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EVALUATION OF THE WEDGE CLEAVAGE TEST FOR ASSESSMENT OF DURABIL--ETC(U)
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SUMMARY 18 DRIC

The Boeing wedge test was applied to joints of aluminium alloy made with three adhesives differing widely in toughness. Bonding pretreatments chromic-sulphuric acid pickle, chromic acid anodise and wet alumina grit blast were compared for two of the adhesives, and clear differences in crack length appeared after exposure to 50°C and 96% r.h. Rates of crack growth were determined in both ambient and warm humid environments, and suitable times are suggested for measurement of crack length. Wedge driving speed can affect results and should be standardised.

Results are more informative if shown as fracture energies rather than crack lengths, and the approximations involved are discussed. Practical aspects of the test are described and possible errors quantified. Fracture energies typically showed coefficients of variation of 5-20%.

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* Sandwich student.

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1 INTRODUCTION

Adhesive bonded joints weaken during long exposure in warm humid conditions, and strength loss varies markedly with the bonding pretreatment applied to the adherends. Exposures of hundreds or thousands of hours are necessary to discriminate between pretreatments if conventional lap shear or peel joints are used, and this led the Boeing Commercial Airplane Company to develop a simple constant-displacement cleavage fracture test that reveals durability differences in only a few hours[1,2] (Fig 1).

A thin wedge is driven between two bonded adherends to form a crack which grows further during warm humid exposure: high crack growth is correlated with poor surface treatment and low durability as shown by other tests. This wedge test was at first used on samples from components showing disbonds in service and as a process control for bonding pretreatment, but has also found an R and D role[3,4]. The present work is concerned with the validity and limitations of the wedge test for research studies, and also with its practical aspects on which little has been published.

Aluminium alloy adherends were used, with three pretreatments to give a range of resistance to warm humid conditions, and with three contrasting adhesives differing widely in toughness. The effect of wedge driving speed was examined, and crack growth rate in ambient conditions monitored to determine suitable times for initial crack length measurement. Crack growth rate at 50°C and 96% r.h. was followed for up to 200 hours in most cases, and for a few specimens up to 3000 hours, to determine suitable times for final crack length measurement. Practical aspects are discussed and related to the expected accuracy and precision, and typical variability observed over a large number of tests is presented.

Discrimination between pretreatments was satisfactory, although the toughest adhesive gave severe permanent bending of the adherends so that fracture energies could not be calculated. Some bending is likely with most modern structural adhesives but fracture energies can be measured approximately. Variability is higher than usually found with lap joints, although it is acceptable for detecting substantial differences between pretreatments.

2 EXPERIMENTAL METHODS

2.1 Adherends

Clad aluminium alloy of 10 SWG (3.25 mm) was cut into strips 25 mm by 150 mm. Alloy BS 3L73 was used for initial trials, but non-availability forced a change to 3L72 for most of the work. This difference is unlikely to affect the bonding surface, but the latter alloy has a lower yield point which affects the accuracy of fracture energy values (section 4.2).

2.2 Adhesives

Adhesives of contrasting properties were chosen to represent a range of toughness and durability. Primers were not used.

(a) Redux 312/5, a modified epoxy, 120^{o}-curing film, supported on a knitted nylon cloth, and was selected as a typical, moderately tough structural adhesive.

(b) AF130 is a modified epoxy supported film adhesive capable of service up to 180^{o}C, and as such has relatively low fracture energy.

(c) FM1000 is an epoxy-nylon unsupported film noted for its toughness.

2.3 Adherend pretreatments

Three conventional processes were chosen for their known differences in durability[5]. All adherends were first swabbed in white spirit to remove protective grease then in propan-2-ol to dissolve identification paint, and finally vapour degreased in 1,1,1-trichloroethane.

(a) Alumina grit blast

A wet process was used with 240 grade grit. Adherends were rinsed in cold tap water after blasting, partially dried with tissues and then in a cold air blast. Traces of grit were removed by brushing in white spirit and adherends were then vapour degreased.

(b) Chromic-sulphuric acid pickle

This was preceded by additional degreasing in a conventional non-caustic proprietary alkaline cleaning solution. Pickling was for 30 minutes at 62-65^{o}C in an industrial bath maintained to the requirements of DEF-STAN 03-2/1, Method O.

(c) Chromic acid anodise

Chromic acid concentration was 40 g/l and Cr^{III} content would have been low. Anodising was carried out at 40^{o}C to the requirements of DEF 151.

(d) Washing and drying after pickle and anodise

All washing was in cold tap water, with a first wash for about 2 minutes started within 0.5 minute of finishing pretreatment. A 20 minute soak then followed in a separate tank of clean water. Adherends were dried in fan circulated ovens at about 60°C, 10 minutes for the pickle treatment and 20 minutes for anodise.

2.4 Preparation of joints

Joints were laid up within 5 hours of completing pretreatment and cured either the same day or within 24 hours. Adhesive 312/5 was cured for 1 hour at 120°C in a press at 25-60 kPa. Adhesives AF130 and FM1000 were given autoclave cures of 1 hour at 170°C and 200 kPa.

Edges of joints were filed smooth and then semi-polished on silicon carbide paper (down to 600 grade) with white spirit as lubricant.

2.5 Measurement of crack length

A side view of the joint with wedge inserted is shown in Fig 1. During initial work with adhesive 312/5 wedges were pressed in at about 100 mm/min using a vice, but for AF130 and FM1000 controlled rates of 20 mm/min and 200 mm/min were compared.

Crack length (a) was measured with a travelling microscope. Joints were then exposed over saturated K_2SO_4 solution at a relative humidity of 96% at 50 ± 2°C and removed at intervals to re-measure crack length. Fracture energy (G) was calculated from crack length and specimen dimensions using the equation

$$G = \frac{Ed^2h^3}{16} \cdot \frac{[3(a + 0.6h)^2 + h^2]}{[(a + 0.6h)^3 + ah^2]^2} . \qquad \text{(Ref 2)}$$

see list of symbols

In most cases five replicates were used.

3 RESULTS

3.1 Crack growth in ambient conditions

Initial fracture energy serves as a datum confirming that pretreatment and cure have been satisfactory. Since some crack growth is to be expected even in ambient conditions the time between driving in the wedges and crack measurement should be standardised.

