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Corrosion Resistance of Silane Coatings on Aluminum and Magnesium Alloys

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Corrosion Resistance of Silane Coatings on Aluminum and Magnesium Alloys

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The Williams Honors College

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Honors Abstract Addendum

The purpose of this project was to increase the corrosion resistance of aluminum and magnesium alloys by coating the coupons with a silane on the surface. Four different silanes: 3-aminopropyltriethoxysilane (APTES), Bis(triethoxysilylpropyl)amine (Bis), (Heptadecafluoro-1,1,2,2-tetrahydrodecyl)trichlorosilane (FTS), and (Heptadecafluoro-1,1,2,2-tetrahydrodecyl)triethoxysilane (non Cl FTS) will be used and compared to see which silane would give the best corrosion resistance. Each silane solution was coated on the coupons by using a solution method in which the coupon was submerged for 24 hours and then thermally cured for another 24 hours. Once the coating was applied, the water contact angle and the thickness of the coating were measured. Then the metal coupons were subject to a corrosion test by immersing them in sodium chloride and oxalic acid solutions for 7 days. The weight loss of the metal coupon was measured after the immersion test. All the silane coatings provided an increased corrosion resistance for aluminum alloy AA2024-T3, and the only silane, APTES, tested on magnesium alloy AZ 31 also significantly reduced its corrosion in both corrosive solutions. The FTS coating provided the best corrosion resistance on the aluminum alloys in an oxalic acid solution and the Bis solution had the best corrosion resistance in the sodium chloride solution. With the addition of a silane coating, aluminum and magnesium alloys used in industry will have an increased corrosion resistance and will increase the life of the part so it will not need to be replaced.

Executive Summary

This project was conducted in order to increase the corrosion resistance of aluminum alloy AA2024-T3 and a magnesium alloy AZ 31 with the addition of a silane coating. Metals are often used in corrosive environments, and when exposed can rust over a certain period of time. When the metal rusts, it loses its functionality and needs to be replaced. By adding a silane coating to the surface of the metal, it will increase its corrosion resistance and increase the life of these metal parts. Aluminum alloys are often used in the textile industry where they often come in contact with oxalic acid and corrode. Thus, a solution to the corrosion problem needs to be found. For this project, multiple different silane coatings were applied to determine which coating would provide the best corrosion resistance. The silane coatings evaluated were 3-aminopropyltriethoxysilane (APTES), Bis(triethoxysilylpropyl)amine (Bis), (Heptadecafluoro-1,1,2,2-tetrahydrodecyl)trichlorosilane (FTS), and (Heptadecafluoro-1,1,2,2-tetrahydrodecyl)triethoxysilane (non Cl FTS). The coating was mainly applied by using the solution deposition method followed with thermal curing. Once the coatings were applied, the metal coupons were subject to the immersion corrosion testing (ASTM G31) for a week using an oxalic acid solution or a sodium chloride solution. The weight loss of the coupon after testing was determined. The metal coupons also went through water contact angle and thickness measurements before and after the corrosion test to assess if the coating had been applied or retained and if so, how thick was the coating. As a comparison to the solution deposition method, the dip coating method, was briefly investigated. The dip coating is the more commonly used method in other

experiments, but it was determined that once applied and dried, the coating was inconsistent and uneven. The solution method gave a more even coating and was used throughout the entire project.

After the experiments concluded and all the data was compiled, it was determined, for AA2024-T3, that the FTS silane provided the best corrosion resistance in the oxalic acid solution and the Bis silane provided the best protection in the sodium chloride solutions. Depending on where the metal is being used and the applications it is being used for, a specific silane can be used to increase the life of the part. More replications of the tests need to be done in order to prove that these two silanes provided the best corrosion resistance in each solution.

An APTES coating was applied to a magnesium alloy AZ 31 to briefly evaluating its ability to increase the corrosion resistance of the coupon. The magnesium alloy was extremely reactive in the oxalic acid solution, the APTES coating still gave some corrosion resistance compared to the untreated surface. The APTES also provided some increased corrosion resistance to the sodium chloride corrosion test.

The results from this project can be used to improve the corrosion resistance of aluminum and magnesium alloys in field such as the textile industry or automotive or aircraft industry. In order to improve upon the results in this experiment, more replications can be done as well as testing the same variables under different conditions.

Introduction

The purpose of this project is to improve the corrosion resistance of certain aluminum alloys and magnesium alloys with the use of multiple different silane coatings. Metals are often used in corrosive environments, and when exposed can rust over a certain period of time. When the metal rusts, it loses its functionality and needs to be replaced. By adding a silane coating to the surface of the metal, it will increase its corrosion resistance and increase the life of these metal parts. Multiple different kinds of silanes will be tested against a control group with no silane coating to determine their corrosion resistance capability for the aluminum alloy AA2024-T3 and a magnesium alloy AZ 31. The silanes that are going to be tested, which can be seen in **Table 1**, are 3-aminopropyltriethoxysilane (APTES), Bis(triethoxysilylpropyl)amine (Bis), (Heptadecafluoro-1,1,2,2-tetrahydrodecyl)trichlorosilane (FTS), and (Heptadecafluoro-1,1,2,2-tetrahydrodecyl)triethoxysilane (non Cl FTS). In order to apply the silane coating to the metal, two methods: dip coating and solution deposition, will be tested to see which applies evenly and more efficiently. To determine if the coating was deposited evenly across the metal surface, a thickness measurement and a water contact angle measurement were conducted, and the coated surfaces were further examined using an optical microscope. In order to assess the corrosion resistance of the metal alloy with and without a silane coating, they were subject to a corrosion immersion test (ASTM G31) in a 3.5 wt.% (~ 0.6M) sodium chloride solution and a 0.068 M of oxalic acid for a period of 7 days. The corrosion rate was estimated based on a standard weight loss

