Society for Adhesion and Adhesives

SEALANTS

One-day Symposium 7th December, 2000

Society of Chemical Industry, Belgrave Square, London

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Programme

10.00	**Registration and coffee**
10.30	**Sealing and re-sealing of buildings** A R Hutchinson and T G B Jones, (Oxford Brookes University)
11.00	**Influence of fuel, water and water/FSII combinations on the mechanical properties of aircraft fuel tank sealants** P W Duke, J Day and S J Shaw, (DERA Farnborough)
11.30	**Development of a durability test method for curtain-wall sealants** A T Wolf (Dow Corning S A, Belgium)
12.00	**The durability of sealed insulating glass units** S L Garvin, J Ridal and M Phillipson, (BRE East Kilbride)
12.30	**Lunch**
14.15	**Usefulness of contact angle measurements for understanding the adhesion of Aerospace sealants to aircraft substrates.** P Bons, (Chementall GmbH)
14.45	**Sealants in the automotive industry** M Lavery, (Evode Limited)
15.15	**Sealant fracture; multi-technique analysis of the interface** S J Harris, S R Church and M A Taylor, (BAE SYSTEMS)
15.45	**General Discussion**
16.00	**Tea**

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

Society for Adhesion and Adhesives Committee

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The next symposium due is "Pressure Sensitive Adhesion" to be held at the SCI on Thursday 25th April 2001.

Details from

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What is the SAA?

The Society for Adhesion and Adhesives was inaugurated in July 1999 and officially launched at the Institute of Materials' (IoM) Congress 2000 in Cirencester, on 13th April 2000.

The Society is based at The Institute of Materials in London and is associated with The Welding and Joining Society [WJS], The Macro Group [a joint body of the Royal Society of Chemistry and the Society of Chemical Industries], The Adhesion Society Inc. in the United States of America and the Beijing Adhesion Society.

It is intended that this Society will be a focus for all those with an interest in the science and technology of adhesion and adhesives. To this end, it will organise international conferences and seminars on all matters related to these fields. These will include triennial Adhesion and Structural Adhesives in Engineering [SAE] conferences and the seminars and workshops traditionally organised by the Adhesives Section of the IoM and the Adhesives Technical Group of the WJS.

What are the benefits?

- Initially, all members will be entitled to free membership. A small membership fee may be charged when the SAA is fully operational but this will not happen before April 2001
- Access to a dedicated website with worldwide links to other major societies and organisations which deal with adhesion and adhesives [Website address is: http://www.me.ic.ac.uk/materials/SAA/SAA1.html]
- Reduced fees for SAA-organised conferences and seminars
- An annual list of members, including affiliations and addresses
- Two SAA newsletters per annum

Who can join?

Anyone who has an interest in the science and technology of adhesion and adhesives.

How do I join?

- Fill in the attached application form and send to: Mr. Malcolm Bowditch, 45 West Way, Broadstone, Dorset, BH18 9LW, UK - Telephone/fax: +44 (0)1202 696610 or e-mail: malcolmbowditch@45westway.freeserve.co.uk

  Or

- Register through the website.

Society for Adhesion and Adhesives

Application for Membership

please complete the following:

Title:..Name: ....

Date of birth: ....

Highest academic qualification: ....

Are you a member of The Institute of Materials ☐ yes ☐ no

Are you a member of any of the associated societies:

☐ WJS ☐ Macro Group ☐ Adhesion Society Inc.

Home address (optional): ....

....

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email: ....

Business details (if applicable):

Job Title: ....

Organisation: ....

Address: ....

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email: ....

Declaration

☐ **I wish to join the Society for Adhesion and Adhesives**

I do not wish the following to be published in the Member' List:
(tick item/s which is/are applicable)

☐ Home ☐ Business

Signature .... Date ....

SEALANTS SEMINAR

Society for Adhesion and Adhesives
7th December 2000

SEALING AND RESEALING OF BUILDINGS

A. R. HUTCHINSON and T.G.B. JONES
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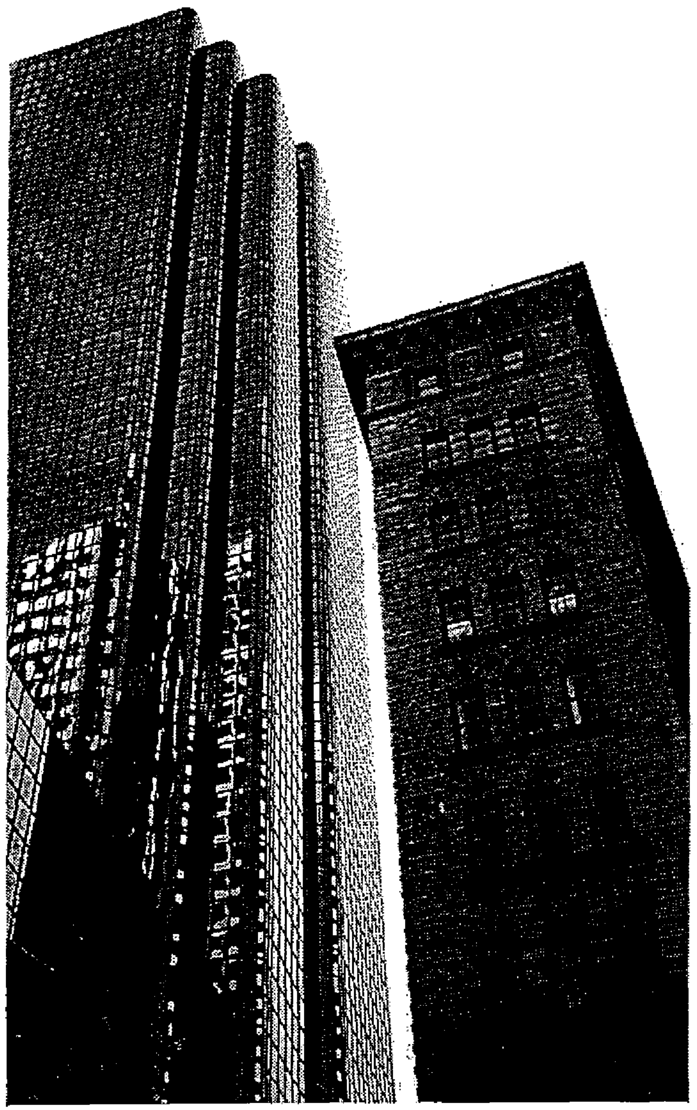
Abstract
The Joining Technology Research Centre has been involved with research into, and the testing of, wet-applied construction sealants since the late 1980's. This paper will focus specifically on factors affecting sealed joint performance in the context of both new sealing and resealing.

