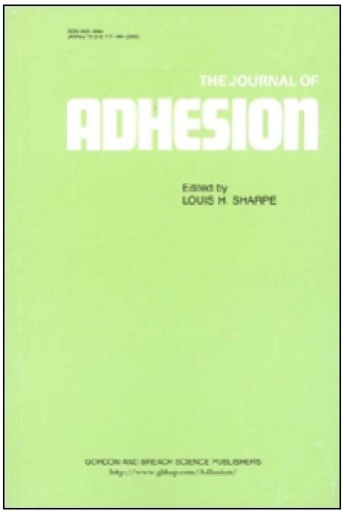

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Cleavage and Static Toughness*

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The cleavage of adhesive joints allows the experimental study of the process of fracture in the low speed range. The value of the fracture energy deduced from the fracture length is the static toughness of the adhesive. This value, which determines the endurance limit of the joint, is much larger than can be explained by the current theories. It depends on the surface treatment of the substrate and results from the damage of the adhesive bonds. To take into account these results, the equation describing the fracture of adhesive joints as it was proposed by A. N. Gent and J. Schultz has to be extended. When that is done, it applies to viscoelastic adhesives, whether pressure sensitive or hot melts, and probably also to cross-linked adhesives.

If G is the fracture energy of the joint, the equation $\mathbf{G} = \mathbf{G}_0 + \alpha.\ \mathbf{K}_2 \cdot \mathbf{v}^n$ accounts for most experimental results and even for the fatigue of adhesive joints.

KEY WORDS: adhesive; fracture; fatigue; cleavage; joints

1 INTRODUCTION

In 1972 Gent and Schultz[1] observed that the energy, W[†], necessary to separate interfacially, by peeling, a film of butadiene-styrene rubber from a polyester film varied with the speed of separation, as:

$$W = W_a(1 + 8.64 \times 10^5 v^{0.42}) \tag{1}$$

in the range of separation speed from 10^{-6} to 10^{-1} m.s^{-1}. W_a is essentially the thermodynamic value of the energy of adhesion. From the variation of W upon peeling in various wetting liquids they concluded that: "W must be factorizable in two terms one representing the equilibrium energy (W_a) and one denoted $f(v)$ representing the magnification factor because energy is expended in irreversible process in the adhesive" and wrote:

$$W = W_a \cdot f(v) \tag{2}$$

setting $(1 + 8.64 \times 10^5 v^{0.42}) = f(v)$. Later Andrews[2] proposed a generalized theory of fracture mechanics which expresses the work of debonding as a function of the energy

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[†] J. Schultz writes the fracture energy as w, but it is more generally denoted G as we will do in this paper, although we keep his notation when we quote him. Also, in equation (1), v was given in cm.s^{-1}, hence a coefficient of 1.25×10^3 instead of 8.64×10^5 given here.

to break interatomics bonds (W) and a loss function Φ (v, T, ε) dependent on crack velocity, temperature and strain but independent of the locus of fracture. They extended equation (2) to both cohesive and adhesive fracture, W being equal to adhesion energy, θ, or cohesive energy, J, depending upon whether the fracture is interfacial or cohesive, respectively, the dissipation function being the same in both cases:

$$J = J_0 \cdot \Phi(v, T, \varepsilon) \quad \text{or} \quad \theta = \theta_0 \cdot \Phi(v, T, \varepsilon) \tag{3}$$

In the case of rubber adhering to various polymers, the value of W was also about the same as θ_0.[3]

In 1978 Maugis and Barquins,[4] studying the debonding by a punch of a polyurethane in contact with a glass plate, showed that their results could be expressed as:

$$G = W_a(1 + \phi(a_T v)^n) \tag{4}$$

G is the energy release rate, ϕ an energy dissipation factor, a_T the WLF correlation coefficient ($-\log a_T = 17.4(T - T_g)/51.6 + T - T_g$), v the separation speed and W_a the energy of adhesion. Equation (2) gives an explicit relationship between the fracture energy, the temperature and the crack velocity in the case of viscoelastic adhesives. It has been verified in the range of speed between 10^{-7} to 10^{-1} m.s^{-1}, at room temperature.

