# Green Anodizing Process for Aerospace Applications

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## WeARE Research Area

- Materials Engineering
- Corrosion Engineering
- Electrochemistry
- Anodization

## Motivation or Background

Corrosion is everywhere and never sleeps. Corrosion impacts not only the economy but also people's safety as well as the environment. The economy is adversely affected by corrosion due to the increase of cost that comes with the inadequate planning of the maintenance, which is required and of paramount importance. Failing or faulty equipment or infrastructure can lead to serious injury or even death. Failing or faulty equipment for chemical and oil storage and transport can cause major damage to the environment [1]. Therefore, it is very important for engineers to consider the effects of corrosion and to implement corrosion control methods. Aluminum alloys are commonly used in aerospace applications, but the oxide layer that is formed atmospherically needs to be increased in thickness to augment the corrosion resistance of the structure.  One control method used in this situation is the anodizing process. Current technologies contain harmful chemicals that deleteriously impact the environment and the health of the people dealing with the chemical. Therefore, the use of more sustainable green materials is currently being explored. In this project, mixtures of acid and natural substances with low environmental impact were used to produce anodized aluminum and the resulting organic coating adhesion to the substrate was tested.

## Objective

***Evaluate the effectiveness of green anodizing process on increasing the adhesion strength of commercial Epoxy coating on aluminum alloy 7050***

## Methodology

**Polishing**
Two sizes of aluminum samples were cut into squares: 5x5 $cm^2$ and 10x10 $cm^2$. These samples were then polished with SiC papers grit size of 60 to 1200 followed by polishing with cloth and a 1µm diamond suspension solution. The samples were then cleaned with soap, 5% NaOH solution, and 25% $HNO_3$ solution. After polishing, a copper cable was adhered to a corner of each sample with epoxy.

**Anodizing**
The samples were then anodized by placing samples in a cell and running a current through them (Figure 1), according to previous literature [2]. For each solution, the current was set to 1 Amp and the anodizing process lasted one hour. Within the cell, three electrodes were used. One was used as the anode (sample to anodized) and two were used as cathodes. Furthermore, a Mercury/Mercury Sulfate reference electrode was used to measure the electrochemical potential. The solutions for the anodizing process included oxalic-citric solution, sulfuric acid with triethanolamine (TEA), and sulfuric acid with TEA and an extract from the plant Caesalpinia Coriaria, also known as dividivi. For the oxalic-citric solution, the concentrations of the acids were 10% for citric acid and 4% for oxalic acid. For the sulfuric acid with TEA, the concentrations were 10% m/v Sulfuric acid and 1000ppm of TEA. For the sulfuric acid with TEA and dividivi, the concentrations were 10% m/v Sulfuric acid and 1000ppm of a mix between TEA and dividivi. The TEA and dividivi mixture was made by adding 42.4 mg of dividivi to 0.89 mL of TEA and heating the mixture at a low temperature. 0.8 L of the TEA and dividivi mixture was used in the 1 L solution.

**Adhesion Strength measurements following ASTM D4541 [4].**
After anodizing, the thickness of the samples were measured. Then a coating (02GN084 Chrome-Free Epoxy Polyamide Primer) was applied by Southwest Research Institute (SwRI). The coating was chosen based on PPG and SwRI's recommendations. For the adhesion testing, dollies were adhered to the coated samples using a DEVCON 2 Ton epoxy adhesive following the ASTM D4541 standard as shown in Figure 3. The test was performed using a Positest AT-A adhesion tester (Figure 4).

