Pressure-Sensitive Adhesive Tack Using the ARES Rheometer

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Introduction

While the definition of tack is somewhat mysterious, it is arguably the most important parameter for assessing the performance of a pressure-sensitive adhesive (PSA). Tack most commonly refers to the ability of an adhesive to form a bond at short contact times and low pressures[1]. A wide variety of tack tests have been developed. For example, the American Society for the Testing of Materials (ASTM) and the Pressure-Sensitive Tape Council (PSTC) have a series of tests which attempt to quantify tack, including:

1. Pressure-Sensitive Tack of Adhesives Using an Inverted Probe Machine (ASTM D2979-95)
2. Standard Test Method for Tack of Pressure-Sensitive Adhesives by Rolling Ball (ASTM D3121-94)
3. Rolling Ball Tack (PSTC-6)
4. Quick Stick of Pressure-Sensitive Tapes (PSTC-5)

Each of these tests is actually only an indexer, as it provides only one measurement of tack. Specifically, ASTM D2979-95 and PSTC-5 report tack as a maximum force during separation, while ASTM D3121-94 and PSTC-6 report tack as the distance of travel of the rolling ball. In addition, each of these tests is fairly crude and requires a large number of samples to obtain statistically significant results.

A more appropriate measurement of tack would provide a force versus distance failure curve as a function of contact time, contact area, temperature, pulloff rate, and applied load. The inclusion of vertical motion control in the Advanced Rheometric Expansion System (ARES) rheometer allows each of these variables to be controlled with acceptable precision. In addition, the force (in fractions of a gram) can be measured as a function of time (in fractions of a second) to provide the force versus distance failure curve. This paper summarizes a series of tack tests which has been performed with the ARES under ambient conditions on 3M Post-it Notes, 3M Scotch Tape, and Bemis Packaging Tape.

Experimental Procedure

Sample Mounting

Four configurations were used, all with the disposable plate fixtures. In all cases the lower plate was made of aluminum and was 50 mm in diameter. All of the probes were truncated cylinders with finely polished surfaces. These are summarized in Table 1.

Table 1: Arrangement of plates and fixtures for ARES probe tack experiments.

Configuration	Samples Tested	Probe Material	Probe Diameter	Lower Plate	Method Used to Affix PSA
1	Post-it Notes, Scotch tape	Aluminum	10 mm	Solid	Double-sided tape
2	Packaging tape	Stainless steel	10 mm	Solid	Superglue
3	Packaging tape	Stainless steel	5 mm	Solid	Superglue
4	Packaging tape	Stainless steel	5 mm	15 mm hole	Superglue

The samples were prepared 24 hours in advance, to allow the Superglue to fully cure.

Test Parameter Determination

As described later, the ARES tack test brings the probe into contact under constant force control, and withdraws the probe at constant rate. If the default proportional (P) – integral (I) control settings are used, considerable overshoot and oscillations about the commanded force can occur. With Orchestrator version 6.4.4 and later, the PI parameters can be adjusted to eliminate this. Figure 1 demonstrates the effect of changing the I value while keeping the P value constant at 1 and a commanded force of 100 g. Note that the default values are P = 1 and I = 1 which overshoots by nearly a factor of 4 and then oscillates (unstable) about the commanded force.

