Advantages of Inherent Semi-Structural Pressure Sensitive Adhesion in Challenging Environments

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Abstract

While numerous high adhesion solution acrylic pressure sensitive adhesives (PSAs) exist in the marketplace, the vast majority of these products cannot provide substantial shear holding power, especially at elevated temperatures, in the range of 70 °C to 150 °C. The majority of acrylic PSAs that do provide elevated temperature shear performance, in conjunction with high adhesion, rely on the use of a secondary crosslinker that requires a separate activation step via heat, UV, EB, or other stimuli. The ability to achieve such performance using a solution acrylic PSA that relies only on the aluminum salt crosslinker results in a more simplified and robust tape manufacturing and application process and is likely to lead to a more reproducible product performance in the field. Herein, we present a new solution acrylic (PSA), which exhibits high peel adhesion, along with exceptional cohesive strength both at ambient and elevated temperatures. Unlike adhesives that utilize a secondary crosslinker, no further chemistry is necessary for this PSA to achieve its ultimate cohesive performance.

Introduction

Many high-performance pressure sensitive tapes require adhesives that can achieve both high bond strength quickly and have permanent bonding features. While structural adhesives are capable of delivering exceptionally high bond strength, the time to achieve it depends on the duration to cure, which needs to be balanced with the pot-life of the adhesives. It might take several hours for a structural adhesive to reach ultimate bond strength. PSAs on the other hand have the advantage of providing a high bond strength rapidly. While the ultimate bond strength of high shear PSAs is often inferior compared to structural adhesives, the advantages inherent to PSAs have resulted in tape technology growing into several transportation, building, and construction applications that have been traditionally served by structural adhesives.

In addition to polymer molecular weight, cohesion in solution acrylic PSAs is primarily driven by the crosslinking chemistry and concentration of that the crosslinker. These crosslinkers are primarily classified by their impact on the shelf stability of the solution acrylic, resulting in one and two component systems.[1] One component solution acrylic adhesives are shelf stable and do not crosslink until either the removal of the inhibiting solvent(s) (metal chelate) or until the dried adhesive is exposed to sufficient heat to activate the primary crosslinking mechanism (melamine formaldehyde, blocked isocyanate, etc.). Two component solution PSAs, which are typically crosslinked using multifunctional isocyanates, undergo crosslinking as soon as the isocyanate is added to the acrylic polymer, which significantly limits storage stability. It is commonly accepted that covalent crosslink sites resulting from chemistries such as isocyanates, result in better elevated temperature resistance than the metal ligand bonds created in metal chelate crosslinking.[2,3]

Traditionally, primary crosslinking chemistries (one and two component) have been utilized in combination with elevated temperature activated secondary crosslinkers whenever significant temperature resistance was required in a solution acrylic PSA. Several high-strength PSAs have been reported in the past[4,5,6] wherein a typical PSA is converted to a structural adhesive by a secondary cure mechanism by incorporating thermal or radiation curable functionalities into the adhesives. In addition, high performance transfer tapes are available to the market that provide performance that can exceed the performance of solution PSAs that utilize secondary crosslinking chemistries. However, these adhesives are not available to the market in liquid form.

Through this work, we would like to demonstrate that there is still room to improve the combination of peel, shear and chemical resistance of conventionally crosslinked solvent acrylic PSAs. While some existing combinations of covalent primary and secondary crosslinking mechanisms can achieve significant elevated temperature performance, these chemistries are not as readily utilized in tape manufacturing as simple metal chelate crosslinking systems. The manufacturing challenges include issues before and after coating, including the limited pot life of two component systems,[7] as well as additional challenges that can impact product consistency. These additional challenges result from the high activation temperatures and/or times that are often needed to achieve full cure in many isocyanate,[8] as well as epoxy & carboxylic acid[9] crosslinked systems

Furthermore, we demonstrate the performance benefits of a recently developed Very High Performance Solvent Adhesive (VHPSA) in a variety of elevated temperature and other harsh environments. This performance spectrum will be compared to two well-established acrylic adhesive controls in the industry, namely a High Performance Solvent Adhesive (HPSA), available to the market as a solution acrylic PSA, as well as a High Performance Transfer Adhesive (HPTA), which is available to the market in transfer tape form. The VHPSA technology has been optimized to provide not only high peel adhesion, but also extraordinary cohesive strength. This enhanced cohesive strength provides superior shear behavior not only at ambient, but also and especially at elevated temperatures. The performance of these PSAs could further be enhanced by the use of the aforementioned secondary cure mechanism.

