A PRELIMINARY STUDY OF USING FILM ADHESIVES CONTAINING ALIGNED AND UNALIGNED NANOTUBES AND NANOFIBERS FOR BONDING CFRP LAMINATES AND STEEL PLATES

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ABSTRACT

Well-dispersed and unaligned multi-walled carbon nanotubes (MCNTs) infused liquid epoxy adhesive have been reported for significantly improving the adhesive-joint of carbon fiber reinforced polymer (CFRP) composite laminates. However, it has not been determined in the literature if the alignment of MCNTs would provide an additional improvement than the randomly aligned case. In this study, various epoxy film adhesives embedded with 1wt% through-thickness aligned MCNTs, unaligned MCNTs, aligned carbon nanofibers (CNFs), and unaligned CNFs were used for bonding CFRP laminates. These variants have been used to bond two CFRP laminates for the ASTM D5868-01 single lap test as well as a steel variant for the same bonding process. The average shear strengths of the samples bonded by the various film adhesives were compared with the samples bonded by the pure epoxy-films. Microscopic analysis has been used to examine the fracture surface after testing. It was also used to visualize how the film adhesives fail while experiencing shear. This study has investigated the effectiveness of infusing through-thickness directionally aligned vs. unaligned nanoparticles in an epoxy film adhesive for bonding CFRP laminates and steel plate. It also indicates the potential future research direction of using nanoparticles in advanced adhesive technologies.

Keywords: Carbon nanotube, Carbon nanofiber, Z-aligned, Randomly-aligned, Film adhesive

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1. INTRODUCTION

Nanomaterials and the products infused with them have become more commonplace in recent years following the discovery of carbon nanotubes (CNT) back in 1991 [1]. CNT and its similar counterpart carbon nanofibers (CNF) are investigated thoroughly to this day. An admirable pairing of either of these materials with adhesive resins have yielded composites which have both high electrical and thermal conductivity as well as a high interlaminar shear strength [2,3,4].

In this study, it is intended to use either CNT or CNF modified epoxy film adhesive to enhance a single lap adhesive joint. In a single lap adhesive joint shear test, the adherends and their surface preparation could also affect the testing results. In the literature [5], several configurations mechanical sanding and chemical baths were used to reduce the inherent impurities of the bonding surfaces for metallic testing coupons. With appropriate bonding surface preparation, they were able to increase the shear strength of their MCNT adhesive film roughly 500% percent with a maximum shear strength of 6.72 MPa, although a direct correlation was impossible due to the differences in bonding area and bond line thickness. In this study, CFRP laminate adherends and galvanized steel adherends were bonded by various epoxy film adhesives containing aligned or unaligned CNT or CNF for the single lap shear tests.

2. EXPERIMENTATION

2.1 Materials

The materials used for this experiment include Toray T700S unidirectional fabric (680 g/m^2 areal weight, 1.8 g/cm^3 fiber density, 12k tow size), MCNT-COOH (COOH-functionalized multi-walled carbon nanotube, Cheap Tubes, Inc.), CNF PR-25-LD-HHT grade (Pyrograph Products/Applied Sciences, Inc.), Epon 862 resin and Epikure-W curing agent (Miller-Stephenson Chemical Co. Inc.), BYK S-191 and BYK S-192 surfactants (provided by BYK), and Tie Down Engineering 59150L Stainless Steel Galvanized Strap (purchased from Home Depot). The CNF used was reported to have a tensile strength of 2.35 ± 0.4 GPa, and a Young's modulus of 245 ± 52 GPa [1] and has a mean length of 50-100 µm as well as a mean diameter of 100 nm. The MCNT was claimed to have a mean length of 10-30 µm and a mean diameter of 20-30 nm.

CFRP laminates were prepared by hand-layup processing. Four layers of T700S fabrics were trimmed just above the ASTM D5868-01 required dimensions for easy sanding with a range of 1 cm to 2 cm extra space and stacked into the out of autoclave-vacuum bag only (OOA-VBO) setup. The Epon 862/Epikure-w resin was impregnated into the fabric using a resin roller. Then the peel-plies and the flow distribution media were arranged. The whole assembly was vacuumed and cured in-between the space of two plates of a hot press. The temperature cycle was 10 minutes at room temperature, 120 minutes held at 120ºC, then demolded and then post-cured at 120 minutes held at 180ºC. The coupon was trimmed into 101.6 mm by 25.4 mm by 2.5 mm CFRP coupons to be bonded with the film adhesive later. The steel strap of 1.0 mm thickness was cut into steel coupons of 31.8 mm by 101.6 mm.

The unaligned MCNT-resin film and unaligned CNF-resin film followed the same procedure for manufacturing with the exception of the nanofiller for each case. For example, to make the batch for aligned and unaligned CNF epon-862 would measure out as 100 wt% of the total calculated mass. Next dispersion chemicals S-191 and S-192 were both measured out for 1 wt% each. Then

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manual stirring was imposed on the batch to mix the ingredients evenly. After the nanofiller, in this case CNF, was measured out at 1 wt% on the scale to get an accurate reading of CNF before mixing. After that high shear mixing and sonication converted the non-homogeneous mixture into a uniform one and lower in agglomerations. The next step was a second degassing, at which point Epikure-W was mixed in at 26.5 wt%. To create the film, the resin undergoes B-staging. This process was monitored for roughly 40 minutes at 120ºC with samples taken in 2 to 5 minute increments. Then the B-staged resin is passed through a proprietary method of creating uniform sheets of film of equal thickness. This marked the end of the unaligned MCNT or CNF modified epoxy film adhesive manufacturing.

