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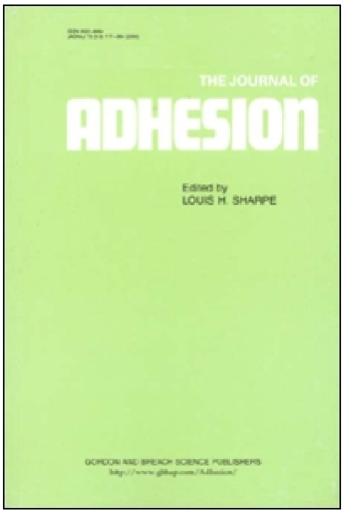

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The Fracture Resistance of a Toughened Epoxy Adhesive

A. J. Kin Loch[a]; S. J. Shaw[a]
[a] Ministry of Defence, PERME, Waltham Abbey, Essex, England

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The Fracture Resistance of a Toughened Epoxy Adhesive

A. J. KINLOCH and S. J. SHAW
Ministry of Defence, PERME, Waltham Abbey, Essex EN9 1BP, England

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The fracture resistance of a rubber-modified epoxy adhesive has been studied using a continuum fracture-mechanics analysis. The fracture energy, G_{Ic}, has been ascertained over a range of test temperatures and rates, both in bulk and in adhesive joints, as a function of specimen geometry. The results obtained are described and a semi-quantitative model discussed for predicting the joint failure behaviour from the bulk adhesive properties.

INTRODUCTION

In recent years rubber-modified epoxy materials have become the basis of a new generation of advanced structural adhesives.[1] The cured adhesive usually exhibits a two-phase microstructure, as illustrated in Figure 1, consisting of small particles of rubber-epoxy copolymer embedded in a matrix of the epoxy. This microstructure results in the material possessing a considerably higher toughness or crack resistance, compared to the unmodified system, but with only a minimal reduction in other important properties such as modulus and high-temperature and creep resistance.

However, the fracture behaviour of joints employing such toughened adhesives is extremely complex and they present the designer with a challenging task if they are to be employed with maximum efficiency. The present work was therefore initiated (i) to gain an understanding of the effect of joint design parameters, such as adhesive bond thickness and width, and of service conditions, such as temperature and rate of loading and (ii) to enable the joint failure behaviour to be predicted from the bulk adhesive properties. The fracture resistance has been measured employing a continuum fracture-mechanics analysis.

Presented at the International Conference on "Adhesion and Adhesives" of the Plastics and Rubber Institute held at Durham University, England, September 3–5, 1980.

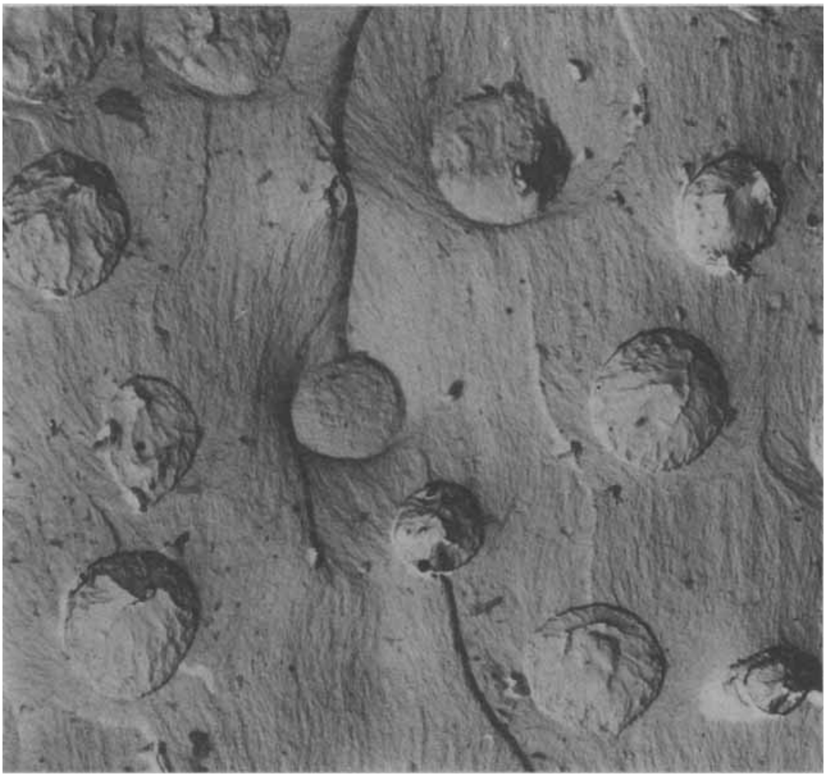


FIGURE 1 Replica transmission electron micrograph of fracture surface of rubber-modified epoxy material. Magnification × 7500.

EXPERIMENTAL

Materials

The rubber-modified epoxy material consisted of an epoxy resin, which was a diglycidyl ether of bisphenol-A (DGEBA), together with 15 parts per hundred of resin (phr) of carboxyl-terminated butadiene-acrylonitrile rubber (CTBN) and 5 phr of piperidine as the curing agent. To prepare this toughened epoxy the CTBN was added to the DGEBA and hand-mixed for approximately five to ten minutes. This mixture was then heated to 65 ± 5°C in a water bath and mixed for five minutes using an electric stirrer and then degassed in a vacuum oven at 60°C until frothing stopped, *i.e.* until most of the entrapped air was removed. When the mixture had cooled to below 30°C the piperidine was mixed in gently to minimise air entrapment.

The rubber-modified epoxy was then poured into a mould or between the substrates to form a joint, as required. The metal mould or substrates were always pre-heated to 120°C. Finally, to effect cure of the adhesive, it was heated at 120°C for 16 h and then allowed to cool slowly.

Bulk compression tests

Previous work[2, 3, 4, 5] has highlighted the importance plastic flow mechanisms around the crack tip may have upon a material's fracture behaviour. However, when tested in uniaxial tension the rubber-modified epoxy suffered brittle fracture prior to yielding. The yield behaviour was therefore examined by testing in uniaxial compression. Samples were cast and machined into blocks with a height-to-diameter ratio of about 2 to 1. The blocks were deformed in a compression cage between polished steel plates, lubricated with molybdenum disulphide grease, in an Instron mechanical testing machine. A constant cross-head displacement rate, $\dot{y}$, was used for each test and this was converted to a strain-rate, $\dot{e}$, using the specimen dimensions. The nominal strain, e, was determined from the crosshead displacement corrected for the machine deflection using a steel blank. The load, P, at yield was measured from the Instron chart and converted into a true compressive yield stress, σ_{yc}, using the initial specimen cross-sectional area, A_0, in the equation:

$$\sigma_{yc} = P(1-e)/A_0 \tag{1}$$

which assumes constant volume deformation.