Most applications wherein high-performance adhesives are used also require adhesives to exhibit good resistance to harsh environments such as in marine, automotive, building exteriors or applications that utilize aggressive cleaning agents., We would like to also report that the VHPSA exhibits extraordinary resistance to a variety of solvents, both aqueous and organic solvents in addition to its high adhesive and cohesive performance. Based on this combination of performance enhancements, we strongly believe that this can enable new application areas, which were unreachable for PSAs previously.

Experimental methods

Materials

Acetone (HPLC grade) and isopropanol (ACS reagent grade) were purchased from VWR and toluene (technical grade) was purchased from Nexeo Solutions. Isooctane was obtained from Aldrich. Motor oil (10W30), transmission fluid, winter grade windshield washer fluid, engine coolant, salt and vinegar were purchased in a retail store. Stainless steel panels were purchased from ChemInstruments. Aluminum foil face-stock of 2 mil nominal thickness was purchased from

Metal Associates Inc. Aluminum (Al 6061) lap shear panels and cold rolled steel (CRS) substrates were purchased from Q-Panel and ACT Test Panels, respectively. The H-shaped aluminum bars were bought from Orange Aluminum (SKU OA5575-6M). These bars were then machined in-house to a size of 1” x 1”. The plastic panels used to conduct peel adhesion testing were obtained from American Plastics Distributors. The HPTA transfer tapes (2 mil and 5 mil adhesive thickness) were obtained from Uline. The VHPSA and HPSA solution acrylic PSA samples were polymerized and formulated at Ashland.

PSA-coated film preparation

Liquid acrylic PSA samples were coated onto Kraft Liner using a Baker Bar. The bar was set to the wet film thickness appropriate for producing the appropriate thickness, 2 or 5 mil dry PSA films. After air-drying the wet sample for 15 min the films were dried in a forced-air oven at 93 °C for 15 min. The films were then transferred onto 2 mil thick Aluminum foil by laminating at a 40 psi nip pressure.

Peel testing

Peel testing was carried out in accordance with PSTC-101.[11] Stainless steel panels were cleaned with acetone and stored under constant temperature and humidity for at least 1 hour prior to use. Plastic substrates used were cleaned with isopropanol and conditioned under constant temperature and humidity before use. 1” wide coated stock (2 mil dry PSA on 2 mil Aluminum foil) were bonded to the panels by rolling twice using an automated 4.5 lbs roller. After allowing the samples to dwell at various times the samples were peeled at 90 or 180° at a rate of 12 in/min. using an Instron® Model 4411 mechanical tester. Average peel values are reported in pounds per linear inch.

Static shear

The static shear testing was carried out in accordance with PSTC-107, Procedure A.[11] Stainless steel panels were cleaned using acetone and stored under constant temperature and humidity conditions for 1 hour prior to use. The coated stock was bonded to the panels with a ½” x ½” contact area and allowed to dwell for 15 min. The panels were then hung vertically (178°), and a 1 kg weight was attached to the film. The shear failure times were recorded in hours. No additional dwell time was utilized during elevated temperature testing. The weights were hung on the samples as soon as the samples were placed in the oven.

Shear Adhesion Failure Temperature (SAFT)[11]

Cold rolled steel (CRS) substrates were wiped with toluene followed by acetone and allowed to equilibrate under constant temperature and humidity for 1 hour. Following this equillibration, the 2 mil coated foil stock was bonded to the CRS surface to cover a 1 in^2 area. The bonded substrates were rolled with a manually controlled 10 lbs roller three times and allowed to equilibrate in a constant temperature and humidity room for 24 hours before testing. Samples were hung vertically in a forced air oven and a 1 kg load applied to the adhesive. The oven was heated at a constant rate of 1°C/min and the temperature at which the load falls was noted. Tests were conducted in triplicate and the average was reported as the SAFT value.

Lap shear testing

All plastic and composite test panels were wipe cleaned with isopropanol. Al 6061 panels were cleaned with acetone, while the CRS was cleaned with toluene followed by acetone. All the panels were then conditioned under constant humidity and temperature for 1 hour prior to use. Free films were bonded to the first surface to cover an area of 1 in^2 and the excess was trimmed with a knife. After removing the release liner the exposed adhesive layer was bonded to the second substrate surface and the bonded substrates were subjected to a pressure of 20 psi for 30 secs using a modified vise clamp (Figure 1, left). The samples were then continued to bond at ambient pressure for 72 hours under constant temperature and humidity. Lap shear testing was conducted using an Instron Model 5566 mechanical tester at a debonding rate of 0.4 in/min.

