A NEW APPROACH TO MEASURING PSA COHESIVE STRENGTH USING A TEXTURE ANALYZER

Lars Kilian, Ph. D., Senior Staff Scientist, Ashland, Inc., Dublin, OH
Charles M. Lewis, Ph. D., Technical Director, Ashland Inc., Dublin, OH

Abstract

Current commonly-used techniques to measure cohesive strength in pressure sensitive adhesives, such as Williams Plasticity and Static Shear (PSTC-107), can be time consuming, highly operator dependent and have poor repeatability. Recently, a stress relaxation technique was investigated to address some of the issues cited above. The technique uses a Texture Analyzer to measure stress relaxation over a short period of time and then a relative force retention (FR) value can be calculated. This technique gives rapid and reproducible measurements of cohesive strength directly from 1 mil PSA-coated stock. These measurements exhibit a strong correlation to Williams Plasticity and shear tests. As a result, Force Retention may be predictive for key performance attributes, such as PSA converting ability and resistance to adhesive edge-ooze.

Force Retention testing is applicable to all forms of coated acrylic PSA, including solvent-based (crosslinked and thermoplastic), emulsion-based, and radiation-cured PSAs. Force Retention allows direct comparisons between the creep resistances of all types of acrylic pressure-sensitive adhesives at the same coating weight. Our current test constraints include a minimum coat weight of 0.7 mil and the need for relatively incompressible facestocks, such as PET and BOPP. The FR technique has already found utility in accelerating product design evaluations by rapidly screening structure-property relationships such as molecular weight and crosslinking sensitivity, additive effects, and process repeatability. This paper will focus on the methodology, benchmarking comparisons and application examples of Force Retention. The most significant advantages of FR relative to shear and Williams Plasticity are increased quality of the measurements and reduced measurement times.

1. Introduction

While the benefits of tack and peel adhesion are readily apparent in PSA performance, achieving and maintaining the necessary cohesive strength is also a key component to enabling PSA-coated tapes to succeed. The optimization of PSA cohesion is critical, since greater cohesion is typically achieved at the expense of either peel adhesion, tack, or both.[1] Achieving consistently sufficient cohesion in the production of both the adhesive and the adhesive-coated article is a prerequisite to avoiding various problems that can occur due to insufficient cohesion.[2]

Static shear[3] is the most broadly used cohesion test that is currently utilized in the PSA industry and is the standard PSTC test method 107.[4] The test consists of bonding a PSA-coated film to stainless steel at a precisely defined contact area, followed by hanging a weight from that film after a defined bonding period. The mean shear failure time of an adhesive is one of the three core attributes most commonly used to characterize PSAs in the tape ands label industries. The other two properties are adhesion to stainless steel and loop tack on stainless steel. Advantages of static shear include moderate predicting ability for cohesive performance and ease of use.

Unfortunately, there are several drawbacks to static shear. Even under optimal sample preparation conditions (machine-coated stock, no defects in the PSA contact area, etc.), the shear test results are often highly variable.[5] The sample contact area is reduced by any air bubbles that can become entrapped during sample bonding. Any contact area reduction will reduce shear failure times. The duration of shear testing can lead to issues in both product development and production quality control. Frequently, shear testing can take days or weeks if too little force relative to the PSA cohesive strength is utilized. Although PSTC-107 typically features a 0.25 sq. in. contact area and a weight of 1.0 kg, numerous other configurations are utilized for adhesives exhibiting higher cohesive strength. Increasing the force / area enables the realistic testing times for high-cohesion PSAs. Unfortunately, it prevents the data from being compared directly to systems utilizing a different contact area and weight configuration.

While the shear stress-based techniques dominate PSA cohesive characterization, there are other capable techniques that are used with less frequency to determine cohesion. Determining the rheology of a PSA via dynamic mechanical analysis is a powerful tool that is utilized to predict all aspects of adhesive performance including adhesion and cohesion.[6,7] Utilizing a temperature sweep of both G' and G", predictions of adhesion can be made via the viscoelastic window technique,[8] and cohesion can be predicted by tracking G' as a function of temperature. Unfortunately, DMA cost and testing time has limited the utilization of this test in the industry. Static lap shear is also used to quantify cohesion of pressure sensitive adhesives.[9] Advantages of this test include relatively short test times and the ability to carry out the test using equipment that is already present in most PSA labs, either Instron or Texture Analyzer.