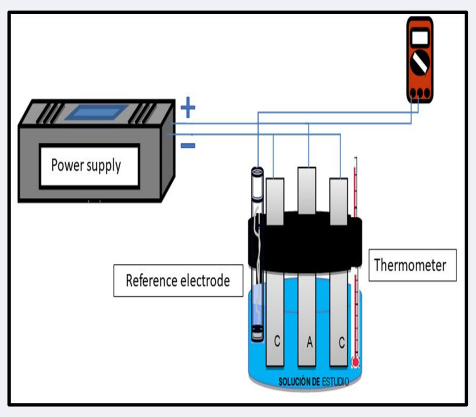


Fig. 1
*Diagram of the electrochemical cell*

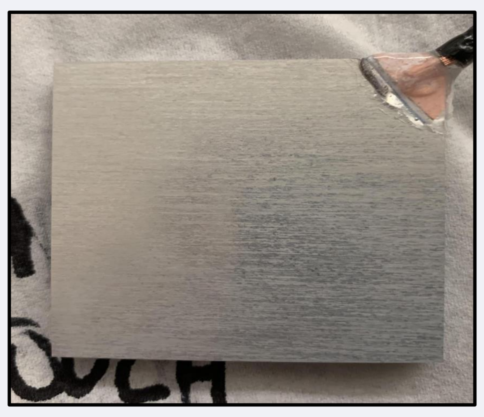

Fig. 2
*Sample after the anodization process*

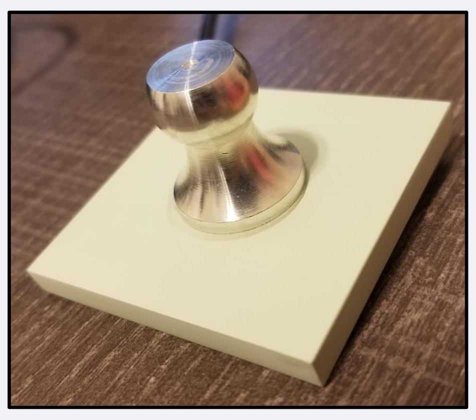

Fig. 3
*Sample with Adhered dolly for pull-off test following ASTM D4541.*

Fig. 4
*Positest AT-A adhesion tester.*

## Results

Figures 5 through 10 depict the different samples after the pull-off test was performed. The table in Figure 11 contains the data from the pull-off test. Figure 12 shows the adhesion strength (Pressure) vs. Time (Duration) plot from the pull-off test for Sample #5 from the Sulfuric Acid with TEA and dividivi solution. As can be seen in the Figures and Tables no adhesive failure in the substrate coating interface was obtained and therefore the values provided do not represent the adhesion strength of the coating to the substrate.

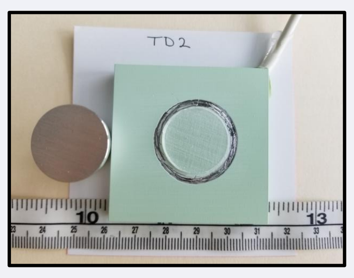

Fig. 5
*Sample #2 from Sulfuric Acid with TEA and dividivi solution post pull-off Test*

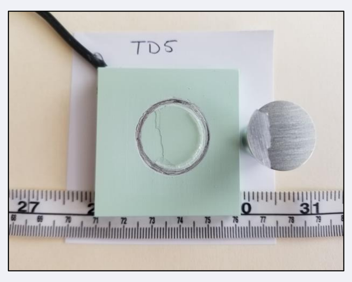

Fig. 6
*Sample #5 from Sulfuric Acid with TEA and dividivi solution post pull-off Test*