method (ASTM D2688-05), and corrosion morphology was examined using an optical microscope and an infinite focused microscope (i.e., surface profiler).

Background

Metal finishing industries have been looking for ways to provide a corrosion resistant treatment to the surfaces of metal for the past couple years. Chromates were often used in the past, but there has been a push to eliminate the use of chromates due to the discovery that hexavalent chromate ions can be carcinogenic and toxic. Silane surfaces are becoming more and more relevant as a safer alternative for corrosion resistance. The silane adds a protective layer onto the surface as to not allow any corrosive material to attack the metal surface underneath.

One of aluminum's most desirable characteristics is its versatility. There are more than three hundred commonly known alloys and many new variations have been developed. Aluminum exhibits numerous physical properties including its high reflectivity. Radiant energy, visible light, radiant heat, and electromagnetic waves are reflected. Aluminum is also a great conductor of both electricity and heat. Aluminum is often used in applications that require high electrical conductivity because it is nearly twice the level that copper is at roughly the same weight [1]. Pure aluminum naturally forms a thin surface layer of aluminum oxide when in contact with oxygen. Through the process of oxidation, a physical barrier is created against corrosion and further oxidation. This makes the surface highly resistant to most acidic and neutral solutions

from the protective oxide layer. Aluminum alloys however, provide a little oxide layer, thus making the surface still prone to corrosion. Aluminum alloys are often used in the textile industry, which often utilize oxalic acid. Oxalic acid is a relatively strong, simple dicarboxylic acid. In the textile industry, oxalic acid is used as a bleacher/neutralizer during the processing of fibers [2]. Oxalic acid is also used in wood industries, an additive for metal cleaning, a feedstock chemical for synthesizing biodegradable polymers and metal oxides, and an active chemical for recovering metals in hydrometallurgy [3]. Thus, a corrosion resistant coating is very important and highly sought after.

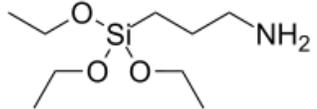
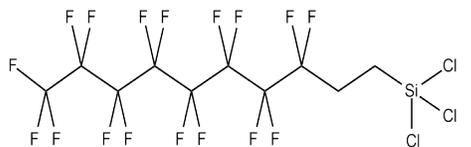
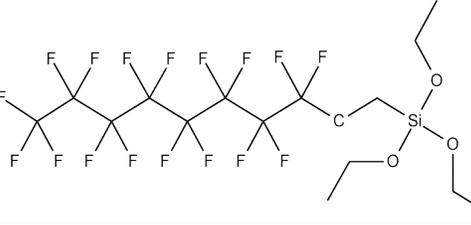
While aluminum generally has a relatively good corrosion resistant, it can still be highly corroded in an acidic environment with a $\text{pH} < 4$ or an alkaline solution with a $\text{pH} > 8.5$. The natural oxide layer that is formed on the surface of the aluminum can be affected by its dissolution in aggressive environments.

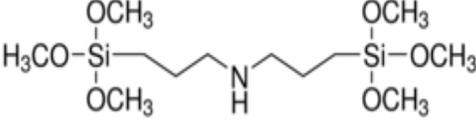
Magnesium and its alloys have been determined that they can possibly be used as implants in the human body because of their similarities to human bones in terms of degradability, mechanical, and physical properties. However, one of the main difficulties in using magnesium for biodegradable implants is because of their low corrosion resistance in solution or in the body. When magnesium gets corroded, it loses its functionality and it weakens mechanically. Some reports have confirmed that the rapid corrosion of magnesium causes hydrogen evolution and alkalization and does not give enough time to regenerate around the implants which can be harmful in the body.

With a proper coating on the magnesium surface, the corrosion rate can be slowed to a point where it can be safe to use magnesium as implants in the body [4].

Silanes are monomeric chemicals that are used as coupling agents that were first discovered in the 1940s. If the silane contains at least one silicon-carbon bond, it is known as an organosilane. The carbon-silicon bond in the silane is very stable and nonpolar, but in the presence of an alkyl group it exhibits hydrophobic effects and low surface energy. Organo-functional silanes are able to bond with numerous different surfaces because they have two different reactive groups on their silicon atom. They can also enhance adhesion between dissimilar materials of their low surface tension which ensures good wetting surface. Because of the silanes functionality they have been known to provide great benefits as an adhesion promoter, mineral filler treatment, corrosion resistant layer and cross-linking agent.

