# IDENTIFYING INCONSISTENCIES IN SURFACE PREPARATION THROUGH ROBOTIC WATER CONTACT ANGLE MAPPING

*Sonia T. Stanciu, Senior Materials Scientist*
*Alan Jechort, Director of Software Engineering*
*Giles Dillingham, Chief Science Officer*
*Brighton Science*
*Cincinnati, OH 45232*



## ABSTRACT

It can be challenging to refine process controls in a manufacturing environment due to the vast number of chemical and physical factors that dictate final performance. The requirements for coating, bonding, and sealing make process control even more difficult because adhesion is highly dependent on unseen surface chemistry. Surface chemistry can be tuned through a variety of surface modification techniques ranging from solvent cleaning to plasma treatment. Better control of these processes can be achieved through quantitative water contact angle (WCA) measurements, which utilize the surface tension of water to probe surface energy - thus providing a fast, reliable, and non-destructive method for tracking inconsistencies in surface preparation across diverse stages of manufacturing and quality control.

We prepared composite and metal surfaces using a variety of manual and automated surface preparation techniques, including solvent wiping, manual sanding, grit blasting, and plasma treatment. We then compared spatial consistency and reproducibility for each technique, as well as sensitivity to surface evolution up to 24 hours after surface preparation, using robotic WCA mapping. This approach illuminated the issues inherent to manual surface preparation, particularly when applied to composites, as well as the effect of initial surface composition on surface aging.

# 1. INTRODUCTION

## 1.1 The dynamic nature of surfaces

The successful manufacture and repair of composite structures that require bonding, coating, painting, or sealing is contingent on preparing pristine surfaces of a controlled composition and subsequently maintaining those surfaces until the intended coating or adhesive is applied. Many failures arise when surfaces are prepared heterogeneously, re-contaminated after preparation, or there is excessive out time before bonding/coating [1]. There are numerous methods for preparing surfaces - ranging from lower-control techniques such as hand sanding

with abrasive papers to higher-control techniques such as plasma treatment for surface functionalization [1-3].

Each technique has its own inherent level of preparation heterogeneity across various substrates, which can be challenging to identify through nondestructive inspection (NDI) [3]. Because the surface of a material is only comprised of its first few molecular layers, many traditional spectroscopic techniques probe too deeply into a material to detect minute changes to surface chemistry [4]. Such nm-range interactions are responsible for the highly sensitive and dynamic nature of surface chemistry - leading to a contaminant as small as a fingerprint being significant enough to transform an interface into an undesired chemical landscape (Figure 1) [4].

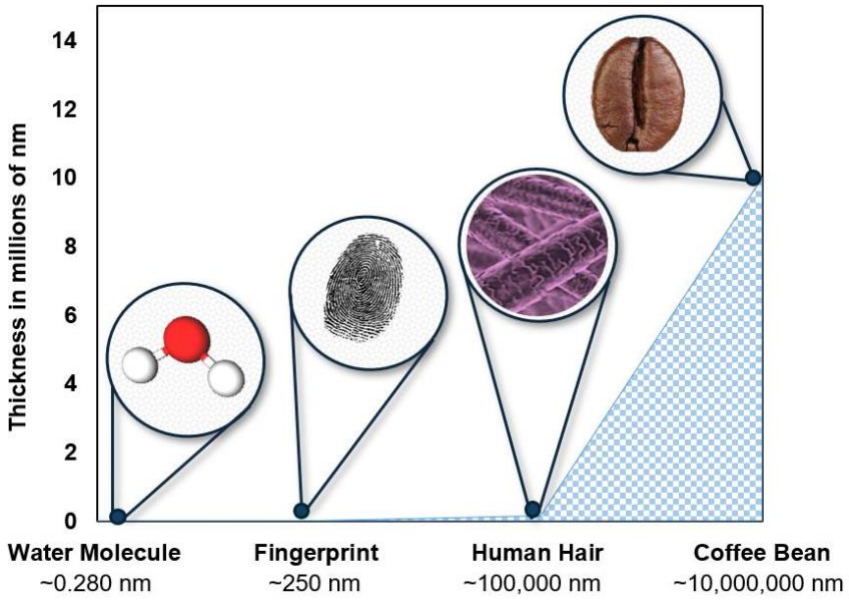


*Figure 1. Comparison of the thickness of a single water molecule (~0.280 nm) [5], to residue left behind from a human fingerprint (~250 nm) [6], a single human hair (~100,000 nm) [7], and a single coffee bean (~10,000,000 nm) [8]. The surface of a material is only 3-5 molecular layers deep, making it about 1000x thinner than a human hair.*

## 1.2 Surface energy and its impact on water contact angle

To understand the complicated nature of bonding, one must understand the impact that surface energy has on facilitating and maintaining a bond. Surfaces have a specific potential energy for chemical and physical reactivity with other materials because the forces at a surface are unbalanced compared to the bulk [4, 9-10]. Thus, a material with a high surface energy is more capable of attracting and maintaining a bond to an unlike surface [4, 9-10]. Some materials have inherently high surface energies due to their strong polarity (i.e. metals) but can become contaminated or oxidized - reducing their practice surface energy and thus their ability to maintain a bond.

