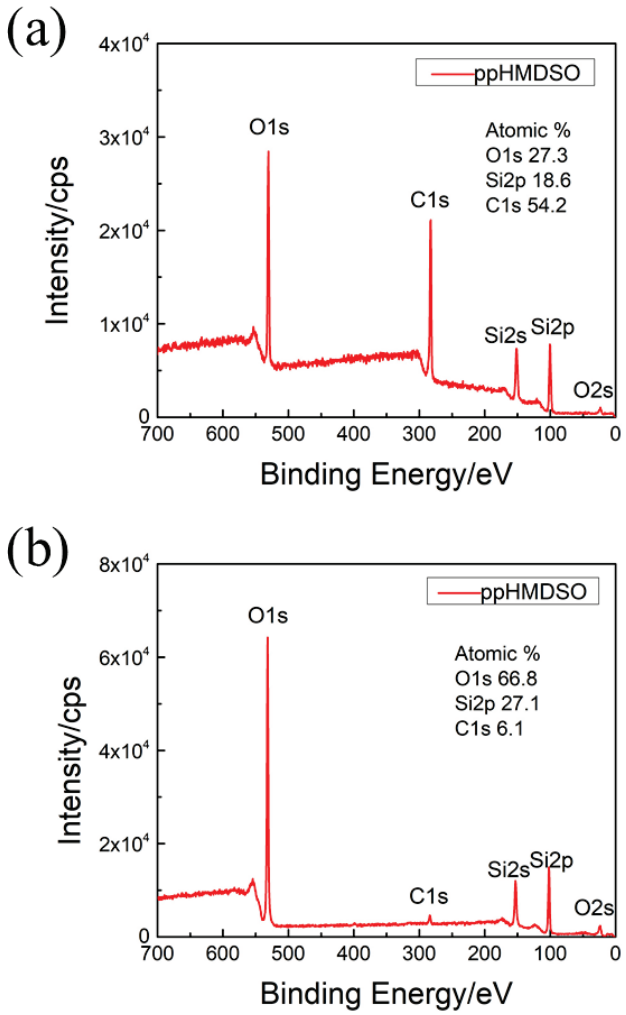

Figure 5. XPS survey data from (a) a vacuum pp-HMDSO coating showing a carbon content of 54.2 at. % and from (b) an atmospheric plasma deposited pp-HMDSO coating showing a carbon content of 6.1 at. %.[4]

XPS data (Figure 6) show that atmospheric pp-TMS films have carbon contents about half that of vacuum pp-HMDSO (~25 at. %), while atmospheric pp-HMDSN films have carbon contents very similar to those of atmospheric pp-HMDSO films. Using HMDSN introduces a small amount of nitrogen (2 at. %) with a concomitant decrease in oxygen content (-2 at. %), but does not significantly affect the oxygen content, suggesting that this oxygen is primarily incorporated from the surrounding atmosphere rather than coming from the monomer structure itself. In support of the XPS data, Fourier transform infrared (FTIR) spectra of atmospheric pp-HMDSO, -pp-HMDSN, and pp-TMS (Figure 7) show a present C-H stretch in pp-TMS and a relatively strong Si-N peak in pp-HMDSN considering the film contains only 2 at. % nitrogen.