Table 1. The different silanes used during this project.

silanes used	Chemical Formula/Structure	Molecular weight (g/mol)
3-aminopropyltriethoxysilane (APTES)		221.37
(Heptadecafluoro-1,1,2,2-tetrahydrodecyl)trichlorosilane (FTS)		581.56
(Heptadecafluoro-1,1,2,2-tetrahydrodecyl)triethoxysilane (non Cl FTS)		610.38

Bis(triethoxysilylpropyl)amine (Bis)		425.71
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Silanes are often characterized in two different groups in terms of their hydrophobicity, alcohol-based and water-based. The problem with silane solutions is that their hydrophobic nature requires a large amount of organic solvents such as ethanol or methanol. The silanes used during this experiment were alcohol based and required a large amount of ethanol to create the solution. Due to the volatility and flammability of ethanol, this poses as a major problem when using it in large quantities when taking to industry [5]. The reduction of volatile organic compounds (VOCs) is mandated by legislatures and end users due to safety concerns.

There are many theories as to how the silane coating is bonded to the surface of the metal. Silanes are known as coupling agents, which is defined as a compound which provides a chemical bond between two dissimilar materials, usually an inorganic and an organic. Organofunctional silanes have a functional group on one end and a hydrolyzable Si ester group on the other end. The trialkoxysilane contains alkoxy groups, which are hydrolyzed to form a silanol containing compound. Once the hydrolysis is complete, condensation to oligomers occurs. Once the metal coupon is submerged in the hydrolyzed silane solution, the oligomers form a hydrogen bond with the OH groups of the substrate. Then during the curing process, water is removed and a covalent linkage is formed with the substrate. Although the steps are described

sequentially, these reactions can all occur simultaneously after the initial hydrolysis step. It is assumed that the MeOSi and SiOSi covalent bonds are responsible for the bonding of the silane to the metal substrate. [6]. One of the most important features of the silane and when it bonds to its substrate is that it assembles a very dense self-assembled silicon and oxygen rich network. This film is homogenous, hydrophobic, and resists water uptake and has chemical stability. This allows for a great corrosion resistance to acidic solutions. The thickness of the siloxane layer is determined by the concentration of the siloxane solution. One notable defect in the silane film is that small pinholes or cracks can be present in the film, which may be susceptible to a small amount of corrosion damage, but still provides more corrosion resistant than without the film in general [7].

APTES is widely used as an adhesion promoter/coupling agent that links an organic coating to an inorganic substrate. Due to its reactive amino terminal group and silanol head groups, APTES is able to bond to the surface with hydrogen and covalent bonds [8]. It also forms a tightly cross-linked network upon thermal curing [8-11], and the layer consists of such network is hydrophobic [11] and remains hydrophobic even after submersing in water for 3 days [11]. Therefore, the thermally cured APTES layer deposited to a metal surface could serve as a barrier against water penetration, consequently protecting the metal from corrosion. Bis silanes have often been used as crosslinkers for coupling agents. They are known to provide a good corrosion resistance, but are often dependent on the surface condition of the metal substrates [5].

The research done in our group has shown that Bis silanes, like APTES, form a cross-linked hydrophobic layer upon thermal curing [12]. Both the FTS and non Cl FTS contain fluorocarbons, which are known to have a super hydrophobicity that repels water from the surface [13]. When they bond to the surface of aluminum or magnesium alloys, they make the surfaces resistant to water, hence minimizing the contact of water, a criterion for reducing metal corrosion.

Experimental Methods

The aluminum alloy AA2024-T3, when purchased, comes in large sheets and had to be cut in order to test individual pieces. The alloy was cut into 2.5 x 1 cm² coupons. Each test required 3 coupons and a control group. The aluminum coupons needed to be cleaned in order to give an uncontaminated surface to allow for the silane to bind to the surface of the aluminum alloy. This was done by immersing and sonicating the aluminum coupons in a series of chemical baths for 10 minutes each. The coupons were first submerged in a bath of hexane, followed by acetone, and then an alkaline cleaning solution. The coupons were then rinsed thoroughly in DI water and air dried. The silane solution was prepared by making a 5 wt.% solution of silane using ethanol as the solvent. In order to apply the silane coating to the surface of the aluminum coupons, the coupons were completely submerged in the silane solution in a glass container for a total of 24 hours. A solution method was used instead of the more traditional dip method because the dip method provided inconsistent results while the solution method provided a more even coating. In addition to having the aluminum coupons in

the solution, at least one silicon wafer was added to the container as well in order to test the thickness of the silane coating applied. After the 24 hours, the coupons were taken out of the solution; they were rinsed with ethanol to remove any of the loose, unreacted silanes from the surface. The coated coupons were then positioned in a glass dish and placed in a vacuum oven for 24 hours at approximately 130°C to cure and lock in the silane coating on the surface of the aluminum alloy. The water contact angle was measured, using a contact angle goniometer, for the modified and unmodified aluminum coupons to ensure that the coating was applied. An ellipsometer was then used on the modified Si-wafer to see how thick of a coating was deposited on the surface. It is then assumed that the thickness on the wafer is similar to what it would be on the aluminum coupon. The mass of the modified and unmodified aluminum coupons were recorded prior to the corrosion test. In order to test the corrosion resistance, each coupon was submerged in either a 3.5 wt.% NaCl solution or .07M oxalic acid solution for 1 week. Each test was done in triplicate with a control group. Per ASTM standards, 20 mL of solution was used per cm² of surface area. After 1 week in the corrosion test, the coupon was removed from solution and any corrosion products accumulated on the surface were brushed off thoroughly under running water. The mass of each coupon was recorded after the corrosion test to calculate the corrosion rate. The corrosion rate was a uniform corrosion rate and not pitting corrosion. The contact angle and thickness was measured once again to see if/how much of the coating remained after the corrosion test. Some of the aluminum coupons were then placed

under an optical microscope in order to get a better look at the corrosion and compare different tests.