The most common treatment of surface energy assumes that there are two major contributors to the surface (free) energy of a material: non-polar forces due to random electron cloud fluctuations, and polar forces due to attraction of permanent dipoles (Equation 1) [4, 9]:

$$\gamma_S = \gamma^d_S + \gamma^p_S \quad [1]$$

where γS is the total surface free energy, γd(S) is the dispersive (non-polar) contribution to surface energy, and γp(S) is the polar contribution to surface energy. The dispersive contribution accounts for nonspecific long range intermolecular forces such as London dispersion forces. Temporary fluctuations in electron clouds lead to weak, short-lived attractive forces that exist in all materials regardless of functional group structure. In contrast, the polar contribution to surface energy arises from permanent dipole-dipole interactions and is impacted by stronger short-range interactions like hydrogen bonding and donor-acceptor chemistry [4, 9]. While both contribute to total surface energy, only the polar component of surface energy can be readily tuned through preparation techniques such as physical cleaning or chemical functionalization [1].

Despite the strong impact that surface energy has on adhesion, there remains a practical challenge surrounding the reliable and nondestructive measurement of surfaces [3]. Many techniques to predict surface energy are highly operator dependent, notably the use of solvent-rich dyne inks, which rely on the individual to visually determine whether the ink separates in a way that indicates lower surface energy. Conversely, water is both highly polar and generally benign to most surfaces at miniscule concentrations, thus the angle it forms when a droplet is deposited on a surface is a good reflection of how strongly that surface attracts polar molecules. The stronger the polar contribution to surface energy, the lower the angle of contact the water will present on the surface, making quantitative water contact angle (WCA) measurements an effective NDI for predicting a surface's inclination for attracting and maintaining bonds/coatings (Figure 2) [3].

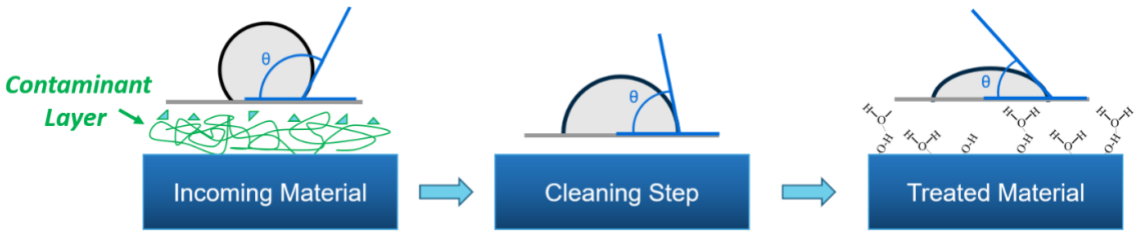


*Figure 2. Theoretical depiction of the decrease in WCA throughout a solvent cleaning and plasma treatment process on a previously contaminated surface. By measuring the change in WCA one can probe the increase in surface energy that occurs throughout treatment steps.*

When a droplet of liquid encounters a minimally porous surface, the attractive net forces between the individual liquid molecules will force the drop into a semi-spherical shape [3]. Surface tension (i.e. the surface energy of a liquid) can be used to calculate the surface energy of the substrate the liquid is in contact with by analyzing the magnitude of the liquid's self-

attraction compared to its attraction to the solid's interface [1, 3]. Thus, the angle between the perimeter of the liquid drop and the solid surface scales inversely with the surface energy of the substrate. The relationship between the angle of the liquid droplet and the surface can be quantified via Young's Equation (Equation 2) [3]:

$$\gamma_S = \gamma_{SL} + \gamma_L \cos\theta \quad [2]$$

where γS is the surface energy of the solid substrate, γSL is the interfacial energy between the liquid and solid surface after their molecules come into contact with one another, γL is the surface tension of the liquid, and θ is the angle of the semi-sphere formed by the liquid after being deposited on the solid surface.

## 1.3 Work of adhesion and modes of bonding failure

In fields related to bonding and coating, the work of adhesion is a critical parameter to probe and scales directly with surface energy. The work of adhesion can be defined as the reversible thermodynamic work required to separate two surfaces from one another (Figure 3) [11-14]. When the surfaces of two different materials encounter one another, a new interface is formed (γSL). Through Dupre's equation (Equation 3) the solid-liquid work of adhesion (WSL) is defined as [12]:

$$W_{SL} \equiv \gamma_S + \gamma_L - \gamma_{SL} \quad [3]$$

One can combine Young's (Equation 2) and Dupre's equations and use both the known surface tension of a liquid and the angle (θ) the liquid forms at the solid-liquid interface to access the work of adhesion term (Equation 4) [12]:

$$W_{SL} = \gamma_L(\cos\theta + 1) \quad [4]$$

Once an adhesive or coating has been cured, the solid-liquid work of adhesion is simplified to work of adhesion (WA) and the liquid term is replaced by the newly formed solid (Figure 3). In our previous work, we show that increasing the "wettability" of the solid adherend increases the energy required to separate it from its cured bond or coating [13-14].

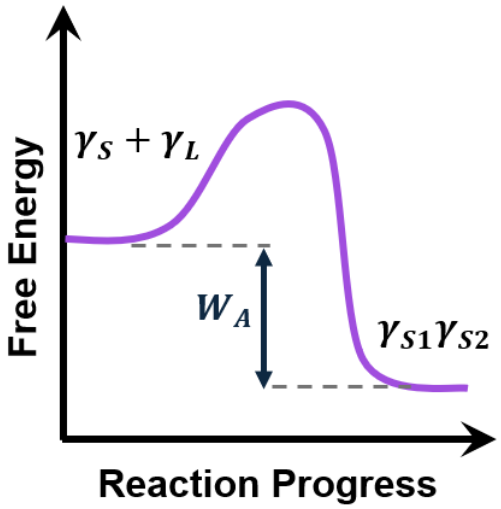


*Figure 3. Free energy diagram of the work of adhesion (WA) towards the formation of a new interface with a cured bond or coating (γS1γS2).*

There are numerous modes of adhesion failure in systems that rely on bonding and coating. Depending on the failure mode, one can often determine the weakest link in their system. Adhesion failures can be separated into three major categories: interfacial failure, cohesive failure within the adhesive, and cohesive failure within the substrate (Figure 4) [11].

