Novel characterisation methods for Polymer Applications

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Introduction

The requirement for novel physical test methods has grown in recent years because as new technology is made available from instrument suppliers.

The aim of this paper is to illustrate the value of non-standard techniques to product screening, investigation and optimization.

A number of new combinatorial techniques are emerging which offer an ability to integrate into a high throughput experimental (H.T.E) work flow [*references*]. However, these techniques can find broader applicability. An instrument which offers a force measurement actuator, allied to a three dimensional position control is capable of a number of test configurations. A configuration has been devised for testing polymer films or in thicker section in indentation, puncture, impact, adhesion or scratching.

Experimental

Specimen preparation

The remit for the mechanical test platform requirement was to accommodate a broad range of materials types and presentation forms. The range specimen formats that can be measured spans free standing films, small cylinders, large coated panels together with a range of combinatorial well-plate sample formats. The materials of interest that have been assessed using this test platform include paint films, coated panels, bulk polymeric components, polymer films starch gels and films, and adhesives (in neat form as well as in adhesive joints).

In order to achieve this range of flexibility, novel sample preparation techniques have been developed. Well plate design to allow thin films to be formed (paints, food starches, adhesives, polymers). Figure 1 shows techniques for preparing high quality test samples in an array, as part of an experimental design for high throughput methods.

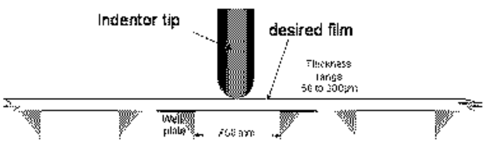

Figure 1. Indentor tip and specimen geometry for puncture testing showing a series of test locations.

Test platform.

An Instron Microtester, which offers excellent vertical axis precision, has been coupled with an XY translation stage, together with dedicated control software to give a test platform. Figure 2 shows the configuration. A number of tests have been made possible for high throughput measurement.

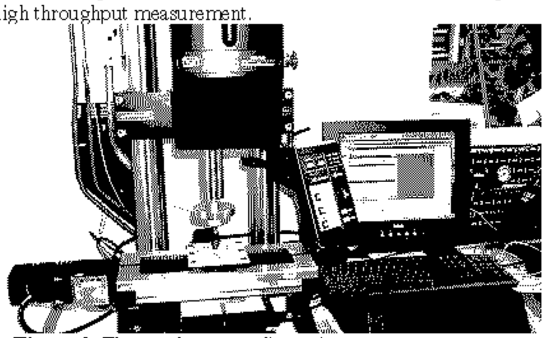

Figure 2. The test frame configuration.

The control of the translation stage to close tolerances allows a mechanical measurement to be taken to within a very small volume of a test specimen. This leads to advantages in taking accurate measurements at precise locations, but also allows novel test geometries or property mapping to take place.

Examples will be given that show the versatility of this testing platform.

Applications

Adhesion testing

An adhesion test has been developed for screening the suitability of conductive adhesives to act as an adhesive to various lead-frame substrate metals. Using combinatorial methods to produce the adhesives of interest has led to the preferred geometry being a blister test.

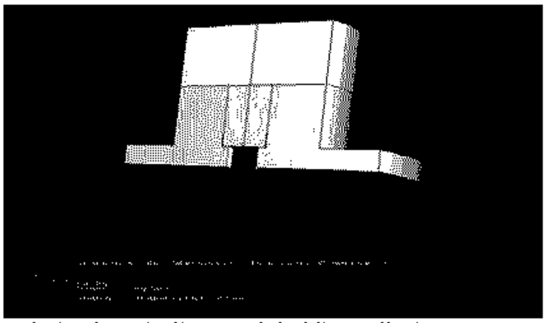

Figure 3. A schematic diagram of the blister adhesion test geometry showing a cross-section through the centre of the indentor, specimen holder and adhesive specimen (grey). The interface of interest is the boundary between holder (orange) and adhesive (grey).

This geometry has the advantage that there is an analysis available for calculating the Interfacial work of adhesion, if required.

The miniature test samples used give highly resolving data [*ref*] and give a range of adhesive and cohesive failure surfaces.

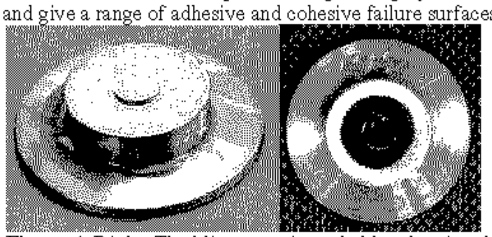

Figure 4. Right: The blister specimen holder showing the central loading plug. Left: A fracture specimen with a mixed mode (adhesive then cohesive) fracture surface.

Some example data for three commercial adhesives of different chemical and physical characteristics is show in Figure 5.

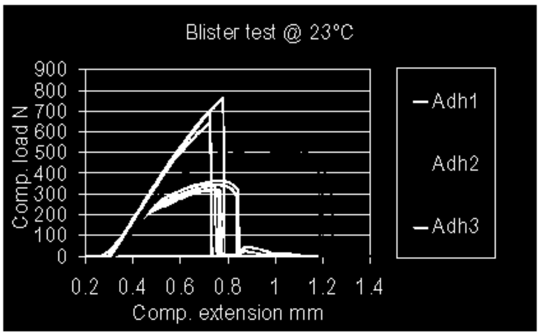

Figure 5. Blister adhesion test data for 3 adhesives.

The test platform that has been developed using the XY translation stage can be used in order to provide sequential testing by mounting an array of test specimens in a known matrix. This could make use of an experimental design with arrays of differing composition or could assess the effect of temperature, ageing, time at temperature and so on.