Data and Results

The first thing tested during this project was how to efficiently apply the coating to the surface of the aluminum alloy. AA2024-T3 coupons that were dip coated and solution deposited with APTES were subject to the immersion corrosion testing. The corrosion rates were gathered from a week test using a .07M oxalic acid solution and presented in **Figure 1**. The coupons that were solution deposited had a corrosion rate of approximately .42 $\mu\text{m}/\text{day}$, while the coupons that were dip coated had a corrosion rate of approximately .47 $\mu\text{m}/\text{day}$. Both of the modified surfaces provided better corrosion resistance than the unmodified surface that had a corrosion rate of .99 $\mu\text{m}/\text{day}$.

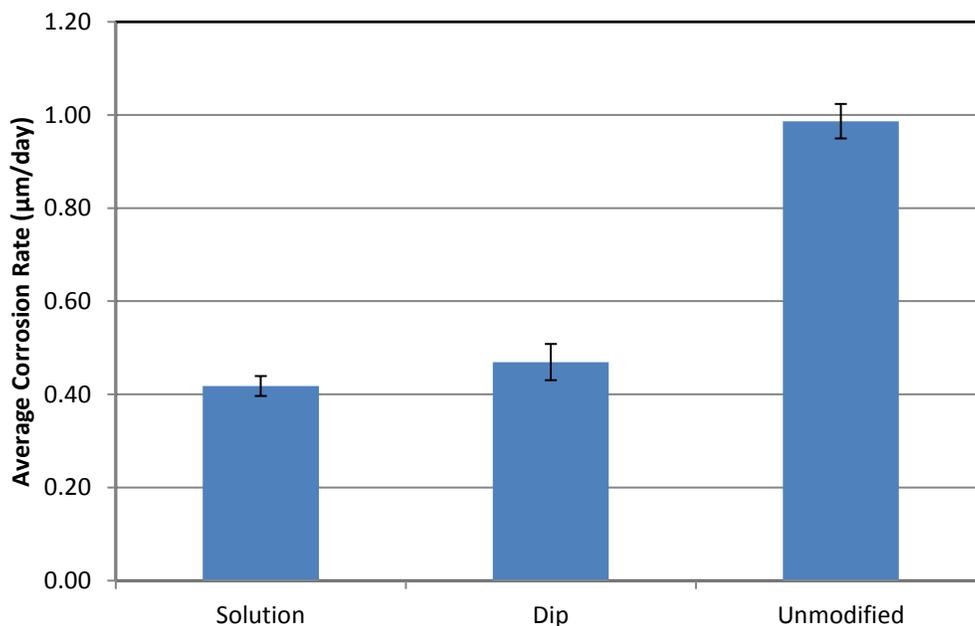


Figure 1. The corrosion rates of aluminum alloys that are unmodified, modified with APTES by dipping in the APTES solution followed with thermal curing, and modified with APTES by submerging in the APTES solution for 24 hours. The values reported are the averages of measurements from at least three samples, and the error bars are the standard derivations.

In a second set of experiments, multiple different silane solutions were tested to see which provided the best corrosion resistance. There were a total of four silanes tested, APTES, Bis, FTS, non Cl FTS, as well as an untreated aluminum. Each silane was tested using the experimental procedure listed above. Again, to ensure the coating had been applied to the surface of the metal coupon, the contact angle was measured with water. All of the silane coatings are hydrophobic, so if the water droplet on the surface looks like a ball then the surface is hydrophobic and the silane coating was applied. A larger water contact angle means a more hydrophobic surface. The results for the contact angle before and after the corrosion tests can be seen in **Table 2 and 3**. All of the contact angles before the corrosion test were greater than 50° showing that the coating was applied due to its hydrophobic nature. The contact angle for most of the coatings after the corrosion test were reduced due to the fact that the coating was removed in the corrosive environment.

