

RHEOLOGICAL AND CURE EXAMINATION OF SILICONE PRESSURE SENSITIVE ADHESIVES

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1.0 Introduction

The composition of a silicone pressure sensitive adhesive (PSA) is based on a polymer filled system. The two main components that dictate the performance of the silicone PSA are a high molecular weight, linear siloxane polymer and a highly condensed, silicate tackifying resin (MQ resin). Figure 1 shows the structure of a typical silicone polymer. Commercially available silicone PSAs utilize either a polydimethylsiloxane polymer or polydimethyldiphenylsiloxane co-polymer that may contain silanol or vinyl functionality at the polymer chain ends.

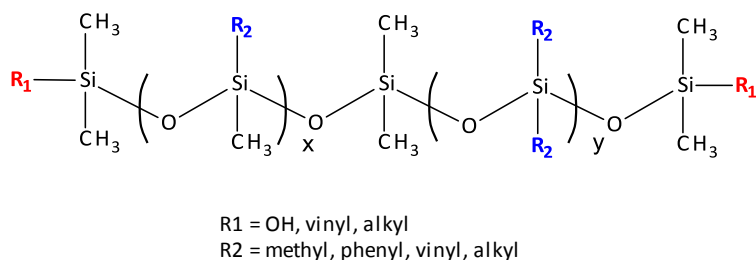
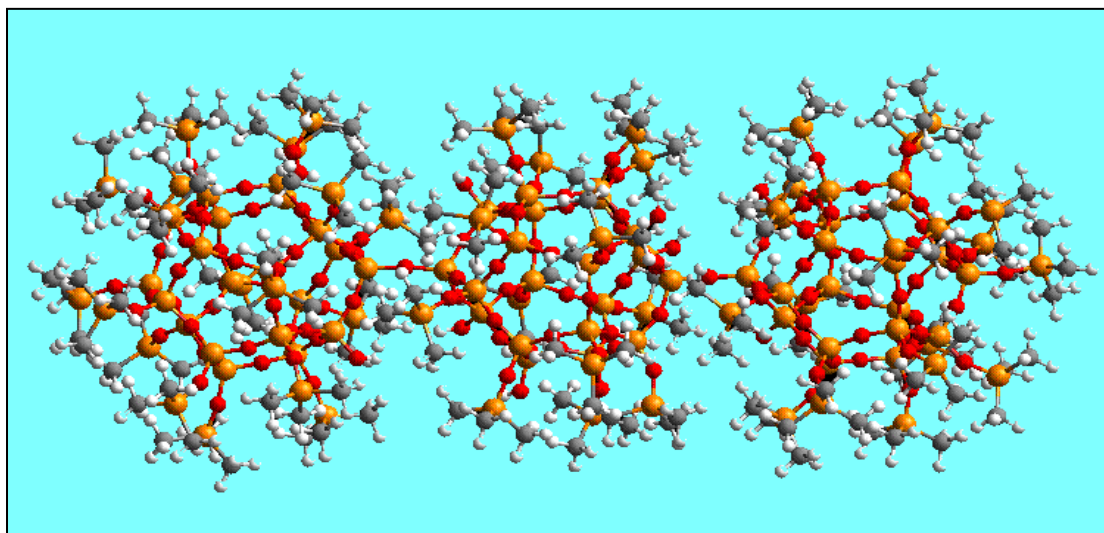


Figure 1. General chemical structure of a silicone polymer.

The silicate resin, often referred to as a MQ resin, is a solid particle supplied in a hydrocarbon solvent. The MQ name derives from the fact that its structure consists of a core of three-dimensional Q-units ($\text{SiO}_{4/2}$) surrounded by a shell of M-units ($\text{Me}_3\text{SiO}_{1/2}$). The resin also contains a low level of silanol functionality on the surface. The ratio of M:Q is typically in the range of 0.6-1.2:1. Figure 2 shows a



computer generated molecular model of a silicate resin.

Figure 2. Molecular model of a MQ resin.

Silicone PSAs are produced by blending a specified ratio of a MQ resin and siloxane polymer together in a hydrocarbon solvent. Heating the mixture to promote a condensation reaction between the available silanol functionality on the resin and polymer can further enhance the initial cohesive strength of the adhesive. The ratio of resin to polymer is the most important formulation detail when trying to optimize the balance of performance properties for a given adhesive. Figure 3 shows an example of how the balance of resin and polymer can affect the tack, peel adhesion, and shear performance for a silicone PSA. The exact positioning of these curves with respect to the x and y axes and each other is determined primarily by the resin composition.

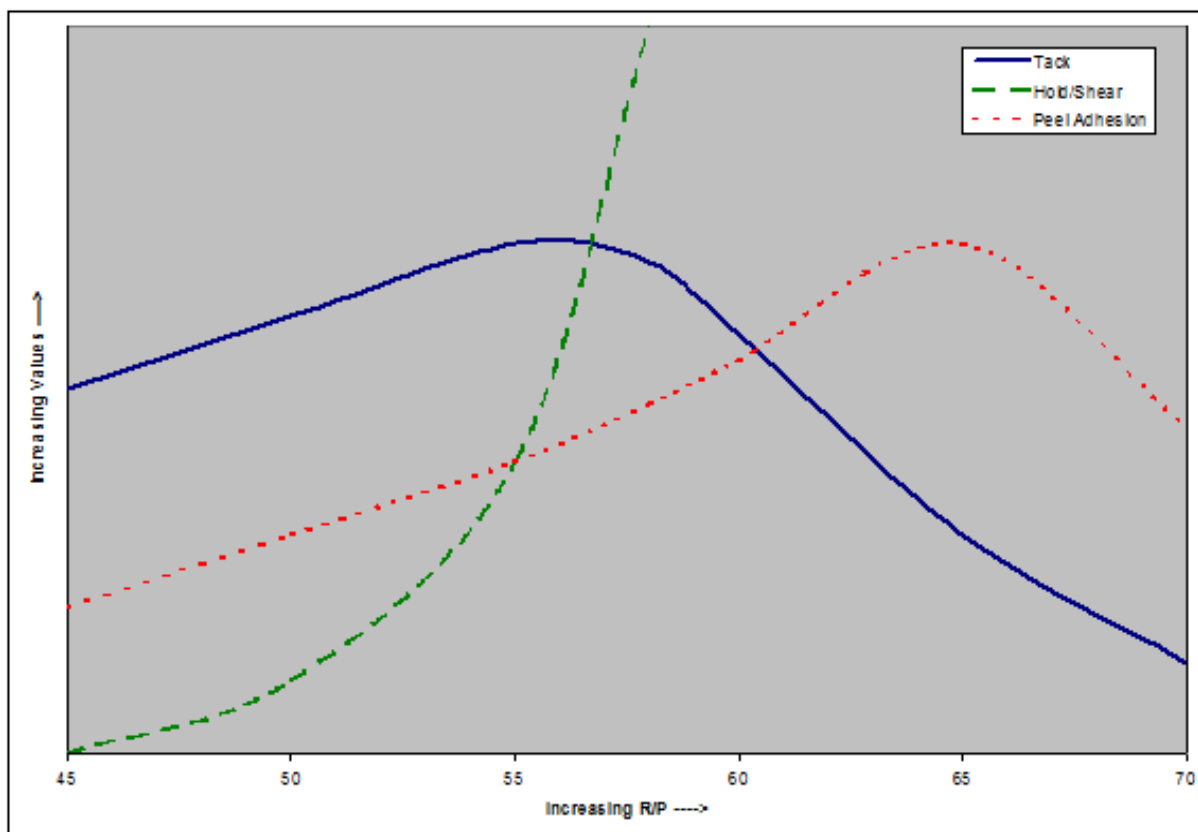


Figure 3. The effect of composition on silicone PSA properties.

1.1 Adhesive Cure Chemistry

Although most silicone PSAs will exhibit pressure sensitive behavior immediately after solvent removal, further crosslinking is done to reinforce the adhesive network. The level of additional crosslinking will depend on the intended application needs of the PSA construction. There are two basic cure systems available for silicone PSAs: peroxide catalyzed free-radical cure and platinum catalyzed silicon hydride to vinyl addition cure. The majority of commercial silicone PSAs employ the use of a peroxide catalyzed free-radical reaction to achieve additional crosslink density. Curing of these types of adhesives is done in multi-zoned ovens due to the use of non-specific peroxides. Solvent removal is first required at lower temperatures (60 to 90°C) to ensure the peroxide does not inadvertently cure solvent in the PSA matrix which would result in reduced performance and poor temperature stability. At elevated temperatures (130 to 200°C), the catalyst decomposes to form free radicals which primarily attack the

organic substituents along the polymer chains to extract protons and generate free radicals¹. The free radicals then combine to form crosslinks as shown by the general reaction mechanism in Figure 4.