Fig 2 shows the decrease in fracture energy with time of exposure for three replicates, zero time being that of the first measurement made within 5 minutes of wedge insertion. Two replicates showed crack growth of 0.1 and 0.04 mm between 0 and 4 hours while the third gave 3.74 mm in 20 hours. Results for five replicates in Table 1 show that between 0.5 hour and 24 hours the average decrease in fracture energy was 0.15 kJ/m^2, corresponding to an average crack growth of 0.44 mm. A further indication that the initial crack soon slows down occurred with AF130 in conjunction with alumina blast, for which audible cracking ceased about 10 minutes after wedge insertion.

The effect of ambient exposure on subsequent crack growth at 50°C and 96% r.h. is also compared in Table 1 and Fig 3. The set exposed for 24 hours at ambient appears to show subsequently a higher energy, but this was due solely to one specimen giving consistently high values for no apparent reason.

It was concluded that initial measurements could be made between 0.5 and 1 hour after wedge insertion and this has proved satisfactory. No cracks have ever been observed under the microscope to be extending during measurement.

3.2 Crack growth and decrease in fracture energy during exposure at 50°C and 96% r.h.

(a) Adhesive 312/5 with pickle pretreatment

The results in Table 1 and Fig 3 show little further crack growth after about 30 hours exposure, and the initial difference between sets had disappeared. Initial cracks were cohesive whereas crack growth was almost entirely in adhesion.

(b) Adhesive AF130

Fracture energies are presented in Table 2 and Fig 4 for the three pretreatments and two wedge speeds, and show that after about 50 hours crack growth was very slow. Two each of the pickled and anodised joints were re-measured at intervals up to 3000 hours exposure and showed continued very slow and erratic crack growth.

Pickle pretreatment gave almost entirely cohesive failure for both the initial crack and crack growth, whereas anodise gave cohesive initial cracks but growth was mainly in adhesion. With alumina blast there was up to 30% adhesion failure even for the initial cracks, higher at the 20 mm/min wedge speed, and crack growth was entirely in adhesion. In all cases the support cloth debonded from the adhesive.

Wedge speed had no effect on fracture energies with pickle or anodise treatments, nor on alumina blast during exposure. However, the faster speed gave a significantly higher initial value with alumina blast, consistent with a higher proportion of cohesive failure.

Differences in durability between pretreatments are as expected[5], and correlate with the proportions of adhesion failure in the crack growth region. However, it is not clear why anodise gave a lower initial fracture energy than pickle when both showed cohesive initial cracks. Possibly crack growth at ambient in the short time between wedge insertion and measurement underwent a transition from cohesive to interfacial. This would be consistent with the lower peel strength given by anodised surfaces[5].

(c) Adhesive FM1000

Crack lengths are shown in Table 3 and Fig 5. Fracture energies were not calculated because considerable inelastic deformation of the adherends with this tough adhesive renders the equation invalid (see section 4.2). With pickle and anodise treatments crack growth almost stopped after about 30 hours, whereas alumina blast gave slow continued growth.

Modes of failure varied widely between specimens for all pretreatments. Pickle gave up to 20% adhesion failure in the initial crack, while crack growth ranged from completely cohesive to completely interfacial. Ahead of the crack the adhesive had elongated greatly with the formation of craze voids. In some cases this had so relieved the stress that the wedges became loose and this accounts for the lack of growth after about 10 hours. Water vapour absorption measurements on a sheet of the cured adhesive showed that it reached almost constant weight between 6 hours and 24 hours exposure at $50^{\circ}C$ and 96% r.h. This stress relief is consistent with the marked effect of water absorption on modulus and glass transition temperature[6]. Anodise pretreatment gave 30-100% adhesion failure in the initial crack and alumina blast 50-100%, and for both pretreatment crack growth was interfacial. Proportion of adhesion failure and crack length were not correlated.

Wedge driving speed had no significant effect on crack length with pickle and alumina blast treatments, nor on initial crack length with anodise. However, the latter gave significantly higher crack growth at the 20 mm/min speed. At that speed the pretreatments also gave significantly different crack lengths, in the expected order of durability.

4 DISCUSSION

4.1 Calculation of fracture energy

The system property determined by the wedge test is fracture energy G , and this depends on elevated powers of joint dimensions and crack length. This becomes clearer if the equation

$$G = \frac{Ed^2h^3}{16} \cdot \frac{[3(a + 0.6h)^2 + h^2]}{[(a + 0.6h)^3 + ah^2]^2}$$

is reduced to an approximate form accurate enough for this test.

The h^2 and ah^2 terms are derived from the shear contribution to adherend elastic energy and for $a = 25$ mm and $h = 3.25$ mm their neglect gives an error of only +3%. The error becomes even smaller as a/h increases. The equation then becomes

$$G \approx \frac{3Ed^2h^3}{16(a + 0.6h)^4} .$$

The 0.6h term is an empirical average correction for rotation of adherends ahead of the crack tip, permitted by ductile strain or crazing of adhesive. For $a = 25$ mm and $h = 3.25$ mm omission of this term would overestimate G by 35%, falling to 21% at $a = 40$ mm. The proper correction must however be unique to each adhesive and will moreover change as water absorption affects modulus and ductility. Since the test is usually employed in a comparative way, typically to assess pretreatment variations using the same adhesive, this correction could be omitted without serious error for the relative G values.

4.2 Sources of error, variability observed

(a) Yielding of adherends

The equation for G depends on adherend bending being entirely elastic, and it may be shown that adherend surface strain ε is given by

$$\varepsilon = \sqrt{\frac{3G}{Eh}} .$$

Thus adherend bending will be elastic only for

$$G \leqslant \frac{Eh\varepsilon_y^2}{3}$$

where ε_y is alloy yield strain. For alloy 3L72 (used with AF130) G values up to 0.5 kJ/m^2 would give only elastic deformation, in fair agreement with the observation that these adherends had no permanent bend after splitting open the joints. Alloy 3L73 (used with 312/5) permits elastic deformation up to

$G = 1.2\ kJ/m^2$, less than half the apparent value for this adhesive, and these adherends were appreciably bent. Fracture energies of modern structural adhesives commonly fall in the range $1\text{-}4\ kJ/m^2$ and so can be measured only approximately by the standard wedge test[3,7]. Adherend yield may be prevented by leaving sufficient unbonded length at the wedge end, such that initial crack length exceeds the critical value below which yield occurs[8]. However, in this case only the reduced fracture energy following warm humid exposure can be measured.