Table 2. The water contact angles of the silane coated coupons pre corrosion test

Sample	Contact Angle Pre-Corrosion Test (°)			
	APTES	Bis	FTS	Non Cl FTS
1	67.2	54.1	118.2	101.6
2	65.2	51.8	127.6	99.2
3	-	53.3	52.5	100.5
Average	66.2	53.1	99.4	100.4

Table 3. The water contact angles of the silane coated coupons post corrosion test

Sample	Contact Angle Post Corrosion Test (°)			
	APTES	Bis	FTS	Non Cl FTS
1	78.3	36.3	84.6	40.1
2	52.6	45.2	73.3	42.1
3	-	96.8	74.4	34.8
Average	65.4	59.5	77.4	39.0

Once all of the aluminum alloy samples were coated with silane, they went through a corrosion test in either a sodium chloride solution or oxalic acid solution. The corrosion rates were plotted in **Figure 2**. In the oxalic acid solution, the FTS solution provided the best corrosion resistance with an average corrosion rate of .47 $\mu\text{m}/\text{day}$. However, it had the worst corrosion resistance in the sodium chloride solution with a .42 $\mu\text{m}/\text{day}$ average corrosion rate. This Bis silane solution had the best corrosion rate in the sodium chloride at .12 $\mu\text{m}/\text{day}$ and an average corrosion rate of 1.12 $\mu\text{m}/\text{day}$ in the oxalic acid solution. All of the silane solutions provided some corrosion resistance compared to the untreated aluminum alloy coupons.

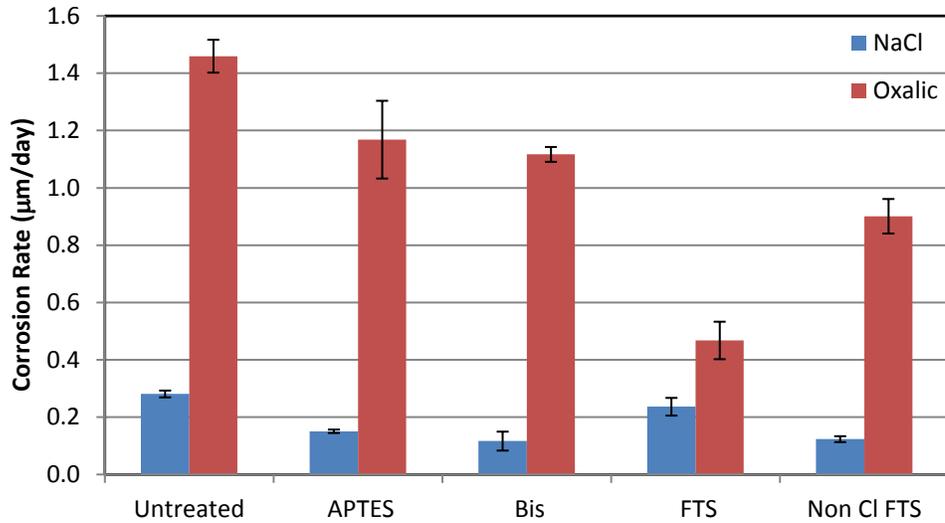


Figure 2. The corrosion rates of aluminum alloy in 3.5 wt.% NaCl solution and .07 wt.% oxalic acid solution of untreated AA2024-T23 coupons and coupons treated using four different silanes (APTES, Bis, FTS and non Cl FTS).