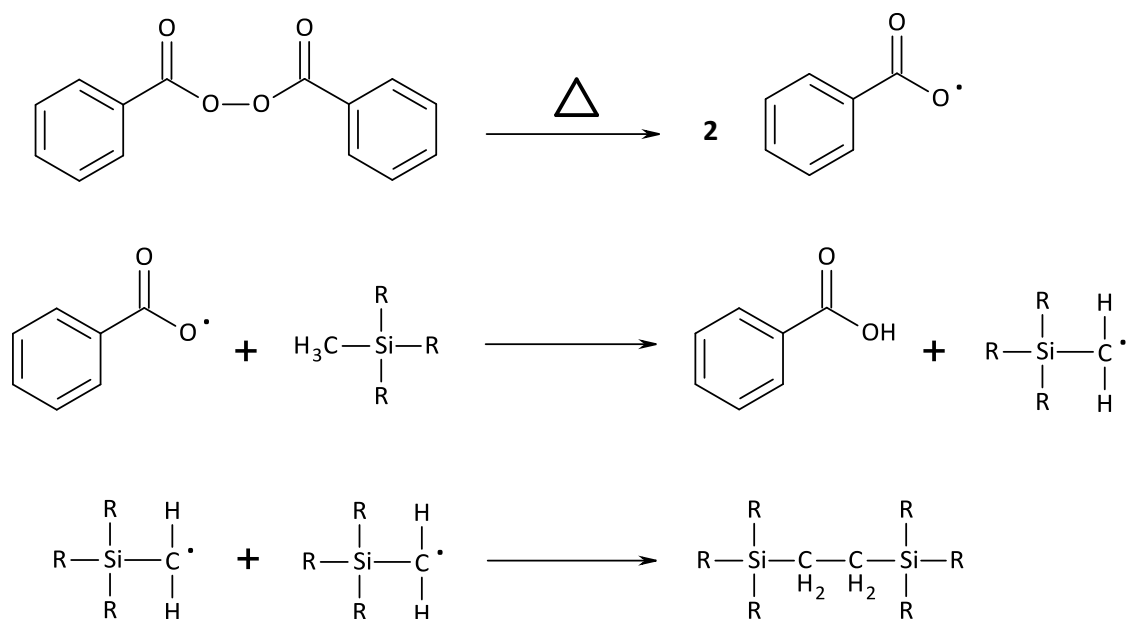


Figure 4. General steps in the peroxide catalyzed crosslinking of silicone PSAs.

The main benefit of the peroxide catalyzed system is the ability to control properties by addition level of peroxide used. The tape producer has the flexibility to use a range from 0 to 4 wt% peroxide. The additional curing with the peroxide results in a more tightly crosslinked PSA. An increase in cohesive strength, as evidenced by performance in shear tests, is generally observed. The increase in cohesive strength is accompanied by a slight decrease in adhesion and tack. Some of the disadvantages of this type of silicone PSA system include the handling of volatile solvents, generation of peroxide by-products, more sophisticated curing ovens, and the need for priming of certain substrates to improve adhesive anchorage in the construction of self-wound tapes.

As an alternative to the peroxide catalyzed system, silicone PSAs can utilize a platinum catalyzed addition cure in which a silicon hydride reacts with a silicon vinyl to form a crosslink site. This chemistry is analogous to the typical solvent-based and solventless platinum catalyzed silicone release coating systems used for release liners of organic PSAs. The curing of this type of silicone PSA can be accomplished in a single-zone oven at lower overall temperatures (100 to 150°C) even though these systems are supplied in hydrocarbon solvents. As the solvent evaporates, the platinum catalyzed reaction occurs without any generation of by-products as shown in Figure 5.

The ability of the system to be cured at a single, lower temperature offers benefits that are not seen with a peroxide catalyzed system. These benefits include faster line speeds (or cure time), lower sensitivity to temperature variation, the ability to use substrates with lower thermal stability (polyethylene, polypropylene, etc.) and no generation of volatile by-products. Another benefit of the platinum catalyzed silicone system is the fact that it does not inherently need the hydrocarbon solvent for anything other than viscosity control. The peroxide catalyzed system not only needs the solvent for viscosity control, the solvent keeps the peroxide dissolved within the adhesive bath prior to coating on the web. This advantage of the platinum catalyzed system allows for the ability to manufacture solventless platinum catalyzed silicone PSAs.

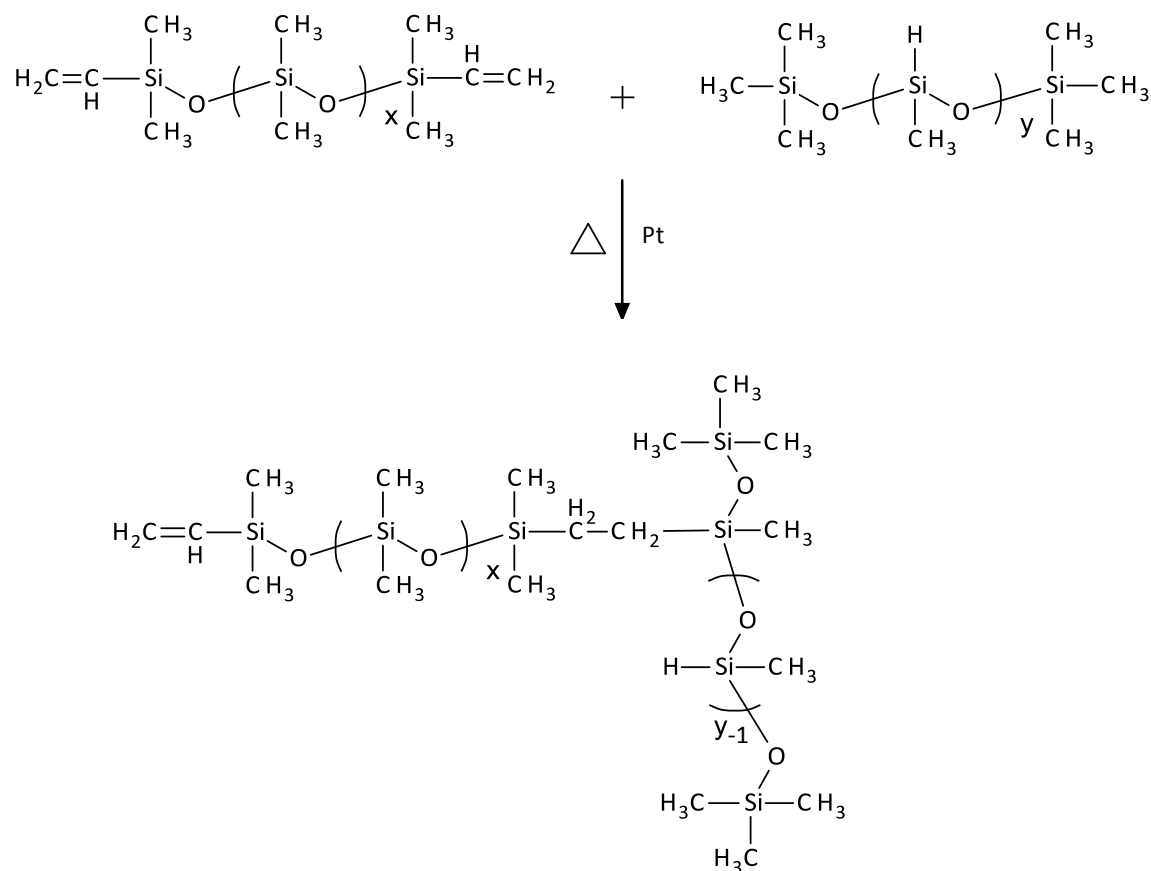


Figure 5. General reaction mechanism in the platinum catalyzed crosslinking of silicone PSAs.

2.0 Basic Physical Properties

Three different types of silicone PSAs were evaluated in this study. A polydimethylsiloxane peroxide cure PSA (Dimethyl Peroxide), a polydimethyldiphenylsiloxane peroxide cure PSA (Diphenyl Peroxide), and a polydimethylsiloxane platinum cure PSA (Dimethyl Platinum). The Dimethyl Peroxide PSA would be considered a general purpose silicone PSA with good releasability for label and transfer adhesive applications. The Diphenyl Peroxide PSA would be considered for applications that require performance in extremely high temperature applications. The Dimethyl Platinum PSA is designed to have low adhesion properties for applications such as protective films. The non-volatile content (NVC) and viscosity for each material prior to catalyzing and coating is listed in Table 1.

