EBBLE GROUP
MANUFACTURING SURFACE TREATMENT TEST & RESEARCH
tantec

# Polypropylene (PP) Bonding Using Polyurethane Adhesive SikaForce 803 With Different Atmospheric Plasma Treatment Settings

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# Abstract

Bonding polypropylene using a surface treatment, often plasma or corona treatment, is common through various industries due to the low bonding performance of polypropylene.

Although difficult to bond, it's chemical resistance, temperature use properties and cost to manufacture make it an exceptional plastic for many applications.

In applications where bonding is critical, such as safety critical print on a medical device or product quality that needs to last product lifetime such as leather bonding to automotive trim, Plasma treatment is often used to improve adhesion and longevity, as well as providing a consistent bonding surface to give confidence in the products lifetime ability.

Treating a polymer in this way gives a different chemistry on the surface that's key to bonding, but this change in chemistry isn't guaranteed to work with every adhesive, printing ink and coating as the chemistry of the substrate, bonding agent and where applicable, the other bonding surface need to be aligned. For example, using a printing ink which works by absorbing in to a porous surface such as paper will still provide the wrong results on a solid surface. Adhesion may improve in this case, but the final product will still not be correct.

To test the ability of bonding on PP, we chose a single PP blend, bonded with a single batch of SikaForce 803 adhesive and changed the level of treatment using a Tantec PlasmaTEC-X atmospheric plasma system.

# Aims and Assumptions

The goal of this experiment is to determine the usefulness of plasma treatment on polypropylene and the compatibility with SikaForce 803 Polyurethane adhesive.

Multiple tests will be run to reduce bias from any results, but it is assumed that adhesive from the same batch, lap shear samples from the same board and plasma treatment from the same system will give consistent and repeatable results.

All tests should be performed at standard lab conditions.

# Method, Equipment and Experiment Parameters

To determine this, we will follow the method below:

1. Pre cut lap shear samples, 25mm x 100mm x 3mm in White PP
2. Measure surface energy of a selection of the received samples
3. Treat at determined treatment levels
4. Measure surface energy of treated parts
5. Bond three lap shears sets together for each parameter with a 20mm over lap using SikaForce adhesive, bond layer should be 1mm thick.
    a. Bond three control untreated lap shear sets together
6. Allow at least 24 hours cure time for product
7. Pull using tensile test system

Measuring surface energy of the material gives us an understanding of what is happening on the surface, on it's own, it is not enough to say bonding will be better, or good enough to meet high performance levels. However, when used as a gauge alongside tensile testing, surface energy can be correlated to adhesion strength and a pass mark can be determined. This is often sensible because a surface energy reading can be taken in seconds by an unskilled operator, where as a tensile test is usually bonded and run in a lab over 24 hours after manufacturing has begun, which may miss important manufacturing issues.

Plasma treatment using a Tantec PlasmaTEC-X system is used for a wide variety of applications, and it's key parameters are speed of travel over the part and distance from the parts surface. Typical starting parameters for a system are 10mm from the surface an 300mm/s. In practice, systems may run in excess of 1,500mm/s and be only 6mm from the surface, or they could be running at 50mm/s and be 15mm from the surface.

The linear drive we use goes from one side to the other and back again, so this experiment consists of two passes as the head moves along.

Taking an experimental spread of 250mm/s to 1,000mm/s, the following table of samples seems sensible to get initial results:

| PP - AP Treatment, 10mm Height, 12.5mm overlap | | | | |
|---|---|---|---|---|
| | Lap Shear 1 - MPa | Lap Shear 2 - MPa | Lap Shear 3 - MPa | Lap Shear Average |
| Untreated | | | | |
| 1000mm/s x2 | | | | |
| 500mm/s x2 | | | | |
| 250mm/s x2 | | | | |

Units used for measurements:

- Surface energy: mN/m (millinewton per meter)
  - This is the same as dynes/cm and $mJ/m^2$
- Adhesion strength: MPa, mega pascals
  - This takes into account the surface area of a surface, it can also be expressed in newtons.

The equipment that will be used will be:

- Surface energy
  - Kruss DSA25 Contact Angle Meter
    - Measuring surface energy using contact angle is generally considered the most accurate way of measuring and the most repeatable. It also allows for the breakdown of the total surface energy in to the dispersive and polar components, with the polar component generally considered the important part for bonding.
- Tensile Testing
  - Tinius Olsen ST5
    - A tensile test unit with enough load to pull a lap shear to failure.
- Dimensions
  - Generic electronic callipers used for thickness and length measurements.