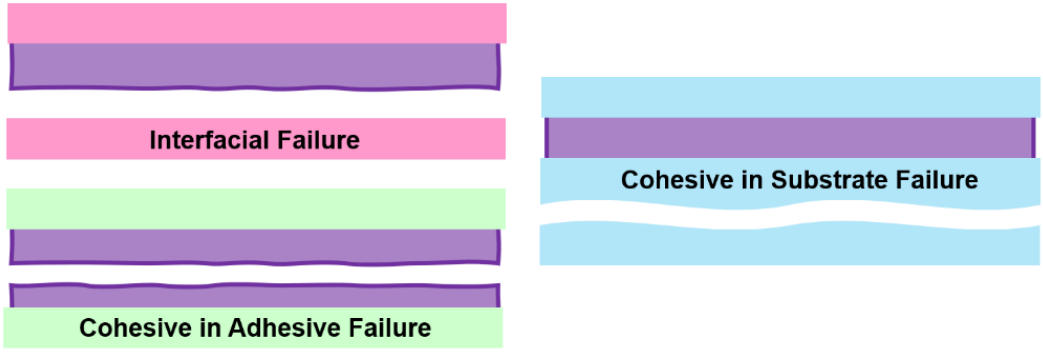


*Figure 4. Cartoon depiction of the three major modes of adhesion failure (interfacial: pink adherends, cohesive in the adhesive: green adherends, and cohesive in the substrate: blue adherends), where purple is the adhesive bonding two like adherends.*

Interfacial failure is defined by total failure at the interface between the solid adherend and whatever had been bonded/coated to it [11]. This failure mode occurs when the cohesive forces holding the adhesive or coating together are stronger than the work required to separate it from the adherend. Interfacial failure is a strong indicator of an aged, contaminated, or ill-prepared surface [11, 13].

Cohesive failure within the adhesive is when the cohesive forces within the adhesive or coating are weaker than the forces maintaining its bond to the adherend(s) [11]. When cohesive failure within the adhesive occurs, parts of the adhesive or coating will remain residually bonded to the surface of the adherend.

The last major category of bonding failure - cohesive failure within the substrate - involves failure of the adherend outside of the adhesive zone [11]. These failures occur when the cohesive forces of the adherend are weaker than the cohesive forces of the adhesive and the work of adhesion at the interface of the bond. While substrate failure is by no means optimal, it does indicate that the surface energy of the adherend was high enough to maintain a bond despite forces strong enough to damage the substrate's bulk. It is important to note that bonding failures often occur via mixed-modal path, rather than wholly one mode or the other.

Previously, we demonstrated the relationship between surface energy and adhesion by using WCA measurements to study their relation to adhesion failure mode on composites with varying cleanliness [13]. Double cantilever beam (DCB) tests and failure mode inspection illustrated the propensity for interfacial failure with increasing WCA. By establishing a system-dependent maximum WCA threshold, manufacturers could prevent interfacial failure in their systems.

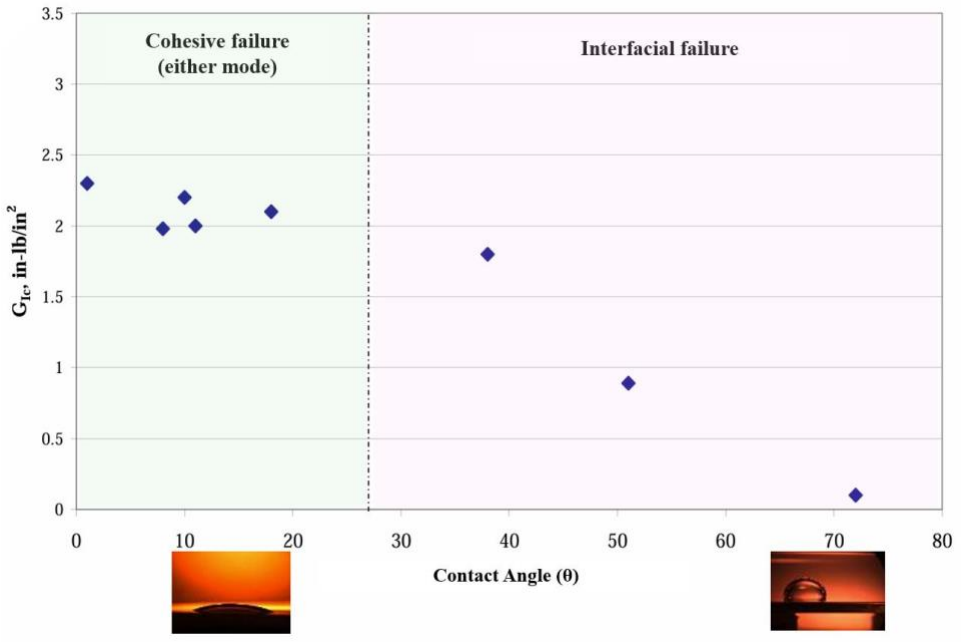


*Figure 5. Decrease in critical energy release rate (GIC) and increase in interfacial failure with increasing contact angle (θ) of composites bonded with epoxy adhesive [13].*

## 1.4 Robotic WCA mapping

Controlling the condition of a prepared surface is critical for reducing bonding and coating failures. In this work, we address the dynamic heterogeneity of surfaces by systematically comparing the WCAs that arise after implementing common industrial techniques for surface preparation - including solvent cleaning, manual abrasion, grit blasting, and atmospheric plasma treatment across both composite and metal substrates. Because of the dynamic and sensitive nature of surfaces, it can be challenging to identify if areas of the adherend have been neglected, re-contaminated, or aged towards a lower surface energy environment. We identify the inherent weaknesses in many of these preparation techniques, as well as elucidate the phenomenon of over or under preparing surfaces to their detriment, especially in more mechanically sensitive polymer-based substrates.