Bulk fracture studies

Sheets of the rubber-modified epoxy were cast possessing widths, H, between 3 and 50 mm. The sheets having a width between 6 and 50 mm were machined and drilled to form the compact-tension specimen, shown in Figure 2. This specimen geometry was unsuitable for 3 mm wide samples and sheets of this width were made into the single-edge crack geometry.[6] In both cases a sharp crack was formed at the base of the slot by tapping carefully a fresh razor blade into the base which caused a natural crack to grow for a short distance ahead of the razor blade. The specimen was then mounted in an Instron tensile testing machine and loaded at various constant displacement rates and temperatures. The maximum load, P_c, at which crack growth occurred was recorded. Values of the fracture energy, G_{Ic} (bulk adhesive) were calculated from[6, 7, 8]

$$G_{Ic}\,(\text{bulk adhesive}) = (P_c Q/HW)^2 (a/E_a) \tag{2}$$

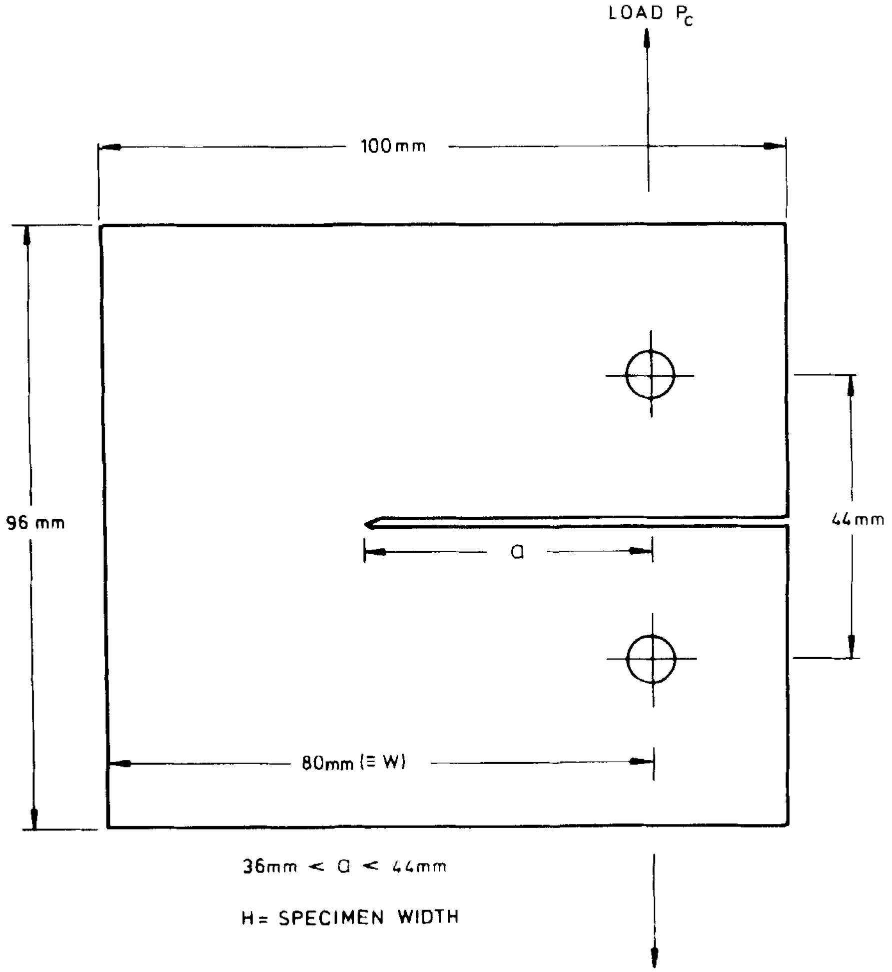


FIGURE 2 Compact tension specimen.

where:

a = crack length
H = width of specimen
W = effective length of specimen
E_a = modulus of adhesive
Q = geometry factor
for compact tension specimen:
$= [29.6 - 185.5(a/W) + 655.7(a/W)^2 - 1017(a/W)^3 + 638.9(a/W)^4]$
for single-edge crack specimen:
$= [1.99 - 0.41(a/W) + 18.7(a/W)^2 - 38.48(a/W)^3 + 53.85(a/W)^4]$

The value of E_a was taken to be the uniaxial compressive modulus. The bulk, and adhesive joint, fracture data was related to the bulk compressive data by approximately equating the time-to-failure in the former tests to the time-to-yield in the latter. This resulted in a displacement rate, $\dot{y}$, of, say, 8.3×10^{-7} m/s being equivalent to a strain rate, $\dot{e}$, of 8.3×10^{-5} s^{-1}, *i.e.* a two decade difference.

Joint fracture studies

The specimen geometry employed for the adhesive joints was a contoured-double-cantilever-beam joint, as shown schematically in Figure 3. The substrate material was mild steel, to specification British Standard 970, EN3B which was machined into cantilever beams 308 mm long, with the height, h, varying between 15.8 mm and 51.0 mm and of widths, H, 3, 6, 12, 25 or 50 mm. The surfaces to be bonded were first subjected to a liquid- and vapour-degreasing bath of trichloroethane, then grit-blasting with 180–220 mesh alumina, then after degreasing again were finally allowed to air-dry. Two beams were pressed down firmly on adhesive tape, supported on a glass plate, with small pieces of plastic sheet inserted between the beams at each end to control the gap between them. Further, a piece of Teflon tape the width of the joint, about 30 mm long and 0.08 mm thick was placed approximately in the centre of the gap and at the narrow end of the joint to assist in propagating a "starter" crack.