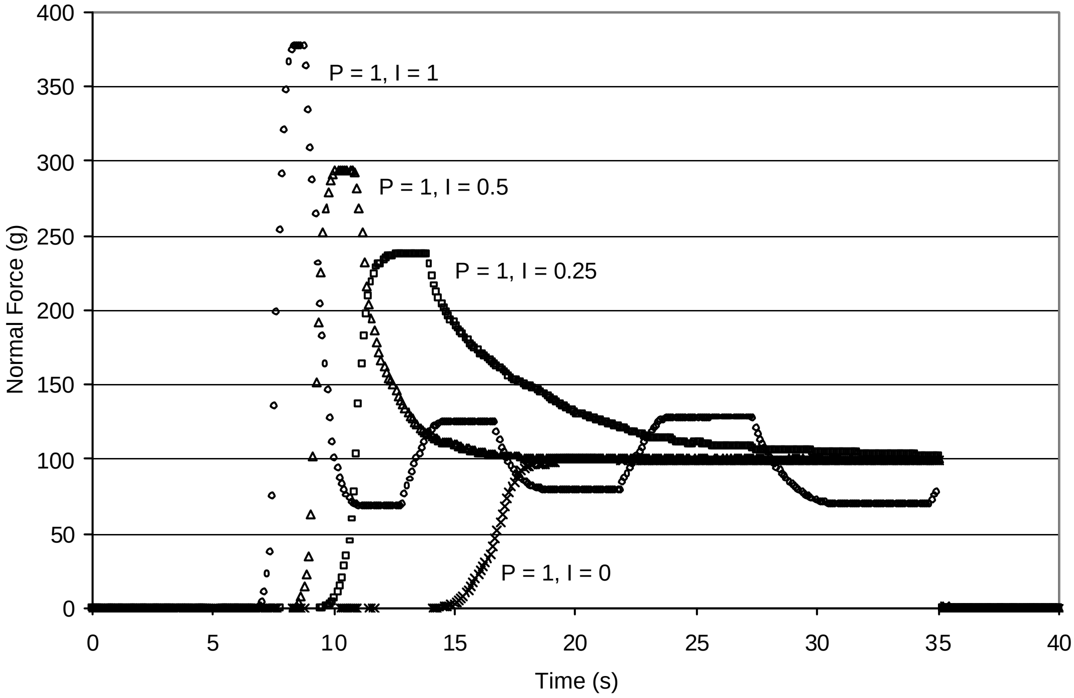

Figure 1: Effect of changing the integral (I) control parameter under an applied force of 100 g and a constant P-value of 1.

In virtually all cases, the integral parameter must be set to zero to avoid oscillations. This has the undesirable consequence of extending the time to contact the adhesive. Increasing the proportional parameter can compensate, but care must be taken to not drive the probe into the lower plate. Figure 2 demonstrates the improvement in contact rate with the integral parameter set to zero.

While Figures 1 and 2 suggest that P = 3, I = 0 are appropriate for tack tests, it is important to realize that the control parameters must be changed depending on the adhesive (and backing) to be tested. For example, for configuration 1 the settings were P = 3, I = 0, but for configuration 3 the parameters were P = 0.9, I = 0. Thus, extra samples must be prepared to determine the appropriate parameters before any tack tests can be performed.

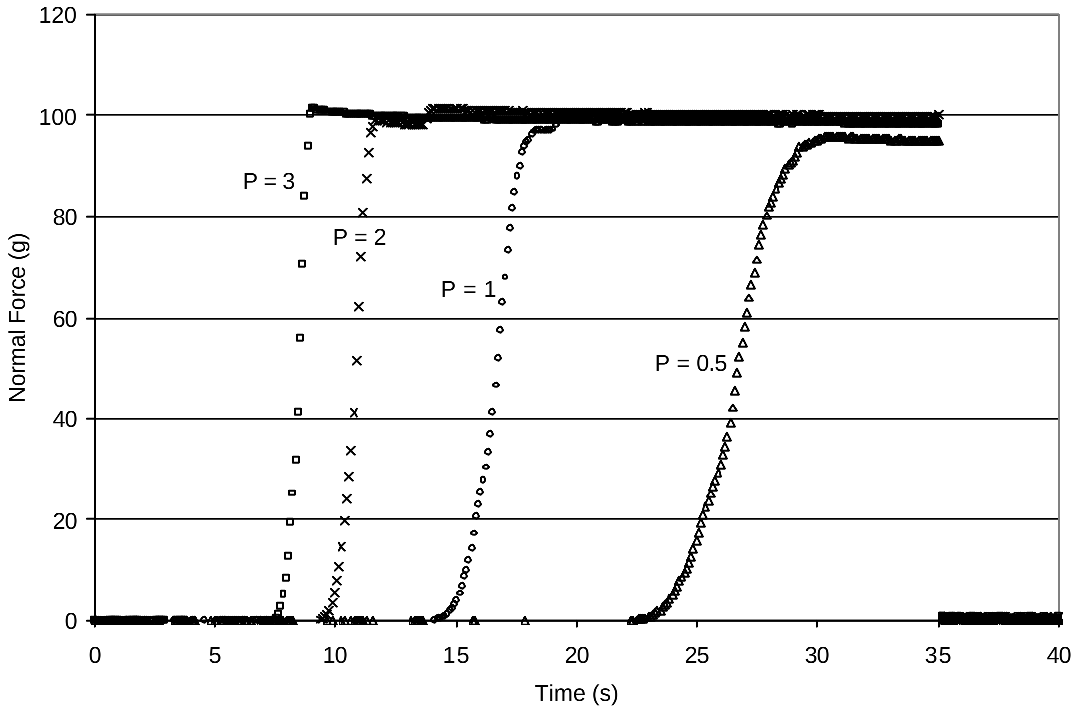

Figure 2: Effect of changing the proportional (P) control parameter under an applied force of 100 g and a constant I-value of 0.

