ADHESIVES FOR PACKAGING

29 April 2004

Society for Adhesion and Adhesives

Society of Chemical Industry
15 Belgrave Square London

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Programme

10.00 Registration and coffee

10.30 Adhesives in packaging
Robert Ashley, BA Consultants

11.00 Recent developments in packaging hot melts
Dave Carter, National Starch & Chemical

Adhesive bond testing – beyond fibre tear
Richard Roberts, Pira International

12.00 Lunch

14.00 Rheology and dispensing of adhesives
Martin Rides, NPL

14.30 Packaging systems for adhesives
Alan Crampton, Loctite

15.00 Blister Sealing Process: An Overview
Keith Allen, Eli Lilley

15.30 Measurement of Adhesive Tack
Bruce Duncan, NPL

16.00 Discussion

Tea

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Adhesives in Packaging

The packaging industry is one of the major markets for adhesive products. However, the applications are diverse with a wide spectrum of substrates and types of adhesive used. Some uses include combination of several differing materials to form the basic packaging composite. This may also involve coatings and print layers. These processes may often involve high speed application methods where rapid bonding is required. In later stages adhesives will be used to close the package after contents filling.

A review will be made of these various stages and an indication of the range of materials involved and changes that have occurred. It will become evident that good surface preparation is important. Some of the test methods used to assess the bonding process will be considered and the significance of noting locations or modes of failure.

Packaging materials are supplied to diverse industries such as heavy duty wrappings for engineering to food and medical packs. In the later cases strict controls are required in terms of residual matter that could migrate into products. The materials can often be exposed to some harsh environments and handling conditions such as temperature extremes. Some of the products packed such as cleaning agents, shampoos and oils in foodstuffs can affect the adhesive layer over storage time.

Apart from the conventional adhesive uses some other areas where adhesion science relates to the performance of packaging materials and a bond formed will be reviewed showing that the subject is of greater value.

Some examples of typical packaging failures will be discussed and how these tend to be tackled.

R J Ashley

Recent Developments in Packaging Hot Melts

Dave Carter & Thierry Pasquier , National Starch & Chemical

The adhesive R&D chemist is confronted with many challenges in the current consumer packaging environment. Packaging designers are responding to criteria such as:

1.Demographic changes where populations are aging – but much design is focused and aimed at the youth market.
2.An increasingly fragmented consumer market where sophisticated consumers demand change impacting on pack size , shape , material and content .
3.Technology changes in terms of new packaging materials , processing advances and products themselves. The adhesive chemist can respond by using new materials and processes so driving innovation and change.
4.Legislation changes are a significant and important factor encompassing changes from improved health and safety in production environments to recycling of packaging materials.
5.Competition between retailers , producers and not least adhesive suppliers will push forward new ideas and concepts that offer challenges and opportunities.

This presentation will discuss four product packaging areas involving change that National Starch is closely involved in , these being:

a) Labeling of profiled and shaped containers
b) Low temperature application packaging hot melts
c) Recycling of labeled PET bottles
d) 'Invisible' labeling of glass bottles

These examples will show how National Starch has responded to recent changes in packaging and also how we are driving change through innovation and new technology.

Adhesive bond testing – beyond fibre tear

Richard Roberts, Pira International

Introduction

This work was largely carried out as part of project MMS9 "Efficiency Improvements in Rapid Bonding Processes", run by the NPL. The objectives of the project were:

- to validate a range of simple methods for assessing the hot melt glueability of common packaging substrates
- to develop a range of simple methods for characterising packaging hot melt adhesives with respect to critical performance properties.

The benefit to the packaging industry will be that by reducing the burden of production trials, it will be possible to assess and optimise a wide range of materials enabling the packaging industry to:

- produce adhesively bonded products more reliably
- introduce innovative new materials
- cut scrap and reject rates.