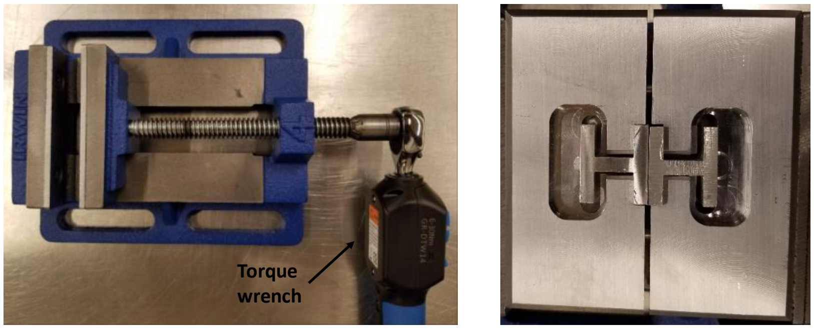

Figure 1. Aligning fixture for the "H-profile" tensile test substrates (*left*); aligning fixture mounted on a vise clamp (*right*).

Arcan fixture tensile testing

For the test samples a "H-profile" aluminum bar was cut to 1" x 1" square pieces. Each test surface was polished using a 1000 grit sandpaper to remove the thin layer of oxide and polish to a smooth surface. The substrates were then rinsed with acetone, followed by an acetone wipe until clean. Finally, the first surface was dried with Kimwipes® and bonded to the test adhesive transfer tape immediately. The excess adhesive was trimmed with a knife to give a test area of 1 in^2. After exposing the other transfer tape adhesive surface by removing the liner, the second test substrate was brought in contact with the adhesive using the aligning fixture shown in Figure 1 (right). The aligning fixture was then placed in the vise clamp described earlier and subjected to 20 psi pressure for 30 s and continued to bond in ambient pressure for another 72 hours.

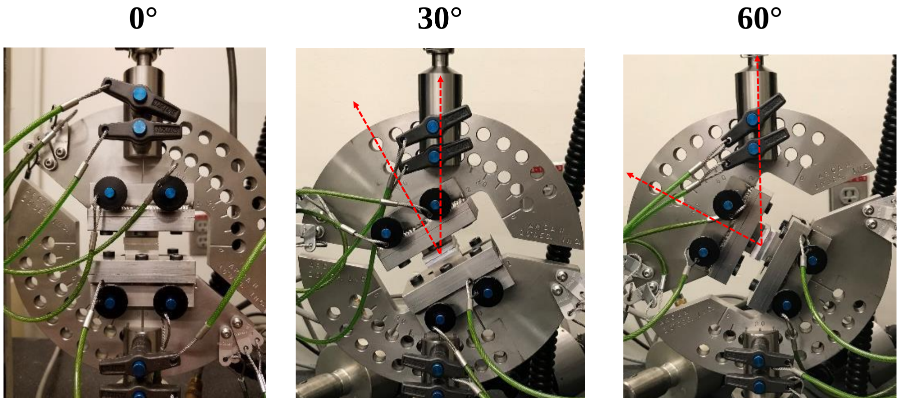

Figure 2. Arcan fixture used for the tensile testing at three different angles (0, 30 and 60°)

A modified Arcan fixture as shown in Figure 2 was used to perform the tensile testing. Hennage[10] reports the use of this fixture to measure tensile strength of PSAs. This fixture was fabricated in our facility as per basic design in the cited thesis, with slight modifications for ease of loading samples. The circular Arcan fixture has holes located precisely every 10° so that it can be rotated to run the debonding tests at 10° angle increments. Tensile testing was conducted using the Arcan fixture mounted in an Instron® Model 4411 mechanical tester.

Chemical resistance

One inch wide adhesive coated Aluminum foil face-stock was bonded to stainless steel panels (2” x 6”) using the same sample preparation methodology as described the peel testing section. The bonded samples were allowed to dwell in the constant humidity and temperature lab for 24 hours. The samples were then immersed in the various media for the required times. After removing from the immersion media, excess solvent was gently removed from the samples using Kimwipes® and the specimen were allowed to recover for 24 hours before measuring the peel adhesion. All the immersion experiments were carried out at ambient temperature, unless specified. For elevated temperature immersion experiments, the bottle containing the immersion media along with the samples were placed in a forced air convection oven at the specified temperature and storage times.