Williams Plasticity was one of the first tests developed to determine the cohesive properties of elastomeric materials, especially natural rubber.[10] A spherical sample is placed between two plates and a 2 kg force is applied for a specific time in order to determine the creep resistance of the sample. The original test was designed to define the creep resistance of the sample in relation to the viscoelastic recovery by removing the force and measuring recovery.[11] However, Williams Plasticity has also been adapted for use in pressure sensitive adhesives to quantify the compressive creep resistance only.[12] The two types of plasticity tests can be readily differentiated by the units of the results. The original William Plasticity yield dimensionless numbers, while the PSA version of the test provides results in mm of sample thickness. The PSA version of the test, which typically provides results in the range of 1.0 to 6.0 mm sample thickness at the end of testing, provides a good degree of predictive ability for PSA converting ability and end-use cohesive performance.

However, Williams Plasticity also suffers from several shortcomings, some of which are similar to shear and some of which are unique to this test. Like the shear test, Williams Plasticity is impacted by significant variability in the results. This variability is strongly driven by the sample preparation sensitivity of the 2.0 g adhesive ball sample. Experience in plasticity sample preparation improves error, but it remains significant. The test also involves significantly greater labor relative to the shear test due to the effort required to produce the spherical sample. In addition, the test provides a bulk creep resistance measurement. Coated PSA film cannot be tested directly using Williams Plasticity. As a result, plasticity cannot be used to determine important properties such as the relationship between coating weight and the risk of problems relating to creep (edge ooze and halo effects worsen with increasing coating weight), as well as sample behaviors related to sample aging.

Texture Analyzers have been utilized in the PSA industry since the development of the Avery Adhesive test.[13] Although developed for the food industry, this instrument provides an excellent platform for PSA testing. In addition to the initial ball probe tack testy, multiple Texture Analyzer tack tests, based on cylindrical probes, have been utilized to test a large variety of pressure sensitive adhesives).[14-16] In addition, the instrument has been adapted to perform numerous adhesion tests, including temperature profile,[17] 90° peel-wheel,[18] and standard 180° tests.[19] Static lap shear[20], as well as dynamic tensile testing[21] testing of PSA-coated stock has also been carried out using a Texture Analyzer. These are useful techniques for characterizing cohesion, especially the dynamic tensile test, which was shown to correlate highly to static shear. In addition, Texture Analyzers have also been utilized to develop viscoelastic profiles of thick PSA samples.[22]

Ashland has developed a compressive creep resistance technique utilizing a texture analyzer and a cylindrical probe typically utilized for PSA tack testing. The instrument applies a static force to the PSA sample, which responds to the force by diffusing away from the probe contact area. The diffusion decreases the measured force and provides a very facile method to determine creep resistance measurements that correlate linearly with Williams Plasticity.

2. Experimental Methods

PSA-coated film preparation

Static shear and Force Retention were performed on 1 mil dry PSA coated on polyester. Except in cases where adhesive was production-coated, the adhesive-coated stock was produced via the transfer-coating method. The solution acrylic PSA was coated to 90# Kraft Liner (Loparex®) using a Baker Bar. The bar was set to the wet film thickness appropriate for producing 1 mil dry PSA films. Following draw-down, the films were air-dried for 15 minutes prior to forced-air oven drying for 10 minutes at 100 °C. The dry coated PSA films were laminated to 2 mil polyester film after being allowed to cool from oven temperature to room temperature.

Williams Plasticity
Solution-based acrylic PSAs were drawn down onto release liner using a Baker Bar set to a wet film thickness of 10 mil. Following 15-minute air drying and 12-minute oven drying, spherical samples were hand-prepared from these films. Once the spheres were rolled to a mass of 2.0 ± 0.1 g, they were placed between two pieces of release liner. The balls were then placed into the plastometer oven and allowed to equilibrate at 93 °F for a period of 20 minutes. After equilibration, the plastometer applied a force of 2 kg onto the spheres for 14 minutes. The sample thicknesses were measured after 14 minutes to determine the plasticity.

Static Shear
The static shear testing was carried out in accordance with PSTC-107, Procedure A. Stainless steel panels were cleaned using acetone and stored under constant temperature and humidity conditions for 24 hours prior to use. The coated stock (1 mil dry PSA on 2 mil polyester film) was bonded to the panels with a 1” X ½” contact area and allowed to bond for 15 minutes. After conditioning, the panels were hung vertically and a 1 kg weight was attached to the film. The shear failure times were recorded in hours.

Figure 1. Apparatus utilized for Force Retention testing of acrylic PSAs.

Force Retention Test Apparatus

Force Retention testing was performed on a TA.XT2 Texture Analyzer (Texture Technologies Corp., Scarsdale, N.Y./ Stable Micro Systems, Godalming, UK). In order to maintain a constant sample temperature between 32 and 35 °C, a modified Fisher Isotemp Incubator was utilized to enclose the entire TA.XT2. The instrument was controlled using a PC running Texture Expert software, version 1.22 (Stable Micro Systems, Godalming, UK). The entire testing apparatus and a close-up of the sample test area are depicted in Figure 1.

