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Optimizing Plasma Treatments to ***RESOLVE*** Challenging Adhesion Issues

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The process of determining the ideal plasma treatment that will resolve a challenging bonding or adhesion issue is an exact science, often the domain of chemists and physicists.

Plasma is a state of matter, like a solid, liquid, or gas. When enough energy is added to a gas it becomes ionized into a plasma state. The collective properties of ions, electrons and radicals, can be controlled to change the properties of surfaces without affecting the bulk material. In this way, plasma is a powerful tool in solving surface preparation problems such as precision cleaning and decontamination, increasing surface wettability, and promoting adhesion. In addition, plasma can also be used to polymerize monomers onto surfaces through Plasma Enhanced Chemical Vapor Deposition (PECVD) to provide thin film coatings.

However, with multiple options and chemistries at their disposal, coming up with the ideal plasma "recipe" often involves having a precise understanding of the surface free energy. Unfortunately, many of the traditional devices used to measure surface energy are limited in the extent of information provided. In addition, many fail to measure the polar and dispersive components of surfaces that otherwise have the same surface energy.

This is critically important, says Dr. Michael Barden, Head of Research & Development at PVA TePla America, because different types of adhesives will form a superior bond with a surface that is polar versus dispersive. Organic adhesives, for example, often bond better to dispersive surfaces, whereas two-component epoxies, carbonates or ureas work better with a polar surface.

"When all these factors can be more precisely measured, plasma experts are able to dial in the best treatment solutions faster, with more consistent results," says Barden.

Dyne Tests

Plasma treatments are often utilized to create a high surface energy to increase the "wettability" of surfaces and assist the spreading of chemical adhesives. Surface energy is defined as the sum of all intermolecular forces that are on the surface of a material, the degree of attraction or repulsion force a material surface exerts on another material. When a substrate has a high surface energy, it tends to attract. For this reason, adhesives and other liquids often spread more easily across the surface. This wettability promotes superior adhesion using chemical adhesives.

On the other hand, substrates that have a low surface energy – such as silicone or PTFE – are difficult to adhere to other materials without first altering the surface to increase the free energy. There are several plasma methods to increase surface energy, including physical and chemical plasmas along with PECVD coating surfaces. In addition, plasma can increase the surface area of bonding by nano-roughening a surface. Surfaces that are highly ordered, or very crystalline, tend to have very low surface energies. To disrupt that order, ionized plasma gas is utilized to bombard the surface. This creates a surface with a high dispersive effect, another way to say high wettability.

Another method of increasing surface energy is to create a polarizable group on the surface by utilizing chemical plasma. For example, O_2 plasma can be used to create surface hydroxyls, which allow liquids to spread through hydrogen bonding mechanisms. To measure this surface energy, dyne tests are often performed to derive a value in dynes/centimeter. A dyne is the amount of force required to produce an acceleration of 1 cm/sec^2 on a mass of 1g. The dyne level of a material is called its surface energy.

These tests come in many forms, but include dyne pens that come with a specific dyne level listed on them. When run across a solid surface, the liquid from the pen will spread, or wet-out, if the dyne level is lower than the material's surface energy; if the ink's dyne level is equal or higher, it tends to remain as droplets. Although economical and simple to perform, dyne pen and ink tests provide only an approximation of the surface energy.

"In about 70 percent of the projects we work on, dyne testing roughly correlates to the adhesion," says Barden. "However, it can only determine if bonding is working within a specific range."

Barden says customers regularly ask for dyne tests to determine the surface energy is, for example, "55 dyne/cm or better." However, deriving a quantitative number can be important if a plasma treatment works best at, say, precisely 60 dyne/cm.

"By more completely measuring the surface free energy, we can come up with a measure of the quantitative change that occurs before and after plasma treatment," says Barden. "This allows the customer to correlate those measurements with their actual bonding experiments to ensure the best possible bond is being achieved."

To do this, designers and manufacturers of plasma systems often turn to the most advanced drop shape analyzers from global leaders such as KRÜSS.

Drop Shape Analyzers

Because surface energy of a solid cannot be directly measured, surface energy values must be calculated from a set of liquid/solid contact angles, developed by bringing various liquids in contact with the solid. Drop shape analyzers measure these contact angles visually. A droplet is deposited by a syringe that is positioned above the sample surface, and a high-resolution camera captures the image from the profile or side view. The image is then measured using image analysis software. Most of these systems are software-controlled to enable direct determination of the precise contact angle and surface free energy, measured in millinewton per meter (mN/m). This value has a direct 1:1 relationship to dyne/cm.

In addition, the KRÜSS equipment can simulate surface treatment process conditions using precision temperature-control methods and other accessories. This enables wetting processes to be optimized in relation to the overall process. For example, contact angle measurements can be carried out at accurately controlled temperatures between -30 and +400°C or at exactly set humidity.

According to Barden, one of the most important benefits of using advanced drop shape analyzers is the capability to

The KRÜSS system can tell an engineer if the assorted polymers they want to bond are oleophobic as well as hydrophobic.

In addition to designing and manufacturing plasma systems, PVA TePla also serves as a contract manufacturer with sophisticated in-house equipment like this KRÜSS Mobile Surface Analyzer used to process materials and conduct a full range of experiments.

evaluate the polar and dispersive components of surface free energy. Although water is often used for contact angle measurement, it is not sufficient for determining if the surface is polar or dispersive. Instead, multiple types of known liquids must be used, the data fed into complex mathematical equations, analyzed and then graphed.

This type of approach can also be used to determine if a surface is "oleophobic," or repels oils. In applications such as fluorinated coatings that ideally need to repel water and other organic materials, drop shape analyzers play a key role. When an engineer wants to bond assorted polymers, they'll often want to know if they are oleophobic as well as hydrophobic (repels water), and the KRÜSS system can tell them that," explains Barden.

Armed with this type of quantitative and qualitative information, plasma experts can then alter surfaces by altering the plasma treatment applied. Physical plasma like Helium, for example, would create a dispersive surface that allows liquid to spread broadly; oxygen plasma along with a hydroxyl or carboxyl coating could be used to create a polar surface that would alter the spreading mechanism.

Leveraging Surface Analysis

PVA TePla makes the use of this advanced technology available as a service that is supported by their in-house team of materials scientists and engineers. With this type of system users can dial-in precise equations that provide the best options for successful surface treatments.

In addition to designing and manufacturing plasma systems, the company also serves as a contract manufacturer and therefore has the in-house equipment to process materials and conduct a full range of experiments. This provides customers an opportunity to brainstorm with their technical team and run experiments together. According to Barden, these technical customer/supplier meetings often produce the best experimental matrices and ideas.

This article was written by Jeff Elliott, Technical Writer, PVA TePla America (Corona, CA). For more information, visit ***http://info.hotims.com/69513-598.***