



The Surface Analyst™: Advanced Goniometry for Manufacturing

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The Evolution of Goniometry

The relationship of the shape of a liquid drop on a solid surface to surface energy and surface tension dates back at least to Thomas Young in the early 19th century. Contact angle goniometry (literally “angle measurement”) as a means to characterize liquid-solid interactions took a major step forward in the 1960’s with the development of the NRL (Naval Research Laboratory) goniometer by William Zisman. Most current contact angle goniometers are based on this enduring design.

The NRL goniometer is a precision instrument well suited for laboratory investigations; we used one extensively in our work on the Composites Affordability Initiative (CAI) program in the late 1990’s when we helped establish the relationships between surface treatment of composite materials, surface energetics, and adhesive bond performance. At the close of the CAI program, the value of these measurements was recognized, but it was also recognized that a standard NRL-style goniometer was not suitable for making these measurements in a manufacturing environment. What was needed was an instrument that could be used on the variety of surfaces that are encountered in aircraft assembly. The Air Force Research Laboratory (AFRL) provided support for development of such an instrument, and this support resulted in development of the SA1000, the first Surface Analyst™.

The Surface Analyst vs a Benchtop Goniometer

There are fundamental differences between an NRL-style goniometer and the Surface Analyst, most of which contribute to the value of the measurement for development and control of surface sensitive manufacturing processes. These include the method of liquid deposition and the method of contact angle calculation once the liquid drop is deposited. The original motivation for these differences was to allow for a more compact and convenient instrument that could be easily handheld. However, they also significantly improve the speed and accuracy of the measurement as well as the flexibility of the types of surfaces that can be measured.

One of the most important innovations of the Surface Analyst technology was the development of Ballistic Deposition to deposit the liquid drop on the surface. A standard goniometer creates a liquid drop on the end of a piece of tubing, which is then transferred to the surface by touching the drop to the surface. Adhesion of the liquid to the surface will then detach it from the tubing. In Ballistic Deposition, a pressurized microfluidic system constructs a drop on the surface through a pulsed stream of microdroplets. This deposition process allows drops to be rapidly built on surfaces regardless of orientation or curvature.

The kinetic energy imparted to the growing drop during Ballistic Deposition plays a major role in the utility of the measurement. The perimeter of the growing drop expands under impact of the microdroplets, then contracts to an equilibrium shape between impacts due to liquid surface tension. In this manner the perimeter of the growing drop advances and recedes multiple times during deposition

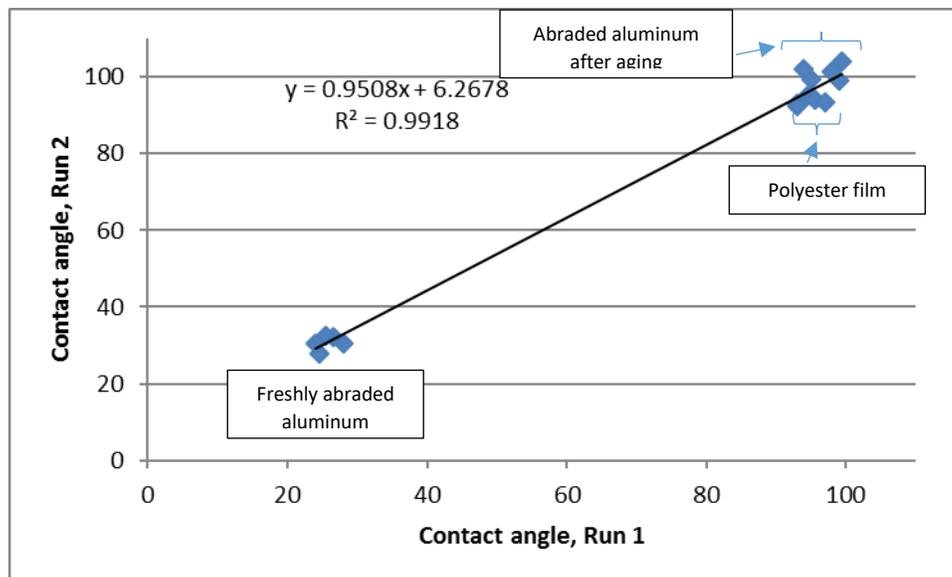


and avoids becoming pinned by features that are part of the surface of almost all manufactured articles, such as roughness, dust particles or chemical heterogeneities. Unlike a syringe-deposited drop, the final contact angle is influenced very little by variations in surface roughness. The amount of kinetic energy imparted to the drop is controllable, which allows tuning the instrument to provide an advancing or receding contact angle.