Common desirable attributes of hot melts are:

- low cost
- clean running (absence of stringing or dripping)
- long pot life (thermal stability)
- low temperature application – to reduce worker exposure to fumes
- wide application temperature window
- taint free – food packaging applications
- generates substrate failure – fibre tear is a universal measure of satisfactory bond performance in the packaging industry.

For specific applications, deep freeze, heat/creep resistance performance or adherence to difficult substrates may be required.

In addition:

- open time and setting time are important characteristics of the adhesive, which need to be matched to the specific application
- universal application – manufacturing companies prefer to use a single adhesive grade for all applications to simplify housekeeping , minimise inventory levels and to avoid mistakes.

Tappi paper, TIP 0304-74 states "when dealing with hot melt products, the operator should record and save open time and set time for each individual line ... if possible, the line speed should be slowed until the maximum open time for the adhesive is found, then increased until the minimum set speed is determined. These measurements help to define the operating window for that particular adhesive, at a particular application rate and temperature on a particular application system. If these values are unsatisfactory to production's goals, the adhesive vendor should be consulted for another product that will perform under the desired application conditions and field requirements."

This is a sensible recommendation but raises the issue of how the supplier assesses his hot melt adhesive against this performance requirement without access to the production line.

A simple test widely used in industry involves spreading a film of the adhesive at the appropriate temperature and correct thickness onto a piece of card, and pressing a second piece of board/card onto the film at appropriate time intervals. These joints are then hand ruptured - relatively uncontrolled test which is a combination of tension and peel depending on operator variability and substrate stiffness.

Fibre tear is usually taken as an indication of satisfactory performance, but this can lead to mis-judgement of bond performance where:

- the fibre structure is of lower strength than the application demands
- failure is not fibre tear, but the strength is greater than the application demands
- long term response to stress on the bond is different to short term response – creep effects occur.

The ring and ball softening point test is often used as a quality control measure, for predicting creep performance, but this has limited value.

To overcome these issues, Pira has developed a creep test which applies a sustained load to an adhesive joint. It is designed to:

- occupy minimum time
- be applied equally to rigid or semi-rigid and flexible substrates
- be compact in design and avoid heavy weights so that a number of samples will fit in an environmental cabinet.

The Pira Adhesive Performance Tester (PAPT)
Equipment of varying sophistication has been developed over the years to produce more accurate end performance evaluation.
In the UK the PAPT is often used. Adhesive application temperature, film thickness, open time, compression pressure and time can all be controlled. However, the machine has some limitations. For example, because the compression and rupture forces are measured using a pneumatic piston, the rupture force rises as a controlled rate, but at some time the bond starts to fail causing an expansion of air and a momentary drop in pressure. Hence, the strain rate is not constant.

A new PAPT is in the process of development. This has constant velocity cams driven by servo motors. In addition a low deflection load cell records the progressive rupture strength of the developing bond at each of 5 individual rupture stations.

The first results have been obtained from the new machine.

Hot melt receptivity
The test uses the Dennison Wax system (Tappi T459), consisting of a series of graded hot melt waxes of increasing adhesive power. The standard test requires finding the wax number which will give substrate failure. However, the test is being adapted to assess the "pull-off" force below substrate failure levels which will depend on the ability of the wax to 'wet' onto the substrate. As the wax adhesive power is graded the result will depend upon the wettability of the substrate.

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Rheology and dispensing of adhesives

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Introduction

The reliable dispensing of adhesives is critical to the manufacture and performance of packaging products. Their dispensing behaviour, for example the tendency for adhesives to "string" (also known as "tailing"), is influenced by the adhesives' physical properties, in particular their rheological (or flow) behaviour. Avoidance of stringing is important, for example, for minimising down-time of packaging processing equipment. In the manufacture of electronics components stringing can lead to component failure. Accurate data under process conditions are necessary to help predict behaviour and tune materials and processes.