Force Retention Test Method

The PSA coated films (1 mil adhesive on 2 mil polyester) were allowed to equilibrate for at least 2 hours at test temperature (32-35 °C) prior to testing. A strip of coated film measuring approx. 1” X 6” was applied to an indexed mounting plate (Mfg. part no. TA-303) immediately prior to testing to minimize the impact of dust and other surface contaminants. The film and mounting plate were then placed on the instrument.

Identical software settings were used to test all force relaxation samples. Testing was carried out in the HLDD (Force Relaxation) test mode. The instrument lowered the tapered cylindrical probe (Mfg. part no. TA-57R) at a pre-test rate of 0.5 mm/s until a trigger force of 10 g was reached, indicating impingement of the PSA film. At this point, the PC began to acquire force data at a rate of 25 points per second. The probe was lowered at a rate of 0.1 mm/s until a target compressive force of 150 g was reached. The probe was held stationary for a period of four minutes and then retracted at a rate of 1.5 mm/s. Percent force retention was determined by dividing the probe force after four minutes by the initial force.

3. Results and Discussion

Test Principles and Data Analysis

Force retention is designed to provide a facile and practical measurement of the compressive creep resistance of pressure sensitive PSAs. The tapered cylindrical probe utilized in force retention applies a constant force to a small area (< 1.0 mm^2) of pressure sensitive adhesive for four minutes. Due to the relatively high force exerted by the probe tip (~250 psi), the soft pressure sensitive adhesive diffuses rapidly away from the contact area, which lowers the force that the instrument measures. Significant force decay occurs in very soft polymers, which can be due to a combination of very low molecular weight and glass transition temperature. In contrast, some highly crosslinked removable pressure sensitive adhesives exhibit very little force decay.

The force retention technique determines the overall force decay over the testing time, which provides a predictive measurement of the cohesive strength of the PSA. This technique can quantify the relative compressive creep resistance of all types of pressure sensitive adhesives, from non-crosslinked and low molecular weight solution PSA base polymers through highly crosslinked removable PSAs that exhibit almost no peel adhesion.

Due to instrument limitations, the combination of probe speed and target force used in this test resulted in a typical maximum force range of 170-210 grams. However, it was determined over a broad range of PSA types that peak force was not the primary contributor to the differences observed between these creep resistance measurements. Repeated measurements of the same adhesive resulted in similar percent reductions in force and similar shapes of the force decay curves (Figure 2). In order to normalize for variations in the initial force, the percent force retention, defined as the final force divided by the initial force, is the key measurement of this test.

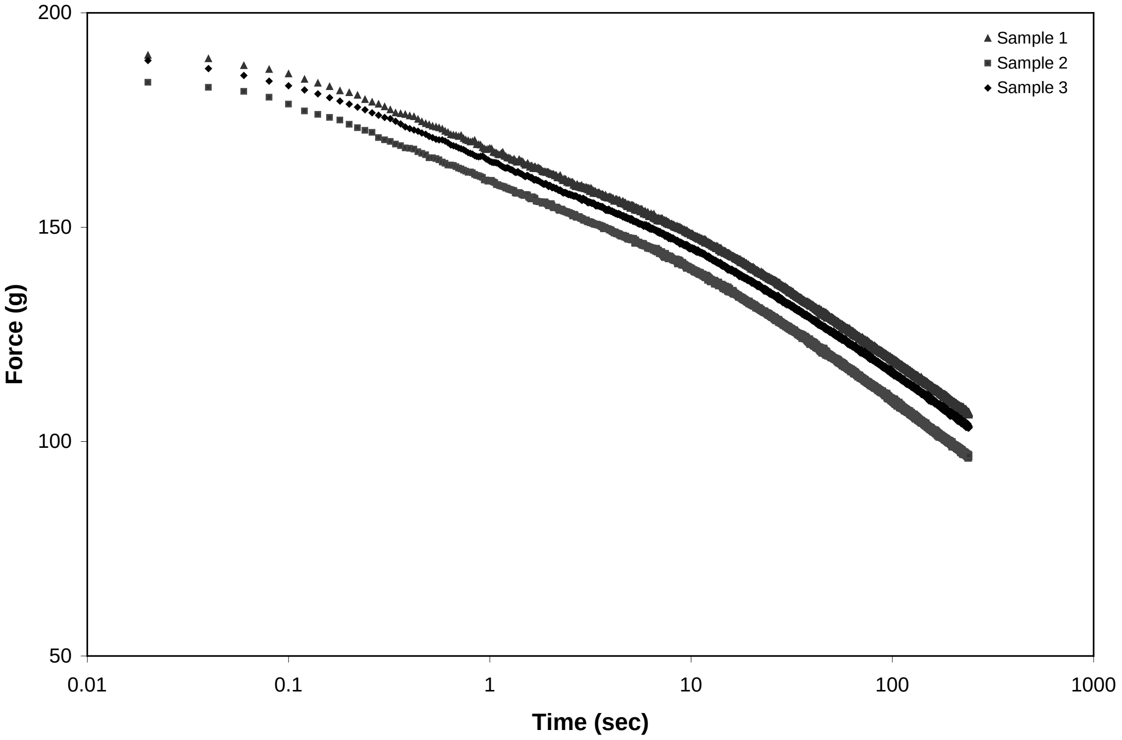

Figure 2. Typical repeatability observed in Force Retention testing of a moderately creep-resistant PSA.