The rubber-modified epoxy adhesive was cast into the gap and cured. To obtain natural starter cracks for subsequent experiments the arms of the specimens were separated at a constant rate of 8.5×10^{-3} mm/s using an

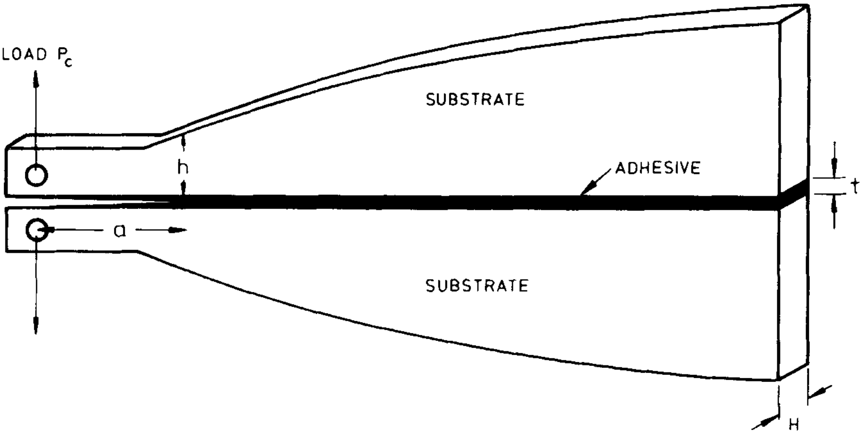


FIGURE 3 Contoured-double-cantiliver-beam adhesive joint specimen.

Instron tensile testing machine until the crack was about 70 to 100 mm long. The specimens were then reloaded at various constant rates of displacement at 20°C until crack propagation was observed. The adhesive fracture energy, G_{Ic} (joint), was determined from the relationship[7]:

$$G_{Ic}\text{ (joint)} = (4P_c^2 m)/(E_s H^2)$$

where P_c = load at onset of crack growth

E_s = modulus of substrate (210 GPa)

H = width of specimen

$m = \{(3a^2/h^3)+(1/h)\}$ = constant = 2 mm^{-1}

RESULTS

Yield and modulus data

Values of the true compressive yield stress, σ_{yc}, and modulus, E_a, of the rubber-modified epoxy as a function of strain-rate, $\dot{e}$, and temperature are shown in Figures 4 and 5 respectively. The viscoelastic behaviour of the material may be readily observed from the time-temperature dependence of σ_{yc} and E_a; values of σ_{yc} and E_a increasing as the strain-rate increases or the temperature decreases.

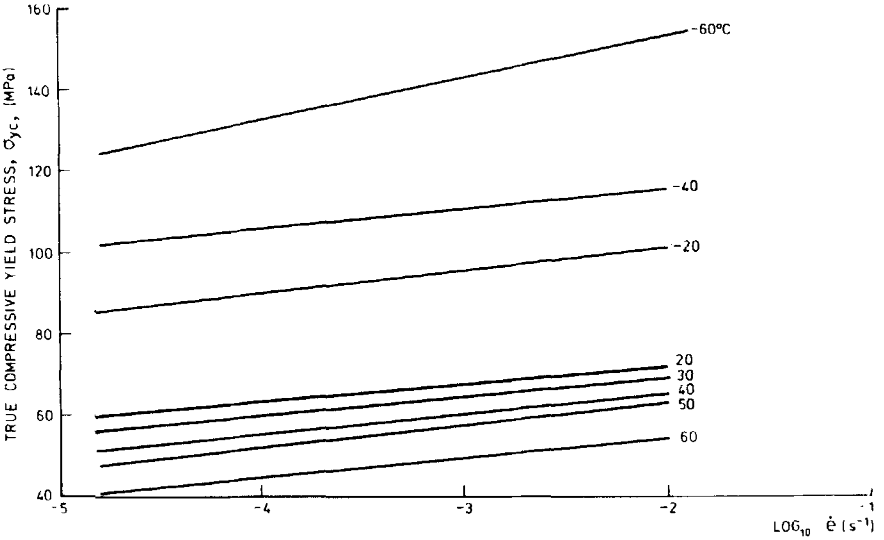


FIGURE 4 True compressive yield stress, σ_{yc}, v. logarithm of strain-rate, $\dot{e}$, for rubber-modified epoxy.

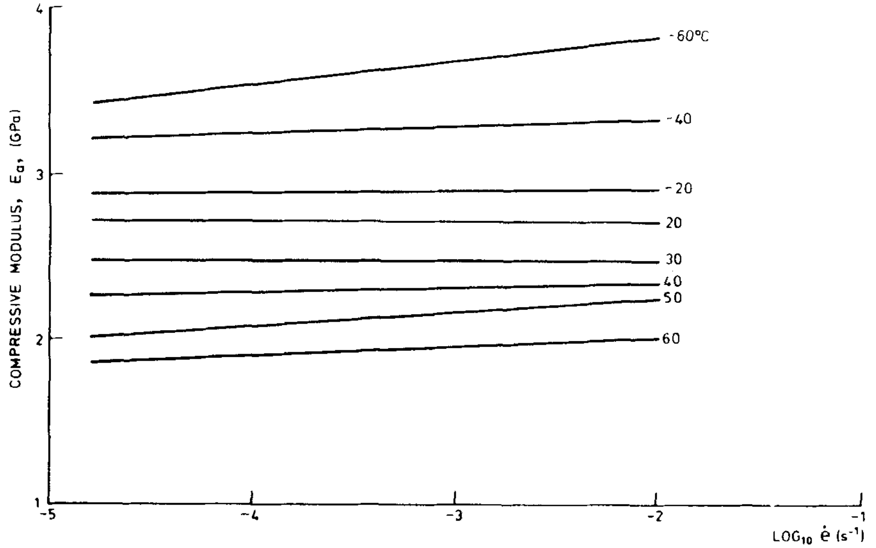


FIGURE 5 Compressive modulus, E_a, *v*. logarithm of strain-rate, $\dot{e}$, for rubber-modified epoxy.

Adhesive joint fracture

The adhesive fracture energy is shown as a function of adhesive bond thickness, t, at various displacement rates, $\dot{y}$, (temperature: 20°C, specimen width, H; 12 mm) in Figure 6 and at various specimen widths (temperature: 20°C, $\dot{y}$: 1.67×10^{-5} m/s) in Figure 7. In all cases the fracture behaviour is dominated by a strong dependence upon the adhesive bond thickness, t, employed, as previously found by Bascom and co-workers[9,10] for tough adhesive systems. The adhesive fracture energy passes through a maximum value, G_{Icm}, at a certain bond thickness, t_m. The value of this maximum adhesive fracture energy, G_{Icm} (joint), is, however, dependent upon the rate, temperature (data taken from reference 10) and specimen width, as may be seen from Figures 8, 9 and 10 respectively.