Dynamic Mechanical Analysis (DMA)

The DMA properties of the adhesive were measured on a Rheometrics ARES Rheometer in the dynamic shear mode using the 8.0 mm diameter disposable parallel plates. The PSA film was applied to the bottom parallel plate to give a thickness of approximately 1.8 mm. The plate gap was adjusted to a 1.5 mm gap. The normal stress in the specimen was allowed to equilibrate for 30 min. The sample was then kept at this compressive force while being cooled to a lower temperature of -40°C that allowed continual adhesion to the plates. The sample was then tested isothermally as a function of frequency between 0.4 and 100.0 rad/s with a measurement interval of 5 data points per decade. The temperature was increased by 5 or 10 °C, depending on the rate of change in the measured DMA properties. The strain was set at 0.1% at -40 °C. The strain was increased to 0.5% at 5 °C, 5.0% at 40 °C, and 15.0% at 80 °C. The sample was allowed to equilibrate for 3 min when the selected isothermal measurement temperature interval equaled 5 °C. Otherwise, the sample was allowed to equilibrate for 5 minutes at larger temperature measurement intervals. The sample was kept at the initial starting compressive force until the test temperature exceeded 25°C. Above this temperature, the compression was allowed to decrease due to the softening of the PSA.

Results and Discussion

In the following sections, the performance of Very High Performance Solvent Adhesive (VHPSA) will be compared to the High Performance Solvent Adhesive (HPSA) and High Performance Transfer Adhesive (HPTA) to demonstrate that it is possible to simplify the manufacture of high-performance acrylic pressure sensitive tape by use of a coater ready solvent based PSA whose performance significantly exceeds that of a currently available high-performance solution acrylic PSA. A significant advantage of using a coater ready solvent formulation is the ability to vary the thickness or other construction parameters such as face-stock and/or liner types to suit application needs.

Peel performance

Figure 3 shows a comparison of the peel performance of the three adhesives, namely VHPSA, HPSA and HPTA on stainless steel (SS), plastic, glass and aluminum panels. For the peel data shown in Figure 3 (right) the samples were bonded to the substrates and further allowed to dwell under constant temperature and humidity for 72 hours. Overall, the peel performance of the VHPSA is comparable or better than the other two adhesives at 2 mil coating thickness on SS and other high surface energy substrates such as glass and aluminum. On the plastic substrates, the HPSA shows a slightly improved performance possibly due to its ability to flow and easy wet-out.

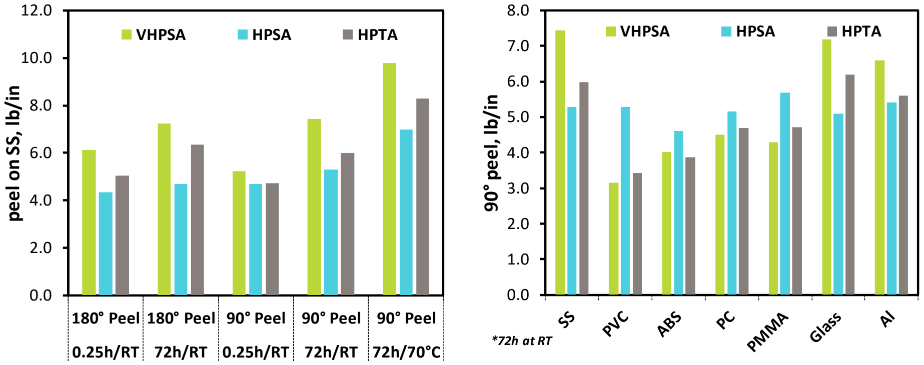

Figure 3. Peel performance comparison of VHPSA, HPSA and HPTA on stainless steel *(left)* and various surfaces *(right)*.

During its service life, adhesives are bound to be subjected to variety of temperatures, and in Figure 4 the peel adhesion VHPSA, HPSA and HPTA are compared as a function of temperature cycling. Adhesive samples on Al foil were bonded to SS panels and subjected to a cold-hot cycles of 4 hrs at 70°C, 4 hrs at -29 ° C and finally 16 hrs at 23°C.

All three adhesives showed good resilience to the extreme temperature cycling and even after 4 cycles and no negative impact observed for all three adhesives' peel adhesion. Despite the similar levels of resilience to thermal cycling, the adhesion of the VHPSA noticeably outperformed the two control adhesives throughout the temperature cycling tests.

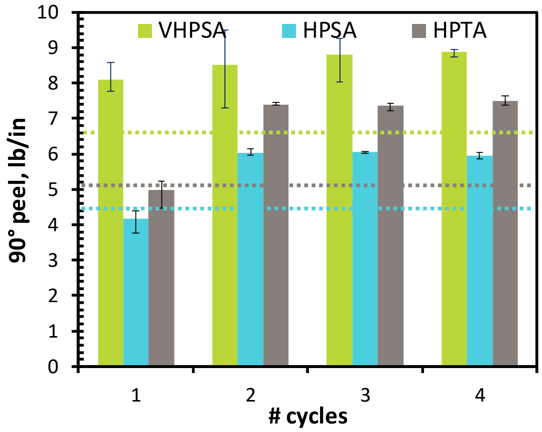

Figure 4. 90° peel adhesion as a function of temperature cycling. *Colored dashed lines are control data after 24 hours of bonding at RT*