Seals and sealed joint systems

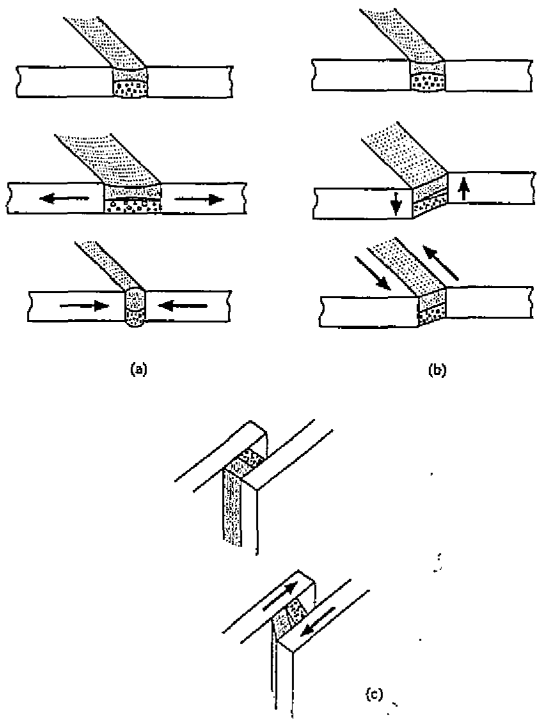
Sealants play a vital role in maintaining the weather tightness of buildings and engineering structures. Buildings and structures comprise many different components and materials, giving rise to joints or gaps between different elements. Joints located between either similar or dissimilar materials in a structure must be designed to enable movements to take place between its component parts. They should not be regarded as convenient discontinuities for a contractor to accommodate tolerances, or as unfortunate gaps to be made as small as possible or even invisible. Joints represent vital parts of buildings and structures which need to be designed, constructed and sealed in a professional manner if they are not to become the weak link in the performance of that structure. Contemporary façade design actually reduces the dependence on sealants for the principal waterproofing function by careful joint design; nevertheless, sealed joints continue to play a vital role, both as primary and secondary barriers.

In traditional forms of construction for buildings involving massive component parts, movement was accommodated by minor cracks and fissures in thick sections or by design features such as overhangs and flashings (Figure 1b). Oil-based caulks such as bitumen or putty were sometimes used to fill cavities to prevent leakage. The modern building sealant industry is a post-Second World War phenomenon because contemporary structural frame buildings are characterised by lighter, thinner, and generally larger external sections such as the elements of a curtain wall (Figure 1a). The generally frequent cyclic movements at joints must be dealt with by sophisticated synthetic sealant systems. The cost of sealing buildings as part of new build represents typically about 3% of the cost of the curtain walling itself; however, the cost of resealing is of the order of 15% because of the complexities of access and material application.



(a) Modern curtain walling (b) Traditional construction

Figure 1. Modern and traditional forms of construction



(a) Butt joint in tension/compression; (b) butt joint in shear; (c) lap joint in shear

Figure 2. Movement in different types of joint.

Joints in buildings and structures today may be sealed by one, or a combination, of three techniques:

- wet-applied sealants
- pre-formed gaskets
- cellular sealing strips or tapes.

This paper is concerned primarily with wet-applied sealants and their durability in joints. However a sealed joint comprises two major elements: the gap into which the sealant system is placed and the sealant system itself. The performance of a joint and the durability of the seal is dependent upon both, and may be reduced significantly by the use of an inappropriate system, incorrect joint detail or by poor application and workmanship.

The gap between the two similar or dissimilar surfaces in a joint requires serious attention in design and specification. For instance, wet-applied sealant must adhere to the relevant surfaces; this generally requires the need for a good standard of surface preparation to be achieved. Sealant systems comprise the sealant itself, a primer (if applicable) and a backer rod or bond breaker tape; joint fillers may also be used in combination with a bond-breaker or back-up material. The backer-rod controls the depth and geometry of seal, provides support for tooling the sealant, and should serve as a bond-breaker for preventing sealant from adhering to the back of a joint; two-sided adhesion only is essential if the seal is to accommodate appreciable movement.

Sealant systems do not have an indefinite life span, so that resealing of joints needs to take place a number of times within the life-time of a building or structure. Resealing is therefore a widespread and continuous activity, accounting for up to 75% of all sealant usage.

Movements in buildings and facades

Movement occurs in all buildings and structures. Modern buildings are characterized by relatively lightweight construction involving new materials and innovative design, and this has given rise to greater emphasis on the design and integrity of joints which can accommodate significant movement. The heavily worked external sealant joint in a building is subject to continual motion as well as various physical and chemical degradation mechanisms, and the life expectancy of the sealed joint is usually less than that of the building façade.

Movement is caused by a variety of effects related to materials, construction, weather and use. Movement takes place in various ways depending on the causes, which include settlement, shrinkage, creep, thermal expansion and contraction, loading, wind and others. Most joints in a building structure are subjected to several types of movement, both separately and in combination, one of which may be initial shrinkage movement.

Movement due to thermal effects can be very significant in joints in curtain walling. The magnitude and rate of movement depends on factors related to the curtain walling (colour, mass, size, etc.), the compass orientation, the time of day and the season.

The movement potential of a joint is difficult to determine accurately. Estimates may be based upon theoretical structural movements of the materials involved, together with experience of the type of construction and anticipated construction tolerances. Butt joints are very common and are generally considered to be the most critical form of joint in terms of the consequences of failure; thus, most estimates of joint movement are based on the geometry of butt joints. Sealant materials in butt joints tend to be in compression when it is hot, and in tension when cold.

The authors studied the joint movements on a concrete-clad building and on an aluminium-clad building in London over an 18-month period in 1996/7. Weather data were also collected for the same time. Some typical data for a vertical joint in the aluminium-clad building are shown in Figures 3 to 5.

Requirements of sealants

Sealant joints must satisfy many requirements in a building structure, providing a seal throughout its design life and under all combinations of joint movement. Since joints in the building envelope are often in highly visible areas of the building façade, there are also a number of aesthetic considerations related to joint design and the appearance of the seals. The requirements of sealants comprise their application and curing characteristics, adhesion abilities, movement-accommodation characteristics, resistance to environmental factors, retention of aesthetic qualities, and their durability. Typical base polymers used today for applications in building envelopes are acrylics, polysulphides, polyurethanes and silicones, including blends and modifications of these materials.

