

AVOIDING FAILURE WITH ADHESIVES AND SEALANTS

7th December 2006



**Society of Chemical Industry
15 Belgrave Square London**

www.uk saa.org/

Programme

- 10.00 Registration and Coffee.**
- 10.30 Ensuring correct application through training.
Philip Bell , All Mastic Ltd.**
- 11.00 Surface treatment to avoid failure.
Tony Kinloch, Imperial College.**
- 11.30 Quality assured metering of adhesives and sealants.
Laurie Penn, Failsafe International Ltd.**
- 12.00 Stress and strain limits.
Bob Adams, Bristol University.**
- 12.30 Lunch**
- 14.15 Why floors fail?
Bob Mabbutt, Laybond Products Ltd.**
- 14.45 Test methods and standards.
Bill Broughton, National Physical Laboratory.**
- 15.15 A catalogue of disasters.
John Comyn, Loughborough Universty.**
- 15.45 Discussion, Close and Coffee.**

This one-day symposium is one of an on going series organised by the Society for Adhesion and Adhesives.

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The next symposium is due to be held at the SCI on Thursday 19th April 2007, entitled "Structural Validation and Health Monitoring of Adhesive Joints".

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“Surface Treatments to Avoid Failure”

A.J. Kinloch

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1. Introduction

The choice of surface treatment may play a key role in influencing whether an adhesively-bonded joint will resist the failure loads and environments to which it may be subjected during its expected service life. The present paper will consider two very different examples of where the choice of surface treatment has played such a key role. Namely:

- The bonding of polymer fibre-composites, where the polymeric matrix is based upon a thermoplastic polymer.
- The bonding of metallic substrates, where the adhesive joint has to be durable in the presence of moist environments especially when cyclic-fatigue loads and/or elevated temperatures are also present.

2. The Bonding of Polymer Fibre-Composites

Composites based upon thermosetting or thermoplastic polymeric matrices, and containing glass, aramid, or carbon fibres, are being increasingly used in many diverse applications. The former polymers are usually based upon epoxy or polyester resins and are, therefore, more polar than fluorocarbon polymers or polyolefins. This leads to epoxy and polyester matrices possessing relatively high values of surface free energy and this makes them more receptive to adhesive bonding. This is because relatively high values of the surface free energy of the composite substrate will lead to good ‘wetting’ by the adhesive and to good intrinsic

adhesion via the 'adsorption' mechanism of adhesion. The 'adsorption' mechanism of intrinsic adhesion simply proposes that, providing intimate molecular contact between the substrate and adhesive (i.e. good 'wetting') is established, then the intrinsic adhesion may arise from interatomic and intermolecular forces acting across the adhesive/composite interface. Examples of such forces are dispersion forces, dipole forces, hydrogen bond forces and primary chemical (e.g. covalent bonding) forces [1].

On the other hand, the composites based upon thermoplastic polymers typically employ matrices such as poly(ether-ether ketone), poly(ether sulphone), polypropylene, etc. These polymers tend to have low values of surface free energy and hence are difficult to bond using standard engineering adhesives. Hence, for these composites more complex surface treatments are often needed prior to bonding when using standard engineering adhesives [1-4].

3. The Durability of Metal Bonded Joints

A major concern when using structural adhesives is that the mechanical performance of adhesive joints involving metallic or ceramic substrates may deteriorate upon being exposed to aqueous environments [5-12]. Further, previous research has clearly revealed that it is the interphase of the joint, i.e. the region adjacent to the interface between the substrate and the polymeric adhesive, which is susceptible to such attack and on which attention must be focussed. Also, a common type of mechanical loading encountered by aerospace structures, especially adhesively-bonded components, is cyclic-fatigue loading. For most materials, the presence of this type of loading is found to lead to a much lower resistance to crack growth than under monotonic loading, and polymeric adhesives are no exception to this observation. Thus, as would be expected, the combination of an aqueous environment and cyclic-fatigue loading is a severe test for any adhesive system.

Hence, in the work described in the present paper the cyclic-fatigue behaviour of adhesive joints has been investigated, and an assessment has been made of the durability of the joints under such loading conditions. A typical epoxy-film adhesive, as used by the aerospace industry, has been employed. The fracture energy, G_c , of the adhesive is 1700 J/m². In particular, the effect of using various surface treatments has been studied, namely: (i)

grit-blasted and degreased (GBD); (ii) chromic-acid etched (CAE), (iii) phosphoric-acid anodised (PAA); and (iv) phosphoric-acid anodised with a typical aerospace formulated-primer also applied (PAAP). Fracture-mechanics tests have been used to obtain the relationship between the rate of fatigue crack growth per cycle, da/dN , and the maximum strain-energy release-rate, G_{max} , applied during the fatigue cycle. These cyclic-fatigue tests have been conducted in both a 'dry' environment of $23\pm 1^\circ\text{C}$ and 55 % relative humidity and a 'wet' environment of immersion in distilled water at $28\pm 1^\circ\text{C}$. X-ray photoelectron spectroscopy and electron microscopy techniques have been used to identify the locus of joint failure and the mechanisms of environmental attack.

4. References

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'AVOIDING FAILURE WITH ADHESIVES AND SEALANTS'

SAA/BASA, London – 7th December 2006

Speaker – Laurence R Penn

When any 2K liquid system, such as an epoxy resin, is metered, mixed and dispensed through a conventional machine, can the machine user be sure that, day in and day out, every fraction of the 'make up' of that mixed resin system is correct to specification? And, if so, can they prove it? Also, when automatically dispensing a bead of single component sealant, can you guarantee the output of an exact volume in the right place? Until recently, the answers to these questions were that the machine user couldn't neither prove the quality of the 2K mix ratio or the volumetric mixed output accuracy.

In terms of liquid metering in general and also specifically in regard to 2K metering, mixing, dispensing and application equipment, no-one was able to provide 'quality control and assurance' over liquid metering processes (I suspect that no-one made a 'big deal' over that situation because there was no answer to it!). And in terms of single components, the guaranteeing of the dispensing of the right quantity, and in the right place, was also becoming a major issue, especially in relation to automated production. Also, I am sure that all of us could see that the use of two components was moving towards more and more critical functions and having discussed some of these new applications with manufacturers and chemical suppliers, most of us must have predicted a potential for 2K application failures and some likely application disasters 'down the road'. The key, therefore, was the lack of quality control and assurance over liquid metering and liquid placement accuracy.

