Durability of Adhesive Joints

Thursday 7th December 1995

held at Society of Chemical Industry, 14/15 Belgrave Square, London

Programme

10.30	Coffee and Registration
11.00	THE INTERFACIAL MICROCHEMISTRY OF ADHESIVE BOND FAILURE IN HOSTILE ENVIRONMENTS S J Davis and J F Watts (University of Surrey)
11.30	DURABILITY OF ADHESIVELY BONDED METAL MATRIX COMPOSITES L M Gardham and M R Bowditch (DRA, Holton Heath)
12.00	PEELING OF PRESSURE-SENSITIVE ADHESIVE TAPES R A Chivers (Smith and Nephew Group Research)
12.30	ON THE DURABILITY OF RUBBER-TO-METAL BONDS M A Ansarifar and G J Lake (MRPRA)
13.00	Lunch
14.30	CYCLIC FATIGUE FAILURE OF STRUCTURAL ADHESIVE JOINTS J K K Jethwa, A J Kinloch (Imperial College) and J F Watts (University of Surrey)
15.00	CHARACTERISING FATIGUE CRACK GROWTH AT ALUMINIUM/EPOXY INTERFACES BY DIRECT MEASUREMENT OF MIXED-MODE STRESS INTENSITY FACTORS H Y Ahmed, M E Fitzpatrick and L Edwards (The Open University)
15.30	Tea
15.45	THE APPLICATION OF SILANE COUPLING AGENTS FROM AQUEOUS SOLUTION AND BY INCORPORATION IN THE ADHESIVE R P Digby and D E Packham (University of Bath)
16.15	TENSILE BUTT OR LAP SHEAR? - THE TESTING QUESTION A Beevers (Oxford Brookes University) and M R Bowditch (DRA)
16.45	Close

The Adhesives Section of the Institute of Materials holds regular symposia on aspects of Adhesives and Adhesion Science.

The Committee consists of the following members

Professor R D Adams	Chairman
M R Bowditch	Secretary
S G Abbott	
K W Allen	
J A Bishopp	
Dr J Comyn	
Dr G J Lake	
Professor A J Kinloch	
Dr D A Tod	

The next major event to be organised will be Adhesion 96 which is to be held at Cambridge University from 3rd-6th September 1996.

The next symposia will be on Curing of Adhesives and will be held at the SCI on Thursday 18th April 1996.

If you wish to receive details of the above please contact:

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The Interfacial Microchemistry of Adhesive Bond Failure in Hostile Environments

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ABSTRACT

The strength and mode of failure of adhesively bonded iron substrates has been investigated. Joints were assembled from polished iron and from iron substrates that had been pretreated in a novel manner by the cathodic deposition of yttrium. Joints were exposed to an aqueous environment and a reduction in joint strength with increasing exposure time was noted.

Failure surfaces of joints exposed to water for 1200 and 7500 hours were examined by small area XPS imaging XPS and ToF-SIMS. An interfacial failure was observed, and although all surfaces were rich in nitrogenous species from the curing agent, no evidence of epoxy residues on the interfacial metal failure surfaces was recorded by ToF-SIMS. The metal failure surface at extended exposure times shows that there are two delamination fronts advancing from the exposed edge of the adhesive joint: one is associated with bond cleavage and gives rise to a "zero-volume debond" and proceeds faster than the other which is associated with gross separation of the adhesive and substrate, oxide growth on the exposed metal adherend and mass transport within the disbondment crevice.

This investigation illustrates the need for surface analysis, at high spatial resolution, if a complete understanding of the failure mechanisms that occur when adhesive joints are exposed to a hostile environment is to be achieved. Although imaging XPS is not yet generally available it is clear that it has much to offer in future investigations.

KEYWORDS

Adhesive joint failure, cathodic disbondment, interfacial failure, small area XPS, imaging XPS, ToF-SIMS.

Durability of Adhesively Bonded Metal Matrix Composites

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Metal matrix composites are high stiffness, low density materials and as such have much potential in the construction of light weight components. Adhesive bonding processes are being investigated partly in order to exploit fully the properties of these materials but partly also because of the unsatisfactory nature of more conventional joining methods when used with these materials.

The work to be presented has involved the use of two types of particulate reinforced metal matrix composites (PMMC) using Al/Li and Al/Cu as matrix materials with silicon carbide being the reinforcement common to both types. In order to provide a benchmark against which success could be measured, a common aerospace alloy, NS4, was also used as a substrate material. Both epoxide and separate part application acrylic adhesives were used against surfaces prepared in a variety of ways.