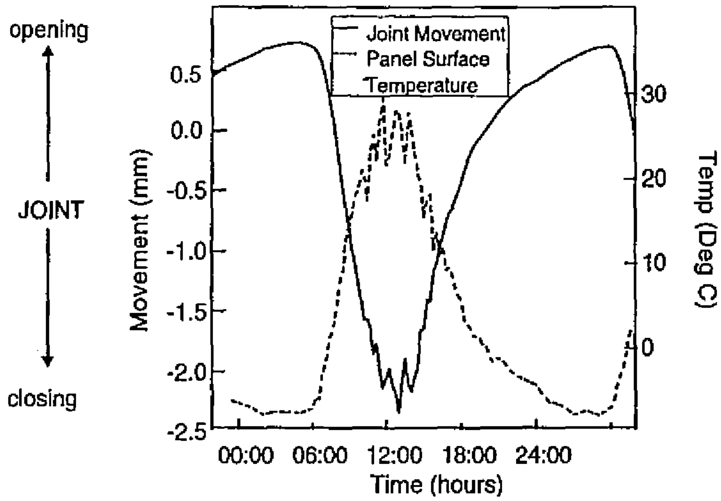


Figure 3. Springtime temperature and movement for aluminium cladding

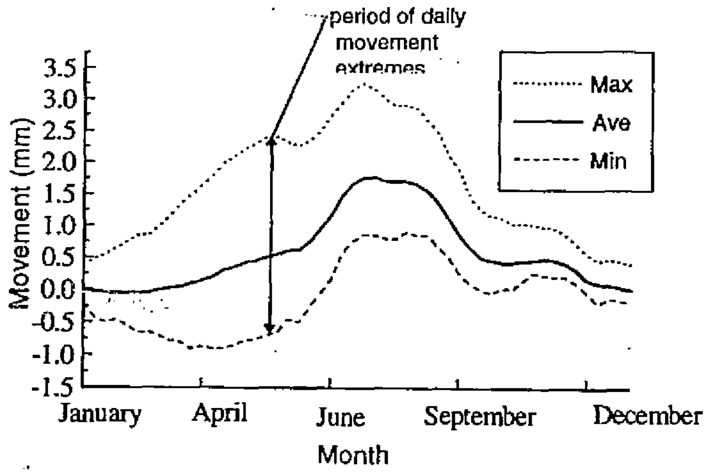


Figure 4. Annual pattern of joint movement for aluminium-clad building

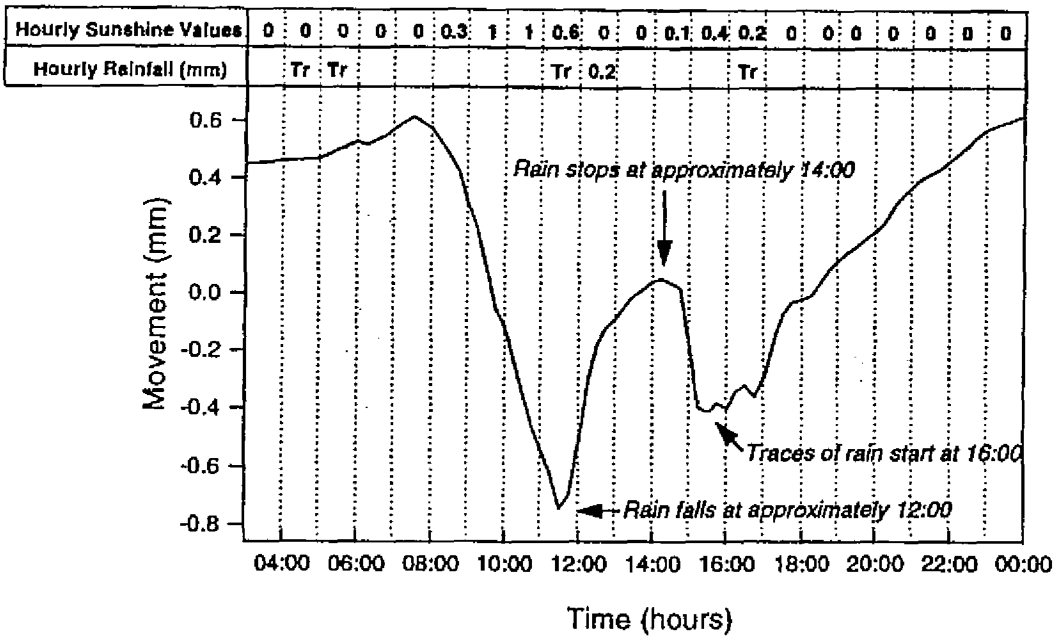


Figure 5. Daily pattern of movement for aluminium-clad panel joint

Performance-based testing and selection, as embodied in ISO 11600, should ensure that the most appropriate selection for different application and post-cure movement accommodation requirements.

Sealant installation

It is essential that the constituent parts of the sealant system are handled correctly and applied to a high standard by trained installers. In addition to fundamental design flaws, poor workmanship is responsible for a large number of failures. Some of the key issues leading to premature joint failure highlight the factors involved in installation. These include: lack of training; poor cleaning of joint surfaces; lack of priming; inadequate sealant bead depth control; incorrect backer rod; poor mixing of multi-component products; lack of tooling, and; lack of protection during cure.

Specification

A detailed specification represents one of the cornerstones for ensuring adequate quality; without it, early sealed joint failure is likely to occur. The specification should include the aspects of: joint sizes; requirements for cleaning and preparing the joint surfaces, including methods for removing existing sealants (if appropriate); priming; type and geometry of backer rod; sealant specification, and; tooling requirements.

The key materials elements which comprise the sealed system are the primer, sealant and backer rod. Suitable sealants should be tested to ISO 11600 performance requirements as a starting point. Durability tests should then be undertaken used parameters relevant to the intended application.

Further reading

BS ISO 11600 (1993) *Building Construction Sealants. Classification and Requirements*, London

CIRIA Report 178 (1998) *Sealant Joints in the External Envelope of Buildings*, Construction Industry Research and Information Association, London

WOLF, A T (ed.) (1999) *Durability of Building Sealants*, 2nd Volume, Proc. Int. RILEM Symposium, E & F Spon, London

WOLF, A T (ed.) (1999) *Durability of Building Sealants*, A State-of-the-Art Report, No. 21, RILEM Publications, Paris

WOOLMAN, R and HUTCHINSON, A R (1994) *Resealing of Buildings: A Guide to Good Practice,* Butterworth-Heinemann, Oxford

Influence of fuel, water and water/FSII combinations on the mechanical properties of aircraft fuel tank sealants

P W Duke, J Day and S J Shaw (Structural Materials Centre, DERA Farnborough)

For reasons primarily associated with both weight considerations and fuel capacity, modern aircraft are usually designed to carry fuel in integral tanks rather than bags, with elastomeric sealants employed to seal mating surfaces, mechanical joints etc. Unfortunately, experience with many aircraft types has shown that this approach to fuel carriage can result in substantial problems regarding fuel leakage.

Although several types of elastomer composition can be considered for applications requiring resistance to hydrocarbon based fuels, those based on polysulphides are currently employed in most fuel tank sealing operations. Newer compositions based on polythioethers have been formulated and are now in common usage. Although these sealant types have a sound reputation for resisting hydrocarbon liquids, the continuing fuel leakage problem has necessitated a detailed consideration of this troublesome issue.

In considering the question of fuel leakage, it is important to recognise that the environment in which the sealant is required to operate is an extremely harsh one. For example, in addition to the obvious presence of aviation fuel, other potential degradants will include water and icing inhibitors contained within the fuel, the effects of which will be superimposed on temperature extremes and dynamic fatigue loading. Under such extreme conditions, failures associated with either degradation of the bulk sealant and/or the sealant/substrate interface have been observed in practice.

In an attempt to generate a greater understanding of the effects of these principal fuel tank fluids, research has been carried out to assess the extent to which each of these fluids is absorbed by typical fuel tank sealant materials, together with their effects on bulk sealant properties. Four types of sealant were investigated, two being polysulphide formulations, the third a modified polysulphide and the fourth a polythioether.

Some of the results obtained from this study are presented and discussed.

DEVELOPMENT OF A DURABILITY TEST METHOD FOR CURTAIN-WALL SEALANTS

Andreas Wolf, Dow Corning S.A., Belgium

Abstract
The paper discusses the work carried out over the past decade towards the development of a durability test standard for sealants, especially the efforts undertaken by the ISO TC59/SC8 and RILEM TC139-DBS committees. The progress made in the development of a test method is being discussed and specific recommendations are made for a future durability test standard.