(b) Anticlastic bending

The adherends are wide and thin so that longitudinal bending is accompanied by appreciable transverse bending in the opposed sense. The crack front on the fracture surface is therefore bow-shaped, more advanced in the centre by up to several millimetres.

(c) Errors in a, d and h

Assuming that wedge bearing edges and crack tip can be clearly located the measurement error in crack length should be negligible. However, differences between opposite sides of a joint suggest variability in a crack length of perhaps up to ±1 mm, probably due to local differences at the edges. For $a = 25$ mm this would give corresponding variability in G of +17% to -15%, decreasing to ±10% at $a = 40$ mm for example.

Nominal or average values are often used for d and h. Stainless steel sheet used for the wedges has thickness tolerances of +0.15 mm to -0.18 mm, giving possible errors in G of -9% to +12%. The adherends have tolerances of +0.11 mm to -0.09 mm giving corresponding errors in G of -10% to +9%.

(d) Observed variability

The frequency of observed coefficients of variation of G are shown in the histogram Fig 6, from 114 sets of measurements on 29 sets of joints including five adhesives, and both aluminium and titanium alloy adherends. Variations in d and h would contribute little to the variability because these were either measured for each joint or were measured averages for sheets of consistent thickness. The small number of high values are believed to reflect real differences between replicates, caused by pretreatment variability. Thus the typical 5-20% coefficient of variation arises mainly from variability in crack length. This variability is less than observed elsewhere[8].

4.3 Practical aspects of the test

(a) Preparation of specimen edges

Adherends are first filed co-planar, to avoid edges of the types shown in section in Fig 7, where a layer of adhesive covers the edge of one or both adherends. Such an external layer is much deeper in the stress direction than the adhesive in the bond and is therefore less strained, so that the visible external crack is retarded relative to the internal crack.

Final smoothing on 600 grade abrasive paper gives an adequate finish for clear location of crack tips with a low power microscope.

(b) Wedge driving speed

This can affect fracture energy, but pressing in the wedges by slow steady closure of a vice should give acceptable consistency. Effect of driving speed is likely to vary with adhesive, pretreatment and time to crack measurement.

(c) Time elapsed before crack measurement

Time between wedge insertion and measurement of initial fracture energy is not critical but should be standarised because slow crack growth occurs even in ambient environments. In warm humid conditions exposure times over about 40 hours may be preferable, because crack growth is by then very slow and discrimination between pretreatments is clearer.

(d) Determination of true displacement, d

An unbonded length is often left at one end of the joint for ease of wedge insertion, leaving a gap between adherends nominally equal to adhesive thickness. Thus wedge and adherends are in direct contact and true displacement is $w - t$. The true gap may be appreciably greater or less than the adhesive thickness if adherends are slightly bent (*eg* by guillotining).

(e) Location and definition of crack tip

Two cases are common. Fig 8a shows an interfacial crack wandering between adherends, and scanning the crack from the opened end to its apparent tip could give a low value for crack length by up to several millimetres. Fig 8b represents crazing, visible as whitening or opacity up to 2 mm ahead of the crack. If well-defined and consistent this region might be regarded as part of the crack.

5 CONCLUSIONS

(a) The wedge test is useful for comparison of pretreatments, giving consistent results for resistance to warm humid conditions in short exposure times. Correlation with longer term tests at moderate loads would be desirable.

(b) Fracture energies of many adhesives are high enough to cause inelastic deformation of the adherends, and cannot be calculated accurately. Nevertheless, results are preferably expressed as fracture energy rather than crack length, to emphasise the full differences between systems.

(c) Apparent fracture energy can be determined with coefficients of variation typically in the range 5-20%.

Table 1

EFFECT ON FRACTURE ENERGY OF TIME OF EXPOSURE TO 50°C AND 96% R.H.

Adhesive 312/5
Pretreatment pickle
Five replicates

Exposure condition and time (hour)	Fracture energy and standard deviation (kJ/m^2)			
	Exposed 24 hours at ambient		Exposed 0.5 hour at ambient	
Ambient 0.5	2.85	0.46	2.58	0.18
Ambient 24	2.70	0.34	-	-
50°C and 96% r.h. 1	2.50	0.35	2.22	0.35
2	2.42	0.33	2.18	0.23
4	2.25	0.38	2.05	0.21
8	2.17	0.35	1.95	0.19
14	2.00	0.25	1.83	0.11
30	1.74	0.34	1.66	0.09
100	1.61	0.40	1.53	0.10

Table 2

EFFECT ON FRACTURE ENERGY OF WEDGE SPEED AND TIME OF EXPOSURE TO 50°C AND 96% R.H.

Adhesive AF130

Adherend pretreatment	Wedge speed (20 mm/min)			Wedge speed (200 mm/min)		
	Exposure time (hour)	Fracture energy (kJ/m^2)	Standard deviation	Exposure time (hour)	Fracture energy (kJ/m^2)	Standard deviation
Pickle	0	0.719	0.070	0	0.724	0.099
	4	0.621	0.126	5	0.618	0.074
	10	0.523	0.048	11	0.541	0.059
	32	0.477	0.031	33	0.534	0.065
	50	0.474	0.034	51	0.505	0.049
	74	0.470	0.023	75	0.488	0.062
	98	0.460	0.017	99	0.454	0.027
	124	0.452	0.016	121	0.454	0.027
	220	0.442	0.021	220	0.454	0.027
Anodise	0	0.482	0.047	0	0.550	0.079
	2	0.482	0.047	4	0.485	0.048
	5	0.466	0.056	7	0.452	0.036
	19	0.463	0.060	21	0.426	0.046
	37	0.435	0.049	39	0.420	0.051
	41	0.403	0.029	42	0.369	0.031
	66	0.371	0.041	66	0.359	0.031
	90	0.323	0.056	88	0.359	0.031
	110	0.319	0.054	108	0.358	0.031
	206	0.317	0.054	204	0.357	0.031
Alumina blast	0	0.392	0.046	0	0.487	0.077
	4	0.254	0.049	5	0.254	0.040
	9	0.205	0.046	12	0.192	0.028
	15	0.181	0.033	19	0.167	0.024
	21	0.157	0.020	41	0.150	0.018
	45	0.130	0.010	65	0.141	0.014
	66	0.133	0.028	85	0.130	0.015
	86	0.130	0.025	181	0.113	0.022
	182	0.118	0.010			

Table 3

EFFECT ON CRACK LENGTH OF WEDGE SPEED AND TIME OF EXPOSURE TO 50°C AND 96% R.H.