The $(a_T v)^n$ dependence has been verified in many cases for various substrates and adhesives, measured in different modes, notably by Schultz and co-workers[5-7] for rubbers, polyethylene or EVA, as well as by Aubrey and Sheriff[8] for various acrylic pressure sensitive adhesives and by Barquins *et al.* for rubber (Ref. 9 and other references therein) and adhesive tape.[10]

Actually, the relationship described by the above equations is followed only in a given range of speed (*ca* 10^{-10} ms^{-1} to 10^{-1} ms^{-1}) which is the common range of tensile testing machines (using the WLF temperature shift).

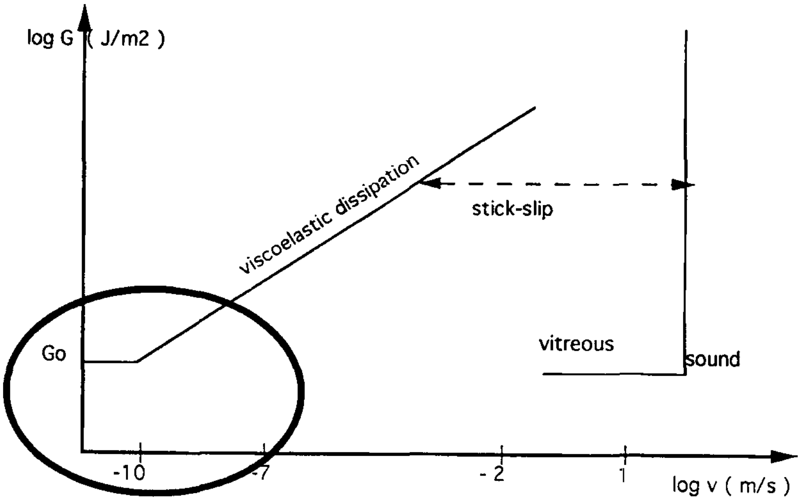


FIGURE 1 General form of the variation of the fracture energy with the speed of measurement (separation) for an elastomeric adhesive. The circle indicates the range of interest in this work.

The general form of the variation of G with speed[8, 10] is given in Figure 1.

One observes five domains:

A very low speed G is constant, then above some $10^{-10}\,\mathrm{ms}^{-1}$ G increases following equation (2); around $10^{-3}\,\mathrm{ms}^{-1}$ stick-slip occurs; at still higher speed the fracture is brittle with little energy dissipation and, finally, when the speed of separation approaches that of the sound in the adhesive, G increases sharply. In this paper, we consider the phenomena that occur in the low speed limit, such as we may observe through cleavage experiments.

2 THE LOW SPEED LIMIT

The events occurring at low speed are important both because they are easier to describe by the laws of physics and because they are those that occur during the slow delamination of adhesive joints and limit their life-time.

In the low speed limit, De Gennes[11] proposed that an adhesive had a viscosity of separation, η_{sep}, and showed, in the case of Newtonian viscosity, that $G \cong G_0 + \eta_{sep} \cdot v$ (5)

The viscous behaviour of polymers is non-Newtonian, so that their viscosity varies with the shear rate, $\dot{\gamma}$, as $\eta = K_2 \dot{\gamma}^{1-n}$ where K_2 (Pa.s^n) is the "consistency" of the polymer.

Prentice[12] considered that when pulling a polymeric chain out of its reptation tube the shear rate is equal to the speed, v, of pulling, divided by the distance, h, between the chain and the tube. Then he found that the energy of fracture by pulling of the chains is $G = \alpha K_2 v^n$, where $\alpha = \pi \cdot r \cdot a \cdot L^2/h^n$, r being the radius of the chain, L its length and a the density of chains crossing the plane of fracture.

So, for most polymers, equation (5) could be written:

$$G = G_0 + \alpha K_2 v^n \tag{6}$$

From equation (2) G should tend toward W_a when v tends toward 0.