Throughout these tests the locus of joint failure was cohesive in the adhesive. However, in accord with previous observations,[9,10] at bond thicknesses greater than the maximum ($t > t_m$) the crack tended to grow in the centre of the bond in an unstable, stick–slip manner whilst at $t < t_m$ the crack propagated near an interface in a continuous, stable manner. The deformation zone at the crack tip was clearly visible due to the stress whitening which occurred, particularly at relatively low rates or high temperatures. In those instances where unstable crack growth occurred the G_{Ic} value for

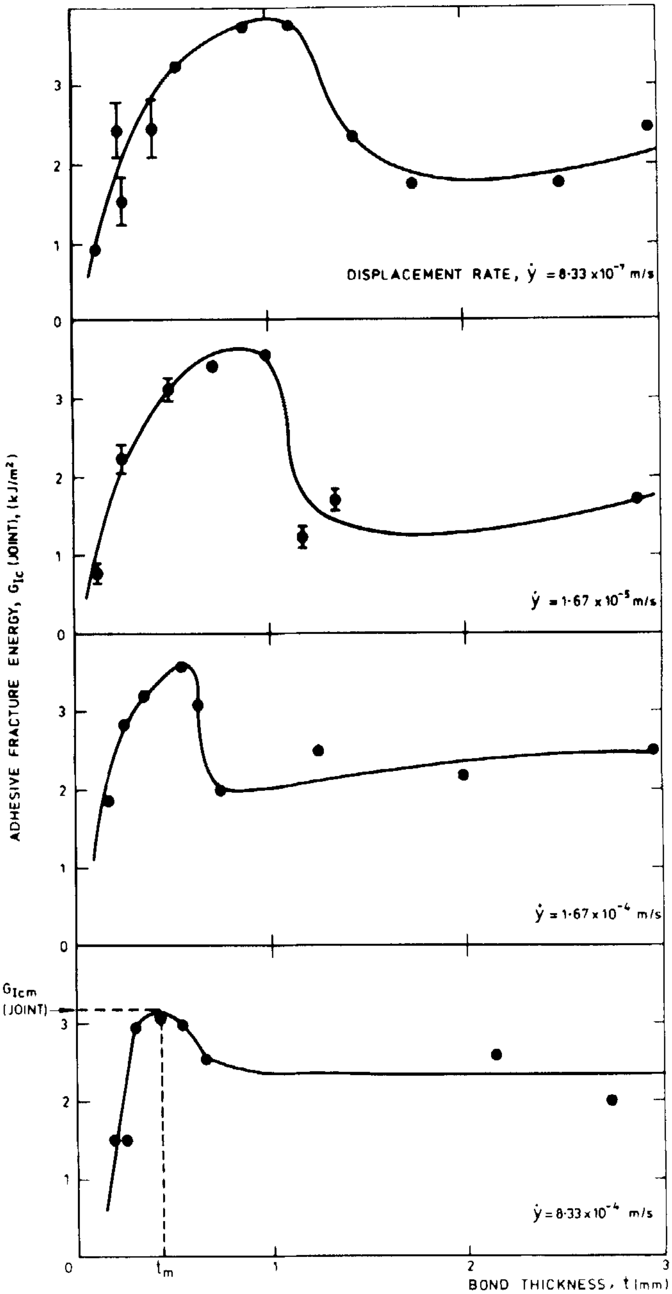


FIGURE 6 Adhesive fracture energy, G_{Ic}, as a function of bond thickness, t, for various constant rates of displacement, $\dot{y}$. (Temperature: 20°C; joint width: 12 mm.)

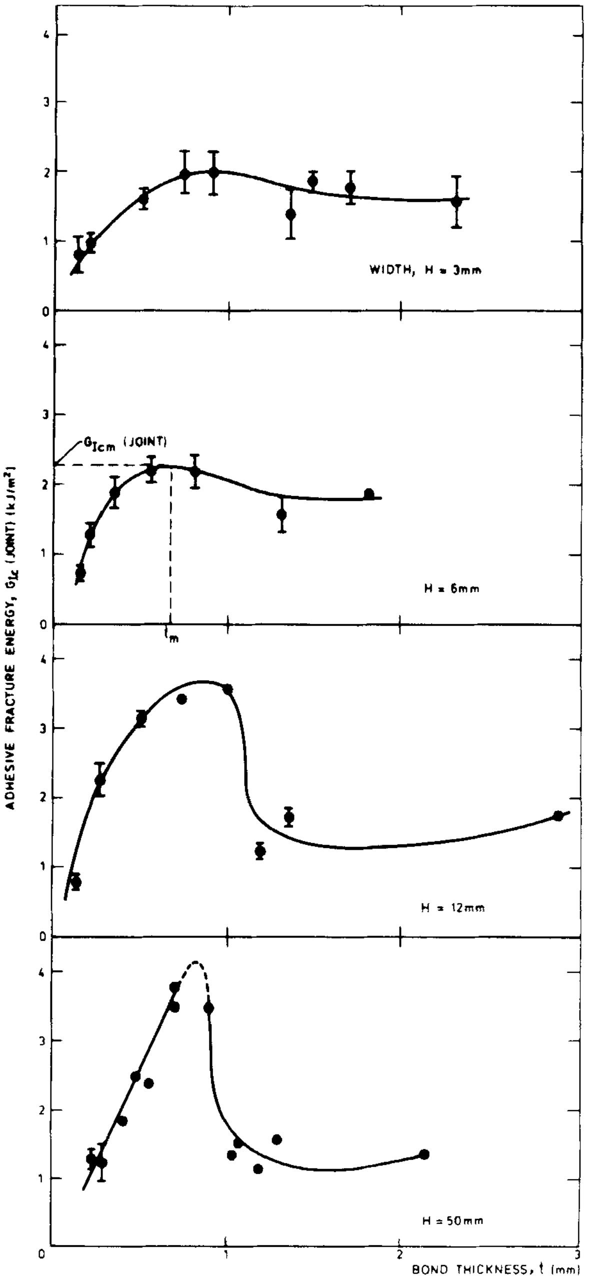


FIGURE 7 Adhesive fracture energy, G_{Ic}, as a function of bond thickness, t, for various joint widths, H. (Temperature: 20°C; $\dot{y}$: 1.67×10^{-5} m/s.)