Adhesive FM1000

Adherend pretreatment	Wedge speed (20 mm/min)			Wedge speed (200 mm/min)		
	Exposure time (hour)	Crack length (mm)	Standard deviation	Exposure time (hour)	Crack length (mm)	Standard deviation
Pickle	0	11.1	0.94	0	11.7	0.89
	1	12.2	0.63	2	12.7	0.74
	3	12.6	0.99	4	12.9	0.77
	4	13.1	1.08	8	12.9	0.81
	8	13.2	1.07	22	12.9	0.84
	15	13.3	1.11	28	13.0	0.90
	21	13.4	1.12	44	13.0	0.90
	28	13.5	1.09	70	13.0	0.90
	51	13.7	1.10	100	13.0	0.92
	72	13.7	1.10	141	13.3	1.22
	101	13.7	1.10			
	142	13.8	1.10			
Anodise	0	12.7	0.55	0	12.5	0.71
	2	15.2	1.53	2	12.9	1.41
	5	15.8	1.99	4	13.2	1.41
	8	16.0	2.00	7	13.3	1.42
	14	16.3	2.26	10	13.3	1.47
	21	16.5	2.14	15	13.4	1.46
	37	16.7	2.28	31	13.6	1.42
	61	16.8	2.35	55	13.8	1.19
	86	16.8	2.38	79	13.8	1.22
	102	16.9	2.41	103	13.9	1.30
				127	13.9	1.30
				150	14.0	1.50
Alumina blast	0	14.6	0.81	0	14.2	1.19
	1	17.2	1.17	1	16.5	1.54
	2	17.7	1.23	2	16.9	1.45
	4	18.0	0.87	4	17.1	1.60
	8	18.3	1.01	8	17.4	1.68
	16	18.6	1.03	15	17.6	1.68
	23	18.9	1.06	23	18.3	1.88
	28	19.3	1.21	31	19.6	1.82
	32	19.8	1.46	49	19.9	2.07
	50	20.8	1.65	74	19.9	2.09
	75	21.2	1.85	98	20.0	2.06
	99	21.7	1.82	121	21.8	2.06
	122	23.4	2.91	162	23.3	2.94
	163	24.5	3.07			

LIST OF SYMBOLS

a	crack length, from bearing edges of wedge to crack tip (Fig 1) (m)
d	displacement of adherends by wedge (d may equal w or $w - t$) (m)
E	Young's modulus of adherends (Pa)
G	fracture energy (J/m^2)
h	thickness of adherends (assumed equal) (m)
t	thickness of adhesive layer (m)
w	thickness of wedge (m)
ε	strain at surface of adherends
ε_y	yield strain of adherends

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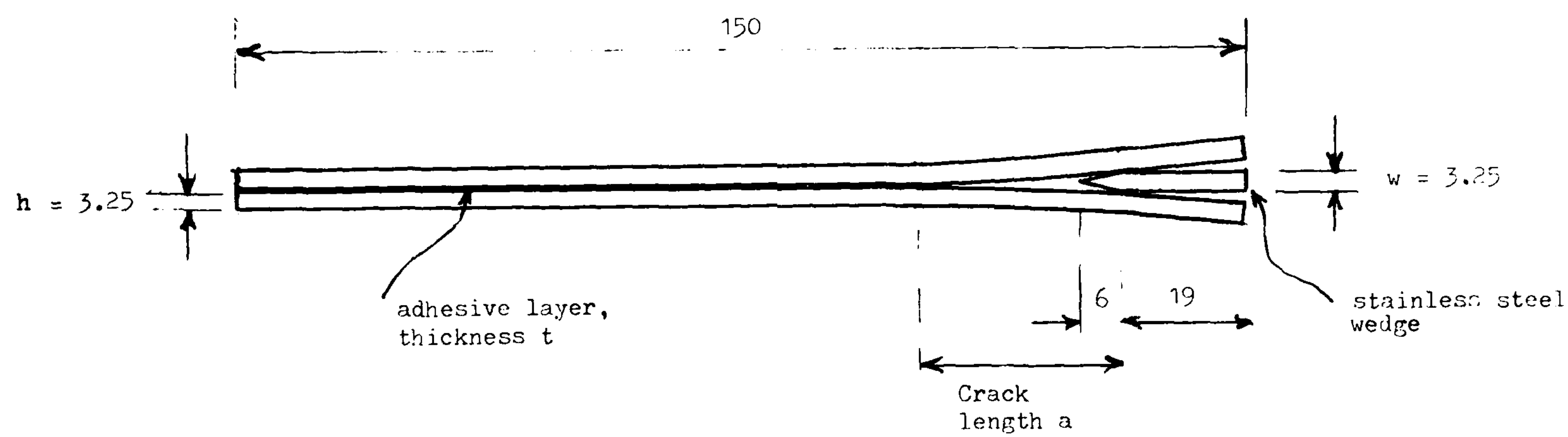


Dimensions mm
Adherend width 25 mm

Fig 1 Side view of wedge test specimen with wedge inserted, dimensions for aluminium alloy adherends

