Evaluation of Atmospheric Plasma Treatment Detection Methods for Thermoset Substrates

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Introduction and Background

The aerospace industry has increasingly turned to carbon fiber composites as a primary material to build lighter and more fuel-efficient planes [1]. The Boeing 787 Dreamliner has parts that are primarily made of carbon fiber [2]. These composites facilitate enhanced structural adhesive bonding, which increases the mechanical properties of the aircraft by distributing stress more evenly across the bonded area [2]. Traditional materials, such as metals, rely on mechanical fasteners, rivets, and spot welding, which add weight and create stress concentrations that weaken the overall structure [1][2]. In this project, we used a composite made of carbon fiber and thermoset resin. Prior to bonding the composite materials, surface preparation is required. Surface preparation (Figure 1) ensures a high-quality bond between the substrate and the adhesive. Plasma treatment increases the surface energy of a material by depositing plasma onto a surface. Plasma is generated (Figure 1) by exposing compressed air to extremely high voltages, ionizing the air and creating charged particles. When these particles contact the composite surface, they form free radicals and carboxyl groups, which improve adhesion. However, these changes are not visible to the naked eye.

Project Statement and Objectives

Boeing tasked us with developing methods to analyze the plasma-treated surface of thermoset composites using non-destructive techniques that, ideally, do not weaken the bonding strength of the surfaces. The constraints of this project were to devise methods that are portable, cost-effective, accurate, and do not affect the bond strength of the thermoset composite surface. We initially explored three plasma detection methods: dyne pens, water contact angle, and FTIR. This project presents an opportunity to create a method for field testing during on-site repairs of thermoset composite materials.

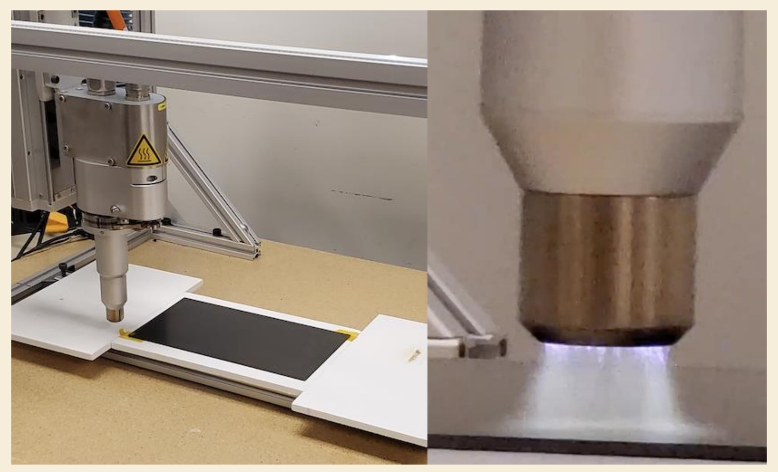

Figure 1: Plasma treatment apparatus located in MEB and a close up of the plasma being applied to the surface.

Methods

Large panels of thermoset were plasma treated, the detection method was applied, and then the panels were bonded. Once they cured, they were cut into the desired coupon size. Then a diamond blade saw was used to cut the fixture notches into the coupons.

Detection Method 1: Water Contact Angles (Figure 2)

- Measures the angle of a water droplet when placed on a surface.

Pros:
- Relatively simple test
- Quantitative
- Portable

Cons:
- Requires flat and level surface
- Contact method

Detection Method 2: Dyne Pens

- Fill pen with deionized water to visually determine surface energy.

Pros:
- Easy to administer
- Qualitative
- Cheap
- Very portable

Cons:
- Non-quantitative
- More abrasive
- Contact method

Figure 2: Water contact angle apparatus courtesy of Professor Aniruddh Vashisth

Detection Method 3: Fourier Transform Infrared Spectroscopy (FTIR)

- Spectroscopy method often used to identify atomic groups and materials.

Pros:
- Portable methods exist.
- Very precise and analytical measurements

Cons:
- Portable method may be expensive

Mechanical Testing: ASTM 5528 Double Cantilever Beam Test (Mode I)

This test assesses the bonding strength by pulling apart the bonded panels. Panels were bonded using EA 9696 film adhesive. Test was performed using the Instron mechanical testing apparatus (Figure 3). Data was collected from coupled software. From each panel, five 1in x 8in coupons were cut out via water jet.

