SLOW AND STEADY – ALTERNATE TEST METHODS TO CONSIDER FOR PSA MATERIALS

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Abstract

Those involved in pressure-sensitive adhesive (PSA) products know that the three critical properties to consider in designing products or in trouble-shooting issues are peel, tack and shear (cohesion). Most of the time, tests are performed at well-known industry standard speeds, such as peel strength at 12 inches(300 mm)/minute. Shear strength is most often tested by hanging large weights on small applied areas to initiate shear failure. However, there are some cases where products are expected to withstand stresses, perhaps very small stresses over a long period of time, where standard speeds or static methods may not define performance very well. In these cases, using small stresses (low weight or force) or very slow rates (0.05 inches/minute) can reveal differences in product performance or potential causes for issues. This paper will show how slow-rate or creep methods can be very valuable in preventing downstream problems during product design, or in solving difficult performance problems.

Background

We first need to review the basis for pressure-sensitive adhesive performance. Figure 1 shows a spring and dashpot according to the Maxwell analogy shows both elastic and viscous components in a viscoelastic material, such as a PSA.

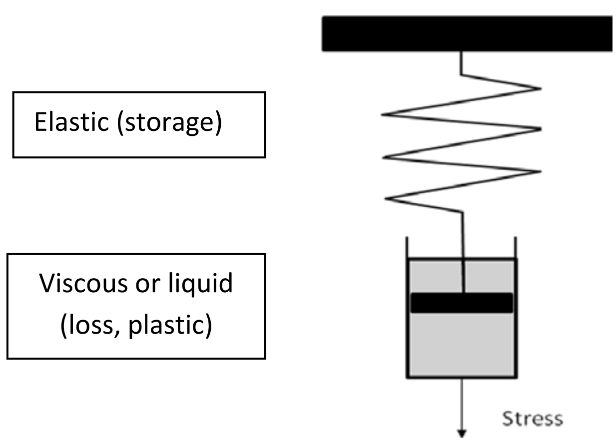

Figure 1. Maxwell Analogy

In this analogy, the dashpot in series with the spring demonstrates stress relaxation.

Figure 2 shows the Kelvin-Voigt analogy below shows the viscous component in series with the elastic component, demonstrating retardation of the spring extension with applied stress:

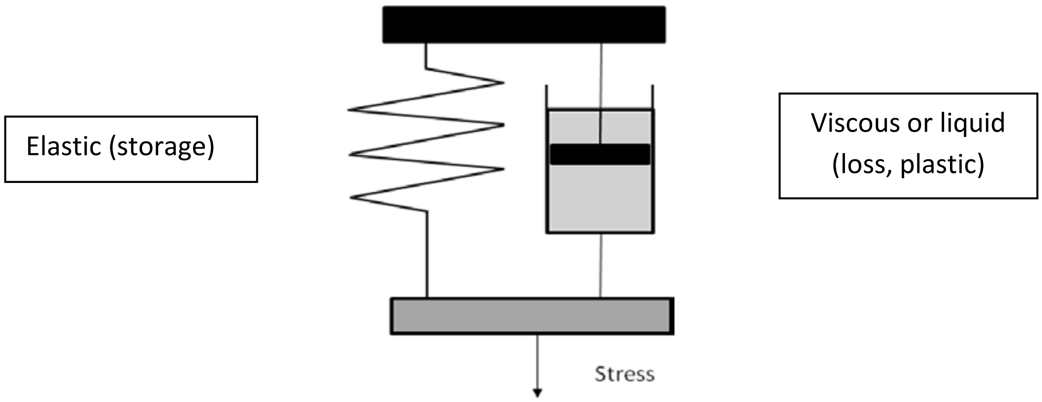

Figure 2. Kelvin-Voigt Analogy

With a pressure sensitive adhesive, the molecular weight distribution and/or the blend of ingredients in a formulated adhesive give a broad range of springs and dashpots, displaying both stress relaxation and retardation during use. Most testing focuses on the strength of the springs, while the slow moving dashpots often are overlooked [1].

For a material to display pressure-sensitive adhesive properties, it must first have enough of a viscous (liquid) component to show deformability and gain adhesion to the applied surface [2]. Once this is established and bonds are formed to gain adhesion, the question becomes how well the adhesion has developed. Here the viscous and elastic components of a PSA contribute in different ways, depending on the property of importance, time after application and exposure conditions.

PSA properties are time and temperature dependent. The rate of adhesive deformation in attempting to debond a PSA from a surface determines the contribution of the viscous and elastic components. A rapid rate of deformation or debonding (correlating to low temperature) mainly brings in to play the elastic portion of an adhesive, while slow rates of deformation (correlating to high temperature), after extending the polymer chains, bring the viscous portion into play [1].

In some applications, the immediate tack and adhesion properties of the PSA are most important. For example, a PSA label applied to bottles at 200 bottles/minute needs to rapidly develop adhesion, granted under a relatively light load, on a low surface energy plastic. For this type of application, it is critical to develop an adequate bond within a short time before the bottle proceeds down the line. An application with a similar type of immediate tack/adhesion need is corrugated packaging tape. Corrugated containers automatically sealed with packaging tape may be subjected to flap holding stresses immediately on closure. For these situations, physical property tests immediately after application or with short residence times are of critical importance, often in conjunction with slower rate/longer dwell time tests.

In other applications, longer rates of deformation, possibly under very light loads per unit area, are of more importance. Mounting and bonding applications may have to exhibit good immediate tack for mounting shelves or dispensers on walls , but the difference is in the expectation of maintaining a bond over an extended time without significant slippage. A 12 mm wide mounting tape that fails after slipping 4 mm over a 1 week period has a debonding rate of 300 mm over 756,000 minutes. Testing at 300 mm/minute may be of interest but it is not indicative of the stress/strain conditions of the failure. Even light load failures over a shorter time period, for instance if 50 grams of force applied over a 25 mm. width causes a slippage of 4 mm. over one day, this translates to a rate of 300 mm./10,800 minutes under a force per unit width not normally used in a standard peel adhesion test for PSA's.

Pressure Sensitive Adhesive Testing – What we normally do.

Before discussing specific tests, we need to start with why we test in the first place. There are four primary reasons for testing product performance:

- Product Development – creating a new product for a current or new application area. This involves both developing the product and providing technical sales tools for product launch.
- Quality Assurance – making sure the latest lot is compared with previous lots and falls within known control limits and meets end-use performance needs.
- Product change – either a voluntary (cost reduction, second sourcing, etc.) or a forced change (vendor shut down, product pruning, etc.).
- Problem Solving – an end use performance issue that requires immediate attention and involves variables outside of the manufacturing process.

Since most testing is comparative in nature, it is important to have a good performing control on which to establish a base performance level, and then compare candidates or different lots to the control. With problem solving efforts, having both "good" and "bad" performing samples is essential to confirm the problem and aid in establishing a test that can truly differentiate between samples that meet or do not meet requirements.