The development of this approach to combinatorial testing and screening has led to other test geometries. Indeed, automated scratch mapping, compression testing, toughness and puncture tests have all been investigated.

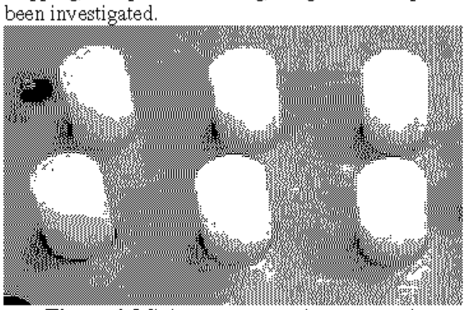

Figure 6. Miniature compression test specimens.

The driving force for developing test methods has been for screening techniques, but in many cases the miniature experiments have shown surprising potential for utilization as characterization techniques in their own right, and in some cases show advantages over the industry standard tests. A good example of this is the puncture test which was originally used as a means to compare film strength. This can be used as a means for characterizing many polymer, paint or other films which can not easily be handled. If a film can be formed in a well then it can be studied as it cures or dries, it can be tested in-situ and no specimen preparation is required whatsoever. This avoids the need to handle delicate or intractable materials, and of course with puncture testing, no specimen preparation or generation of free edges is required at all. This eliminates the concern that stress concentrations at the free edges initiate fracture. The data below shows the consistency of puncture testing for a commercial polyethylene film.

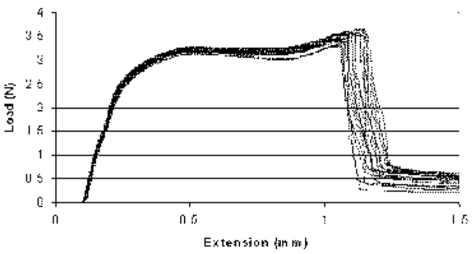

Figure 7. Puncture test data for PE film. Note the consistency in the fracture extension. This is better than many film strength tests.

In the control software development of this test platform, it has been necessary for the test frame to 'sense' surfaces. This has been achieved using the load cell and z-axis motor to work interactively. Machine vision has also been implemented. This allows fast an accurate positioning to achieve register with wells or test specimen location.

The surface sensing capability allows surface profilometry to be possible. It also allows property mapping on a macro scale using indentation. An illustration of this useful spin-off is shown below.

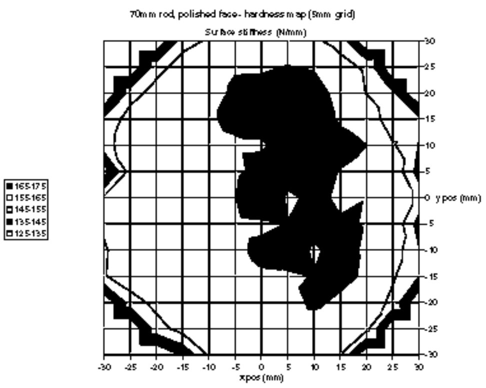

Figure 8. A hardness map for a a polymer component in cross section, showing a harder region.

The property mapping potential using this type of test platform is not yet fully exploited. In the example given, the principle is well established for conventional hardness, indentation and nano-indentation mapping techniques. The resolution using this approach, allied to the versatility of the control software offers some new possibilities. It is possible to carry out toughness or puncture mapping on a film – over a closely packed array of holes.

In this vein, a development version of scratch mapping is in prototype. The scratch map below shows a scratch morphology array using the contact tip geometry (apical angle) and the load applied as the variables.

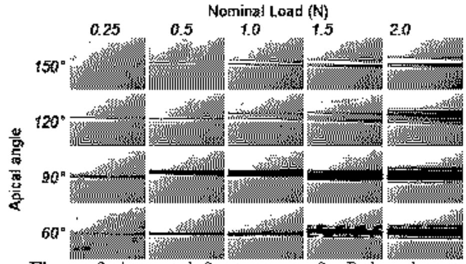

Figure 9. A scratch fracture map for Polycarbonate.

Using the test platform described, it would be possible to generate scratch behaviour (shown in images or as a physical parameter such as scratch hardness) as a function of rate, temperature or as a parameter derived from the force deflection curve. One useful parameter that is emerging as for discrimination is the critical force observed as shown below:-

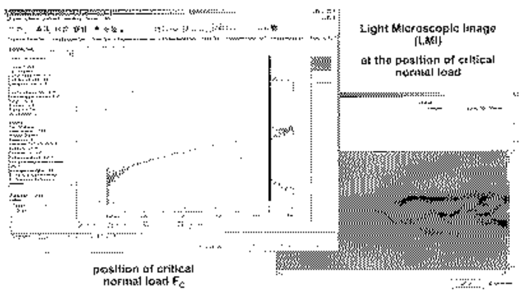

Figure 10. A scratch ramp test curve showing the critical force at the onset of fracture in the polymer being associated with the scratch. (taken from the IUPAC project on 'The scratch resistance of commercial plastics' contribution by H. Steininger (BASF).[ref].

Conclusions

The role of investigative techniques

References

[1] Denton, F. R., III. and Lahti, P. M. in Electrical and Optical Polymer Systems - Fundamentals, Methods, and Applications; Wise, D. L., et al., Eds.; Marcel Dekker: New York, **1998**; Chapter 3.
[2] Ahn, K.-D.; Chung, C.-M.; Koo, D.-I. *Chem. Mater.* **1994**, *6*, 1452.
[3] R. A. Wessling, and R. G. Zimmerman, U.S. Patent **1968**, 3401, 152.