Fig 2

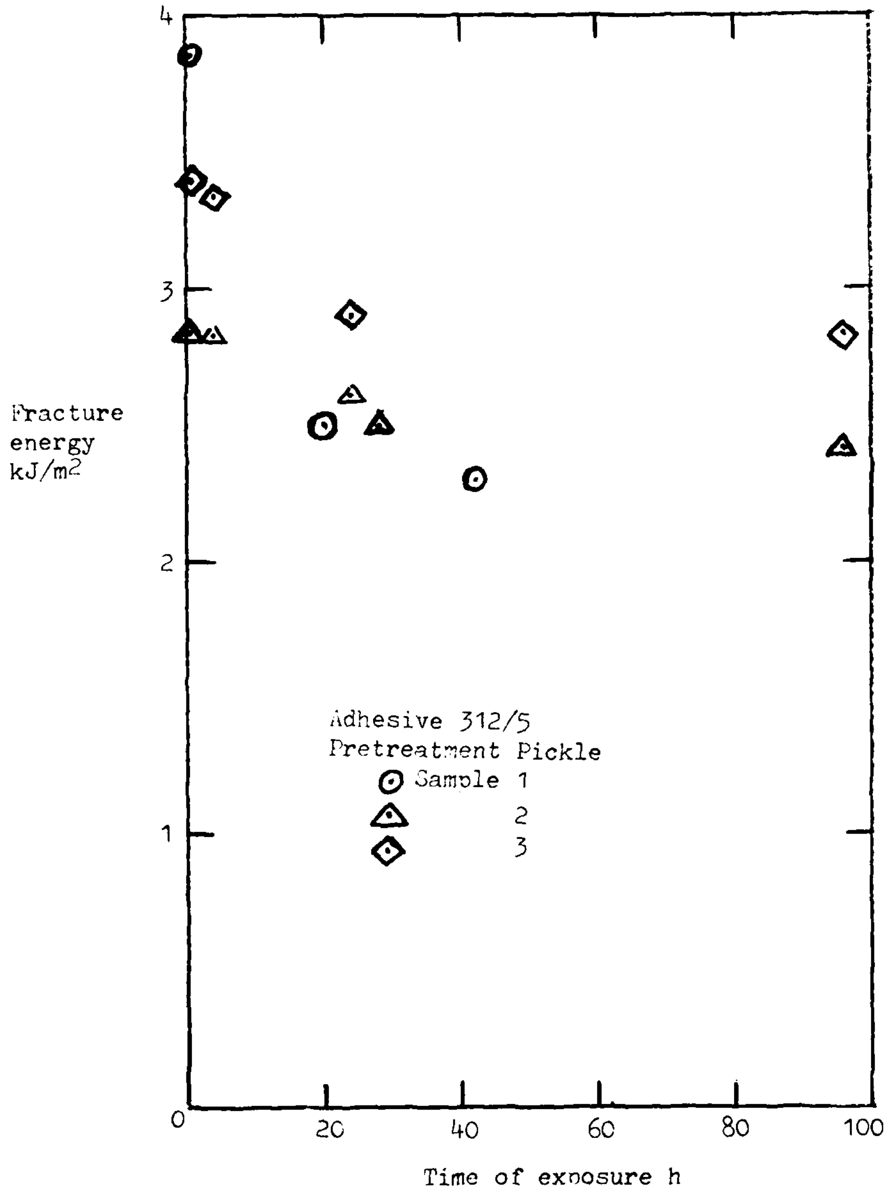


Fig 2 Decrease in fracture energy with time of exposure to ambient conditions (three replicate joints)

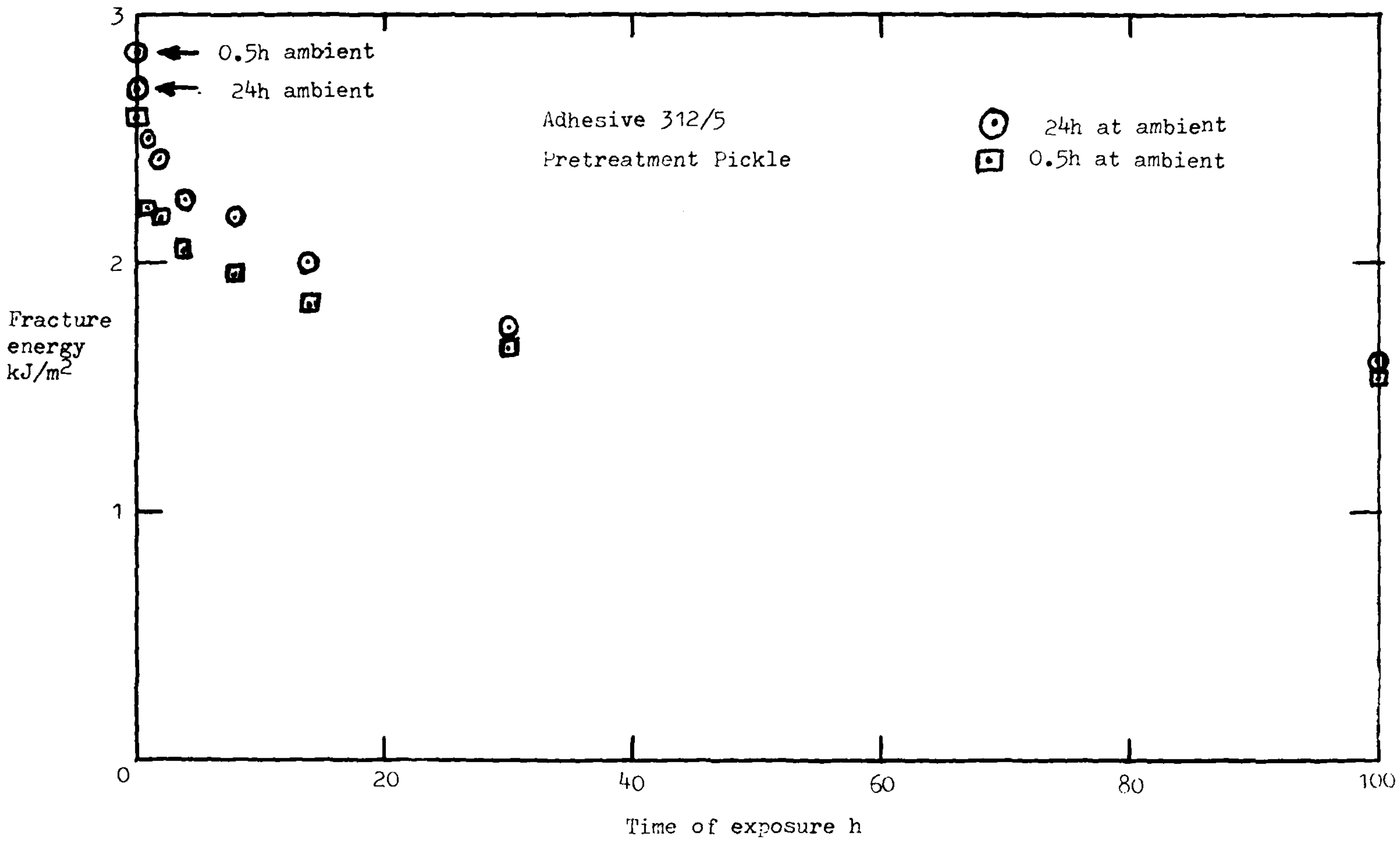


Fig 3 Decrease in fracture energy with time of exposure to $50^{o}C$ and 96% r.h., effect of previous ambient exposure

