TECHNICAL PAPER

Correlating Water Contact Angle with Water Break Measurements as an Indicator of Surface Energy

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Introduction

WATER BREAK TEST

This is a common pass/fail method used to evaluate surfaces for the presence of hydrophobic contaminants, which can be detrimental to adhesion of paint or an adhesive. It is a qualitative means of evaluating surface energy, which is directly related to surface cleanliness. In this test, a stream of water is visually evaluated as it flows over a surface. If it spreads out into a continuous, unbroken sheet, it indicates that the surface is substantially free of hydrophobic contaminants. If the surface is contaminated with low surface energy substances, the flowing water will not sheet uniformly over the surface but rather it will break into rivulets and tend to bead up (termed “water break”).

Water break tests are not ideal as a quality control tool. They are messy: a relatively large amount of water is used which has to be removed and the component must be dried before coating or bonding. Cases of water break tests contaminating sensitive surfaces because of impure water or transfer of contaminants during the drying process are not uncommon. Because the result is only a binary ‘water break free/not water break free,’ it is unknown whether it is too sensitive for some applications or not sensitive enough.

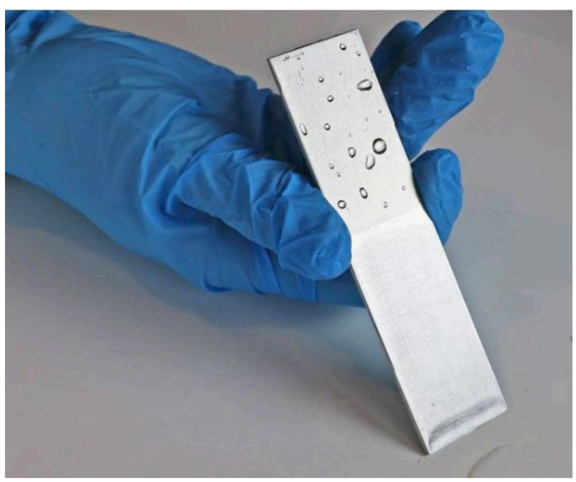

Figure 1. *Depiction of water break test results. Top portion of the panel displays a non-water break free surface (water beads up). Bottom portion displays a water break free surface (water spreads out).*

WATER CONTACT ANGLE ANALYSIS

Like a water break test, contact angles are sensitive to the top few molecular layers of a surface, the region that is responsible for adhesion. Unlike water break tests, water contact angle (WCA) measurements provide a rapid and quantitative measure of surface energy that can be directly and proportionally related to surface cleanliness and adhesion. A clean surface with high energy will display a low contact angle: the surface tension of the water droplet will be overcome by the energy of the surface and spread out (i.e. the water molecules are more strongly attracted to the high energy surface than to themselves). Conversely, a surface that is contaminated will display low surface energy and produce a high contact angle: the water molecules are more attracted to themselves than the surface, and the droplet will bead up. This is shown in Figure 2. The amount that the drop spreads out or beads up is quantified by the contact angle (the angle formed by the drop perimeter and the surface), and is directly proportional to the amount of contamination on the surface. As an example, Figure 3 shows the relationship of the contact angle of water to the amount of a silicone contaminant applied to a carbon fiber reinforced composite material surface.