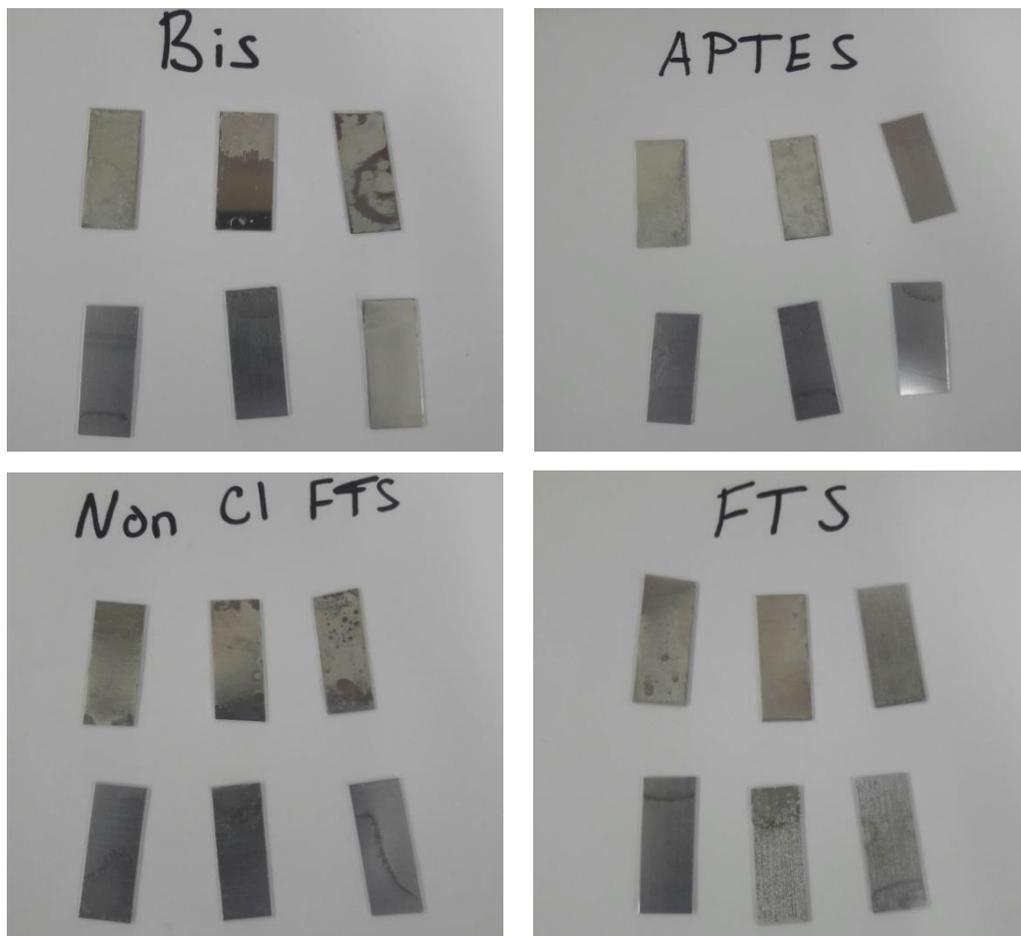


Figure 3. Pictures of the aluminum alloy coupons after they went through the corrosion test with the four different silanes. In each of the pictures, the top three pictures were in NaCl and the bottom three were in oxalic acid.

After the coupons were pulled out of the corrosion solutions, any corrosion product was brushed off and then cleaned in an acid bath. The pictures of the aluminum alloy coupons can be seen in **Figure 3**.

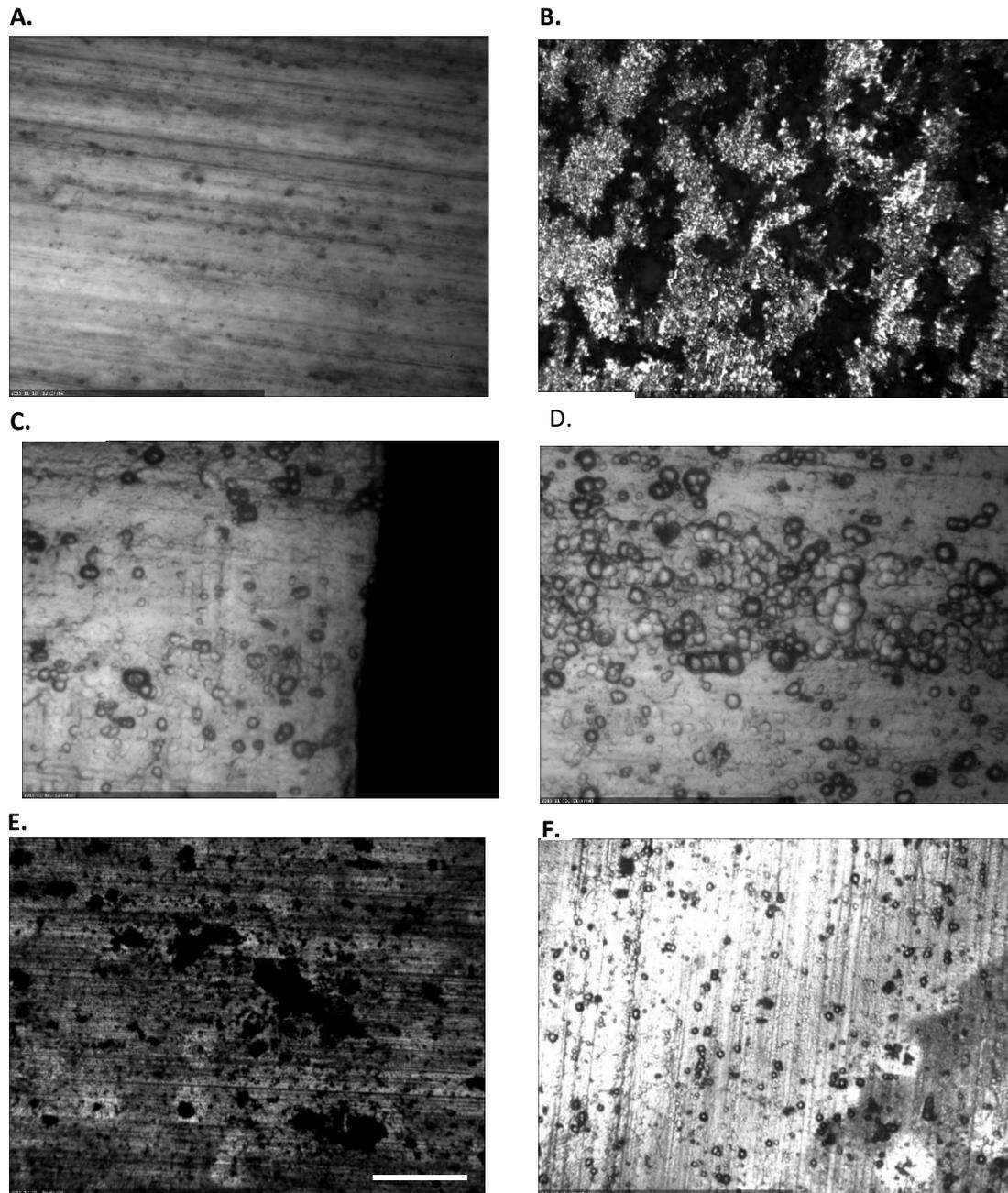


Figure 4. Optical microscopic images of the AA2024-T3 coupons. A. Untreated AA2024-T3 before corrosion test. B. Untreated aluminum after corrosion test. C. APTES surface after corrosion test. D. Bis surface after corrosion test. E. FTS surface after corrosion test. F. Non Cl FTS after corrosion test. All of the corrosion tests were in a .07M oxalic acid solution. All images have the same size, and the scale bar is 200 μm .