In this study, we quantified spatial uniformity of treated surfaces immediately after preparation and subsequently aged up to 24 hours after treatment using an in-house automated robotic WCA tester (Figure 6). This TRL-7 robotic WCA tester is currently in the laboratory demonstration phase and is the culmination of a year-long effort aimed at enhancing the software capabilities of our current commercialized TRL-9 robotic system. Through grid-style robotic WCA mapping of diversely prepared surfaces, we elucidated weaknesses that arise with manual sanding, as well as highlighted the critical step of sufficiently preparing a surface before plasma treatment to produce stable surface states conducive to reliable bonding and coating.

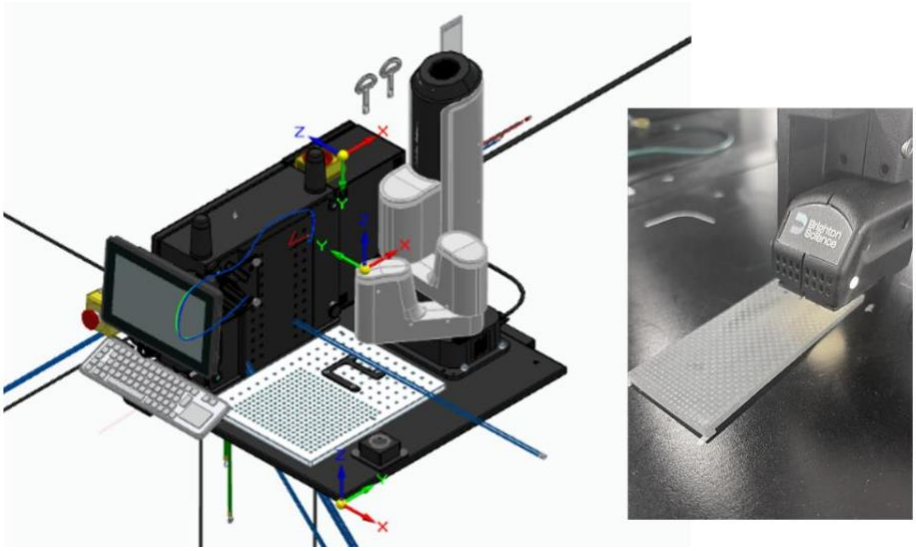

*Figure 6. Computer-aided design (CAD) image of robotic WCA tool (left) and close-up of measurement head analyzing a deposited water droplet on sanded composite (right).*

# 2. EXPERIMENTATION

## 2.1 Materials and sample preparation

Two species of carbon fiber-reinforced (CFR) composite were purchased from commercial sources and machined into approximately 2 mm thick rectangular coupons (surface area specified when relevant). On-site direct attenuated total reflectance Fourier transform infrared spectroscopy (ATR-FTIR) using a Thermo Fisher Nicolet iS20 spectrometer equipped with a germanium crystal was used to probe the surfaces of the composites. Aluminum coupons approximately 5 mm thick were purchased from commercial sources and used as received.

### 2.2 Surface treatments

We prepared composite and metal surfaces using a variety of manual and automated surface preparation techniques, including unidirectional hand wiping with Kimwipes and 99.9% purity isopropyl alcohol (IPA), manual sanding with 220 grit abrasive paper, grit blasting under vacuum with 220 grit aluminum oxide, and atmospheric plasma treatment. Atmospheric plasma treatment was performed using a Plasmatreat OpenAir-Plasma® system with an RD1004 nozzle. Surfaces were plasma treated at a standoff height of 10 mm using a single-pass parallel sweeping pattern to ensure uniform exposure.

### 2.3 Robotic WCA measurements

The robotic WCA tool was calibrated prior to testing according to specified guidelines, and daily performance checks were performed to ensure WCA reliability across different days and map geometries. All robotic WCA measurement arrays were initiated less than 5 minutes after surface treatment (unless specified) to limit surface contamination and aging, with a 1.5 µL sessile drop comprised of high purity HPLC water deposited at the same height at every measurement position. Measurements were automatically quantified through in-house software and mapped in accordance with the geometry of the samples to best articulate the surface heterogeneity of each coupon throughout treatment steps. On the WCA maps, one square box containing a number value indicates a singular contact angle interpretation from one 1.5 µL water droplet, with an inherent statistical uncertainty of ±2˚ due to software interpretation limits.

## 3. RESULTS

### 3.1 WCA mapping of CFR composite coupons

As key industries such as aerospace and automotive shift away from metals, they must broaden their scope of knowledge regarding surface preparation. It is critical to understand that surface preparation is not one-size-fits-all. There is a common misconception in the materials science community that roughening a surface is distinctly important for improving adhesion, mainly through offering greater surface area for bonding [1, 3, 14]. But adhesion failures still occur when surfaces are aggressively roughened, especially with thermoplastics and thermosets. Meanwhile, strong adhesion can often be achieved on relatively smooth

surfaces with higher-control techniques such as chemical functionalization through plasma treatment [2, 13-14].