Characterisation of rheological properties

There is a wide range of rheological techniques available for characterising materials and each one has its advantages and disadvantages. To maximise the value of the data generated it is important to ensure that the most appropriate technique is used for the application. In selecting the appropriate rheological technique it is important to consider various issues, in particular; the intended use of the data, the deformation modes occurring in the processing of the material, and additional complicating factors. A simple rule of thumb is to employ a technique that mimics the process for which the data are required: for example for dispensing an extrusion based characterisation technique is appropriate.

The complete rheological response of an adhesive is comprised of both its viscous and elastic components. In addition, flows can be shearing, for example as in flow through a cylindrical tube, or extensional (stretching) as occur during filament formation. These flows may also be steady or transient; an example of the latter being creep flow. Slip flow may also occur. Properties are typically shear rate and temperature dependant. Changes in material properties may also result from moisture uptake or loss, degradation or cross-linking. Furthermore, materials may also exhibit changes in properties during testing due to changes in their structure, for example for heavily filled adhesives. A time dependence resulting in a reduction in viscosity is referred to as thixotropic behaviour. Thus the rheological behaviour of a material can be very complex.

Stringing is considered to be a complex process that is governed by the physical properties of the materials, in particular their rheological (especially the extensional rheological properties) and surface tension properties, as well as processing conditions, e.g. non-isothermal flow.

This paper reports on rheological measurements carried out on a range of hot melt adhesives, and on developments to the melt flow rate method [1,2] aimed to make it more suitable for testing such materials.

Oscillatory rheometry

The viscoelastic properties of polymer melts can be measured using an oscillatory rheometer [3]. The principle of the technique is to subject a specimen, held between two plates, to a sinusoidal torque or displacement. The response of the sample to that input is measured. Typically, shear storage G' and shear loss G" moduli are determined, the first being a measure of the elastic behaviour and the second of the viscous behaviour. The method is suitable for generating quantitatively accurate data for modelling as well as for quality control.

Results clearly demonstrate that there are very significant differences in the properties of the hot melts, both in terms of the magnitude of the values, the dependence on temperature and also the rate response of the adhesives, Figures 1 & 2. Two of the hot melts exhibit very sharp transitions and are dominated by the material's viscous behaviour, whereas the third material has a significantly broader transition and at high rates its elastic behaviour is considerably more significant.

Melt flow rate testing

The melt flow rate method is a measure of the ease of flow of a material. The principle is to determine how much material is extruded through a die in a given time when a load is applied to the molten sample in a barrel [1]. The melt flow rate technique is appropriate for quality control purposes and provides a simple, qualitative measure of the processability of the material. The results normally obtained are not fundamental rheological properties, and tend to be obtained at relatively low shear rates. However, recent work at the NPL has demonstrated that the melt flow rate instrument, with minor modifications, can be used to determine quantitatively accurate shear viscosity and entrance pressure drop data [2]. From the latter, extensional viscosity data can be derived that are more likely to correlate with stringing behaviour than traditional shear flow data. Results are presented in Figure 3 for hot melts that show melt flow rate values obtained using short and long dies that correlate with extensional and shear flow properties. Furthermore, as a result of recent improvements to the instrument significantly higher melt flow rate values were obtained, with confidence, than was previously possible. Thus it is possible to use this method to produce data under conditions that are more comparable with those occurring in processing.

Acknowledgement

This work is being carried out as part of a programme of underpinning research funded by the Department of Trade and Industry, UK.

References

1. ISO1133: Plastics - Determination of the melt flow rate and melt volume flow rate of thermoplastics.
2. Measurement Good Practice Guide No. 61. Improved testing using the melt flow rate instruments – multi-rate and extensional flow measurements, M. Rides C.R.G. Allen and A. Dawson, National Physical Laboratory.
3. ISO 6721-10: Plastics - Determination of dynamic mechanical properties, Part 10: Complex shear viscosity using a parallel plate oscillatory rheometer.

