Experiment 3: Measurement of the Bonding Strength of METLBOND on Carbon Fiber, Aluminum, and PTFE in Shear

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ME 411 Engineering Measurement and Instrumentation Systems

Portland State University

2017-03-21

Workload Distribution

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Introduction

The purpose of the experiment is to measure the shear strength of an adhesive called METLBOND 1515-3M (Grade 5). This material was donated to Portland State Aerospace Society and is to be used by our capstone team in a prototype of a composite cryogenic fuel tank. The prototype will be made primarily from three materials. These materials are resin-impregnated carbon-fiber (CF), PTFE, and Aluminum. We hypothesized that this adhesive could potentially be the critical failure mechanism in our current design. Some properties of the adhesive were provided by the manufacturer (see appendix A), however, shear strength data is dependent upon the materials being bonded, and there was no available test data regarding the interface of CF and Aluminum, or PTFE and Aluminum.

To test the shear strength of the METLBOND adhesive, two sets of test coupons were developed for tensile testing--one of Aluminum ends and lapped CF, and one of Aluminum ends and lapped PTFE. The coupons were subject to tensile testing, so that the adhesive experienced a stress that approximates pure shear, which is the type of stress we expect to see applied to the pressurized fuel tank. By measuring the force applied at failure to a known shear area, we can infer the ultimate shear stress for the material and compare it to stresses expected in the current tank design (~44 psi internal pressure). This knowledge will help us decide if we need to increase or decrease the lapping areas in our tank interfaces or make different material or design decisions to mitigate the risk of failure in shear.

Theory

Stress cannot be directly measured, but can be inferred by measuring force and the area over which the force is applied. Materials experiencing stress will also experience some strain, but for this experiment we are only interested in the ultimate stress at which our material fails. A tensile force can be applied by hanging a weight from a specimen, or by placing it into a tensile testing machine. In the first case, an accurate force measurement can be obtained by slowly filling a hanging container with a liquid and measuring the weight it takes to cause failure in the test specimen using an off-the-shelf strain-gauge scale.

Stress is defined as the force applied per unit area of application. In this case, the stress applied to the adhesive, σ_{bond}, is dependent upon both the weight of the hanging liquid and the container apparatus, $W_{container}$:

$$\sigma_{bond} = \frac{W_{liquid}+W_{container}}{A_{bond}} \quad [1]$$

Where A_{bond} is the area of the bond material.

If the test sample is subjected to pure shear loading, where the applied force is perfectly parallel to the interface in question, then equation 1 can be used to calculate the shear stress on the test sample without any consideration given to other stress modes.

<u>Methods</u>

Equipment

The required equipment includes: Several custom-made aluminum/carbon fiber and aluminum/PTFE test coupons (see Figure 1), three s-hooks, two eight-inch chains, a frame to hang the coupons from, a one-gallon jug, a six-gallon graduated bucket, a 32 gallon watertight bin, a water source, and a scale (with an associated uncertainty of +/- 0.1 lbs).

Setup

Two sets of test coupons were prepared. A set was made for each material interface and test type combination. Each test coupon was made using two rectangular pieces of aluminum cut from the same piece of 0.25” thick plate. A hole was drilled into each aluminum piece along its vertical axis to allow a hook to pass through.

Figure 1: Test Coupons-- Aluminum & CF (Left), Aluminum & PTFE (Right).

The material being bonded to the aluminum was lapped over both pieces, on both sides, as shown in Figure 1, for a total of four bonding zones per coupon[1].

For each coupon, the four bonding zones were equal in area. This was accomplished by cutting 0.25” x 0.25” squares of METLBOND film for the carbon fiber coupons.. The size of the aluminum PTFE interface was controlled by cutting the PTFE strips 0.25” wide and marking 0.25” from the edge (marking the edge of the bonding surface). The Adhesive film portions were measured out with a ruler, marked with a Sharpie, and cut using a razor blade. (The accuracy of these areas was restricted by half the resolution of the ruler used, 1/32”, resulting in an area uncertainty of +/- 0.015 in^2 per bonding zone, or +/- 0.06 in^2 per test coupon). Once the coupons were assembled, they were wrapped in a special release film, further wrapped in a breather fabric, vacuum sealed, and cured in an oven (see Figure 2) using a time and temperature regime developed by a previous PSAS airframe capstone team and optimized for maximum adhesion[2].

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1 Also shown in figure: hole-free coupons to be tested with a tensile testing machine at a later date.