And so, in the mid 1990's, and with the increasing use of liquid chemical systems, I came up with the same question over and over again: Why can't we have Quality Control and Quality Assurance over liquid chemical metering processes? Now the term 'Quality Control' is defined as a '**system** for maintaining standards in manufactured products by the testing of a sample of an output against the specification'. And as many of us know, within the context of our specialist field of

liquid use, such a 'sampling' system is inadequate in that the testing of a sample does not represent the testing of all the rest of the product!

And so, back to our Failsafe Metering technology, a good example of a recent application was via our licensee, Naka Failsafe Metering, in Japan. The project is the manufacture of helicopter blades. Their customer's 'quality control' procedure is to 100% x-ray of those composite helicopter blade structures after their manufacture. Whether they intend to X-ray all of their future production is up to them, but at Failsafe, we know that our machines are, continually and totally, checking for, and ensuring, a 100% quality assured metered output from our metering system. The customer's resultant approval of our 'Failsafe' process for this project has led to another application with that same customer, but this time covering the insulation of liquid propane gas tankers.

Another example was with a chemical manufacturer in Switzerland, where the Failsafe Metering process was used to produce a 2K adhesive bead for glazing. This time, their method of checking the 'Failsafe' machine output was by examining the continuity of the bead 'exotherm' via infra-red photography. Once again, this showed our 'Failsafe' system as 100% correct and we appreciated this alternative form of independent confirmation of the Failsafe Metering end product quality.

Failsafe Digital Metering – What was the background to this technology and how did it come about?

In the 1990's, and with some forty years experience within the international field of single and two part liquid chemical system use (epoxies, polyurethanes, silicones, polysulphides and polyesters) and as a pioneer/inventor within the field, including being the founder of Liquid Control in the UK and the USA and having licensed Liquid Control in Asia, I realised that, despite the sophistication of most manufacturing processes and extraordinary as it might seem, there was no such thing as an ability to 'Quality Assure' the automatic measurement and application of low viscosity/solvent thin to high viscosity/putty thick, liquids. As the result of several years of development, and in the year 2000, an entirely new principle of metering, 'Failsafe - Quality Assured Metering', came into being.

Failsafe Metering – How does it work?...

As we know, most liquid adhesives and sealants are not degassed and, therefore, they are compressible. For this reason, the 'Failsafe' principle starts with high pressure liquid input which ensures that the liquid is squeezed to a maximum density per unit volume, prior to it being broken down into adjustably sized, highly accurate, individual dots/small shots. For typical adhesive and sealant applications, the size of a metered liquid dot/shot size may be from 0.01ml to 0.1ml. Each dot/shot is preset and automatically checked for volumetric accuracy. Only if correct, the accuracy check will cause an electric 'OK' signal to be generated. For instance, a typical 2K ratio and output accuracy check rate can be 35 checks per second (beat that if you can!). These high speed metered shots are then fired forward in multiples, so as to form either a larger metered shot or a metered flow rate. Should, just, one shot accuracy check fail, the electronic control system will shut down the metering immediately and before that failure (that is to say the one dot/small shot) can affect any output or end product. Therefore, with two liquid components, the Failsafe Metering system checks 100% of each of the two, metered outputs relative to each other in terms of the correct ratio, and in terms of the correct metered and mixed output flow rate or mixed shot requirement!

Needless to say, we 'went to town' in terms of International patents. Therefore, numerous aspects of this new 'failsafe' metering technology are now either patented or 'patent pending' worldwide.

Now having briefly covered the background and principle of 'Failsafe Metering', I hope you agree that 'Failsafe* Metering' is a logical 'liquid process' requirement which sooner or later, needed to happen. Certainly, our unique capability of eliminating metering errors, and our ability to 'Quality Assure' the metering and placement of single and two component liquid chemical systems, so as ensure their maximum quality of function and conservation, makes perfect sense and has come 'just in time'.

Avoiding Failure with Adhesives and Sealants- Stress and Strain Limits

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Adhesives

- What properties are we looking for?

Joints

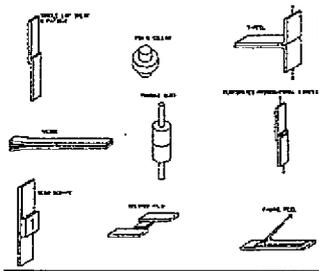


Figure 1: General configurations for the various test methods

Single Lap Joint

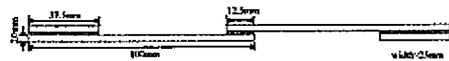


Figure 4-15: Schematic drawing of the single lap joint (not to scale)

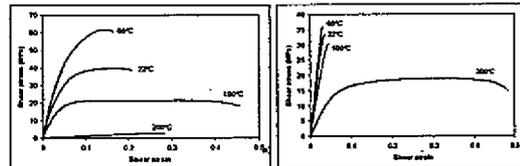
Lap Joint FEA



Figure 1: Lap Joint FEA

Adhesives

- Adhesive shear curves obtained with the TAST: a) Supreme 10HT, b) Redux 326 paste.



What properties do we need for joint strength prediction?

- Sometimes strain to failure
- Sometimes stress at failure
- Usually some type of elastic[ish] modulus

SEALANTS

- Generally much more compliant than adhesives, especially structural adhesives.
- Sealants must
 - Seal
 - Adhere

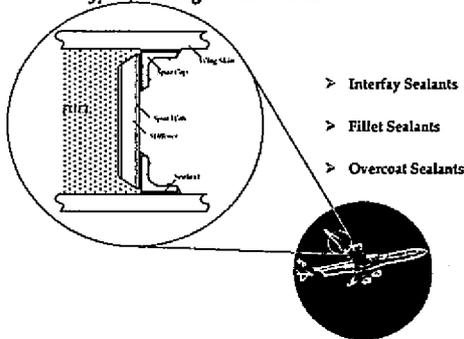
Sealants

- Unless there is a lot of compression, we must have adhesion. Otherwise, we can just use some sort of rubbery sheet.