With the exception of the reverse bend fatigue tests which were carried out in the atmosphere, all environmental exposure involved total immersion of single overlap shear joints in distilled water at either 40 or 50°C. Stress rupture tests were carried out in the wet environment and conventional debonding of joints using an Instron tensometer and impact testing using an instrumented Ceast apparatus were carried out on specimens cooled to room temperature after removal from the immersion tanks.

Peeling of pressure sensitive adhesive coated tapes

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Pressure sensitive adhesives are used in a very wide range of applications. The requirements for load-bearing and duration of bonding in these applications vary over a wide spectrum. Pressure sensitive adhesives for medical applications occupy a small part of this range, with very specific requirements. Loads sustained in use (during wear) are not usually large, and are frequently in the plane of the tape, while it is essential that these tapes be removable by peeling with minimal pain and trauma to the skin after no more than a few days.

Several models have been published which attempt to describe the peeling of joint.s These consider either the forces and stresses in the backing, substrate and adhesive, or the energy balance. The most significant of the former models is that due to Kaelble, which gives remarkable agreement with experimental data, despite various criticisms which have been levelled at his assumptions over the years since publication. The energy model states that the energy to debond should be independent of the debonding process, and this disagrees with the peel energy calculated from the Kaelble model. Recent analysis by Kinloch and Williams has shown that an intrinsic peel energy is indeed obtained from a rigorous analysis of experimental data of peeling systems.

The above models make simple assumptions, and, especially, tend to make little reference to the adhesive layer in the joint. For a pressure sensitive adhesive, this is of great importance. Equally, the models (and, indeed, test procedures) tend to treat the backing in fairly simple mechanical terms, and the substrate as rigid. For pressure sensitive adhesive coated tapes on skin, both of these are unrealistic. The paper will discuss the existing models of peel adhesion, and consider their relevance to practical applications of pressure sensitive adhesive coated tapes in a medical environment.

On the Durability of Rubber-to-Metal Bonds

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ABSTRACT

In may applications rubber is bonded to metal for fixing purposes or to alter the stiffness. Integrity of the bond is often vital to maintain the required stiffness and ensure adequate life. The use of fracture mechanics methods to assess failure is more difficult than for the cohesive failure case in that, for strongly bonded systems, the failure locus may vary with the test geometry and method of loading. In the present work, the mechanics of bond failure is being studied for various test geometries. It has been observed that when a constant force is applied to a bonded joint, failure can occur in a time-dependent manner with a locus that is close to the bond and similar in peeling (at different angles), pure shear and various combinations of simple shear and compression. If such a joint is pulled apart at a constant (high) rate, very different failure loci are observed for the different geometries and a simple energetics approach does not apply. Initial constant force results suggest that the rates of failure, for at least some of the geometries used, can be quantitatively interrelated by an energetics approach. It appears that the time-dependent failure, which is observed with elastomers that do not normally show time-dependent cohesive failure, may be associated with cavitation-like processes occurring in the failure zone.

Cyclic Fatigue Failure of Structural Adhesive Joints

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The cyclic fatigue failure of epoxy/aluminium alloy and epoxy/electrogalvanised steel joints has been studied using a fracture mechanics approach. Experiments have been undertaken in both a relatively dry environment and in water. The effect of water on the cyclic fatigue properties will firstly be discussed. Secondly, the mechanisms of attack will be considered. For these studies techniques such as X-ray photoelectron spectroscopy, atomic force microscopy and electron microscopy have been employed. Thirdly, the use of the fracture mechanics data to predict the lifetime of adhesively-bonded components will be discussed.

Characterising Fatigue Crack Growth at Aluminium/Epoxy Interfaces by Direct Measurement of Mixed-Mode Stress Intensity Factors

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Adhesive bonding of aluminium for structural applications has attracted increasing attention in recent years from both the automotive and aerospace sectors. The drive to improve fuel efficiency has led to a desire for lighter structures, though aluminium is disadvantageous in that it cannot be joined economically using existing techniques.

Adhesive joining of aluminium has therefore become a viable proposition for many applications, and prototype road vehicles have been constructed using aluminium chassis that feature boned joints.