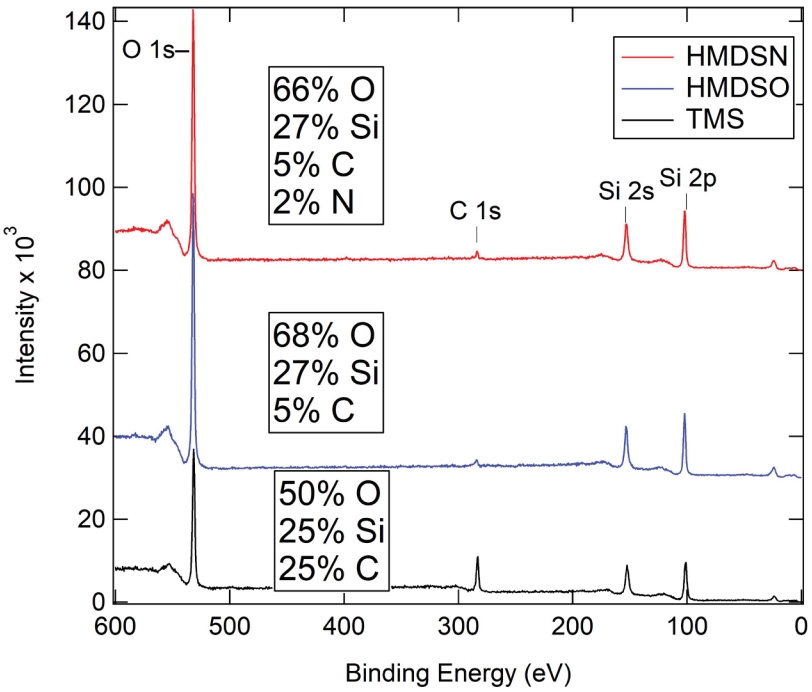

Figure 6. XPS survey data from (top) atmospheric pp-HMDSN coating and (middle) atmospheric pp-HMDSO showing carbon contents of 5 at. % and from (bottom) an atmospheric pp-TMS coating showing a carbon content of 25 at. %.

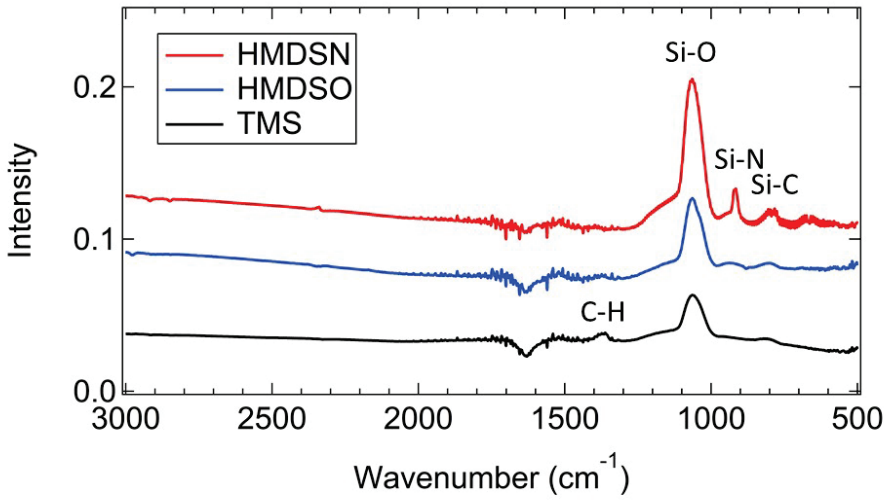

Figure 7. FTIR spectra of atmospheric pp-HMDSN, pp-HMDSO, and pp-TMS showing the key differences in their compositions, namely the visible C-H stretch in pp-TMS and Si-N peak in HMDSN.

Better Film Uniformtiy

Another objective in early experiments with this instrumentation has been to deposit films (1.5x 1.5 cm^2 or bigger) of good lateral thickness uniformity. These experiments were performed with HMDSO exclusively because it deposits easily and consistently. Determining the optimal pattern

of movement of the plasma head over the substrate was the first target because some patterns of movement produced films that were visibly nonuniform as pictured in Figure 8.1. The plasma head is like a shower head; the plasma is generated within the head and the monomer fragments exposed to the afterglow of the plasma "rain down" on the substrate through an array of holes. Initially, the shower head was positioned in a stationary position above the substrate. The coating deposited as visible spots mirroring the array of the holes on the shower head, indicating that little dispersion of the flow from the holes occurred before reaching the substrate. Circular motions of the head were next tried; moving the head in a small circle resulted in an array of slightly more diffuse dark spots, and large circles produced dark splotches, the darker color corresponding to an area with thicker coating. We suspect that the path of the circles resulted in points of overlap, exposing those points to the plasma afterglow and reactive particles more frequently than the other points along the path. A serpentine or "square raster" pattern in which the head moves all the way across the sample in the Y direction and makes a step in the X direction before moving back in the Y direction produced dark stripes across the film until the step size was reduced to a length less than the space between holes in the shower head. The square raster pattern with a small step size yielded films macroscopically uniform in color. For films that were not visibly nonuniform, film thickness uniformity was determined by measuring the thickness of three points on a film (as illustrated in Figure 9) using ellipsometry.