The issue is that surface roughening is rarely an isolated process and can be challenging to perform homogenously. Sand-to-black is a colloquialism used frequently in industrial operating procedures to dictate surface preparation of CFR composites. But over-abrasion is a common and significantly detrimental phenomenon in organic systems due to polymer chain disruption and/or damage to the CF. Sanding can also add detrimental contaminants to the surface, as many sandpapers contain low-energy stearates to increase the abrasives' lifetime. Sanding wheels may also transfer detrimental contaminants to the surface; thus, any abrasion step typically requires an additional cleaning step afterwards.

It can be challenging to identify if a composite has been over-abraded by appearance alone; this is where quantitative WCA measurements are incredibly useful. A polyamide grinding wheel was used to abrade the center of a 100 mm x 25 mm IPA-wiped CFR epoxy composite for approximately 30 seconds (Figure 7). A technician may not have recognized over-abrasion anywhere but within the centermost portion of the composite's surface. But mapping of the broad range of WCAs across the surface tells a more complex story. Despite the outermost edges of the abraded section looking visually different than the heavily abraded center, they still share the same range in WCAs (53-65°) – meaning they both have a lower surface energy than the unabraded sections. The sections of the composite that were IPA-wiped but unabraded had WCAs ranging from 36-46° - indicating a higher surface energy, and thus, a superior environment for bonding or coating.

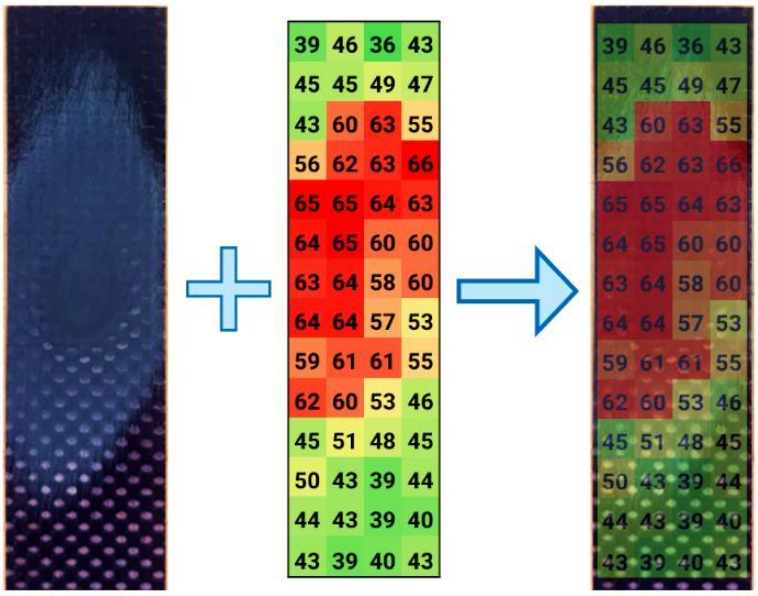


*Figure 7. WCA mapping of CFR epoxy composite abraded in the center by a polyamide abrading wheel (Mean: 53°, Standard Deviation: 9°). Surface was cleaned with an IPA wipe before and after abrasion.*

We confirmed through ATR-FTIR analysis that abrasion resulted in the removal of the epoxy resin at the center of the composite (Figure 8). The key peaks at 3000 cm-1 and 1500 cm-1, identified as belonging to epoxy resin, disappeared after abrading the center of the composite - thus indicating exposure of carbon fiber. By combining FTIR measurements and WCA mapping one can elucidate the weaknesses in their surface preparation process - thus predicting where and what mode of adhesion failure could occur first.

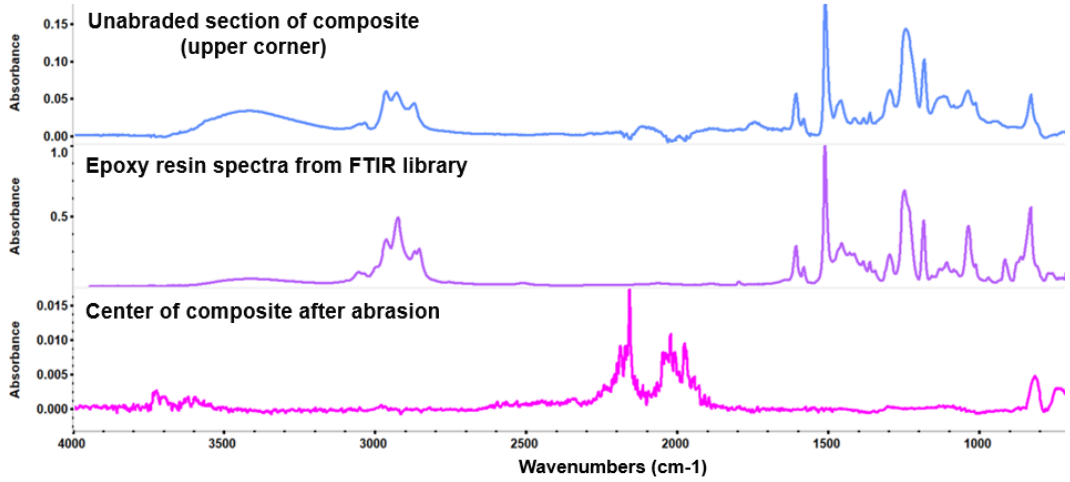


*Figure 8. ATR-FTIR spectra (in order from top to bottom) of (1) an unabraded section of the CFR epoxy composite, (2) library spectra of epoxy resin, and (3) region of CFR composite with the deepest level of abrasion.*

Many of the most common surface preparation techniques implemented in industry today were initially developed for metals. This means that their use may not be ideal when transitioning from inorganic to organic systems. Manual sanding is one of the lowest-control techniques one can implement to change the surface energy of a composite, but higher control techniques such as grit blasting using small abrasive particles can still damage a composite surface when performed for even brief durations of time. We used a 50 mm x 50 mm section of the same species of composite mapped in Figure 7 and utilized robotic WCA measurements to study the change in surface energy throughout a standard grit blasting treatment process (Figure 9).