# Results and Discussion

Initial surface energy results gave interesting results:

| | Polar SFE mN/m | Dispersive SFE mN/m | **Total SFE mN/m** |
|---|---|---|---|
| PP As Received | 0.05 | 31.97 | **32.02** |
| 1000mm/s x2 | 7.03 | 34.17 | **41.21** |
| 500mm/s x2 | 8.21 | 36.01 | **44.22** |
| 250mm/s x2 | 11.04 | 35.37 | **46.41** |

Depending on the manufacturer, material blend, and also it's additive and colour content, the surface energy can vary significantly. My expectation was to see the total surface energy starting around 30mN/m and going to around 72mN/m, but even with high levels of treatment (low speed), the maximum treatment was only 46mN/m.

While the surface energy figures here didn't look like the treatment had done much, it is worth noting that the polar component and the total did increase. A rule of thumb is normally that when you get to around 10m/N/m of polar, you will get good adhesion.

Tensile testing then gave the following results:

| PP - AP Treatment, 10mm Height, 12.5mm overlap | | | | | |
|---|---|---|---|---|---|
| | Lap Shear 1 - MPa | Lap Shear 2 - MPa | Lap Shear 3 - MPa | Lap Shear Average | Notes |
| Untreated | 0.451 | 0.543 | 0.421 | 0.472 | All adhesive failure |
| 1000mm/s x2 | 3.946 | 3.545 | 3.177 | 3.556 | All cohesive failure |
| 500mm/s x2 | 5.003 | 4.188 | 4.266 | 4.486 | 1x Cohesive failure, 2x substrate failure |
| 250mm/s x2 | 4.211 | 4.552 | 4.494 | 4.419 | 1x Cohesive failure, 2x substrate failure |

| Specimen ID | Width mm | Overlap Length mm | Area mm² | Shear Force N | Shear Stress MPa |
|---|---|---|---|---|---|
| 1 - Untreated | 25.0 | 19.0 | 475 | 214 | 0.451 |
| TANTEC PP T4 | 25.0 | 19.0 | 475 | 1870 | 3.95 |
| TANTEC PP T7 | 25.0 | 18.0 | 450 | 2250 | 5.00 |
| TANTEC PP T10 | 25.0 | 18.0 | 450 | 1900 | 4.21 |
| TANTEC PP T12 | 25.0 | 19.0 | 475 | 2160 | 4.55 |
| TANTEC PP T11 | 25.0 | 19.0 | 475 | 2110 | 4.44 |
| 2 - Untreated | 25.0 | 19.0 | 475 | 258 | 0.543 |
| 3 - Untreated | 25.0 | 19.0 | 475 | 200 | 0.421 |
| TANTEC PP T5 | 25.0 | 18.0 | 450 | 1600 | 3.55 |
| TANTEC PP T6 | 25.0 | 18.0 | 450 | 1430 | 3.18 |
| TANTEC PP T8 | 25.0 | 20.0 | 500 | 2090 | 4.19 |
| TANTEC PP T9 | 25.0 | 20.0 | 500 | 2130 | 4.27 |
| Average | | | | 1520 | 3.23 |
| SD | | | | 816 | 1.73 |