The aligned MCNT and aligned CNF film adhesives (i.e., resin films containing through-thickness directionally aligned MCNT or CNF) were produced by placing the unaligned MCNT-film and CNF-film under an electrical field and an elevated temperature, and the MCNTs or CNFs begin alignment [4]. The film was cooled down to solidify the film adhesive to maintain the alignment of MCNTs or CNFs. More details about the resin film processing is described in Section 2.4.

2.2 Single-Lap Shear Test

The standard used in the adhesive-joint test experiment was ASTM D5868-01 which describes the procedure of testing the lap shear bond strength using composite materials. The lap shear bond strength was determined by putting the test sample into tension until fracture (Figure 1). The CFRP coupons measured 25.4 mm by 101.6 mm with a thickness of 2.5mm stated by the standard. It was critical for the bonding site to preserve a 1 inch^2 area to ensure the higher end of possible shear stress, deviating could produce less than optimal measurements in shear stress. The standard required a 13 mm/min loading rate. Prior to testing, the samples were gently fastened into the testing machine. Due to the nature of this step, it was easy to accidentally snap the bonding site and destroy the sample before it could be tested. To avoid this, we had fastened the testing clamps onto the sample grippers without introducing torsion into the anchoring process. The machine used for this test was a Tinius Olsen 10ST universal testing machine.

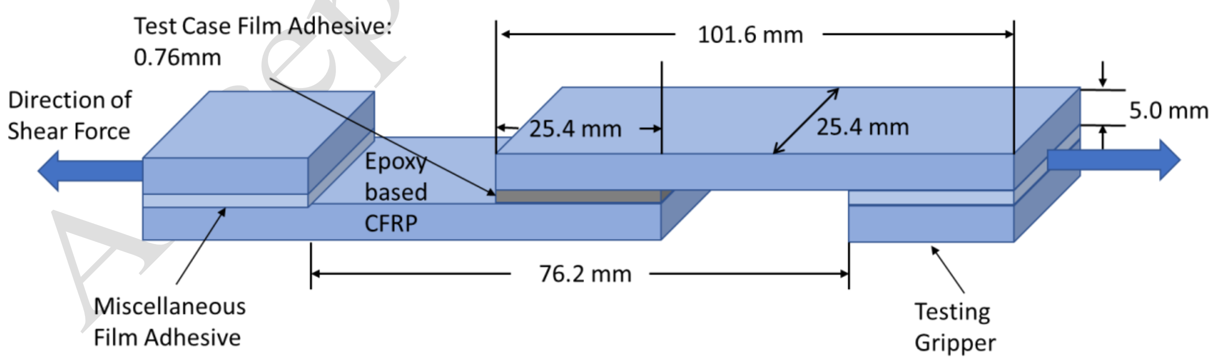

Figure 1: The overall testing dimensions of ASTM D5868-01.

2.2.1 Sample Preparation and Bonding Process:

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The CFRP coupons were split into pairs based on the similarity of the overall dimensions they shared to create stable testing data. CFRP coupons were sanded by 220, 320, 400 grit 3M SandBlaster Advanced Sanding Sheet sandpaper. Further sanding used 800, 1,500, and 3,000 grit 3M Wetordry Sanding Sheets. The steel coupons followed an identical sanding process. Sanding the coupons created a mirror-like shining surface.

All coupons were fitted with their film adhesives. Each assembly was pressed together with medium-sized alligator clips to ensure no shifting to the overall thickness of the film occurred during the curing process. The curing takes place inside an oven at 120ºC for two hours. The coupons and its various film adhesives were post-cured for 2 hours at 180ºC. After the curing, the samples were lightly sanded with a hand Dremel to remove any of the cured film residual from the edge of the assembly.

Post-curing took place later into the sample manufacturing. After curing, the samples were sanded with the sandpaper stated above from 220-400 grit. to achieve a uniform dimension of 101.6 mm x 25.6 mm x 2.5 mm. For the complete experiment focusing on CFRP, at least 30 sample coupons were required to cover 15 tests at three variations: control, aligned MCNT, and unaligned MCNT. Although testing showed a marginal increase in shear strength which yielded a second test using CNF instead. This resulted in recycling ten of the previous tests. These recycled coupons were sanded once again to remove any outside structural damage following the same range of sanding grits used in the original CFRP coupons. Additional leftover coupons were reconstituted into 25.4 mm x 25.4 mm x 5mm cubes which were bonded to the opposite sides of the bonding site using any of the film adhesives.

The number of adhesive-joint assemblies being tested are summarized in Table 1.

Table 1: Coupon layout throughout the experiment.

Experiment	CFRP Tensile Testing					Steel Tensile Testing		
Case Type	Control	A. MCNT	U.A. MCNT	A.CNF	U.A. CNF	Control	A.CNF	U.A. CNF
# Coupons	10	10	10	10 (Recycled)	10 (Recycled)	10	10	10

2.2.2 CFRP Laminate Preparation

The CFRP coupons were prepared and trimmed as described in section 2.2. The sides of the coupons were sanded by using 80 to 400 grit sandpaper of the same 3M SandBlaster Advanced Sanding Sheets listed above in section 2.2.1. until they reached the standard dimensions. The now sanded samples were washed with soap and water, dried in the vacuum oven for 30 minutes at 100ºC, then taken out to cool on a metal plate at room temperature for 10-15 minutes, then the samples were rinsed in acetone and dried off manually. Then the samples were placed back into the 100ºC oven for another 15 minutes to evaporate the remaining traces of acetone. Then the

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coupons were washed again with soap and water and finally placed into the oven at 100ºC for 10-15 minutes for the water to evaporate, then the coupons were taken out of the oven and placed back on the cool metal plate at room temperature. The CFRP coupons were then ready for film adhesive bonding after resting for 2 more hours covered with tin foil to prevent dust interacting with the bond area.