One common factor in the need for testing is that we are either trying to prevent a failure or investigate and solve a failure. We need to understand why things fail; not just pressure sensitive adhesives, but any product or process [3]:

- Stress – mechanical, chemical, electrical
- Environment – substrate, ambient conditions, outdoor exposure to UV, etc.

- Temperature – higher temperatures accelerate physical changes and chemical processes, such as oxidation
- Time – any of the factors listed above will affect the product or process more as time passes.

For all these situations and failure components, standard test methods are used for at least a starting base. Quality assurance test methods primarily follow standard procedures and test surfaces (typically stainless steel) to provide reliable and effective comparison data across years of production. The most common pressure sensitive adhesive test methods are:

- Peel Adhesion (PSTC-101, ASTM D3330, FINAT FTM-1, AFERA 5001) – these methods cover 180° or 90° peel adhesion, normally tested off stainless steel (or glass for FINAT FTM-1). The tests are normally done with a carrier/adhesive peeled away from a test panel, bringing the carrier stiffness and modulus into the peel value, especially with 180° peel angle. These tests are normally run at 12" (300 mm)/minute. The tests as they are normally run are more of a substrate/adhesive/carrier test than a test of the adhesive alone [4, 5, 6, 7].

- Tack – either loop tack (PSTC-16, ASTM D6195, FINAT FTM-9), probe tack (ASTM D2979) or rolling ball tack (PSTC-6, ASTM D3121) are used for determining the immediate deforming and adhesion of an adhesive to a surface, usually stainless steel. These are very short residence time tests, from possibly less than one second (rolling ball tack) to one second (probe tack) to about 8 seconds (loop tack). Loop tack brings into play the backing stiffness as part of the test result, and especially with firm adhesives such as crosslinked acrylics, may show wide standard deviation because of the small amount of contact time in these tests. Probe tack uses a very small surface area probe (5 mm diameter) for a short residence time. Rolling ball tack is very sensitive to set-up variation and finds the best fit in QA testing for determining significant process and lot formulation issues. Again, these are all very short residence time tests [8, 9, 10, 11, 12, 13].

- Static Shear (ASTM D3654, D6463, PSTC-107) – due to the relative inexpensive equipment and simple set-up and test procedure, this method is the standard for examining the cohesive strength of an adhesive (if a cohesive failure occurs). Though widely used, this method is subject to wider standard deviations in test results, with minor variations in mounting, adhesive surface area, etc., causing more significant error between samples. In a survey of static shear tests on pressure sensitive adhesive tapes on stainless steel (cohesive failure only), we found the following result shown in Table 1, compared to a similar survey on peel adhesion test results:

Table 1. Comparison of One Standard Deviation as a Percent of Mean

Test	Average Sample Deviation	Standard Deviation Range
Static Shear	24.4%	1.3% - 74.1%
Peel Adhesion	6.2%	2.0% - 12.0%

If an adhesive failure occurs, the test becomes more of a holding power test, and even wider variation can be seen. This test is designed to give a single value per test; hang time in minutes [14, 15, 16].

All of these standard tests are designed to provide a performance indicator of the pressure sensitive adhesive in a relatively short period of time. Each method has its limitations, and in the case of tack and peel adhesion tests, are really measuring the elastic properties of the adhesive due to the short dwell times of these tests and the rapid rate of testing as they are normally performed. Still, these methods are valuable for QA testing to compare to previous runs and to be able to share information with suppliers, customers, etc.

Product change and product development involves both standard methods and modified methods customized to specific substrates, exposure conditions and dwell times to provide both a standard evaluation (same language between supplier, coater and end-user) and an application specific evaluation of performance based on end-use needs. Problem solving needs to focus on application specific surfaces, to take into account the surface energy of the substrate, surface roughness, etc., and application and exposure conditions in the interest of understanding cause and potential solutions for the problem as quickly as possible.

To adequately investigate problems or to do a good job of product development, we must venture into alternative methods and be creative in how we approach testing. Most failures occur over relatively long periods of time, and are significantly influenced by the viscous component of adhesives. We need to focus on this component to adequately understand product performance and to solve adhesion problems that occur over long dwell times. Standard methods in peel adhesion or shear strength, modified in load, peel rate, surface or test conditions, can be useful in accurately representing end-use conditions. Other test methods, primarily created for specific end-use circumstances, can be of equal value [17].

Review of Alternative Test Methods

This discussion will focus on possible alternative test methods, as opposed to the more standard test methods across the PSA industries.

- **Dynamic Shear (FINAT FTM-18, ASTM D1002) [18, 19]**

Known as lap shear in the adhesive and sealants industry, it has been shown in many studies to be useful in testing the behavior of PSA’s and other adhesives under relatively slow deformation. While this test is often used to generate a single point (peak strength), the information gathered goes beyond a single point result from a static shear test (hang time in minutes until failure), to give a graphical representation of the deformation of the adhesive layer from the application of initial stress to the point of irreversible failure of the adhesive layer (complete disentanglement of the polymer structure). This type of longer deformation rate test has been shown to accurately depict shear or tensile failure behavior in a more timely fashion than static shear tests [20, 21]. A diagram of test configurations is shown in Figure 3 below:

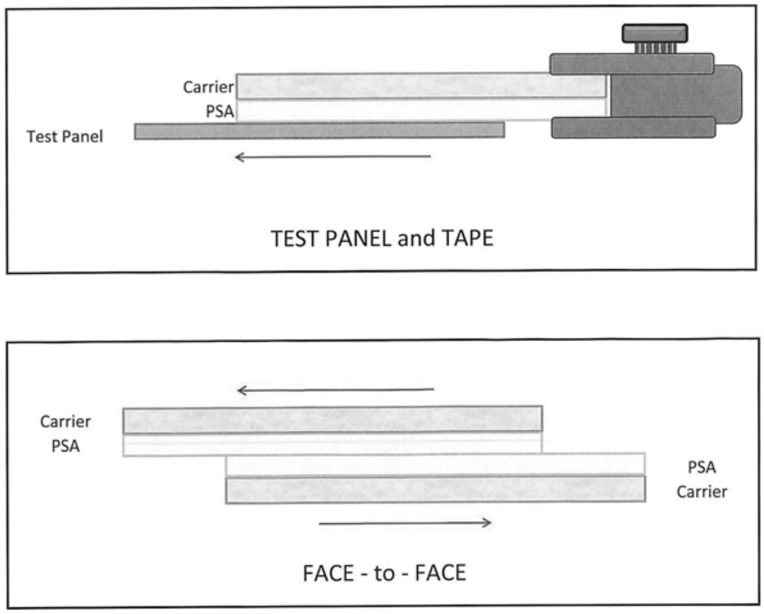

Figure 3. Two Possible Sample Configurations for a Dynamic Shear Test

In the adhesives and sealants industry, the bond dimensions are normally 1 in. (25mm.) wide by 1/2" overlap [19]. For PSA applications, an adjustment to the dimensions may be desired. In trying to replicate the frontal area of a progressing debonding zone of a PSA tape, a wide but short area may be more suitable, such as 25 mm. wide by 6 mm. long [17]. This wide width, narrow applied length is better suited for concentrating on the adhesive and trying to remove backing effects. In all these configurations, the idea is to slowly pull apart the adhesive joint area to reveal its ability to withstand forces over an extended period of time to better understand the long term properties of an adhesive without waiting too long for results. A graph of a dynamic shear result is shown in Figure 4 below:

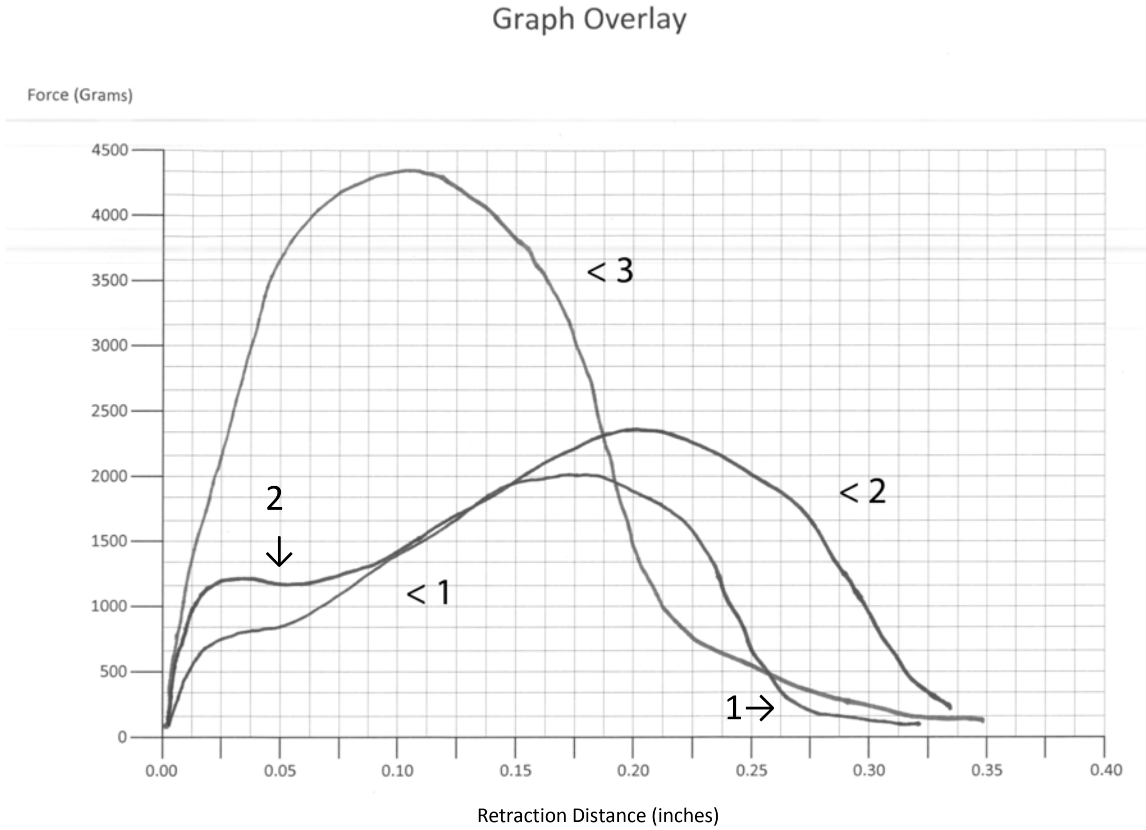

Figure 4. Dynamic Shear Results on 3 Tape Samples

Samples 1 and 2 are general purpose reinforced tapes showing a low slope to peak, a lower peak and total work compared to sample 3, a high performance duct tape.

Figure 5 is a sketch of how the polymer chains are stretched, disentangle and eventually the adhesive layer pulls apart during a dynamic shear test:

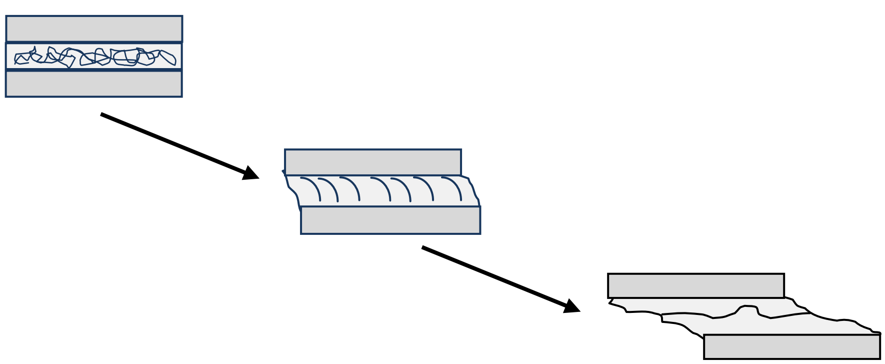

Figure 5. Depiction of Progression of Strain in Dynamic Shear Test

- **Modified Static Shear (ASTM D3654, D6463, PSTC-107)**

While this is a normal test in the tape industry using stainless steel or NIST 1810a Linerboard, this test does fall into the category of relatively slow speed and is meant to test the liquid character of an adhesive. Depending on the end-use conditions, this test can be modified very easily for substrate, test conditions and load, to simulate specific application conditions [14, 15, 16].

One of these modifications is the Shear Adhesion Failure Test (SAFT, ASTM D 4498). Samples are mounted in the same way, but testing is done in an oven that is programmed to ramp up in temperature at a rate of 0.5° C per minute [22]. Hang time is then correlated to the temperature based on the ramp rate. The standard method is designed to indicate the relative ability of an adhesive to withstand temperature and maintain holding power. This method does bring out the liquid behavior of an adhesive, using heat to accelerate the physical flow of the adhesive. Table 2 displays an example of testing done on three tape samples, comparing peel, static shear and SAFT test results:

Table 2. Comparison of 3 Tape Samples

	A				B				C			
	AVG.	σ	n	MOF	AVG.	σ	n	MOF	AVG.	σ	n	MOF
Peel Adhesion on Stainless, ASTM D 3330, lb./in.												
90°, initial	***10.9***	0.5	4	A9T1, LG2	2.7	0.07	5	A, GH3	***4.0***	0.2	5	A
90°, 7 day dwell	***12.8***	0.3	5	T9C1, LG3	3.4	0.05	5	A, GH3	***5.5***	0.2	5	A, GH2
180°, initial	***10.4***	0.9	4	A9T1, LG2	3.3	0.05	5	A, GH3	***3.3***	0.1	4	A
180°, 7 day dwell	***12.0***	0.5	4	C, LG3	6.4	0.08	5	CD	***5.9***	0.3	4	A, GH3
Shear Resistance on Stainless, ASTM D 3654A, minutes												
initial	***63.3***	9.9	5	C	26.3	2.2	5	C	***47.6***	13.7	5	C
Shear Adhesion Failure Temperature (SAFT), ASTM D 4498, °C												
	67.1	1.0	5	C	54.4	0.9	4	C	***121.9***	3.5	4	C