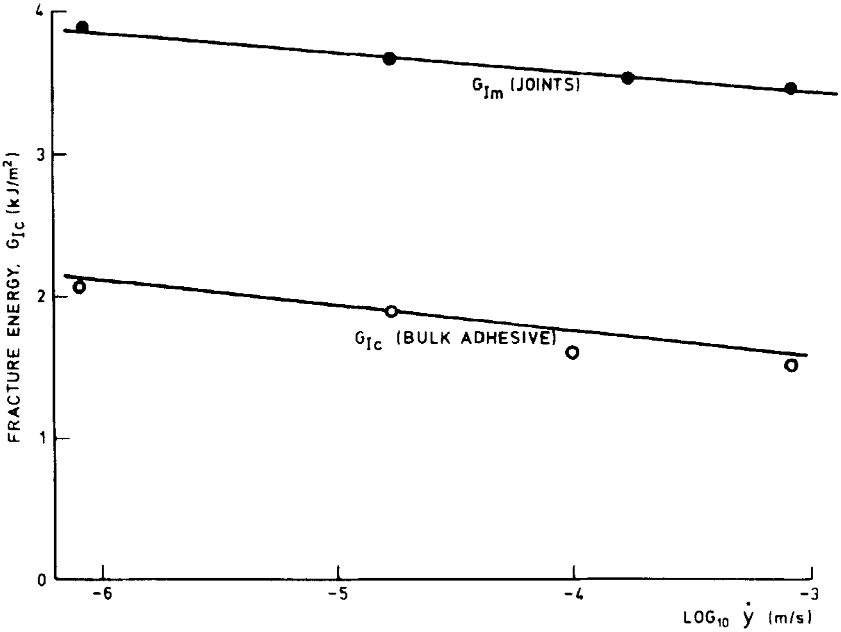


FIGURE 8 Fracture energy *v.* logarithm of displacement rate, $\dot{y}$. (Temperature: 20°C; specimen width: 12 mm.)

crack arrest was about 0.4 kJ/m^2 and virtually independent of rate, specimen width and temperature.

Bulk fracture

Values of the fracture energy, G_{Ic} (bulk adhesive) of the rubber-modified epoxy material are shown as a function of rate, $\dot{y}$, temperature and specimen width, H, in Figures 8, 9 and 10 respectively. The mode of crack growth was unstable stick–slip under the conditions studied.

DISCUSSION

Effect of adhesive bond thickness

The results shown in Figures 6 and 7 clearly confirm the attainment of a maximum, G_{Icm} (joint), value at an optimum thickness, t_m although the maximum is far less pronounced when the specimen width, H, is narrow. Bascom and co-workers[10] have previously reported the attainment of a

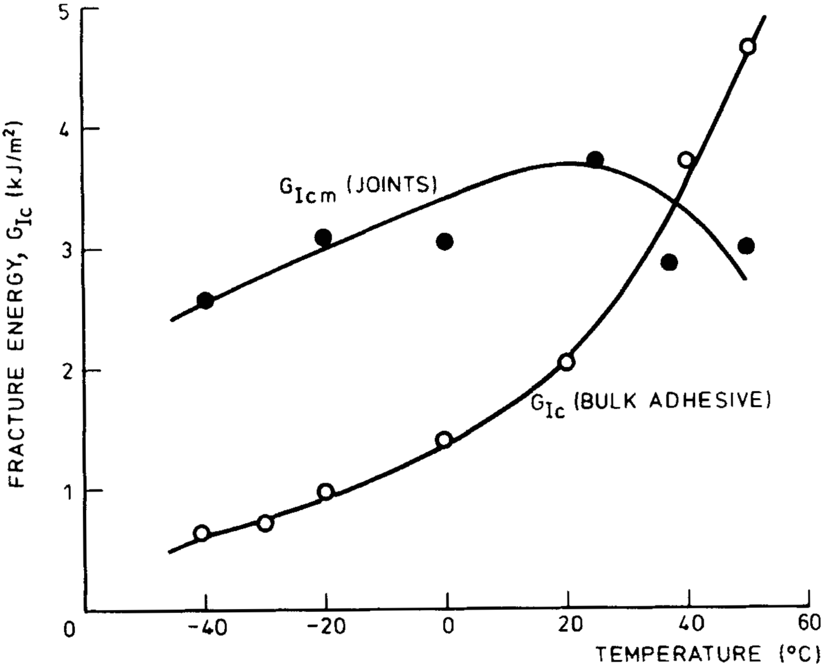


FIGURE 9 Fracture energy *v.* temperature. (Specimen width 12 mm; $\dot{y}=2.19\times10^{-5}$ m/s.)

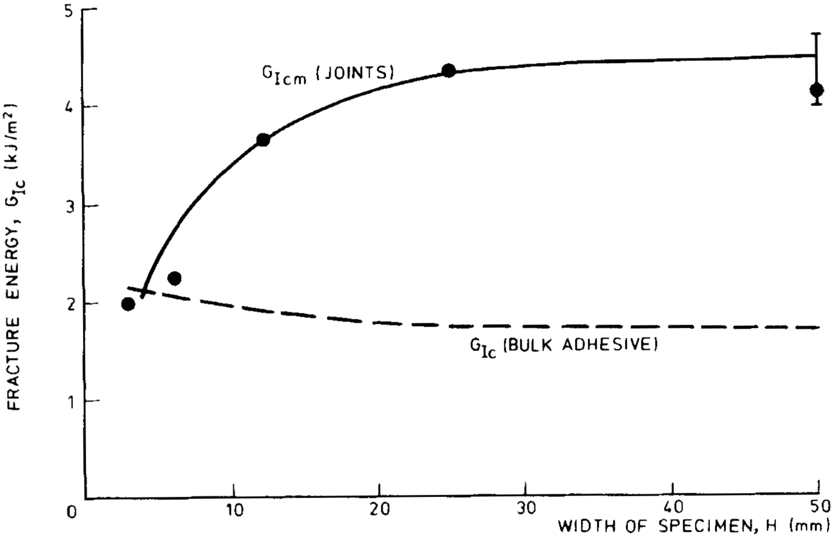


FIGURE 10 Fracture energy *v.* specimen width, H. (Temperature = 20°C; $\dot{y}=1.67\times10^{-5}$ m/s.)