2 https://github.com/psas/sw-cad-carbon-fiber-process/blob/master/LayupInstructions.docx

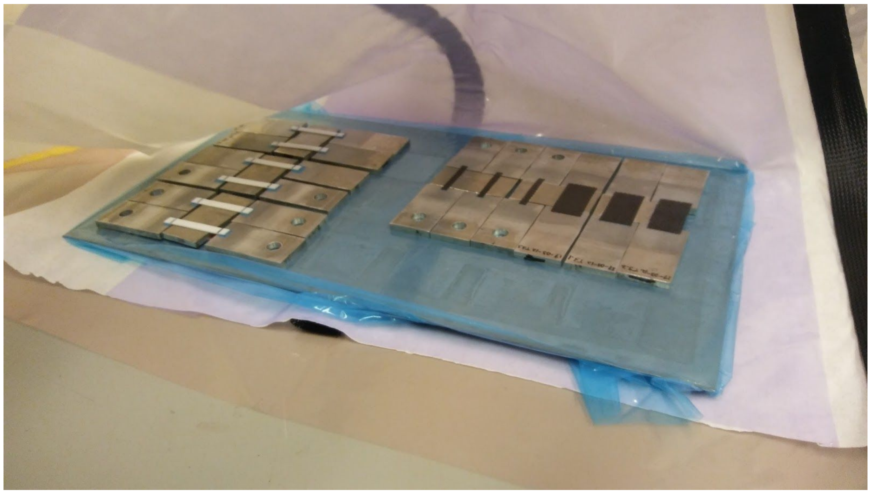


Figure 2: Pre-oven prep- release film (blue), breather fabric (white), vacuum bag (pink)

During the experiment, the coupons were hung by one end from a steel support frame using an s-hook. On the other end, a 32-gallon bin was hung using another s-hook attached to two chains, as shown in Figure 3. The chain lengths were altered symmetrically on the hook to ensure that when failure occurred, the testing apparatus would only fall approximately one inch to the ground, eliminating the possibility of water loss or tipping (so that the weight applied to the coupon could later be measured).

Figure 3: Manual Tensile Test Apparatus

Experiment

Hanging Container (Manual Test)

For each replicate of the shear strength experiment, the 32 gallon bin was slowly filled with water until failure occurred. Water was poured into the 32 gallon bin in four gallon increments until roughly half full. The bucket was graduated ahead of time with six-one gallon increments, and only filled to the four gallon mark for each step of the experimental procedure for ease of pouring. Then water was slowly added from a one gallon jug (see Figure 3) until failure of the coupon was apparent (failure was easily visible and audible). Upon failure of the testing coupons, the weight of the loaded testing apparatus (including bin, chains, hooks, and lower-half of test coupon) was measured on an electric scale in order to determine a pound-force subjected to the testing coupon.

Tensile Tester

A formal tensile test was scheduled in the Ondine Materials Lab to compare with our manual test results. Due to a fluid leak, the machine was unavailable until the week of 3/27/17. As such, the testing coupons created for this apparatus will be tested on 3/27 to gather more data to be compared with theoretical values and results obtained from our manual experiment.

<u>Results</u>

Analysis and Calculations

Table 1 represents a summary of our raw results from the manual tensile test experiment along with the calculated stress at failure and an abbreviated failure mode interpretation.

Replicate	Force (lbs)	Calculated Stress (psi)	Failure Mode
PTFE 1 (T4.1)	118	472	Etched Coating Shear separation
PTFE 2 (T4.2)	101	404	Etched Coating Shear separation
PTFE 3 (T4.3)	100.6	402.4	Etched Coating Shear separation
Carbon Fiber 1 (T6.1)	~258.2	~1032	Carbon Fiber failure, mode indeterminate
Carbon Fiber 2 (T6.2)	180.6	722.4	Carbon Fiber failure, mode indeterminate
Carbon Fiber 3 (T6.3)	233	932	Carbon Fiber failure, mode indeterminate

Table 1: Volume, Force, Stress values, and failure modes from manual tensile test

The tensile tests resulted in multiple failure modes, none of which were due to failure at the METLBOND interface, as seen in Figure 4 below. Therefore, METLBOND failure at room temperature can be dismissed as a limiting factor in our design. Further analysis is required to investigate what changes are present at cryogenic temperatures. Despite this, we obtained useful data and were able to assign a bare-minimum failure stress value to the METLBOND (see Table 2, Min Failure Stress).