Sealants

- How to test?
- Example of aircraft fuel tank sealants

Sealant Types for Integral Fuel Tanks



Aircraft Fuel Tank Sealants - General Requirements

- The materials to be used for sealing the integral fuel tanks have to:
- > Adhere to the metal or composite surfaces under any circumstances
 - > Be elastic enough in order to compensate micro-movements of the joint without developing any cracks
 - > Retain their properties in a very wide temperature range -60 °C to +60 °C



Sealants: Elastomeric materials with very low T_g and in general rubber-like behavior

Sealant Testing

Several Tests should be performed to obtain the sealant characteristics

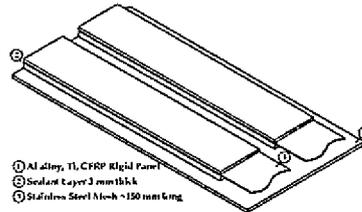
- Tension in order to obtain $\sigma - \epsilon$ data
- Shear in order to obtain $\tau - \gamma$ data
- Single Lap Shear
- Dynamic Mechanical Analysis to obtain T_g and dynamic modulus
- Peel Testing to check adhesion on various substrates

All the tests are performed before or after environmental exposure such as fuel immersion, water immersion and hot air exposure

Peel Testing

Sealant approval process in aerospace industry

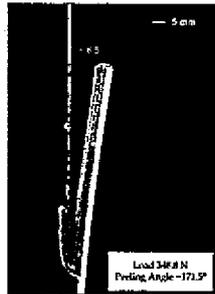
➔ 180° Peel Test



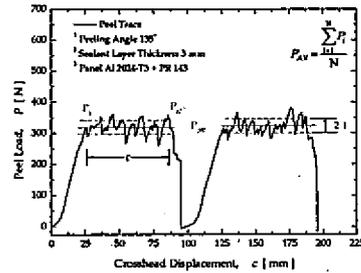
- ① Al alloy, TL CFRP Rigid Panel
- ② Sealant Layer 3 mm thick
- ③ Stainless Steel Mesh - 150 mm long

180 degree peel test

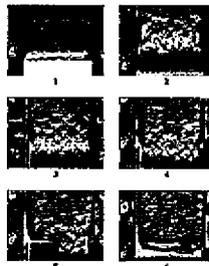
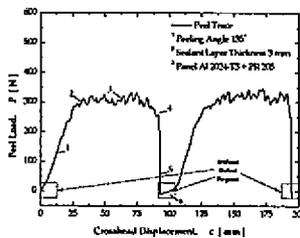
- During test



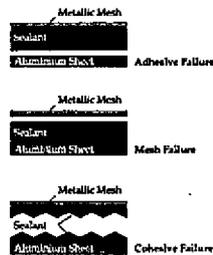
Typical Force-deflection curves



The Role of Artificial Defects



Failure Modes For Specimens on Aluminum Panels



- Cohesive failure in the sealant layer for all the specimens independent of the panel treatment

- Some spots of adhesive failure near the PTFE defect

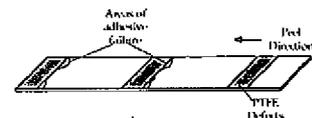


Figure 4.13 Schematic tearing load-versus-the displacement curves for constant rate of extension of a specimen (cf. V. Greenleaf & Thomas (1955)).

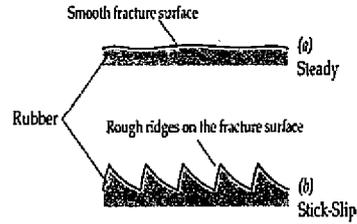
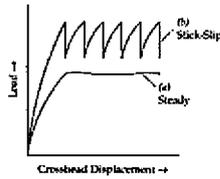


Figure 4.14 Schematic representation of the torn surface of rubber for (a) steady and (b) stick-slip fracture propagation.

Surfaces after failure

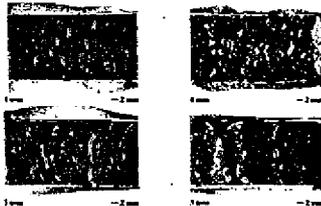


Figure 4.47 Photographs showing the fracture surface of both Sealant A and Sealant B after peeling specimens with various sealant layer thicknesses bonded to painted (Prime-D) aluminum panels at 90°.

Peel force vs sealant thickness

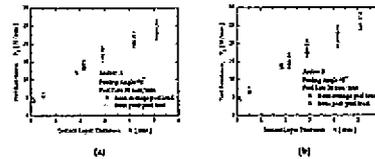


Figure 4.45 Plots of the peel resistance, evaluated from both the average and the peak peel force, as a function of the sealant layer thickness when peeling (a) Sealant A and (b) Sealant B at 90°.

Where does the energy go to?

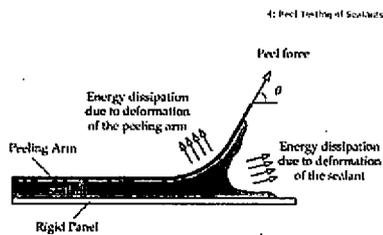


Figure 4.51 Energy dissipation during a peel test.

Peel Energy Calculations

Energy Balance Approach

$$G = \frac{1}{b} \left(\frac{dU_{ext}}{du} - \frac{dU_s}{du} - \frac{dU_{de}}}{du} - \frac{dU_{pl}}{du} \right)$$

dU_{ext} : external work

dU_s : strain energy stored in the peeling arm

dU_{de} : energy dissipated during tensile deformation of the peeling arm

dU_{pl} : energy dissipated due to bending plastic deformation of the peeling arm

Peeling arm (Metallic mesh)

> Infinite tensile stiffness

> Zero bending stiffness



$$G = \frac{P_{AV}}{b} (1 - \cos \theta)$$

Peel energy vs sealant thickness

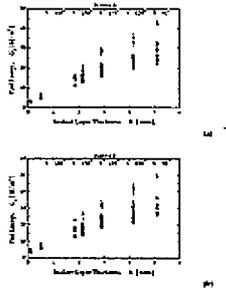
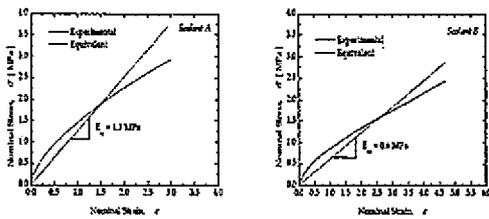


Figure 2.16 Peel energy vs sealant thickness as a function of initial Stress Ratio for various peel angles for all Solvent A and B Solvent B.

FEA of sealant peel test



Sealant properties



Rate Effects

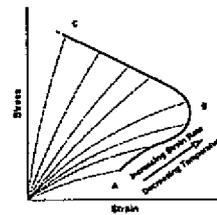


Figure 2.17 Schematic representation of the dependence of stress-strain curves on strain rate and temperature (Redrawn from Smith (1963)).