The morphology of the AA2024-T3 coupons after corrosion test using 0.07 M oxalic acid can be seen in the optical microscopic images shown in **Figure 4**. In the untreated aluminum, the lines from the metal are very visible as well as the normal defects in the metal from cutting. The untreated aluminum that was subject to the corrosion test was severely corroded and showed very large pits. All of the coupons that were treated with a silane coating still exhibited some corrosion and pitting, but not as much as the untreated samples.

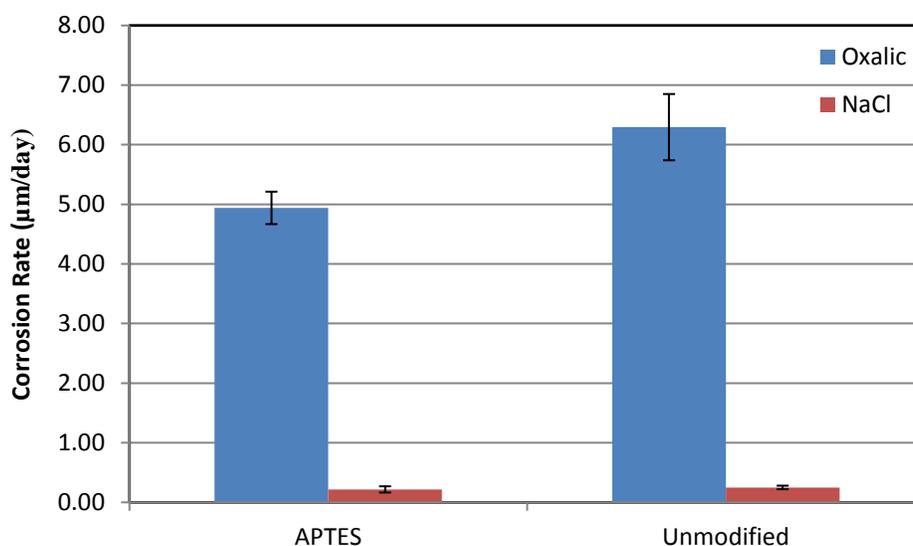


Figure 5. The corrosion rates of magnesium alloy coupons that were unmodified and modified with APTES.

Magnesium alloys were also tested to see if the corrosion resistance can be improved with an APTES coating. The corrosion results can be seen in **Figure 5**. The magnesium reacted considerably with the oxalic acid and began dissolving immediately upon entering the oxalic acid solution. The average corrosion rate of the modified APTES magnesium coupon in the oxalic acid was 4.9 µm/day while the untreated was 6.3 µm/day. The magnesium coupons under an optical microscope can be seen in

Figure 6. The untreated magnesium before the corrosion test had defects from the cutting of the sample. The modified and unmodified magnesium after the corrosion test did not have noticeable pitting because the surface was dissolving, so the surface actually became smoother after the test.