Once of the problems with metal/adhesive bonds is that the mechanics of crack growth at the interface between the two materials is not well-understood. The problem is theoretically complex and difficult to model, and it is not trivial to relate the results of laboratory experiments to real applications where they can benefit the design process.

Work at the Open University has applied the optical method of caustics to the study of this problem: this is an optical interferometry technique that allows direct measurement to be made of the stress intensity at a crack tip. Results have been obtained for bimaterial samples of aluminium/epoxy, and the work is now being extended to model joints.

Results from this technique can be obtained from real joint geometries, and combined with experimentally-determined fatigue crack growth rates from model systems, in order to produce information on the in-service lifetime of a bonded joint.

The Application of Silane Coupling Agents from Aqueous Solution and by Incorporation into the Adhesive: Bond Strength, Durability and the Location of the Silane

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ABSTRACT

In recent years concerns have been expressed about the effects on the environment of traditional chemical etching and anodising pretreatments for adhesive bonding of aluminium. This has led to considerable interest in the use of silanes in structural bonding. Silane coupling agents have been used for many years in the manufacture of fibre-composite structures to increase bond strength and, more importantly, durability. Silanes have been shown to substantially increase the durability of adhesive bonds with a number of adhesive/adherend combinations. The potential of silanes to provide a relatively simple, less harmful pretreatment for adhesive bonding is undeniable; however, in order for this potential to be realised it is necessary to gain a more complete understanding of the role of silanes in the bonding process.

In this work, two methods of silane application to clad aluminium adherends have been compared: deposition from aqueous solution and incorporation into a supported-film epoxy resin adhesive. The efficacy of amino- and epoxy-functional silanes has been assessed using both application methods. Bond strength and durability have been determined by use of the boeing wedge test and the peninsular blister test. Scanning and transmission electron microscopy, in conjunction with ultramicrotome sectioning, have been used to examine the pretreated adherends, the bondline and the post-failure surfaces. Surface and interfacial chemistry, before and after failure, have been examined using x-ray photoelectron spectroscopy, energy dispersive x-ray spectroscopy and wavelength dispersive x-ray spectroscopy.

The data gathered have been used to produce graphs of the variation in crack length and strain energy release rate on exposure to a warm, wet environment. The chemical analysis data have given an insight into the location of the silane coupling agents and their role in the bonding process.

Tensile Butt or Lap Shear? - The Testing Question

A Beevers (Oxford Brookes University) and
M R Bowditch (DAR Holton Heath)

As part of a major national programme on *'Measurement Technology and Standards for Adhesive Bonding'* an extensive study has been carried out to compare different durability tests. Nine different test methods have been evaluated to determine their effectiveness in measurement of relative durability of adhesive joints. The assessment of suitability of the test methods was based on a number of different criteria including sensitivity, reproducibility, comparative accuracy, ease of application and interpretation. Some particularly interesting observations emerged from the comparison of the tensile butt and lap shear joints, and this presentation discusses the different mechanisms which may contribute to the degradation behaviour in the two systems.

For both these methods, bonded joints were immersed in water at 60°C for selected periods and then tested to measure residual strengths. The parametric test matrix included two substrate materials (mild steel and aluminium alloy), two surface treatments (grit blast, and grit blast and silane), and two adhesives (a two-part cold-cure epoxy and a one-part, hot-cure epoxy). These studies showed that the tensile butt test is generally more reproducible and exhibits a more rapid degradation during early stages of ageing. The tensile butt test is also more discriminating on high durability systems.

An anomalous characteristic with the one-part epoxy adhesive on grit blasted mild steel lap joints was an initial loss of strength during the first three weeks of ageing, followed by almost total recovery of original strength after a further three weeks of ageing. This behaviour was not observed in the tensile butt test.

A possible explanation of this characteristic has been developed from a consideration of the mechanisms associated with water absorption and the stress distributions within the different joints. Redistribution of stresses during the early ageing period are shown to be more damaging to the butt joint, thus causing a more rapid strength loss. The recovery of the strength in the lap shear test during the secondary period may be due to beneficial plasticisation and compressive swelling forces which develop later due to the flower diffusion rate in the central thickness of the glue line.

The effects of the silane coupling agents in the locus of failure and behaviour of the joints are also considered. The observations illustrate the complexity of the possible mechanisms involved during the ageing process and highlight the difficulties in interpretation and prediction of behaviour from durability tests.