Utilizing identical testing conditions, the Force Retention technique was used to quantify the creep resistance of an extremely broad range of solvent PSAs. In the absence of crosslinking, acrylic polymer creep resistance is driven by both glass transition temperature and molecular weight. Figure 3 shows the drastic impact these two factors have, with force retentions of 13% and 48% measured for two very different uncrosslinked polymers. In crosslinked polymers, the degree of crosslinking becomes the primary driver for the amount of flow under constant force, as evidenced by the results for the moderately and highly crosslinked PSAs in the figure.

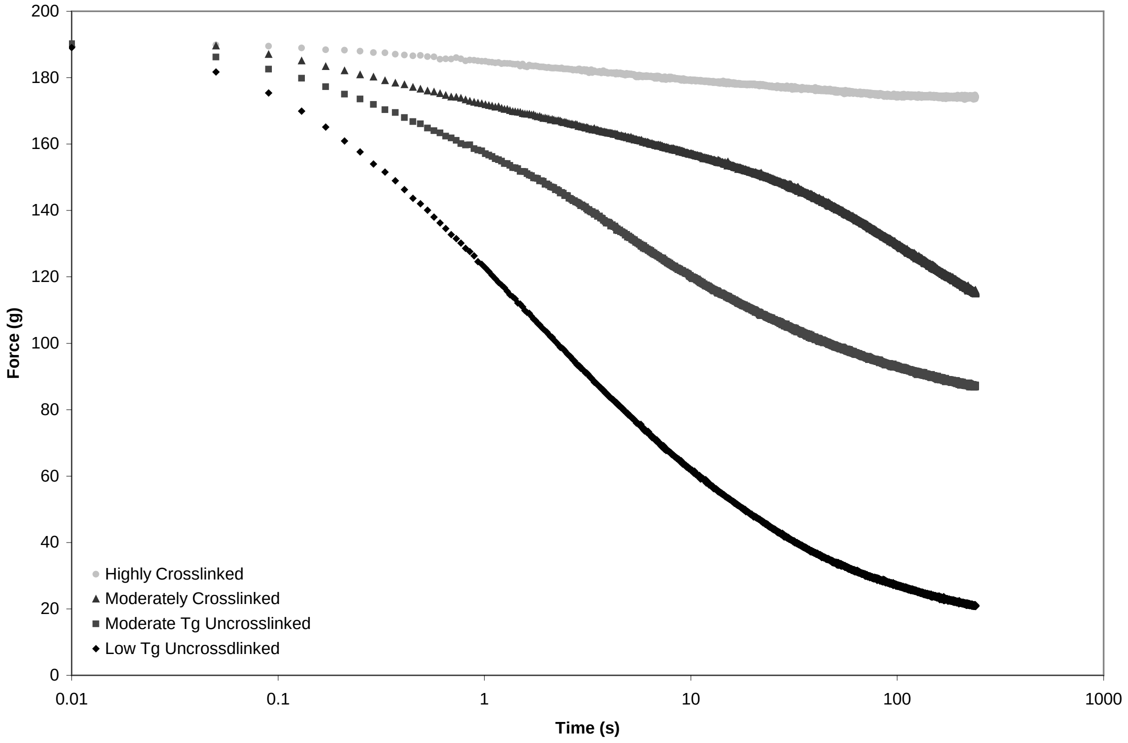

Figure 3. Force Retention measurements of a very broad range of acrylic solvent-based PSAs. Force Retentions measurements ranged from 11% to 93%.

Williams Plasticity Correlation

One of the key methods to determine the validity of the Force Retention technique was to directly correlate the results with Williams Plasticity measurements. For this study, eight unique solvent-based acrylic pressure sensitive adhesives were tested for both Williams Plasticity and Force Retention. The adhesives were selected to cover a very broad range of cohesive strengths and compositions. Over the range of Williams Plasticities typically found in solvent acrylic PSAs (1.8 – 5.3), an extremely high linear correlation was observed between the two test techniques, with r^2 equaling 0.96. The correlation between the two tech techniques is plotted on Figure 4.

