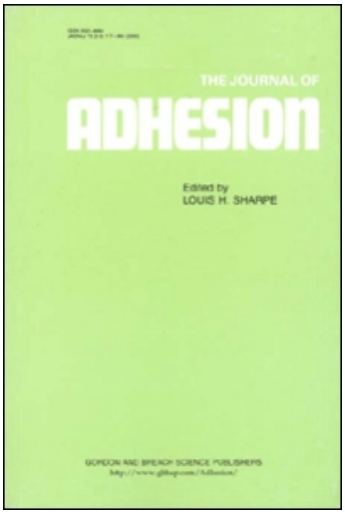

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The Fracture Mechanics of the Pin and Collar Test for High Temperature Anaerobic Adhesives

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A mechanical test method for the studies of high-temperature anaerobic adhesives has been established, based on fracture mechanics, by modifying the standard test method of collar and pin test. Linear Elastic Fracture Mechanics approach was applied to the establishment of the relationship between adhesive fracture surface energy "R", fracture load and crack length. Hence, from the joints containing a given artificial flaw the adhesive fracture surface energy can be determined; alternatively, from the strength of the joints without artificial flaws the inherent flaw size "a_i" can be calculated to account for the decrease of joint strength.

The experimental techniques were applied to examine the mechanical behaviour of the joint system based on high temperature anaerobic adhesives. It was found that the joints cured at room-temperature had higher adhesive fracture surface energy but lower joint strength than the joints postcured at high temperatures. The "a_i" data explained this interesting phenomenon. The joints cured at room-temperature had extraordinarily large "a_i", which was found to be formed by the uncured adhesive near the edges of the joints and the adhesive further cured in the postcure processes to reduce the "a_i". Also, the growth of intrinsic flaw was found to be responsible for the deterioration of the joints in a short-term, high-temperature ageing process.

INTRODUCTION

"The acrylic acid diesters (anaerobic curing materials) remain in liquid form so long as they are exposed to oxygen. If oxygen is removed as, for example, by confining the material in a thin film between the threads of a metal nut and bolt, the material hardens. A new series of one part anaerobic adhesives has recently become available which has strength characteristics of the structural adhesives. They cure at room temperature in several hours, but their cure can be accelerated to a matter of minutes at slightly elevated temperatures."[1] Although this was written by Lou Sharpe in 1966, there is still a considerable lack of understanding of the mechanisms which control the mechanical properties of these anaerobic adhesives.

Since anaerobic adhesive technology is newly developed, mechanical properties of the adhesives (as opposed to the mechanical properties of the joints formed using the adhesives) are rarely reported and the appropriateness of some test methods to specific applications may be questioned. Further development of these adhesives

will require test methods for the determination of the behaviour of the adhesives in the bonds.

Anaerobic adhesives are one-part, room-temperature quick-curing adhesives which are generally catalogued into four groups, sealing, threadlocking, retaining and structural bonding. Recent developments have enabled them to be used in applications in which they are exposed to high temperatures, and commercial products, (e.g. Loctite 620), have become available for retaining applications up to 230°C either continuously or intermittently. Although the chemistry of anaerobic adhesives has been well reported, their mechanical properties and behaviour appear less frequently in the literature. Mechanical test methods for assessing the performance of the adhesives have commonly employed standard test methods for which an average failure stress is reported. The American Military Specifications (AMS) for Testing Anaerobic Adhesives is an exemplar and the test methods are followed by many of the manufacturers of anaerobic adhesives.

Applications of retaining anaerobics frequently involve in the bonding of cylindrical parts.[2,3] Consequently the standard test method described in the AMS for retaining anaerobics has adopted the pin and collar test under shear mode loading. The strength of the adhesives, is expressed as the maximum failure force per unit area, and is evaluated by testing the joints under a set of standard conditions. The performance of the adhesives at high temperatures is evaluated by the residual strength of the joints when tested at room temperature after ageing. The average stress criterion which is often quoted may result in a large error when the conditions in practical situations are not identical to those in laboratory tests.[4] Also, although the deterioration of joint strength is associated with the chemical degradation of the adhesive polymers, the pin and collar test method does not easily yield details of any changes in the mechanical properties of the adhesive associated with the ageing processes. Moreover joint strength is not a single function of cohesive strength of the adhesive and may be much less relevant to the joint strength when failure occurs adhesively. Flaws in the bonds, which may nucleate cracking may be a factor reducing strength of adhesive joints and the growth of flaws in the ageing processes may be cause for the decrease of joint strength.