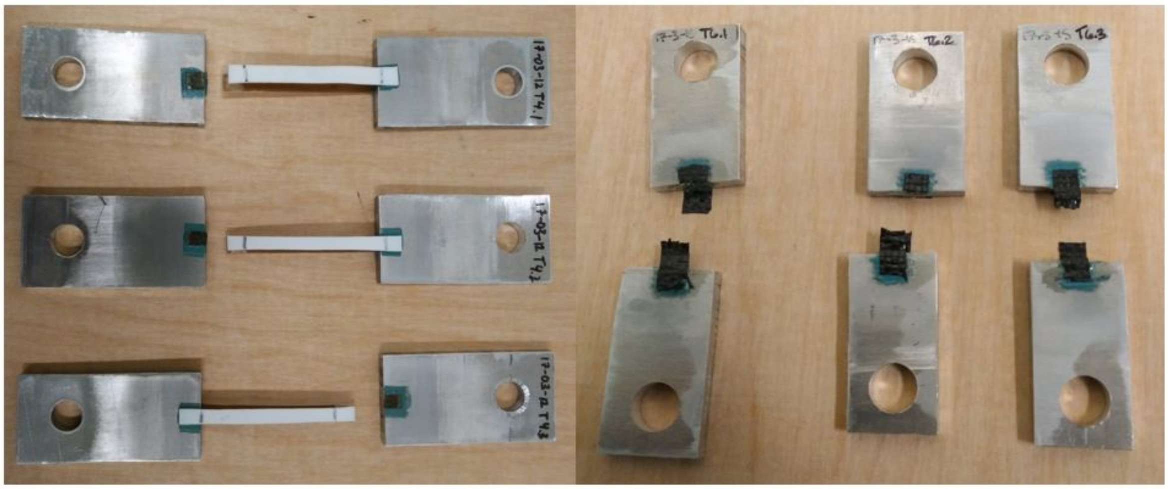


Figure 4: PTFE & Carbon Fiber Testing Coupons Displaying Multiple Failure Modes

The Carbon Fiber tests all failed in the carbon-fiber layer in a suspected tearing mode rather than the hypothesized aluminum-CF adhesion surface. The PTFE failed at the interface between the virgin PTFE material and its etched coating. The etched coating remained adhered to the METLBOND. Failure of these testing coupons can be referenced in Figure 5 below. The 95% confidence minimum failure stress of the PTFE samples can therefore be taken as a conservative ultimate shear stress for the Virgin/Etched PTFE interface. In no cases did the METLBOND separate from the Aluminum.

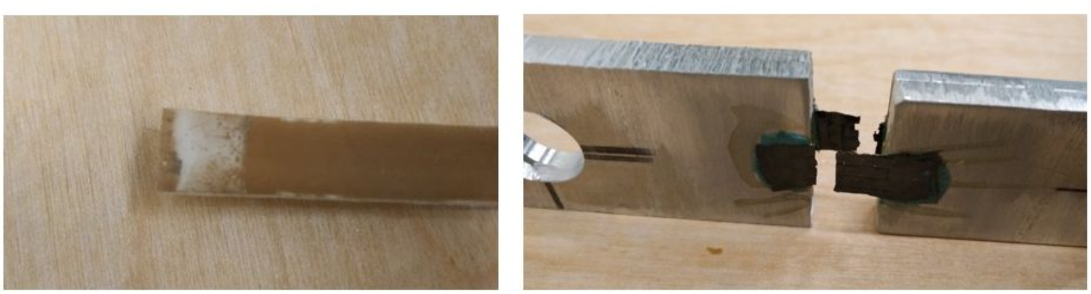


Figure 5: Failure at PTFE Etching (left) & Carbon-Fiber Layer Failure (right)

Shear Stress at Failure		
Analysis:	*PTFE (psi):*	*CF (psi):*
Sample Mean:	426.13	895.47
SD:	39.73	158.00
Degrees Freedom:	2	2
Confidence Level	0.95	0.95
T value	4.303	4.303
Std. Error	22.94 (5.38%)	111.72 (7.07%)
Population Mean	426.13	895.47
Min. Failure Stress	327.43	215.59
Confidence Interval	98.70	679.87
Estimated Internal Tank Stress	44	44
Factor of Safety (N)	7.44	4.89

Table 2: Statistical Analysis of calculated ultimate stress values using confidence interval of 95%

The minimum failure stresses calculated for the PTFE and CF tests both exceeded the estimated internal tank stress (a rough estimate of the internal pressure of the tank) by a factor of safety of 4.89 and 7.44 respectively, well above our capstone's required factor of safety of 2.

