Water Contact Angle Correlation to Crosshatch Adhesion Tape Test in Accordance with ASTM D3359 -09

Introduction

Manufacturers are familiar with adhesion challenges when attempting to bond, paint, etch, or coat a substrate. Poorly prepared surfaces can undoubtedly produce problematic adhesion issues. A common standard for measuring the effectiveness of paint adhesion relies on an archaic method: The Tape Test. While the Tape Test can provide some insight on adhesive ability, its analysis is subjective, the process is time consuming, and it is destructive to the part/surface being tested. There are also many conditions that are difficult to control and maintain consistency: the force at which the tape is peeled, the speed at which the tape is peeled from the surface, as well as the pressure at which the crosshatch grid is applied all contribute to the test's downfalls. Furthermore, the test can only be done post treatment and application or in the lab for research and development. These limitations render the Tape Test impractical or impossible to use as a surface readiness indicator prior to bonding on the manufacturing floor.

There exists an objective, quantitative, and non-destructive surface energy gauge, the Surface Analyst. This portable instrument measures surface energy using water contact angle that directly relates to surface cleanliness and preparedness for adhesion. These characteristics are ideal for in-line process monitoring on a factory floor as well as research and development in a laboratory.

BTG Labs' Materials and Processing Engineers performed an analysis to compare surface measurements with the Tape Test and water contact angle measurements taken with the Surface Analyst.

Experimental

Polypropylene samples were precleaned prior to treatment by washing with Dawn detergent and warm water, followed by a DI water rinse. The precleaned samples were treated with air plasma with a standoff height of 13.4mm and with varying speeds; 50mm/s, 100mm/s, 150mm/s, 250mm/s, 350mm/s, and 500mm/s. These correspond to plasma residence times of 0.5, 0.25, 0.1, 0.07, and .05 seconds.

Surface Analyst water contact angle measurements were taken on polypropylene samples both before and directly after plasma treatment. Samples were then primed with 2 coats of DPLF

Epoxy Primer. Primer was applied according to technical data sheet with adequate times allowed for drying between coats, in accordance with industry standards. Black Envirobase High Performance (EHP) waterborne basecoat automotive paint was then applied to the samples within 30 minutes of primer coats and allowed to dry for 24 hours prior to testing.

Adhesion testing was done in accordance with ASTM standard D3359-09, *Standard Test Method for Measuring Adhesion by Tape Test (see Figure 1).* This included using a crosshatch tool to scratch a grid into the surface. A piece of adhesive tape was then placed over the grid and pressed firmly into place. The tape was then removed at a medium pressure at 180 degrees. The percentage of paint pulled off with the adhesive in relation to the grid was examined.

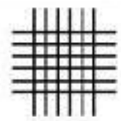
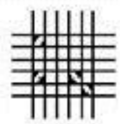
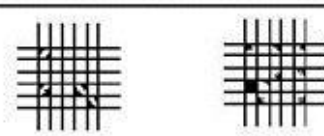
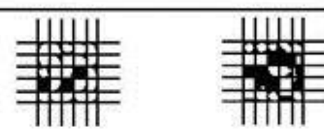
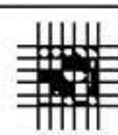
Classification of Adhesion Test Results		
Classification	Percent Area Removed	Surface of Cross-Cut Area From Which Flaking has Occured for Six Parallel Cuts and Adhesion Range by Percent.
5B	0% None	
4B	Less Than 5%	
3B	5-15%	
2B	15-35%	
1B	35-65%	
0B	Greater Than 65%	

Figure 1. ATSM D3359 Classification of Adhesion Test Results

Results

Figure 2 shows the water contact angle measurements obtained from each part as a function of plasma residence time. Increasing the plasma residence time decreased the measured contact angle, indicating increased surface energy. Figure 3 shows the images of the cross-hatch tape test and the correlation between adhesion and plasma residence time as measured by ASTM D3359. Figure 4 demonstrates the results from the water break images and their correlation to water contact angle measurements. Although the nature of the ASTM classifications make precise correlation with treatment level subjective for intermediate adhesion levels, this test shows that contact angles below about 40° are associated with significantly improved adhesion, and that contact angles of approximately 20° are associated with no observable paint delamination.