Chemical resistance

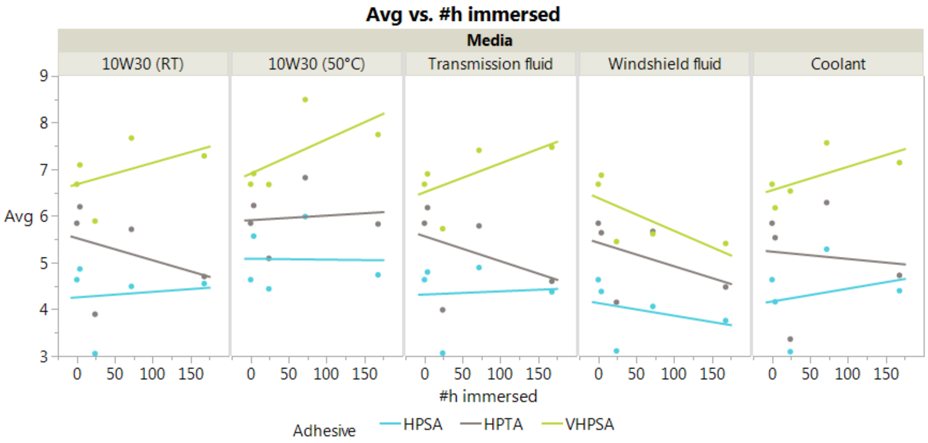

Figure 5. 90° peel adhesion of VHPSA, HPSA and HPTA after a week-long immersion in various automotive fluids

Figures 5 and 6 show the impact of full immersion of the bonded samples in various automotive fluids and aqueous solutions, respectively. As shown in Figure 5, VHPSA exhibits little to no change in peel adhesion to most automotive fluids even after a week-long full immersion study compared to the other two adhesives. Of all the automotive fluids, the winter-grade windshield fluid was the most aggressive towards all the adhesives. VHPSA retained a significant amount of peel adhesion compared to the other adhesives even after a week of immersion in this fluid.

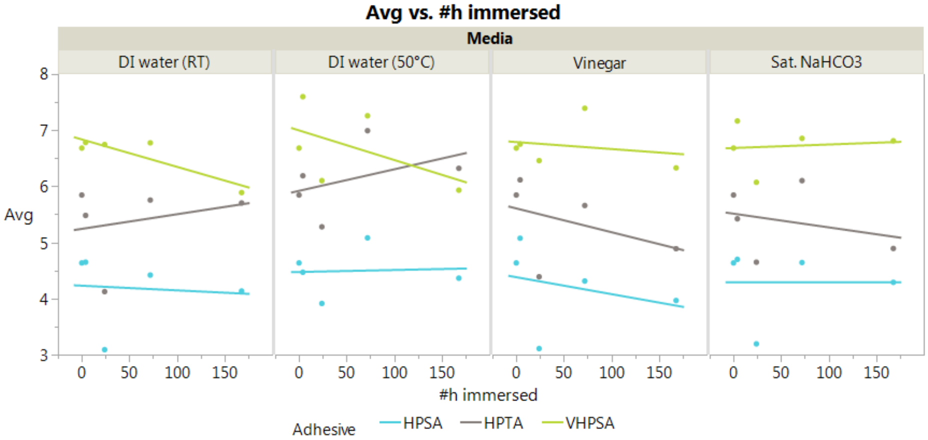

Figure 6. 90° peel adhesion of VHPSA, HPSA and HPTA after a week-long immersion in various aqueous solutions

Aqueous media had the least impact on any of the adhesives. Most of the observed changes seems to be coming from the liquid affecting the foil face-stock rather than the adhesive. No edge lift was observed for the bonded samples corroborating this statement.

Along with good chemical resistance VHPSA demonstrated excellent gasoline resistance (Figure 7). Samples were immerged for 1 or 24 hour into a mixture of isooctane and toluene that simulates gasoline, then were dried with a tissue and tested without recovery. After 1-hour immersion in gasoline peel adhesion for both VHPSA and HPTA did not change. HPSA peel reduced from 4.4 to 3.3 lb/in. While peel for all three adhesives reduced after 24 hour immersion vs 48 hour control, VHPSA has the smallest reduction in peel (31%) in comparison to 43 and 82% reduction for HPTA and HPSA respectively.