Stress vs Engineering Strain

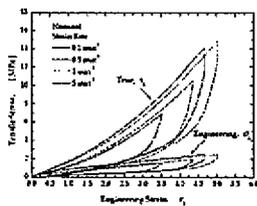


Figure 2.16 Typical loading-unloading stress-strain curves for Solvent B at various strain rates.

Lap joints with sealants

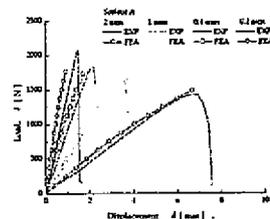


Figure 2.21 Comparison of FE-predicted force-displacement curve and experimental results for single lap joints of Solvent A with various glue layer thicknesses.

Poisson's Ratio

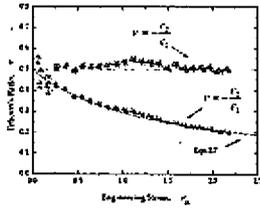


Figure 2.31 Variation of Poisson's ratio calculated from both engineering and true strains with respect to the engineering strain for Soda-Lime Glass.

Sealants

- So what mechanical properties are we looking for in a sealant?
- *Strength?*
- *Ductility?*
- *Adhesion?*

Answer

- All three parameters, but in different proportions

CONCLUSIONS

Sealants

- High adhesion strength is always good
- High ductility is always necessary 300%+
- Sufficient strength is always helpful 2MPa+
- FEA is not so easy

CONCLUSIONS

Adhesives

- High tensile strength is important in structural applications 40MPa+
- Modest ductility is essential 10%+

Thank you

Any Questions ?

Why floors fail

This article is to investigate a specific type of floor failure and enquire why current test methods are inadequate for predicting them.

Design and build

Design and build is the current terminology for an integrated construction project managed by a main contractor from start to finish. Specialists are usually employed to install the components

As with any other fast moving industry, construction techniques are constantly being re-evaluated at the design stage with suppliers identifying opportunities to provide materials they consider, or are proved to be, fit for purpose. The usual criteria apply of cost and functionality under conditions of installation and use, creating the need to share knowledge between designers, installers and manufacturers. Floor design is presently devolved to the structural engineer and to the component suppliers of the multitude of services and decorative features requested by the client. The designer's task is to integrate them.

The floor

The floor is a multifunctional structural component. It is in effect a large laminate with a wearing upper surface and strength to suit its location and purpose. There are many types of floor but for this exercise consider it to be a modern steel reinforced concrete slab, ground supported, suspended, or cantilevered. The steel reinforcement is placed closer to the lower face and the concrete will be cut at regular intervals in the upper face to anticipate cracking due to shrinkage during hydration and moisture loss. The design is robust and follows models created and developed over 150 years. The type of failure under consideration is delamination, the separation of one layer against another, not untypical of spalling where the surface layer separates without noticeable damage to the lower layers.

The construction site

To justify the failures it is necessary to understand the processes involved on a modern construction site. Across Europe there are significant differences in working practice, originating from both tradition and the environment. Although the construction site is now strongly regulated for H&S, there is less protection afforded to the building itself. Although the UK and Ireland suffer more wet or damp weather, the cost of protection is not justifiable and, in common with European practice, there remains a substantial proportion of wet construction technique. Many main contractors are applying risk/cost benefit analysis to manufacturer's specifications because they know that they can save considerable sums of money by imposing restraints on sub-contractors and the materials they use and still have the benefit of transferring experience to subsequent sites.

The building

For the floor there will be several specialist contractors. A typical floor will consist of a power floated steel reinforced concrete slab, a pump applied levelling screed and adhesively bonded floorcovering. The timings of each stage are less important than

the condition (because the floor is a functional component, essential to the construction operation), so measurements and assessments are usually taken at the appropriate time to ensure that adverse interactions are minimised. This inevitably leads to risk assessments by the installer in conjunction with the main contractor. Guidance is given in British Standard installation Codes of Practice (COP's), the architect's instructions, a suppliers specification, advice and experience. For most installations the single largest risk is associated with moisture, either residues in the floor or the damp and cold conditions during the construction.

Generally large scale floor failures are infrequent but following an investigation into the failure of a recently laid floor slab the question raised was why? Considering the abuse a floor receives from start to finish, why do they not fail more frequently, what if anything do current methods of test tell us about the potential of a floor design to become problematical.

Once the question is asked the answers are not obvious, why does one timber floor fail following a flood whilst another, seemingly identical, survives. When a blister appears under the covering, is it the fault of the covering, the combination of materials, the subfloor, the activity above the covering or the construction of the floor itself. Could a thin flexible covering really damage a rigid subfloor. How can you tell whether there is any residual stress or strain in a particular combination and how close is it to failure?

This is not wear and tear, progressive deterioration, it is a fault implicit in the building design, the combination of products and their installation which could contribute to a problem some time, hours, days or even years later.

Differential movement

Differential stress in a building is not necessarily a problem. It accounts for the structural strength of a floor. It benefits a paper based wall covering during the drying process but can lead to joints opening or cracks forming in brittle materials.

Movement in a floor slab is anticipated with expansion joints. For a blister to form there must be stress relief accompanied with differential movement. The possibilities are shrinkage of the lower layer or expansion of the upper layer.

Laboratory modelling

The model selected was a laminated beam which would deflect with stress.

It is well known that hydrating cement products shrink and that calcium sulphate and ettringite will expand during hydration. With a mixed system, such as in a levelling compound, the development of each phase occurs at different times but generally a correctly formulated product remains essentially the same size, or, in jargon, is shrinkage compensated. However in common with many other building materials they reversibly shrink when dry and expand when wetted.

The materials were 6mm smoothing or levelling compound applied as a liquid to a primed and protected steel sheet. The concept was that small changes in dimension would result in curvature of the laminate with steel, which could then be used to measure the forces involved and the long term stability of the products. It should also be possible to replicate the site conditions and identify the risks, especially in the long term when there is the possibility of recrystallisation (reversion) and chemical interference from other building components.

For testing over long periods, 2 years and more, a flexible corrosion protected 0.5 x 50 x 350mm tin-steel sheet was used. For dynamic testing, compressive flexure at 40mm per minute, 1mm x 50 x 150mm steel was selected.

Many different smoothing compounds were cast. Each was held flat on a magnetic table until it had reached Vicat final set then transferred to a climate chamber.

Supported at 340mm centres, deflection was measured at the centre.

Measurements were taken of deflection under self weight or after loading at timed intervals. In early tests the force to restrain was ambitiously measured, but this was abandoned in favour of monitoring deflections, dead load bending, and varying the storage conditions to simulate installation.