The strong correlation between Force Retention and Williams Plasticity carries multiple advantages. In addition to validating the test results with an established and capable test method, this correlation also suggests that it may be possible to complement or replace Williams Plasticity with a faster, easier, and more capable test method. Test versatility is also improved versus Williams Plasticity since coated stock can be tested directly; plasticity requires the preparation of spherical specimen from PSA transfer films.

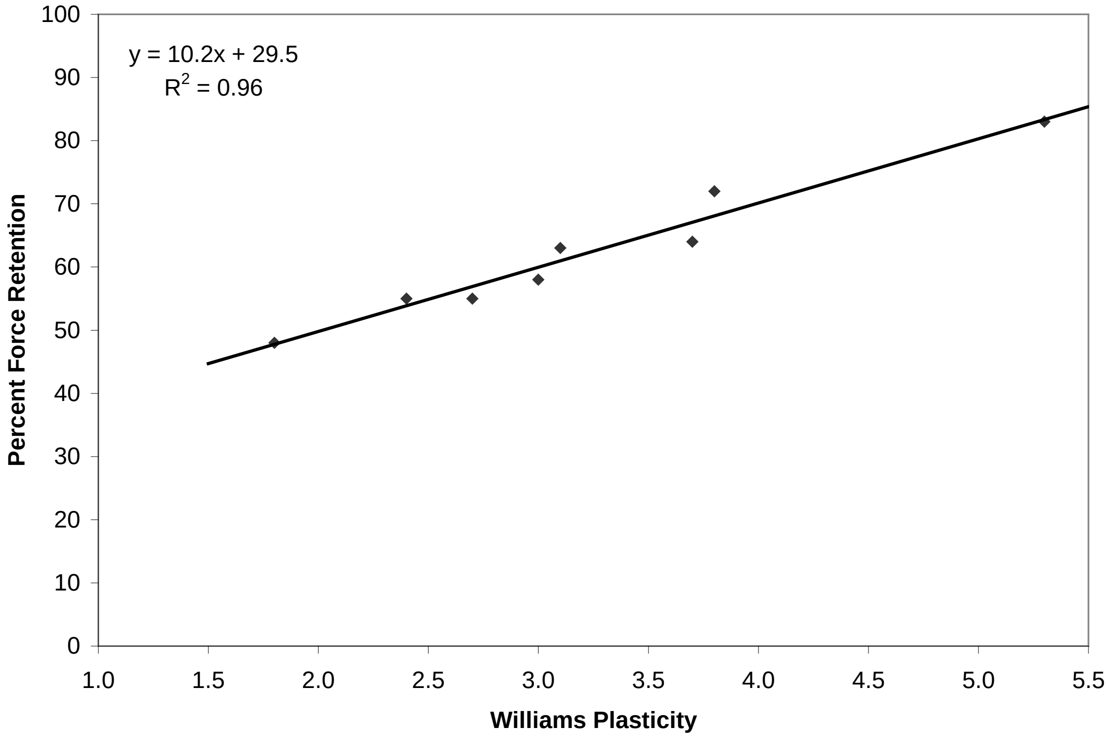

Figure 4. Direct comparison between average Force Retention and Williams Plasticity measurements of eight unique PSAs indicates excellent linear correlation.

PSA Performance Optimization

The overall PSA performance of acrylic solvent-based pressure sensitive adhesives can be modified by changing the crosslinker concentration. The goal of most solvent PSA crosslinker optimizations is to maximize adhesion and tack while maintaining sufficient cohesive strength to ensure robust product converting ability and cohesive performance in the application. The traditional approach has been to create a crosslinking "ladder" which, plots PSA performance values, including shear failure times, versus crosslinker concentrations. In most cases, shear failure times start very low (less than 1 hour) and rapidly increase with increasing crosslinker concentration (Figure 5). Shear failure times become highly sensitive to crosslinker concentration at increasingly high levels.

One challenge of the shear test sensitivity to higher crosslinking is that it can be challenging to determine the crosslinker concentration that provides sufficient cohesion over typical lot-to-lot variation of adhesive. In contrast, Force Retention measurements for the same pressure sensitive adhesive samples over this crosslinking range gave rapid initial rise of creep resistance, followed by a significant flattening of the crosslinking curve. The advantage of this data set is that it clearly identifies 85% relative crosslinker content as the point at which creep resistance does not rise very rapidly with additional crosslinker. As a result, it was determined that 90% relative crosslinker was the minimum acceptable crosslinker loading needed to ensure product cohesion capability.