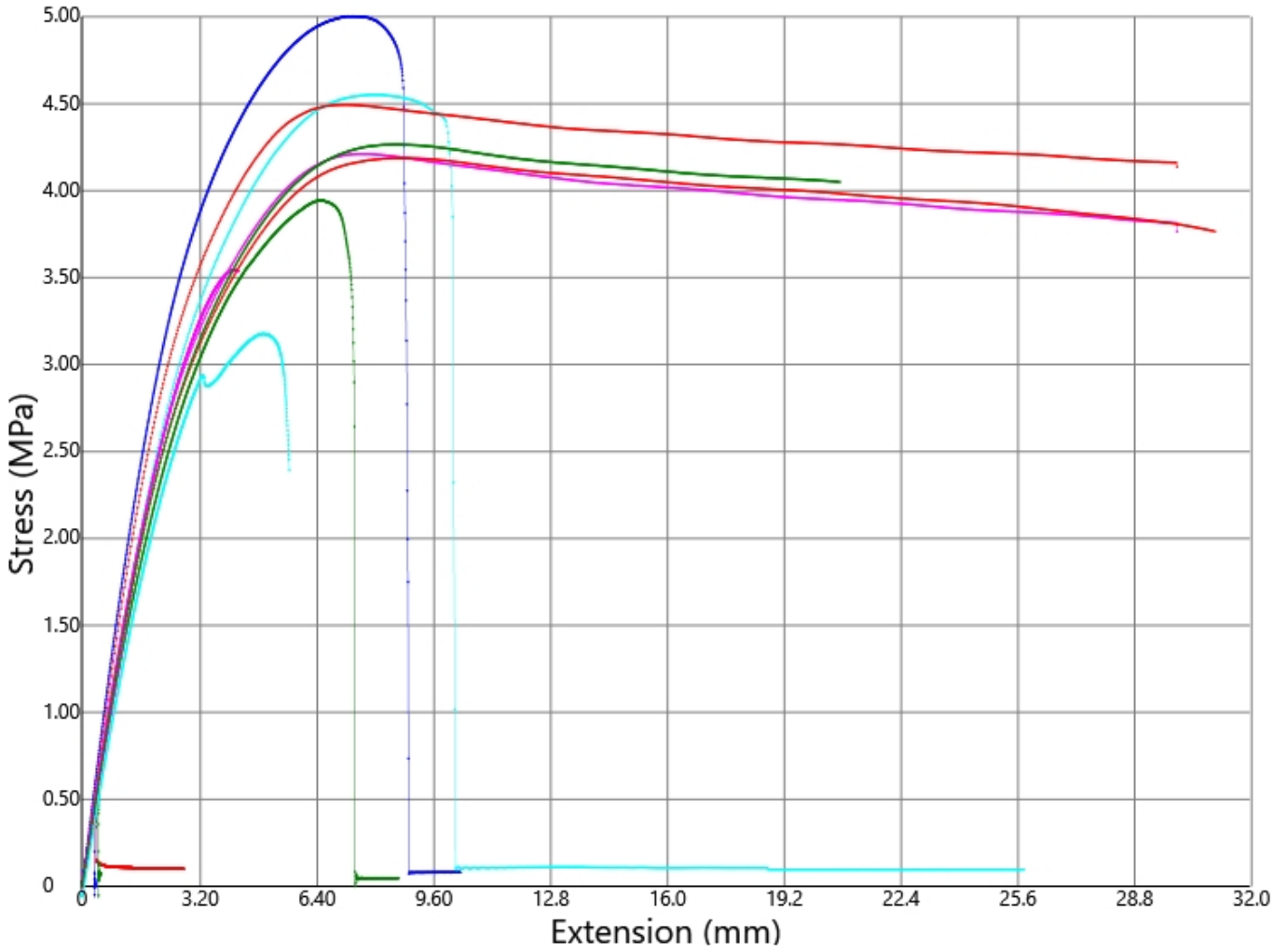
Samples marked untreated have no treatment

Samples T4 – T6 1,000mm/s x2

Samples T7 – T9 500mm/s x2

Samples T10 – T12 250mm/s x2

Adhesion strength and results were really interesting, showing very low adhesive failure results for the untreated control, where the adhesive left behind could be easily picked off in one sheet, through to complete substrate failure of the PP substrate, without any movement of the adhesive bond. Cohesive failure, leaving adhesive behind on both surfaces was also seen.

