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MTS Adhesives Programme 1996-1999 - Project PAJ1

Failure criteria and their application to Visco-Elastic/ Visco-Plastic materials

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Tack Loop tack round robin

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1 Introduction

One of the aims of Project PAJ1 - Failure criteria and their application to visco-elastic/visco-plastic materials - of the DTI Performance of Adhesive Joints Programme was to improve methods for measuring the tack of pressure sensitive adhesives. As part of this study, a round robin exercise was performed to assess the reproducibility of loop tack testing.

FINAT test method no. 9 describes a method of assessing the tack of a pressure sensitive adhesive. (FINAT is an organisation based in the Netherlands. The main priority of the FINAT Technical Committee is the production of technical standards and test methods for suppliers and users of pressure sensitive laminates primarily for label applications.) This is one of the test methods described in a literature review on tack⁽¹⁾ carried out as one of the earlier tasks in this project.

This test method allows an end user a means of comparing "initial grab" or "application tack" and is useful for those working with automatic labelling equipment, for example. "Grab" is the subjective estimate of "tack" - the property of a material that enables it to form a bond immediately on contact with another surface which may be an adherend or another layer of adhesive. "Quick stick" is sometimes used to describe the tack force immediately after the bond is made. The test has the advantage of testing the tape as supplied rather than having to coat the adhesive onto a polyester film which is then brought onto a test substrate.

Initial enquiries suggested that the test is used by a wide range of manufacturers and users but in some cases irregularly.

The objective of the round robin was to assess the reproducibility of measurements within and between laboratories.

2 Procedure

Initially a letter was sent to in excess of 50 companies, mostly manufacturers and users of pressure sensitive adhesives. The prime sources for company names and addresses were:

- Pressure Sensitive Manufacturers Association
- Adhesive Tape Manufacturers Association
- Adhesives and Sealants Directory (The Yearbook of the British Adhesives and Sealants Association)
- Packaging Industry Directory

Positive responses were received from 13 companies, with a further five requesting a copy of the report on completion but being unable to participate in the round robin for various reasons.

Each participant was supplied with:

- a reel each of three different tapes
- a box of microscope slides, to provide a standard surface
- FINAT test method no. 9 (Appendix A)
- further instructions including advice on cleaning the microscope slides with methanol, removing the outer three turns of each reel of tape before commencing testing
- sheets on which to record results and equipment used.

The three different tapes were manufactured by Scapa Tapes (previously Sellotape Industrial Ltd) and were:

- clear polypropylene ref. 1240 (rubber resin adhesive)
 packaging tape
- green polyester ref. 1627 (silicone adhesive)
 splicing tape
- black paper ref. 2566 (rubber resin adhesive)
 masking tape

3 Test procedure

The test procedure as defined by the FINAT test method is given in Appendix A.

Briefly the procedure is to take a strip of the adhesive tape 25 mm wide, minimum length 175 mm, and to form it into a loop with the adhesive surface facing out. The ends of the loop are clamped in the top jaw of a tensile tester with the loop hanging vertically downwards.

The glass plate is clamped in the lower jaw of the tensile tester with the long axis of the plate at right angles to the long axis of the strip.

The loop is lowered onto the glass plate at 300 mm/min and immediately full contact has been achieved over the glass plate (25 x 25 mm) the machine is reversed and separation takes place at 300 mm/min. The delay in reversing direction should be kept to a minimum.

The maximum force necessary to completely separate each loop from the glass plate is recorded, and "quick-stick" tack is expressed as the average value (ignoring the initial peak) and range for the replicates measured (minimum five).

4 Results

Results were obtained from 13 laboratories including NPL, SATRA and Pira International. These are listed in Appendix B.

The "quick-stick" tack results are summarised for each type of tape in tables 1, 2 and 3, giving means and standard deviations for each set of results.