Table 1. Basic physical properties of the silicone PSAs.

PSA type	NVC (%)	Viscosity (cP)
Dimethyl Peroxide	56.91	26,200
Diphenyl Peroxide	55.73	39,700
Dimethyl Platinum	40.91	31,000

3.0 Cured Adhesive Characterization

3.1 Formulating, Laminate Preparation, and PSA Testing Procedures

The peroxide catalyzed materials were formulated by adding a solvent solution of benzoyl peroxide (BPO) in an amount to yield 0, 1, 2, or 4 wt% BPO per silicone solids. Each formulation was subsequently diluted with additional solvent to reach a final solids content of 50 wt%. The platinum catalyzed materials were formulated by adding a platinum catalyst and a silicone-hydride (SiH) crosslinker to the vinyl-functional PSA in an amount to yield 0.5, 1.0, or 1.5 wt%.

PSA laminates were prepared by coating the formulated adhesives onto 2-mil untreated polyester (PET), 2-mil fluorosilicone treated PET, 10-mil untreated PET, and 1-mil untreated polyimide (PI) films using a vacuum coating table with an appropriate application bar to target a 1.5-mil dry adhesive thickness. The PET films coated with the peroxide catalyzed formulations were placed in an air-circulating oven at 80°C for two minutes to remove the solvent followed by 180°C for 2 minutes to cure. The second cure zone was increased to 204°C when PI films were coated. The platinum catalyzed formulations were cured at 150°C for 3 minutes on all film types. Each prepared laminate was cut into test strips using a 1" specimen tape cutter and the thickness was measured using a digital micrometer.

The 2-mil untreated PET laminates were characterized by 180° peel adhesion per PSTC-101 and probe tack per ASTM D-2979. The 2-mil fluorosilicone treated PET laminates were characterized by peel release and rheometry. Prior to testing for release from the fluorosilicone liner, a 2-mil untreated PET film was laminated to the PSA to serve as a facestock. The rheology samples were further prepared by stacking several laminates together until a target thickness of 1.5 mm was reached. The fluorosilicone liner remained on both sides of the rheology sample. The 1-mil untreated PI laminates were characterized by high temperature static shear. The 10-mil untreated PET laminates were characterized by Texture Analyzer.

3.2 Dimethyl Peroxide PSA

Samples of a Dimethyl Peroxide PSA were prepared at levels of BPO catalyst varying from 0 to 4 wt% to evaluate the impact that the extent of cure would have on the resulting performance properties. The laminate thickness, peel adhesion, and tack results on 2-mil PET are shown in Table 2.

Table 2. Laminate thickness, peel adhesion, and tack results for the Dimethyl Peroxide PSA.

BPO (%)	Avg Thickness (mil)	Adhesion (g/in)	Tack (g)
0	1.68	1446.5	917.3
1	1.72	1432.0	741.1
2	1.68	1373.5	706.3
4	1.65	1101.0	577.0

As the level of catalyst was increased for the Dimethyl Peroxide PSA, it was found that there was a decreasing trend in both the adhesion and tack performance. This indicates that increasing the crosslink density of the Dimethyl Peroxide PSA has a direct, inverse relationship to these performance properties.

Release from the 2-mil fluorosilicone treated PET liner (or fluorosilicone release liner) was tested at 1, 7, and 14 days of room temperature (RT) aging. The release testing was completed using a delamination

rate of 12 inches per minute while removing the liner at an angle of 180° from the laminate. The results are shown in Figure 6.

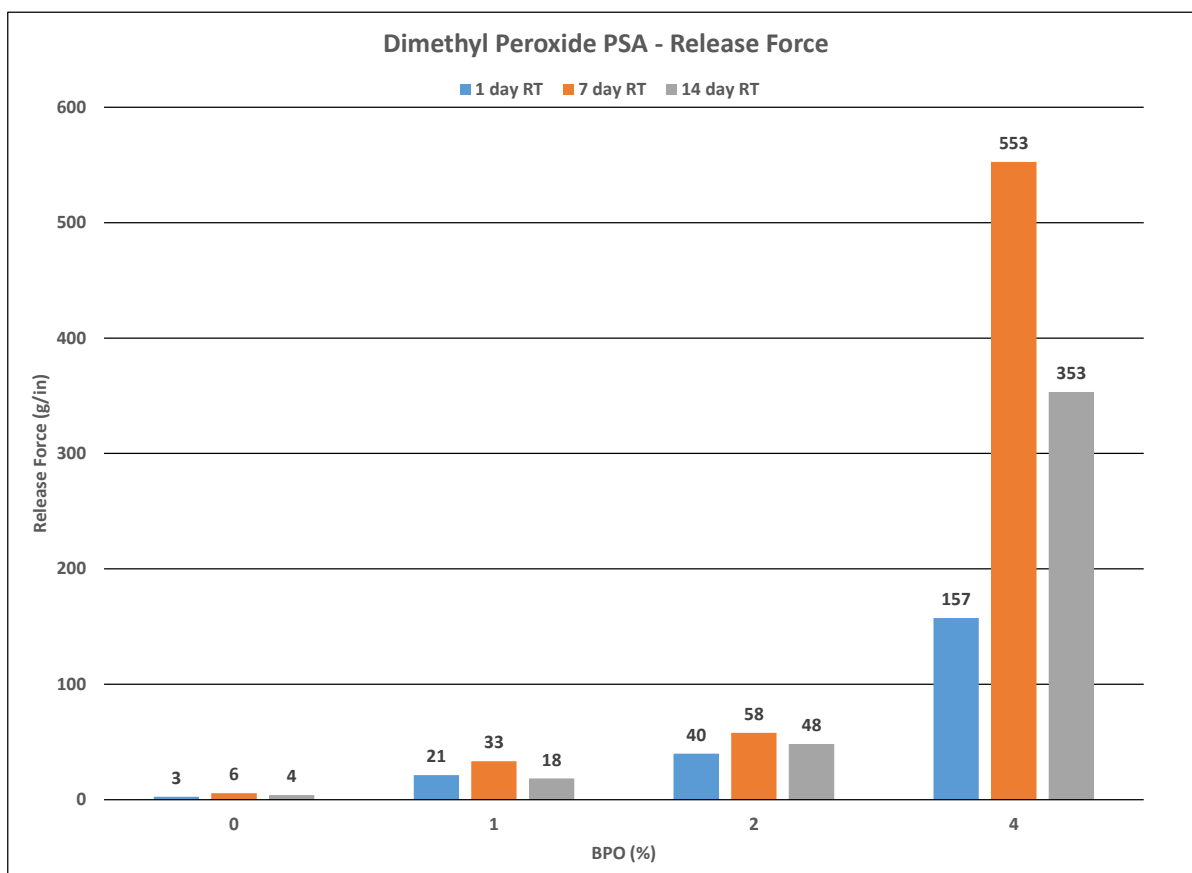


Figure 6. Release testing of the Dimethyl Peroxide PSA from a fluorosilicone release liner.

The release force increased as the level of peroxide increased for the Dimethyl Peroxide PSA. There was a significant increase in release force when moving from 2 to 4 wt% peroxide. Within each set of cured material (0, 1, 2, and 4 wt% BPO), the release force peaked at 7 days of aging. Although the increasing release force appears to have a direct relationship to the catalyst loading, the increase in crosslink density may not entirely account for the performance exhibited. Each formulation was coated directly onto the fluorosilicone release liner prior to cure. This has the potential to allow the peroxide catalyst to interact with the liner during the cure cycle and likely contributes to part of the increasing release forces.

The high temperature static shear performance was tested at 300°C using a 1 kilogram weight. The laminates were tested until complete failure was reached. The results are shown in Table 3.

Table 3. High temperature static shear results for the Dimethyl Peroxide PSA.

BPO (%)	Fail Time	Failure Mode
0	15 min	Cohesive
1	30 min	Cohesive
2	30 min	Cohesive
4	9 Day	Cohesive

As the level of catalyst was increased for the Dimethyl Peroxide PSA, the static shear performance exhibited an increasing trend. This indicates that increasing the crosslink density of the Dimethyl Peroxide PSA has a direct relationship to increasing static shear performance.

The stress-strain curves generated by the TA-XT2i Texture Analyzer² can be used as a supplement to evaluate the performance of the PSA. Studies have shown that the failure energy, as calculated by integration of the area under the curve, relates to the behavior of the adhesive during the bonding and debonding process^{3, 4, 5}. The calculations on each curve include the peak force, total area under the curve, and the area ratio (area post-peak / area pre-peak). Each 10-mil PET laminate was tested on the Texture Analyzer using a 7mm, stainless steel punch probe with a 1” radius at a test speed of 0.2 mm/sec, dwell time of 0.5 seconds, and test force of 100 grams. The results are shown in Figure 7 and Table 4.

