LASER SURFACE PREPARATION AND BONDING OF AEROSPACE STRUCTURAL COMPOSITES

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SUMMARY

A Nd:YAG laser was used to etch patterns conducive to adhesive bonding onto CFRP surfaces. These were compared to typical pre-bonding surface treatments including grit blasting, manual abrasion, and peel ply. Laser treated composites were then subjected to optical microscopy, contact angle measurements, and post-bonding mechanical testing.

Keywords: *composites, CFRP, surface treatment, adhesive bonding, laser*

INTRODUCTION

Adhesive bonds are critical to the integrity of built-up structures. Disbonds can often be detected but the strength of adhesion between surfaces in contact is not obtainable without destructive testing. Typically the number one problem in a bonded structure is surface contamination and preparation [1,2]. Standard surface preparation techniques, including grit blasting, manual abrasion, and peel ply, are not ideal in terms of adhesively bonded composite structures because of variations in their application (i.e., there can be dissimilarities across surfaces because of different operators, operator error, or other inconsistencies inherent with these techniques) [3]. However, etching of carbon fiber reinforced plastic (CFRP) panels using a Nd:YAG laser appears to be a promising way to both clean a composite surface prior to bonding as well as provide a bond-promoting patterned surface akin to that from a peel ply without the inherent drawbacks from the same (i.e., debris and curvature from the peel ply). Such a technique can provide high surface reproducibility and greater precision. Comparison of optical micrographs, contact angle measurements, and mechanical testing data (single lap shear) will be discussed for several CFRP panels prepared and bonded under various conditions.

EXPERIMENTAL

Materials and Methods

Composite panels were fabricated from 16 plies of unidirectional Torayca P2302-19 prepreg (T800H/3900-2 carbon fiber-toughened epoxy resin system) [4]. Silicon carbide (220 grit) was used for grit blasting (80 psi) and silicon carbide sandpaper (320 grit) was used for manual abrasion. Hysol® EA9895™ WPP (Henkel) pre-impregnated polyester peel ply (wet) and PF 60001 (Precision Fabrics) polyester dry peel ply were used as received. After surface preparation, regardless of technique, two CFRP panels (10.2 cm x 20.3 cm [4" x 8"]) were bonded together using a strip (dimensions: 1.59 cm x 20.3 cm [0.625" x 8.0"], areal weight: 244 g/m² [0.050 lb/ft²]) of Scotch-Weld™ Structural Adhesive Film AF-555M (3M) shimmed to a final bondline thickness of 203 μm (8 mil). Bonding was done in a 13600 kg (15 ton) capacity hydraulic press (Technical Machines Products) at 0.310 MPa (45 psi). Temperature was raised to 177 °C (350 °F) at a fixed rate (2.78 °C/min [5 °F/min]) and then maintained for 2 h. After curing the press was cooled to ambient temperature at the same rate. Prior to adhesive bonding the entire layup was held under vacuum overnight.

Laser Etching

Laser etching of CFRP panels was done using a PhotoMachining, Inc. laser ablation system with a Coherent Avia® frequency tripled Nd:YAG laser (7-watt output at 355 nm). Two different patterns were etched onto CFRP surfaces (see Figure 1). Pattern A was created to replicate that of a peel ply treated surface while pattern B was a 0°/90° crosshatch. The following parameters could be adjusted: laser power, frequency, beam width, beam spacing, scan speed, and number of passes. For all work the final two parameters were maintained at 25.4 cm/sec (10 in/sec) and 1, respectively. Beam width and spacing was kept at the maximum resolution of the laser (25 μm [1 mil]). Laser power was varied among 4.9, 5.6, and 6.3 W while frequency was set to 30, 40, or 60 kHz depending on the experiment.