2.2.3 Steel Coupon Preparation

Thirty steel coupons were prepared. A 1 inch^2 region at the end of each steel coupon was sanded with the 220 to 3,000 grit 3M sandpaper listed above in section 2.2.1. The cleaning process is identical to the CFRP laminate preparation. Each of the 30 steel coupons were assigned to another coupon with similar dimensions, mirroring the CFRP coupons, to ensure a uniform force distribution and minimize the inherent moment the bonding site will experience. By following the ASTM D5868-01 standard five test specimens were made for each case conducted (i.e., control, aligned CNF, unaligned CNF, aligned MCNT, and unaligned MCNT).

2.3 Microscopic Morphology Study

The failure modes experienced by the test cases were examined using the microscopy analysis. The microscope pictures were taken with a Nikon Eclipse LV150 upright microscope with ranges from 100x to 3000x magnification.

2.4 Resin Film Adhesive Preparation

The resin film process can be described as follows (Figure 2). At the start 100 wt% Epon 862 and 26.5 wt% Epikure-w was measured out (stoichiometric ratio 100:26.5), the Epikure-w was set aside for later use. Then from the combined weight of Epon 862 and Epikure-w dispersion agents S-191 and S-192 at 1.0 wt% each were added into the Epon 862. For the different concoctions of film adhesive having CNF instead of MCNT, the only difference in manufacturing the film was the nanofiller. The same process was done for both cases at the start. The nanofiller used in this explanation will be MCNT. Once 1 wt% of MCNT was deposited into the resin and then the mixture was gently stirred with a sterile stirring rod to avoid the nanofiller from spilling. After manual mixing, the batch was placed on a hot plate for 90ºC and high shear mixing (HSM) ran for 1 hour. Every 30 minutes the direction was switched to ensure a consistent blending of the materials. HSM concluded the batch was placed into a sonicator to agitate and disperse any agglomerations HSM did not break down. This process proceeded for 1 hour at which a quality control sample was taken for microscopic analysis to gauge how well the dispersion was. The curing agent Epikure-W was mixed into the batch and swiftly underwent HSM for 10 minutes with the direction alternating every 5 minutes. The resin was taken to be degassed once more following the same configuration as before. At the end of this process the batch was ready to begin B-staging.

B-staging took place inside a vacuum chamber at 120ºC until the resin reached B-stage. This process was monitored for roughly 40 minutes at 120ºC with samples taken in 2 to 5 minute increments. The batch underwent our proprietary process of making homogenous sheets of resin film of various thicknesses stated by ASTM D5868-01. For making the films containing through-thickness directionally aligned CNF or MCNT (i.e., ZT-Film), the resin films were subjected to a

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high voltage to align the nanofillers in the z-direction [4]. This electrical field alignment was done on a proprietary R2R assembly line. Figure 2 shows the schematic of the aligned CNF or MCNT film manufacturing process

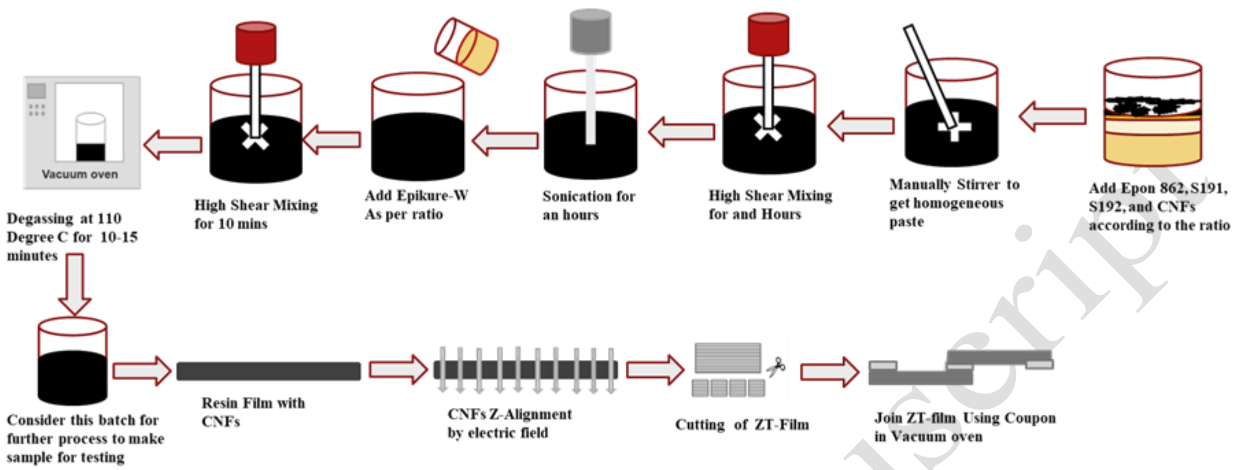

Figure 2: The resin film adhesive containing aligned CNF or MCNT, i.e., CNF Z-threading resin film or ZT-Film, was manufactured using this process for each variation of film adhesive involving nanofillers.

The thickness described in the ASTM D5868-01 standard called for a film thickness of 0.76 mm which was made with a minimum of 0.65mm to a maximum of 0.86 mm. For this experiment, both aligned and unaligned film adhesives were made at this stage. Once the alignment process was complete the resin had converted into sheets of resin which could then be cut to the dimensions stated in ASTM D5868-01.

The films were cut into 1 inch^2 pieces and then applied at the bonding site at the end of each coupon. A small amount of force was necessary to keep the films and coupons from moving while curing. This need was fulfilled by using medium sized alligator clips clamping the coupons together at the bonding area. This step was important as without it the bonding edge could be larger and cause the coupons to cure at an angle which would cause a stress concentration and create inconsistent data.

