

PLASMA PROCESSING FOR OPTIMAL MEDICAL DEVICE MANUFACTURING

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ABSTRACT

Reliability and reproducibility are crucial in the manufacture of medical products. Non-compatible materials (e.g., plastic/metal, mix-matched polymers) used in the manufacture of medical devices pose difficulties in the assembly process. Gas plasma surface treatment modifies the surface energies, allowing designers and manufacturers to maintain their desired bulk properties with few compromises. The use of plasma technology removes contaminants at the nanometer level, allows micro-level etching, promotes cross-linking, and activates and changes surfaces from hydrophobic to hydrophilic.

Plasma is the only successful method for treating the surface of chemical-resistant materials. Plasma cleaning removes organic materials that can prohibit desired bond strengths. In addition, plasma treatment can add functional groups to the surface, changing the surface chemistries for improved lubricity and surface adhesion. The following paper will explore the plasma processes used for surface modification of the various material types used in the manufacture of medical devices.

Key words: gas plasma, plasma treatment, hydrophilic, hydrophobic

BACKGROUND

One of the most prevalent concerns when bonding materials is how to get a strong bond between mismatched substrate surfaces. Generally, when bonding like materials, there are adhesives available that will strongly bond the two surfaces at the interface of the substrates. However, in many manufacturing scenarios the need to bond different materials with very dissimilar physical and chemical properties frequently arises. For example, a reasonably rigid material such as polycarbonate (PC) may need to be bonded to a more elastic polyethylene (PE) material. Since the chemical makeup of these materials is quite different, they will tend to bond differently to various adhesives. A certain adhesive may bond well to the PC, but not well – if at all – to the PE, and vice versa. There are methods to chemically treat the surface of one or both of the materials using primers, or other mechanical means, such as sanding, to get a strong adhesive bond. While many of these chemical methods work quite well, they require the use of materials that may

expose the manufacturer to dangerous or toxic matter. Such materials may also require expensive or time-consuming disposal methods. Most importantly, many toxic or dangerous materials cannot be used in the manufacture of medical devices that are implanted in the human body.

This paper will examine the use of low temperature-gas plasma to modify the surfaces of several plastics; describing how plasma can be used to make strong adhesive bonds between mismatched polymers without changing the bulk properties of either polymer.

METHODOLOGY

Sample Preparation

Tecothane[®] 75, ultra-high molecular weight polyethylene (UHMWPE), cast polypropylene (PP), polyimide (PI), and high-density polyethylene (HDPE) materials were chosen for these trials. These materials are a good representation of the common polymer varieties found in medical device manufacturing. Their material characteristics and bondability with common adhesives are well known.

The UHMWPE, PP, and PI materials were cut into 1.5 x 0.5-inch (38.1 x 12.7 mm) blocks from 0.5 inch-thick (12.7 mm) stock. Once the blocks were cut from the bulk and sanded to shape, they were briefly washed with isopropanol to remove possible bulk contaminants, such as finger oils, cutting oils and dust introduced to the bonding surface from the cutting and forming processes.

The Tecothane material was cut into bars that measured 3.0 x 0.5 inches, ¼-inch thick (76 x 12.7 x 6.3 mm). The HDPE material was purchased in sheet form: 0.03-inch (0.76 mm) thick and cut into strips 3-in. x 0.63-inches (76 x 16 mm). The block and bar stock were marked one inch from the ends so the HDPE sheet stock could be bonded at the same point on all blocks.

The adhesives used to bond the different polymeric materials to the HDPE strips were extra-thick cyanoacrylate (CA), RTV, and 12-minute, two-part epoxy. These adhesives, like the polymers selected, represent bonding agents commonly used in the medical device industry and their adhesion characteristics have been well studied.

Plasma Treatment

Two types of plasma technologies were used to treat the samples. The first was a traditional, vacuum batch system. An experiment matrix was created to explore the time and power spaces of the process operating window (see Table 1). Argon was the process gas of choice because it physically cleans and activates the surface through ion bombardment and molecular scission. The operating pressure in the system was set at 220 milliTorr.

Variable	High Value	Low Value
Time	5 minutes	1 minute
Power	500 W	100 W

Table 1
Experimental Matrix

The second type of plasma technology utilized was an atmospheric plasma wand system using helium and oxygen. The system parameters were set at a comparatively standard setting used for most work on plastic substrates (see Table 2). Plasma exposure under the wand was 10 seconds at a height of 0.25 inches (6 mm) over the substrate. The wand temperature was set to 100 °C and allowed to warm up prior to any treatment.