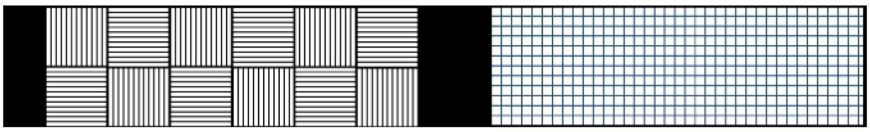

Figure 1. Two patterns used for laser etching: A was designed to replicate the peel ply pattern while B is a simple 0°/90° crosshatch (drawings not to scale).

Optical Microscopy

Micrographs were taken with an Olympus BH-2 optical microscope equipped with a Hitachi KP-D50 digital color camera. As shown in Figure 2, CFRP surfaces varied greatly depending on the surface preparation technique. Additionally, a lack of precision is present within each individual surface treatment, be it pitting (Figure 2.B.), unevenness (Figure 2.C.), or debris (Figure 2.D.). It is of note that curvature was also present in the pattern remaining from use of peel ply (Figure 3.A.). Bénard, et al., recently confirmed via confocal microscopy that curvature resulted from the peel ply [5]. In comparison to the current state of the art peel ply, the laser etching process leaves not only a debris free surface, but also one that is flat and of high fidelity (Figure 3.B. and C.).

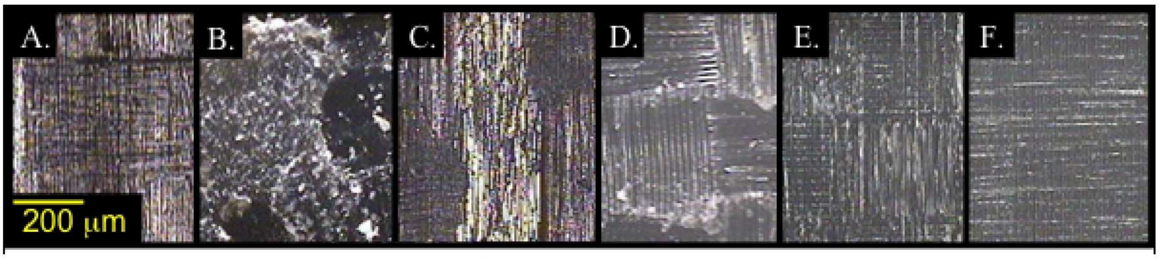

Figure 2. CFRP surface (A.) as is, (B.) grit blasted, (C.) manually abraded, (D.) treated with peel ply, (E.) laser etched with pattern A from Figure 1, and (F.) laser etched with pattern B from Figure 1.

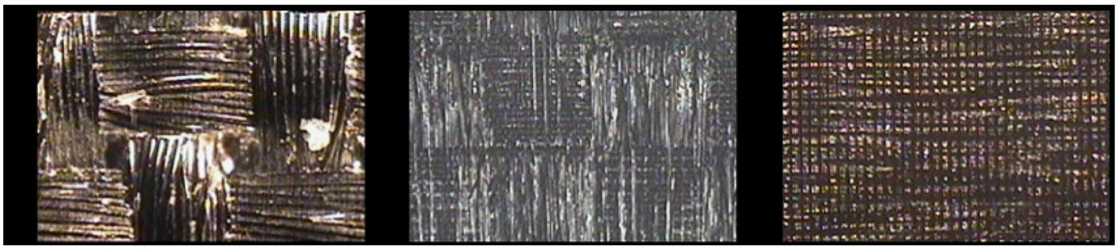

Figure 3. (A.) Curved CFRP surface arising from peel ply and (B. and C.) flat, debris free CFRP surface after laser etching (patterns A and B from Figure 1, respectively).

Contact Angle Goniometry

Contact angle goniometry was performed using a FTA 1000B system (First Ten Angstroms). Sessile drop contact angles were measured for each sample using 3 µL drops of water, 3 µL drops of ethylene glycol, or 2 µL drops of diiodomethane, where appropriate. Interfacial tension of a suspended drop of each liquid was measured prior to analysis to verify the purity of the liquid and precision of the focused image. Contact angles were determined by drop shape analysis and standard deviations were calculated by comparison of the contact angles observed for each frame of a 40 frame movie collected after drop deposition on the sample surface. Each sample was measured at least twice. Water contact angle data for various CFRP surface treatments, including laser etching, are summarized in Table 1.