In many applications, failure of brittle adhesive joints is by fracture and this mode of failure is controlled by the stress irregularities which are introduced by flaws and which are ignored in those test methods based on average stress criteria. For such systems fracture mechanics provides a feasible approach to the characterisation of adhesive joints. The deterioration of adhesive joints may be studied by an examination of the quantitative relationship between the fracture toughness (or adhesive fracture surface energy for adhesive joint systems), crack length and the critical fracture load (which can be interpreted as joint strength for adhesive joint systems). The decrease of joint strength can then be directly related to the decrease of fracture toughness (which would indicate a change of the properties of the adhesive materials) and the growth of crack in the ageing processes.

The objective of this work is therefore to develop an appropriate test method based on fracture mechanics to approach the understanding of the mechanical behaviour of high temperature anaerobic adhesives.

THE FRACTURE MECHANICS OF THE COLLAR AND PIN TEST

The application of fracture mechanics to structural adhesive joints has in the past been largely restricted to joint configurations in which mode I crack propagation was examined.[5,6,7] However, in many practical applications structural adhesive joints are designed to take advantage of high mode II toughness of adhesives, e.g. the applications of retaining anaerobic adhesives. Therefore, in order to examine joint properties of retaining anaerobic adhesives under conditions which approach practical situations, the 0° cone (collar and pin) specimen geometry from the standard test method is utilised and modified for the fracture mechanics test.

For adhesive joints, an energy criterion approach may be preferred to the stress intensity factor approach because of the complicated nature of the stress analysis at the crack tip for heterogeneous systems. Thus the critical strain energy release rate may be calculated by the Linear Elastic Fracture Mechanics[8] expression,

$$G_c = (1/2b)\, F_c^2 (dC/da)$$

where "G_c" is the critical strain energy release rate (which is equal to the adhesive fracture surface energy for the quasi-static crack growth), "F_c" is the critical fracture force, "b" is the thickness of the bondline, "C" is compliance and "a" is the crack length. The relationship between adhesive fracture surface energy, critical fracture force and crack length may be derived assuming that the adhesive is a Hookean solid; the adherend is much more rigid than the adhesive so that the energy dissipated in the deformation of the adherend can be ignored and that the stress distribution is homogeneous along the bondline.

Considering the 0° cone specimen containing a flaw in the middle of the bond under mode II (forward shear) loading as shown in FIGURE 1, the compliance can be expressed as a function of crack length,

$$C = \mu/F = w/E_s \pi d(h-a) \tag{1}$$

hence,

$$(dC/da) = w/E_s \pi d(h-a)^2 \tag{2}$$

and

$$R = G_c = (1/2b)\, F_c^2 (dC/da)$$
$$= w\, F_c^2/2E_s[\pi d(h-a)]^2 \tag{3}$$

The symbols are shown in the diagram. "E_s" is the shear modulus of the adhesive and "d" is the average diameter of the collar and the pin.

The conditions (linear behaviour of the adhesive, a fracture process zone which is small compared with the joint dimensions etc.) for the theoretical relationship (equation 3) might not be fully satisfied in practice. However the relationship between compliance and crack length may be expressed as a power relation, $C = K(h-a)^n$, and the plot of lnC versus $\ln(h-a)$ should be a straight line if the relationship is as expected. An experimental compliance calibration method may then be used to enable the fracture toughness of the joints to be calculated by equation 3.