Experimentally this has rarely been verified, whatever the locus of fracture. It is only in the case of very low adherence, where only Van der Waals forces are in play as, for instance, a silicone rubber in contact with PMMA[13] or a polyurethane pressed against a piece of flat glass,[4] that $G(v \to 0) \cong W_a$. In general, much higher values are found.[6, 13]

Contrary to other methods, the cleavage of adhesive joints allows one to examine fracture processes at very low speed and to study what occurs at the low speed limit.

2.1 Results Obtained from the Cleavage of Adhesive Bonded Joints

We have described in detail how the cleavage of adhesively bonded joints can give quantitative values of the fracture energy.[14]

A wedge is introduced between two rectangular plates (Fig. 2) which produces a crack of length, l, from which G is deduced. In the cases where the adherend is brittle, or when we want to observe the fracture tip through (transparent) glass or sapphire, a chip-test[15] configuration (Fig. 3) is used instead.

Upon the introduction of the wedge between the two substrates, a crack forms in the adhesive layer. It starts so rapidly that we cannot measure its speed with a video

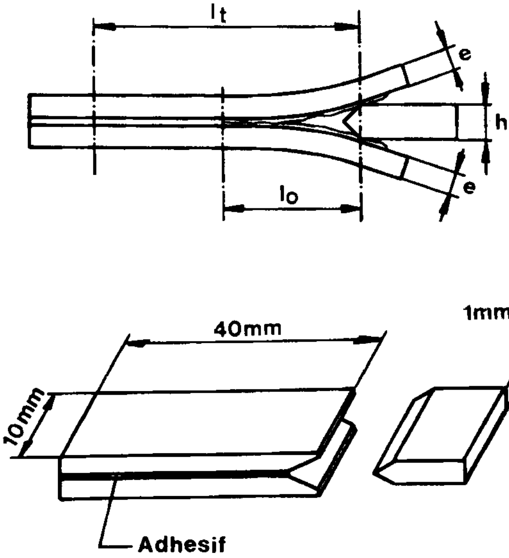


FIGURE 2 Schematic drawing of a cleavage test with the indication of the dimensions used here. The fracture length, l_0, gives G_0.

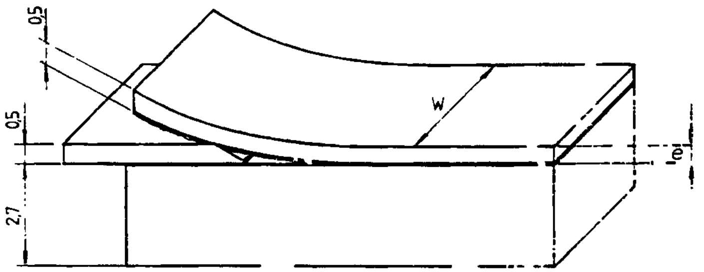


FIGURE 3 Schematic drawing of a chip test, with the indication of the dimensions used here.

camera. After 8 seconds the speed reaches 10^{-4} ms^{-1} and after 2–3 days it stops; after that it does not change at least during 10 years (or it progresses more slowly than 10^{-12} ms^{-1}, which is unmeasurable in 10 years). The fracture energy value deduced from cleavage experiments thus can be considered to be the low speed limit value of $G(v \to 0)$ or G_0.

That this is indeed the case can be shown by comparison of the result of cleavage of a stainless steel /EVA/ stainless steel joint (in the dimensions shown in Fig. 2) and that of the peeling, at various speeds, of an adhesive joint made of a plate and a film of stainless steel bonded with an EVA hot melt adhesive.

The measured values are shown in Figure 4. The variation of G with the separation speed is low in that case, showing a variation $G(v^n)$ with $n = 0.26$. At low speed, $G = 1200$ J.m^{-2} which is almost the value found in the cleavage experiment ($G_0 = 1000$ J.m^{-2}).