3. RESULTS AND DISCUSSION

3.1 Single-Shear Lap Test Results

3.1.1 CFRP Coupon Testing

For this experiment the following film adhesive cases were investigated: aligned MCNT/resin, unaligned MCNT/resin, control resin, aligned CNF/resin, and unaligned CNF/resin. Figure 3 shows all the samples' shear stress vs displacement curves. All samples had a similar shear strength level.

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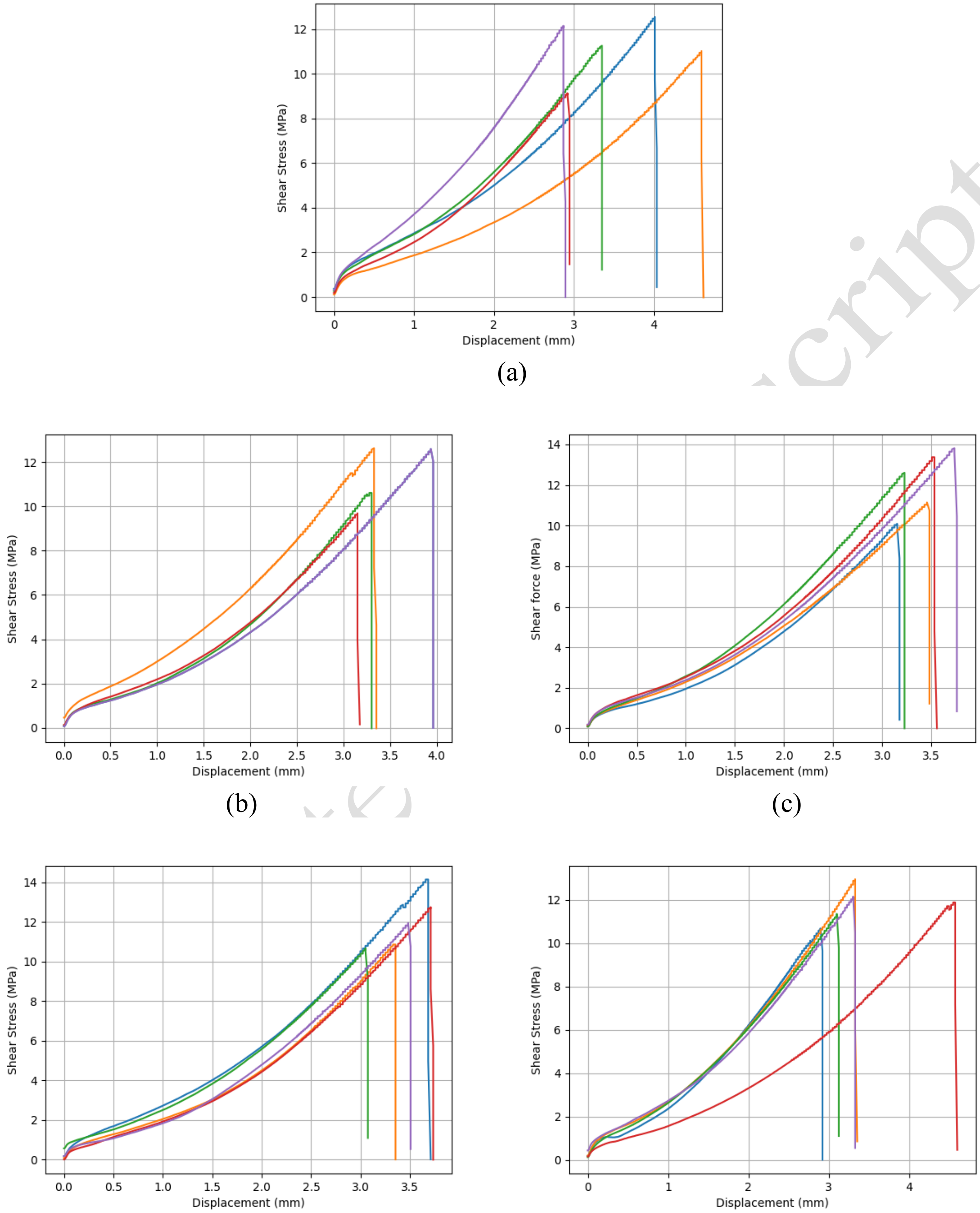

Figure 3: The shear stress vs crosshead displacement curves for the CFRP bonding test: Control (a), Aligned MCNT (b), Unaligned MCNT (c), Aligned CNF (d), Unaligned CNF (e).

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Table 2 has the similarities between the ultimate shear strengths documented from the test cases. A visual inspection of the fracture surface regardless of testing case showed a consistent fiber failure (adherent) instead of a film adhesive failure (adhesive). If the CFRP adherent was stronger than the ones being tested, one would expect a higher shear strength. Hsiao et al. showed the ultimate stress found with 1.0wt% MCNT film adhesive was around 17 MPa in the same experiment standard procedure, ASTM D5868-01 but using resin transfer molding process produced CFRP adherends [3].

Table 2: CFRP Coupon Testing Case Results.

	Control Shear Strength (MPa)	Aligned CNF Shear Strength (MPa)	Unaligned CNF Shear Strength (MPa)	Aligned MCNT Shear Strength (MPa)	Unaligned MCNT Shear Strength (MPa)
Sample 1	12.53	14.14	10.67	12.59	10.09
Sample 2	11.00	10.85	12.93	12.62	11.14
Sample 3	11.26	10.66	11.34	10.60	12.60
Sample 4	9.12	12.73	11.87	9.68	13.37
Sample 5	12.14	11.94	12.14	11.42	13.81
Mean	11.21	12.06	11.79	11.38	12.20
Stdev.	1.32	1.43	0.84	1.27	1.56
C.O.V. (%)	11.81	11.87	7.18	11.20	12.76
Minimum	9.12	10.66	10.68	9.68	10.09
Maximum	12.53	14.14	12.93	12.62	13.81

Figure 4 below shows the majority of CFRP bonding samples, regardless of the type of resin film being used, yielded similar failure modes. The T700 unidirectional carbon fiber used inside the CFRP composite testing coupons failed first with heavy damage to one or both sides of the bonding area.