To determine the adhesive fracture surface energy, specimens containing artificial flaws of known size may be tested. Then, from the strength of the joints without

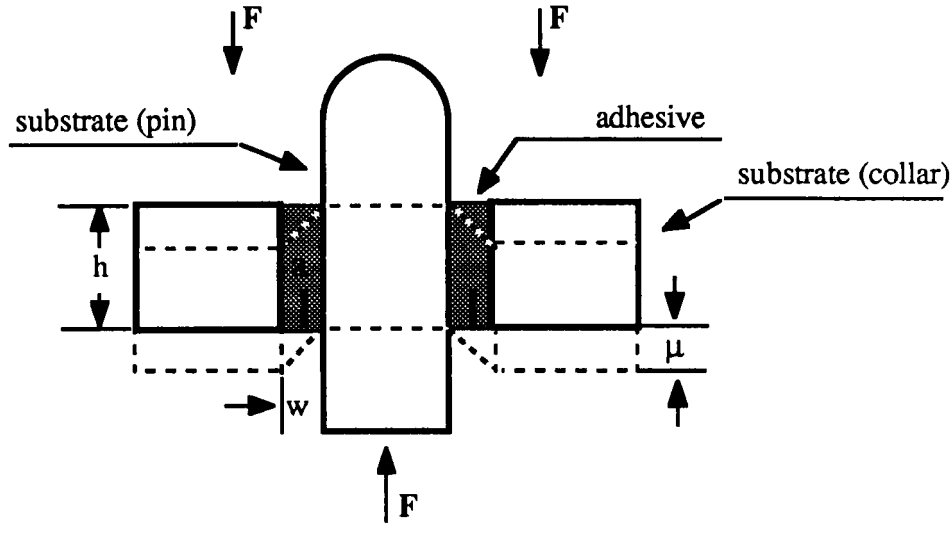

FIGURE 1 Diagram of the 0° cone speciment containing a flaw (a) in the middle of the bond, and the deformation of the joint under mode II loading.

artificial flaws, it is possible to calculate the flaw size (a_i) (termed the *inherent flaw size* by Anderson,[9] which will account for the strength of the adhesive joint. This concept of *inherent flaw size* relates the effects of the flaws in the bonds to an equivalent size of flaw located at the point of crack initiation. Although the size of this inherent flaw might not be identical with the initiating flaw in the bonds it may be used as a parameter to monitor whether the change of joint strength is caused by the growth of intrinsic flaws or a change in the Fracture Surface Energy.

EXPERIMENTAL

The objective of this experimental work is to establish the relationship between the adhesive fracture surface energy, fracture load and crack length for the collar and pin joint system by the compliance calibration method.[7] A commercial high temperature anaerobic adhesive was used for the tests as being typical of this kind of adhesive.

1 Materials and Specimen

The adhesive used is Loctite 620. It is recommended for retaining applications at temperatures ranging from 150°C to 230°C.

Specimens are made of mild steel CS-1020 (Australian specification). Each specimen comprises a pin 12.00 ± 0.02 mm in diameter and a slip collar 12.10 ± 0.02 mm inside diameter. The height of the collar is 12.00 ± 0.05 mm. The specimen gap

between the collar and pin is controlled at 0.05 ± 0.01 mm. All the specimens were vapour degreased using acetone before bonding.

2 Experimental Techniques

The compliance calibration involves determining the compliance of the joints with a range of artificial flaws. A computerized testing system has been developed for recording the load-displacement curves so that the compliance can be determined from the curves. The testing system is shown in FIGURE 2. The relative displacement between the collar and the pin is detected by a very sensitive LVDT (Linear Variable Differential Transformer). A computer program has also been developed for simultaneously acquiring the signals from the load cell and the LVDT to determine the elastic compliance from the acquired data. Other modifications to the standard test include a change to one end of the pin so that it is half-balled head, and minimises the effects of misalignment of the specimens in the test by the use of a loading plate with a same radial half ball in the material. The artificial flaws were made by coating a thin layer of wax of measured length on the pins. The specimens were tested at a loading range of 2 mm/min on an Instron tester at room temperature.