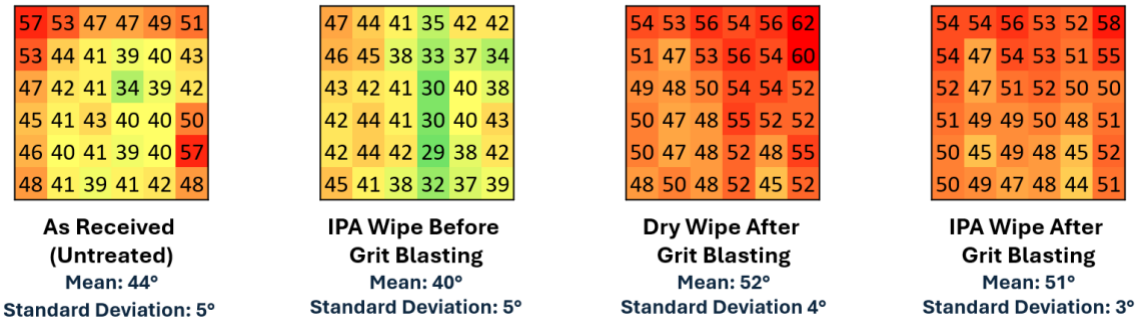


*Figure 9. WCA mapping (from left to right) of CFR epoxy composite (1) untreated, (2) directly after IPA wipe, (3) directly after grit blasting and dry wiping (to remove residual grit), and (4) after additional IPA wipe.*

The IPA wipe before grit blasting was not sufficient in preparing the composite surface in a truly homogenous manner, but it did lower the mean WCA from 44° to 40° - indicating removal of some surface contaminates. And while grit blasting reduced the homogenized the surface, it increased the mean WCA to 52°. The IPA wipe after grit blasting only lowered the mean WCA to 51°, which indicates that the sharp increase in WCA after grit blasting is not a result of surface contamination after the abrasive process, but rather, damage to the composite. While prepping surfaces homogenously is certainly important for promoting long-lasting adhesion, a mismatch in the technique selected for a particular substrate may transform the landscape to its detriment.

Atmospheric plasma treatment, a higher-control method for surface preparation, uses hot ionized gas from ambient air to clean and functionalize surfaces with polar functional groups such as -OH, -COOH, and -NH2. Thermoplastics and thermosets benefit greatly from surface functionalization because they have inherently less surface energy than clean metals. Additionally, there is significantly less danger in exposing composite fillers when plasma intensity and number of passes is tuned to the affected system. We treated a 100 mm x 25 mm CFR epoxy composite coupon by IPA wiping, performing a single pass with atmospheric plasma, and allowing the surface to age overnight to study the effects that plasma treatment had on surface stability (Figure 10). While the mean WCA (32°) and standard deviation (3°) of the composite may have been sufficiently low to promote good adhesion after IPA wiping, the desired WCA range for any bonding or coating process is system dependent. For example, composites that are bonded together by an elevated temperature cure adhesive may be more forgiving of contamination. By taking quantitative WCA measurements across different treatment steps and correlating WCA to mechanical testing results (lap shears, peel testing, etc.) one can establish a system-dependent WCA tolerance limit and reduce bonding or coating failures without repeated use of destructive tests.

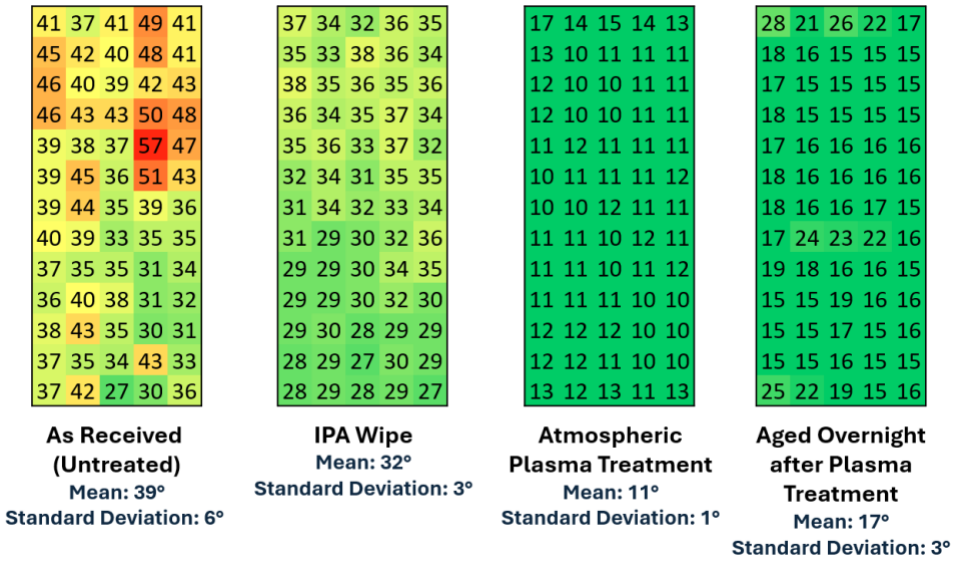


*Figure 10. Mapping of WCA measurements on, from left to right, (1) untreated CFR epoxy, (2) directly after IPA wipe, (3) directly after atmospheric plasma treatment, and (4) aged approximately 24 hours after plasma treatment.*

Atmospheric plasma treatment greatly decreased the mean WCA (32° → 11°) for the IPA-wiped composite coupon and produced a stable surface for at least 24 hours after treatment (mean WCA: 17°). The WCAs of the composite directly after atmospheric plasma treatment are on the same order of freshly abraded aluminum (Table 1, Section 3.2), indicating strong polar contributions to surface energy.