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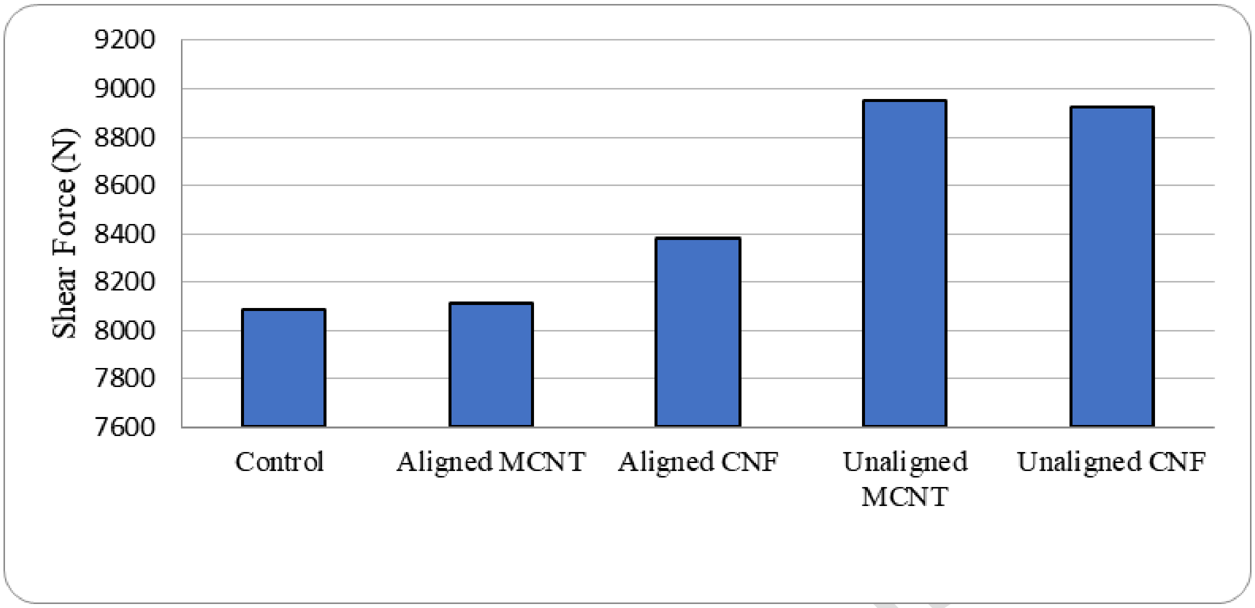

Figure 4: Average Ultimate Shear Stress per CFRP Case.

This damage indicated a fiber-tear failure in the CFRP coupons. Due to this, the ultimate shear stress exerted onto the samples at failure could be inferred to be from the fiber failing rather than the resin film failing in every case. This conclusion can be seen clearly in Table 3, which summarizes all the testing cases using the CFRP adherents. Since all cases of the CFRP adherent bonding test were limited by the adherent failure they cannot tell the significance of the adhesive film compositions effect over the other. A stronger adherent was needed for addressing the issue. In the following cases, the steel adherents were used.

Table 3: CFRP-bonding Cases Testing Summary

Film Adhesive Type	Average Bonding Area (mm2)	Average Shear Strength (MPa)	COV of Shear Strength (%)	Improvement over control shear strength (%)	Improvement over control shear strength COV (%)
Control	7.34	11.21	11.81	N/A	N/A
Aligned CNF	7.25	12.06	11.87	7.62	0.50
Unaligned CNF	7.73	11.79	7.18	5.19	-39.17
Aligned MCNT	7.10	11.38	11.20	1.54	-5.16
Unaligned MCNT	6.87	12.20	12.76	8.84	8.05

3.1.2 Steel Coupon bonding Testing

While not specifically made the standard used before, ASTM D5868-01, the steel testing was machined in a similar fashion with the exception being a larger bonding area resulting in a rectangular form of 645.16 mm^2 (1 $inch^2$) bonding area. The thickness was also different with an average 1.25mm compared to the CFRP test coupons at 2.5mm thick. A similar process was

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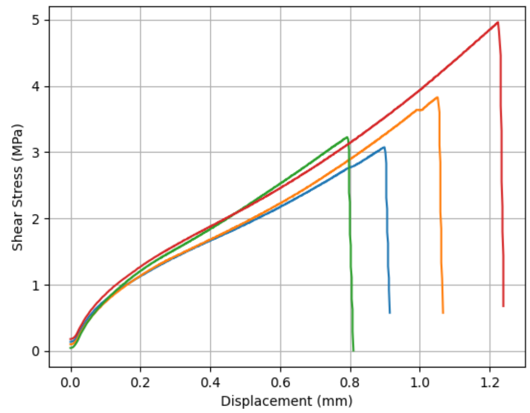
repeated from the CFRP coupons to create the film thickness used for this round of tests. This round of testing ensured no adherent failure in the coupons would occur again and should yield failure modes either on the interface or the adhesive. Figure 5 shows the shear stress vs displacement curves. Note that MCNT was not used in the steel-bonding studies.