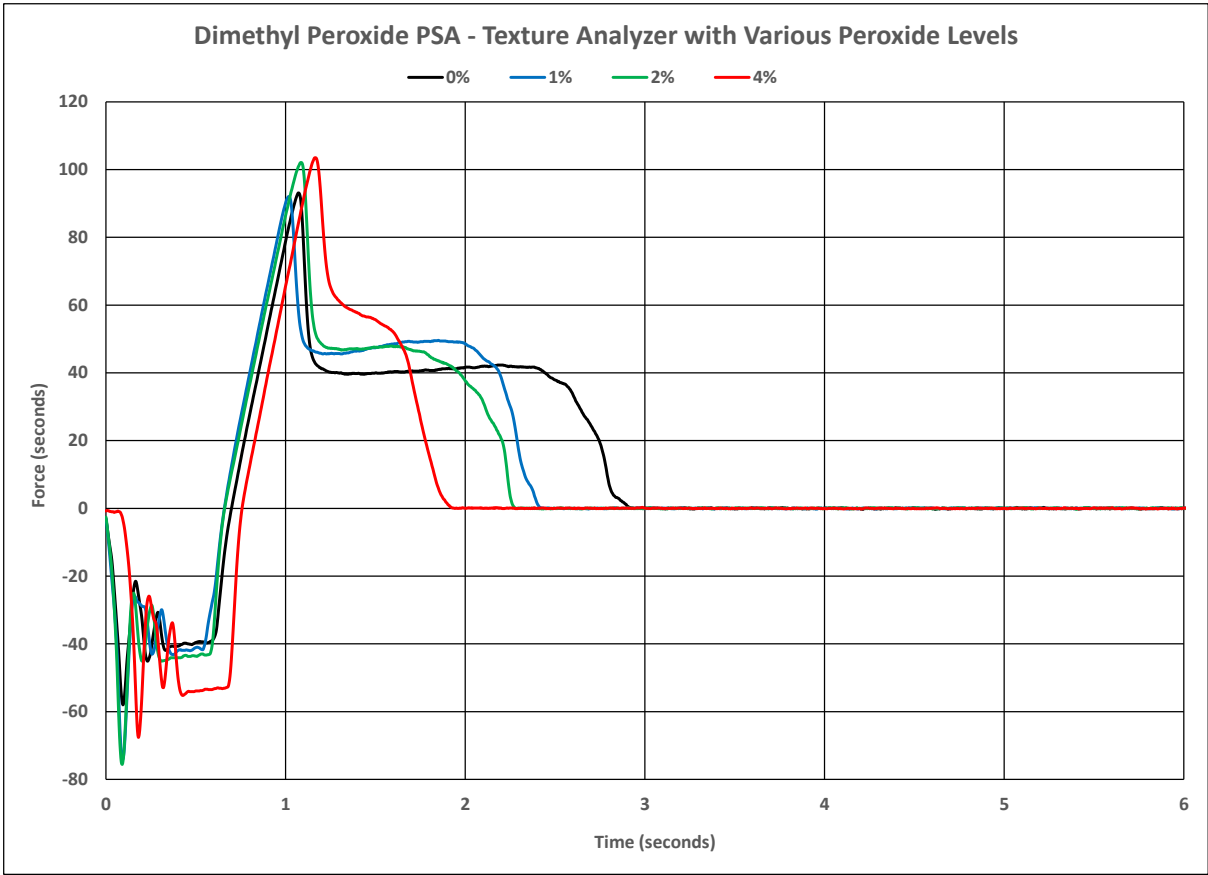


Figure 7. Texture Analyzer results for the Dimethyl Peroxide PSA.

Table 4. Texture Analyzer results for the Dimethyl Peroxide PSA.

BPO (%)	Average Peak Force (g)	Average Total Area (g.s)	Average Area Ratio
0	93.23	90.57	3.91
1	92.59	82.18	3.46
2	102.47	77.64	2.45
4	103.03	57.25	1.60

As can be seen in the TA curves, there is a secondary shoulder generated during the debonding step which indicates that fibrils are forming for these PSAs. The secondary shoulder and subsequent distance to probe separation decreases as the level of catalyst increases. The decrease in Total Area and Area Ratio correlates well with the adhesion and tack testing results for the Dimethyl Peroxide PSA.

Understanding the viscoelastic nature of silicone PSAs can help guide the proper selection of PSA type (dimethyl, diphenyl, etc.), crosslink density, and additives for a particular application. In addition, the rheological profile of a system can be used as a troubleshooting tool for determining sources of performance failures, as well as correlating against other performance tests. The rheology of each formulation was conducted using a dynamic temperature ramp at a rate of 2°C per minute. Each sample was tested using 8 mm stainless steel parallel plates at a frequency of 10 radians per second and a constant strain of 0.1%. The results are shown in Figure 8 and Table 5.

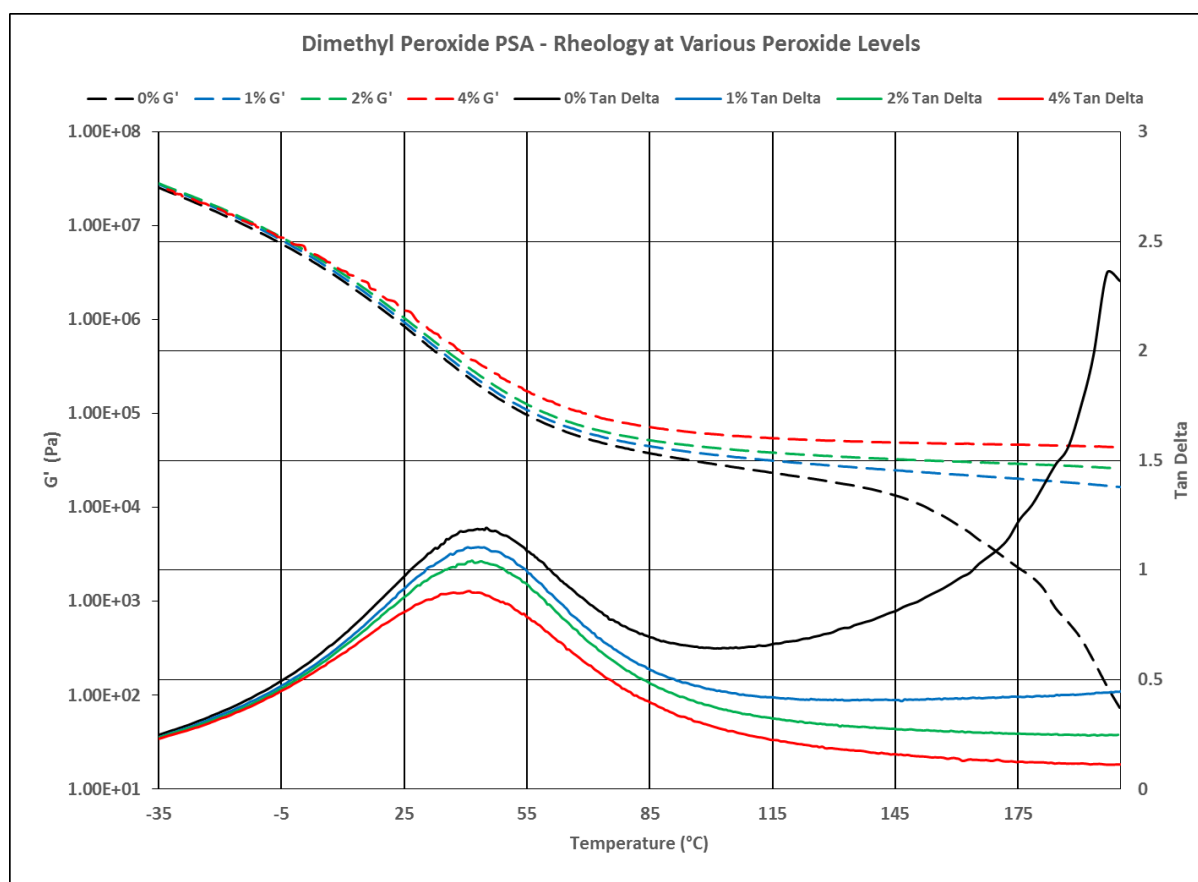


Figure 8. G' and tan delta curves for the temperature ramp testing of the Dimethyl Peroxide PSA.

Table 5. Selected rheology values for the Dimethyl Peroxide PSA.