maximum value when they examined the variation of G_{Ic} (joint) with t as a function of temperature. To interpret the occurrence of this maximum they employed an elastic-plastic model for the deformation zone surrounding the crack tip, which has been widely used for epoxy[2–5] and other polymeric[4,11] materials. The model is shown schematically in Figure 11 and the radius of the plastic-deformation zone, r_{Iy}, is given by:[7,8]

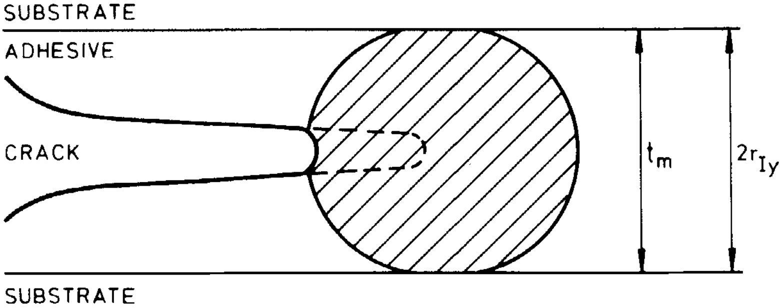


FIGURE 11 Simple elastic-plastic model for deformation zone at crack tip. As drawn $t = t_m$.

$$r_{Iy} = \frac{1}{2\pi} \frac{E_a G_{Ic}}{\sigma_{yt}^2} \quad \text{in plane-stress} \tag{4}$$

$$r_{Iy} = \frac{1}{6\pi} \cdot \frac{E_a G_{Ic}}{\sigma_{yt}^2} \cdot \frac{1}{(1-\nu^2)} \quad \text{in plane-strain} \tag{5}$$

where ν is Poisson's ratio and σ_{yt} is the uniaxial tensile yield stress. Now in a sheet containing a crack the state of stress near the crack tip varies from plane-stress in a very thin specimen, or in the edge regions of a thick specimen, to plane-strain in the central regions of a thick specimen. The increased constraint introduced under the influence of plane-strain conditions elevates the tensile stress necessary for yielding and thus the plastic zone is smaller in plane-strain; cf. Equations (4) and (5). Bascom *et al.*[9,10] employed Equation (5) and assumed the value of σ_{yt} to be the uniaxial tensile fracture stress and showed that at the maximum, G_{Icm} (joint), the deformation zone, $2r_{Iy}$, was approximately equal to the bond thickness, t_m. In the present paper, however, the actual value of the tensile yield stress, σ_{yt}, may be deduced, since:[12–14]

$$\sigma_{yt} \approx 0.75\sigma_{yc} \tag{6}$$

Furthermore, since the value of r_{Iy} is greater at the edge of the joint (plane-stress conditions acting) we would suggest that Equation (4), employing G_{Ic} (bulk adhesive), is the more applicable.

The values of $2r_{Iy}$, calculated from Equation (4) are shown together with those of t_m in Table I. As may be seen the correlation is very good and thus the present work supports the proposals of Bascom and co-workers, although via a slightly modified argument.

The decline in G_{Ic} (joint) values at adhesive bond thicknesses, t, less than t_m may now be readily understood. Namely, the presence of the high modulus substrates restricts the full volume of the plastic-zone from developing and, since the toughness is mainly derived from the energy dissipated in forming the plastic-zone, then the adhesive fracture energy is steadily reduced as the bond thickness is decreased. The decline, often very rapid, in G_{Ic} (joint) when $t > t_m$ is not fully understood but will be considered below.

TABLE I

Comparison of adhesive bond thickness, t_m, at maximum fracture energy, G_{Icm}, and plane-stress plastic-zone diameter.

Temp (°C)	$\text{Log}_{10}\,\dot{y}$ (m/s)	Joint width, H (mm)	t_m (mm)	$2r_{Iy}$ (mm) (b)
20	−6.08	12	1.0	0.85
20	−4.78	12	0.8	0.70
20	−3.78	12	0.55	0.49
20	−3.08	12	0.4	0.43
20	−4·78	3	0.85	0.75
20	−4.78	6	0.65	0.72
20	−4.78	12	0.8	0.70
20	−4.78	25	0.7	0.65
20	−4.78	50	0.8	0.61
50	−4.66	12	1.1 (a)	1.6
37	−4.66	12	0.9	1.16
25	−4.66	12	0.6	0.57
0	−4.66	12	0.5	0.39
−20	−4.66	12	0.25	0.15
−40	−4.66	12	0.1	0.05

Note: (a) After reference 10
(b) Calculated from Equation (4)

Effect of rate and temperature

Considering first the bulk adhesive material; the previous studies[3–5] have shown that the failure of this material is governed by the need to achieve a critical stress acting over a certain distance ahead of the crack tip. Thus, as the yield stress of the material decreases then crack tip blunting becomes more severe due to the larger extent of plastic deformation around the tip. The lower stress-concentrating effect of the blunt crack means that a higher applied load is required to attain the value of the critical stress. This in turn, implies a high initial G_{Ic} value, *i.e.* G_{Ic} (initiation) > G_{Ic} (propagation), and the relatively large amount of stored elastic energy in the sample at the onset of crack propagation results in fast unstable crack growth until the energy supply is insufficient to sustain crack growth and crack arrest occurs. Thus, since the yield stress falls with decreasing rate and increasing temperature the fracture energy would be predicted to behave in the reverse manner, as indeed it does; see Figures 8 and 9. This inter-relation between rate and temperature clearly emphasises the importance of the viscoelastic response of the adhesive. Recent elegant work by Hunston *et al.*,[15] has even demonstrated that it is possible to apply the time-temperature superposition procedure to the fracture energy data to produce a well defined master curve.

Turning to the adhesive joints, the situation is far more complex since the value of G_{Ic} (joint) at any particular rate and temperature is also a function of bond thickness, t. For example, if G_{Ic} (joint) is plotted against rate, $\dot{y}$, at various constant bond thicknesses, $t = 0.15$, 0.30 or 1.0 mm as shown in Figure 12, then the dependence of G_{Ic} (joint) upon $\dot{y}$ is clearly a function of the bond thickness selected. A similar effect is recorded[16] if the variable is temperature.