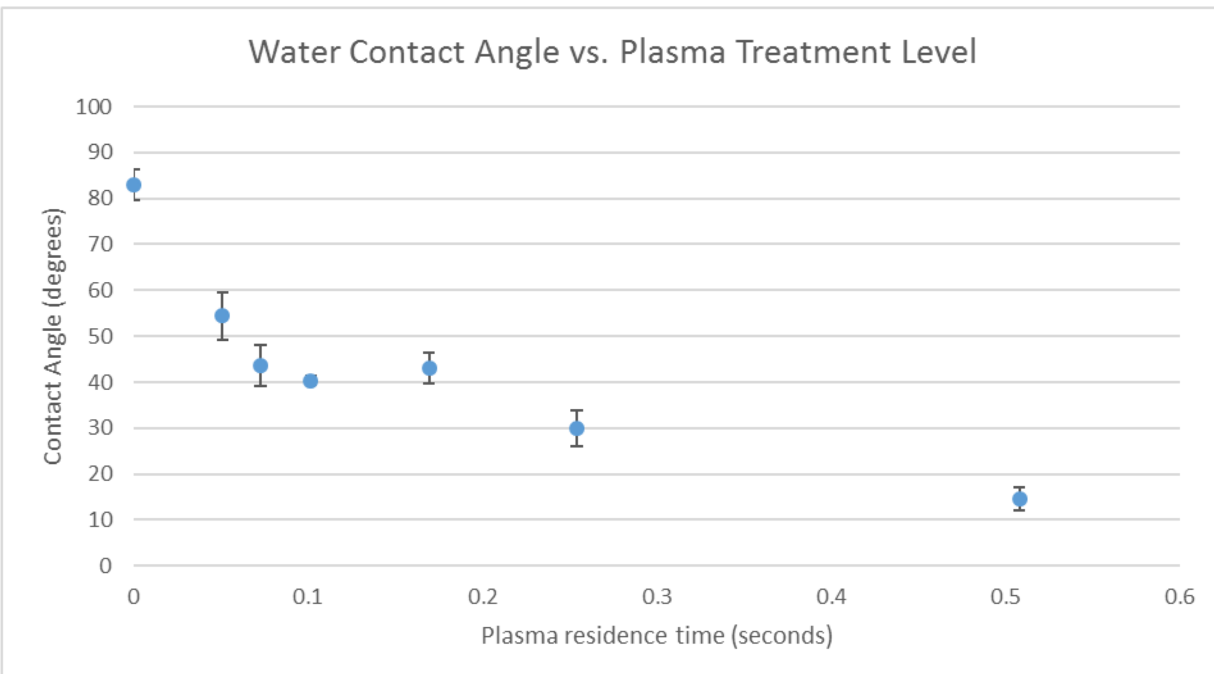


Figure 2. Water contact angle measurements as they correspond to plasma treatment on polypropylene surfaces.

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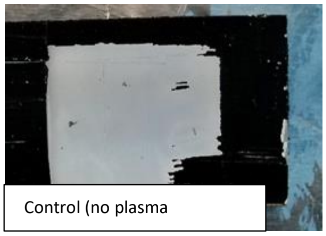
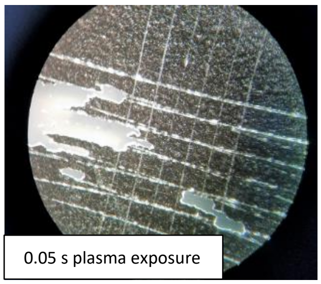
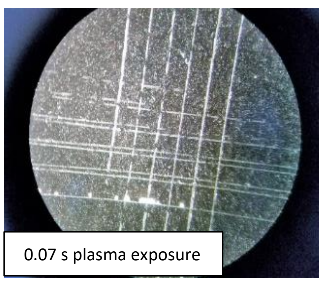
Figure 3. Cross hatch images ordered according to ASTM classification


Control (no plasma

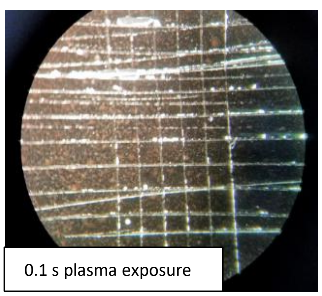
Greater than 65% adhesion failure, classification 0B (ASTM).