1. Introduction

Over the past two decades, the sealants industry has undergone rapid technological and structural changes. On the one hand, advancements in technology have enabled the launch of a multitude of new sealant products based on novel polymers, cure chemistries and formulations. On the other hand, increasing competitive pressure and customers that are more demanding have required shorter product development cycles. Unlike the well-established sealants, which have been sold for more than twenty years based on the same formulation, the new sealant products do not have well-established performance histories. At present, generating a reliable performance history for a new sealant product still requires long-term outdoor testing and extensive in-service field evaluations. Attempts at avoiding this tasks, by employing various forms of short-term laboratory-based ageing tests, have had limited success and are viewed with suspicion by construction specifiers, mainly because of the lack of an established correlation with the actual in-service performance of sealants.

Thus, the sealants industry urgently needs a method for generating long-term performance data rapidly and with assured reliability. Accelerated laboratory ageing experiments are the most promising method for acquiring durability information within the shortest possible time; however, a methodology for conducting and interpreting these experiments needs to be developed that improves the predictive value of this technique.

The need for improved longevity of sealed joints is well recognised. Work undertaken by RILEM Technical Committee 139-DBS "Durability of Building Sealants" during the period 1991-2000 has helped progress towards the development of testing and classification standards for the durability of building joint sealants. Information has been gathered in the following areas: factors causing degradation, correlation of artificial weathering and outdoor exposure results, and suitability of laboratory based methods for predicting the service life of sealants. These contributions are in direct support of the work of ISO Committee TC59/SC8 "Jointing Products" and will be eventually allowing the development of an International Test Standard on Durability of Building Sealants.

2. Objectives of RILEM Technical Committee 139-DBS

RILEM TC 139-DBS was inaugurated in 1991. The objectives that RILEM TC139-DBS set itself are to review and disseminate the present knowledge regarding the assessment of the durability of sealants, to promote research in this field, and to make recommendations for suitable experimental methods. These objectives are being achieved by preparing a state-of-the-art report and conducting international symposia, by conducting integrated research programmes, and by preparing a RILEM Technical Recommendation (RTR) on a sealant durability test method in close liaison with the ISO standardisation activity.

3. Development of an accelerated durability test method

Work towards an accelerated durability test method was started in 1989 within the International Standardisation Organisation Committee ISO TC59/SC8 (Work Group 6). Later, in 1994, the activity was transferred to RILEM TC139-DBS. The committee has now developed a RILEM Technical Recommendation (RTR) [1], which will be considered by ISO for the development of a future durability test standard. The purpose of this technical recommendation is to provide a framework for assessing the effects of cyclic movement and artificial weathering on curtain-wall sealants in a laboratory-based procedure.

During their entire service life, joint seals are exposed to cyclic mechanical strain and environmental degradation factors. Cyclic joint movement, sunlight, temperature variations (heat, cold) and moisture (water) are considered to be the primary environmental and service degradation factors leading to sealed joint failure. Weatherproofing joint seals in lightweight building façades (curtain-wall cladding) are exposed to frequent cyclic movements. This joint movement imposes cyclic mechanical strain on the seal, which, depending on the exposure conditions and the construction design, can vary substantially in rate and amplitude.

A first test method was devised in 1998. Research by Oxford Brookes University (UK) [2,3] on the effect of early joint movement on the performance of sealed joints led to the incorporation of a 'dynamic cure' conditioning procedure in the test method. In the final RTR method, sealant specimens are cured either statically (no movement) or dynamically (exposed to cyclic movement). The cured sealant specimens are then exposed to repetitive cycles of artificial weathering (light, heat and moisture) and cyclic movement under controlled environmental conditions (degradation cycles). Weathering is carried out in an artificial weathering machine. This is followed (optionally) by rapid mechanical fatigue cycling. Then the specimens are exposed to the two thermo-mechanical cycles as defined in ISO 9047 (section 8, first week) [4], using the full amplitude suggested as the movement range of the sealant under test. After completion of each degradation cycle, the specimens are extended to their full rated extension and held there as the sealant beads are visually examined for changes in appearance, cohesion and adhesion. The depth of any cohesive or adhesive flaw is determined according to the rules provided in ISO-DIS 11,600 [5] and the general condition of the sealant is reported. The weathering exposure, the cyclic movement, and the examination for failures constitute a degradation cycle and the degradation cycle is repeated as often as desired to achieve a certain exposure (see Figure 1 for a schematic representation of the test method).

The use of this method is intended to induce property changes in sealants associated with typical end use conditions. The repeated exposure of sealant specimens to cycles of artificial weathering and cyclic movement is meant to simulate a natural weathering environment of sealants installed in curtain-wall joints exposed to high joint movement.

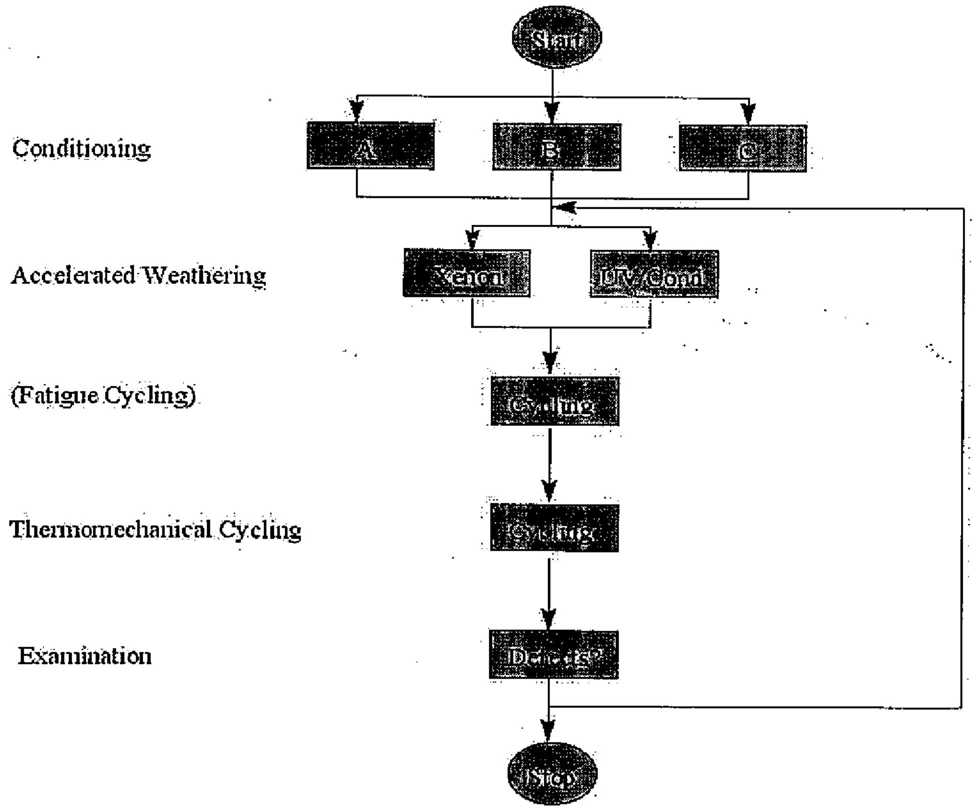


Figure 1. Schematic flow-chart of test procedure

The newly developed RILEM Technical Recommendation is based on the expertise and experience of the many researchers involved in sealant durability research. While this test method is still based firmly upon the current state-of-the-art, it allows test results to be generated, which can be utilised in the future development of a service life model. What is needed now is a close co-operation between academic and industrial research aimed at improving our understanding of cumulative damage models for sealed joints.

