The Use of Contact Angle Analysis to Determine Surface Cleanliness

Introduction

Recent advances in coating, painting and cleaning technologies have pushed surface preparation and cleaning requirements to new limits. Most everyone knows that surfaces need to be clean but unfortunately not many have any idea of just how clean these surface need to be. Worse, there seems to be no reliable and quantifiable method to determine just how clean a surface is. One method of qualifying surface cleanliness that is gaining in popularity is contact angle measurement.

What is contact angle measurement?

Contact angle measurement is a simplified method of characterizing the interfacial tension present between a solid, a liquid, and a vapor. If a liquid with well known properties is used for the measurement, the resulting interfacial tension can be used to identify the nature of the solid used in the measurement. The technique is extremely surface-sensitive, with the ability to detect partial monolayers.

When a drop of liquid rests on the surface of a solid, its shape is determined by the balance of the interfacial liquid/vapor/solid forces. For example, when a droplet of a high surface tension liquid is placed on a solid of low surface energy, the liquid surface tension will cause the droplet to form a spherical shape (lowest energy shape). A common illustration of this phenomenon is the behavior of water droplets on a freshly waxed surface such as an automobile fender. We say that the water "beads up" as it forms spherical shapes which seem to slide off the surface.

Conversely, when the solid surface energy exceeds the liquid surface tension, the droplet is a flatter, lower profile shape. This is easily seen in the case of a red wine glass as the wine wets or "sheets" the surface.

By viewing small droplets of the liquid of interest in profile the effects of interfacial tension can be readily observed. In order to define these droplet profiles it is common to draw a line tangent to the curve of the droplet at the point when the droplet intersects the solid surface. The angle formed by this tangent line and the solid surface is called the contact angle (see Figure 1).

How does contact angle measurement detect contamination?

Contact angle measurement can be used to detect the presence of films, coatings, or contaminants which have a surface energy that is different than the underlying substrate. This detection capability is directly related to the difference in the relative surface energies of the materials.

Perhaps the most common example of this contaminant detection is the testing of metal surfaces for the presence of oil. Most common fabrication metals such as aluminum or steel have very high surface energies. Accordingly, a clean metal surface would cause a water droplet to exhibit a very low contact angle. However, organic materials such as cutting or

forming lubricants have very low surface energies. If the surface of a metal is coated with a film of oil, the contact angle observed will be much higher than expected. This will be true even for films only one molecule thick.

How does contact angle measurement compare to other methods of surface evaluation?

There are many other methods which utilize interfacial tension to evaluate surfaces for cleanliness. Many in the industry are familiar with the "water break test". This test is a crude method of determining surface cleanliness which basically involves the ability of water to wet a surface completely and uniformly. The science behind this test is well suited for the task, but the actual methods used are completely subjective and non-quantifiable. However, this method can be useful for evaluating large surface areas.

A more accurate test of wettability involves the use of dyne pens to determine the point at which the surface of interest is wettable. For many coating and plating applications this test provides adequate information about the cleanliness of the surface. The critical surface energy of the solid can be determined within a certain range of the pen fluids used. Unfortunately, this method also suffers from a degree of subjectivity as the tester must reliably observe the wetting of the dyne fluid each time and report the results consistently. In theory this is simple; in practice it is somewhat difficult.

Contact angle instruments

The traditional contact angle measurement instrument consists of a sample stage, a method of applying a drop of liquid (usually a syringe), a light source which can illuminate the droplet in a silhouette mode, and a set of optics for magnifying the image for observation. A typical instrument is shown in Figure 2.

These optical instruments are operated by placing a small (1 - 25 microliters) drop of liquid onto the surface to be studied. The silhouetted image of the droplet is observed through the optical magnifier. A rotatable reticule allows the operator to position a reference line at the edge of the droplet to represent the tangent to the curve of the droplet. A protractor within the optics then provides a direct readout of the contact angle.

Unfortunately, operator subjectivity again comes into play in this measurement technique. The positions of the tangent line and the base line of the droplet are estimated by operator judgement. Because the images are often small and the optics difficult to use, a fairly large error (5 - 10°) is possible. In addition, the optical phenomenon known as parallax can cause a wide degree of inconsistency between operators.

New systems available (see Figure 3) utilize precision optics and CCD cameras in conjunction with image processing hardware and software to perform contact angle analysis quickly, easily, and accurately. In practice a droplet of liquid is dispensed onto the surface of interest then a CCD camera displays the profile of the droplet on a computer screen. The image is captured into the computer memory and analyzed to determine its shape. The computer software then calculates the tangent to the droplet shape and computes the contact angle.