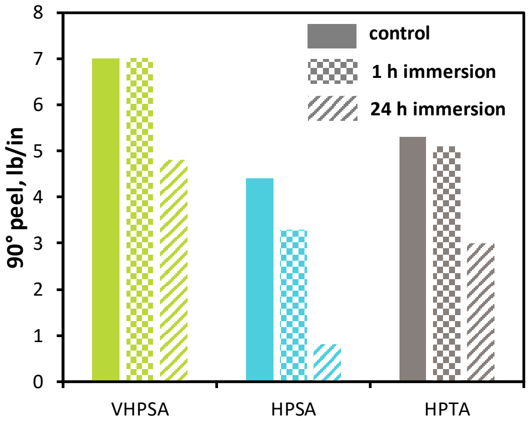

Figure 7. Gasoline immersion: 2 mil samples on Al foil. Gasoline used herein was ASTM reference fuel C (isooctane - toluene, 50/50 (v/v)). No recovery time prior to peel testing

Static shear

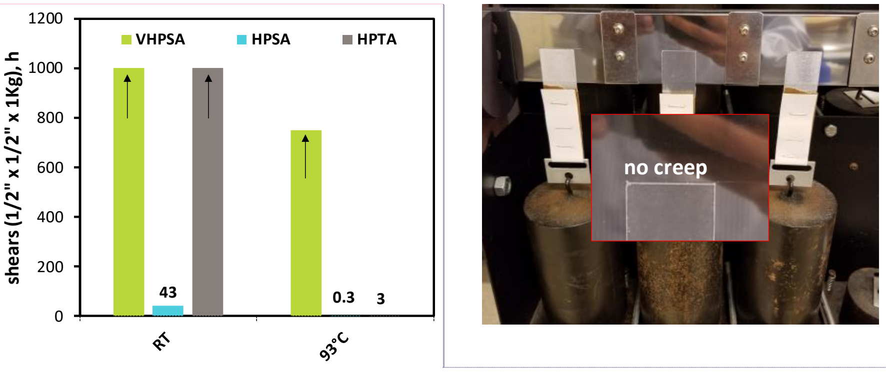

Figure 8. ½” x ½” x 1kg shear performance comparison of VHPSA, HPSA and HPTA at RT and 93°C *(left)* tested to a maximum of 1000 h; VHPSA 1" x 1" x 10 lb shear after 3000 hr *(right).*

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It is very difficult to achieve both high peel performance and high shear. Those characteristics are known trend opposite to each other. Generally, as cohesive strength is increased, adhesion strength is reduced, which makes it is easier to peel PSA from substrates. As shown in Figure 8, although the HPSA control demonstrates relatively high peel, it has poor ambient and high temperature shears (43 and 0.3 hour respectively), compared to the other two adhesives. On other hand, the HPTA control has ambient shears (>1000 hr) comparable to VHPSA. However, the HPTA control came up significantly short on 93 °C static shears (3 hr vs 750 hours). VHPSA has such a high cohesive strength, even for 10 lb load at 1 inch^2 overlay we did not see failure or slippage after 3000 hr (see Figure 8 (right). To date, this team has been unable to induce any VHPSA shear samples (up to 5 mil) to failure (accompanied by cohesive failure) at room temperature under any test configurations.

Shear adhesion Failure Temperature (SAFT)

SAFT is the temperature at which the sample fails in shear mode at constant load. This test can be viewed as good indicator of real life high temperature applications, wherein typically a constant load is applied while the temperature can fluctuate significantly above ambient.

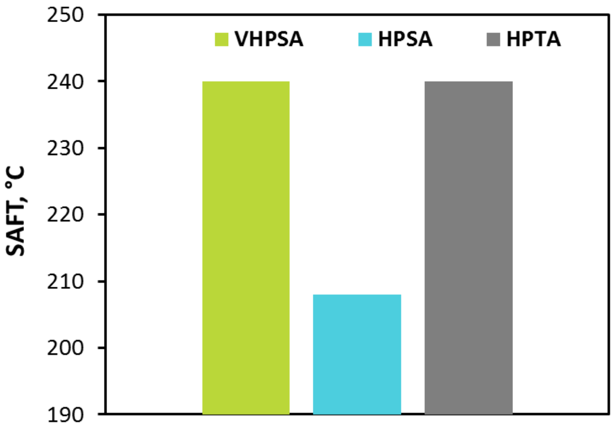

Figure 9. SAFT of VHPSA, HPSA and HPTA

The samples were heated at a constant rate of 1°C/min., while a constant load of 1 kg was applied in shear mode. Figure 9 compares the SAFT values of VHPSA, HPSA and HPTA under these conditions. HPSA exhibited a lower SAFT value compared to VHPSA and HPTA, consistent with the elevated temperature shear data. It is interesting to note that both VHPSA and HPTA have similar SAFT values although the VHPSA has a better elevated temperature shear strength

Lap shear

Lap shear is the most common test used for structural adhesives to evaluate the strength of adhesive joints. Single lap joints are easy to make and to test. To ensure complete bonding of the adhesive to the substrates, lap shear joints were subjected to 20 psi pressure for 30 sec using a modified vise clamp. The joints were then allowed to dwell for 72 hour under constant temperature and humidity prior the testing. We also tested various other industry-relevant substrates, including Nylon PA6, PA6/6 & PA6/6 GF, polycarbonate (PC), and sheet molding compound (SMC), as well as aluminum and cold rolled (CR) steel.