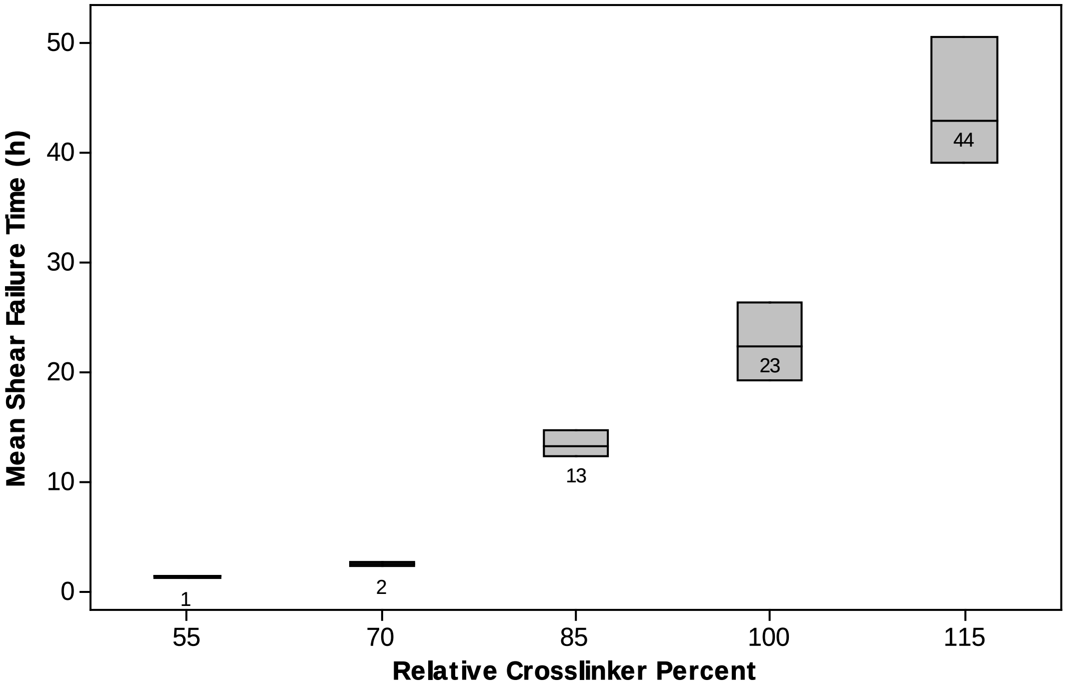

Figure 5. Solvent PSA shear failure time increases drastically with increasing crosslinker amounts.

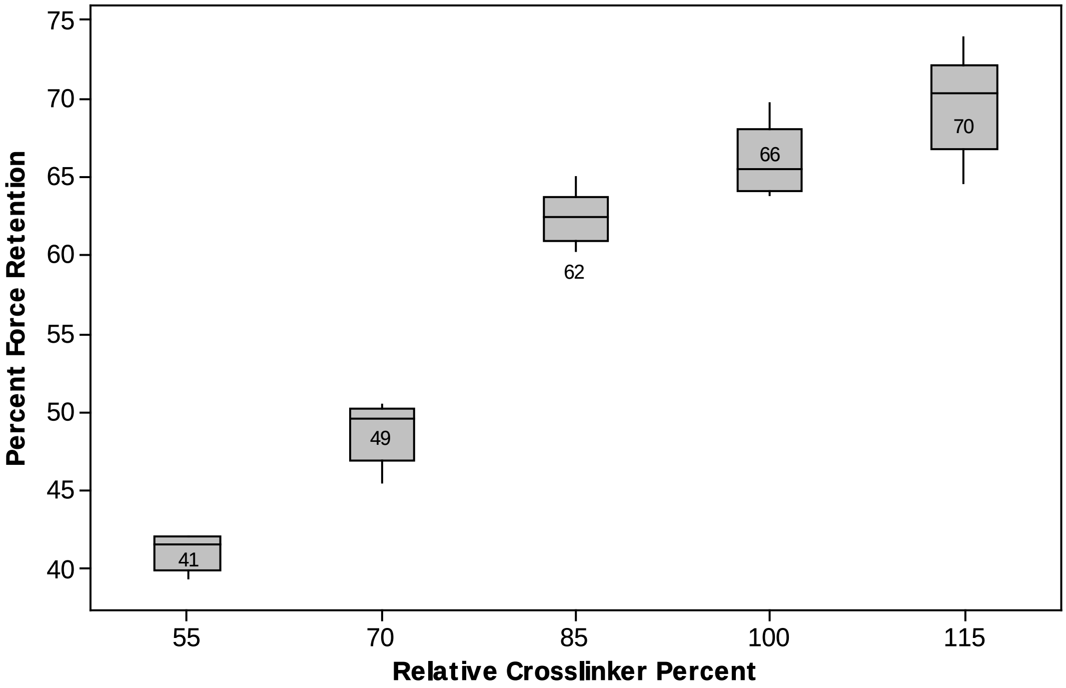

Figure 6. Force Retention data for increasing crosslinker indicates inflection point at 85%, beyond which limited cohesive strength is gained with increasing crosslinker.