By careful examination of the results, it was possible to differentiate between chemical cure and environmental changes. All the test pieces were particularly sensitive to changes in humidity, age usually revealed small changes and temperature variation had very little effect.

(Diagrams)

Reducing the humidity during cure led to permanent deflections. Changes in humidity after initial cure had a significant, reversible effect on deflection. Response was rapid with increasing humidity but slow to reducing humidity.

Loading and stress relaxation curves were obtained by applying a force through a dial gauge to return and maintain the beam to a flat position. The loading curve was logarithmic with time predicting very high forces at short time intervals.

The dynamic tests, carried out at 40mm/min, using the smaller laminate, in compressive flexure, resulted either in cohesive failure just under the surface, like spalling or cleanly at the interface with the steel. In tensile flexure they simply cracked. For a given primer, the controlling factors for delamination at the interface were the thickness of the smoothing compound the degree of deflection and the moisture content of the assembly. There was no obvious correlation with load at failure.

A profile of a floor blister was recorded for comparison with mathematical predictions

Mathematical modelling

Because the deflection in the test laminates is 'self imposed' rather than due to point load, the curvature should be symmetrical. This agrees with profiles measured.

Arguably it can be resolved and represented as a segment of a circle.

The radius, r , is expressed by $(c^2+4h^2)/8h$

Where c is the chord length and h is the perpendicular distance between the chord and the perimeter of the circle.

The subtended angle, a , $=2*ASIN(c/2/r)*(57.2957795)$ degrees

The circumference of the arc can be derived, $=2*3.14159*r*a/360$

A blister

Mathematically the blister can be resolved into a 3 dimensional sinusoidal curve which comprises a central circular segment with two half segments, one on either side. It is arguable that the radius of the centre and side segments are different and that all should be represented by elliptical geometry but this makes the calculations unnecessarily complex and the small refinements add little to the end results.

The central part represents half the blister's diameter and height, easily obtained from practical site measurements, which enables the shrinkage/expansion to be calculated, from the ratio of chord and circumference lengths.

Examples

A flexible tile 500x500mm lays flat on the floor along its perimeter but forms a blister in the centre 5mm above the surrounding floor. The expansion of the tile or shrinkage of the aperture is 0.03% .

A 200 mm floor slab deflects by its maximum design load of span/360 under imposed loads. The difference in length between outer and inner faces is 0.18% whilst the supporting beams would need to move only 0.002% closer to accommodate the sag.

A test piece 340x 6mm deflects 5mm at the centre in a simple arc. The difference in length between the inner and outer faces is 0.167% the difference in aperture is - 0.037%.

Discussion

It is often forgotten that a building is constrained only by itself, gravity and the land it stands on, but suffers internal, external and self imposed stresses. On the basis that internal movement is the result of constrained force, it is not unreasonable to conclude that these forces are significant.

In order to interpret the test results it is necessary to identify the type and extent of movement to which the products are likely to be subjected.

Physical movement due to settlement is an unknown. Compaction or flexure, due to the everchanging weight of the structure and dynamic load deflection due to superimposed loads, will be the largest known contributor. Reversible moisture movement, typically 0.1% for 40%rh differential, represents the next largest source followed by irreversible shrinkage of concrete due to chemical cure, which is claimed to be between -0.03% to -0.08%. Diurnal temperature changes in a building will add to the overall stress in a floor particularly from south facing glazed corridors. It is well accepted that movement between different components will, and do occur and this is accommodated in movement joints.

The test results suggest that under normal conditions the products are well capable of withstanding compression due to load and moisture change on the upper face of a properly prepared concrete slab with a considerable safety margin.

One of the significant characteristics of failure in a building environment has been attributed to slip-stick movement, where stresses are relieved in a short time frame. The forces measured by conventional test methods dismally fail to predict a failure condition. The load-time results from this test programme however show that the safety margin is considerably reduced during sudden small movement. This safety margin is further reduced if the surfaces are not correctly prepared

Smoothing compounds are fluid at the time of installation and therefore flat, at least on the upper face. Samples removed from site are sometimes permanently distorted and the suspicion was that the compound had created this shape by expanding and was therefore the cause rather than the result of failure. The relaxation results following loading in this test work provide further justification that after a failure the stress relief can result in permanent distortion. However that may not be the only explanation. Unsupported prisms when allowed to dry can also 'grow' into new shapes. It is probable that crystal growth in the pores simply follows moisture movement. <http://www.cmse.ed.ac.uk/MatWorld-Mar05.pdf>

In earlier times, the only way of successfully determining fitness for purpose of a primer was to hit an assembly with a hammer, if it shattered off then it was not as good as another which didn't. Dynamic testing in flexure provides a reproducible alternative.

This work has concentrated on a group of polymer modified hydraulic setting products which some may say is not an adhesive. It does however have all the

attributes of a highly filled adhesive. It is a Maxwell material, viscoelastic, which cannot be described by the usual engineering material constants, such as shear or elastic modulus. Some BS standards do recognise the importance of creep but only as a limitation to performance, BS4071 for timber products and EN1902 for floorcoverings. Few standards recognise viscoelasticity as a positive benefit for bonding a product prone to generate considerable stress at the bond line to a rigid base. Adhesives for parquet wood flooring, prEN14293, deals with the concept ungainly by introducing 'shear elongation' and permitting lower dynamic shear strength. The adhesives referred to for bonding timber to concrete are moisture curing viscoelastic products (Laybond 19), which show a leap forward in terms of reduced risk of failure in adverse conditions.

Summary

This test programme behind the review "Why floors fail" reveals that the rate of test is critical to understanding adhesive performance. It examines and mathematically quantifies the dimensions of a floor blister and uses two methods of testing a laminate to demonstrate viscoelastic behaviour in a polymer modified cement, a hydraulic setting product.

RJM, Laybond Products, July 2006

Mathematics of a bulge on a floor

To calculate expansion and % expansion based on a bulge

A bulge on a floor is the result of differential movement between the base and an upper layer. It is fairly easy to compute the expansion necessary to create a bulge if two assumptions are made

- 1 It is symmetrical
- 2 Each component is a segment of a sphere.