3 Results and Discussion

The failure mechanism of the joints has been examined. The characteristics of the load-displacement curves indicate that failure, under the experimental conditions, of joints cured at room-temperature for two weeks is by fracture after a limited yielding. Joints which had been post-cured at high temperatures failed by a more brittle fracture mechanism.

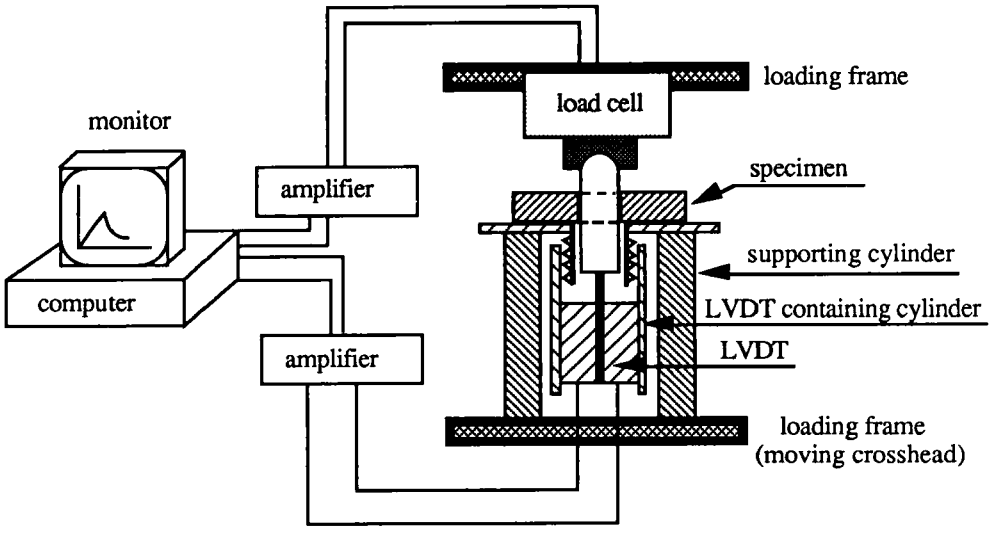

FIGURE 2 Diagram of the testing system.

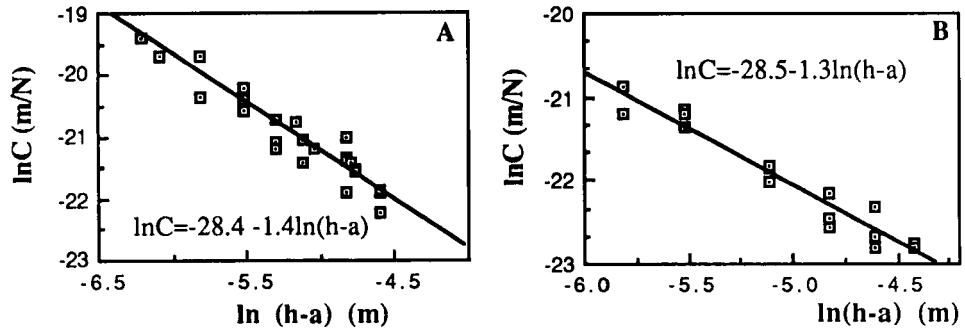

FIGURE 3 Plots of lnC versus ln(h − a) for the joints cured under different conditions. A. room-temperature for 2 weeks. B. postcured at 180°C for 2 hrs.

FIGURE 3 shows two sets of data from the compliance calibration tests. As expected, the plots in lnC versus ln(h − a) clearly demonstrate the linear relationship expected from equation (3). The equations for the two straight lines in the diagrams are obtained from a least square fit. "n," the exponent in the equation is equal to 1.45 for the joints cured at room temperature and 1.3 for the joints postcured at 180°C. This indicates that the high-temperature postcuring has little effect on "n". Furthermore, the small variation of "n" does not much affect the value of the adhesive fracture surface energy evaluated by this method especially in small crack length range.