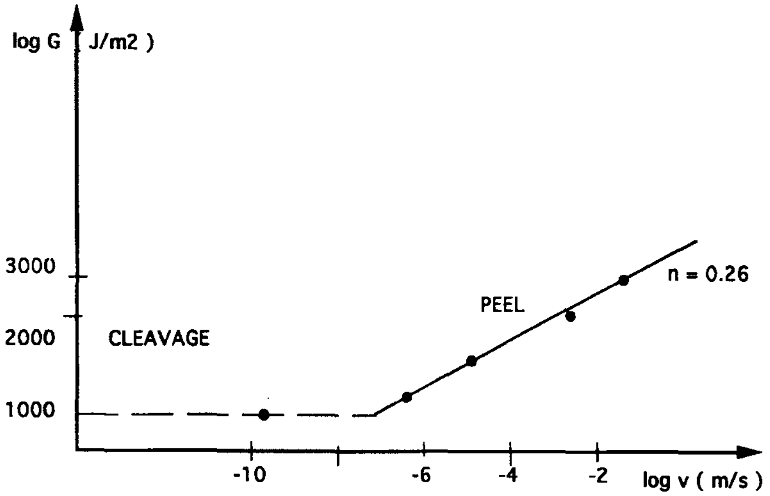


FIGURE 4 The low speed limit of peeling of a stainless steel/EVA/stainless steel joint is nearly equal to the value found in a cleavage test.

2.2 The Static Toughness of Adhesives

The values of G_0 deduced from the fracture length of cleaved joints are generally high for the commonly-used adhesives, in ambient conditions, as shown in the Table I, which summarises the measurements we made for stainless steel bonded with various commercial adhesives.

This means that adhesives are able to dissipate energy even at very low or null speed. This had already been noticed by Kendall[16] and called by him the "static toughness" of adhesives. That static energy dissipation is due to the damage of the adhesive.

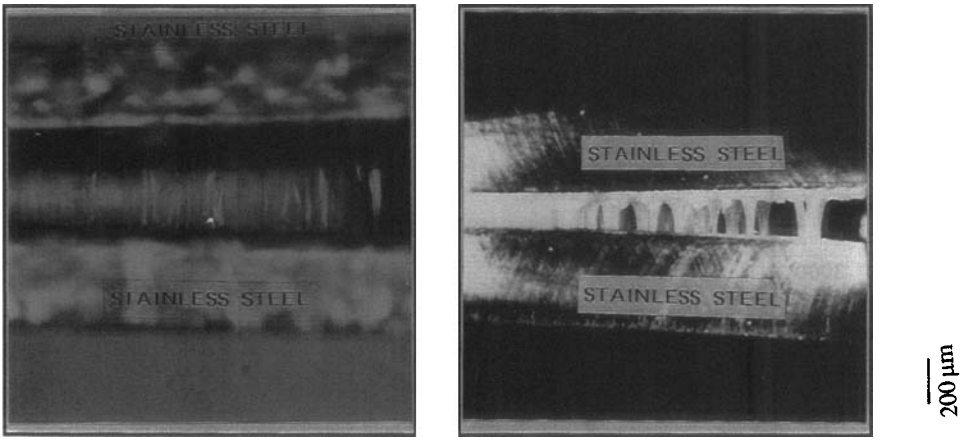
Under the imposed stress the polymer, depending upon its degree of crystallinity or of crosslinking, forms fibrils, crazes or cavities ahead of the crack tip. At low speed the fracture propagates through the damaged zone, as shown in Figure 5 for some characteristic cases. When it stops, although the damaged zone may extend over distances as long as 6 mm, the joint does not debond. The damaged polymer is stable in indoor conditions.[17]

TABLE I

Static toughness of various adhesives deduced from the fracture length of cleaved joints. (*These ranges of values have been obtained* in the course of evaluation of many commercial adhesives)