Two segments each with half base and half the height can be used to represent the bulge. The difference between the aperture chord length, and the perimeter, arc length, identifies the movement required to create it

The maths for each segment is

			units	Data.	Units
Diameter	d			680	Mm
Bulge height	b			5	Mm
Material thickness	t			6	Mm
The calculation		Formula		Calculation	
segment height	h	b/2	mm	2.5	Mm
Chord length	c	d/2	mm	340	Mm
Outer radius of arc	r	$(c^2 + 4h^2) / 8h$	mm	5781.3	Mm
Inner radius of arc	r _i	r-t	mm	5775.3	Mm
Subtended angle	a	$2 * \text{Asin}(c/2r)$	radians	3.4	degrees
Length of arc	l	$2 * 3.14159 * r * a / 360$	mm	340.0	Mm
Inner length of arc	l _i	$2 * 3.14159 * r_i * a / 360$	mm	339.7	Mm
% change in length top face to bottom	l _i /l-1		%	-0.1038%	
% change in aperture	c/l-1		%	-0.0143%	

The calculation for an elliptical bulge is more complicated (<http://www.1728.com/ellipse.htm>)

The same maths can be used to identify the difference in length between the upper and lower faces or for a simply supported test piece, using c for distance between supports and h for deflection.

Data.	beam length	beam span/360		simply supported
		5000	mm	
	sag	13.9	mm	
	Material thickness	200	mm	6 mm

The calculation						
segment height	h	6.9	mm	4	mm	
Chord length	c	2500	mm	340	mm	
Outer radius of arc	r	112503.5	mm	3614.5	mm	
Inner radius of arc	r _i	112303.5	mm	3608.5	mm	
Subtended angle	a	1.3	degrees	5.4	degrees	
Length of arc	l	2500.0	mm	340.1	mm	
Inner length of arc	l _i	2495.6	mm	339.6	mm	
% change in length top face to bottom		-0.1778%		-0.1660%		
% change in aperture size		-0.0020%		-0.0368%		

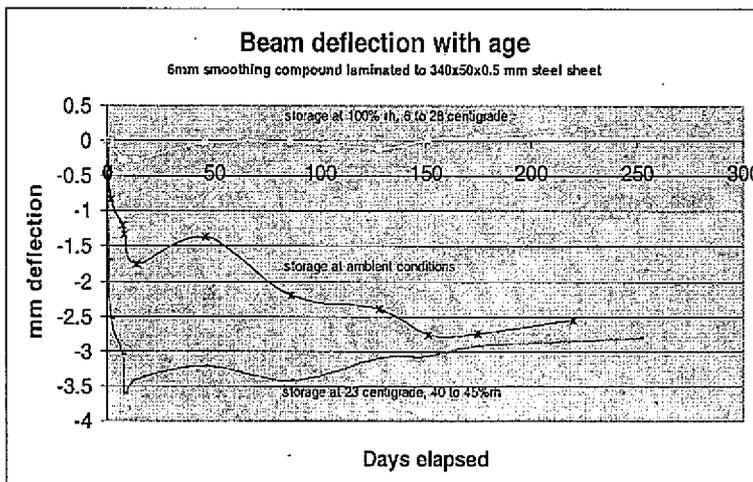
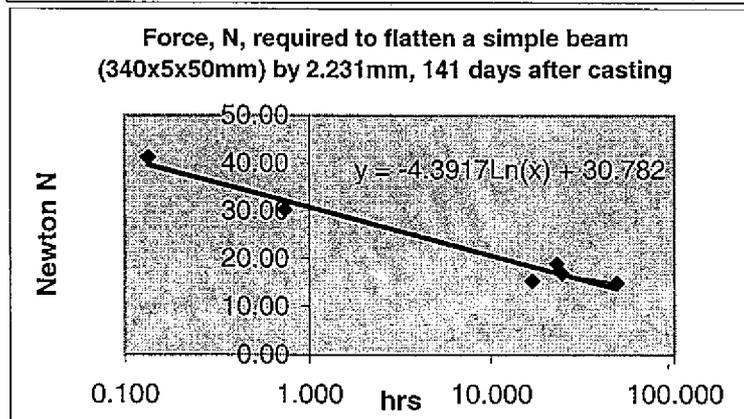
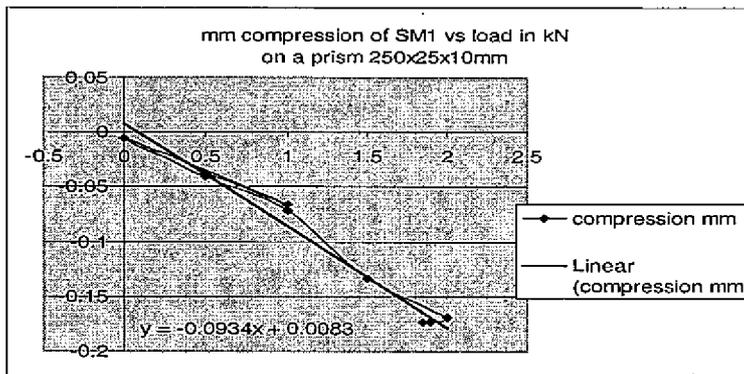
Expansion due to differential thermal movement

Coefficient TE concrete $10 \cdot 10^{-6}$

0.00001 mm per degree C

temperature range	10	degreesC
length	1000	mm
expansion	0.1	
	0.010%	

Longitudinal load compression curve for a smoothing compound prism with 40N/mm² compressive strength



TEST METHODS AND STANDARDS

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INTRODUCTION

Numerous test methods exist for characterising adhesives and bonded joints, and may be used to determine fatigue resistance, environmental durability and creep behaviour. Adhesive tests can be divided into those methods that provide mechanical property data for the adhesive, which aids the selection of adhesives, and those methods that can be used to determine the quality of adhesively bonded structures, and thus aid the design process of adhesive joints. Although an extensive range of test methods is available as national and international standards, most of these tests can only be used for qualitative measurements, providing a means of checking the effectiveness of different surface preparations and comparing mechanical properties of different adhesive systems (i.e. ranking adhesive formulations). A limited number of test methods are suitable for generating engineering data, particularly for determining structural integrity of adhesively bonded structures subjected to static, cyclic and environmental effects. An overview is presented of test methods for characterising tension, compression, shear and fracture toughness properties for design and quality assurance purposes for all stages in the life cycle (i.e. manufacture, assembly, testing and service).