4. References

1. Anonymous, 'RILEM TC 139-DBS: Test method - Determination of changes in adhesion, cohesion and appearance after exposure to artificial weathering of elastic weatherproofing sealants for high movement façades', *Materials and Structures*, to be published.
2. Jones, T.G.N., Hutchinson, A.R. and Lacasse, M.A., 'Effect of movement waveforms on the experimental performance of newly sealed joints', in 'Durability of Building and Construction Sealants, RILEM Proceedings PRO 10' (RILEM Publications, Paris, France, 1999) 211-227.
3. Lee, T.C.P., Jones, T.G.B. and Hutchinson, A.R., 'Proposed test procedures incorporating dynamic cure', in 'Durability of Building and Construction Sealants, RILEM Proceedings PRO 10' (RILEM Publications, Paris, France, 1999) 297-313.
4. Anonymous, 'ISO 9047 – Building Construction – Jointing Products – Determination of adhesion/ cohesion properties at Variable Temperatures' (International Standardisation Organisation, Geneva, Switzerland, 1989).
5. Anonymous, 'ISO/DIS 11600 – Building Construction – Sealants – Classification and Requirements' (International Standardisation Organisation, Geneva, Switzerland, 2000).

THE DURABILITY OF SEALED INSULATING GLASS UNITS

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Presentation to the
Society for Adhesion and Adhesives
London, 7 December 2000

Introduction

Insulating glass units (IGUs), otherwise known as sealed double- or triple- glazing units, are now commonly installed in all types of windows, doors, curtain walling and structural glazing applications. They can be installed in appropriately designed timber, PVC-U, aluminium, steel and composite frames. They have better thermal insulation properties than single panes of glass and therefore enable reductions in energy use in buildings and allow designers to meet the requirements of building regulations.

Insulating Glass Units

An IGU is a composite of two panes of glass that are sealed together under factory conditions. The performance of an IGU is substantially different from a single pane of glass. The two panes of glass are held at a fixed distance apart by a spacer bar that is located around the perimeter of the glass. The glass pieces are sealed together with sealants that are applied to seal the perimeter of the IGU. The spacer bar contains a desiccant that absorbs moisture trapped within the unit during manufacture or which naturally permeates through the edge seal during service. There are other types of edge seals available, such as pre-extruded butyl-based tapes that account for some of the market.

The IGU can either be of the single seal or dual seal variety. A dual seal system contains a primary sealant that is compressed between the spacer bar and the glass. Dual seal IGUs have a lower moisture vapour transmission than comparable single seal IGUs. The primary sealant is normally polyisobutylene or other butyl compounds, and its primary function is to provide a moisture barrier. The secondary (outer) seal is usually polysulfide, polyurethane, silicone or hot melt butyl. These types of sealant have different moisture vapour permeation properties that should be taken into account when considering the specific installation.

Life Expectancy

The design of IGUs means that moisture will permeate as water vapour through the edge seal of the unit under normal conditions. Where the IGU has been properly designed, manufactured and glazed the lifetime should achieve 25 – 30 years. However, premature failure of IGUs has been found after short periods of time, ie five years or less. Failure is due to excessive moisture penetration through the edge seal that leads to condensation on the internal surfaces of the glass. This becomes progressively worse over time, but any appearance of misting or condensation under natural conditions indicates failure. A number of explanations for failure have been suggested, including the following:

- Poor quality and poorly manufactured IGUs.
- Poor glazing workmanship or the use of the wrong glazing system or window frame.
- Poor installation of the window into the building.
- Loss of adhesion of the sealant to the glass.
- Chemical degradation of the edge seal by aggressive agents (eg solvents released by inappropriate glazing materials).
- Ultra-violet degradation from sunlight if not properly glazed.

A number of environmental influences affect the performance and durability of the IGU and these include heat, sunlight, rain, atmospheric pressure, etc. Proper design and glazing is required to ensure that the IGU and the window frame can resist these influences over time. If glazed incorrectly then moisture, from rain and condensation, will lie in contact with the edge seal for long periods of time and result in failure. This 'ponded' water will increase the vapour pressure differential across the edge seal and so increases the moisture vapour transmission rate. The bond between the edge seal and the glass may also break or swelling and softening of the edge seal can occur.

Evidence from survey work carried out by BRE indicates that the failure rate of BSI Kitemarked IGUs was less than half that of non-kitemarked products. Specifiers are advised to give preference to dual seal systems that carry the BS5713 kitemark, and eventually its European successor. However, poor glazing of even the best IGU can lead to failure soon after installation.

BRE Research

BRE has been undertaking research on the lifetime of quality assured (kitemarked) IGUs glazed using currently recommended methods. This work is intended to assess the durability when glazed into various types of frames.

At present the measurement of durability is based on short term laboratory test procedures. These normally involve the exposure of a number of units to cycles of humidity and temperature. A dewpoint measurement is made before and after cycling. At present in the UK such a test requirement is made under BS 5713 and other countries have their own standards. CEN have been developing a future European Standard for moisture resistance of insulating glass units and also for gas retention where appropriate. This new standard will bring into line the different requirements for testing currently used in Europe.

There appear to have been few studies to determine the actual long term performance of insulating glass. Those that have been undertaken have consisted of surveys, either questionnaires or condition surveys. The earliest records refer to the Norwegian West Coast study undertaken in the 1960's, more recently in the USA and the UK.

The UK work has led to a long term exposure experiment being set up at BRE East Kilbride in Central Scotland. The climate at the site is severe with high annual rainfall, low temperatures during winter and warm wet summers. In the window frames, which include timber, PVC-U, aluminium and steel, continuous monitoring systems were developed for the measurement of the following:

- relative humidity in the ventilated glazing cavity of the frames;
- temperature in the ventilated glazing cavity of the frames;

- electrical conductance to detect liquid moisture (water) on the rebate platform, in contact with the edge seal and moisture bridging between the rebate platform and the edge seal.

The windows were exposed to the prevailing weather conditions, predominantly to the south and west. This meant that the windows were more subject to rain, winds and sunshine than would have occurred on other elevations.

Highest temperatures encountered at the edge seal during summer and the greater daily temperature range would have placed greater stress on the edge seal area and cause greater thermal movement of the glass and sealant. During winter the temperatures around the edge seal were generally lower indicating the effect of the external environment on the IGU. However, relative humidity was higher in the winter and indicated a presence of moisture in the rebate area. Higher humidities, indicate moisture or moist air was present and this could have an influence on the durability of the IGUs.

The electrical conductance results have shown that some window frames have remained dry for long periods of time, including during the summer and winter periods. However, other frames were more prone to leakage of rain into the glazing cavities that has given rise to higher electrical conductance and relative humidity.

All the windows had the same specification and type of IGUs installed. A representative sample were tested for moisture penetration to the method being adopted through CEN. In all cases the IGUs passed the test. Ultimately, it is intended that the results of the CEN test will be compared with the results of five years natural exposure at this site. The experiment has now been completed and a full report on the experiment will be available in early 2001.

Conclusions

The long term service life of insulating glass units in windows needs to be assured if the full environmental and economic benefits are to be realised. This paper has briefly described some research that is intended to address the issues of service life under natural exposure and provide a link to accelerated durability tests being developed by CEN.