The standard Surface Analyst configuration is adjusted to eject microdroplets at a velocity of about 1 meter per second. This level of kinetic energy results in a contact angle is close to the receding angle and therefore somewhat lower than the static angle that is generally established using a syringe-deposited drop with a NRL-type goniometer. This Ballistic Contact Angle shares some characteristics with both the receding angle (Sensitive to the same surface characteristics that control adhesion) and the advancing angle (able to measure high-energy metal and ceramic surfaces that would show a receding angle of 0°) is very sensitive to subtle changes in surface chemistry.

Repeatability and Accuracy

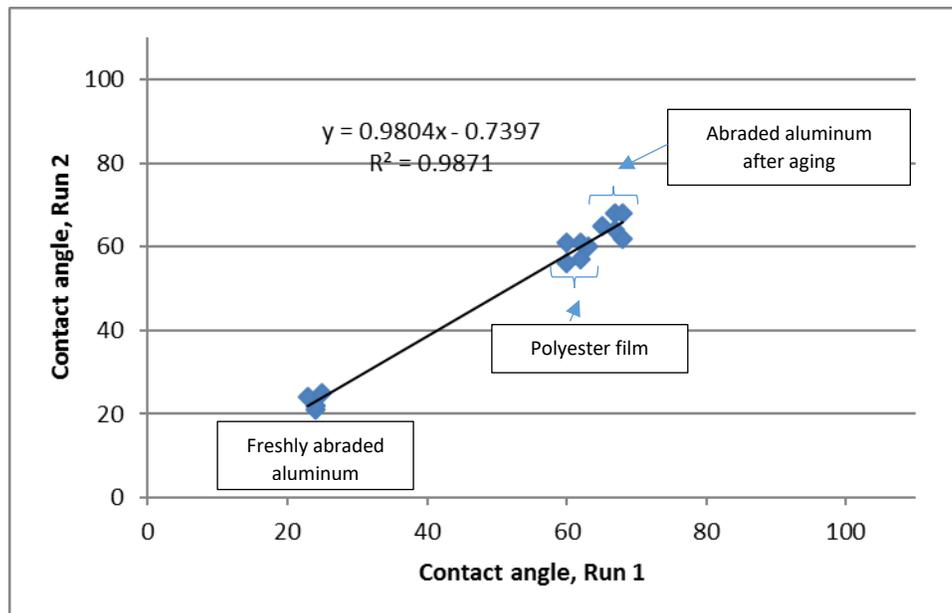
This Figure shows the contact angles established using syringe deposited drops and measured with an NRL goniometer on three surfaces: freshly abraded aluminum, aluminum that was abraded several days before the measurement was taken, and a sample of a polyester film. The first and second set of measurements correlate well ($R^2=0.99$), which shows that the measurement is repeatable. However, the contact angles on the polymer film were indistinguishable from those on the aged aluminum surface.



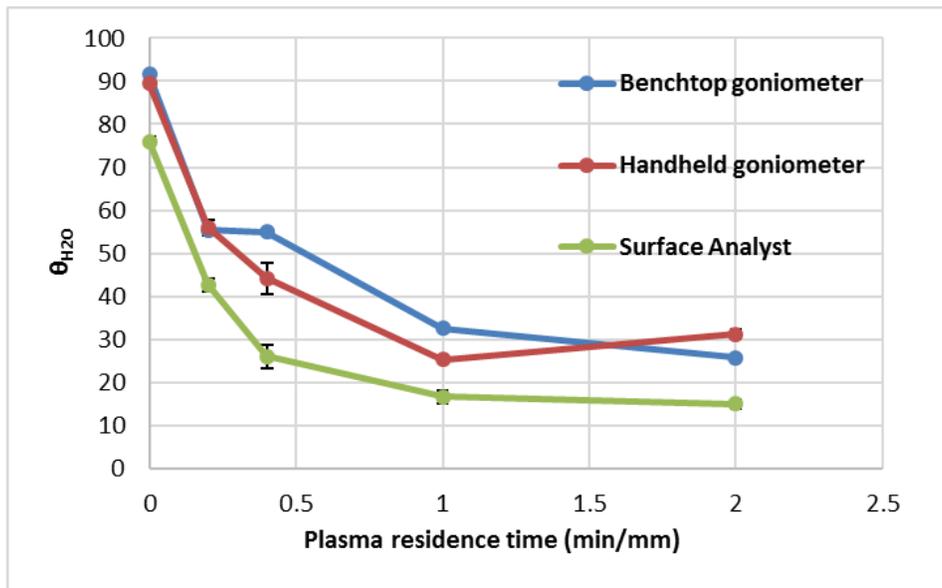
The next Figure shows the results obtained using the Surface Analyst on these same three surfaces. Contact angles on the polymer film and aged aluminum were lower than for the syringe deposited drops, as these surfaces show hysteresis and have a lower receding contact angle. The repeatability was excellent ($R^2=0.99$). Most importantly, the data shows three clearly distinct populations of contact



angles. The Ballistic Deposition process was more sensitive and more readily distinguished between the three different surfaces.