BULK ADHESIVE TESTS

Most of the commonly used test methods are incapable of providing reliable engineering data because the test geometry induces a complex state of stress in the adhesive layer, thus invalidating the results. Two approaches have been adopted in order to overcome this problem. The direct approach is to measure the properties of bulk adhesive specimens. Bulk adhesive specimens can be cast or machined to the required shape (e.g. dumbbell tensile specimens) for measuring tensile, compression and shear properties. It is possible to generate stress-strain curves for all three modes of loading, thus enabling strength, stiffness, Poisson's ratio and strain to failure to be obtained. National and international test methods also exist for tension, compression and shear:

- BS EN ISO 527-2: Plastics: Determination of Tensile Properties. Test Conditions for Moulding and Extrusion Plastics
- BS EN ISO 604: Plastics: Determination of Compressive Properties
- ASTM D 5379: Standard Test Method for Shear Properties of Composite Materials by the V-Notched Beam Method

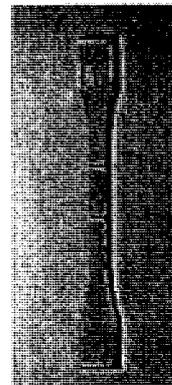
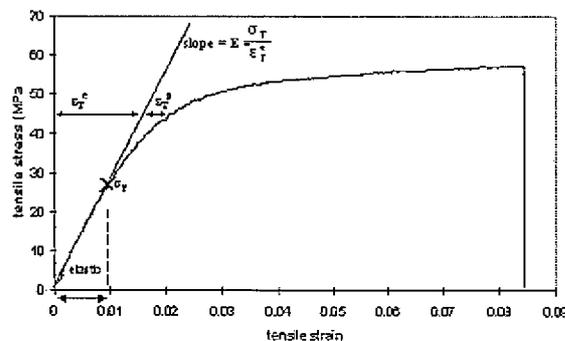


Figure 1 – Dumbbell tensile specimen with typical tensile stress-strain response.

Tensile specimens typically consist of a waisted section with parallel sides (Figure 1) to facilitate strain measurements and to ensure failure occurs within the gauge-section away from the gripped ends. Compression properties can be obtained by loading small rectangular specimens (typically 10 mm x 10 mm and 4 mm thick) between two parallel platens. A number of methods have been developed to measure shear properties including the plate twist, V-notched beam (or Iosipescu) and torsion rod tests. The plate-twist method, developed to measure the shear modulus of plywood, can be used to measure shear modulus ranging from 0.29 GPa to 88.2 GPa. A version of the test method for polymeric materials is now specified in BS EN ISO 15310 (Reinforced Plastics: Determination of the In-Plane Shear Modulus by the Plate Twist Method). The plate twist method is unsuitable for determining shear strength. The V-notched beam method can be used to measure both shear modulus and shear strength. The circular rod specimen can be loaded in torsion to provide shear data and in tension to provide tensile data. The main concern is that the shear stress distribution along and through the specimen under torsion loading is non-uniform. A torsion test machine is also required, a facility not available to most industrial laboratories. Failure mode in torsion as with the V-notched beam method is dependent on the ductility of the polymer. Brittle materials tend to fail in tension whilst thermoplastics undergo shear yielding.

Although bulk adhesive specimens are relatively straightforward to test, there are a number of problems associated with casting or machining bulk specimens (i.e. exothermic reactions, porosity, residual thermal stresses, surface scratches), which may cause premature failure. Strain distribution in the gauge-section is invariably non-uniform. In fact, rapid and large deformation may occur at several locations away from the gauge-section. This is particularly an issue for materials exhibiting large strain to failure, such as toughened epoxies.

ADHESIVE JOINTS

Adhesive joints can be used to provide tensile, compressive and shear data for an adhesive as well as other adhesive property data, such as fracture toughness (Mode I, Mode II and Mixed-Mode I/II), and peel and cleavage strengths (e.g. T-peel, climbing drum test, floating roller method, compact tension and cleavage (Boeing) wedge test). The cylindrical butt joint can be used to test thin bondlines in tension, torsion and compression. The test provides data on the moduli of rigidity and elasticity, and Poisson's ratio. The average strength is taken as the applied load at failure divided by the bond area. Care needs to be taken to ensure good alignment during specimen preparation and testing. A small misalignment can severely reduce strength data. Variations of this test configuration are included in BS EN 26922 (Adhesives: Determination of Tensile Strength of Butt Joints) and ASTM standards.

An alternative approach for determining the shear properties of an adhesive is to apply uniaxial tension or compression load to a specimen consisting of thick, rigid adherends, with a short overlap length. Various thick adherend shear test (TAST) specimen geometries have been suggested, including those specified in BS ISO 11003-2 (Adhesives: Determination of Shear Behaviour of Structural Adhesives. Tensile Test Method Using Thick Adherends). TAST test can be used to generate shear modulus and shear strength data. A quick perusal of the literature and standards will reveal a multitude of shear tests in addition to the TAST, each with the objective of producing a state of pure uniform shear within an adhesive layer (including V-notched beam, Arcan and butt joints).

The most commonly used shear methods are the lap joint configurations (i.e. single-lap and double-lap). These test geometries tend to be used for quality assurance purposes for ranking and comparing adhesive systems and surface treatments. The single-lap and double-lap geometries are unsuitable for generating engineering data. Despite all its obvious weaknesses, the lap-shear test is the most widely used method for producing *in-situ* shear strength data of an adhesively bonded joint. Variations of this test method are included in both national and international standards. Attempts to eliminate eccentric loading, responsible for bending of the adherends and rotation of the bonded region, have resulted in the development of a symmetric variant of the single-lap shear test, known as the double-lap joint. However, bending of the outer adherends is unavoidable, since the load is applied to the outer adherends via the adhesive, away from the neutral axis.

A major consideration in the design of adhesively bonded structures is the possibility of crack growth, either within the adhesive or at the adhesive/adherend interface. Crack propagation can be catastrophic when the fracture toughness of the adhesive/adherend system has been exceeded. Debonds are probably the most life-limiting defects that occur in bonded structures, and may arise during processing or subsequent service. Crack initiation and growth usually occurs under mixed-mode conditions, a combination of Mode I (crack-opening), Mode II (forward-shear) and Mode III (scissor-shear or tear). As a result, fracture toughness tests have been developed to quantify resistance to crack initiation and growth under tensile and shear loading conditions. These test methods are used to assess the influence of adherend surface condition, adhesive, adhesive-adherend interactions, primers, etc., on the fracture strength. Efforts in recent years have been directed towards generating reliable test methods for determining fracture toughness under mixed-mode loading conditions. There are a number of reservations as to the relevance and reliability of fracture toughness data, particularly in applying test results to actual engineering structures. Although serious doubts have been raised, it is recognised that a fracture mechanics approach to studying the failure behaviour of adhesive joints has proved invaluable in understanding the fundamental mechanisms of adhesion. Fracture toughness data obtained from mode I fatigue tests has been used to predict the fatigue life of adhesively bonded joints with good agreement obtained between the theoretical predictions and experimental results.