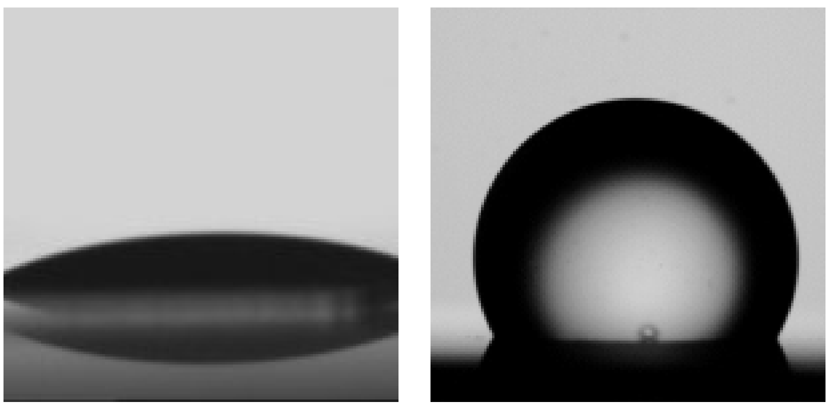

Figure 2. *Image on the left displays a low WCA indicative of a high-energy surface (clean). Image on the right displays a high WCA indicative of a low energy surface (contaminated, or otherwise not clean).*

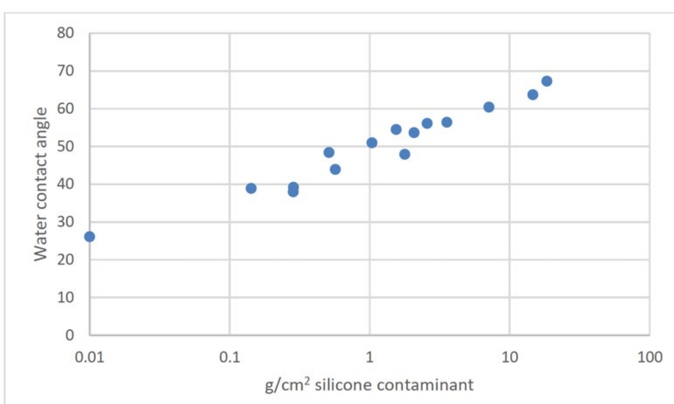

Figure 3. *Quantitative relationship between the water contact angle and the amount of silicone contaminant on carbon fiber reinforced epoxy composite.*

The Brighton Science Surface Analyst™ obtains these measurements in a couple of seconds, automatically interprets the data, and stores the data in an easily retrievable form for documentation of process control and quality. Because of the proportional nature of water contact angle measurements to surface quality, and because they are sensitive to amounts of contamination that are well below those that can cause adhesion issues, these measurements can catch process drifts *before* they result in product quality issues. This white paper explores the relationship between the water contact angle (as determined using the Surface Analyst) and a water break test performed according to ASTM standard procedure and contrasts the quantitative sensitivity of contact angle measurements with the binary nature of the water break test.

Results

EXPERIMENTAL

Peel ply composite laminates utilized for this study were peeled and in some cases immediately contaminated with various amounts of siloxane mold release. The surface energies of these laminates were then characterized using two methods: 1) WCA measurements via Brighton Science's Surface Analyst, and 2) the water break test. For this study, the water break test was performed by applying distilled water to composite laminate surfaces via a spray bottle.

RESULTS

Both water break and water contact angle showed correlation to contamination level. Figure 4 shows the water contact angle correlation to contamination level. The higher the contamination level, the higher the contact angle which indicates a low surface energy and poorly cleaned surface. Figure 5 shows the results from the water break test. While the interpretation of the results is somewhat subjective, it appears clear that 12 µg/cm^2 of the siloxane contaminant produced a surface that was no longer water break free. This value is indicated on Figure 4 by the vertical red line, and shows the quantitative sensitivity of the water contact angle measurement to levels of contamination well below those that result in a non-water break free surface. It is interesting and perhaps significant that 10µg/cm^2 or less of this contaminant has been found in other studies to be the threshold detrimental level to structural adhesive bonds on aerospace composite materials.