One the relationship for the compliance and crack length is found, the relationship between adhesive fracture surface energy, crack length and fracture load can be established.

$$R = (1/2b)\, F_c^2(dC/da) = nwF_c^2/2E_s(\pi d)^2(h-a)^{n+1}$$

FIGURE 4 shows the plots of adhesive fracture surface energy versus crack length. The results show that "R" is a property of the joint system but not a function of crack length. This affords further evidence for the relationship established for this joint system.

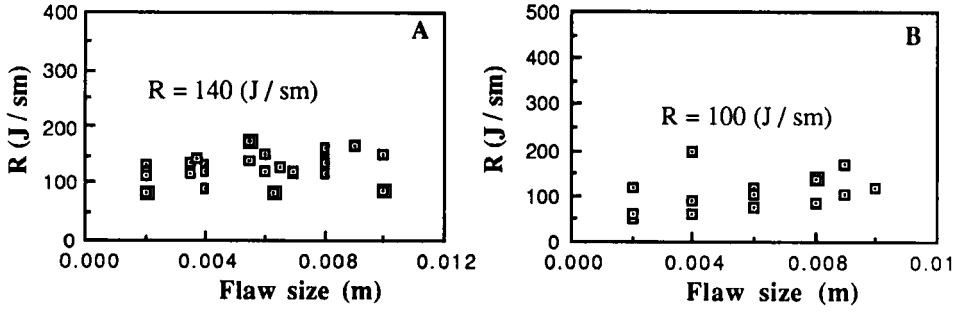

FIGURE 4 Plots of adhesive fracture surface energy versus flaw size. A. room-temperature for 2 weeks. B. post-heat-treated at 180°C for 2 hrs.

The value of "R" is relatively low indicating the joints are very brittle. This may also be proven by the characteristics of the load-displacement curves from the tests and the shear modulus evaluated for this joint system (please see the data in TABLE 1 in next section).

The artificial flaws were made at the interface instead of in the middle of the adhesive as in the theoretical derivation. This change does not affect the experimental expression because the bondline is very thin. Cracks may propagate at the interface or in the bulk depending on the properties of the joints.

MECHANICAL BEHAVIOUR OF HIGH TEMPERATURE ANAEROBIC ADHESIVES

1 Effects of Curing Conditions

The mechanical behaviour and properties of joints made using Loctite 620 and the way in which these are affected by room-temperature ageing and by heat-treatment have been examined. The results are shown in TABLE 1. The joint strength is the maximum force required to bring about failure and so is the fracture load or yield load, dependant upon whether failure is by yield or fracture. The shear modulus is calculated from the elastic compliance data, $E_s = w/C\pi dh$, where "E_s" is shear modulus, "w" is thickness of the bond, "d" is the diameter of the pin and "h" is the height of the collar.

Like most anaerobics, Loctite 620 is reported to develop "finger tight" strength after curing for 30 minutes at room-temperature and to have full strength after 24 hours at room-temperature [Loctite technical data]. However, our results show that curing continues after 24 hours at room-temperature and develops slowly. The load-displacement curves for each cure condition demonstrated that the failure mechanism of the joints cured at room-temperature changed from ductile fracture to brittle fracture after curing for more than 2 weeks. Although the adhesive in the bond becomes very brittle after becoming fully cured, the joint is quite strong because the bond is designed to be subjected to shear load and the bond line is very thin.

Joint strength is also a function of the curing time and reaches a maximum of 16KN after curing for more than 2 weeks at room-temperature. Moreover, high

TABLE 1
Effect of cure conditions on the mechanical properties of Loctite 620

Cure condition	RT-24H	RT-1W	RT-2W	RT-1M	PT180-24H
C (m/N) $*10^{-10}$	6.00	4.37	3.83	2.33	1.30
E_s (N/sm) $*10^8$	3.69	5.06	5.77	9.49	17.0
F_c (KN)	12.1	14.1	15.7	16.3	19.3
R (J/sm)			140		100
a_i (mm)			2.0		0.9

Note: RT-24H is room-temperature 24 hrs., RT-1W is room-temperature 1 week and so on. PT180-24H indicates joints post-cured at 180°C after curing at room-temperature for 24 hours.