Double cantilever beam (DCB) test is used to measure the initiation and propagation values of Mode I fracture energy, G_I , under static and cyclic loading conditions. The test is available as British Standard BS 7991 (Determination of the Mode I Adhesive Fracture Energy, G_{Ic} , of Structural Adhesives Using the Double Cantilever Beam (DCB) and Tapered Double Cantilever Beam (TDCB) Specimens) and has been accepted as a new ISO work item. A tensile load is applied to a specimen with an embedded through-width insert (i.e. debond) at the specimen mid-plane. The tensile force acts in a direction normal to the crack surface. Tapered Double-Cantilever Beam (TDCB), also included in BS 7991, was developed with the purpose of making the measurement of fracture energy G_I independent of the crack length (i.e. constant compliance). The specimen design is well suited to tests where the crack length is difficult to measure, especially for determining the rate of crack growth under various cyclic loading and environmental conditions.

No joint geometry has been standardised for Mode II testing of adhesives. Problems still exist. These problems include the plastic deformation of the substrate arms upon testing, the effect of friction on G_{IIc} and the formation of micro-cracks ahead of the crack tip. There are several potential test methods including the end notched flexure (3-point and 4-point) and end loaded split methods. The most commonly used test method for quantifying G_{IIc} for metallic and laminated polymer composite adherends is the end notched flexure (ENF) test, which is essentially a DCB specimen loaded in either three-point or four-point flexure.

CONCLUSIONS

The paper presented an overview of test methods for assessing adhesives and adhesive joints, demonstrating that a comprehensive set of adhesive tests are available to engineers/designers for characterisation of material performance for all stages in the life cycle (i.e. manufacture, assembly, testing and service). More details on standards and test methods for both design and quality assurance purposes can be obtained by visiting the Adhesive Design Toolkit website (<http://www.adhesivestoolkit.com>).

ACKNOWLEDGEMENTS

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A CATALOGUE OF DISASTERS

J Comyn

IPTME, Loughborough University

WRONG ADHESIVE

AUTOMOTIVE HEADLIGHTS

Polystyrene-polyester body bonded with UV-cured acrylic adhesive to a polycarbonate lens.

Adhesive contained

aluminium hydroxide as a filler and whitener

polyethylene glycol, as a plasticiser. MWt ~ 600.

Lamps failed by steaming up. The Achilles' heel is PEG

Firstly it will tend to adsorb on the surfaces of the $\text{Al}(\text{OH})_3$ particles by H-bonding.

Secondly although soluble in the monomers which constitute the uncured adhesive, it will separate out from the cured material.

Basic thermodynamics – polymers are incompatible due to low entropy of mixing.

ALUMINIUM BONDED TO WOODEN HOUSES

Al foil bonded to long planks with a solvent based polychloroprene.

Stabilising additives were MgO, ZnO (acid absorbers) and BHT (antioxidant).

Al corroding and delaminating.

Adhesive applied to both Al and wood – after partial drying IR heated to increase tack just before bonding. Basic problem is that BHT evaporates with solvent. The polymer degrades with release of chloride ions.

ADHESIVE FOR EXPANDED POLYSTYRENE

Synthetic latices for binding expanded polystyrene (EPS) beads. All copolymers of vinyl acetate.

Made a brick and then broke in 3 point loading.

Mean failure loads (N) after water immersion for 7 days were

A	9.0
B	8.3
C	1.8
D	0
E	10.6
F	7.2

Used contact angles to investigate EPS and dried films of adhesives.

	Water Contact angle °	dispersive component mJm ⁻²	polar component mJm ⁻²	Work of adhesion mJm ⁻²
A	74	16	12	47
B	67	16	19	36
C	65	12	24	28
D	16	5	69	-17
E	55	6	38	11
F	55	6	38	11
EPS	101	0.4	26	

XPS – Except for D, all showed only C and O (does not detect H). D shows 5.2% potassium.

J Comyn, D C Blackley and L M Harding, *J Adhesion* **40** 163 (1993), *Int J Adhes Adhes* **13** 163 (1993), **12** 263 (1992).

DECORATIVE LEAD STRIP FOR WINDOWS

Lead strip bonded to interior of a double glazing unit.

Adhesive clearly degrading (cracks and discolour) and lead falling off.

Reason – Styrene/butadiene adhesive (containing C=C bonds) and with no UV stabilizer.

THIXOTROPY

Free flowing epoxide adhesives just running away.

Barium sulfate as filler.

Aluminium security door, internally bonded, adhesive running everywhere.

DOUBLE GLAZING

Edge seals were traditionally of two-part polysulfide, but now two-part polyurethanes and thermoplastic polyisobutene are used. Their purpose is to hold the unit together and act as a permeation barrier to water. The spacer tube contains a desiccant (zeolite molecular sieve) to absorb any water left at the time of manufacture, and any which permeates the seal.

Calculations show that if the seal remains intact the life of the unit will be long (50-100 years).

Failure of the seal is easily seen through the glass. Wet air will enter through the smallest debond, assisted by pressure changes. It will soon saturate the desiccant and condensation on the inner faces will be seen.

I have seen failures due to plasticiser migrating from a polysulfide sealant into the surrounding glazing compound (linseed oil + calcium carbonate + polyisobutene). Results in shrinkage and debonding.

THERMAL MISMATCH

Differences in the coefficient of thermal expansion of adherends are not a problem when the latter are measured in cm, but can be a problem when measured in m.

This was a problem with

aluminium bonded to wooden houses

polypropylene sheet bonded to the walls of a swimming pool.

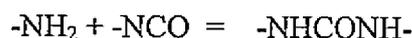
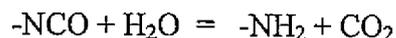
One double glazing failure was due to the outer sheet being tinted and therefore heat absorbing, and a rigid epoxide being used to bond the spacer tube to glass.

Calculations in all these cases show large forces.

PREMATURE CURE

An isocyanate adhesive was being used to bond steel sheet to wood.

Spray the adhesive to both adherends, then spray some water.



No control exercised over open time and temperature.

SILICONE

CONTAMINATION

Frequent occurrence. Silicone fluids seem to get everywhere. Contamination easy to detect, source sometimes difficult to trace.