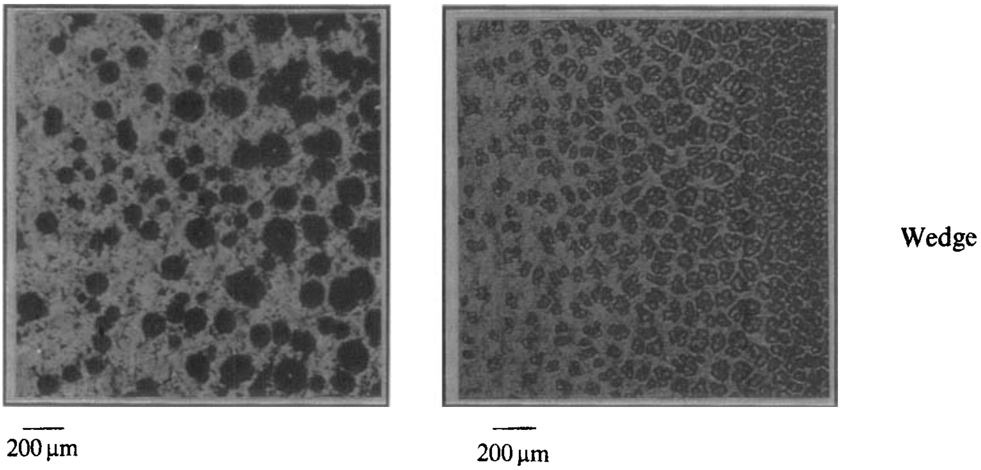
Adhesive	$G_0(JM^{-2})$	Adhesive	$G_0(JM^{-2})$
Epoxy-Nylon E610*	6000	Press.-sens.tape	30–600
Epoxy-CTBN	1200	Hot-Melt	100–1000
Epoxy-2 Comp.	80–200	Acrylics (unmodified)	50–100

* E 610 is a formulation made by Asulab.



Fibrillation ahead of the crack tip in a double sided adhesive tape (speed $< 10^{-12} ms^{-1}$) - Initial thickness 200 µm

Fibrillation ahead of the crack tip in a P.U. hotmelt (speed $< 10^{-12} ms^{-1}$) - Initial thickness 125 µm



Cavitation ahead of the crack tip in an EVA Hot-melt (Photo taken through the glass - speed $< 10^{-12} ms^{-1}$) Magnification x 50 - Initial thickness 40 µm

Cavitation ahead of the crack tip in an epoxy-nylon (Photo taken through the glass - speed $< 10^{-12} ms^{-1}$) Magnification x 50 - Initial thickness 150 µm

FIGURE 5 Some figures of the damage to adhesives ahead of the crack tip.

The static toughness (fracture energy at speed zero) is an important parameter of the adhesive joint as it determines the limit of endurance of the joint: as long as the energy, G, corresponding to the imposed stress, is smaller than G_0 the joint will not debond.

It is remarkable that some adhesive tapes or hot melts show a *static toughness* as high as thermoset adhesives although they are not cross-linked. In the case of hot-melts

TABLE II
Joint surface treatment and fracture energy. Fracture is in each case cohesive

Surf. Treatment	G_0(J M^{-2})
None	900
Solvent wipe	1000
Alkaline	2000
"Asuclean"*	6000

*Asuclean is a cleaning formulation made by Asulab.

this is due to the semi-crystalline structure of the polymer; as soon as the crystallites melt the fracture energy decreases considerably.[18]

The expression:

$$G = G_0 + \alpha \cdot K_2 \cdot (v)^n \tag{6}$$

takes into account that the value of G at low speed is the static toughness.

2.3 The Value of G_0 Depends upon the Surface Treatment

Although one would expect G_0 to be characteristic of the adhesive, mainly when the fracture is cohesive, it is found to depend upon the surface treatment of the substrate. This is shown in the following two experiments.

2.3.1 Cleaning of stainless steel

Wedge tests were made on stainless steel joints bonded with the same epoxynylon adhesive (E 610 made by Asulab), following the same curing procedure (45 min at 180°C). Only the cleaning step was changed and the results of Table II were obtained: they show that surface treatments increase the static toughness, G_a, from 900 to 6000 J M^{-2}, although in all cases the fracture is cohesive.

2.3.2 Anti-adherent treatment of glass

In another experiment, joints were made with a stainless steel plate and a piece of glass 4 mm thick, bonded with a two-component epoxy, cured 4 h at 80°C. G_0 was measured in the "chip" configuration of Figure 3.