Who needs contact angle analysis?

Once confined to academic and corporate research laboratories, contact angle analysis is becoming a popular surface evaluation technique in many industries. The finishing industry has evolved into a high technology industry, employing contact angle analysis as well as many other many analytical techniques to improve existing and innovate new finishing technologies.

The increased accuracy and reliability of the new video contact angle systems has extended contact angle analysis into other areas. For example, silicon wafers are being analyzed for cleanliness prior to processing. Also, the large glass panels used for fabricating flat panel displays for laptop computers are analyzed for cleanliness using contact angle analysis.

Conclusion

The cleanliness of surfaces prior to finishing or processing has become a vital concern in a wide variety of industries. Until recently, subjective methods of qualifying these surfaces have been the only techniques available. New contact angle instruments utilizing computerized video processing offer the ability to quantify the cleanliness levels of these surfaces. As the applications for more critical finishing techniques increase, the use of an accurate, repeatable method of measuring cleanliness will become a necessity.

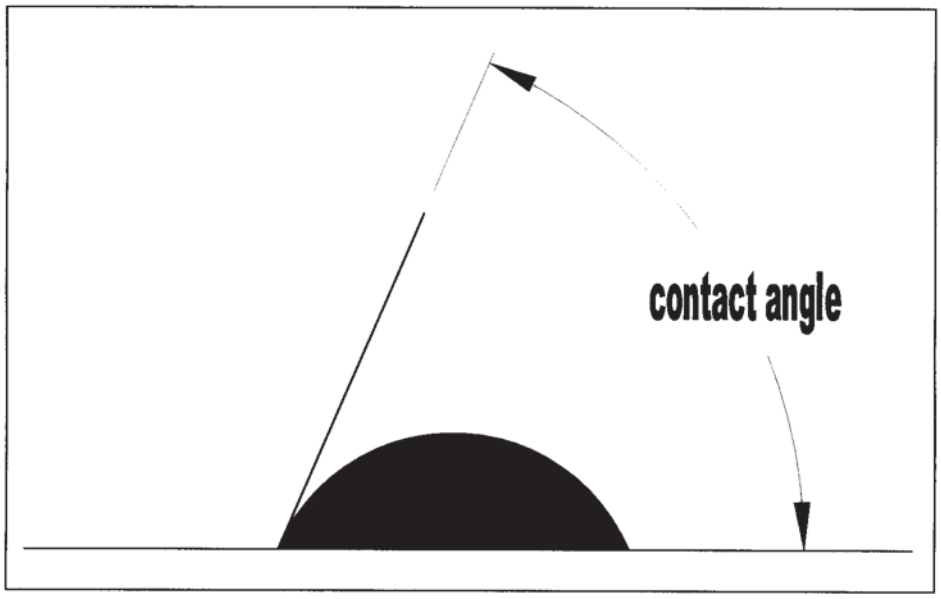

Figure 1: Contact Angle Definition

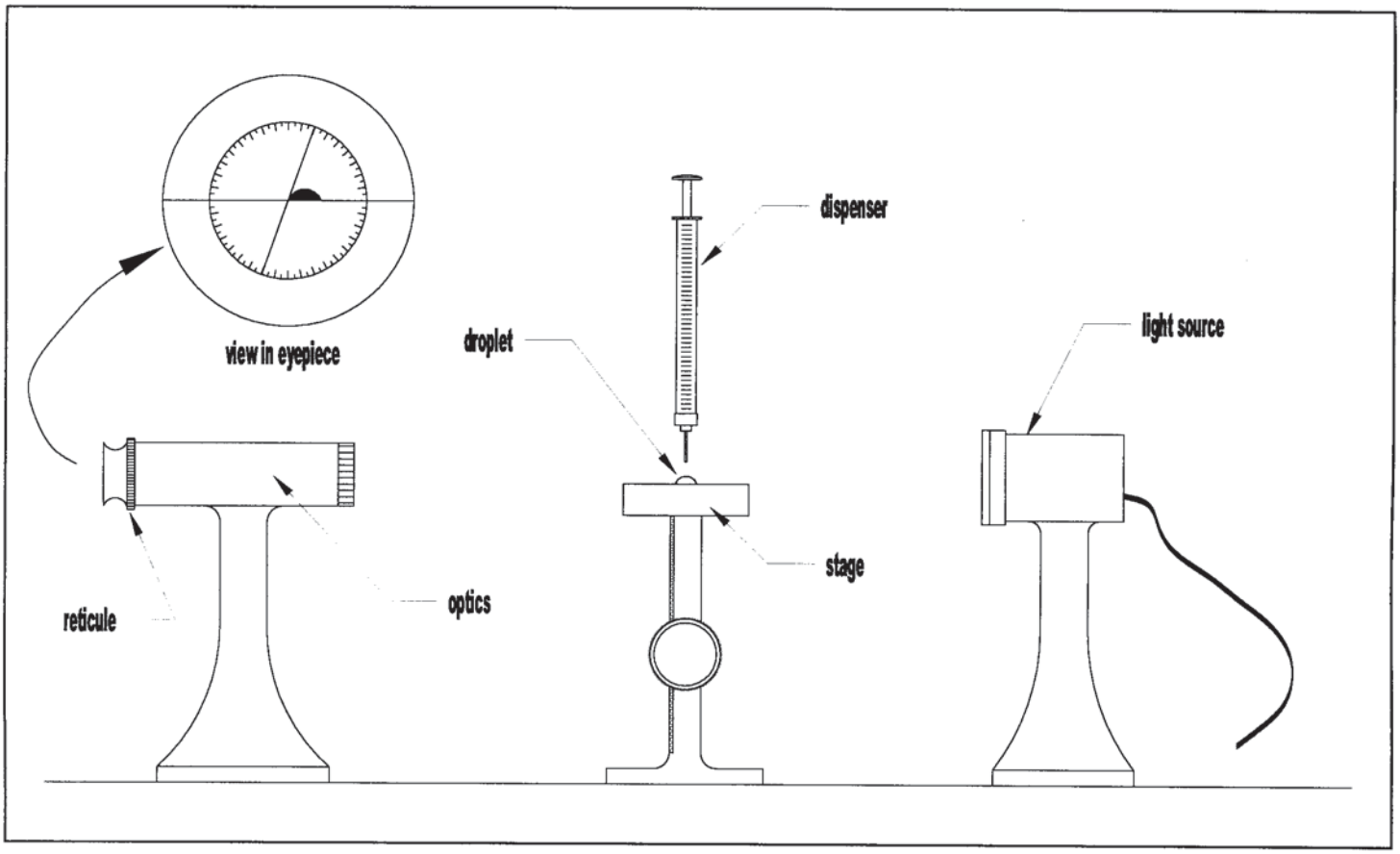

Figure 2: Optical Goniometer

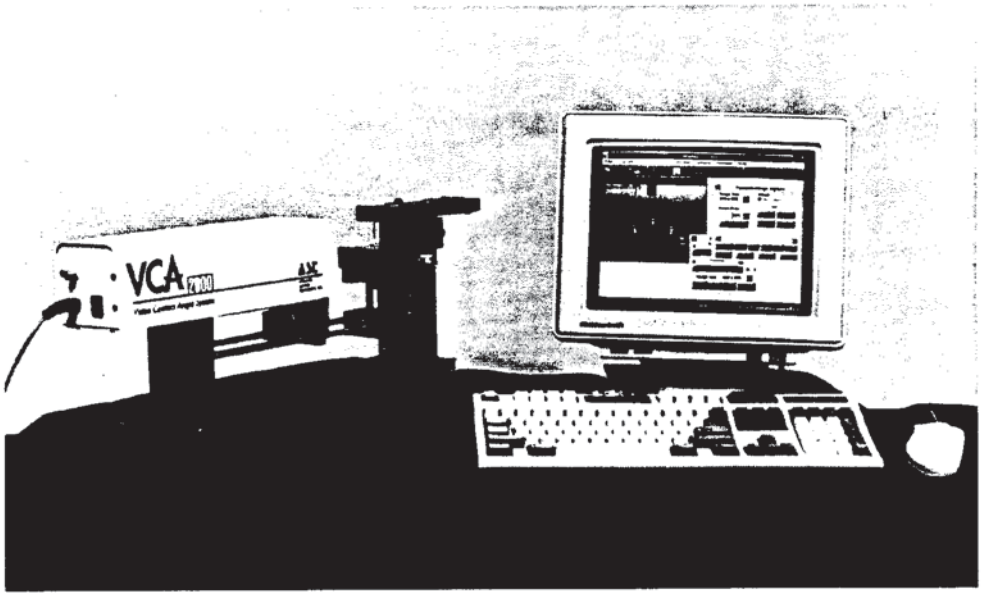

Figure 3: Computerized Video Contact Angle System