Even composite surfaces that have been maintained using peel ply can benefit greatly from additional surface analysis. We mapped an 80 mm x 25 mm poly(aryl ether)-based CFR composite that had been manufactured using wet peel ply on one of its surfaces. Robotic WCA mapping directly after peel ply removal indicated a generally homogenous surface, but a mean WCA of 51° (Figure 11). Atmospheric plasma treatment directly after peel ply removal significantly decreased both mean WCA (19°) and surface heterogeneity towards a maximum WCA of 26°.

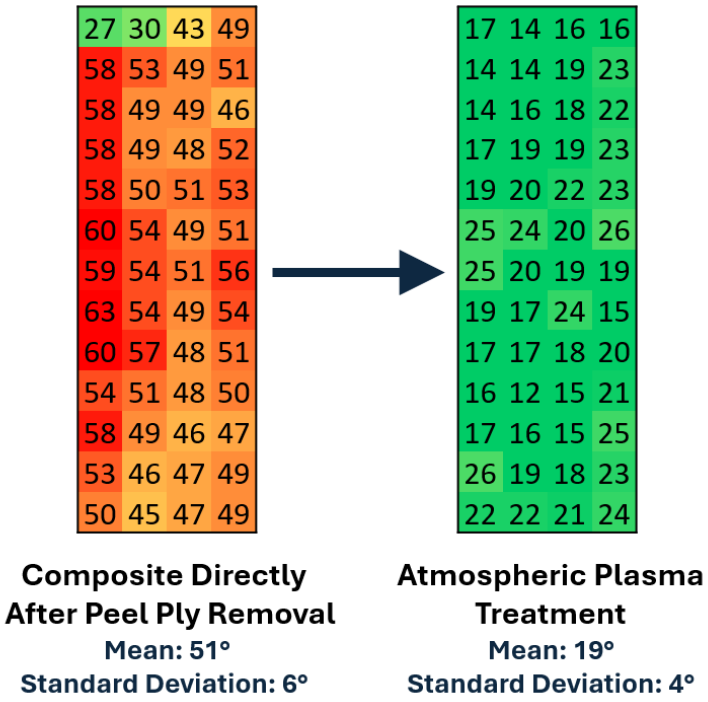


*Figure 11. WCA mapping of poly(aryl ether)-based composite coupon directly after peel ply removal and plasma treatment. No IPA wipe was utilized before or after plasma treatment.*

Using wet peel ply to protect the surface of a composite while it cures results in the first few molecular layers of the composite resembling that of the peel ply resin. Many peel plies have inherently low surface energies, which means that while they are physically protecting the surface from external contaminants, the cured substrate may require additional surface functionalization after peel ply removal.

## 3.2 WCA mapping of aluminum coupons

While clean metallic systems inherently have greater surface energies than thermosets and thermoplastics, their high reactivity leads to the formation of a metal-oxide layer quite rapidly after exposing a pristine surface. These oxide layers can be detrimental to bonding and coating as they are both lower in surface energy than the pristine metal and are weaker in mechanical strength compared to the bulk substrate. Aluminum is an excellent example of an inorganic material used frequently in manufacturing operations that experiences rapid surface aging after treatment (Table 1). One can track the dynamic nature of this and other metals' surfaces and potentially identify when an oxide layer has formed using quantitative WCA measurements.

*Table 1. Approximated WCA ranges of aluminum coupons at various stages of surface preparation/aging. Measurements taken on site across diversely-prepared aluminum coupons.*

| Condition | Approximate WCA Range |
|---|---|
| Fresh (abraded, grit blasted, plasma treated) | 0-15° |
| Aged ~1hr after treatment | 20-30° |
| Aged ~1 week after treatment | 50-60° |
| Solvent wiped after aging | 30-40° |
| Hydrocarbon oil contamination | 60-70° |
| Silicone contamination | 80-100° |

First, we mapped a 100 mm x 25 mm aluminum coupon without any surface preparation (Figure 12). A single IPA wipe lowered the mean WCA from 79° to 40°, indicating the presence of some surface contamination - possibly various machining oils as shown in Table 1. Atmospheric plasma treatment greatly homogenized the surface and lowered the mean WCA to 14°. But allowing the aluminum coupon to age overnight in ambient conditions led to a drastic increase in both surface heterogeneity and mean WCA (37°).