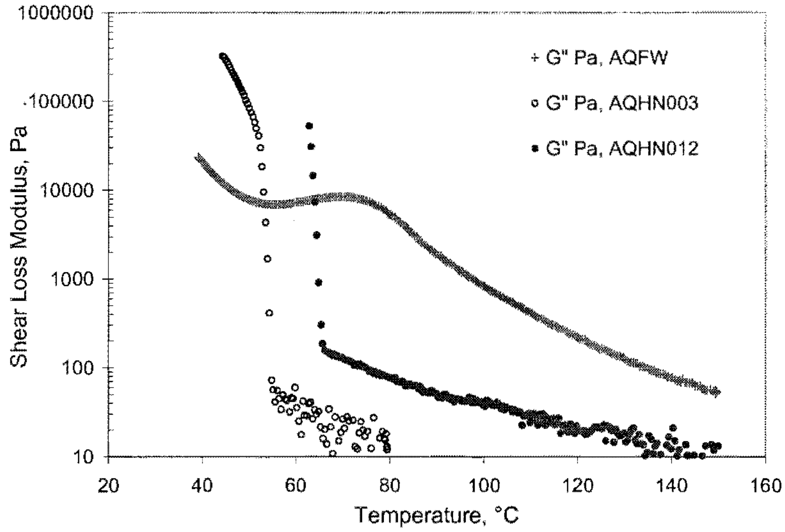

Figure 1: Temperature dependence of hot melt adhesives by oscillatory rheometry, exhibiting significant differences in viscous response. (Material names given are NPL sample codes)

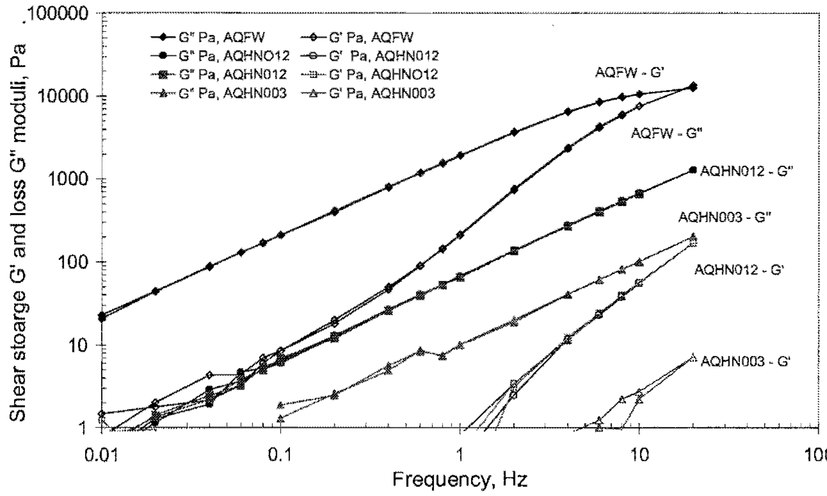

Figure 2: Comparison of the viscoelastic responses of various hot melt adhesives, by oscillatory rheometry.