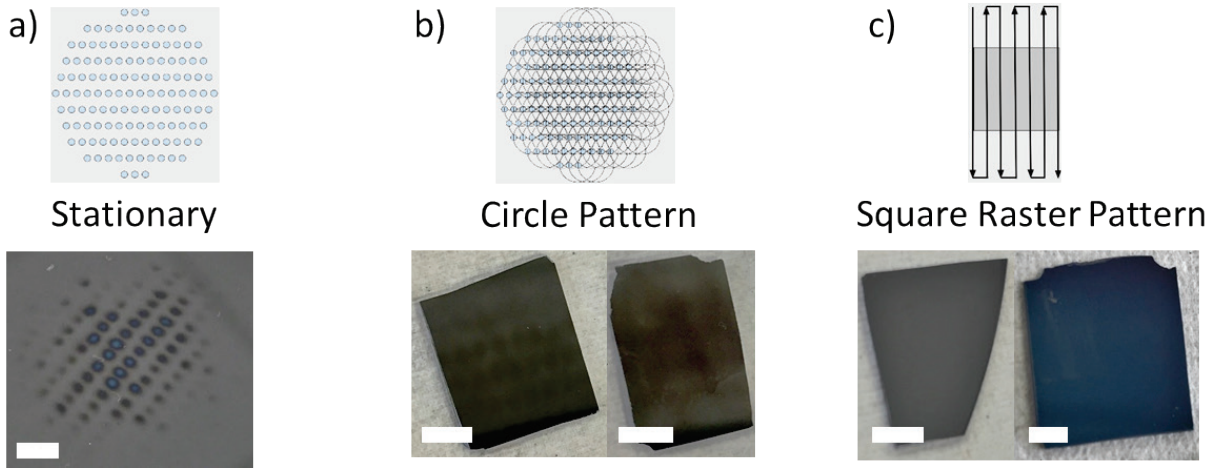

Figure 8.1. The deposited atmospheric pp-HMDSO films a) displayed the pattern of the shower head when the head was held stationary, b) developed dark, thick spots when the plasma head was moved in small circles, and c) appeared uniform in color when the plasma head was moved across the sample in a square raster pattern. The white scale bars represent 5 mm.

As a result of these studies, good lateral thickness uniformity was achieved by starting the square raster pattern with a small step size with the leading edge of the plasma head on the sample, moving the entire head across the sample with each bottom-to-top or top-to-bottom scan, and