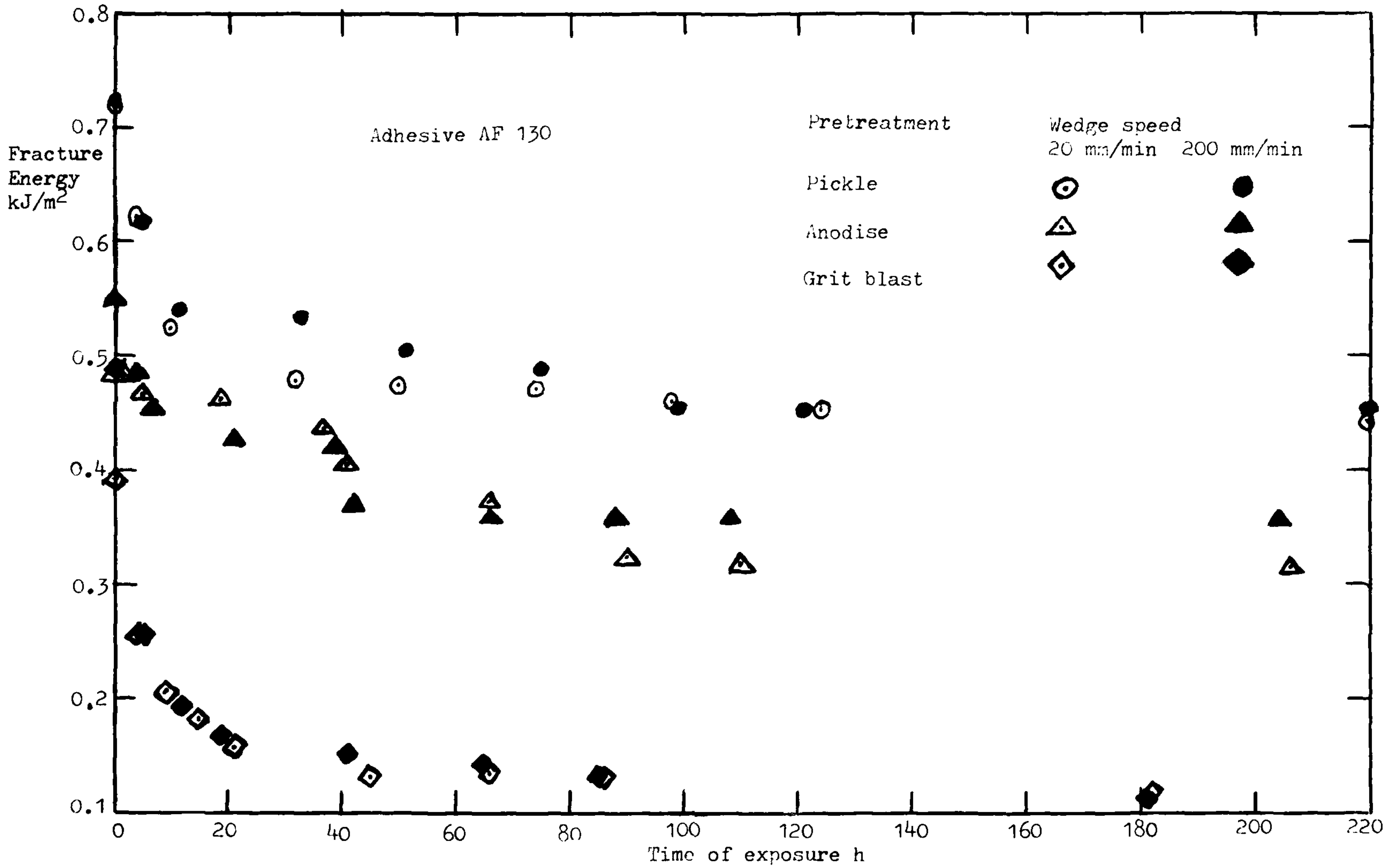


Fig 4 Decrease in fracture energy with time of exposure to 50^{o}C and 96% r.h., as affected by adherend pretreatment and wedge driving speed