Modes of Failure

AVG. - the average value of the replicates, σ - standard deviation, n - number of replicates, MOF - mode of failure
Numbers 1 to 9 = %, as A9T1 is a 90% clean peel with 10% transfer of the adhesive to the substrate.
A - adhesive failure - the adhesive was removed from the substrate cleanly.
C - cohesive failure - the adhesive split, leaving residue on both the face stock and substrate.
T - adhesive transfer - the adhesive transferred from the face stock to the substrate. Usually attributed to poor anchorage.
Numbers 1 to 3 1 = slight 2 = moderate 3 = severe
GH - ghosting - a shadow or stain remained on the substrate.
LG - legging - the condition of a soft adhesive when strings or legs are formed when it is pulled.

If one compared only the standard peel and shear data for Samples A and C, the conclusion might be that Sample A would perform better in general applications. However, if the application was subject to higher temperatures, even exposure to 60° C for significant time, Sample A may not be as good a fit as Sample C, as shown in the SAFT results.

- **90° Static Peel (PSTC-14)**

The standard version of this test for packaging tape applied to corrugated boxes is PSTC-14, with the test geometry shown in Figure 6 below [23]:

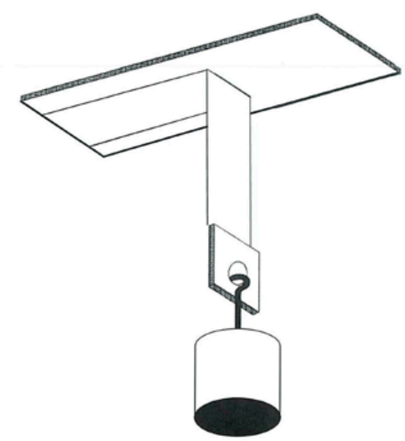

Figure 6. Diagram of 90° Inverted Peel Set-Up

This test can be used to study the low load effects in applications requiring long term holding power, by isolating to the tensile properties of the adhesive under light loads. This is important in applications where a tape applied with some built-in stress can show edge curl, lifting or flagging over time. Examples are packaging tape, athletic tape and wire bundling tape.

This test operates under the premise that over time, even a very light load, 25 grams or less, will cause even an aggressive tape to debond [24]. If a tape shows a peel value of 2 lbs./inch width at 12 inches/minute, this means if a 2 lb. weight was hung in the configuration above, it would peel at a rate of 12 inches/minute. Going down in weight will cause the peel rate to slow, but the tape will still peel from the surface. A slow strain on the adhesive focuses on the viscous or liquid component of the PSA.

- **Butt Tensile [17, 24]**

When testing peel adhesion, the angle of peel determines which type of stress is applied to the adhesive. 90° Peel tests the tensile modulus while all other angles of peel are a blend of tensile and shear modulus. To test the liquid properties of the adhesive in a slow deformation, we must keep the adhesive test area a constant, such as in shear testing. The issue with gathering tensile modulus information in a standard 90° Peel test is that the area being tested is constantly changing as the peel front progresses. In order to clearly understand the tensile behavior under slow strain, we must change the geometry of the test to use a set area of adhesive.

The Butt Tensile configuration is described in Figure 7:

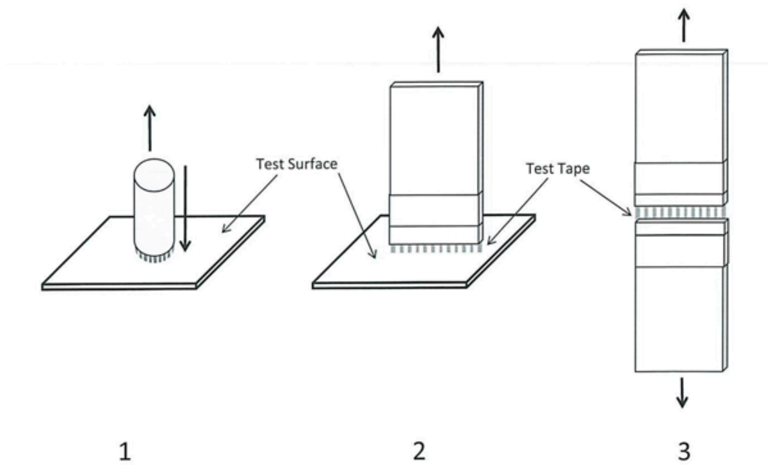

Figure 7. Three Test Configurations for Butt Tensile Tests

Configuration 1 shows a metal probe brought into contact with the adhesive surface on a flat plate. Configuration 2 shows a rectangular, flat metal probe with the adhesive tape wrapped around the panel and secured, then brought into contact with a test surface. Configuration 3 shows two rectangular, flat face panels with adhesive tape wrapped around both panels, brought together adhesive-to-adhesive. Any of these can work, with the type of tape, coat weight of adhesive and other factors determining the best method of testing.

With all these configurations, the method is to bring the surfaces together for a set time and pressure, to allow the adhesive and test surface (or other layer of adhesive) to deform and bond well. The test is done at a very slow rate, preferably 0.01 – 0.02 inches/minute. As the adhesive is put under strain, visible tendrils are formed as the polymer chains are pulled. A photo of an adhesive under test is shown in Figure 8:

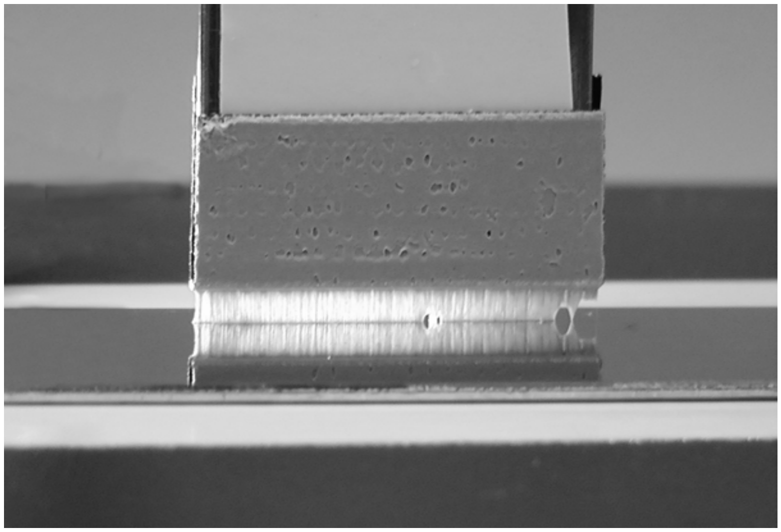

Figure 8. Heavy Duty Tape in a Butt Tensile Test, Adhesive to Steel

An example of a butt tensile graph is shown in Figure 9:

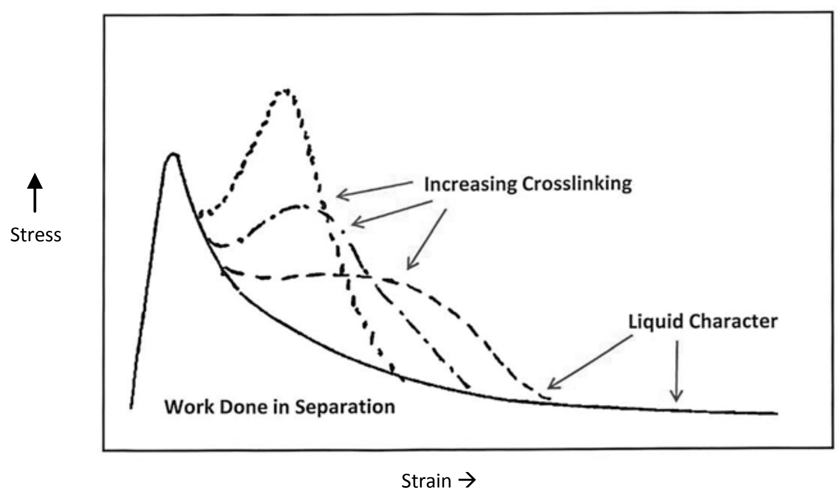

Figure 9. Details of a Butt Tensile Graph [17]

The initial peak and the sharpness of the initial slope indicate more of the elastic component of the adhesive. The area under the curve gives the total work done in separating and stretching the adhesive layer. The length of the tail indicates the liquid component of the adhesive. When crosslinking is present, a secondary peak will appear, getting more obvious and predominant with increased crosslinking.

The butt tensile test gives a graphical result with much more information than the single point determined by the standard static shear test. Degree of crosslinking is shown by the presence of a second peak (in most cases) and a higher peak value with less of a tail (less liquid character after the peak). Softer adhesives tend to show a longer tail and lower peak.

Figure 10 shows a comparison of a general purpose, heavy duty tape (1) and a high performance duct tape (2), with sample 2 showing a second peak, indicative of crosslinking.

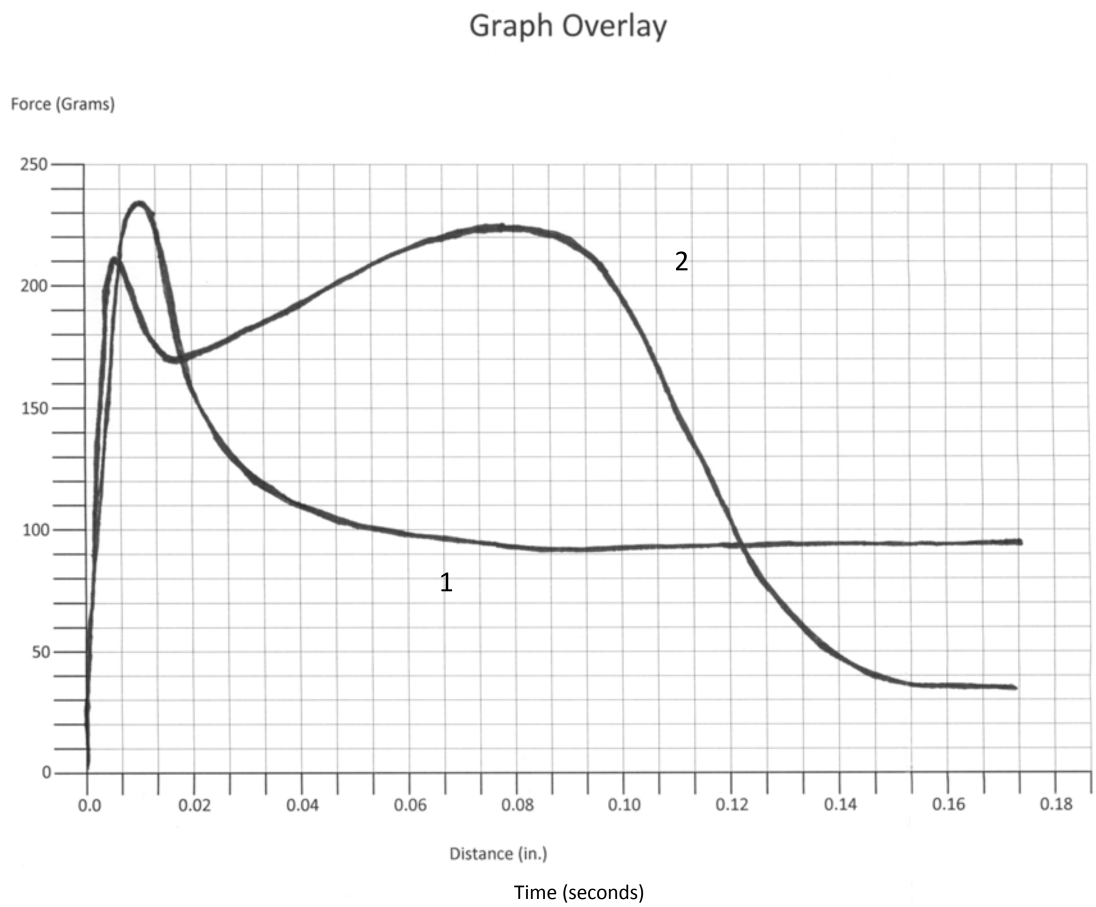

Figure 10. Butt Tensile Graph of Two Tapes

Practical Use of Alternate Methods

Here are some case studies from past projects that involved low load/slow speed type of tests. Some application details and product information have been omitted to demonstrate the value of these methods without revealing confidential information.

- **Example 1**

In this case a modified static shear test was used. Although this did not involve a pressure sensitive adhesive tape, this case still shows how low load/slow speed tests can be of value.

A manufacturer of reinforced plastic grinding pads was experiencing product failures in end-use, characterized by an internal bond failure between a felt layer and a reinforced plastic part. Standard adhesion tests in the bond area at the customer did not show differences between "good" and "bad" parts, and also did not replicate the mode of failure seen in the field (showed internal failure within the felt instead of felt adhesive failure from the plastic surface seen in end-use). Good and bad parts were sent for the purpose of developing a quality assurance test method capable of reproducing failure mode in the bad parts yet showing a differential in the test from good parts.