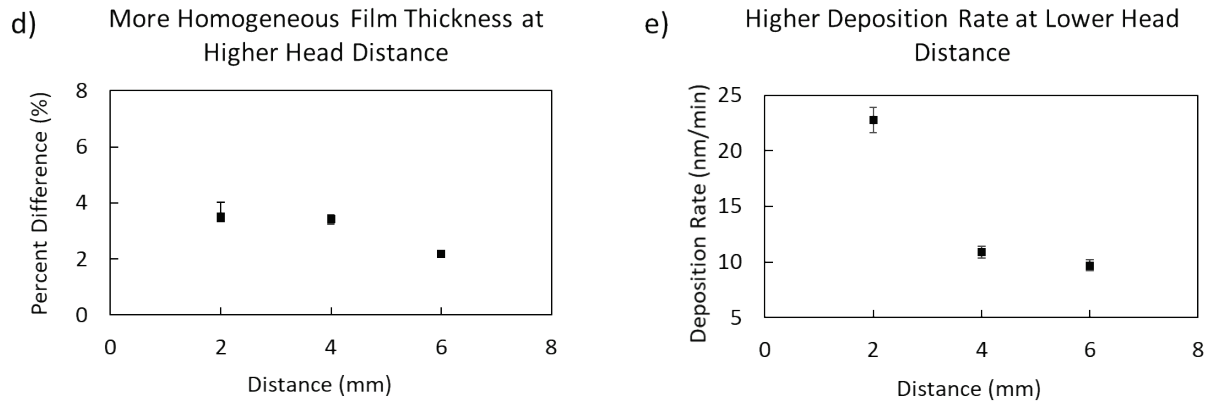

Figure 8.2. (d) Increasing the distance between the plasma head and the substrate improves the uniformity somewhat for a change from 2 mm to 6 mm, but (e) such an increase results in a decrease of factor 2.3 in the deposition rate.

ending with the head completely off the sample to guarantee that every point on the sample was exposed to the plasma afterglow for the same amount of time. "Good lateral uniformity" was defined as keeping the percent deviation from the mean among thicknesses measured at multiple points on a sample under 5%. Uniformity could be improved by increasing the sample to deposition head distance from 2 to 6mm (Figure 8.2d), but with an accompanying strong decrease by a factor 2.3 in the rate of deposition (Figure 8.2e).

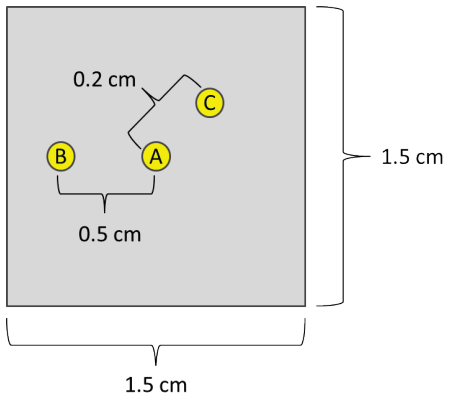

Figure 9. Film thickness was measured at points A, B, and C on each sample to determine thickness uniformity in terms of standard deviation of the measurements.

Investigate Monomers Beyond TMS

A third objective was to investigate monomers beyond TMS, looking again for films with higher C content such as were achieved originally with vacuum plasma deposited HMDSO. Besides the studies with HMDSN, preliminary characterizations have been performed on atmospheric pp-films deposited from TMCTS (see Figure 1) as a monomer. XPS measurements showed that with this monomer a carbon content as high as 25.2 at. % has been achieved.

The deposition rate for each monomer was calculated because monomers that deposit faster require less energy and gas for laying down a coating of a particular thickness. As is shown in Figure 10, TMCTS had by far the fastest deposition rate, but films were visibly nonuniform.

TMS deposits at a fair rate, but the quality of the deposition is very sensitive to environmental conditions such as temperature and humidity. This sensitivity is currently under investigation.

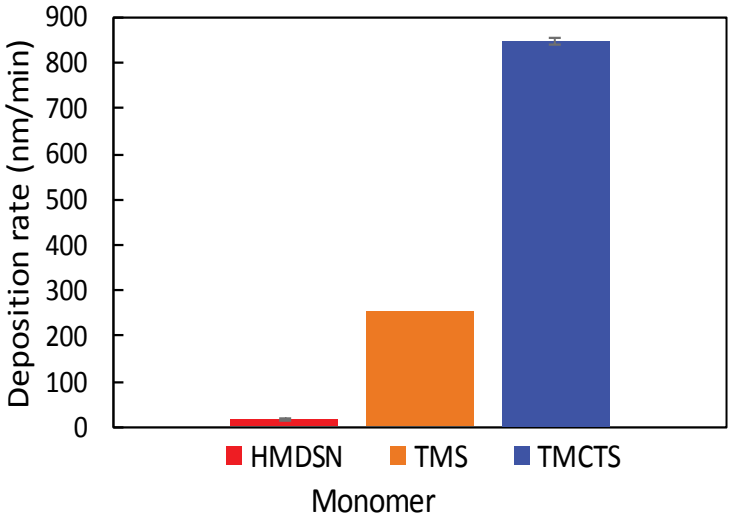

Figure 10. Comparison of the deposition rates for HMDSN, TMS, and TMCTS with constant substrate to head distance, head speed, and head movement pattern, but with the power level and flow rates optimized (roughly) for each. TMCTS deposits the fastest by far.