Helium Flow	40 SLM
Oxygen Flow	500 sccm
Power	100 W

Table 2
Atmospheric Plasma Conditions

The atmospheric system uses a wand through which the process gases flow. The process gases are excited with a radio-frequency field and flow out of the wand through the perforated tip to the substrate.

In both systems, the various polymer blocks, as well as the HDPE strip to be attached to blocks, were treated under exactly the same conditions.

Sample Assembly

A control group of plastic blocks and HDPE strips was not treated with plasma. Each type of polymer block was given a thin coating of each adhesive, the HDPE sheet stock was then placed on the adhesive, and a 0.5 oz (16.5 gm) weight was placed on the HDPE to hold the assembly together until the adhesive cured. After the control group was processed, the procedure was repeated after each plasma treatment step. Samples were assembled immediately following plasma. After the adhesives had cured at least 24 hours, the samples were sent to the Nordson Corporation's Corporate Materials Laboratory in Amherst, Ohio for testing.

Sample Testing

Testing was performed on a pull testing instrument capable of both lap shear and T-peel tests. The pull speed for both test types was set at 0.500 inches/minute and the data was

recorded. (Note: the system and its load cells are calibrated annually.)

Some of the control group samples came apart while being placed in the instrument chuck prior to actual testing. These samples were given a nominal value of 0 lbs.

NOTE: The water droplet contact angle measurement is a good means by which to evaluate surface wettability. However, this measurement was not taken because good contact angle measurement results do not always translate to good adhesion results as the correct functional groups for a given adhesive are not always present on the surface.

RESULTS

The Tecothane material used in these trials is especially challenging. Like the other materials, the surface has to be activated for optimal adhesion performance. Unlike the others, the Tecothane is molded and contains a mold-release compound that resides on the surface and is also contained within the bulk of the material. The mold-release compound must be removed prior to surface activation. Once the mold-release compound has been removed and the Tecothane surface activated, surface cross-linking must be done to create a layer that will help prevent the mold-release compound from blooming back to the surface. Cross-linking is accomplished by ionic bombardment of the surface. The ions scission the surface molecules and recombine with other molecules also scissioned to form a barrier-like layer.

Chart 1 shows the lap-shear and T-peel results for the control group, atmospheric plasma-treated group, and the optimal vacuum-plasma group using cyanoacrylate for the adhesive.

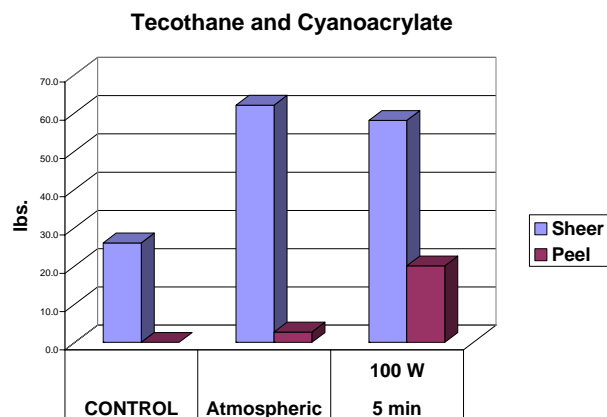


Chart 1

Chart 1 indicates that the control group had moderate lap-shear strengths, but was lacking completely in T-peel strength. Treatment with the atmospheric plasma system significantly improved the lap-shear strength. The atmospheric system showed only minimal gains in T-peel tests. Treatment in the vacuum batch system exhibited a somewhat better increase in T-peel strength while

maintaining lap-shear strength. Reasonable T-peel strength is somewhat more difficult to achieve than lap-strength using cyanoacrylate adhesive on Tecothane material. It is interesting to note that the RF power settings for both the atmospheric plasma system and the batch system were the same. Treatment times were longer in the vacuum batch system.

Chart 2 also compares results between the control group, atmospheric and vacuum batch systems using a two part-epoxy as the adhesive.

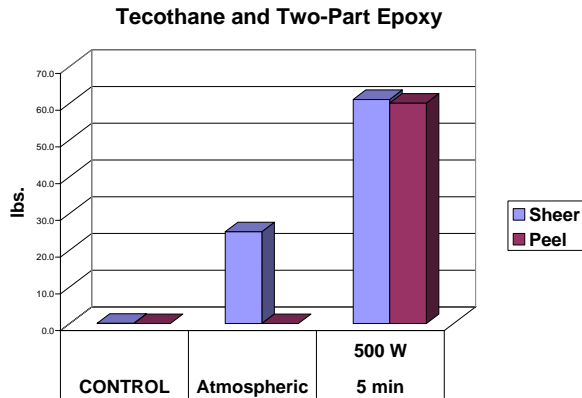


Chart 2

The two-part epoxy did not perform well in the control group or in the atmospheric plasma group. There was only a small increase in lap-shear strength, and the T-peel strength was poor. However, there was a significant increase to both test results with the batch system.