Testing

After mounting the lower fixture, the upper fixture and appropriate probe can be mounted. Next a solid lower plate should be attached to the lower fixture so that the gap can be zeroed. The solid lower plate should then be replaced with a sample so that the appropriate PI control parameters can be determined. In addition, the time to contact and reach the commanded force should be recorded. At this point tack testing can begin.

Between each sample the probe must be cleaned with a volatile solvent, which in all of this work was toluene. After cleaning the probe, the next sample is secured in the lower fixture. The probe can then be lowered until *almost* in contact with the PSA (typically a fraction of a millimeter). The Orchestrator software requires several parameters. First, parallel plate geometry should be selected, with the appropriate starting gap indicated, and the box marked record gap checked. The test is listed under the transient tests, and is called a multimode extension test. Next, the test should be edited so that in the first zone force is controlled to the selected value. The duration of the constant force zone should include the time to contact, time to reach the set value, and the desired contact time. The second zone should then be set at the desired constant rate of withdrawal. Note that the sign of the rate determines the direction of travel! The time for this zone is unimportant, and should be set so that that probe is retracted far enough to remove the sample at the end of the test. Finally, the third zone should be set as "end of test". Under the options menu and the motor control submenu, the PI control parameters can be set. At this point the test can be run. At least five samples should be run at each condition.

Results and Discussion

In order to better evaluate the ARES tack test, a series of tests were performed on a removable PSA (Post-It Notes) and a more aggressive PSA (Scotch tape). The expectation was that the Post-It Notes would have lower tack than the Scotch tape. Six tests were done using Condition 1 and a control force of 100g. The results are summarized in Table 2 below.

Table 2: Comparison of tack in terms of the maximum force of removal for Post-It Notes and Scotch tape.

Sample Type	Test Parameters	Average Tack (N)	Standard Deviation
Post-It Notes	30s hold; 5mm/s pulloff	4.12	0.65
	60s hold; 0.5mm/s pulloff	2.82	0.35
	60s hold; 5mm/s pulloff	4.06	0.49
Scotch Tape	30s hold; 5mm/s pulloff	11.92	1.58
	60s hold; 0.5mm/s pulloff	8.56	1.28
	60s hold; 5mm/s pulloff	13.57	0.73

These data confirm our expectations. In all cases, the Scotch tape has greater tack than the Post-It Notes. It is important to note that other factors, such as adhesive thickness, can affect the tack value and may have contributed to the differences.

While differences between PSAs can be seen clearly, the effect of changing a test parameter must also be determined. Two of these variables are contact time and applied load. Again using Condition 1 and Scotch tape, six tests were performed to isolate any effects of these variables. These are summarized in Table 3 below.

Table 3: Effect of changes in contact time and applied force on the maximum recorded force during withdrawal of the probe.

Force (g)	Contact Time (sec)	Average Tack (N)	Standard Deviation
10	5	6.84	2.28
	30	8.13	0.71
	60	10.07	1.00
100	5	13.43	0.93
	30	11.92	1.58
	60	13.57	0.73

The data indicate that contact time is important at lower applied loads. This is likely due to the wetting (and adhesive bond formation) of the probe by the adhesive. At low forces the adhesive bonding increases with time as the surface wets, while at high forces the adhesive deforms significantly, increasing wetting even at short times.

The final variable that was considered with Configuration 1 was pulloff rate. Assuming that a good adhesive bond has formed between the PSA and the probe, the slower the removal rate of the probe, the greater the opportunity for the adhesive to flow and dissipate energy. Thus, as rate increases the tack should be higher, but the area on a force versus time curve should be less. Scotch tape was again tested with contact times of 30 seconds, an applied load of 100g, and pulloff rates of 5 mm/s and 0.5 mm/s. Figure 3 compares the pulloff behavior at the two rates.