BPO (%)	Tg (°C)	Tan Delta at Tg
0	45.1	1.19
1	43.2	1.10
2	41.6	1.04
4	41.1	0.90

The impact of crosslink density through catalyst addition for the Dimethyl Peroxide PSA becomes evident by the rheology analysis. As the catalyst concentration increases, the position of the Tg and the magnitude of the tan delta peak decreases. The largest effect is found in the rubbery plateau regions where the higher crosslink density increases the storage modulus. This reduces the ability of the PSA to dissipate energy and results in lower peel adhesion and tack. The tan delta profile at temperatures greater than roughly 115°C demonstrate how lower crosslinked systems start to exhibit flow when compared to the same PSA at higher catalyst levels.

3.3 Diphenyl Peroxide PSA

Samples of a Diphenyl Peroxide PSA were prepared at levels of BPO catalyst varying from 0 to 4 wt% to evaluate the impact that the extent of cure would have on the resulting performance properties. The laminate thickness, peel adhesion, and tack results on 2-mil PET are shown in Table 6.

Table 6. Laminate thickness, peel adhesion, and tack results for the Diphenyl Peroxide PSA.

BPO (%)	Avg Thickness (mil)	Adhesion (g/in)	Tack (g)
0	1.62	1164.0	1034.7
1	1.58	1145.0	1002.1
2	1.68	1233.5	872.1
4	1.62	1104.5	719.0

As the level of catalyst was increased for the Diphenyl Peroxide PSA, it was found that there was a decreasing trend in both the adhesion and tack performance. The adhesion did reach a maximum performance level at 2 wt% peroxide before decreasing back to the lowest level at 4 wt% BPO.

Release of the Diphenyl Peroxide PSA from the fluorosilicone release liner was tested at 1, 7, and 14 days of room temperature (RT) aging. The release testing was completed using a delamination rate of 12 inches per minute while removing the liner at an angle of 180° from the laminate. The results are shown in Figure 9.

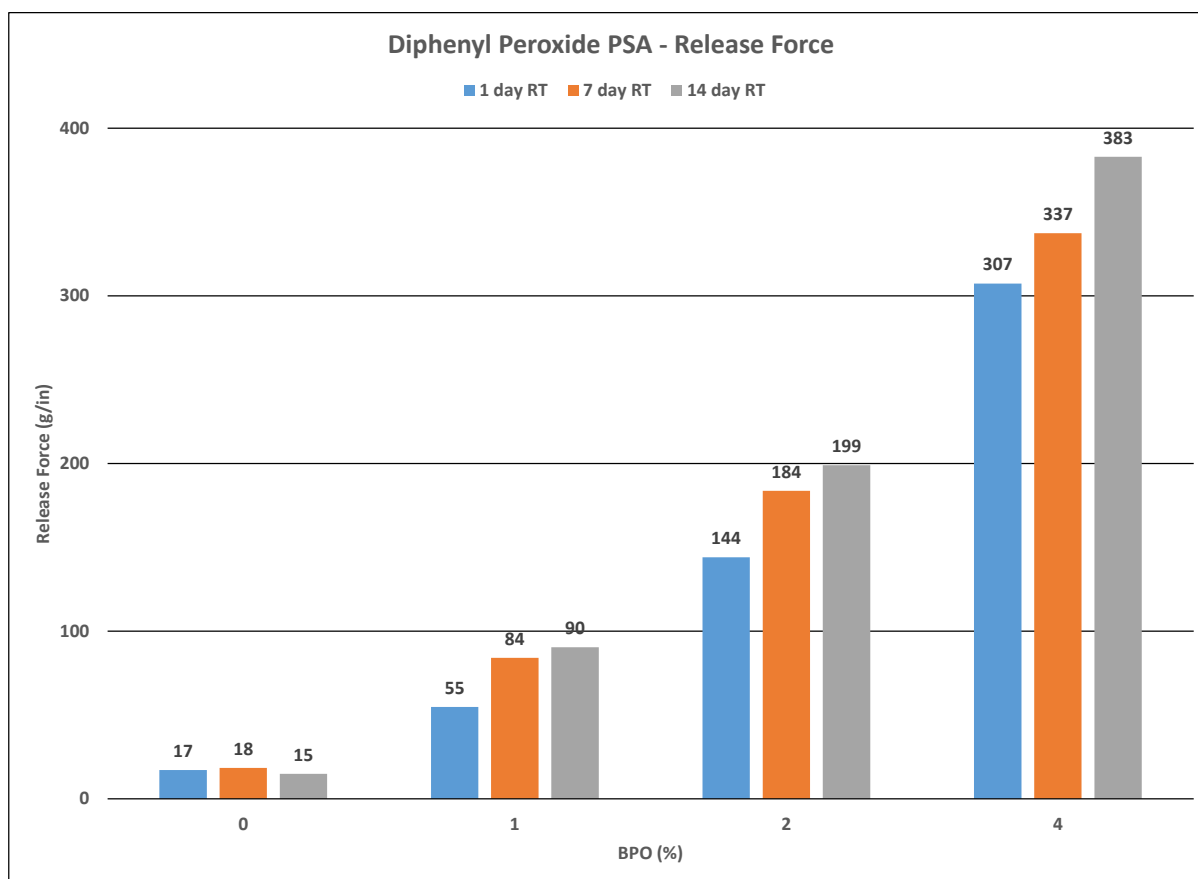


Figure 9. Release testing of the Diphenyl Peroxide PSA from a fluorosilicone release liner.

The release force increased as the level of peroxide increased for the Diphenyl Peroxide PSA. This behavior is similar to that seen with the Dimethyl Peroxide PSA. Within each set of cured material (0, 1, 2, and 4 wt% BPO), the release force continued to increase as the laminates aged.

The high temperature static shear performance for the Diphenyl Peroxide PSA is shown in Table 7.

Table 7. High temperature static shear results for the Diphenyl Peroxide PSA.

BPO (%)	Fail Time	Failure Mode
0	15 min	Cohesive
1	30 min	Cohesive
2	1 hour	Cohesive
4	Ended after 9 Days	NA

As the level of catalyst was increased for the Diphenyl Peroxide PSA, the static shear performance exhibited an increasing trend. This indicates that increasing the crosslink density of the Diphenyl Peroxide PSA has a direct relationship to the increase in static shear performance. At 4 wt% BPO, the testing was discontinued after the sample did not exhibit slip even after 9 days.

Each 10-mil PET laminate coated with the Diphenyl Peroxide PSA was tested on the Texture Analyzer and the results are shown in Figure 10 and Table 8.