The stress applied to the product in end-use was shear in nature, and the part was subject to temperature increase due to frictional heating. We first tried a standard SAFT test on laminated samples in a lap shear configuration. Results are in Table 3:

Table 3. SAFT Results on Lot Samples

Shear Adhesion Fail Temperature		
½" x ½" x 500 grams, **fail at °F**		
Sample ID	Avg.	MOF
Good Lot 1	197.8	A
Good Lot 2	199.6	A
Good Lot 3	194.9	A
Failure Lot	173.8	A

This test did show some differential between known failure and known good performing lots. This test replicated the field mode of failure, indicating this type of test may work, but some adjustments to temperature and possibly the weight used may help to better differentiate "good" from "bad" performing lots.

A screening test was done on temperature, keeping the temperature constant, preheating the oven and inserting the samples in the configuration shown in Figure 11:

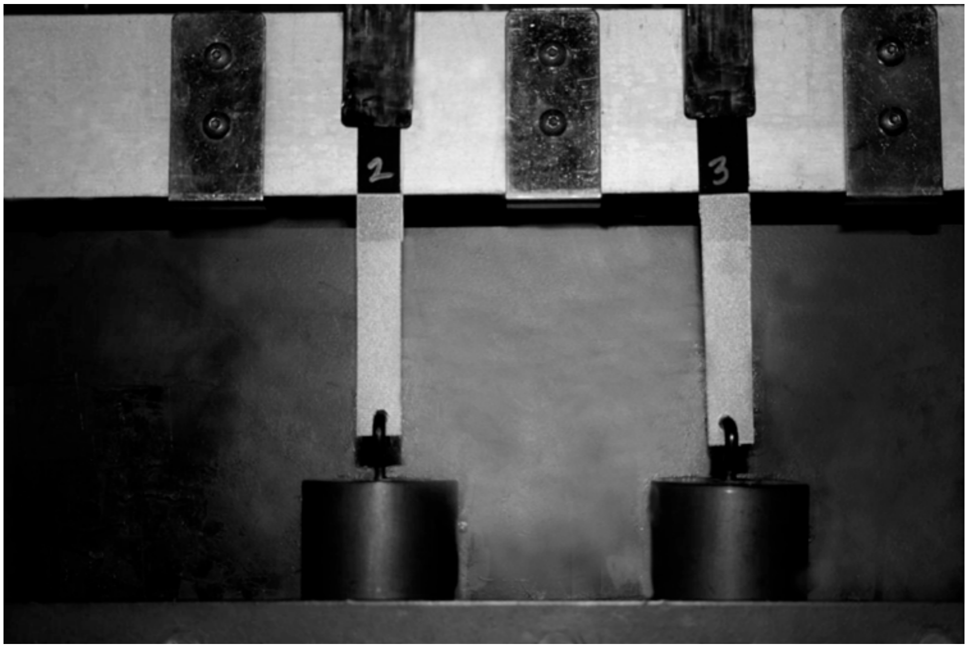

Figure 11. Heated Shear Test

Results from this test are shown in Table 4:

Table 4. Heated Shear Test Results

	Heated Shear (ASTM D 3654 method H)					
	½" x ½" x 500 grams, **minutes to fail**					
	165°		170°		175°	
Sample ID	Avg.	MOF	Avg.	MOF	Avg.	MOF
Good Lot 1	+1440	No Slip	+1470	No Slip	55	A
Good Lot 2	+1440	No Slip	+1470	No Slip	50	A
Good Lot 3	+1440	No Slip	+1470	No Slip	61	A
Failure Lot 1	621	A	96	A	7	A
Failure Lot 2	+1440	No Slip	+1440	No Slip	9	A

This test, run at 175° F, was found to be meet the customer's needs of a fairly quick test (in process QA), inexpensive equipment and set-up and a clear differential between suitable and failure product.

- **Example 2**

Following is a case of an industrial cloth tape where a current product was displaying significant flagging in as little as 4 hours after application. Adhesive formulation adjustments were made to increase adhesion. This did improve performance, as shown with the reduced flagging of Sample “B” in Figure 12, using a modified ASTM D1000 flagging test [25]:

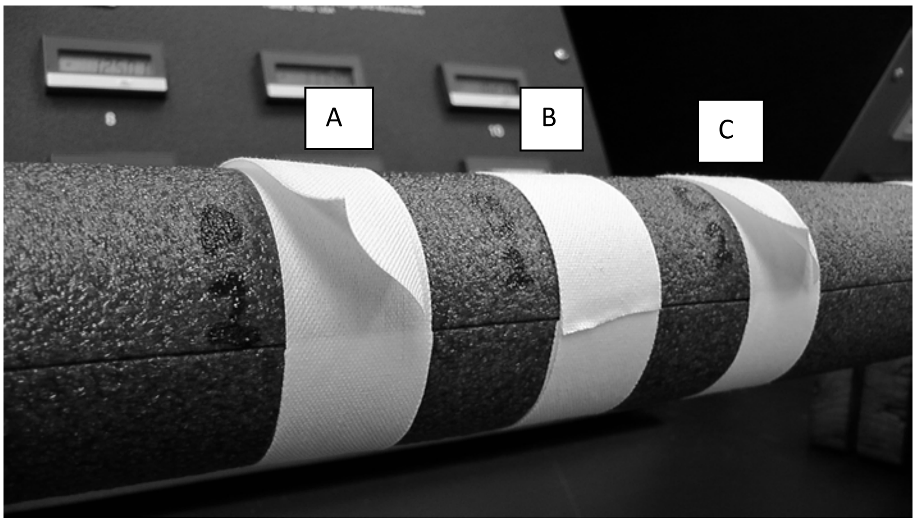

Figure 12. Flagging Test on 3 Cloth Tape Samples, 4 hrs. applied

The test was modified by using foam pipe insulation around a PVC pipe to provide some hoop stress. 1000 gram weights were used for winding. This was found to better replicate the end-use, where the tape was wrapped around a compressible material under tension.

The 90^o Static Peel test was done on the three industrial cloth tape samples, using 25 gram weights, with results listed in Table 5:

Table 5. 90^o Static Peel Results

Sample	Average Hang Time (Minutes)
A	6.4
B	24.9
C	8.5

This test was found to be suitable for a numeric test, and gave a clear difference between known failure samples and one showing some reduction in flagging. The customer decided there was still work to be done to improve flagging resistance, and now they had two tests to help guide their work.

The 90° inverted static peel test is fairly easy to set up and requires minimal capital investment. As in other PSA physical property tests, it does require checking the test angle, good sample preparation and mounting techniques.

- **Example 3**

A paper packaging manufacturer was experiencing sporadic seam failures, especially in warm weather storage after packages were filled. The manufacturer wanted to determine the cause for the failures, and to test possible candidate adhesives that might offer improved performance.