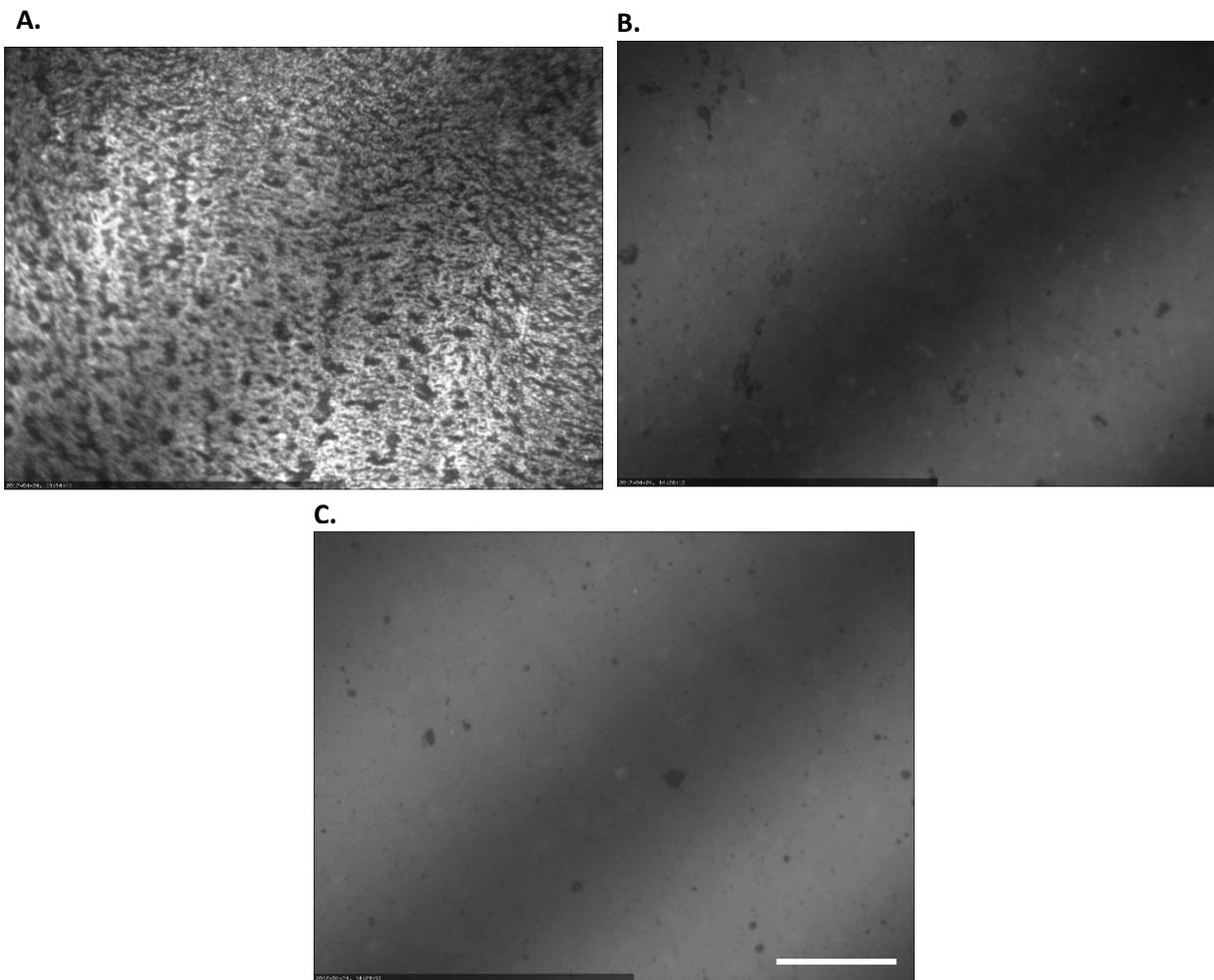


Figure 6. Optical microscopic images of magnesium alloys AZ 31. A. Untreated magnesium alloy surface before corrosion test. B. Untreated magnesium alloy after corrosion test. C. Magnesium alloy treated with APTES after corrosion test. All the images are the same size, and the scale bar is 200 μm .

Discussion/Analysis

In order to determine the best method for coating the coupons, a corrosion weight loss test was done on coupons that were dipped in silane solution versus coupons that were submerged for 24 hours. The more common practice for applying the coating in the past has been using the dip method. However, throughout the experiment when the coupon was dipped in the solution, upon drying, the coupon had a very inconsistent layer that wasn't very even. On the other hand, the solution deposition method gave a very even coating on the surface of the coupon which provided a better corrosion resistant layer. The coupons that were coated using the solution deposition method had a lower corrosion rate than those coated with the dip coating approach. Also the error bars on the dip coated coupons were larger than those coated using the solution method because of the inconsistency with the coatings.

The contact angle for all of the aluminum coupons were recorded before and after the corrosion test. Those values can be seen in **Table 2** and **Table 3**. The contact angle was measured before the corrosion test to see if the silane coating was applied to the surface and it was measured after the corrosion test to see if the coating remained on the surface after being exposed to a corrosive environment. If the silane coating was applied to the surface of the aluminum, the surface would be hydrophobic and the contact angle will be quite large. Each silane sufficiently coated the aluminum alloy because the contact angle for all of them was greater than 50° showing the surface was hydrophobic. After the aluminum coupons were subject to the corrosion test, the

contact angle did not decrease much in most cases, indicating the coating for the most part remained on the surface. The greatest decrease in the water contact angle was observed for the coupons coated with non-Cl FTS after the corrosion testing, which could be the results of weaker bonding or less coverage of non-Cl FTS layer, since the hydrolysis of non-Cl FTS could be less (or takes longer) than the chlorinate FTS [14].

The corrosion rates for AA2024-T3 coated with different silanes were presented in **Figure 2**. The coupons went through a corrosion test in either .07M oxalic acid or 3.5 wt.% NaCl. The silane that showed the most corrosion resistance in the oxalic acid solution was FTS, but it had the highest corrosion rate in the NaCl solution. The Bis silane gave the best corrosion resistance in the NaCl solution, but it was about average against the oxalic acid solution. The FTS had a statistically significant difference in corrosion rate while the Bis silane in NaCl did not have a significant difference. However, all of the silanes that were coated on the aluminum coupons gave a better corrosion resistance than the unmodified surface. All four silanes provided a better corrosion resistant layer than the untreated aluminum oxide surfaces on the coupon in corrosive environments.

The APTES silane solution was also applied to some magnesium alloy coupons. When the magnesium alloys were submerged in the oxalic acid solution, they immediately reacted and began dissolving. However, the magnesium alloy coupons coated with APTES had a lower corrosion rate than the untreated magnesium alloy coupons. The modified surfaces also performed better in the sodium chloride solution.

The magnesium coupons that went through the corrosion test in the oxalic acid did not exhibit any noticeable pitting. However, because the surface was dissolving the surface was considerably smoother.

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