Table 1. Water contact angles for various CFRP surface treatments. [Angles reported as less than one (<1°) were immeasurable, virtually immediately wetting out the surface.]

CFRP Surface Treatment		Angle (°)	CFRP Surface Treatment		Angle (°)
None		79			
Grit-blast		86	Wet peel ply		76
Manual abrasion		88	Dry peel ply		83
Laser etched, pattern A	40 kHz, 6.3 W	32	Laser etched, pattern B	40 kHz, 6.3 W	<1
	40 kHz, 5.6 W	26		40 kHz, 5.6 W	<1
	40 kHz, 4.9 W	96		40 kHz, 4.9 W	93
	60 kHz, 6.3 W	22		60 kHz, 6.3 W	<1
	60 kHz, 5.6 W	40.4		60 kHz, 5.6 W	13.9
	60 kHz, 4.9 W	101.3		60 kHz, 4.9 W	99.9
	30 kHz, 6.3 W	24.8		30 kHz, 6.3 W	<1

Mechanical Testing

Single lap shear specimens were tested according to a slight modification of ASTM D3165-00 using an MTS 810 Test Frame with an MTS 661.20 Force Transducer (25 to 100 kN [5,500 to 22,000 lb]) and MTS 647 Hydraulic Wedge Grips (100 kN [22,000 lb] capacity; 21 MPa [3000 psi] maximum pressure). This test was used as a measure of joint bond quality and to determine comparative shear strengths of joints made with a singular adhesive but different surface preparation techniques (minimum of five specimens per set of conditions). The modification to ASTM D3165-00 regarded how the bonded test specimens were gripped (see Figure 4). All other significant portions of the standard (i.e., adhesive overlap, adherend thickness, gripped portion of the specimen, crosshead speed, etc.) remained the same. This modification allowed for a considerably simpler bonding configuration as well as an overall materials savings in that less CFRP had to be produced and less adhesive was used.

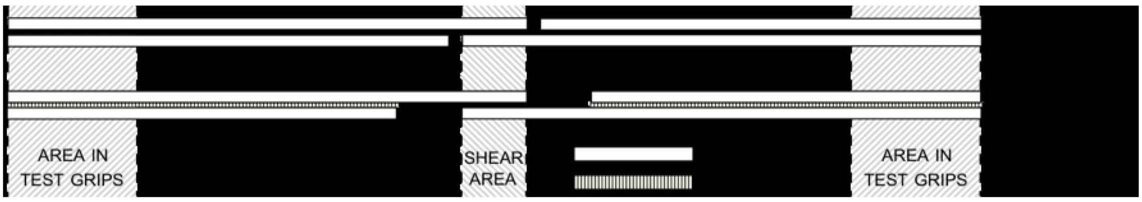

Figure 4. Scheme of single lap shear specimen test configuration according to ASTM D3165-00 and as modified in present work (lateral view).

RESULTS AND DISCUSSION

Laser Etching

As shown in Figure 3, surface preparation via laser etching results in high fidelity and precise topographical modification to the CFRP surface. The resin accumulation or void areas observed in the peel ply treated surfaces, which are an artifact of the weave pattern of the peel ply material, are not present in the laser etched surface due to this surface preparation process occurring after the CFRP panel has been cured. Using laser etching for surface preparation also precludes curvature and debris common to the peel ply process.