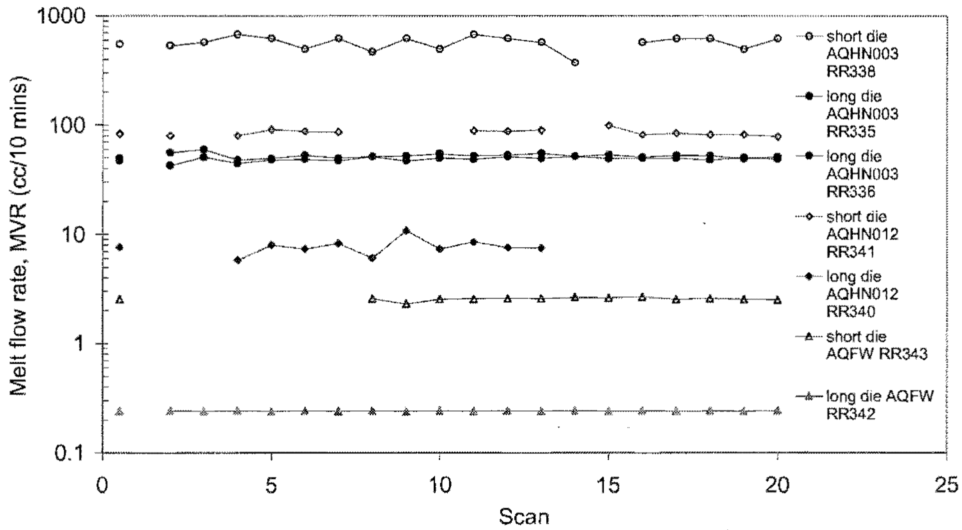

Figure 3: Melt flow rate measurements of three hot melt adhesives using short and long dies, permitting derivation of shear and extensional flow properties.

Packaging for Adhesives

Alan Crampton
Loctite Research Development & Engineering

This presentation looks at the issues concerned with the packaging of adhesives. The basic principles involved in the packaging of products generally are discussed and applied firstly to a familiar consumer product. The example of tomato ketchup is used to understand what the package must do and how the requirements are met. In particular the use of coextrusion technology to achieve the necessary barrier properties is outlined.

These same principles are then applied to the packaging of adhesives. Loctite adhesives are used to demonstrate how the important characteristics of the product are considered by the package technology and how the package contributes to the product shelf life.

The package is an integral part of the product used by the customer. Even for simple manual use applications the package is a critical element of how the product is used. This is even more obvious when a total system consisting of product, package and dispenser is used. Two systems are examined, a collapsible aluminium cartridge for cyanoacrylate adhesives and a bag-in-box for anaerobic adhesives. The benefits of these systems for the two types of adhesives and how they interact with the users are discussed.

Finally some examples of how it can go wrong are examined and used to show how applying the principles of packaging technology can prevent such problems.

An Introduction into blister sealing

Abstract

I can not stress enough, the importance of the adhesive function within the blister pack, it guarantees the protection of the product throughout its shelf-life, protects the material under stressed conditions, both when forming the blister and at extreme climatic conditions, especially in tropical climates.

Blister packaging is not without its problems, some materials are prone to delamination between the plies, such as the Orientated Polyamide (OPA) layer within the formpack base material, pinholes and splits can be experienced after forming. High temperatures are also required for rotary sealing which can cause the print on the lidding material to lift. Ingress of moisture through the seal mainly through the PVC layer within the base film. Retained solvents used as part of the printing process trapped inside the lidding material which can cause handling problems on line, also a distinct odour can be given off, and more recently problems such as the revised International Conference Harmonisation (ICH) guidelines for the Asian market with the increase in temperature and humidity storage conditions for product on stability.

One of the most critical points of the process is material selection at the development stage of a product. Careful consideration to the requirements of the product is essential, i.e. is the product moisture or oxygen sensitive?, is it light sensitive?. All these points must be considered when selecting the material. To give an example, the aluminium / aluminium blister is the ultimate moisture barrier, it also protects the product from light. Aclar is a clear base film that gives a good moisture barrier, but would not be suitable for light sensitive products. If you need total product protection, or if the product will be exposed to extreme climatic conditions then the tropicalised blister could be considered.

During development it is imperative that the blister layout has been appropriately designed with the maximum of seal area around the pocket, ideally 5mm, with a minimum of 3mm.

One of the most important features of the blister design is the type of knurling pattern used, it can be positive or negative, pyramid or dimple design. It is a very complex and detailed subject that requires the knowledge and expertise of the suppliers.

There are two main types of blister sealing machine, rotary (continuous) motion, and platen (intermittent) motion. There are positives and negatives of both, some say rotary gives a more uniform seal because of the continuous seal roll design, thus giving a tighter seal, some say that platen sealing gives a flatter more cosmetically acceptable blister, giving a stronger peal strength between the lid and the base laminates because of the even distribution of the seal pressure between the top and bottom sealing plates.