Quantifiable Sources of Error

Variation in Area of applied METLBOND:	+/-0.015 in^2
Variation in Weight Measurement:	+/-0.1 lbs

Table 3: Uncertainty range of measured variables

Treating all measured variables as independent, the following linear variance formula (equation 2) was applied to calculate the total uncertainty due to random variance.

$$\delta_\sigma = \sqrt{(\frac{\partial\sigma}{\partial W}\delta_W)^2 + (\frac{\partial\sigma}{\partial A}\delta_A)^2} \quad [2]$$

Replicate	Uncertainty (psi)	Uncertainty %
PTFE 1 (T4.1)	29.50	6.25%
PTFE 2 (T4.2)	25.25	6.25%
PTFE 3 (T4.3)	25.15	6.25%
Carbon Fiber 1 (T6.1)	64.50	6.25%
Carbon Fiber 2 (T6.2)	45.15	6.25%
Carbon Fiber 3 (T6.3)	58.25	6.25%
PTFE Mean Uncertainty:	26.64	6.25%
CF Mean Uncertainty:	55.97	6.25%
Test Uncertainty:		6.3%

Table 4: Uncertainty Values Calculated using equation 2 and values from Table 1 and 3

Discussion

During experimentation, there were notable factors that may have led to error in the data. There was a moderate degree of quantifiable uncertainty (6.3%) inherent to the idealized experiment (see Table 4) , which could account for the majority of the standard deviation found in the PTFE sample tests (Table 2: 5.38%), which failed in shear. But, the calculated uncertainty for the carbon-fiber tests was below the calculated standard error (Table 2: 7.07%) and did not fully explain the high standard deviations in our test values (Table 2: +/- 158 psi). This suggests that our sample preparation technique and test apparatus allowed for a high amount of random and hard-to-quantify error to be introduced to the test, much of which changed the form of stress applied to the system and obscured the impact of pure-shear stress on the test specimens. One such source of error was the inherent sway of the testing apparatus. The goal of the experiment was to measure the tensile strength of each test coupon, but as it was a homemade apparatus, it was noted that the hanging bin would swing and rotate slightly when adding weight to the system. This led the test coupons to additionally experience undesired torsion. This issue was much more visible in the PTFE test coupons, as the plastic strips were cut much longer than the carbon fiber, giving the free-hanging weight much more freedom to rotate. But it had a much bigger effect on the brittle CF which, in the case of test coupon T6.3, caused a clear tear failure as a light kick to the bin filled with water led the apparatus to swing and rotate, leading to instant failure at the center of the CF test specimen.

Another point of error was the integrity of the testing coupons themselves. Due to the nature of the materials and coupon assembly process, maintaining exact dimensions of each material was increasingly difficult. In one particular case, the carbon fiber layers on the coupons were obviously skewed/offset from the center of the aluminum, leading the coupon to experience additional stresses instead of the desired direct tensile. The testing apparatus itself also contributed to this problem, as the mounting bolt would shift to one side upon the addition of sufficient weight, skewing the force on the coupon to be off center. Elongation of the PTFE coupons was also a concern. The material properties of PTFE dictate a minimum 350% elongation to failure, and during the testing process the elongation of the testing coupons was clearly apparent once sufficient weight was added to the bin[3]. This led experimentation to be paused in one instance, necessitating the chain links to be shortened so that the bin did not come to rest on the ground and stop providing a tensile force to the testing coupon.

[3]https://www.matbase.com/material-categories/natural-and-synthetic-polymers/thermoplastics/engineering-polymers/material-properties-of-polytetrafluoroethylene-ptfe.html#properties

Summary/Conclusion

Contrary to our hypothesis, none of the samples failed due to shear in the METLBOND adhesive. Therefore, we were unable to determine the shear strength of METLBOND 1515-3M (Grade 5).

The PTFE samples failed in shear between the PTFE-etched surface interface with an average tensile load of 426.13 +/- 26.64 psi. The carbon fiber samples failed due to an indeterminate loading condition at the carbon fiber sheet where the apparent average tensile load was 895.47 +/- 55.97 psi. When compared to our expected internal tank pressure of approximately 44 psi, the factors of safety for a 0.25 in^2 PTFE-aluminum and carbon fiber-aluminum adhesion surface are 7.44 and 4.89 respectively.