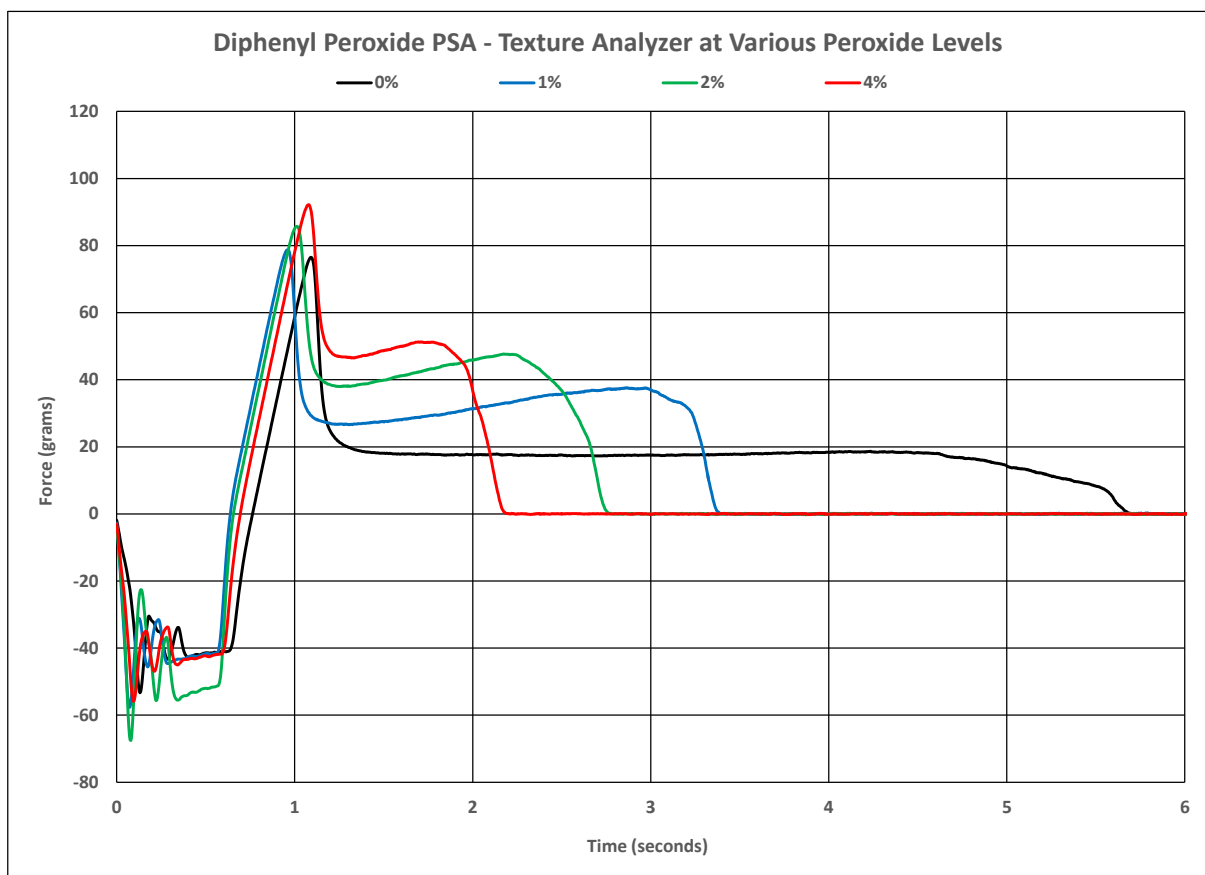


Figure 10. Texture Analyzer results for the Diphenyl Peroxide PSA.

Table 8. Texture Analyzer results for the Diphenyl Peroxide PSA.

BPO (%)	Average Peak Force (g)	Average Total Area (g.s)	Average Area Ratio
0	76.19	91.96	5.91
1	78.74	91.44	5.54
2	85.7	88.27	4.33
4	94.21	68.97	2.52

As can be seen in the TA curves, there is a secondary shoulder for each sample which indicates that these PSAs are forming fibrils during the debonding step. The secondary shoulder was much larger for the Diphenyl Peroxide PSA than the Dimethyl Peroxide PSA. This correlates to a tackier PSA at room temperature. The secondary shoulder and subsequent distance to probe separation decreases as the level of catalyst increases. The decrease in Total Area and Area Ratio correlates well with the adhesion and tack testing results.

The rheology of each Diphenyl Peroxide PSA formulation was conducted using a dynamic temperature ramp at a rate of 2°C per minute. Each sample was tested using 8 mm stainless steel parallel plates at a frequency of 10 radians per second and a constant strain of 0.1%. The results are shown in Figure 11 and Table 9.

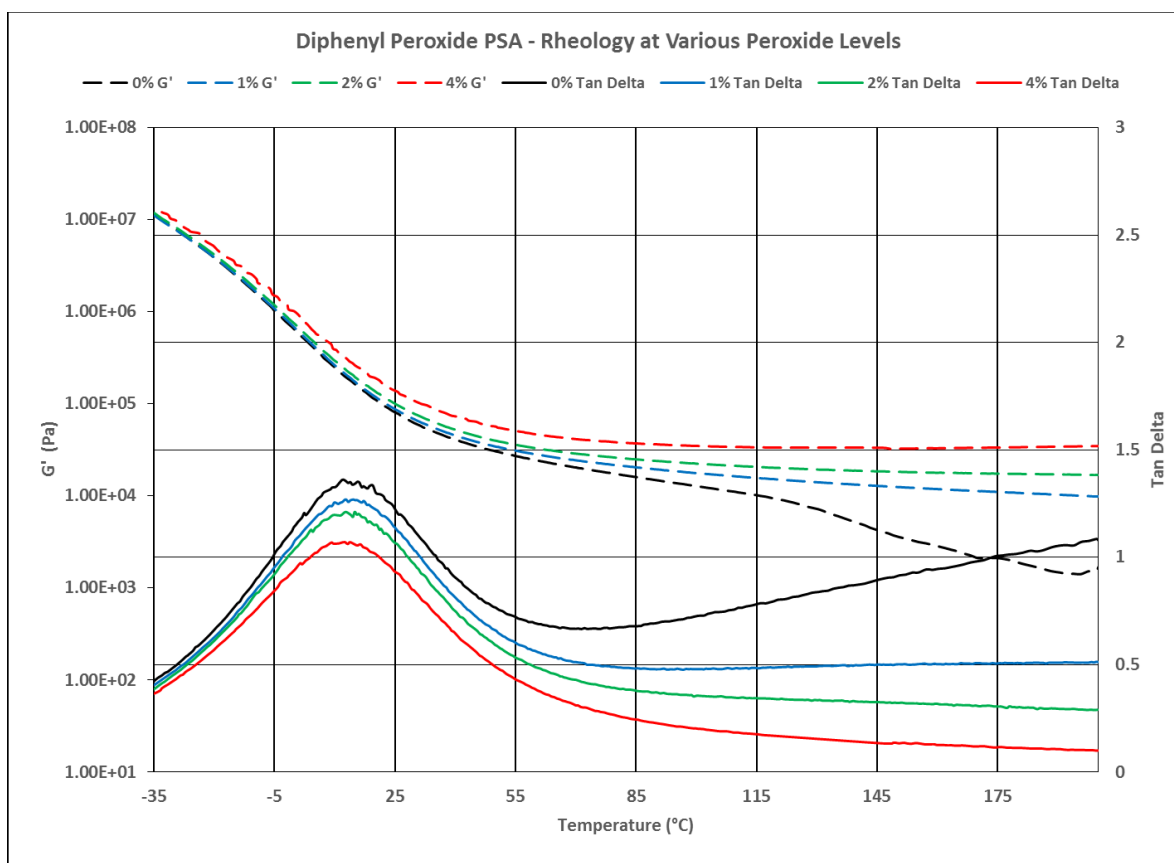


Figure 11. G' and tan delta curves for the temperature ramp testing of the Diphenyl Peroxide PSA.

Table 9. Selected rheology values for the Diphenyl Peroxide PSA.

BPO %	Tg (°C)	Tan Delta at Tg
0	11.9	1.36
1	12.7	1.27
2	12.6	1.21
4	12.4	1.07

The Diphenyl Peroxide PSA in this example has better high temperature stability when compared to the Dimethyl Peroxide PSA at the same peroxide loading. In the rheology analysis, this is indicated by the tan delta plateau and flatter G'. The Tg and magnitude of tan delta is greater for the Diphenyl PSA at all levels of peroxide concentration. As the catalyst concentration increases, the position of the Tg and the magnitude of the tan delta peak decreases. The largest effect is found in the rubbery plateau regions where the higher crosslink density increases the storage modulus. The higher crosslink increases the storage modulus. The tan delta profile at temperatures greater than roughly 85°C demonstrate how lower crosslinked systems start to exhibit flow when compared to the same PSA at higher catalyst levels.

3.4 Dimethyl Platinum PSA

Samples of a Dimethyl Platinum PSA were prepared at levels of crosslinker varying from 0.5 to 1.5 wt% to evaluate the impact that extent of cure would have on the resulting performance properties. A sample at 0% crosslinker was attempted but incomplete cure led to cohesive failure. The laminate thickness, peel adhesion, and tack results on 2-mil PET are shown in Table 10.

Table 10. Laminate thickness, peel adhesion, and tack results for the Dimethyl Platinum PSA.

XL (%)	Avg Thickness (mil)	Adhesion (g/in)	Tack (g)
0.5	1.67	4.1	52.8
1.0	1.69	3.2	41.7
1.5	1.67	3.1	47.3

The unique nature of this “low adhesion” PSA results in minimal adhesion and tack properties when tested against stainless steel substrates. As the level of crosslinker was increased for the Dimethyl Platinum PSA, it was found that there was a decreasing trend in the adhesion and tack performance.

Release of the Dimethyl Platinum PSA from the fluorosilicone release liner was tested at 1, 7, and 14 days of room temperature (RT) aging. The release testing was completed using a delamination rate of 12 inches per minute while removing the liner at an angle of 180° from the laminate. The results are shown in Figure 12.