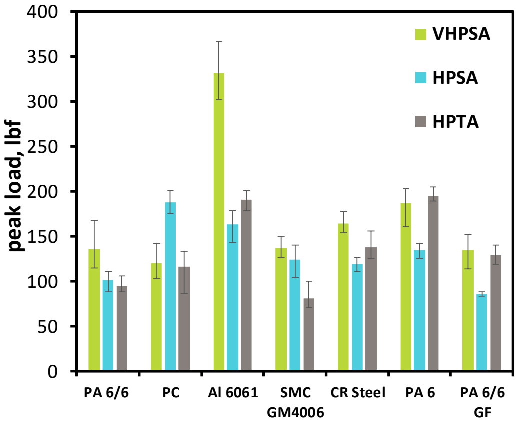

Figure 10. Lap shear of VHPSA, HPSA and HPTA on various substrates

All three adhesives reveal comparable peak load on PA6/6. VHPSA and HPTA performed better than HPSA on PA6, PA6/6 GF, and cold rolled steel (Figure 10). HPSA had much better anchorage to PC and similar performance to VHPSA peak load on SMC. VHPSA demonstrated outstanding performance on aluminum. Peak load for VHPSA on Al 6061 was about twice higher than same for HPSA and HPTA. Such a high performance opens the opportunity to use solution acrylic PSAs in semi-structural applications.

Arcan fixture tensile strength

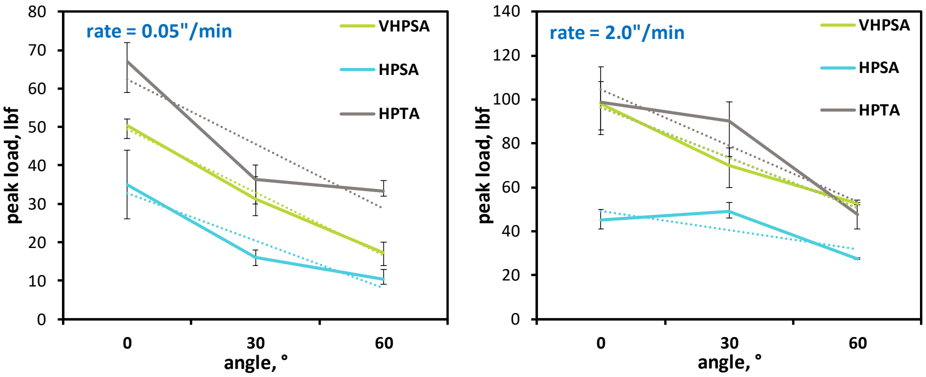

Figure 11. Peak load during tensile testing of VHPSA, HPSA and HPTA at an extension rate of 0.05"/min (left) and 2.0"/min (right)

The “Arcan” fixture is a circular fixture as shown in Figure 2. The test specimens bonded with the adhesive to be tested is loaded at the center. The shape of the specimen concentrates the stress at the center where it has the smallest cross section.[1] The design of the test fixture allows it to be rotated by 10° increments. Herein, the 0° would be the pure tensile and 90° would be pure shear. For this experiment we tested the samples in triplicate at three different angles 0, 30 and 60° and at two different rates, ca. 0.05”/min and 2.0”/min. As shown in Figure 11 the peak load continues to decrease as we go from a normal tensile stress to more shear stress for all three adhesives. The HPTA and VHPSA shows higher peak load to failure compared to the HPSA. It is interesting to note that the while the HPTA shows improved performance over VHPSA at lower debonding rates they behave very similarly at higher rates. The performance measurements at higher rates is more useful to know how the adhesives will perform, for instance when exposed to man-made or natural calamities.

It was observed that the failure mode for most of the samples was an adhesive failure, i.e., the adhesive always remains on the first surface to which it was applied to. Only in a few instances, there was a partial transfer to the second surface and never was there a cohesive failure at any of the angles for any of the adhesive. This shows that the internal strength of the adhesives is greater than the adhesive strength.

Dynamic Mechanical Analysis (DMA)

DMA was performed to get a better understanding of the viscoelastic properties of each adhesive and how they relate to peel strength and shear results. Time Temperature Superposition analysis was performed with each of the three adhesives at 25 °C. Chang’s Concept of the viscoelastic window was applied to compare the adhesives at a standard test temperature of 25 °C and relate adhesive properties to the viscoelastic nature of each adhesive.[12,13] The viscoelastic window for VHPSA, HPSA, and HPTA are illustrated in Figure 12.