Usefulness of Contact Angle Measurement for Understanding the Adhesion of Aerospace Sealants to Aircraft Substrates

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Polysulfide based sealants have been widely used in aircraft manufacturing for many years. The major areas of application are: sealing of fuel tanks, sealing of the fuselage, corrosion protection and aerodynamic smoothing.

As different purposes require different application properties we can distinguish between sealants of different viscosity and application times. **A** grade sealants are used for brush application to cover bolts, nuts and rivets, **B** grades are applied by means of an extrusion gun to form fillets for the protection of edges and **C** grades are distributed on a substrate with a roller to form an interfay between two sheets of material. The material could be an Aluminium alloy, an Aluminium alloy plated with pure Aluminium, Stainless Steel, Titanium, Carbon Fibre Composites or any of the above coated with Epoxy or Polyurethane based coatings. Regardless of the type of sealant or type of substrate there is one requirement that has to be fulfilled by all combinations: the sealant has to adhere to the substrate after curing.

One widely accepted adhesion test, which is called for by many specifications, is the 180 ° peel test. The main disadvantages of this test are that a lot of experience is required in order to obtain reliable and reproducible results and that a certain cure time, dependent on the application time, is necessary before the specimens can be tested. For a product with an application time of e. g. two hours a cure time of two weeks is often specified in the standards. C grade materials could easily have application times of 48 or more hours which then require very long cure times so that it is sometimes very difficult to collect data about the adhesion properties of a sealant in a reasonable time period. The cure is sometimes performed at elevated temperatures but it has been observed several times that heat could influence the adhesion in a positive way.

In the recent past especially the development of new coatings (solvent based - high-solids - water based) has required numerous adhesion tests in order to examine the compatibility with sealants. The desire to obtain a quicker assessment or prediction of the sealant adhesion to new substrates was the driving force for our investigations using the contact angle measurement.

The determination of the surface energy of substrates according to Young gives only a rough estimate of the possible adhesion of an adhesive to an adherend. A general rule of thumb says that reliable adhesion can be observed when the surface energy of the substrate is approximately 10 $^{mJ}/_{m^2}$ greater than the surface tension of the adhesive. But sometimes the peel test proves that adhesion can be achieved although the comparison of the surface energies of adherend and adhesive does not give evidence for this result, and of course vice versa. This gives us reason to assume that the total surface energy, the spreading coefficient or the work of adhesion is not sufficient enough when describing the interaction between the adhesive and the substrate.

Figure 1 shows a variety of substrates and their corresponding surface energies.

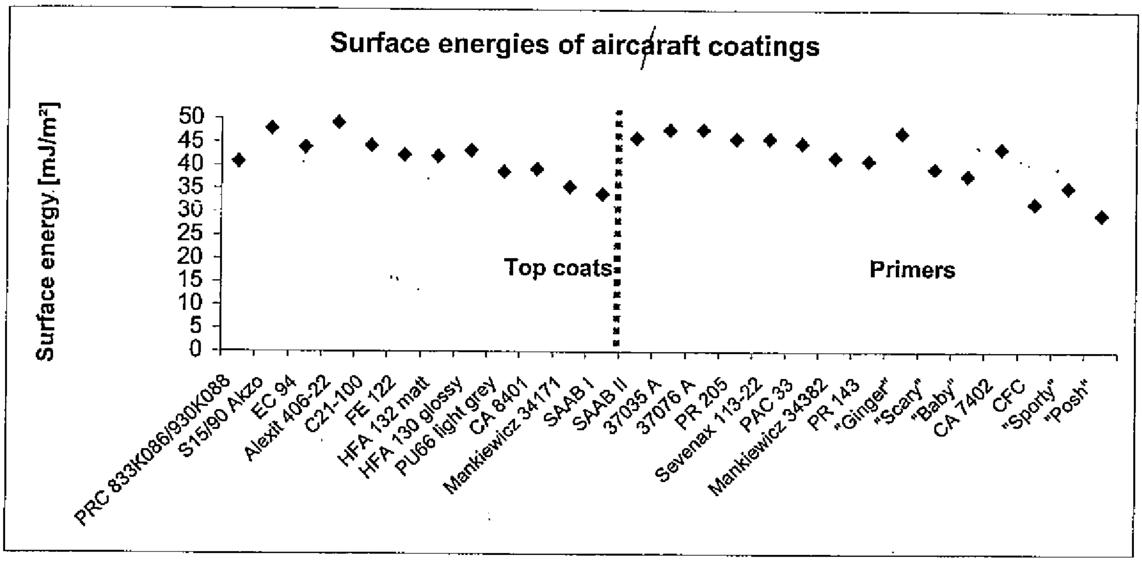

Figure 1: Surface Energies of some Aircraft Coatings

Surprisingly the surface energies of most substrates can be found in a region between 35-45 $^{mJ}/_{m^2}$ regardless of the paint's properties. The surface energies of Epoxy Primers and Polyurethane topcoats are obviously not as different as expected. Due to the facts mentioned above a correlation between the surface energies and the adhesion determined in a peel test can not be found.

The principle of Owens-Wendt, the differentiation of polar and dispersive contributions to the surface energy, allows us to distinguish between more polar or more dispersive surfaces. Substrates with the same total surface energy could differ significantly in their polar and dispersive parts.

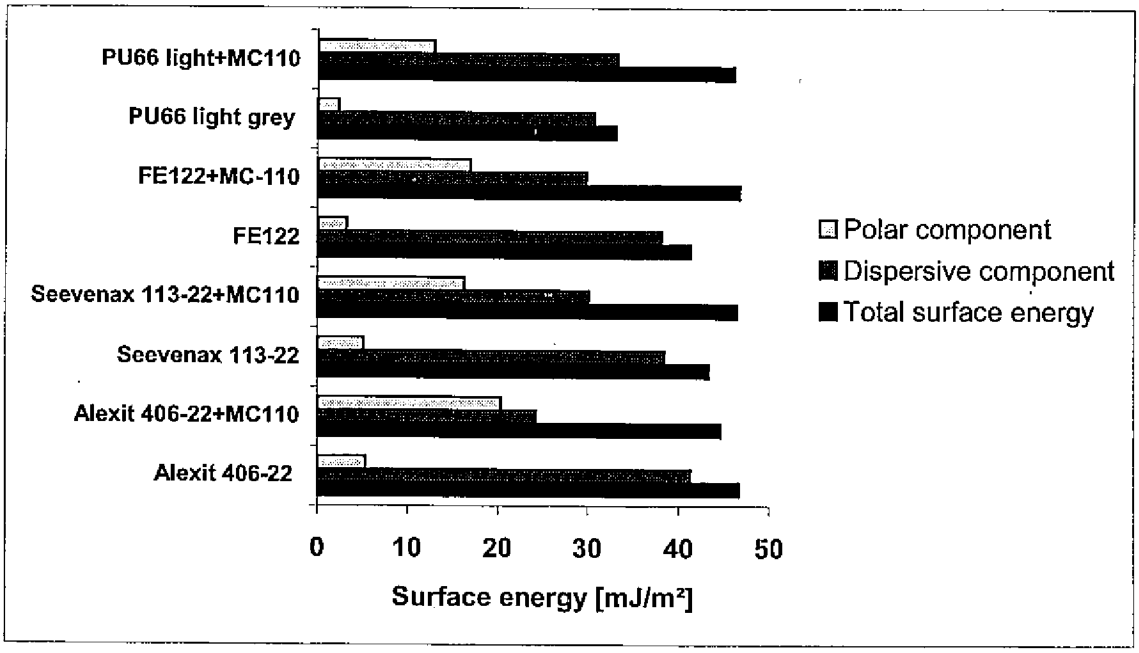

Figure 2: Components of the Total Surface Energy according to Owens-Wendt

Figure 2 shows the polar and dispersive components of different panels according to the Owens-Wendt principle. The surface energies were determined in one run after cleaning and in another after cleaning plus the application of the commercially available external adhesion promoter MC-110. After curing the peel strength of the sealant on both the primed and the cleaned surfaces were measured.