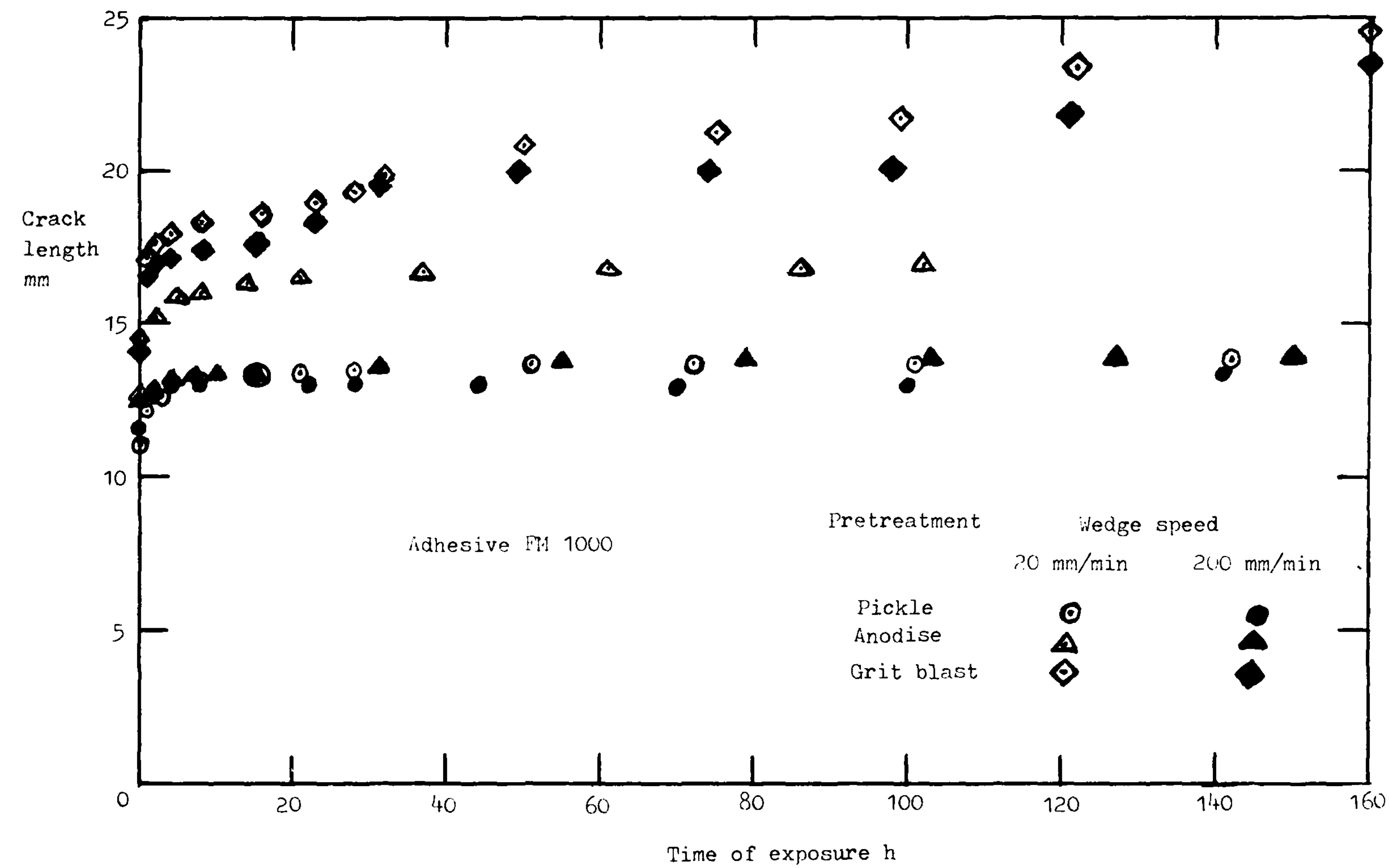


Fig 5 Increase in crack length with time of exposure to 50°C and 96%r.h., as affected by adherend pretreatment and wedge driving speed