Table 1

**Loop Tape “Quick-stick”, Black tape 2566
(units - Newtons)**

Lab no	Mean	Standard deviation	Coefficient of variation	No of reps
1	7.26	0.52	7.16	10
2	4.50	1.04	23.11	10
3	7.32	0.74	10.11	10
4	7.20	0.47	6.53	10
5	7.53	0.69	9.16	10
6	6.60	1.14	17.27	10
7	9.00	0.73	8.11	10
8	5.35	0.23	4.30	10
9	8.42	0.68	8.08	10
10	8.64	0.93	10.76	10
11	4.42	0.65	14.71	10
12	6.46	0.75	11.61	10
13	9.25	0.87	9.41	10

Statistics

Number of labs

13

Grand mean

7.07

SD within

0.76

SD between

1.59

Warning limits

Grand mean - 2,0 SD

3.90

Grand mean + 2,0. SD

10.25

Action limits

Grand mean - 2,6 SD

2.94

Grand mean + 2,6 SD

11.20

Table 2

**Loop Tack "Quick-stick", Green tape 1627
(units - Newtons)**

Lab no	Mean	Standard deviation	Coefficient of variation	No of reps
1	10.81	1.02	9.44	10
2	12.49	1.12	8.97	10
3	13.47	1.07	7.94	10
4	11.40	1.76	15.44	10
5	12.94	0.82	6.34	10
6	12.80	0.87	6.80	10
7	13.10	1.21	9.24	10
8	13.38	1.44	10.76	10
9	12.96	0.94	7.25	10
10	14.60	0.79	5.41	10
11	11.83	1.02	8.62	10
12	12.69	0.31	2.44	10
13	14.80	0.65	4.39	10

Statistics

Number of labs

13

Grand mean

12.87

SD within

1.06

SD between

1.12

Warning limits

Grand mean - 2,0 SD

10.63

Grand mean + 2,0. SD

15.11

Action limits

Grand mean - 2,6 SD

9.95

Grand mean + 2,6 SD

15.78

Table 3

**Loop Tack “Quick-stick”, Clear tape 1240
(units - Newtons)**

Lab no	Mean	Standard deviation	Coefficient of variation	No of reps
1	18.45	3.05	16.53	10
2	5.98	2.24	37.46	10
3	15.01	3.96	26.38	10
4	14.70	1.85	12.59	10
5	16.48	0.95	5.76	10
6	12.22	2.66	21.77	10
7	20.80	0.87	4.18	10
8	10.99	0.68	6.19	10
9	11.64	3.50	30.07	10
10	18.60	2.00	10.75	10
11	20.38	2.18	10.70	10
12	17.19	2.40	13.96	10
13	23.01	2.83	12.30	10

Statistics

Number of labs

13

Warning limits

Grand mean - 2,0 SD

6.37

Grand mean + 2,0. SD

25.23

Grand mean

15.80

Action limits

SD within

2.44

Grand mean - 2,6 SD

3.55

SD between

4.71

Grand mean + 2,6 SD

28.06

The results have been analysed as suggested in ISO 5725.

Mean

The means within each set of results for each tape were checked for outliers. Outliers would have been marked with an asterisk and excluded from the “grand mean”.

Standard deviation

The standard deviations were checked for outliers. Again any outliers would have been marked with an asterisk and excluded from the “SD within”. If a standard deviation had been found to be an outlier the associated mean would also have been excluded.

Number of laboratories

This is the number of laboratories after excluding outliers.

Grand mean

This is the overall mean of all acceptable means.

SD within

The average of all acceptable SD's.

SD between

The standard deviation of the means.

Warning limits

Range of warning limits ± 2.0 SD about the grand mean.

Action limits

Range of action limits ± 2.6 SD about the grand mean.

5 Discussion of results

Repeatability within a laboratory was generally good although it varied with the type of tape. Some laboratories reported problems handling tapes.

In particular it was found that static electricity could be generated when the tape was unwound from the coil. This contributed to the problem that the loop was not always symmetrical when applied to the glass slide; this would vary the peel angle and hence the force to peel the tape from the slide.

The clear polyester tape gave a very variable (jerky) peel force, and results recorded may be a mean of the tack force or a mean of the peaks recorded.

On some equipment computer programs for recording load were ineffective with a very jerky tack force.

The test procedure as defined by FINAT is somewhat ambiguous, as the procedure states that the maximum force to separate the loop from the plate should be recorded, but that "quick-stick" tack is the average value obtained from at least five measured replicates (ignoring the initial peak).

Reproducibility between laboratories is less good probably mainly because some companies have measured an average tack force and others the maximum tack force. Measurements at Pira indicated that the maximum tack force values could be between 4 and 15% greater than average tack force values.