Contact Angle Analysis

CFRP panels that were laser etched with both patterns A and B showed significantly higher surface energies when compared to pristine CFRP according to contact angle measurements with water (see Table 1). Other surface preparation techniques did not result in contact angles markedly different from the untreated CFRP. However, water contact angles for laser etched CFRP surfaces could be varied from 0 to over 100°, thus allowing a singular material to be alternately hydrophilic or hydrophobic depending on laser etching parameters. Water contact angle data were used as a discriminator for selection of patterns to be used for adhesive bonding. Pattern A/40 kHz/5.6 W and pattern B/60 kHz/5.6 W were chosen based on their low measurable contact angles. Additionally, pattern B/30 kHz/6.3 W was selected for bonding due to the lower frequency and higher energy etching conditions that were thought to lend themselves to deeper etching.

Surface Energy and Wetting Envelopes

Young's equation relates the contact angle a liquid makes with a surface (θ) and the liquid's surface tension (γ_L) to the surface energy of the interrogated material (γ_S). Modifications of Young's equation separate contributions to the surface energy of a material into polar and dispersive components (γ^p and γ^d respectively). By measuring the contact angle of multiple liquids on a given surface these parameters can be obtained using extended Fowkes theory (Eq. 1).

$$(1+\cos\theta)\frac{\gamma_L}{2\left(\gamma_L^d\right)^{0.5}} = \left(\gamma_S^d\right)^{0.5} + \left(\gamma_S^p\right)^{0.5}\left(\frac{\gamma_L^p}{\gamma_L^d}\right)^{0.5} \quad (1)$$

Eq. 1 can be rewritten to define a domain representing polar-dispersive liquid surface tension values that would satisfy the criterion of complete wetting (i.e., a contact angle value of 0°). This domain is referred to as the wetting envelope. Fluids with surface tension properties underneath a particular curve (i.e., inside the envelope) will "wet out" the surface spontaneously while those above the curve (i.e., outside the envelope) will not. For optimum adhesion, an adhesive must thoroughly "wet out" the surface to be bonded. "Wetting out" means the attractive forces between the adhesive and bonding surface are maximized. For example, a lower surface energy material like water will spontaneously wet out a higher energy surface, such as that of an un-waxed car bonnet (hood).

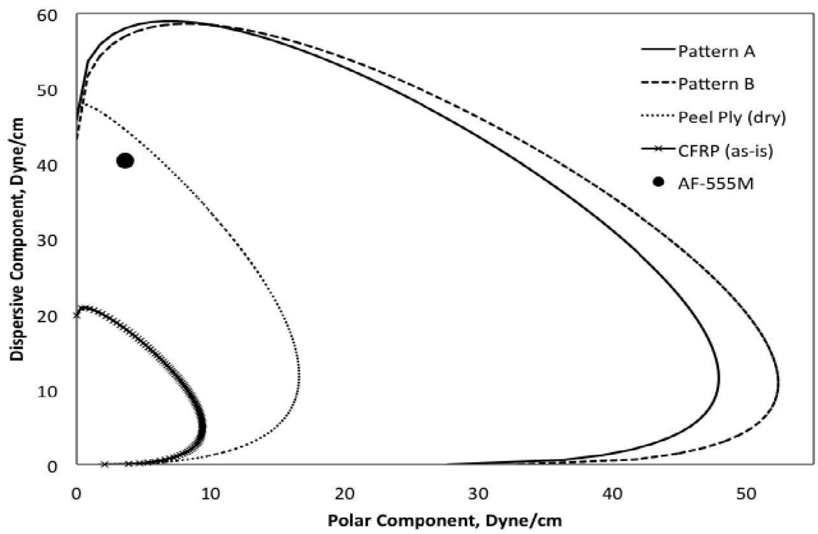

Figure 5. Wetting envelopes for pattern A/40 kHz/5.6 W and pattern B/60 kHz/5.6 W laser etched CFRP, dry peel ply treated CFRP, as-is CFRP, and AF-555M adhesive.