It is quite obvious that in comparison to the surfaces which were only cleaned, the polar component of the surface energy is increased in all cases by the additional application of the external adhesion promoter MC-110.

The approach of Good and van Oss describes the character of the polar components more in detail. According to them acid-base interactions are responsible for the polar part of the surface energy. Due to the fact that a hydrogen bond is only a special case of the interaction between a Lewis base and a Lewis acid, the corresponding terminology is used to describe these effects in general.

With the right choice of test liquids the total surface energy could be separated into the dispersive (Lifshitz-van der Waals) and (Levis)acid-(Levis)base component. Figure 3 gives the results determined with the "three liquid method" according to Good and van Oss. More or less the dispersive part nearly equals the value of the entire surface energy in all cases. The differences between the coatings are mainly caused by the acid-base component of the surface energy. In contrast to FE 122 and PU 66, which have hardly any acid-base interactions, the other systems show this component from the beginning. Treatment with the external adhesion promoter results in an increase of the acid-base component. This is in accordance with the results determined by the application of the Owens-Wendt Theory (see Fig. 3).

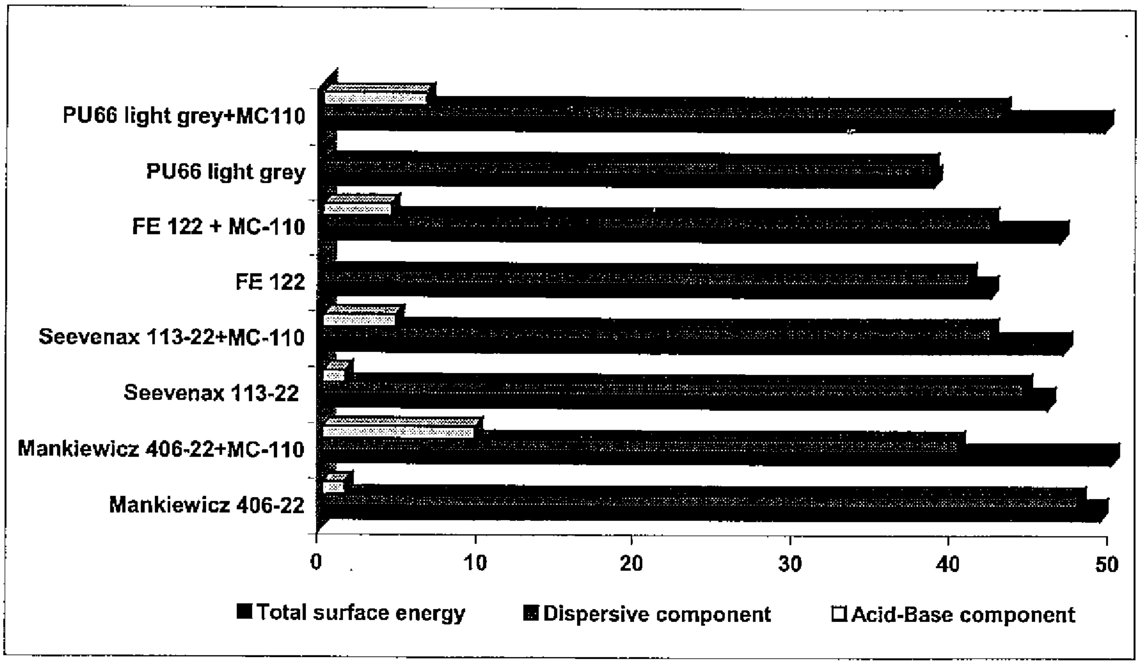

Figure 3: Components of the Surface Energy in $^{mJ}/_{m^2}$ according to Good and van Oss

Figure 4 shows the results of peel tests on the different substrates.

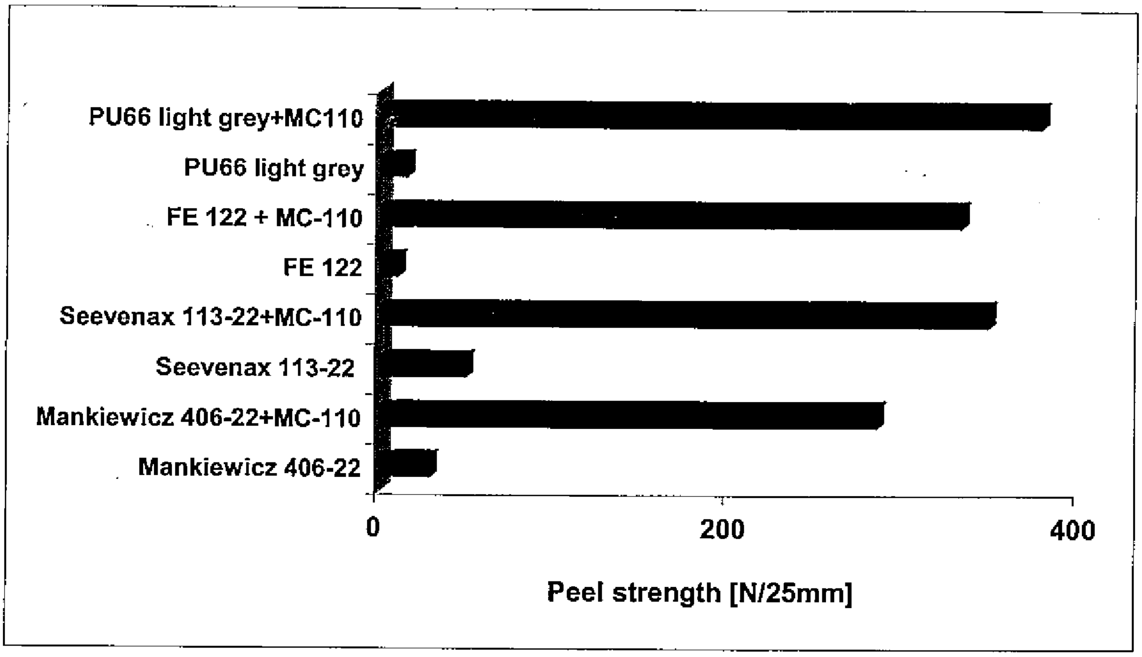

Figure 4: Peel strength with and without adhesion promoter

As a sealant without an internal adhesion promoter was used for these tests, a high peel strength can only be achieved in combination with a 100 % cohesive failure on primed substrates. Up till now it has been shown in quite a lot experiments that reliable adhesion can only be found in adhesion tests when a significant value of the acid-base component of the substrate is given.