temperature post-curing significantly improves the joint strength. A major point of interest is that despite the fact that joints become more brittle after high temperature post-cure, the strength of the joints is much higher than that of the joints without high temperature post-cure. The reason for this particular behaviour is found from an examination of the inherent flaw size (a_i) for the joints. This is calculated by substituting the fracture load from the joints without artificial flaws into the relationship between the fracture load, flaw size and adhesive fracture surface energy established for this geometry of joint system. It is found to be the major factor controlling the strength of the joints. The joints cured at room-temperature are found to have a very large intrinsic flaw size. Examination of the fracture surface shows that this large flaw is not produced from conventional causes such as air bubbles, foreign materials, weak-bonding area, etc. but is formed by the uncured adhesive near the edges of the bond line because the penetration of oxygen inhibits curing. The uncured adhesive further cured in the high temperature post-curing process and this resulted in a large increase of joint strength.

2 Effects of Thermal Ageing

In order to understand the degradation behaviour of anaerobic adhesive joints in the thermal ageing processes, experiments have been carried out to examine the effects of thermal ageing on the fracture mechanics parameters of the joints. The joints were aged at 280°C, which is 50°C higher than the maximum temperature recommended for this adhesive. The experimental data of joint strength (F_c), adhesive fracture surface energy (R) and the inherent flaw size (a_i) are shown in TABLE 2. It can be seen that the inherent flaw size increases after thermal ageing while the adhesive fracture surface energy remains almost the same. The growth of the intrinsic flaw is apparently responsible for the decrease of joint strength in this ageing process. The mechanism of the growth of the intrinsic flaw remains to be investigated in our future work.

TABLE II
Effects of thermal ageing on R, F_c and a_i for the joints aged at 280°C

Ageing time (hr)	F_c (KN)	R (J/sm)	a_i (mm)
2	21.2	129	0.6
12	14.7	175	5.0
24	14.4	159	5.4

CONCLUSIONS

This work demonstrates that the Linear Elastic Fracture Mechanics approach is feasible to the study of mode II crack propagation for rigid joint systems. It provides an experimental method by compliance calibration for the establishment of the relationship between fracture load, flaw size and adhesive fracture surface energy.

This relationship makes it possible to characterize adhesive joints quantitatively in terms of fracture mechanics parameters. It has demonstrated that the fracture mechanics test method is very useful to the understanding of the mechanical behaviour of a joint system based on high-temperature anaerobic adhesives.

References

1. Sharpe, L. H., "Assembling with Adhesives," Machine Design, Vol. 38, No. 19, (1966), p. 177–200.
2. Pearce, M. B., How to Use Anaerobics Successfully, App. Polym. Symposia **19** (1972), p. 207.
3. Murray, B. D., Anaerobic Adhesive Technology, App. Polym. Symposia: Durability of Adhesive Bonded Structure, Vol. 32 (1977), p. 411.
4. Anderson, G. P. et al., Evaluation of Adhesive Test Methods, in Adhesive Joints, Mittal, K. L., 1984 Plenum Press, New York (1984), pp. 269–287.
5. Kinloch, A. J. & Shaw, S. J., A Fracture Mechanics Approach to the Failure of Structural Joints, in DEVELOPMENTS IN ADHESIVES-2, ed. Kinloch, A. J., Applied Sci. Pub., London, 1981, pp. 83–122.
6. Ripling, E. J., Mostovoy, S. and Patrick, R. L., Application of Fracture Mechanics to Adhesive Joints, ASTM STP No. 360, p. 5, Symposium on Recent Developments in Adhesion Science, (1963).
7. Trantina, G. G., Fracture Mechanics Approach to Adhesive Joints, J. COMPOSITE MATERIALS, Vol. 6 (April 1972), p. 192.
8. Knott, J. F., Fundamentals of Fracture Mechanics, Butterworth & co Ltd, London, 1973, pp. 100–106.
9. Anderson, G. P. & DeVries, K. L., Predicting Bond Strength, Inter. J. Adhesion, 1987, Vol. 23, pp. 289–302.