Figure 3: DCB Test of Bonded Panels

Timeline

W - Weeks 1-10	S – Weeks 1-5	S – Week 6	S – Weeks 7-8	S – Weeks 8-9
R&D	Instrument Training	Experiment: Detection Methods	Experiment: Mechanical Testing	Final Report and Presentation

Results and Discussion

Water Contact Angles

Due to the low surface energy of the thermoset, the resulting water droplets had an **average water contact angle of 71.2°**. However, after plasma treatment, the average water contact angle decreased significantly to **15.7°**. It was also found that after cleaning the plasma treated thermoset with a Kimwipe, the plasma treatment degraded as the water contact angle increased to **27.3°**. A visual difference can be seen in Figure 4.

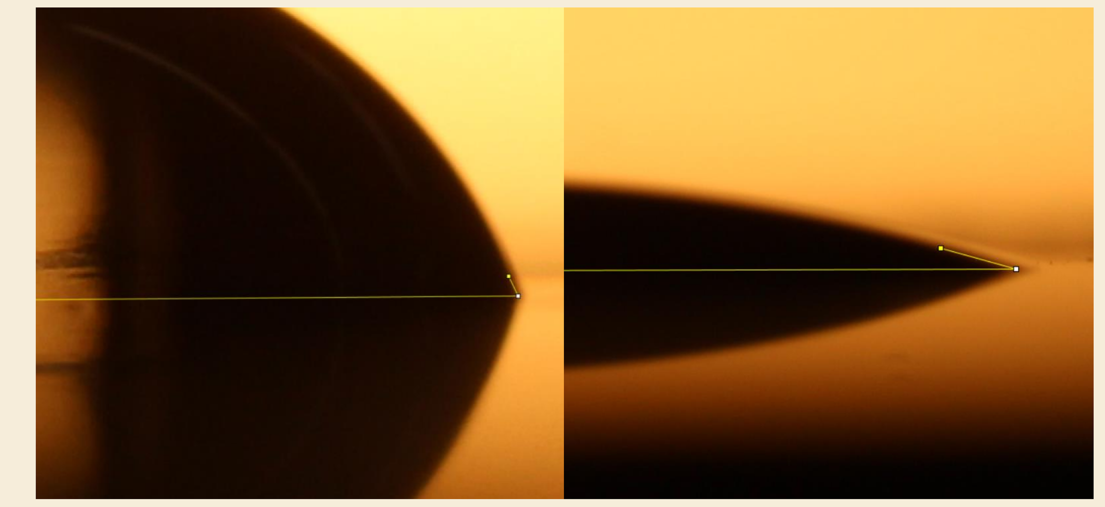

Figure 4: Water contact angles before plasma treatment (left) and post- treatment (right)

Dyne Pens

Dyne pens serve more as a visual and qualitative indication of plasma treatment. The liquid used in the dyne pens was deionized water. As can be seen in Figure 5, prior to treatment, the water placed on the sample beaded up immediately, indicating a low surface energy. Post-treatment, the water spread and wet the surface much more effectively. See Figure 5.