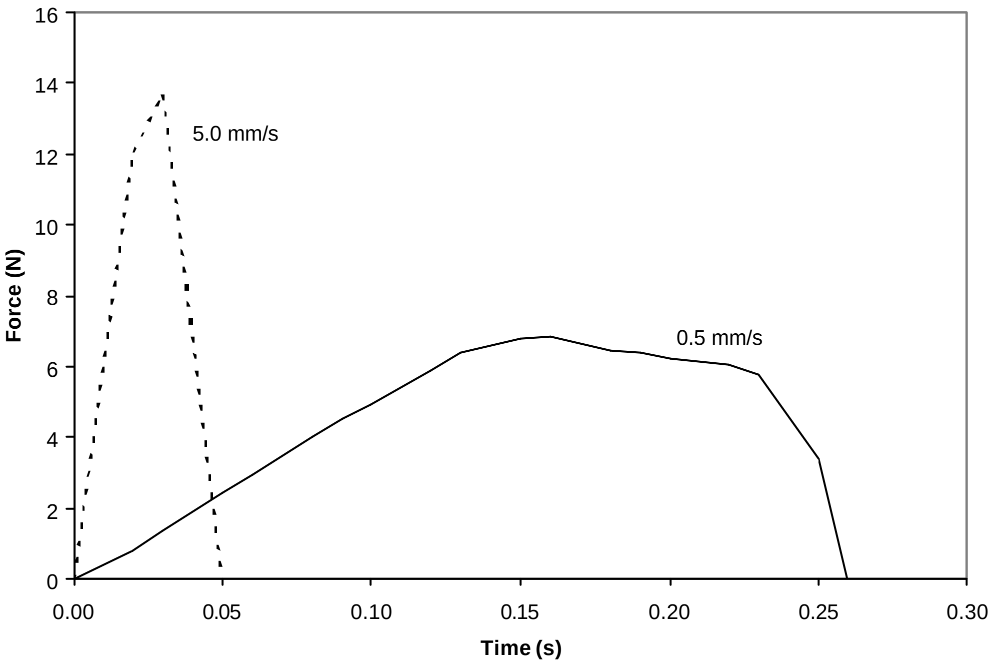

Figure 3: Comparison of failure curves at two pulloff rates for Scotch tape.

To this point most of the discussion has focused on the effects of changing test parameters. Another important consideration is how the test configuration affects the results. To explore this aspect, three different configurations were used (2-4 in Table 1), including a 10 mm probe with a solid lower plate, a 5 mm probe with a solid lower plate, and a 5 mm probe with an annular plate. The hole in the center of the annular plate was 14 mm in diameter and permitted deflection of the PSA backing as the probe was brought into contact with the adhesive. Bemis packaging tape was used in all cases and was affixed to the lower plate using Superglue. While a series of tests were done, only the results for 100 g of applied force, 30 seconds of contact time, and a withdrawal rate of 0.5 mm/s will be discussed. These tack data are presented in Table 4, while the failure curves are shown in Figure 4.

Table 4: Comparison of tack using different experimental configurations (2-4 in Table 1).

Probe / Lower Plate	Tack (N)	Standard Deviation
10 mm / Solid	3.12	0.11
5 mm / Solid	7.13	0.97
5 mm / Hole	8.17	1.13