The critical parameters of temperature, pressure and dwell time must be optimised to ensure the heat seal lacquer on the lidding material is transferred to the contact layer of the base material (usually PVC). If these parameters are not correctly set it will result in leaking blisters. Gross leakers will be detected during the in-process controls, but leakers due to micro channels through the sealed area will not be detected by the in process checks and may cause problems of stability over time.

There has been many new developments in recent years, such as covert and overt anti counterfeiting features, more child resistant materials, advanced forming technology to reduce the amount of material used and to achieve more complex pocket designs. Pure aluminium blisters to reduce cost. New tool designs to achieve a tighter stronger blister pack, and non-destructive type leak testing to replace the methylene blue dye test. This will allow the product to be re-worked, and hence saving thousands of pounds each year.

Although there have been advancements in the blister sealing process we still need to concentrate our efforts on developing new adhesives, new materials, more efficient seal tools, all with the prime objective of enhancing our blister design to improve the effectiveness of our products.

Keith Allen, Eli Lilly
26/04/2004

Measurement of Adhesive Tack

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The capability to rapidly and reliably bond two surfaces is a key performance requirement in many adhesive products, particularly pressure sensitive adhesive tapes and labels. A key property for successful bonding is the tack or 'stickiness' of the adhesive [1]. Tacky materials can be discerned by touch but for material specification or product development purposes a more quantitative approach is necessary. Tack is defined as the ability of an adhesive to form a bond to a surface after brief contact under light pressure. Often thought of as a simple property to measure, tack depends on a complex interaction of many different factors and, hence, there is no single value that characterises tack. Tack is a complex property depending on the visco-elastic properties of the adhesive, the nature of the substrates and the manner in which the test is performed.

Bond formation depends on:

- Adherend surface properties - material, wettability or surface energy, roughness, porosity.
- Preparation - cleanliness, pre-treatments, coating weight and uniformity, adhesive application, open or drying time, environmental conditions experienced prior to bonding.
- Physical and chemical properties of the adhesive – base polymer type, functional groups, flow properties, surface energy.
- Bonding process - contact pressure, duration of contact, rate of pressure change, thermal history, penetration of the adhesive into adherend surface.

Separation is influenced by:

- Separation process - rate of separation, angle of peel, specimen clamping.
- Mechanical properties of adherends - flexibility, modulus, cohesive strength of surface layers.
- Mechanical properties of adhesive - rigidity, cohesive strength, extension to failure, visco-elastic properties, creep, stress relaxation.

Determined values also depend on:

- Quantities measured –
  - separation force or amount of peel.
  - sampling rate.
  - peak force or area under curve.
- Temperature of the parts.
- Humidity.

Over the years, different methods for measuring tack have been developed. Many of these are unique to the industry where they were developed (e.g. the mandrel test in the footwear industry) but a few have gained wider acceptance. These can be loosely grouped into three classes:

Rolling ball tack (Figure 1):
Higher tack samples halt the ball in a shorter distance. A very simple test to carry out but prone to large uncertainties and the tack results are often confused by other factors. The test is useful from a quality assurance perspective as it should indicate changes in a product coming off a production line.

Loop tack (Figure 2):
Higher tack samples require larger forces or energies to pull a flexible loop of tape from a rigid surface. A reasonably inexpensive test to perform requiring mechanical test equipment common to many laboratories. Comparison of results can be hampered by the complex dependence of local peel stresses on the loop materials.

Probe tack:
Higher tack surfaces require larger forces or energies to pull a rigid probe from the sample. Dedicated probe tack equipment is relatively expensive and inflexible although fixtures for standard mechanical test machines can be used to good effect. This test allows a greater degree of control over test parameters than the other techniques.

The first two types of test have a long history of use in packaging development and quality assurance laboratories. The probe tack method is newer and, as it gives greater control over some of the key test variables, has the potential to provide the most accurate results.