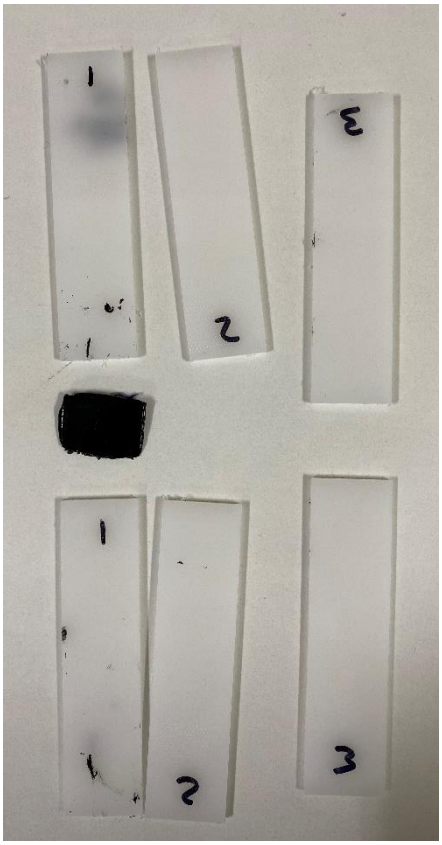


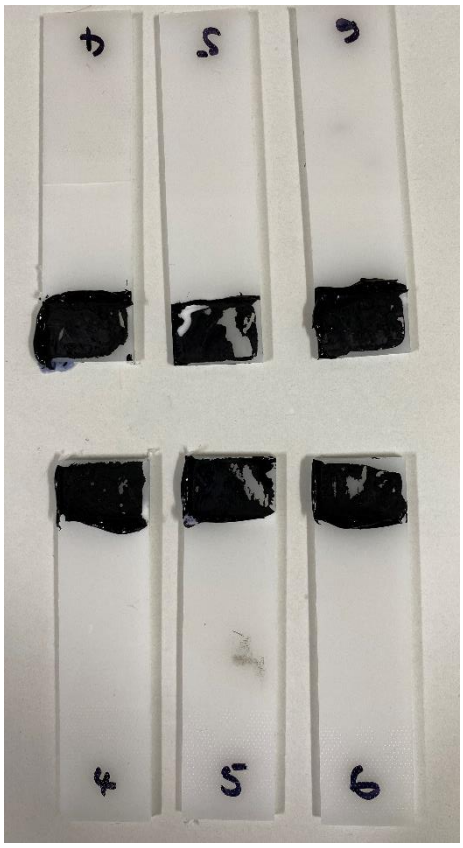

Reviewing the graph data, all untreated samples failed quickly with low extension and under 0.5MPa so are hard to see in the bottom left of the graph.

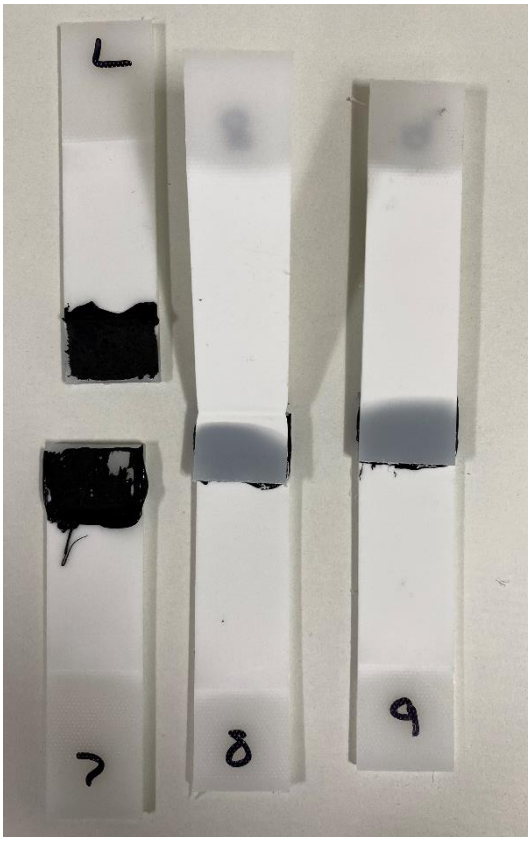
Where the adhesive failed, a shard failure was seen and an extension of less than 10mm was observed.

Where the material failed, the material began to stretch and extended well beyond 10mm. The programme was set to pull a sample for a predetermined amount, which was not enough to pull the samples apart. Samples which were fully destroyed we're then pulled further after the experiment had come to a finish. All of the samples which go on at high load for greater than 10mm were these material failure parts.

The range of samples after pulling looked like this:





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Specific failure method sample photos below:

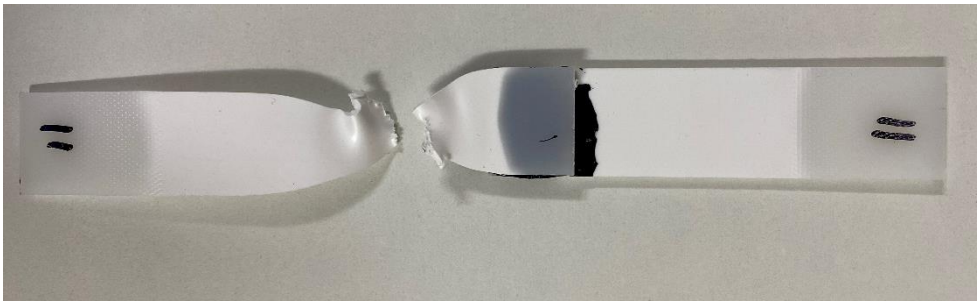

*Figure 1 Treatment at 250mm/s x 2, the material stretched and snapped. Adhesive bond showed no sign of breaking or fatigue.*