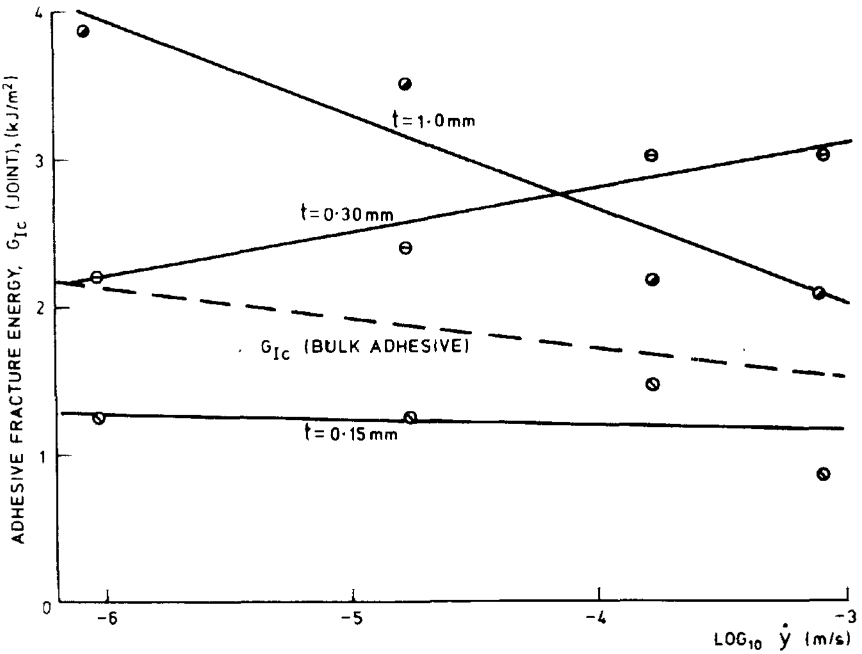


FIGURE 12 Adhesive fracture energy *v.* logarithm $\dot{y}$ for various constant adhesive bond thicknesses, t.

A somewhat more fruitful approach is to plot G_{Icm} (joint) against rate or temperature, as shown in Figures 8 and 9 respectively. However, it should be borne in mind that the relevant bond thickness, t_m, is not now a constant. The value of t_m increases with decreasing rate or increasing temperature, as indicated in Table 1. Examination of Figure 8 reveals that for the different rates studied the behaviour of G_{Icm} (joint) and G_{Ic} (bulk) is similar but G_{Icm} (joint), for this particular width of specimen, is greater than G_{Ic} (bulk). This enhancement of the crack resistance of the joints compared to the bulk is discussed below. A similar observation is recorded when the effect of temperature is examined, at temperatures below about 30°C. At higher temperatures the bulk adhesive begins to exhibit a ductile tearing mode of crack growth[3,4] which is not observed in the adhesive layer, constrained between high-modulus substrates, and hence G_{Ic} (bulk) is greater than G_{Icm} (joint).

Effect of specimen width

It is sometimes found[7,11] that the measured fracture energy of a material varies with the width of test specimen employed, over a certain range of widths. This arises because the state of stress near the crack tip varies from plane-stress in a very thin specimen to plane-strain near the centre of a wide plate. Now, as mentioned above, the tensile stress at which a material yields is greater in a triaxial stress-field (plane-strain) than a biaxial one (plane-stress) and thus in the former a more limited degree of plasticity develops at the crack tip and this results in G_{Ic} (plane-strain) $<$ G_{Ic} (plane-stress).

Examination of Figure 10 reveals that G_{Ic} (bulk) does decrease slightly with increasing specimen width, H. However, G_{Icm} (joint) *increases* significantly with increasing width. Further, G_{Icm} (joint) is usually greater than G_{Ic} (bulk) and this enhancement of the adhesive fracture energy becomes more marked as the degree of constraint on the adhesive layer is increased, *i.e.* as H increases, and this observation is discussed below.

Comparison between bulk and joint fracture

The far more complex fracture behaviour of the adhesive joints, compared to the bulk rubber-modified epoxy material, is obviously caused by the constraints imposed upon the adhesive layer when it is sandwiched between the high-modulus substrates. Indeed, it has already been demonstrated how the presence of the substrates may restrict the growth of the plastic-zone at the crack tip, resulting in comparatively low values of G_{Ic} (joint) when employing thin bonds, see Figures 6, 7 and 12. The value of G_{Ic} (joint) increases as the bond thickness is increased until the thickness is equal to the plastic-zone diameter, $2r_{Iy}$, normal to the crack plane, *i.e.* $t_m \equiv 2r_{Iy}$. The value of $2r_{Iy}$, and hence t_m, may be predicted from a fracture mechanics analysis, using bulk adhesive properties in Equation (4), and the value of t_m represents an optimum thickness at which the adhesive fracture energy is at its maximum value.

Now, the two major questions yet to be addressed are (i) why is G_{cm} (joint) usually greater than G_{Ic} (bulk) and (ii) why does G_{Ic} (joint) decrease when $t > t_m$? If these could be answered quantitatively then the toughness of the adhesive joint as a function of geometry and service conditions could be fully predicted from the mechanical properties of the bulk adhesive, especially if it is remembered that the bulk fracture behaviour can, in turn, be predicted from employing time-temperature superposition principles.

The work of Wang *et al.*,[17] provides at least a semi-quantitative answer to these questions. They used a hybrid stress model finite-element analysis incorporating an advanced crack tip element to examine cracked adhesive bonded double-cantilever-beam specimens. They computed the stresses, using an elastic analysis, in the near and far fields as a function of adhesive/substrate modulus ratio and adhesive bond thickness and compared these results to a monolithic system. They found that in an adhesive layer a given level of the σ_{11} local tensile stress ahead of a crack could act over a very much