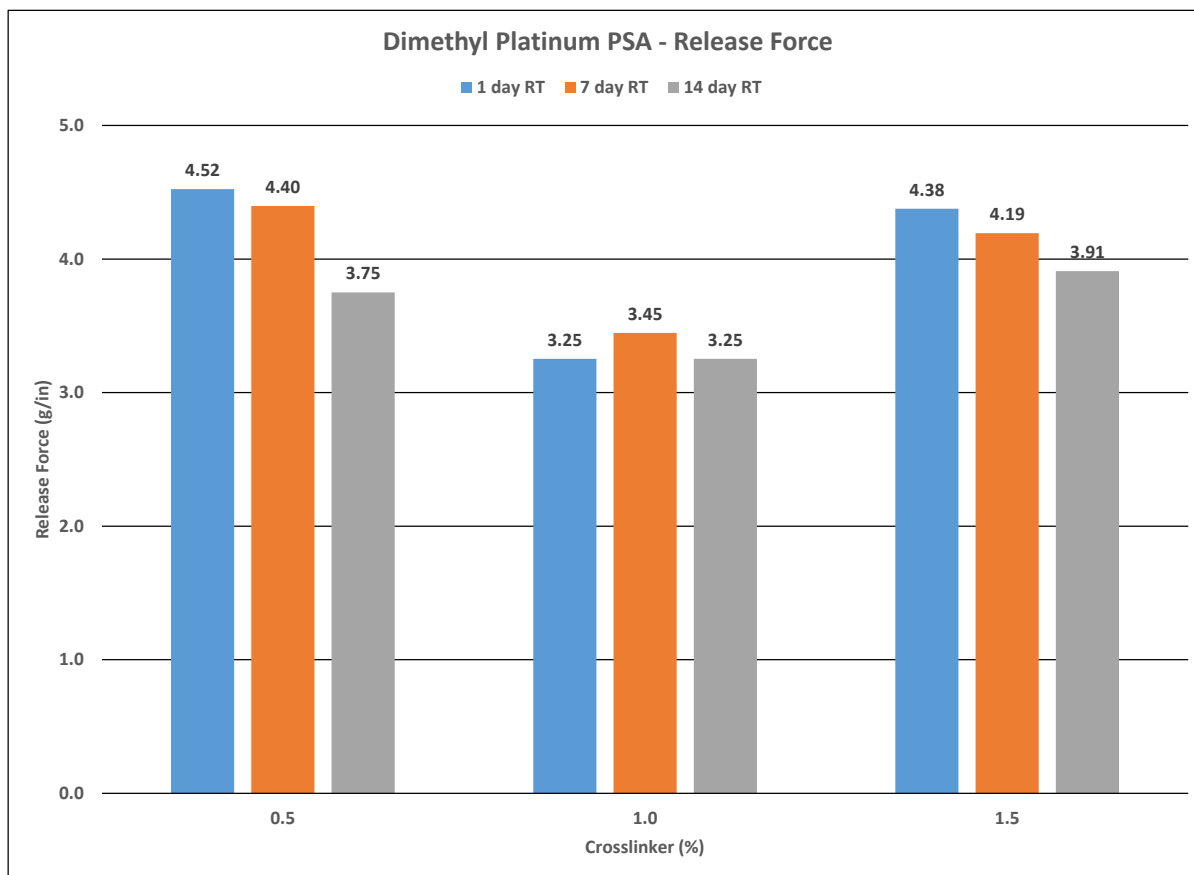


Figure 12. Release testing of the Dimethyl Platinum PSA from a fluorosilicone release liner.

A minimum release force for the Dimethyl Platinum PSA is seen at 1.0 wt% crosslinker. Samples prepared at 0.5 and 1.5 wt% crosslinker show a decreasing trend with time. The 1.0 wt% crosslinker sample was stable over the 14 day test period.

The high temperature static shear performance for the Dimethyl Platinum PSA is shown in Table 11.

Table 11. High temperature static shear results for the Dimethyl Platinum PSA.

XL (%)	Fail Time	Failure Mode
0.5	45 min	Cohesive
1.0	6 Day	Cohesive
1.5	Ended after 14 Days	NA

As the level of crosslinker was increased for the Diphenyl Platinum PSA, the static shear performance showed significant improvement. In fact, the performance of the Dimethyl Platinum PSA was the best of the 3 PSAs evaluated in this study.

Each 10-mil PET laminate coated with the Dimethyl Platinum PSA was tested on the Texture Analyzer and the results are shown in Figure 13 and Table 12.

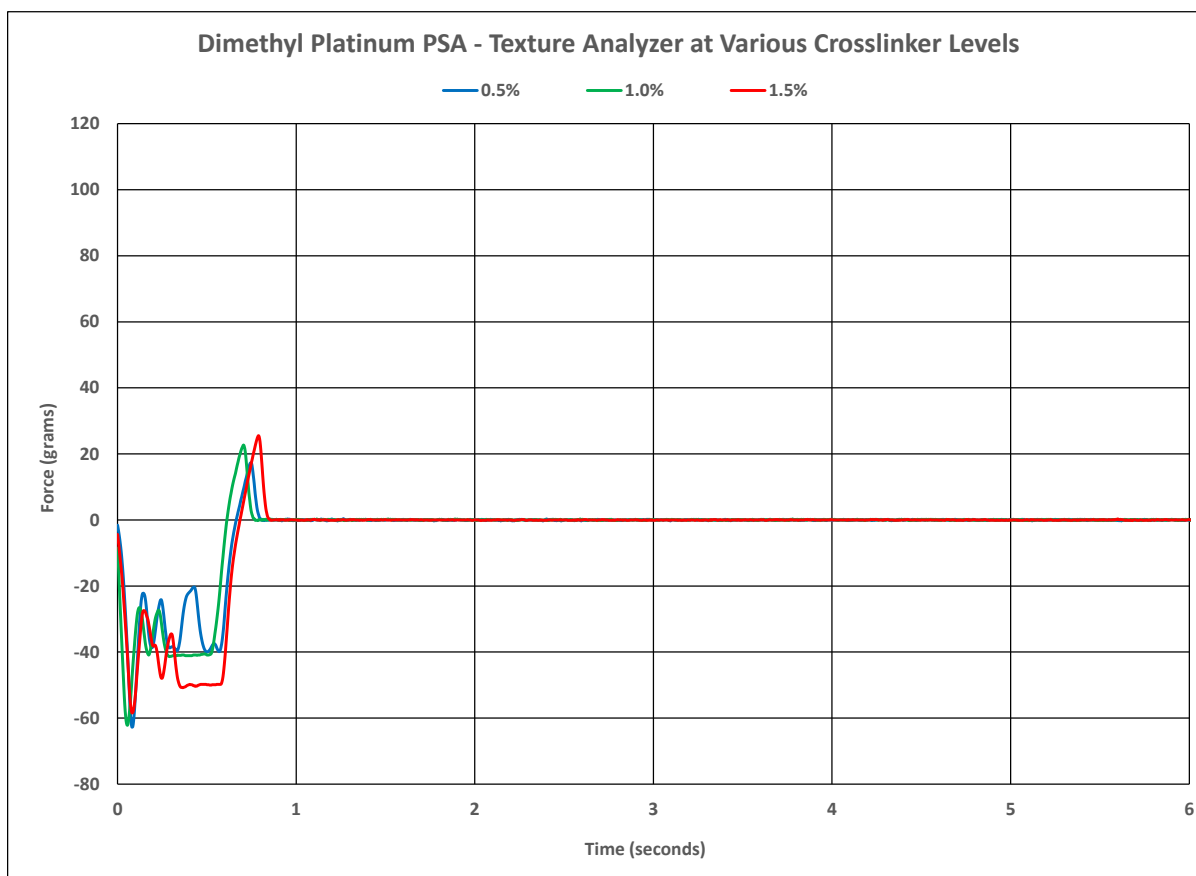


Figure 13. Texture Analyzer results for the Dimethyl Platinum PSA.

Table 12. Texture Analyzer results for the Dimethyl Platinum PSA.

XL (%)	Average Peak Force (g)	Average Total Area (g.s)	Average Area Ratio
0.5	17.58	1.20	0.62
1.0	22.48	1.83	0.48
1.5	24.48	2.10	0.46

The Texture Analyzer testing of the Dimethyl Platinum PSA showed very small peaks indicating low tack and adhesion performance. The results showed very little differentiation between levels of crosslinker.

The Dimethyl Platinum PSA used in this study presented additional challenges when attempting to test rheology owing to the low adhesion nature of the cured material. At low temperatures, the PSA would not remain in contact with the parallel plates and resulted in poor data. The sample was submitted for DMA analysis using testing conditions similar to those used for silicone elastomers. Film tension clamps were used to secure the cured material and a multi rate temperature ramp was utilized. Between -145 °C and -30 °C the rate was 0.5 °C/min, and at temperatures above -30 °C it increased to 3.0 °C/min. A strain rate of 0.25% was applied at 10 radians per second. The results are shown in Figure 14 and Table 13.