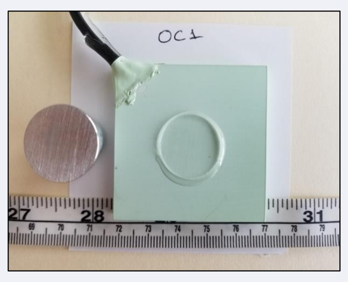

Fig. 7
*Sample #1 from oxalic-citric solution post pull-off Test*

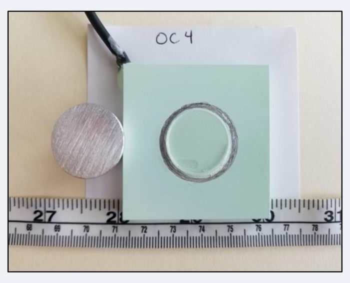

Fig. 8
*Sample #4 from oxalic-citric solution post pull-off Test*

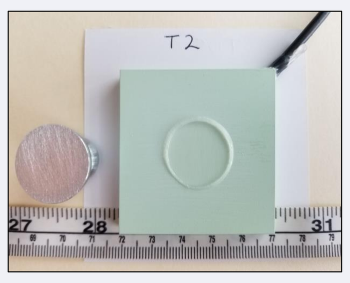

Fig. 9
*Sample #2 from Sulfuric Acid with TEA solution post pull-off Test*

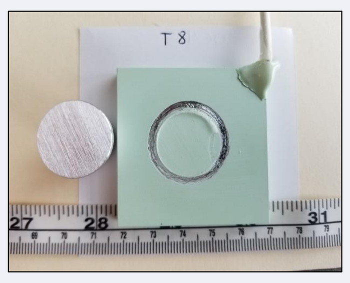

Fig. 10
*Sample #8 from Sulfuric Acid with TEA solution post pull-off Test*

The pull-off test did not go according to plan because the epoxy adhesive used was not as strong as needed to sustain the energy needed to deadhere the coating from the substrate and obtain values of adhesion strength. Improving the ratio used for the adhesive mixture could improve the test.

Table 1. *Average anodized and coating thickness, and pull-off test results following ASTM D4541.*

| SPECIMEN | ANODIZING (µm) | ANODIZING + COATING (µm) | PULL-OFF (psi) | FAILURE LOCATION* |
|---|---|---|---|---|
| TD2** | 10.8 | 62.12 | 504 | D/A 95%, A/C 5% |
| TD5 | 10.4 | 56.37 | 600 | D/A 60%, A/C 40% |
| OC1** | 14.0 | 54.87 | 588 | D/A 100% |
| OC4 | 14.2 | 58.12 | 541 | D/A 100% |
| T2 | 10.2 | 67.50 | 552 | D/A 100% |
| T8 | 9.8 | 52.50 | 516 | D/A 90%, A/C 10% |

*D/A: adhesive failure between the dolly and the adhesive. A/C: adhesive failure between the adhesive and the coating
**coating surrounding dolly was not removed

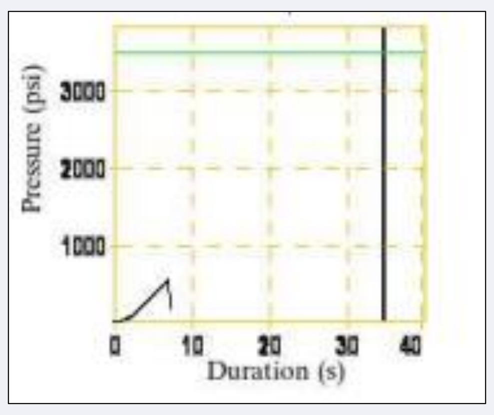


Fig. 12
*Table of pull-off test results following ASTM D4541.*

## Skills and Experience

- Sample preparation.
- Materials Engineering Lab.
- Anodization Process.

## What I Learned

- Basic Corrosion. I attended to the ME 4683 Corrosion Engineering class.
- Polishing methodologies.
- Anodizing processes.
- Electrochemistry basics.

## Future Plans

- Continue to learn more about corrosion while continuing this project during Fall 2020.
- Improve adhesive mixture for pull-off test.
- Anodize 10x10 $cm^2$ samples
- Coat 10x10 $cm^2$ samples.
- Perform Electrochemical testing on coated samples.
- Execute accelerated corrosion testing based on ASTM B117 [5].

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- Support from BOEING,PPG and SwRI in terms of technical guidance, products (PPG) and coating application service (SwRI) is appreciated.

## References

[1] Jones, D. A. (1992). Principles and prevention of corrosion. Macmillan.
[2] Romero, N., Troconis de Rincón, O., Millano, V., Gallo, J., López, G., Polo, E., & Linares, D. (2016). Use of Mixtures of Acid and Natural Substances with Low Environmental Impact in the Production of Anodized Aluminum (UNS A93003) . NACE International Corrosion Conference Proceedings.
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[4] American Society for Testing and Materials. (2017). Standard Test Method for Pull-Off Strength of Coatings Using Portable Adhesion Testers.
[5] American Society for Testing and Materials. (2011). Standard Practice for Operating Salt Spray (Fog) Apparatus.