Measured tack values depend very strongly on the test method. Results from DTI funded Materials Measurement research demonstrate that there is often little correlation, even for qualitative ranking, between different measurement methods. Poor reproducibility between measurement laboratories is common despite well-specified tests, which makes specification and control of tack properties difficult.

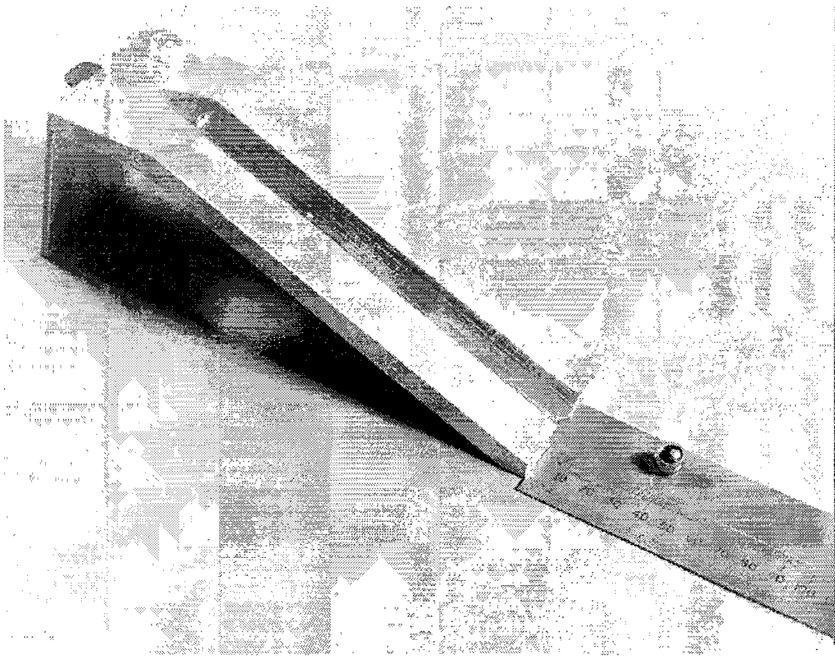


Figure 1: *Rolling ball test*

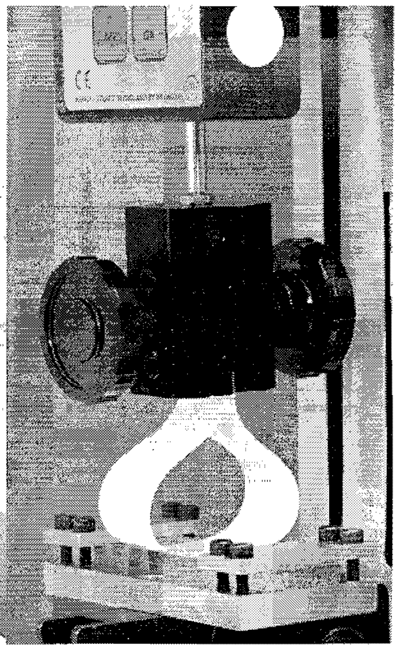


Figure 2: *A loop tack test*

It is often the case that differences between results can be traced back to differences in particular test settings. Understanding these differences provides a means through which measurements can be compared. For example, in one series of loop and probe tack measurements the ranking order of two tapes being studied were reversed. The effects of different test parameters were investigated for the probe tack test. It was found that the adhesives used had different sensitivities to dwell time (the time between contact being made and the substrates being separated). The standard dwell time for the probe tack test was 1 second whilst in the loop tack test it was observed that it took over 10 seconds between the centre of the loop tape making contact and it being pulled off the base plate. The probe tack test results show, in Figure 3, that the rankings for tapes 2 and 3 are reversed between 1s and 10s dwell periods – providing an explanation for the different findings of loop and probe tack tests.