Utilization of Force Retention in Process Control Determination

Shear is commonly utilized as the PSA cohesion quality control tool in acrylic PSA manufacturing. However, the test limitations discussed in the introduction result can result in process data that may not adequately reflect the capability of the production process. In addition, large test variation can increase the likelihood that an atypical production batch of adhesive is detected incorrectly, either as a false positive or undetected negative result. Finally, many shear tests on PSA-coated films can take multiple days, reducing the speed at which lots can be released for shipment to customers.

The control chart in Figure 7 was generated from 15 lots of typical solvent acrylic PSA that was coated on production equipment. Utilization of machine-coated stock minimized testing variation due to sample preparation, enabling an improved focus on the capabilities of the process and test. The shear data generated from these fifteen lots ranged from 30 to 71 hours. The data series was deemed to be typical of other comparable solvent acrylic PSA products for both the sample mean and sample range variation. However, both the sample means and ranges were very significant in relation to the process mean. The process control limits were calculated to be ± 42% of the sample mean. In addition, there were three lots with shear values that deviated significantly from the mean (samples 4, 8, and 9). Based on shear testing alone, it would appear that the process capability was limited. However, without an additional capable cohesion test, it would have been difficult to achieve an accurate determination of process capability.

The fifteen production-coated lots of solvent PSA were also tested using Force Retention and the results were compared to the shear testing. Significant reductions in both the sample mean and range control limits were observed (Figure 8). The potential outlying lots #4, 8, and 9 identified by the shear testing were determined to be typical lots. In contrast to the shear control limits being ±42% of the mean for the shear testing, the force retention process control limits were determined to by ±6% of the process mean (and the average sample ranges of the shear tests were three times greater). Process capability was determined to be significantly better using the Force Retention method, due to the improved reproducibility. In addition, using Force Retention as the primary QC tool for this process instead of relying on shear would reduce the average sample testing time from two days to 30 minutes.

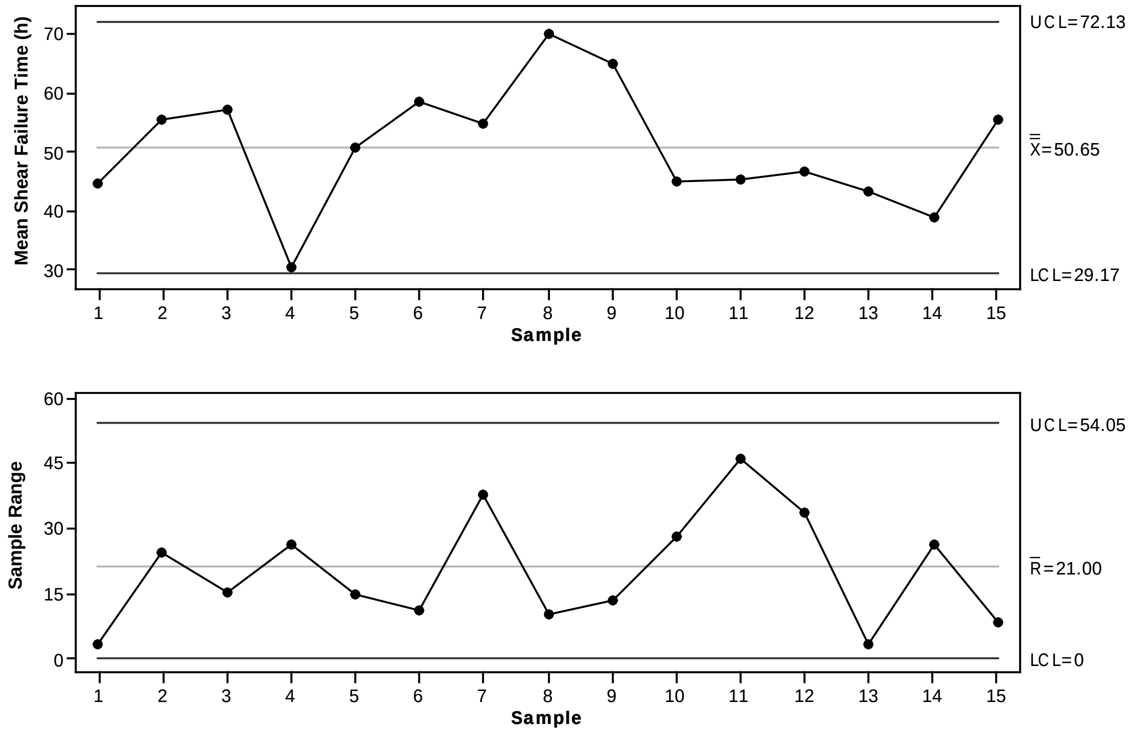

Figure 7. Shear failure times of production-coated PSA lots indicates a wide performance range, despite the fact that the process was known to be under control.