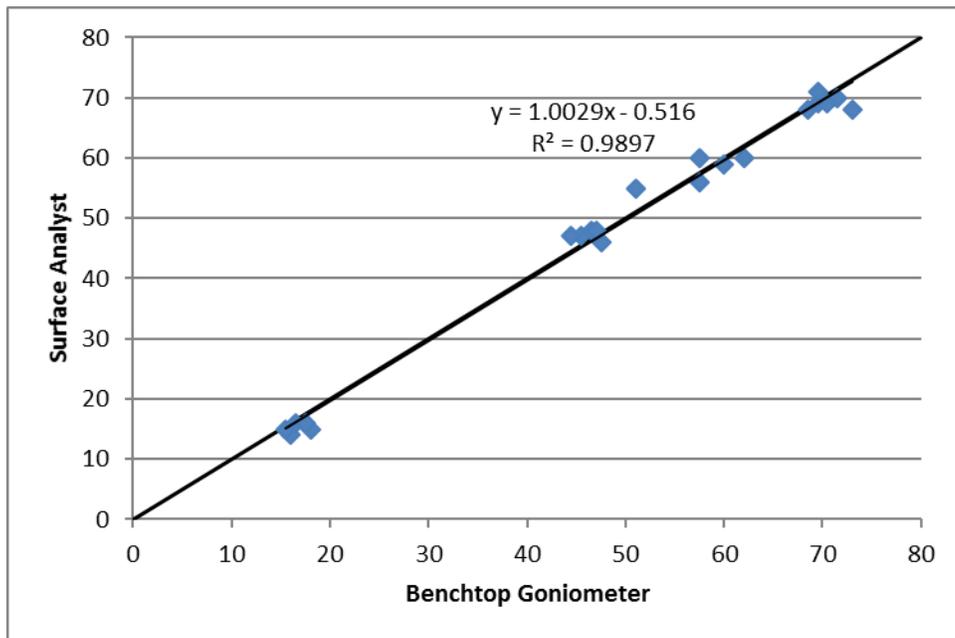


Further evidence of the sensitivity of the Ballistic Deposition process to subtle changes in surface chemistry is seen in the results of a comparison by an independent laboratory of the response of the Surface Analyst and two standard goniometers to the effects of atmospheric plasma treatment on aluminum. The following Figure shows that the contact angle measured by all three instruments decreased as a function of residence time in the plasma, showing that the surface energy increased due to cleaning. However, the data generated using the Surface Analyst showed a much smoother more consistent response to treatment level. This is due to the ability of Ballistic Deposition to overcome surface heterogeneities and establish a true equilibrium drop shape that is characteristic of the underlying surface chemistry and not affected by factors such as surface texture.



Another very useful characteristic of the Surface Analyst technology is the use of a top-down image of the drop to calculate the contact angle, rather than the profile view used by NRL-type goniometers. This simplifies the optics by removing the need for reflex mirrors to acquire an image, which allows the measurement head to be made much smaller and obtain measurements from restricted locations on complex parts. Measurements can even be made from recessed surfaces or surfaces where a profile view is obstructed by other features of the parts. Perhaps most importantly, the calculated contact angle is not from only two diametrically opposed locations on the drop, but represents an average of the entire drop perimeter. This improves the precision of the measurement on surfaces that are textured or chemically heterogeneous. The versatility of the measurement is also greatly increased, as most of the surfaces encountered in manufacturing and field situations (such as aircraft repair) tend to be textured and have some level of heterogeneity.

The algorithm used by the Surface Analyst to calculate the contact angle does an excellent job. To evaluate this, the Surface Analyst was used to deposit water drops on a wide variety of surfaces placed on the stage of a benchtop goniometer. This Figure shows the contact angles for each drop calculated by the Surface Analyst (Y axis) with the value measured for the same drop from a profile image using the optics of the goniometer.



The correlation is excellent ($R^2=0.99$), the slope is indistinguishable from 1.00, and there is less than 1° offset, confirming the validity of the top-down measurement system used in the Surface Analyst.

To conclude, the Surface Analyst is a goniometer in the sense that it measures an angle. However, it differs in fundamental ways from instruments that are based on traditional benchtop contact angle goniometers in the way it uses Ballistic Deposition to establish the contact angle, combined with a top-down imaging approach. These characteristics result in an instrument that is easy for manufacturing personnel to use without lengthy training. It allows objective and extremely sensitive and repeatable measurements of surface chemistry that are especially convenient to use for manufacturing process control.