These preliminary results suggest that the METLBOND adhesive is not the critical failure mechanism in our design at ambient temperatures. To validate the loading condition that the coupons experienced, testing will be conducted on 03/27/2017 using a professional tensile testing machine. Additionally, further testing must be conducted to determine the adhesive's tensile strength at cryogenic temperatures.

Appendix:

A. *Extract from METLBOND 1515-3M Material Data Sheet*

Table 5 | Mechanical Properties: Metlbond 1515-3 0.05 lb/ft^2 (242 g/m^2), BMS 8-245B

Property	Substrate	Test Temperature	Result
Metal-to-Metal Lap Shear psi (MPa)	2024 T3 aluminum, no primer Tested at 270°F (132°C)	-65°F (-54°C) 75°F (24°C) 250°F (121°C) 300°F (149°C) 350°F (177°C)	4600 (32) 4700 (32) 3800 (26) 3000 (21) 1800 (12)
Metal-to-Metal Double Lap Shear psi (MPa)	2024 T3 aluminum, no primer	-65°F (-54°C) 75°F (24°C) 250°F (121°C) 300°F (149°C) 350°F (177°C)	5900 (41) 5900 (41) 4700 (32) 3500 (24) 2500 (17)
Composite-to-Composite Double Lap Shear psi (MPa)	Carbon epoxy composite per BMS 8-212	-65°F (-54°C) 75°F (24°C) 250°F (121°C) 300°F (149°C) 350°F (177°C)	4900 (34) 5400 (37) 2600 (18) 1900 (13) 630 (4.3)
Metal-to-Metal Bell Peel lb/in (KN/m)	0.063 in/0.025 in (1.60 mm/0.635 mm) 2024 T3 aluminum, no primer	-65°F (-54°C) 75°F (24°C) 250°F (121°C) 300°F (149°C) 350°F (177°C)	6 (1.0) 16 (2.8) 14 (2.4) 8 (1.4) 4 (0.7)

B. Raw Data: *Live notes from experiment*

CF run 1 (T6.3) - failed @ 12 gallons at CF, didn't fail until applied light 'kicking'/pressure to testing apparatus with suspended bucket 99% full, weight not centered (shifted to one side on connecting bolt)

CF run 2 (T6.2) - failed @ 20 Gallons, slight sway in suspended bucket, but failed after resting for a few seconds following the 20th gallon poured into bucket. Weighed at 180.6 lbs

CF run 3 (T6.1) - failed @ 27 gallons, weighed at 233 lbs. Slight 'impact' upon final bit of 27th gallon being dumped in which caused failure. All 3 failed at carbon fiber, no failure at Metlbond-aluminum interface.

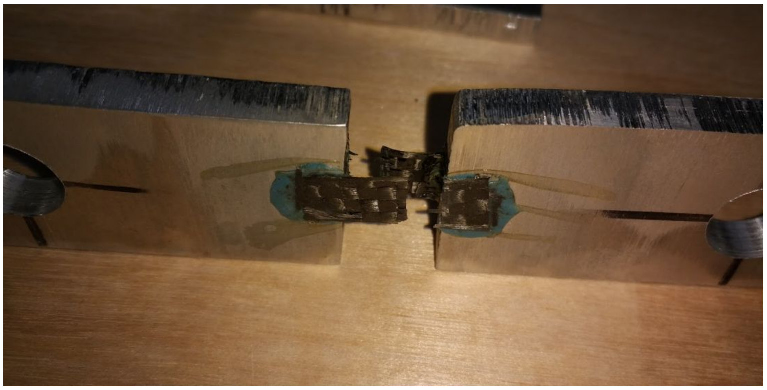
PTFE run 1 (T4.1)- failed @ 12 gallons, weighed @ 118 lbs. Twisting/torsion present during filling, failure appears to be the PTFE etching layer coming off of the PTFE itself as opposed to METLBOND failure.

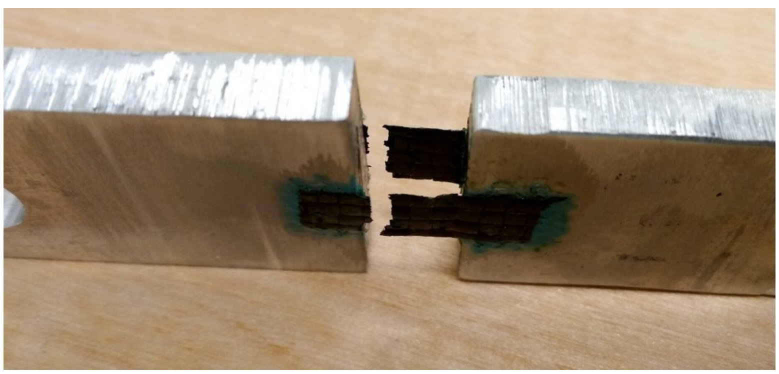
PTFE run 2 (T4.2)- failed at ~10 ⅓ gallons, weighed at 101 lbs. Elongation occurred in PTFE same as 1st run, failure occurred at etching layer on PTFE not METLBOND. @ gallon 10 elongation caused hanging bucket to rest on ground, needed to lift apparatus and shorten chain rung in order to add more water to cause failure.