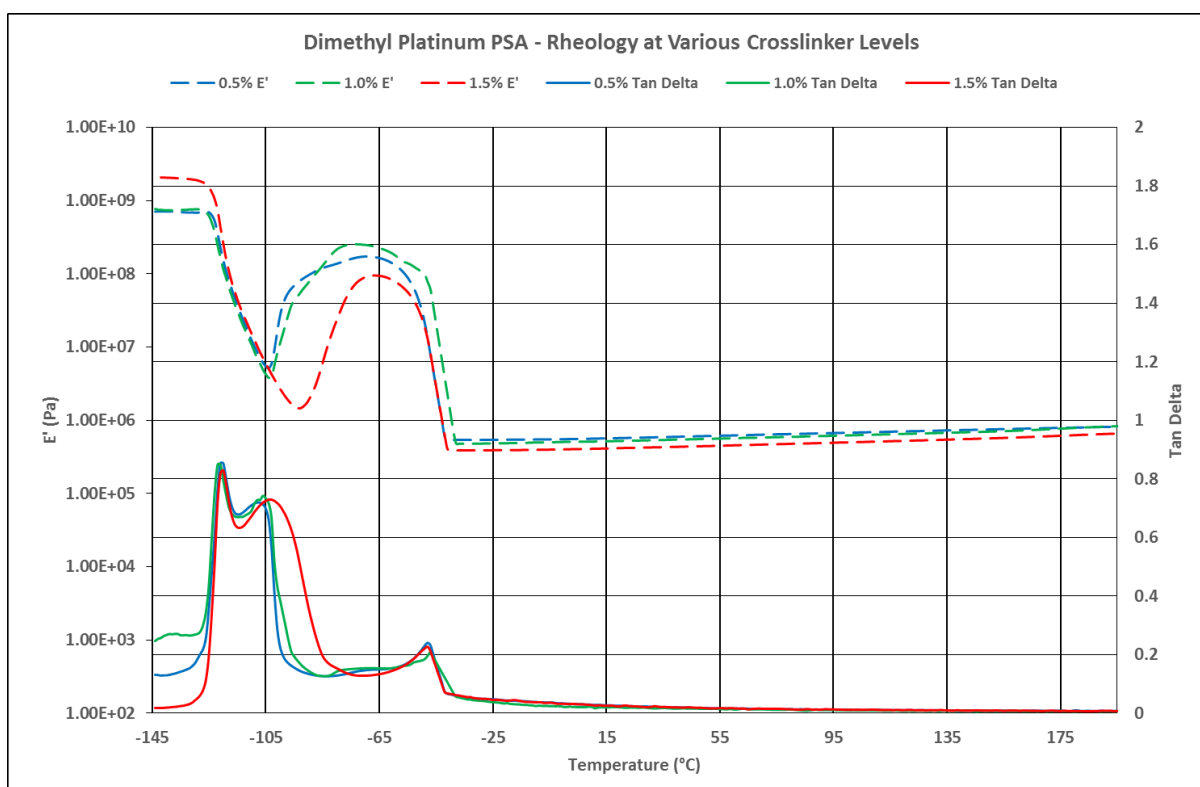


Figure 14. E' and tan delta curves for the temperature ramp testing of the Dimethyl Platinum PSA.

Table 13. Selected rheology values for the Dimethyl Platinum PSA.

XL (%)	T _g (°C)	Tan Delta at T _g
0.5	-121	0.854
1.0	-122	0.849
1.5	-120	0.829

As opposed to the peroxide cured systems, the Dimethyl Platinum PSA does not show appreciable variation when changing crosslinker levels. The vinyl functionality available on the polymer portion of the PSA is limited and therefore adding the additional SiH containing crosslinker did change the elastic modulus significantly above -25°C.

4.0 Summary

Increasing crosslink density through peroxide addition provides a silicone PSA with higher temperature stability for applications such as heat seal and plating tapes. Applications that would require more tack and adhesion from the same PSA, such as splicing tape, may benefit from a lower peroxide loading. Decreasing the amount of peroxide also has the effect of lowering the storage modulus of the PSA allowing for more flow and subsequent wetting of the PSA on low energy substrates. PSAs which utilize a dimethyldiphenylsiloxane co-polymer can tolerate higher temperatures when compared to traditional dimethylsiloxane based materials. The performance benefits would lend themselves to applications such as plasma, high velocity oxygen fuel (HVOF), and other high temperature masking tapes.

Whereas the crosslink density of the peroxide catalyzed PSAs can be modified through incremental catalyst addition, the platinum catalyzed PSAs are more limited due to the dependence on the availability of vinyl and hydride functionality on the polymers used in the formulation. The low adhesion and tack performance of these PSAs make them ideal for use in applications such as electronic device protective films where fast wetting along with low and stable adhesion are desired.

As demonstrated in this study, the rheology and performance of a peroxide catalyzed PSA can be modified by the extent of crosslinking introduced by variations in the catalyst levels. Alternatively, the rheological profiles of a PSA can be more aggressively targeted to meet the needs of a specific application through the use of PSA additives.

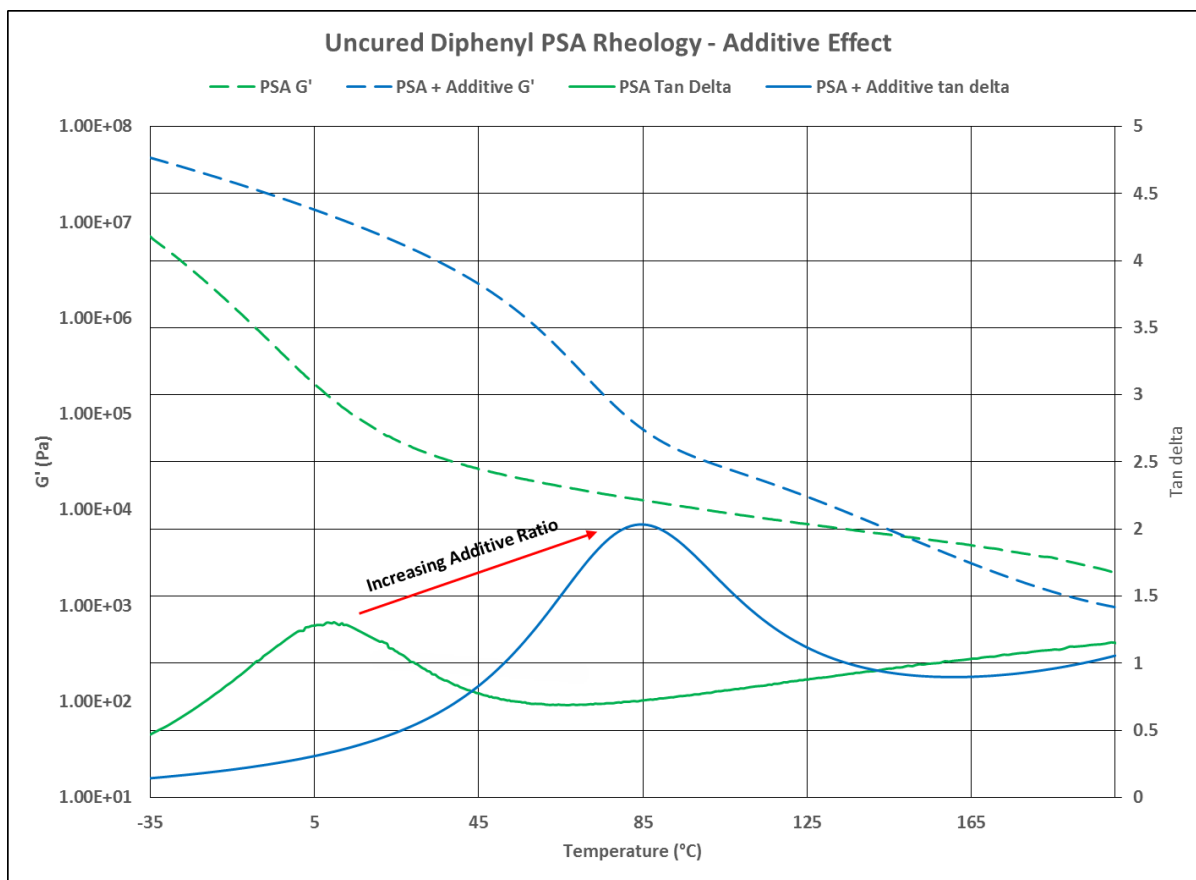


Figure 15. The rheological impact of adding a modifier to the Diphenyl Peroxide PSA.

Figure 15 shows an example of a Diphenyl PSA before and after the addition of a modifier. The effect of the addition is the concurrent increase in the glass transition temperature and the tan delta peak. The higher tan delta peak is an indicator of the energy dissipation potential of the system that could be used to tune vibration dampening.

Understanding the relationships between crosslink density, adhesion, tack, release, and rheology to the end application will give the technologist the tools to make better material selection when designing a PSA construction.

5.0 Literature Citations

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6.0 Acknowledgments

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