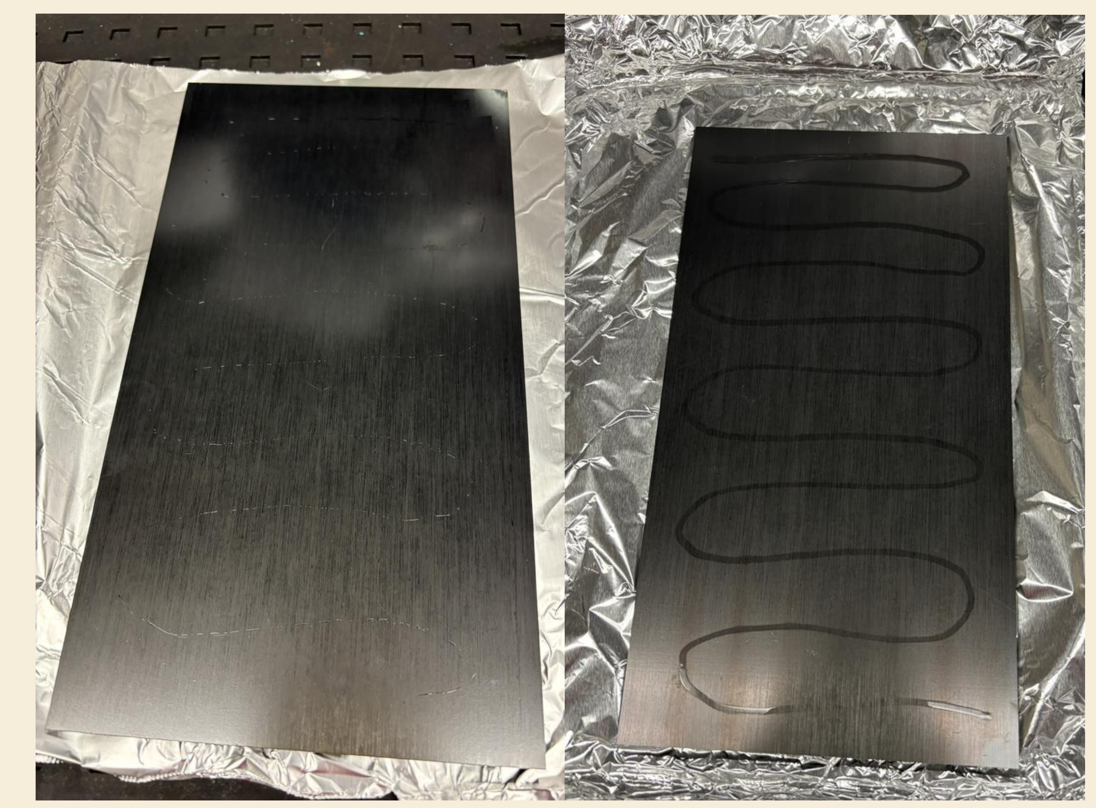

Figure 5: Dyne pens before plasma treatment (left) and post-treatment (right)

Fourier Transform Infrared (FTIR)

Five spectra were collected for each non-treated and treated thermoset sample. The main goal of this particular detection method was primarily to determine if FTIR spectroscopy can detect any noticeable changes in the thermoset after plasma treatment. Thus, the FTIR sample was not used for bonding. The averaged spectra for each sample is shown in Figure 6. There are very slight differences in the spectra. However, since the percent difference between specific treated and non-treated spectra peaks are the same for each peak, this is most likely due to the amount of contact made with the thermoset and the FTIR crystal. This inconsistent contact may have been caused by the rough peel-ply layer on the backside of the thermoset samples. Thus, it was concluded that there were not enough significant differences between non-treated and treated spectra to validate FTIR spectroscopy as a potential detection method.

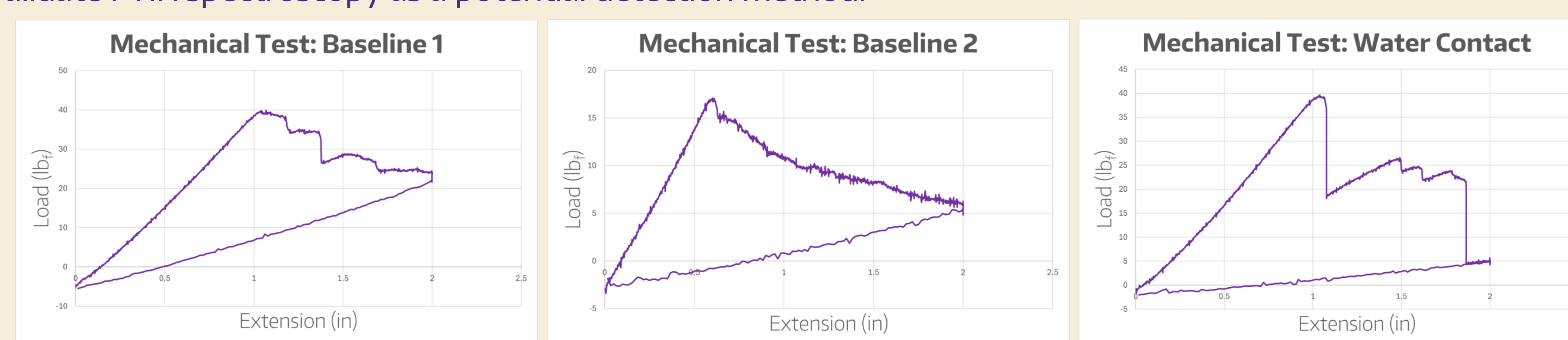

Figure 7: The graph of the fracture energy of Baseline 1(left). Then Baseline 2(middle) and Water contact on the right.