Fig 6

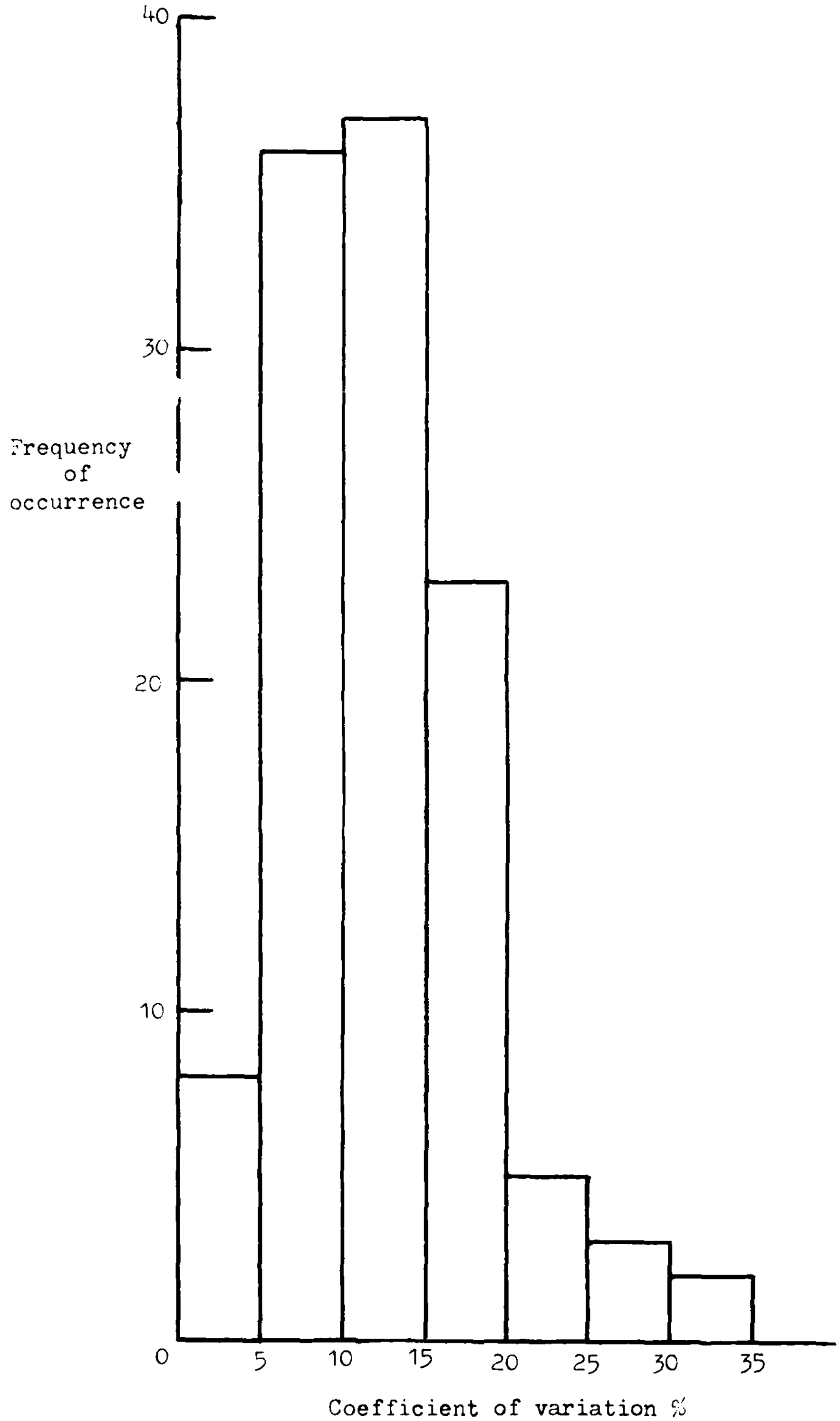


Fig 6 Frequency of occurrence for coefficient of variation by 5% bands

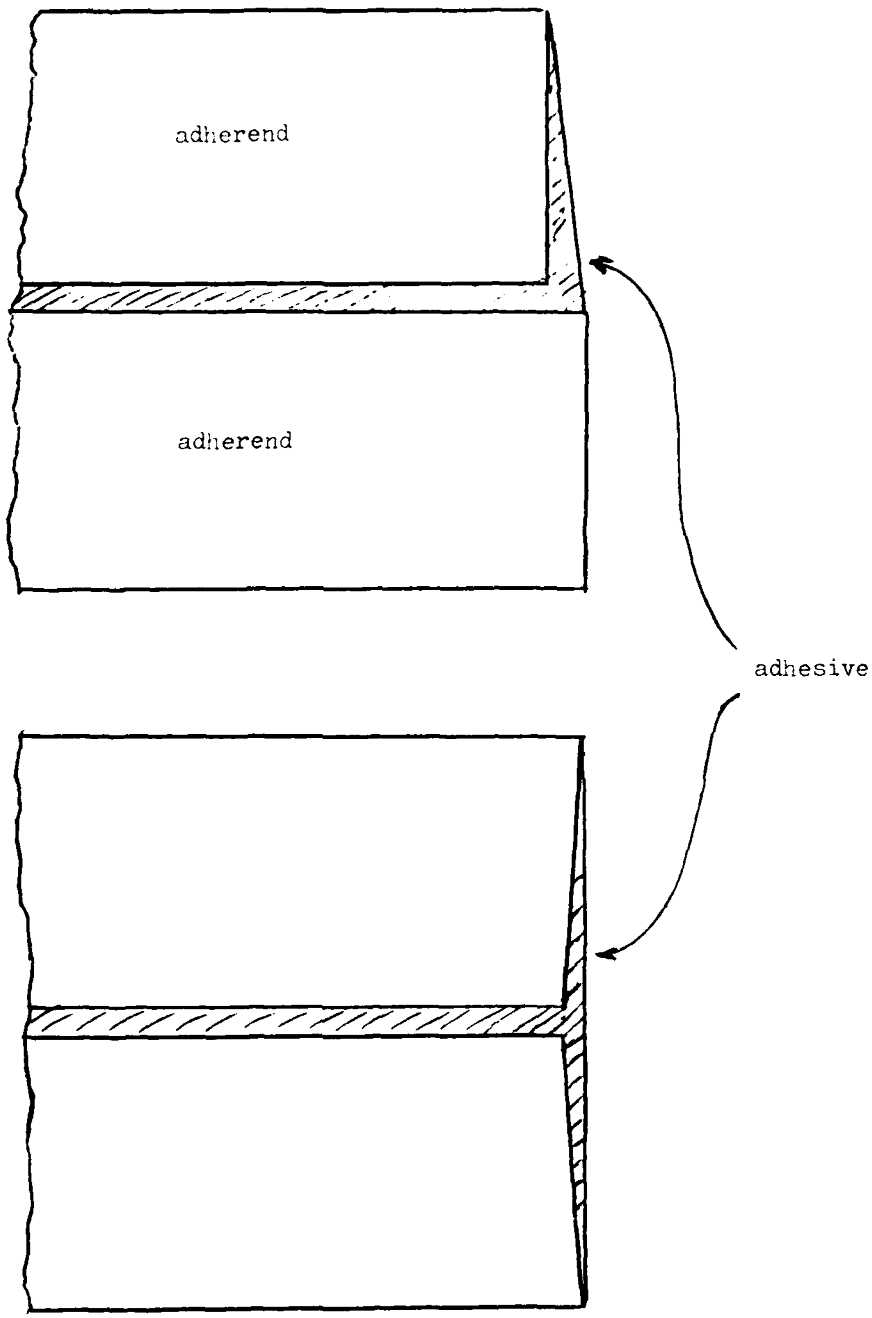
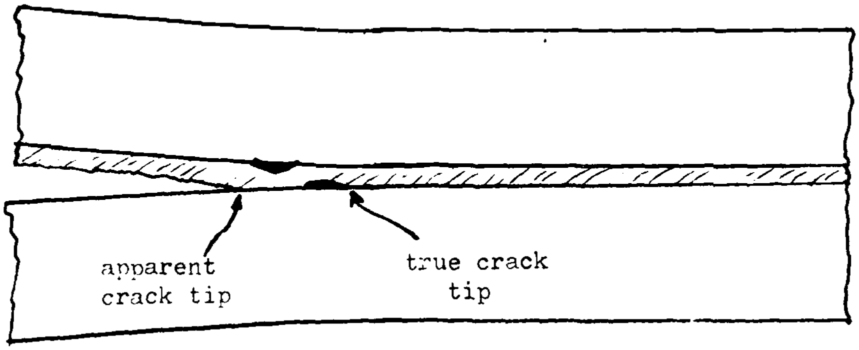


Fig 7 Typical cross-sections of joints at unprepared edges, external adhesive layer masking bond line

Fig 8



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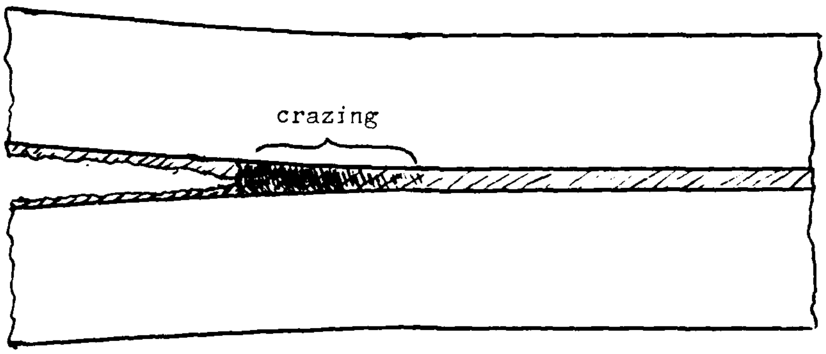

b

Fig 8 Characteristic types of crack front region

REPORT DOCUMENTATION PAGE

Overall security classification of this page

UNLIMITED

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17. Abstract

The Boeing wedge test was applied to joints of aluminium alloy made with three adhesives differing widely in toughness. Bonding pretreatments chromic-sulphuric acid pickle, chromic acid anodise and wet alumina grit blast were compared for two of the adhesives, and clear differences in crack length appeared after exposure to 50°C and 96% r.h. Rates of crack growth were determined in both ambient and warm humid environments, and suitable times are suggested for measurement of crack length. Wedge driving speed can affect results and should be standardised.

Results are more informative if shown as fractures energies rather than crack lengths, and the approximations involved are discussed. Practical aspects of the test are described and possible errors quantified. Fracture energies typically showed coefficients of variation of 5-20%.

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