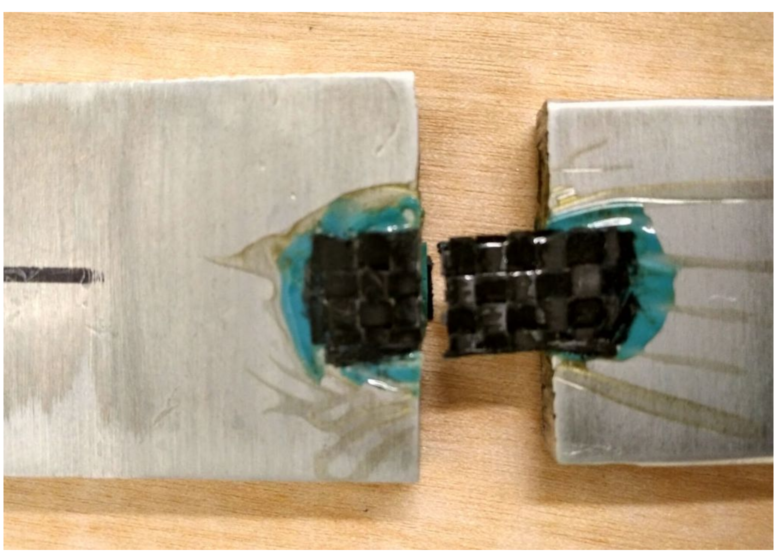
PTFE run 3 (T4.3) - failed @ ~10 ¼ gallons, weighed at 100.6 lbs. Same issues as last runs, etching later failure, ripped off of PTFE.

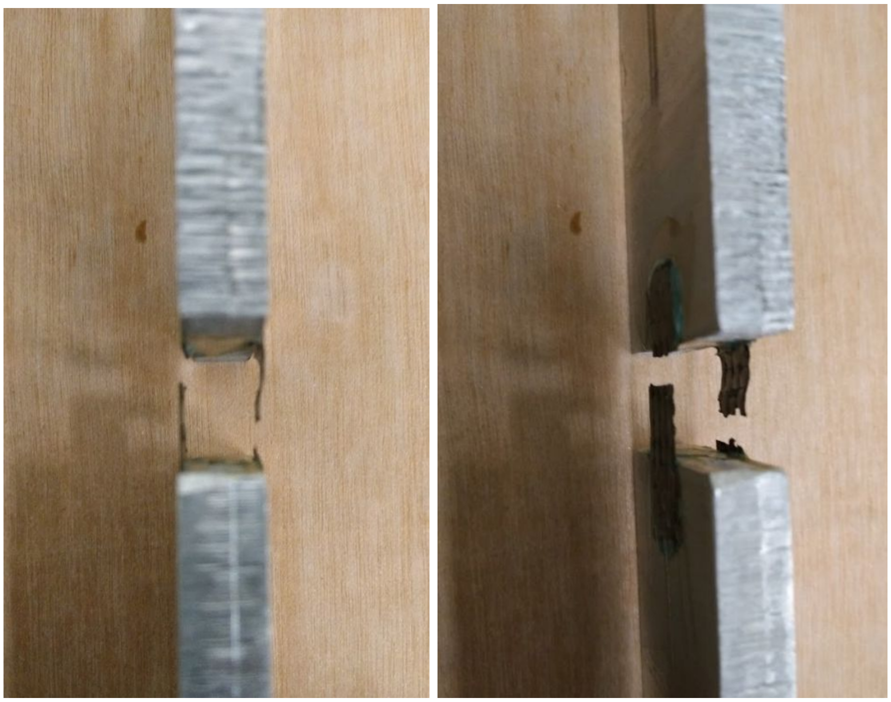
C: Additional Photos of Coupon Failure

Carbon Fiber:









PTFE Etching Failure (Aluminum Side)