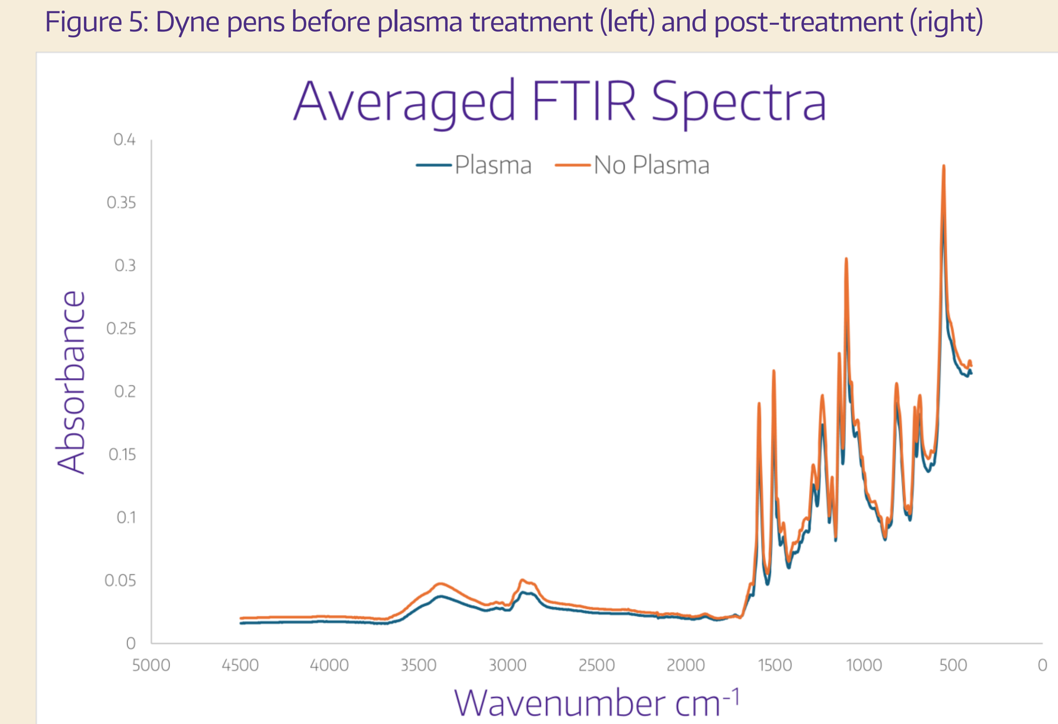

Figure 6: FTIR Spectra of the sample before and after plasma treatment. No difference

Mechanical Testing

Each detection method, except for FTIR, had five coupons for mechanical testing. Unfortunately, about 80% of all coupons failed prematurely through *delamination.* The data for those that were successful can be seen above. The area of the graph (Figure 7) represents the *work* done to separate a coupon. Dividing that by the crack area on the coupon yields the **fracture energy**. As can be seen in Table 1 , there is a lot of variability in the data. The initial hypothesis suggested that any sort of contact on the plasma treated sample would result in decreased bonding strength. Further tests would have to be conducted to support this.

Table1: Data collected after performing DCB Test

Sample	Fracture Energy (lb*in/in^2)
Baseline 1	22.5
Baseline 2	5.0
WCA 1	10.8

Looking Ahead

We believe that the delamination of our sample was due to poorly cut or damaged edges, which might have created stress concentrations. It is possible that the use of waterjet cutting introduced some edge defects. Another plausible cause of delamination could be the thickness of our laminate. These samples were also much thinner, thus creating the fixture notch was significantly more difficult. The 1 in coupons may have also contributed to this as it is harder to create a notch with a wider coupon. Lastly, the composite material itself could be a factor, some of the samples had defects when they arrived. Moving forward, we recommend using a different cutting tool to prepare our samples. We should also use thicker and narrower coupons. Additionally, we would request Boeing to potentially improve the processing of the coupons

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[1] Bowler, T. (2014, January 28). Carbon Fibre Planes: Lighter and stronger by design. BBC News. https://www.bbc.com/news/business-25833264

[2] Joining & bonding of composite parts – the structural ... (n.d.).https://multimedia.3m.com /mws/media/1586280O/iatd-joining-and-bonding-structural-adhesives-white-paper.pdf