According to Chang, peel strength is a combination of bonding (measured at 0.01 rad/s) and debonding potential (measured at 100 rad/s). The storage modulus, G’, and loss modulus, G”, measured at 100 rad/s are the highest for the VHPSA (top right corner of viscoelastic window) when compared to HPTA and HPSA. This indicates that VHPSA will have a high resistance to debonding (or a high peel strength), and VHPSA does have higher peel strength on polar substrates when compared to both HPTA and HPSA. Bonding of the adhesive is indicated by the bottom of the viscoelastic window. In the case of VHPSA, the bottom of the viscoelastic window is higher than both the HPTA and HPSA indicating that bonding may occur more slowly for VHPSA when compared to the other two adhesives. Peel testing at 90° with a 15 min dwell indicates only slightly better peel values for VHPSA when compared to HPSA and HPTA. When the dwell time is increased to 72 hour, the difference in peel strength of VHPSA increases due to an increase wet out time (increased flow) and a high resistance to debonding when compared to the other adhesives.

The bottom of the viscoelastic window indicates the potential cohesive strength of the adhesive. Shear testing like bonding is a low frequency event. Storage modulus, G’, measured at 0.1 rad/s can be an indication of cohesive strength. At room temperature, VHPSA and HPTA do not fail the typical ½” x ½” x 1 kg shear test. At 93°C, the cohesive strength of VHPSA is substantially higher than that of HPTA and HPSA. The bottom of the viscoelastic window predicts that VHPSA has a higher modulus at low frequency G’ measurements. Low frequency G’ measurements correlate to the plateau modulus of a material and predict how an adhesive might resist cohesive failure (or flow) at room temperature and high temperature.

VHPSA when compared to HPTA and HPSA is unique in that it maintains high energy dissipation and peel strength as indicated by 90° angle peel adhesion tests performed at 70 °C. In addition, it also maintains cohesive strength at elevated temperatures as indicated by shear experiments performed at 93 °C. The performance of this adhesive when compared to the others can be predicted by a simple analysis of the viscoelastic window as described above.

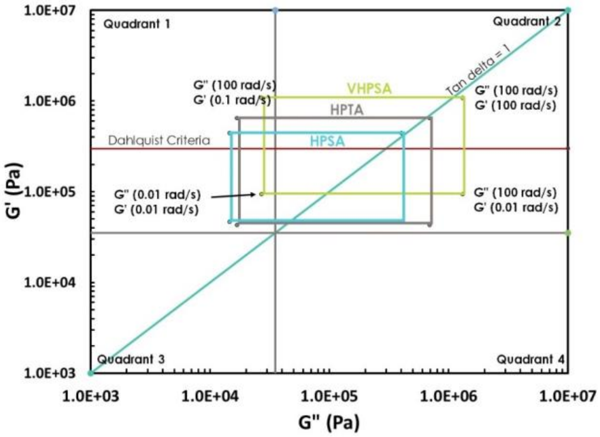

Figure 12. Viscoelastic windows for VHPSA, HPTA, and HPSA with all data collected at 25°C using TTS analysis of DMA frequency scans.

Conclusions

A new solvent-based coater ready PSA (VHPSA) has been shown to exhibit very high peel adhesion as well as high shear strength required for high permanency. This new PSA shows comparable peel adhesion to the HPSA and HPTA over a wide variety of polar substrates and significantly better shear strengths both at ambient and elevated temperatures. Also, the chemical resistance of the VHPSA is far superior to the other adhesives that were tested for comparison.

These gains in the VHPSA cohesive performance were achieved without the use of any covalent bond forming primary or secondary crosslinking mechanisms, which results in the ability of the technology to leverage the processing and process capability advantages that are inherent to aluminum metal chelate crosslinked systems. Frequency sweeps of the adhesive through DMA also confirms the superior performance of the VHPSA compared to the other two adhesives tested. We attribute this improved PSA performance of the new adhesive VHPSA to its unique polymer design and topology, which is also still capable of using the widely utilized and highly robust metal-chelate crosslinker, Aluminum Acetylacetonate.

Furthermore, the VHPSA with its high lap shear could mark the entry of pressure sensitive adhesives into semi-structural adhesive regime without the need for any specialized equipment for the manufacture of the same. This is a significant development as the PSAs are known for it is ability to wet-out rapidly and provide higher bond strength quickly. Achieving very high strength rapidly will be a big boost for automotive, electronics and building & construction industries.

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