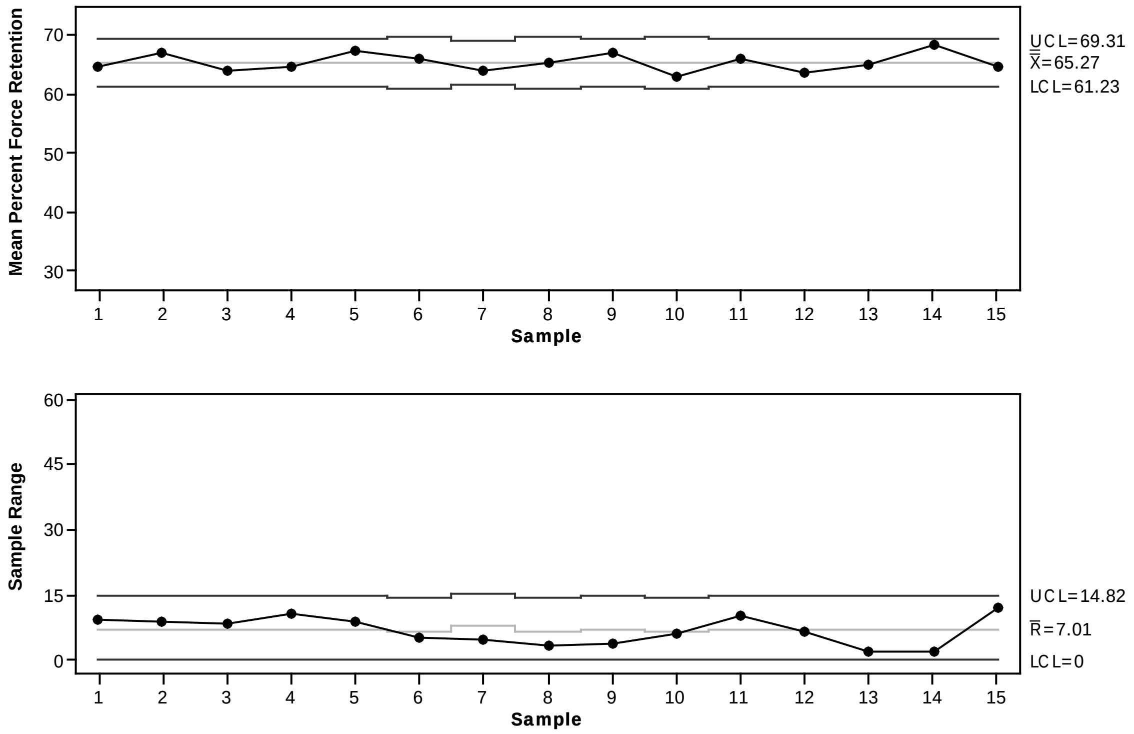

Figure 8. Quality control data of production coated PSA, determined by Force Retention method, and graphed using same axes as the corresponding shear data set.

4. Conclusions

A new method, Force Retention, was developed for rapid and accurate quantification of PSA compressive force creep resistance in coated PSA films. Force retention utilizes pre-existing Texture Analyzer probe tack testing equipment already utilized around the PSA industry. Creep resistance is measured reproducibly by measuring the force decrease caused by PSA diffusion away from the area of the sample that is under probe contact. This technique was designed to mimic the forces applied in the Williams Plasticity test, and correlation testing indicates this goal was achieved. Force Retention was also shown to improve both PSA performance optimization as a result of crosslinker adjustment, and the test method showed that a production process was more capable than was apparent based on static shear testing.

Force Retention testing enjoys multiple advantages over legacy cohesive tests, including shorter test time, greatly simplified sample preparation, and improved data quality. In addition, the same test configuration can be applied to all PSA types, ranging from non-crosslinked low molecular weight thermoplastic to highly crosslinked removable thermoset PSAs. This test technique is still under development and further improvements in test capability and applicability are expected.

5. Future Work

Ongoing work is underway to adapt the Force Retention test to the newest-generation Stable Micro Systems Texture Analyzer, the TA.XTplus. Differences in software and hardware design and capability will require shifting some of the test settings in order to achieve the same initial target force range and reproduce other key test parameters. Additional data analysis will be applied to the pre-existing database of force retention measurements in order to determine if more accurate creep resistance results can be extracted from this type of testing.

Test capability is a critical aspect to improved understanding of this new test technique. Once test conversion to the new texture analyzer is complete, a more thorough measurement systems analysis will be undertaken to determine the impact of all sources of sample variation: sample preparation, operator, probe reproducibility, and instrument variation.

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