*Similar results seen on 250mm/s and 500mm/s samples.*

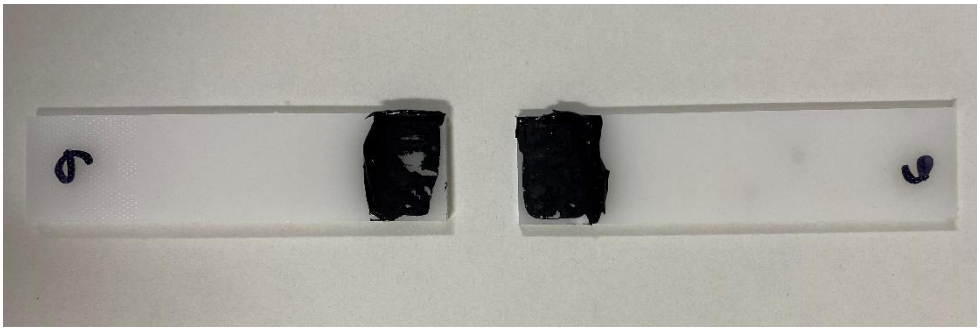

*Figure 2 Treatment at 1,000mm/s x 2, the material showed some signs of stretch, with others in this group having material failure.*

*Cohesive failure seen on 1/3 of the 1,000mm/s and 500mm/s samples.*

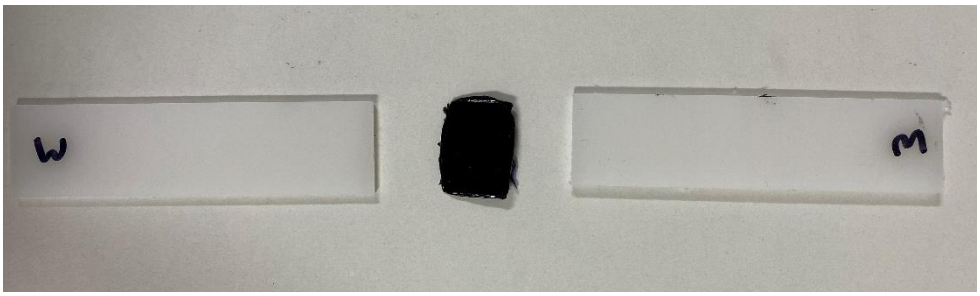

*Figure 3 Untreated samples showed full adhesive to substrate failure.*

Maximum forces greater than 4MPa seemed to show that the limit of the PP material and the limit of the adhesive we're similarly matched, showing the high levels of treatment of the slower moving plasma to have a mix of cohesive and material failure.

While either would be good to see in a real life application, for the purpose of experimental design it would have been sensible to have used a thicker and stronger material to aim for cohesive failure in every attempt.

The experiment was very encouraging and gave good results with all levels of plasma treatment. While 250mm/s isn't especially slow, the treatment that takes place comes out at around 200ºC, which can cause heat damage and in theory, over treatment. Even so, at these speeds, there were no visible witnesses from heat or over treatment damage, and the performance data is as good for 250mm/s as it is for 500mm/s.

The adhesive itself performed well throughout the testing, giving a pull off force in newtons over 2,000N.

# Conclusion and Further Work

Treatment of any kind made a significant difference in adhesion strength, going from very low and now adhesion to full substrate failure.

Interesting, surface energy didn't show to same effect, adding proof to the notion that high surface energy doesn't necessarily mean greater adhesion, in this case, a quality control of only 40mN/m would be sensible. It would be a good further test to use the same PP material and try different variations of testing, such as test inks, force tensiometry and contact angle with different liquids.

Treatment window seems to be fairly wide, allowing for a wide variety of speed without causing variation in performance. It would be good to investigate this further with a wider range of parameters, taking the second parameter of nozzle height in to account too, which was fixed for this test at 10mm from the surface.

The material chosen was polypropylene due to the material being the most common requiring plasma treatment, however, it would be good to run the same test regime using other common materials such as PE, HDPE, PA6, PA66 and PA12.

We ran the treatment with an offset at 10mm to give a good coverage. This seemed to work really well and no banding was seen. It would be good to see a larger offset and determine the effect on adhesion strength to determine the requirement of multi nozzle systems for flat surfaces.

The failure methods seen that seemed to be similar values – cohesive and material failure, both gave failure within the material in some way. Where cohesive failure occurred, the material also stretched and became a more opaque white compared to how it began. Material that failed completely also showed this, indicating stress within the polymer chains. A thicker material variant should overcome this and give a true reflection of the adhesion strength and consistency.

The adhesive worked really well with plasma treatment, while giving exceptional results on the trial too. Altering the adhesive to try other Polyurethanes as well as different chemistries would also be useful.