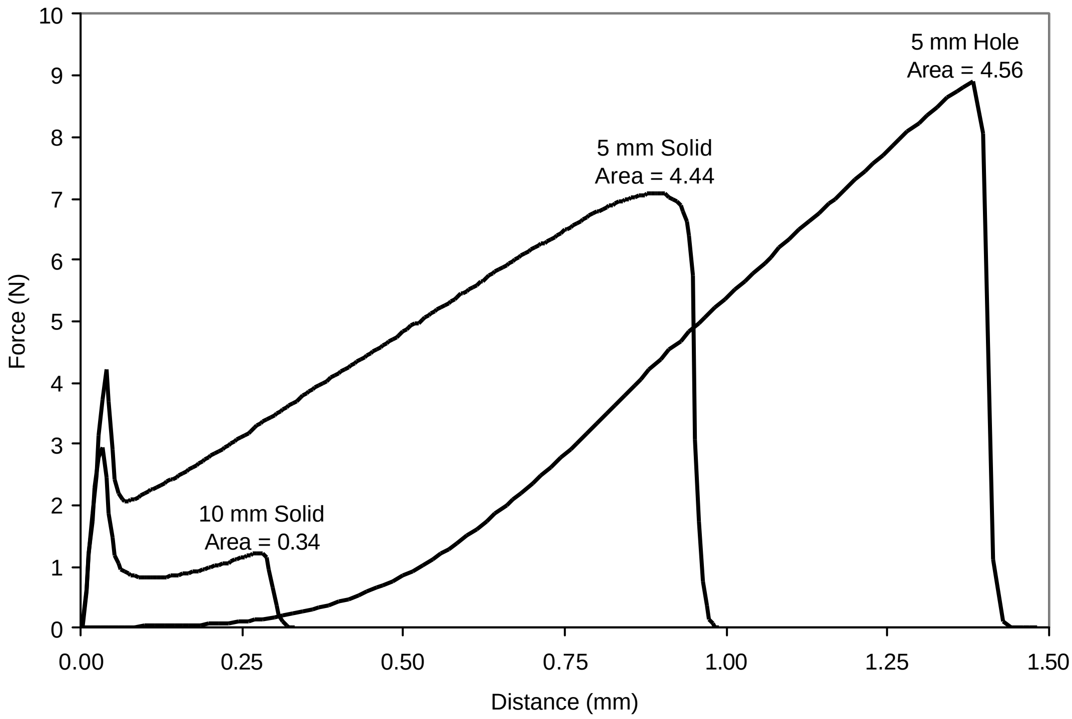

Figure 4: Comparison of failure curves for the three experimental configurations summarized in Table 4.

The shape of the curves in Figure 4 is significant. The 10 mm Solid curve demonstrates the expected behavior for a PSA in a probe tack configuration. The initial peak is directly related to the tack of the adhesive. Additionally, the curve shows that the force increases as the probe is withdrawn following Hooke's law up to a yield point at which the adhesive begins to detach from the probe and form adhesive filaments. The height of this peak depends on the wetting of the probe by the adhesive and the degree to which the adhesive can resist the debonding forces. Since these two factors oppose each other, a high initial peak will be attained when the modulus of the adhesive at the bonding rate is high and the debonding rate is high[2].

The second peak represents the stretching of the filaments. Specifically, the polymer chains are themselves stretched and aligned. This reduction in entropy produces a strain hardening effect. This peak to is dependent on the wetting of the probe by the adhesive. If bonding of the adhesive to the probe is lower than the force needed to stretch the polymer chains then the second peak will be lower, or even absent[2]. This is expected in the case of a weak PSA like the Post-It Notes discussed earlier. In fact, the Post-It Notes did not show a second peak under any testing conditions.

As seen in Figure 4, the 10 mm probe has a large initial peak, but the second peak is barely visible. This suggests that the probe was not fully wetted. This is confirmed by the subsequent 5 mm probe tests with the solid and annular plates. Specifically, the smaller probe can be wet more easily, and as the backing is permitted to flex the wetting further increases. Thus, the second peak increases quite dramatically. The first peak is more complicated. At the slow pulloff rate of 0.5 mm/s the adhesive flows readily, thus the first peak is small in both area and maximum magnitude. In fact, when the backing is allowed to flex, filaments form immediately and no initial peak is observed. At higher rates the first peak would not be smaller than the second peak, and the peculiar behavior seen in Figure 4 would not be observed.

Since ASTM D2979-95 specifies a force of 19.6 g and allows for some flexing of the backing material, additional tests were performed with Configuration 4 using 20 g as an applied force and the Bemis packaging tape. The tack results are summarized in Table 5.

Table 5: Data taken using Configuration 4 and 20 g of applied force.

Contact Time	Pulloff Rate	Tack (N)	Standard Deviation
5 sec	5 mm/s	6.34	1.26
30 sec	5 mm/s	6.48	1.62
5 sec	0.5 mm/s	4.95	0.61
30 sec	0.5 mm/s	5.62	0.65

As with the Scotch tape discussed above, the lower applied force reduces the wetting of the PSA and, therefore, the tack. The shapes of the failure curves are all consistent with the explanations provided above.

Conclusions and Recommendations

The ARES probe tack test satisfies the criteria for a tack test as outlined by ASTM D2979-95. Perhaps more importantly, this test is capable of distinguishing different types of adhesives, accurately reflects changes in test parameters, and can demonstrate different failure mechanisms depending on the test configuration. In addition to the data presented here, other studies should be planned to take advantage of the environmental control available in the ARES rheometer. Specifically, since temperature dramatically affects a PSA's rheological properties, changes in failure behavior should be observable.

References

1. Zosel, A. *Colloid and Polym. Sci.*, **263**, 541 (1985).
2. Chuang, H. K., C. Chiu, and R. Paniagua. *Adhesives Age*, September, 18 (1997).