There is one other consideration when examining this data: over-treatment can become a significant issue, especially when trying to remove a surface contaminant prior to surface activation. Running at elevated RF power levels for extended time periods increases the propensity for over-treatment. While over-treatment was not an issue in this study, the tendency to over-treat should always be considered as the response “sweet spot” can easily be missed.

Chart 3 shows the results for lap-shear data for polypropylene material using all three adhesives. This is very significant because there was no noteworthy increase in T-peel strength using either the atmospheric system or batch system over the control group. Good T-peel results are much more difficult to attain than good lap-shear results.

The atmospheric system produced a four-fold improvement over the control group. The two-part epoxy again showed the most significant improvement. None of the batch system results were as good as the atmospheric results. Perhaps, this is due to over-treatment and the need to decrease process time and RF power levels in both cases. Or, the atmospheric plasma system may be the better

method to treat polypropylene material for increased lap shear strength.

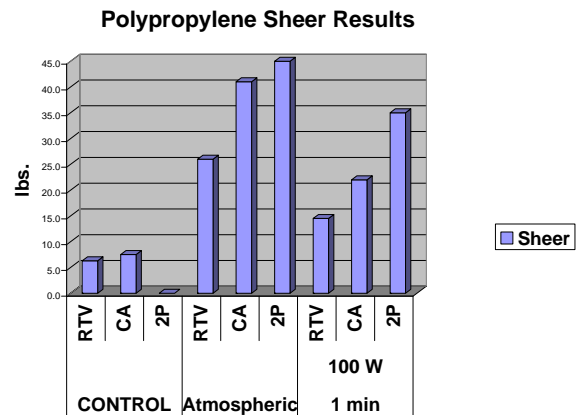


Chart 3

Chart 4 represents the data for bonding HDPE to UHMWPE. Again, the cyanoacrylate adhesive provided the best results for these trials.

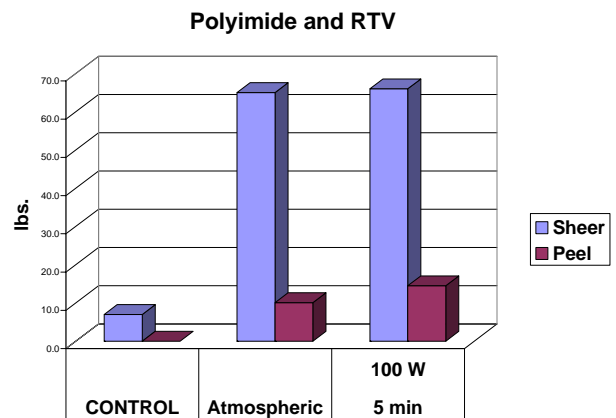


Chart 4

Again, Chart 4 shows that plasma treatment of any kind for a given adhesive produces increased bonding results. It also shows once again that improving T-peel strength is much more difficult than improving lap-shear strength, especially when trying to improve lap-shear strength on the same substrate with a given adhesive.

CONCLUSION

As seen with the data, bonding mismatched plastics can be challenging for engineers and technicians in medical device manufacturing, even when the proper adhesives are utilized for maximum adhesion results. Chemically resistant polymers that are predisposed to poor adhesion cannot be treated to improve adhesion with substances that are harmful to the human body. Low temperature gas plasma can be a simple, safe, inexpensive, and powerful alternative to prepare materials for bonding for the manufacture of medical devices.

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BIOGRAPHY

Les Wood has over 12 years of experience in the surface modification and gas plasma treatment fields. He has worked extensively with various types of non-metallic coatings such as PECVD and sputtering, and is familiar with vacuum metalization processes including CVD, ion beam and sputtering. Mr. Wood is named inventor or co-inventor on several patents pertaining to surface modification and PECVD coatings, and has published articles on those topics. He holds a BS degree in Physics and Mathematics from Lincoln University in Missouri, and an MS in Physics from the University of Idaho. Mr. Wood is currently a senior applications engineer with March Plasma Systems.
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