Failures occurred within 1 to 2 days after processing and storage. Based on an analysis of the package contents and failure samples, this appeared to predominantly be an adhesion failure from the laminated side of the paper (adhesive was coated on one side, with the other seam side pressed together to form the seal). Further testing and examination of adhesive properties indicated that the current adhesive may not have enough flow (liquid character) to consistently flow into the fibrous surface and bond well enough. Candidate adhesives were obtained that showed promise of being able to flow into the substrate better (softer, lower cohesive strength) and displayed better adhesion results (more paper tear) in standard tests. In evaluating the candidates, a test was needed that simulated the stress on the seam, used the actual substrate and could demonstrate that we were not giving up too much in the cohesive strength area with the softer/higher adhesion candidates.

Looking at the way the packages were stored and the package appearance after filling with contents, we decided the stress on the seam was primarily cleavage in nature, as illustrated in Figure 13:

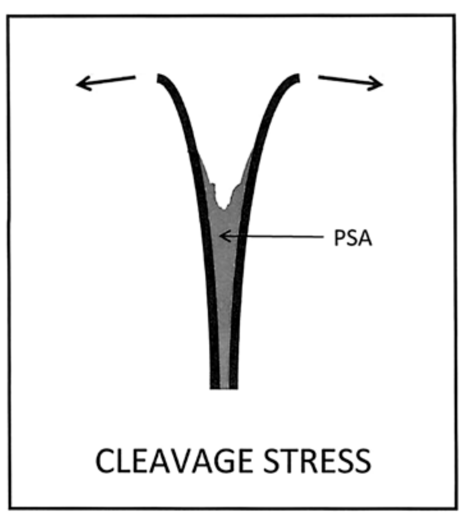

Figure 13. Depiction of Stress on the Package Seam

We also knew that the forces acting on the seams ranged from a few grams/25 mm. bondline width, estimated to be as high as 300 grams/25 mm. bondline width. We also knew that failure rates were low (less than 10%), even in worst case storage conditions (temperatures as high as 120° F/50° C).

We decided on using a Static "T" Peel test at 120° F, using very light shear weights. The test configuration is shown Figure 14:

Figure 14. Static T-Peel Test Configuration

We used the current adhesive as the basis for method development, stating a requirement that the weight chosen must show the current adhesive to have at least 24 hours of hang time before failure. Also, we should be seeing adhesive failures from the laminated side to make sure we were replicating end-use failure mode. This assumption was based on the low failure rate of the current adhesive and information gathered from reported problems. With that, a weight study was done on the current adhesive and one of the submitted candidates with higher peel adhesion, with the results found in Table 6.

Table 6. Weight vs. Hang Time Screening Test

Elapsed Time	Candidate A			Current Adhesive		
	25 gr.	50 gr	75 gr	25 gr.	50 gr	75 gr
1 hour						
2 hours		All failed	All failed			All failed
3 hours						
4 hours	60% failed				20% failed	
5 hours	40% failed				60% failed	
6 hours						
24 hours				***All Pass***	20% Pass	

The failures on the current adhesive were 95% adhesive failure from the laminated side, meaning we were replicating the actual mode of failure. The failure mode on the candidate adhesive was all cohesive, indicating that while showing higher adhesion in bond tests, this candidate was probably not going to help reduce/eliminate seam failures.

Using this test with 25 gram weights, more candidates were screened with the results shown in Table 7:

Table 7. Static T-Peel Test Results, 120^{o} F

Time	Current	A	B	C	D	E	F	G	H
1 hour		All Fail			10% fail		All Fail	70% Fail	
3 hours					10% Fail				
5 hours					10% Fail				15% Fail
8 hours					10% Fail			30% Fail	15% Fail
24 hours	***All Pass***		***All Pass***	***All Pass***	30% Fail 30% Pass	***All Pass***			30% Fail 30% Pass

We tested T-Peel adhesion of these candidates with the results shown in Table 8:

Table 8. Comparison of Candidates, T-Peel and 120° F Static T-Peel Results

Sample	T-Peel, 4 inches/minute, pounds/inch width)	120° F Static T-Peel
Current	3.4 (50% CF/50% PT)	Pass
A	2.6 (80% PT/20% CF)	Fail
B	***2.0 (100% PT)***	***Pass***
C	***3.0 (100% PT)***	***Pass***
D	2.2 (100% PT)	Fail
E	***2.7 (100% PT)***	***Pass***
F	2.5 (100% PT)	Fail
G	Not Tested	Fail
H	2.1 (100% PT)	Fail

The three highlighted candidates were chosen based on passing the heated static T-Peel tests and showing 100% Paper Tear in T-Peel adhesion tests. We decided the combination of these properties would provide an adhesive that was soft enough under application conditions to flow into the paper and develop a strong bond (paper tear), without being too soft and causing seam failure under cleavage stress in end-use.

- **Example 4**

This was a study done on a cross-linkable acrylic adhesive, to compare an alternative test method (dynamic shear) to standard methods, at five different cure levels.

The adhesive was formulated, then cast for a dry caliper of 1 mil on release liner, then laminated to 1 mil Polyester for testing. Standard test results are summarized in Table 9 below:

Table 9. Standard PSA Test Results on Cure Levels 1 through 5

Sample	180° Peel, < 1 min. (oz./inch)	MOF	180° Peel, 24 hr. (oz./inch)	MOF	Static Shear (min).	MOF	Probe Tack (grams)
1	37	AF	40	AF	25	CF	220
2	36	AF	40	AF	36	CF/AF	150
3	31	AF, Zippery	37	AF, Zippery	487	AF	89
4	36	AF, Zippery	33	AF, Zippery	1078	AF	126
5	38	AF, Zippery	31	AF, Zippery	975	AF	128

Figure 15 is a dynamic shear graph of the five cure levels:

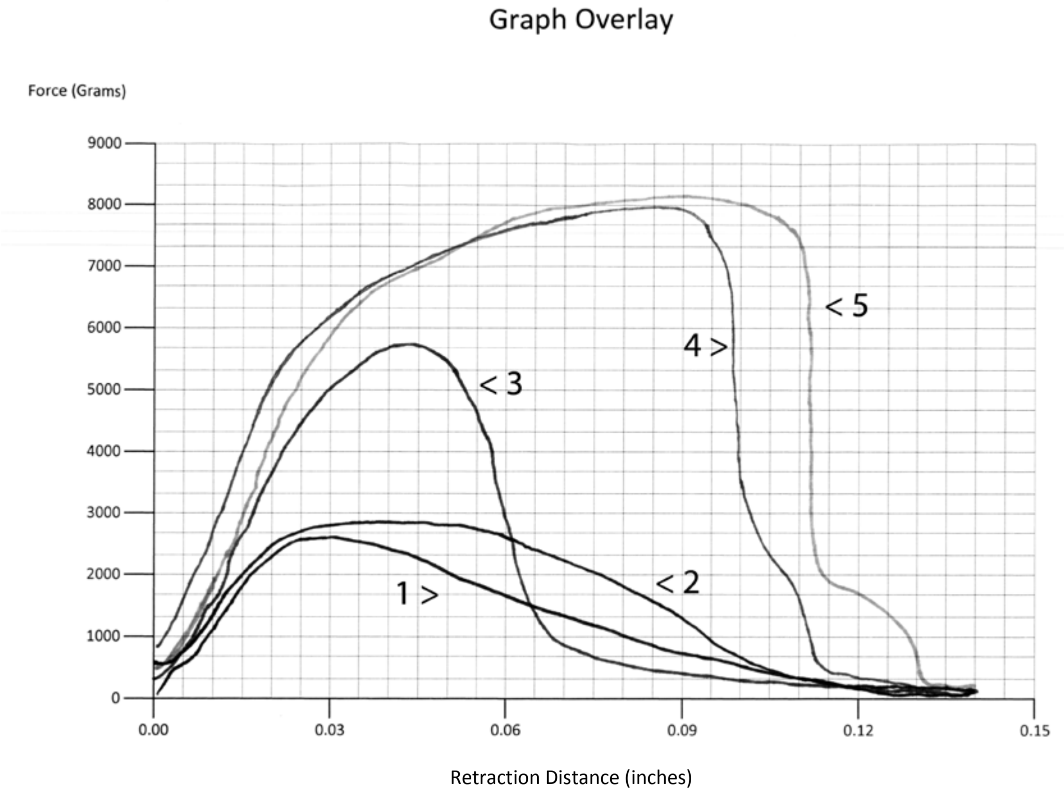

Figure 15. Dynamic Shear Results on Five Different Cure Levels

The level of crosslinking increases from samples 1 through 5. Notice as the crosslinking increases, the peak height increases, the slope to the peak increases and the total work under the curve before the peak increases. This graph also indicates there was not a large difference in shear behavior between crosslink levels 4 and 5, indicating the effect of increased crosslinking may have reached an effective maximum at or before level 4.

- **Example 5 [3]**

A crosslinked rubber based adhesive coated on a paper backing was showing low tack. Butt Tensile tests were done on 7 lots of retains, including the batch in question. The tests showed good reproducibility, with the classification of curves illustrated in Figure 16:

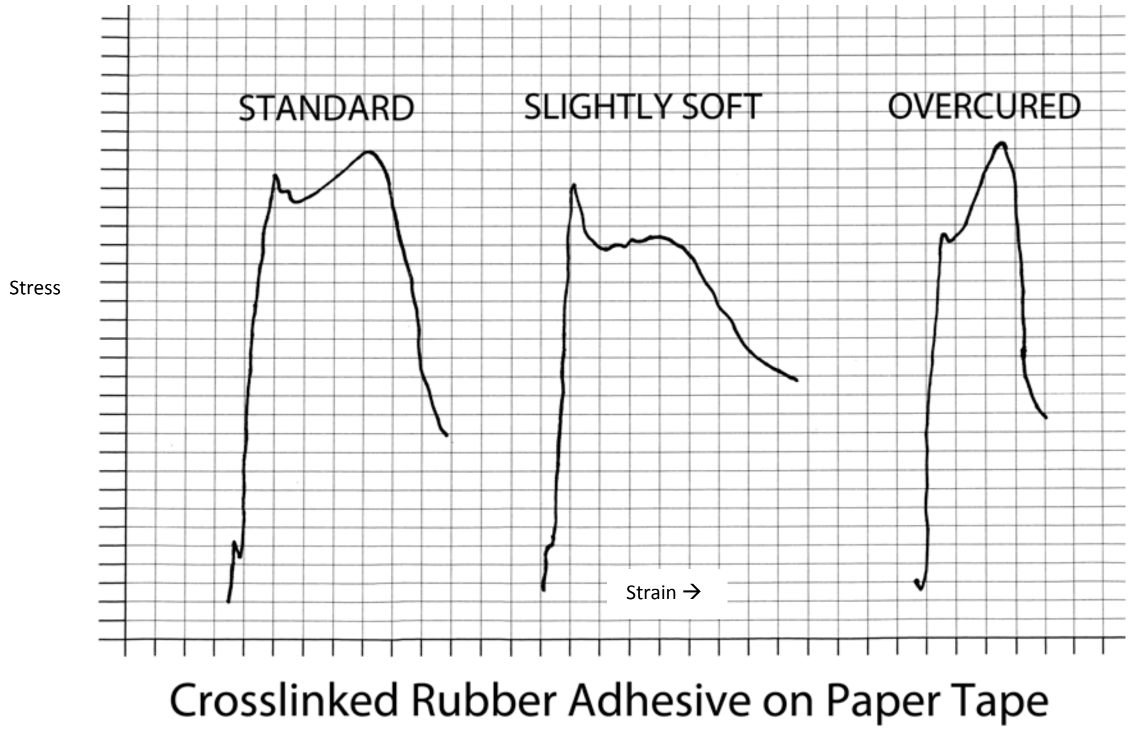

Figure 16. Three Different Butt Tensile Results on Lot Testing

The last batch showed a much stronger second peak, indicating it was overcured compared to the previous batches, leading to lower tack. This low rate of deformation test gave a clear indication of the cause for the problem.

Conclusion

Slow rate and/or low load tests, which allow the liquid character of the adhesive to take a more dominant role in the test, are valuable in predicting performance, solving problems or assuring quality of product. When deciding what tests are needed and how to conduct them, the following factors need to be considered [3]:

- ***Stress*** - What critical stresses are seen in end-use? Tensile, Shear, Combination?
- ***Time*** - If a problem is encountered, what is the rate of failure? Immediate, one day, one week?
- ***Temperature*** - What is the application/exposure temperature? Can a higher temperature be used to accelerate failure or to mimic actual conditions?
- ***Environment*** - What are the conditions encountered in end-use (chemical exposure, humidity).

When deciding what tests to use, be wary of falling into the following traps:

- ***Looking only at one test*** **–** remember the triangle of PSA's. If you only check peel and tack, shear issues may cause problems. Don't just look at one number, like peak strength, when a graph will tell much more.

- ***Looking at a number and missing the mode of failure***.

- ***Testing "standard" but missing the real conditions of failure.***

Technical people tend to use standard methods, but they also need to use creativity in adapting standard methods and creating new tests to accurately replicate end-use conditions in predicting and solving problems, and to assist in functional product design.

Acknowledgements

I would like to thank the following people for their help with this paper:

- John Johnston for his teaching, consultation, coaching and contributions to content.
- Cheryl Saqqa, Physical Testing Project Leader at Chemsultants, for her help with testing and reviewing the paper.
- Don Eppink, Physical Testing Lab Manager at Chemsultants, for his contributions to testing and content.
- Gary Avalon, Chief Operating Officer at Chemsultants, for his coaching and help with reviewing and editing the paper.
- Joe Mausar, Director of Marketing at Chemsultants, for his help with illustrations and content, and for reviewing and editing the paper.

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