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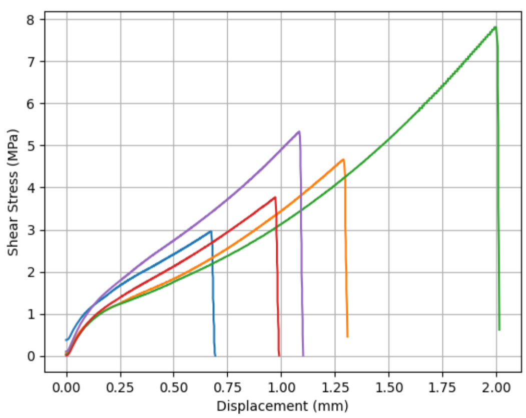

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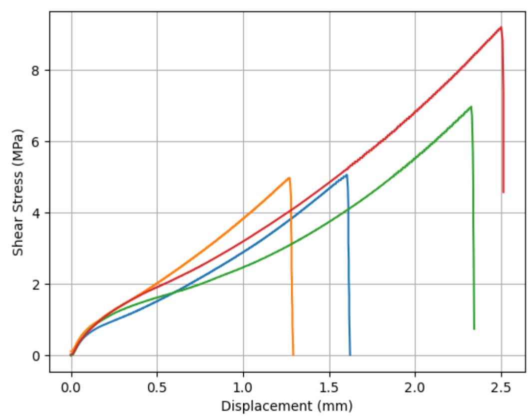

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Figure 5: Steel Adherends Testing Cases: Control (a), Aligned CNF (b), Unaligned CNF (c).

Table 4 summarizes the shear stresses of the steel-plates bonding study. The values were less than the shear stress found in the CFRP tests. However, this was a red herring. While it was true, the reasons why were based on inconsistent results. The majority of the samples experienced interface failure on the CFRP fiber. Yet error could have also been present in the second round of testing. The surface preparation of the galvanized steel coupons could have caused this lower failure shear strength in [5] steel coupons were sanded in ascending grades of sandpaper and chemical washes to remove as many impurities as possible. In Table 4 below, the mean shear strength found was

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similar to the data in [6] with an average shear strength of 3.12 MPa for its epoxy and hand sanding test compared to the 3.772 mean found in the control resin film bonding test.

Table 4: Steel Coupon Testing Case Results.

Ultimate Shear (MPa)	Control Shear Strength (MPa)	Aligned CNF Shear Strength (MPa)	Unaligned CNF Shear Strength (MPa)
Sample 1	3.075	2.956	N/A
Sample 2	3.826	4.669	5.059
Sample 3	N/A	7.815	4.977
Sample 4	3.227	3.768	6.972
Sample 5	4.962	5.331	9.195
Mean	3.772	4.908	6.551
Stdev.	0.857	1.857	1.989
C.O.V. (%)	22.712%	37.841%	30.367%
Minimum	3.075	2.956	4.977
Maximum	4.962	7.815	9.195

Note: Failed before testing (N/A).

After testing, the highest shear strength case from the steel coupon test was found to be the unaligned CNF case as shown in Figure 6 and Table 5. In a cross section of the bonding site, the randomly aligned nanofiber will oppose movement as well as stopping the crack propagation. Because of this, the bonding site must prove difficult to break in tension testing. If one compares the cases with aligned CNF, the alignment of the CNF along the z-direction will appear flat and pointed in the general direction of failure. With unaligned CNF, it would be more difficult for the crack propagation to pass through. Although the aligned MCNT would behave similarly to unaligned CNF in theory. Because the aligned CNF was all pointed in the z-direction it would increase the chance of fiber pullout resulting in a lower ultimate shear strength.

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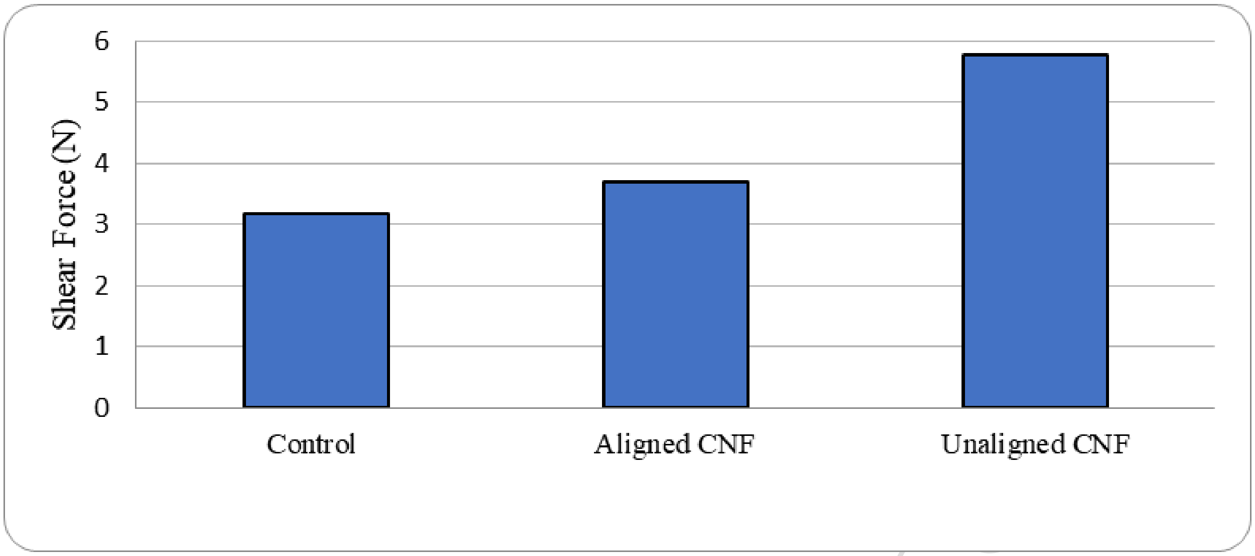

Figure 6: Average Ultimate Shear Stress per Steel Case.

Table 5: Steel Testing Summary.