In addition differences in equipment may have resulted in slightly different values being recorded. For example, in other loop tack measurements carried out by NPL using a Mecmesin Advanced Force Gauge (AFG), a Kipp and Zonen model BD 112 chart recorder, and a Mecmesin Dataplot system, the force gauge had given the highest values, the dataplot software results approximately 3% lower, and the chart recorder results on average 2% lower. However, these percentage differences are in general small compared to the differences in means obtained between laboratories.

A further factor affecting both repeatability and reproducibility was that the experience of using the test varied between laboratories. Some laboratories reporting that they had not carried out the test before.

One company (results 2 and 3) rested a lightweight (3.9g) plastic tube, about 28 mm diameter x 25 mm length, in the loop in order to maintain the shape. This was removed immediately the loop of tape touched the glass plate.

Another company (1) normally tests by supporting the test tape with 200µ PET, which makes the tape easier to handle. This consistently gives higher readings than with an unsupported film. However, on these limited results it would seem that the range of results is reduced using this technique, (table 4).

Table 4
Loop tack force (N) (mean, SD)

	Black 2566	Green 1627	Clear 1240
Unsupported	7.26 ± 0.52	10.81 ± 1.02	18.45 ± 3.05
Supported with 200 µ PET	20.54 ± 2.91	19.85 ± 3.07	28.13 ± 7.36

Table 5
Comparison of the ranking of the tack of the three supplied test materials

Ranking	Test House												
	1	2	3	4	5	6	7	8	9	10	11	12	13
High	1240	1627	1240	1240	1240	1627	1240	1627	1627	1240	1240	1240	1240
Medium	1627	1240	1627	1627	1627	1240	1627	1240	1240	1627	1627	1627	1627
Low	2566	2566	2566	2566	2566	2566	2566	2566	2566	2566	2566	2566	2566

The table shows that 9 out of 13 of the test houses ranked the tapes in the same order, which corresponds to the nominal order supplied by the manufacturer of the tapes. It seems from the additional information supplied by the round robin participants that the main difficulty was in handling the clear polypropylene tape and this led to difficulties when performing the test. The polypropylene tape gave a less even peel trace, sometimes peaking and falling before rising again - this could cause a false low value. The four cases where

the polyester tape (1627) was ranked higher than the polypropylene tape (1240) were associated with a tack value for the polypropylene tape which was low relative to the other laboratories. The ranking order could be changed if the value of the standard deviation was taken into account.

6 Conclusions

1. Test results between companies are variable using this test procedure even when additional guidelines were issued to define the procedure.
2. Some tape materials are more difficult to test than others.
3. The ranking of tapes by loop tack testing is generally the same for all laboratories.
4. Repeatability within a laboratory is generally quite good, although some tapes are more difficult to handle than others.
5. Reproducibility between laboratories is less good, probably at least partly because some companies measured peak "quick stick" force and some measured the mean force.

Reference

1. Roberts, R. A., Review of the Methods for the Measurement of Tack. The Performance of Adhesive Joints 1996 - 1999. Project: PAJ1: Failure Criteria and their Application to Visco-Elastic/Visco-Plastic Materials. Report 5, September 1997.

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Appendix A

Standard FINAT test method for pressure sensitive materials

FINAT test method no. 9 (FTM 9)

„Quick-Stick“ tack measurement

Scope

This test method describes a means of assessing probably the most important and yet the hardest to measure property of pressure sensitive materials, the tack. The method described should allow the end user to compare the „initial grab“ or „application tack“ of different laminates and can be extremely useful to those working with automatic labelling equipment where this property is of particular importance.

Definition

The „Quick-stick“ tack value of a pressure sensitive material is expressed as the force required to separate, at a specified speed, a loop of material (adhesive outermost) which has been brought into contact with a specified area of a standard surface.

Test Equipment

A tensile tester or similar machine with reversing facility and a vertical jaw separation rate of 300mm per minute with an accuracy of $\pm 2\%$. It should also have a capability of measuring direct loads up to at least 20 Newtons with an accuracy of $\pm 2\%$.

A flat „Float Process“ glass plate measuring $25 \pm 0.5\text{mm}$ x $30 \pm 2.0\text{mm}$ x minimum thickness 3.0mm. A metal peg is attached at the centre of one face of the plate. The dimensions of the peg should be such that the peg can be clamped in the lower jaw of the tensometer.