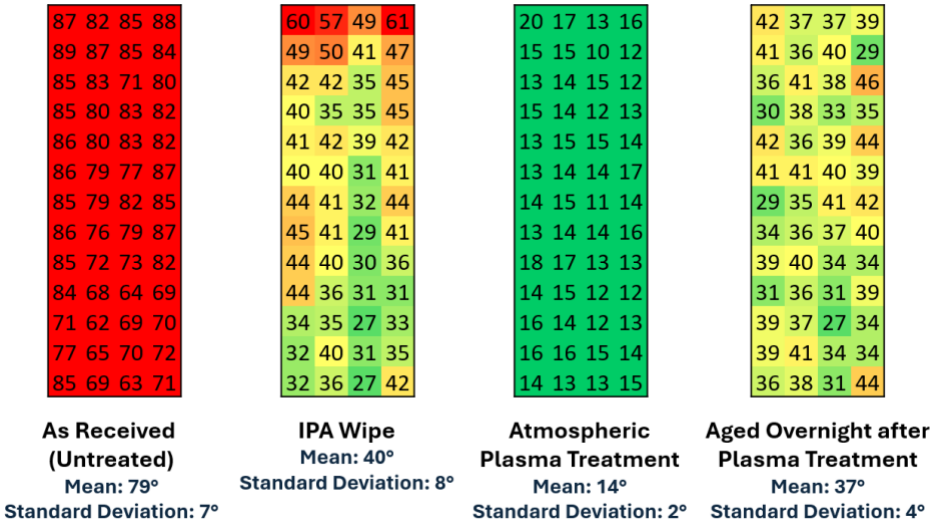


*Figure 12. Mapping of WCA measurements taken (from left to right) on (1) untreated aluminum coupon, (2) directly after IPA wipe, (3) directly after atmospheric plasma treatment, (4) aged approximately 24 hours after plasma treatment.*

While abrasion is not typically necessary for generating pristine surfaces in composite systems, the stubborn oxide layers that form quickly in highly reactive metals makes this step incredible beneficial for maintaining pristine surfaces. One can see how differently an

aluminum coupon ages when the oxide layer has been abraded before plasma treatment (Figure 13).

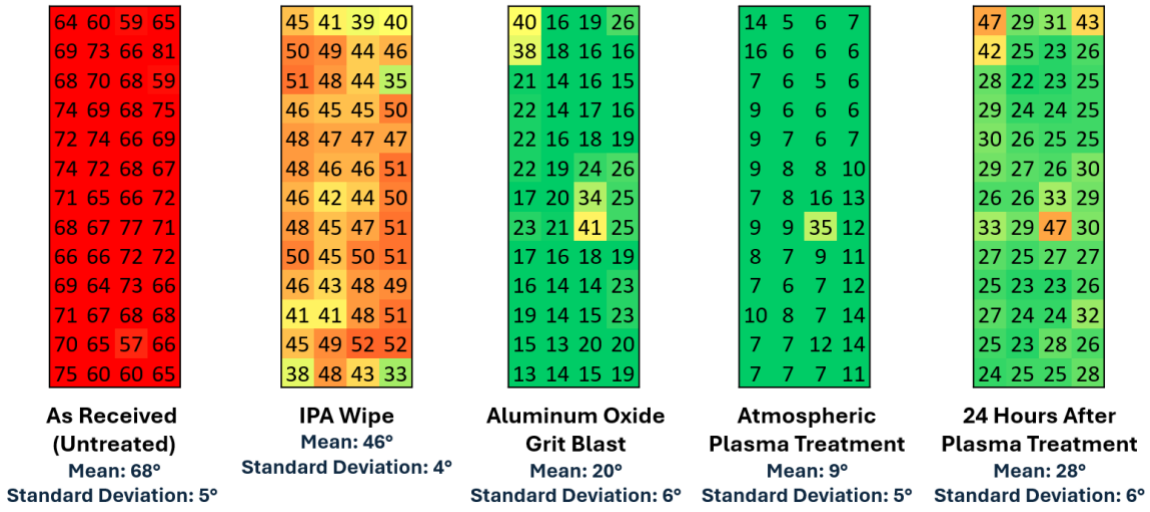


*Figure 13. Mapping of WCA measurements taken (from left to right) on (1) untreated aluminum, (2) directly after IPA wipe, (3) directly after grit blasting and a dry wipe (4) directly after atmospheric plasma treatment, (5) aged approximately 24 hours after plasma treatment.*

Adding a grit blasting treatment to the aluminum coupon before atmospheric plasma treatment led to an overall lower mean WCA of 9°, compared to 14° for the plasma treated aluminum in Figure 12. Additionally, the mean WCA only increased to 28° after 24 hours of aging, compared to 37° in Figure 12. The high reactivity of metals means that their surfaces are inherently less stable than polymers, thus more prone to WCA creep. But by physical removal of the oxide layer on the coupon's surface before plasma treatment, we prepared a more robust surface with a longer out-time.

## 4. CONCLUSIONS

Our study demonstrated that substrate preparation and process control benefit greatly through close monitoring of surface chemistry via quantitative WCA measurements. Here, we showed that automated robotic WCA mapping is a robust, non-destructive technique for identifying both the homogeneity and stability of various surface treatments across diverse substrates. Surface preparation must be tuned to the relevant substrates, adhesives/coatings, and bonding out-times in order to prevent under or over preparation of surfaces. Our results highlighted significant WCA heterogeneity associated with manual preparation techniques and emphasized the sensitivity that composite surfaces have to being prepared with abrasives.

Due to the diverse tolerances of different surfaces, an explicit WCA value cannot be provided here as a pass/fail for manufacturers. But, by combining quantitative WCA measurements, FTIR analysis, and mechanical studies, one can identify common contaminants in their manufacturing areas, as well as determine a maximum allowable WCA threshold for their specific systems [13]. While mapping is not necessarily feasible for all manufacturing environments, it does elucidate locations that have been under or over prepared to their detriment. Ultimately, integrating even selective quantitative WCA measurements into workflows offers a practical path toward improved quality control and earlier detection of weaknesses in surface preparation.

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