Film Adhesive Type	Average Bonding Area (mm2)	Average Shear Strength (MPa)	COV of Shear Strength (%)	Improvement over control shear strength (%)	Improvement over control shear strength COV (%)
Control	8.43	3.77	22.71	N/A	N/A
Aligned CNF	8.06	4.91	37.84	30.10	66.61
Unaligned CNF	8.99	6.55	30.37	73.65	33.70

Figure 7 compares the average shear stresses for using aligned and unaligned CNFs in the resin film for bonding CFRP laminates and steel plates. It can be seen that CFRP bonding failure seems to be independent of the type of resin film used. However, the steel-bonding data showed significant improvement of using CNFs in the resin film. Furthermore, the unaligned CNF infused resin film produced 73.65% improvement as shown in Table 5.

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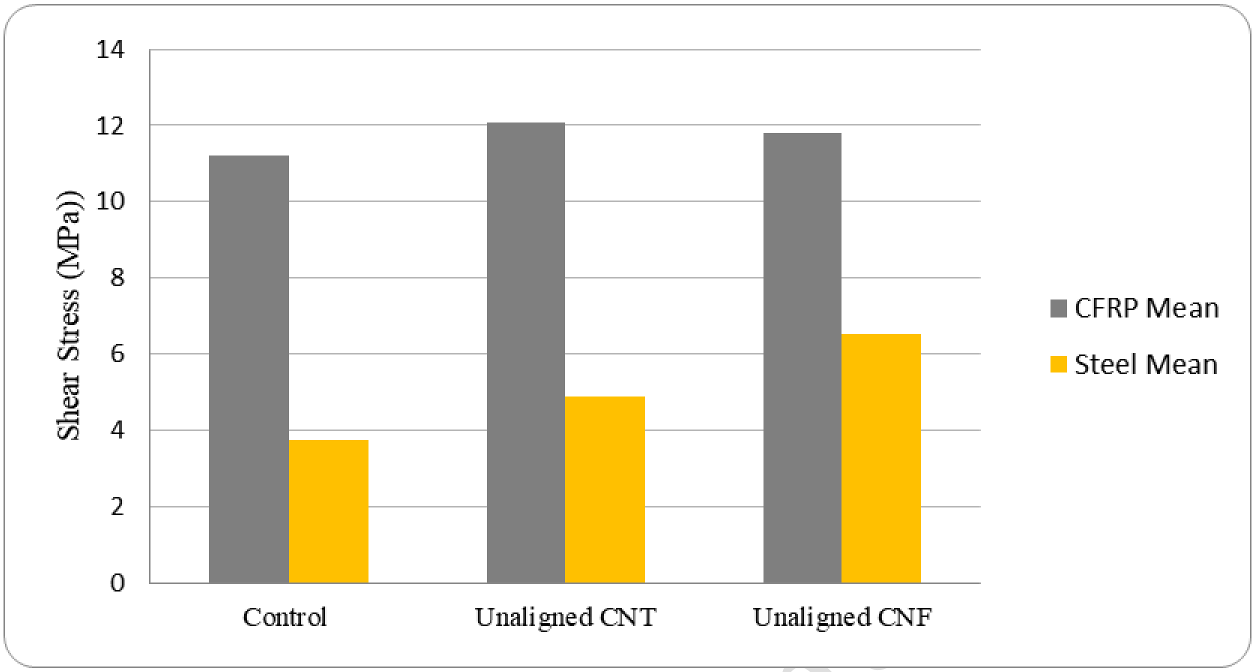
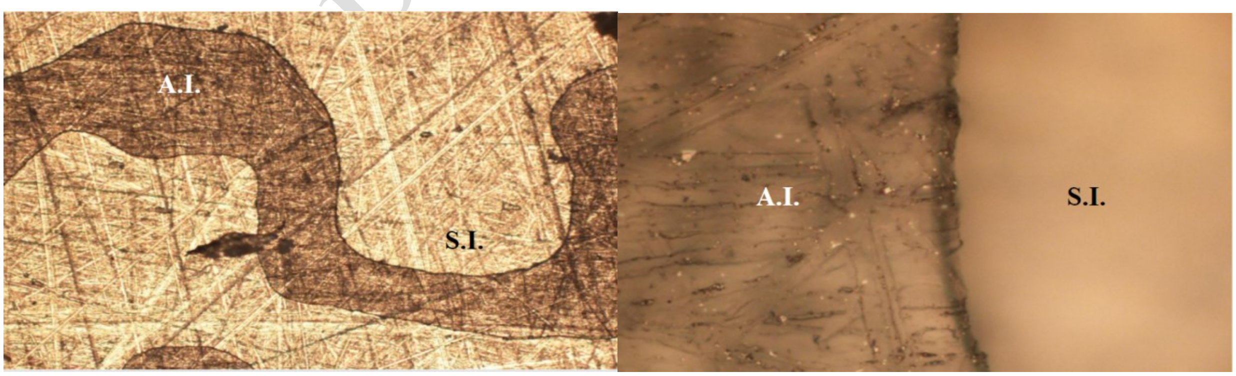


Figure 7: Average Shear Stress for each experiment.

3.2 Fracture Failure Modes

After testing the CFRP and steel coupons, pictures of the failure surface on the bonding site were taken to distinguish a pattern relating the shear stress found in testing and the failure modes. In the figure below each sample following their respective case was documented. Due to the extensive damage to the CFRP test samples a uniform failure outside adherent failure could not be distinguished. Yet the steel test samples provided a clear picture of the failure mode. These pictures were chosen as they resemble the average failure mode from each case.