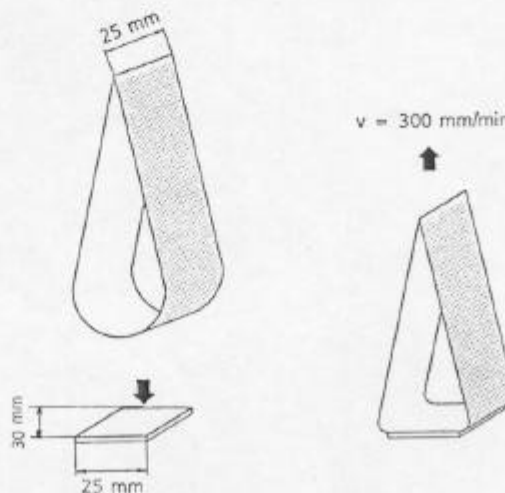
Test Pieces

These should be strips taken from a representative sample of material. The strips should be 25mm wide and have a minimum length of 175mm in the machine direction. The cuts should be clean and straight. At least five strips should be taken from each sample.

Test Conditions

$23^{\circ}\text{C} \pm 2^{\circ}\text{C}$ and $50\% \text{RH} \pm 5\% \text{RH}$.

The test sample or test strips should be conditioned for not less than 4 hours before testing.



Procedure

Remove the backing paper from each strip immediately prior to the test being carried out. Hold the two ends of the adhesive coated facing material strip and from the strip into a loop, adhesive surface outermost, by bringing the two ends together. Clamp the ends of the loop for a distance of 10 mm into the top jaw of the tensile tester leaving the loop hanging vertically downwards. The sides of the jaw should be protected from the adhesive coating.

Clamp the glass plate in the lower jaw with the longer axis of the plate positioned at right angles to the long axis of the strip. Start the machine and bring the loop into contact with the glass plate at a speed of 300mm per minute. When full contact over the glass plate has been achieved (25mm x 25mm) immediately reverse the direction of the machine and allow separation to take place at a speed of 300mm per minute. It is important that delay in reversing direction is kept to an **absolute minimum**. Record the maximum force necessary to completely separate each loop from the glass plate.

Results

„Quick-stick“ tack is expressed as the average value (ignoring the initial peak) and range for the five strips tested in Newtons.

If the force exceeds the strength of a paper facing material, the result quoted should be the maximum reached before the paper tears and this result should be followed by the postscript PT.

If adhesive transfer occurs, this should be indicated by the letters A.T.

Notes

1. The rigidity of the sample affects the results and must be considered when comparing different adhesives on different facing materials.
2. The test plates must be thoroughly cleaned so that no trace of adhesive, grease, silicone or moisture is left on the surface. The recommended method is to suspend the plates over refluxing sulphur-free toluene but other methods which remove contamination properly can be adopted, e.g. ultrasonic cleaning.

Prior to use, the cleaned plates should be left for 30 minutes under standard test conditions. Care should be taken to handle clean plates by the edge only.

3. Any equivalent of „Float Process“ plate glass may be used such as „Kristallspiegelglas“.

4. 1 kgf = 9.81 Newton.

October, 1985

Appendix B

Companies participating in the round robin

Borden Chemical UK Ltd	Rownhams Road, North Baddesley, Southampton SO52 9ZB
Crispen Adhesives Ltd	Coleman Road, Leicester LE5 4NQ
Evode Ltd	Common Road, Stafford ST16 3EH
National Physical Laboratory	Queens Road, Teddington, Middlesex TW11 OLW
Norton Performance Plastics	13 Earlstrees Road, Corby, Northants NN17 4AZ
Pira International	Randalls Road, Leatherhead, Surrey KT22 7RU
Ritrama (UK) Ltd	Lynwell Road, Lyntown Trading Estate, Eccles, Manchester M30 9QG
SATRA	Satra House, Rockingham Road, Kettering, Northants NN16 9JH
Scapa Tapes plc	Holland Street, Denton, Manchester M34 3GH
Stable Micro Systems Ltd	Vienna Court, Lammar Road, Godalming, Surrey GU7 1YL
Tenza	Carlton Park Industrial Estate, Saxmundham, Suffolk IP17 2NL
UCB Chemicals	Anderlecht Str. 33, B-1620 Drogenbos, Belgium

Note: The order of these companies does not imply the order of the test laboratories in the round robin results.