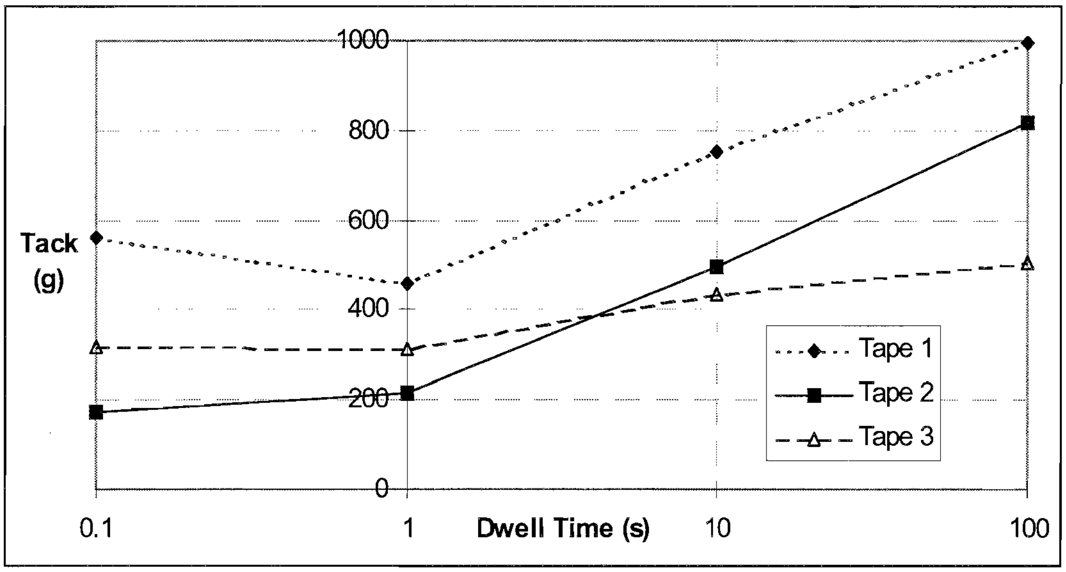
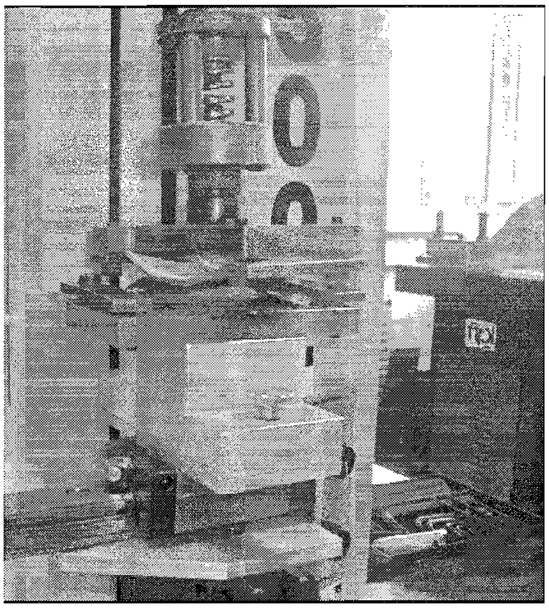


Figure 3: *Influence of dwell time on tack*



In the field of soft metrology, tack is a critical part of the feel of a surface leading to renewed interest in accurate measurements. Work continues to develop robust measurement methods for tack to improve adhesives and application processes. One current area of development is to understand the effects of temperature transients in common bonding processes, such as hot melt adhesive application, where changes in adhesive temperature during the bonding procedure have profound effects on bond strengths. A tack test instrument is being developed to allow more control over critical test parameters such as sample temperature, pressing pressure, dwell time and sample clamping.

Figure 4: *Spotting tack tester jig with test in progress (courtesy of SATRA)*

Reference [1] BC Duncan, SG Abbott and RA Roberts, Adhesive Tack, NPL Measurement Good Practice Guide No. 26, 1999.

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SAA

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