Thus wetting envelopes are one potential method to aid in predicting the suitability of a surface for bonding with an adhesive of known surface tension parameters. Wetting envelopes for pattern A/40 kHz/5.6 W and pattern B/60 kHz/5.6 W laser etched CFRP, dry peel ply treated CFRP, and as-is CFRP, as well as the location of AF-555M adhesive on this type of plot, are shown in Figure 5. While the wetting envelope for the dry peel ply treated CFRP surface encompasses the adhesive, which suggests the adhesive will wet the peel ply treated surface sufficiently, wetting envelopes for both laser etched CFRP surfaces are considerably larger, indicating the adhesive will definitely wet out these surfaces. The other discussed surface treatments (i.e., grit-blasting and manual abrasion) did not have wetting envelopes that encompassed the adhesive and these were omitted from Figure 5 for clarity.

Mechanical Testing Data

Apparent shear strength data, bondline thicknesses, and failure modes for single lap shear specimens of CFRP with various surface treatments are summarized in Table 2. All strength values are of the same order of magnitude regardless of surface preparation technique or bondline thickness. However, it is in the failure modes where major differences can be seen. All laser etched CFRP samples had a light-fiber-tear failure mode while other surface preparation techniques in this study resulted in mixed mode failures. Grit-blasted samples had 90% cohesive, 10% light-fiber-tear failure. Wet peel ply samples had 95% adhesive, 5% light-fiber-tear failure while dry peel ply samples had 80% thin-layer cohesive, 20% adhesive failure. The light-fiber-tear failure mode for all laser etched CFRPs suggested that the laser surface preparation technique produced an adhesive bond strong enough to damage the adherend before breaking the bond itself. [See ASTM D5573-99 (2005) for failure mode definitions.]

Table 2. Averaged single lap shear data for bonded CFRP panels according to different surface preparation techniques. [Samples marked by * were prepared for another study but were otherwise tested under identical conditions.]

CFRP Surface Treatment	Average Bondline Thickness, μm (mil)	Apparent Shear Strength, MPa (psi)	Failure Mode
Grit-blast*	152 (6.0)	25.1 (3648 ±148)	90C/10LFT
Wet peel ply*	229 (9.0)	25.5 (3697 ±105)	95A/5LFT
Dry peel ply*	140 (5.5)	28.0 (4056 ±174)	80TLC/20A
Pattern A/40 kHz/5.6 W	188 (7.4)	26.6 (3851 ±95)	LFT
Pattern B/60 kHz/5.6 W	196 (7.7)	27.6 (4003 ±136)	LFT
Pattern B/30 kHz/6.3 W	208 (8.2)	26.4 (3811 ±81)	LFT

CONCLUSIONS

A Nd:YAG laser was used to etch patterns conducive to adhesive bonding onto CFRP surfaces. These were optically compared to typical pre-bonding surface treatments such as grit blasting, manual abrasion, and peel ply. Laser etched CFRP panels consistently had surfaces free of debris, irregularities, and curvature. Laser etched CFRP surfaces were then subjected to contact angle measurements. Depending on laser parameter selection, water contact angles could be varied from 0 to over 100°, thus allowing the surface properties to be tailored. Wetting envelopes correctly predicted that laser etched CFRP surfaces would be wetted out by the adhesive used in this study. It is of note that the wetting envelopes for laser etched CFRP were significantly larger than that for peel ply treated CFRP, thus suggesting laser etching to be suitable for perhaps a broader array of adhesives. Finally, mechanical testing was done according to ASTM D3165-00. Comparison of this data per surface preparation technique was expected to afford some correlation to respective contact angle measurements. More specifically, it was anticipated that higher surface energies (e.g. lower contact angles) would correspond to greater bond strengths. However, apparent shear strength values showed the peel ply treatment and laser etching to be roughly equivalent, with both being slightly better than grit-blasting. On the other hand, failure modes for laser etched CFRPs strongly suggested that this laser surface preparation technique produced adhesive bonds robust enough to damage the adherend before breaking the bond.

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