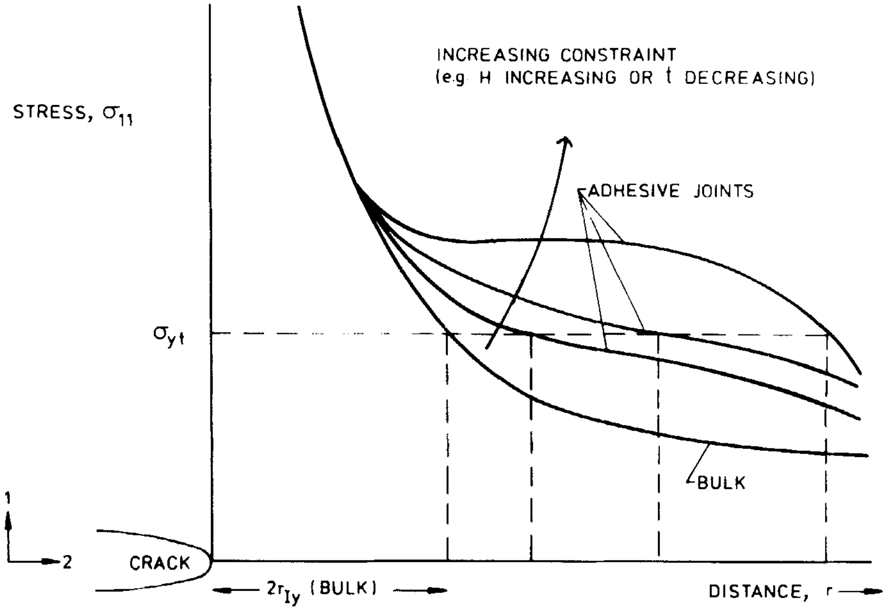
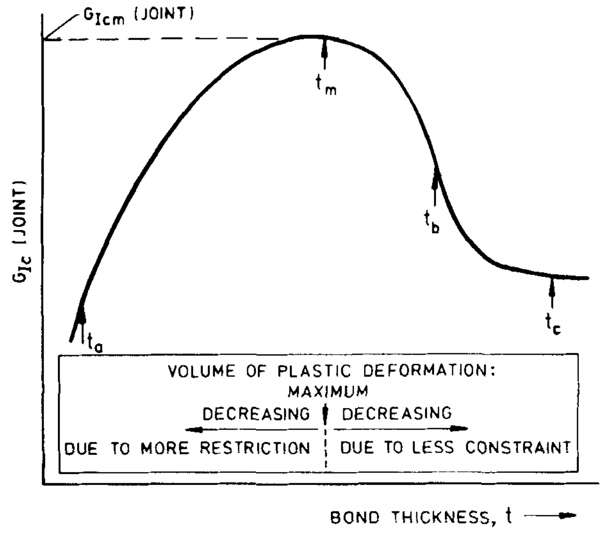


FIGURE 13 Effect of constraint on the σ_{11} stress-level ahead of a crack tip.

longer distance than that expected from comparison to a similar bulk specimen of the adhesive. This conclusion was valid for a common G_I level in the joint and bulk and when the distances involved were greater than about 2.5 μm. The distance over which the given σ_{11} stress-level acted was greater the higher the degree of constraint imposed upon the adhesive layer, *i.e.* the higher the E_s/E_a ratio and thinner the adhesive layer. This effect is shown qualitatively in Figure 13. Now, since the stresses at the crack tip are singular then clearly the yield criterion (taken to be $\sigma_{11} = \sigma_{yt}$ in this case) will be



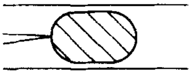
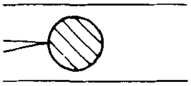
t - VALUE	DEGREE OF CONSTRAINT DUE TO BOND THICKNESS	PLASTIC-ZONE SHAPE	G_{Ic} (JOINT)
t_a ($< 2r_{Iy}$)	HIGH		COMPARATIVELY LOW.
t_m ($= 2r_{Iy}$)	MODERATE		AT MAXIMUM.
t_b ($> 2r_{Iy}$)	LOW		BELOW G_{Icm} (JOINT) VALUE.
t_c ($\gg 2r_{Iy}$)	ALMOST NIL		APPROX. EQUAL TO G_{Ic} (BULK).

FIGURE 14 Schematic model for explaining the G_{Ic} (joint) *v.* bond thickness, *t*, relationship.

exceeded in some zone at the crack tip region. To a first approximation the length of this zone is the plastic-zone length, $2r_{Iy}$.

At the crack tip in the adhesive layer the thickness of the plastic-zone will be, therefore, as predicted from the bulk behaviour but its length ahead of the crack may be longer for distances, r, greater than 2.5 μm. Thus, since the plastic zones in the rubber-modified epoxy are typically of the order of hundreds of microns, the volume of plastic deformation ahead of the crack tip may be considerably greater when the material is constrained (but the development of the plastic deformation not restricted) as an adhesive layer compared to the bulk, unconstrained state. This would obviously result in G_{Ic} (joint) > G_{Ic} (bulk).

This argument is further developed in Figure 14. Essentially, the maximum volume of plastic deformation ahead of the crack tip in the adhesive layer occurs when $t_m = 2r_{Iy}$. That is when the maximum degree of constraint exists, at a given value of joint width, H, commensurate with the condition that there is no restriction on the development of the plastic-zone due to the presence of the high-modulus substrates. Under this situation G_{Ic} (joint) is at its maximum value, G_{Icm} (joint). Now G_{Ic} (joint) < G_{Icm} (joint) at $t < t_m$ due to restriction on the further development of the plastic-zone and at $t > t_m$ due to the decrease in degree of contraint reducing the length of the plastic-zone, and hence reducing its volume. The degree of constraint is obviously also a function of joint width. For example, as H increases the degree of constraint increases and thus G_{Icm} (joint) would be predicted to increase in value compared to G_{Ic} (bulk) and the decline at $t < t_m$ would be expected to be more rapid. Both of these effects are observed, as may be seen from Figure 7. Finally, the shapes of the plastic-zones ahead of the crack are in agreement with observations on the extent of stress-whitening and quantitative measurements of this zone size are currently being pursued.

CONCLUSIONS

It has been found that the adhesive fracture energy, G_{Ic} (joint) of joints consisting of steel substrates bonded with a rubber-modified epoxy adhesive is a strong function of adhesive bond thickness, t. A maximum value, G_{Icm} (joint), is recorded at a specific bond thickness, t_m. The values of G_{Icm} (joint) and t_m are highly dependent upon the width of the joint, test rate and temperature. Further, the value of G_{Icm} (joint) has been compared to the fracture energy, G_{Ic} (bulk) of the epoxy material and under many conditions the former parameter has been found to be greater in value.

From a consideration of the micromechanics of the failure mechanism these observations have been qualitatively explained and, in some instances, quantitatively predicted from a knowledge of the bulk mechanical properties of the adhesive.

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