We have also examined the impact of different methods of surface activation in relation to the surface properties and adhesion results. In practice cleaning, abrading and application of external adhesion promoters are used. The acid-base theory of Good and van Oss provides a basis for better understanding of peel test results which showed that some substrates gave inferior adhesion after cleaning-abrading-cleaning than only after cleaning with solvent. Again a higher value for the acid-base interaction component gives strong evidence that reliable adhesion can be detected in peel tests.

Sealants in the Automotive Industry
(Society for Adhesion and Adhesives)

Introduction
The automotive industry uses sealants in a large number of applications from windscreen bonding to underbody coating. However, within these broad parameters lies a range of rather interesting applications where materials are applied at the "Body-in-White" stage of the car building process. The phrase "Body-in-White" refers to the unpainted car body shell. Here the materials must behave as both sealants and adhesives. As sealants to prevent the ingress of water, salt and dirt etc and as adhesives to have excellent adhesion to a seemingly inexhaustible number of substrates, coated with a similar number of oils. The good adhesion performance must be maintained over a wide range of test temperatures, typically from –40°C to + 90°C and after several types of ageing test of varying severity. These points will now be discussed in more detail.

Typical Car Building Process Conditions
The steel (mild, electrogalvanised, hot dipped galvanised, pre-phosphated etc) is normally covered with an oil applied at the mill for protection during transportation. After delivery to the car plant the steel is cut to size and stamped to shape. Very high forces are required to produce these panels and therefore press oil is applied to lubricate the part in order to prevent stress cracks from being formed during the pressing process. For more complicated parts (deep drawn) a more efficient lubricant is applied to the steel as the chances of stress cracks formation is greater in this situation.

The next stage of the process is the application of the sealants and adhesives. This is achieved by using equipment of varying complexity, from hand operated pumps to sophisticated pumping systems linked to robots. Each robot can be programmed to perform several complicated tasks, for example applying a very accurate bead of sealant / adhesive as well as picking up and moving parts.

After the sealant / adhesive has been applied the panels are joined together to form the part. High-pressure water jets are used to remove oil from the panel. The uncured products must withstand these jets for two reasons: (1) If material falls into the paint bath it will contaminate the system (2) If the material is washed off it will not be able to fulfil its function. The panels are then dried before proceeding through the electrocoat paint system and into the oven. Temperatures here range from 180 – 200°C and the typical time in the oven is 30 minutes. This is the oven where the sealants and adhesives applied in the body shop cure. After the electrocoat oven successive coatings of paint are applied but these topcoats are cured at lower temperatures (130 - 150°C) for shorter times (10-15 minutes).

Typical Property Requirements and Typical Applications
The general requirements for a body shop sealant / adhesive are given below.

1) Suitable rheological properties – The material must be suitable for the application equipment.
2) Modulus requirements – The cured product should meet the modulus requirements after the appropriate stoving conditions. This is controlled by physical test requirements, lap shear & peel strength tests for example, set by the customer. These tests are carried out at –30°C, +20°C and +90°C.

3) Ageing test conditions – Approved materials must pass very stringent ageing tests. Salt spray tests for 500 or 1000 hours – lap shear bonds are prepared and cured according to the relevant specification. Bonds are then continuously exposed to the salt spray atmosphere (5% NaCl solution / 35°C). The bonds are tested after the required periods of exposure.

   Cycling tests – These vary but nearly always include a salt spray element to the cycle. A typical test is 1 day in salt spray, 4 days 98% rh / 40°C, 2 days at ambient conditions – this is one cycle. Bonds are tested after 6 cycles or 10 cycles depending on the application.

   Humid cataplasme – Lap shear bonds are stoved as specified by the customer. The bonds are wrapped in a fixed amount of cotton wool, placed in a polythene bag and a specific amount of de-ionised water added to the bag. The bag is then heat sealed and placed in a 70°C oven for a fixed number of days (7, 14 or 21). After the ageing period the test assemblies are placed in a freezer at –30°C for two hours. At the end of this time the assemblies are removed and allowed to warm up to laboratory ambient. The wet cotton wool is removed and the bonds tested within 4 hours of removing from the freezer.

4) The main applications for sealants / adhesives in the body shop are:

(a) **Anti-flutter**: low modulus materials that prevent vibration between inner and outer panels

(b) **Structural**: high modulus materials that contribute to the torsional stiffness and impact resistance of cars.

(c) **Spotweld / interweld / weld through** sealers: materials that are applied to panels prior to spot welding. These materials seal the joints between spotwelds.

Applications and test results will be illustrated by using examples from the Elastosol product range.

Adhesion to Imperfect Surfaces

The changes in environmental legislation in the early1990's following the Montreal Agreement has resulted in the need for changes in the surface treatments used in the aerospace industry[1,2]This involves the removal of the Cr^{IV} ion from all processes and the use of low VOC (volatile organic content) or water based coatings.

Considering a key part of the airframe, such as the aircraft's fuel tank gives an indication as to the nature of the problem. This is an integral part of the wing and, therefore, the coatings and sealants will be subjected to a wide range of temperatures, fuel and fuel/water mixtures and exposure to fungi that live at the fuel/water interface.

This paper will discuss one of the many problems associated with a system of this kind. This is to understand the chemistry of the interface between the paint and the sealant. Figure 1 is a schematic of a joint inside an aircraft wing showing all of the components present in this corrosion protection and sealant system.

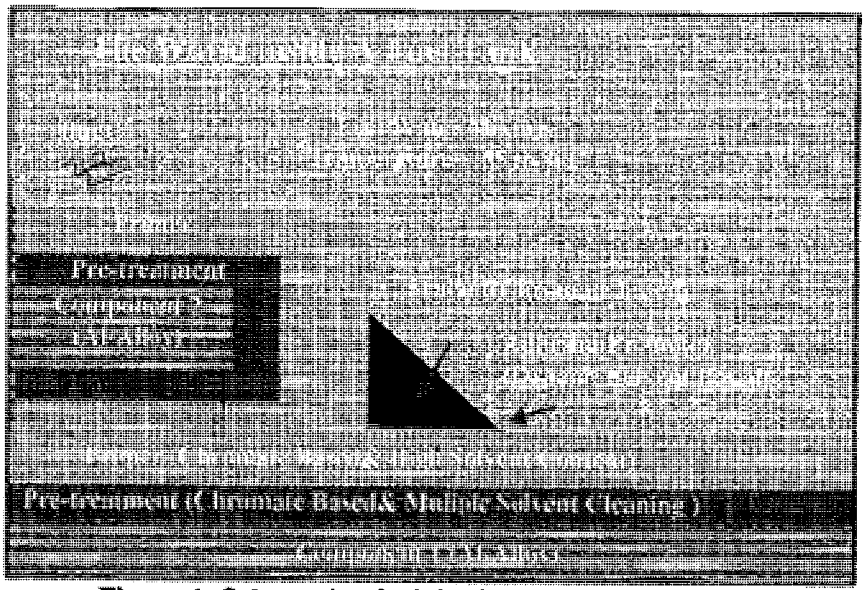

Figure 1: Schematic of a joint inside an aircraft wing

The challenge is to use surface analytical methods to explore the interfaces represented in this diagram, and understand their modes of failureThis report summarises the initial work on "Adhesive" or "Interfacial" failure in paint/sealant joints. The work will be described in a case by case basis rather than a discussion on individual surface analytical results.