0.05 s plasma exposure

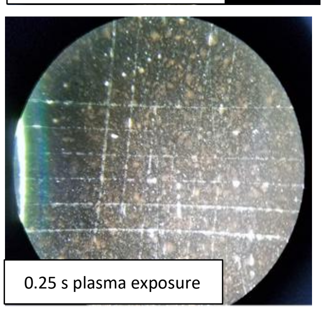
15-30% adhesion failure, classification 2B (ASTM)


0.07 s plasma exposure

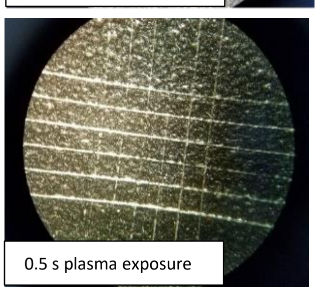
5-15% adhesion failure, classification 3B (ASTM)


0.1 s plasma exposure

Less than 5% adhesion failure, classification 4B (ASTM)


0.25 s plasma exposure

Less than 5% adhesion failure, classification 4B (ASTM)


0.5 s plasma exposure

0% adhesion failure, classification 5B (ASTM)

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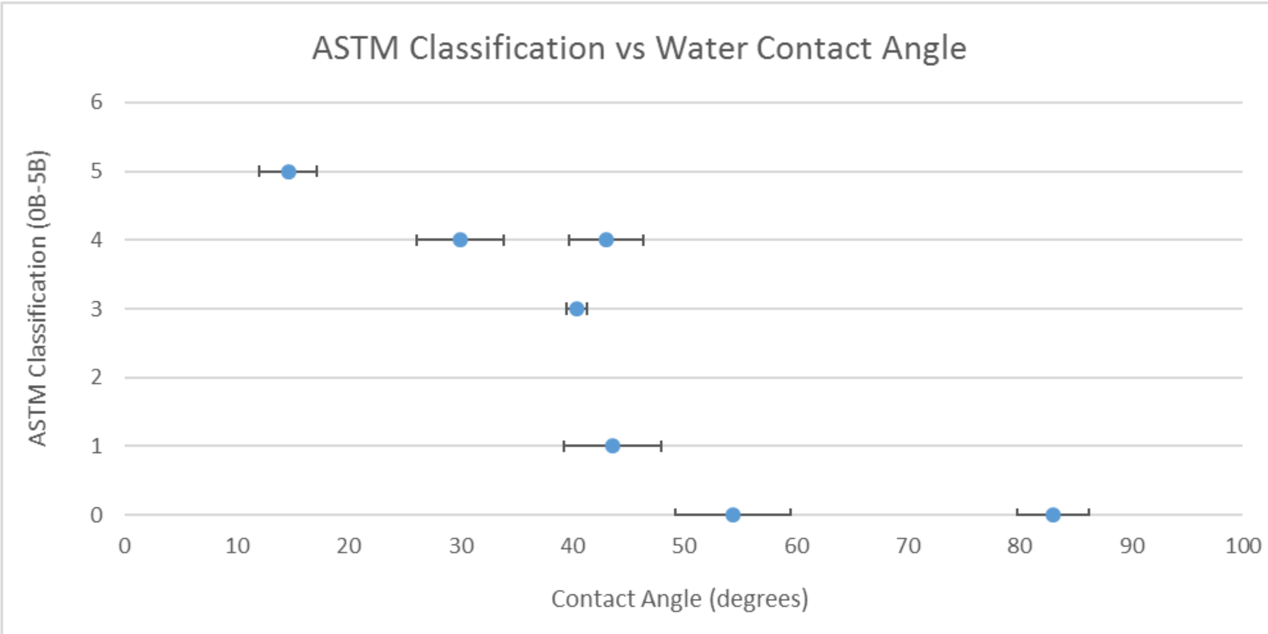

Figure 4. Contact Angle vs. ASTM Classification. Classifications; 5B – 0% adhesion failure, 4B - <5% adhesion failure, 3B – 5-15% adhesion failure, 2B – 15-35% adhesion failure, 1B – 35-65% adhesion failure, 0B - >65% adhesion failure

Conclusions

These data show that water contact angles measured using the Surface Analyst correlate very well with plasma exposure time. Because of the dependence of water contact angles on the same surface characteristics that determine adhesion, these contact angles are an excellent predictor of paint adhesion. The rapid, quantitative, and non-destructive nature of the contact angle measurements when compared to the ASTM D3359 Tape Peel Test make contact angle measurements a more practical, economical, and accurate quality assurance test for evaluating treatment level and treatment consistency. With this paint/substrate/surface treatment system, contact angles with an average of 15° corresponded with 0% adhesion failure. Contact angles with an average of 35° correspond with less than 5% adhesion failure. Contact angles with an average of 40° correspond with a 5-30% adhesion failure. Contact angles greater than 50° correspond with total adhesion failure. The Tape Test does offer some insight on adhesive ability, however the measurements are subjective and not highly precise as they rely on the user's interpretation. Lastly, the test cannot be used in a factory setting to monitor surface preparation prior to adhesion. Water contact angle measurements offer more precise insight into adhesive ability prior to bonding, painting, etching, or coating—directly on the manufacturing floor.