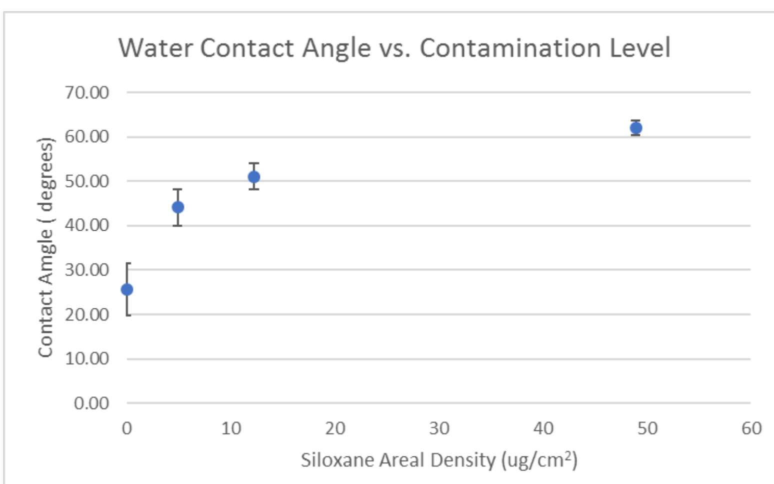

Figure 4. *WCA response as a function of siloxane contamination level. Each point represents an average of 15 measurements.*

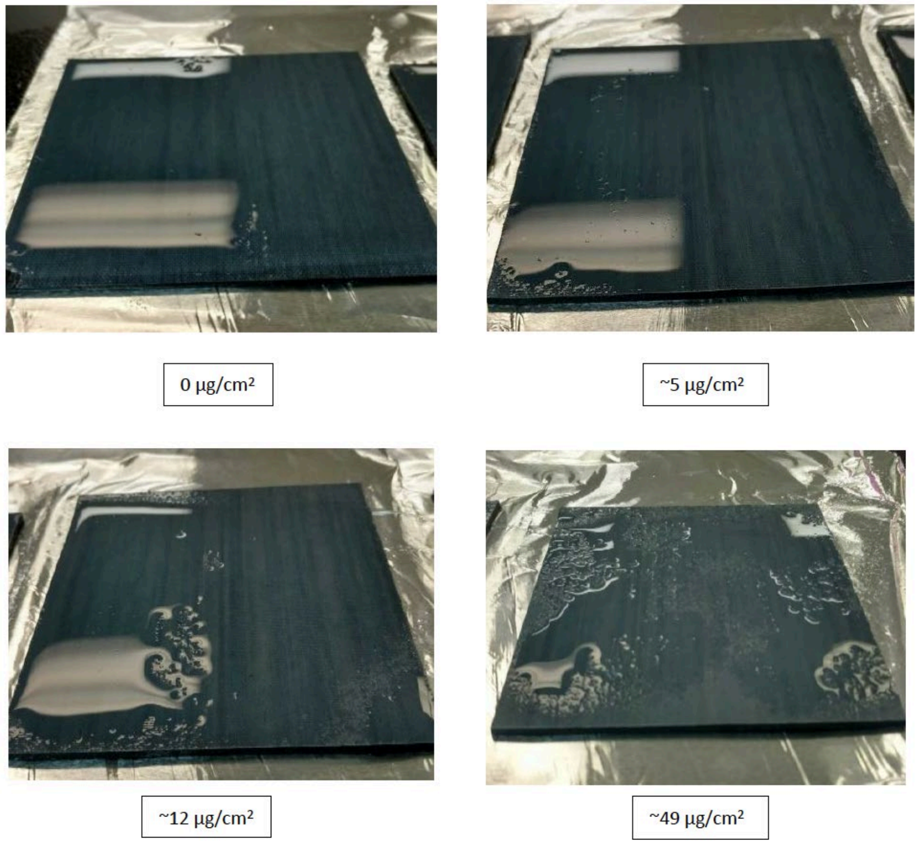

Figure 5. *Composite laminate surfaces subjected to the water break test. Water break free surfaces were observed for 0, 5 and 12 µg/cm2. Above 12 µg/cm2 a completely non-water break free surface was observed.*

CONCLUSIONS

While both water break and water contact angle measurements show a positive correlation with contamination level, water break measurements are highly subjective and are not able to quantify direct to the level of contamination. Conversely, water contact angle measurements are objective and quantitatively proportionally to the amount of contamination.