The EIS measurement described previously was also done for a coating system of PU on atmospheric pp-HMDSN on AL 3003 H14, as shown in Figure 2. The short-term results shown in Figure 11 were similar to those for the vacuum pp-HMDSO; the coating systems maintained its impedance modulus over the course of a week while the impedance modulus of the system of PU laid directly onto Al 3003 H14 experienced a significant drop in impedance modulus.

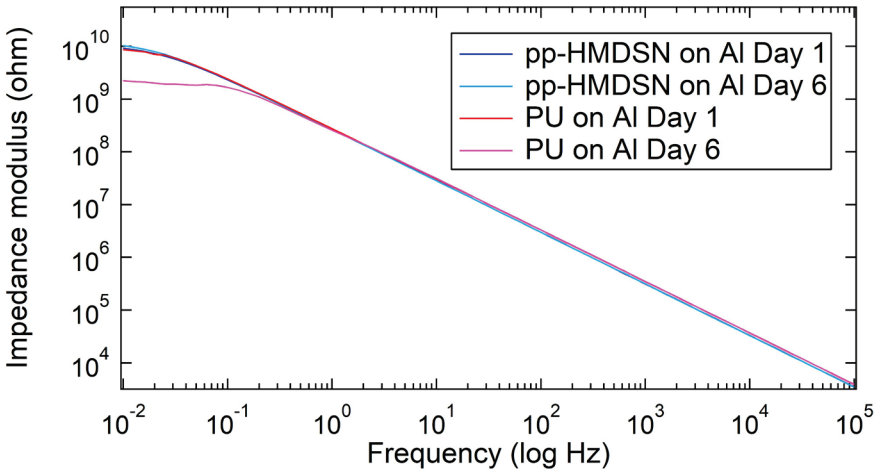

Figure 11. EIS data for sample with PU top coat directly on top of Al 3003 H14 and for the PU coating system with atmospheric pp-HMDSN exposed to 3.5% NaCl solution in an EIS cell. The pp-HMDSN coating prevented the system from experiencing the drop in impedance modulus that the PU directly on Al alloy did.

Summary

The recent acquisition of the new Surfx Atomflo 500 has greatly increased our capabilities to produce results that will aid the DoD in accomplishing its goal to better protect its metal assets from corrosion and human assets from harm to their health. Without using any environmentally unfriendly VOC's, the Atomflo 500 deposits plasma polymerized coatings which—according to our results—show promise for corrosion protection. Depositing coatings at ambient conditions as opposed to in a vacuum is far more conducive to practical applications; an atmospheric plasma deposition system can easily be incorporated into a manufacturing assembly line, and equipment too large to fit in an available vacuum chamber need not be disassembled. If a substrate requires recoating due to damage, the coating can be directly applied to the damaged spot rather than removing the damaged piece for recoating in a vacuum chamber. As is now evident, coatings deposited with the same monomer, but by different methods can have quite different properties. Nonetheless, with the capabilities provided by this acquisition to quantify how the properties of coatings deposited in ambient conditions can be optimized through changing deposition parameters, we have already achieved coatings that provide substantial protection against corrosion by atmospheric plasma polymerization. So far we have:

1.) Identified a monomer (precursor), TMS, that yields films when deposited with atmospheric plasma polymerization that have a higher carbon content than atmospheric deposited pp-HMDSO.

2.) Improved the uniformity of the films we deposit with atmospheric plasma deposition by optimization of the movement pattern for the deposition head.

3.) Surveyed how film uniformity and deposition rate vary with sample to head distance.

4.) Verified that the monomer TMCTS, suggested to us by collaborators at ARL, provides a comparatively high deposition rate in atmospheric plasma polymerization deposition.

5.) Shown that an atmospheric plasma deposited film used as a primer coat under PU can substrantially improve corrosion resistance over short times. .

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