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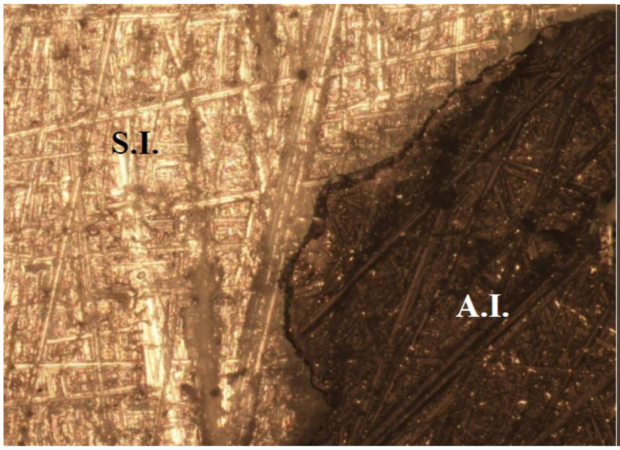

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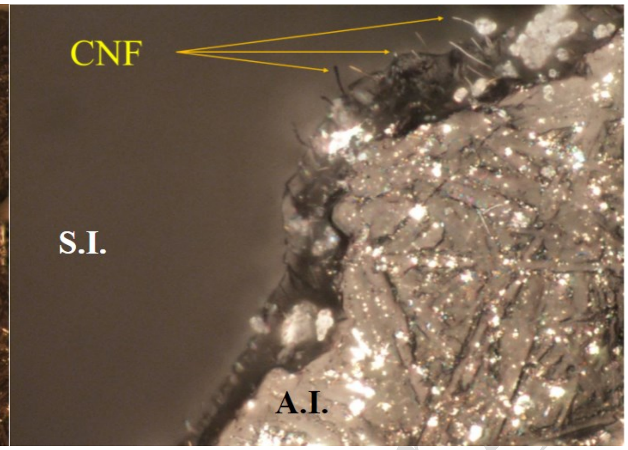

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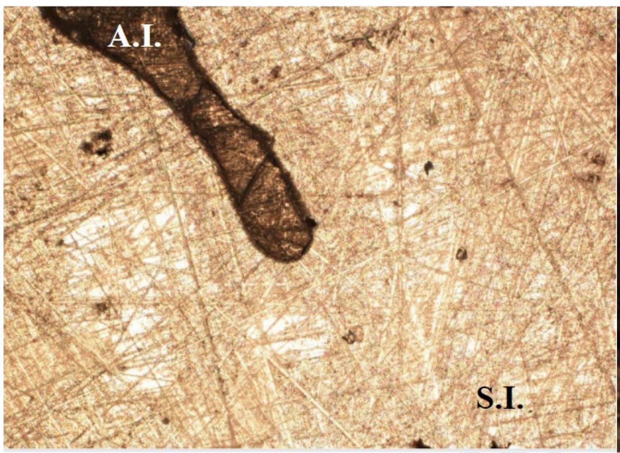

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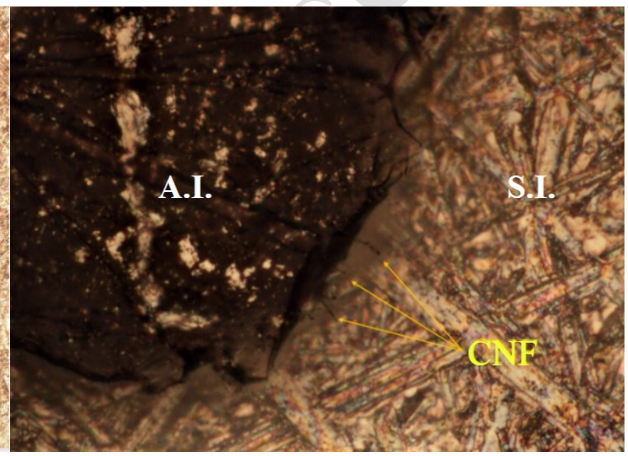

(f)

Figure 8: Steel Bonding Sample Failure Microscopy: Control 100x (a), Control 1000x (b), Aligned CNF 200x (c), Aligned CNF 1000x (d), Unaligned CNF 100x (e), Unaligned CNF 1000x (f). Adhesive Interface (A.I.), Steel Interface (S.I.).

4. CONCLUSIONS

This study characterized the capabilities of aligned and unaligned MCNT and CNF infused film adhesives in shear through unidirectional carbon fiber and epoxy-based film adhesives in various cases. The CFRP coupons used in the first test were expected to outlast the film adhesives and provide true to life shear strength data. However, the outcome of that test proved otherwise. In actuality, the fiber yielded first in every case regardless of the composition of its film adhesive. To rectify the data, another round of testing was conducted with the change to steel adherend coupons. These tests resulted in a very clear correlation to the film adhesive used and the corresponding shear strength. In summation, unaligned CNF proved to be the strongest of the cases during the steel plate-bonding testing. It performed 73.6% better than the neat epoxy-film adhesive, at an average shear stress of 6.551 MPa compared to 3.772 MPa from the control case. Although the CFRP laminate bonding tests showed almost the same results of shear strength for every sample

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case, it did show that ultimate shear strengths for all film adhesives were all possibly higher than what were measured as the CFRP coupons were the first to fail. This meant the average shear strength for unaligned MCNT infused resin as explained in [4] of around 17 MPa might not have been far off from the tested result if the CFRP coupon used in this study did not fail first. In terms of failure mode shown on the bonding sites of the steel plates, the unaligned CNF infused film-adhesive bonding cases seemed to have higher coverage on both coupons at least for the steel test in cohesive failure. The aligned CNF film-adhesive bonding cases had one side clean while the other coupon had heavy cover of its film as well as the coverage found in unaligned CNF. The control film (neat epoxy film) bonded cases had a majority failure which almost had all coverage on one coupon with a small amount on the other. The absence of a trend, at least for the visual inspection, suggests a future experiment with finer surface preparation to determine if the surface treatment could improve the steel plate bonding and provide a clear and consistent failure mode, especially for the CNF infused resin films.

5. ACKNOWLEDGMENT

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