When the glass surface was treated with an anti-adherent layer of DMOAP*, the value of G_0 was very low and the separation was interfacial (A). Then the anti-adherent layer was destroyed by a corona treatment in successive rapid passes. Each pass of the electrode destroyed some of the DMOAP molecules and restored bonding sites at the glass surface. After the first pass G_0 increases. After the second pass G_0 increases further and the fracture becomes cohesive (C) although the fracture energy is lower than that found for untreated glass. After the third, it is equal to that of clean glass. After the fourth pass it becomes higher than that of the cleaned glass. (Table III).

---

* DMOAP is Dimethyl Octadecyl (3-trimethoxysilyl) Propyl Ammonium Chloride.

TABLE III

Destruction of an anti-adherent layer deposited on glass and fracture resistance of the bond. Although the fracture is cohesive beyond one pass, its energy increases with the number of bonds

Glass Treatment	$G_0 (\mathrm{J\,M^{-2}})$
Cleaning	30 (C)
Cleaning + DMOAP*	≤ 3 (A)
+ Corona (1 pass)	8.5 (A)
+ Corona (2 passes)	16.5 (C)
+ Corona (3 passes)	30 (C)
+ Corona (4 passes)	51 (C)

* DMOAP DiMethyl Octadecyl Ammonium (3-Trimethoxy) Propyl Silane (A) = adhesive failure; (c) = cohesive failure

Clearly, the destruction of the anti-adherent layer increases the number of substrate-adhesive bonds and changes the fracture from adhesive to cohesive. However, the cohesive fracture energy increases as the number of substrate-adhesive bonds increases. Although the number of bonds is not known, the evolution of G_0 with the number of substrate-adhesive bonds may qualitatively be represented as in Figure 6.

The cohesive fracture energy depends upon the number of existing bonds between the substrate and the adhesive, *i.e.* $G_0 = f(W_a)$.

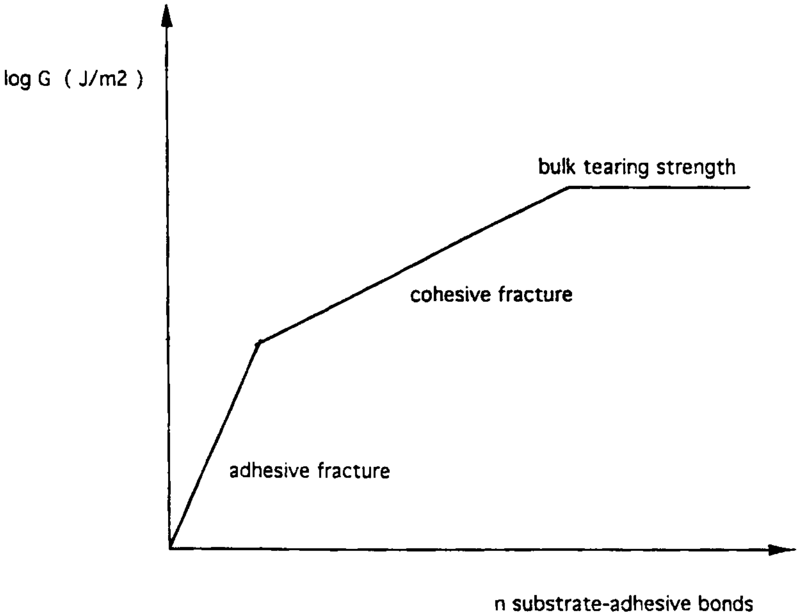


FIGURE 6 As the number of substrate-adhesive bonds increases due to an appropriate surface treatment the fracture energy, G_0, increases. The locus of fracture changes from adhesive to cohesive. However, even when the fracture is cohesive the fracture energy increases with the number of bonds.

3 PHYSICAL MEANING OF G_0

The term G_0 in equation (6) is justified by the experimental results of cleavage experiments. Earlier, Carré and Schultz,[19] studying the fracture of rubber-aluminium joints, noticed that the low speed value of the peel energy was much higher than that calculated for W_a.

They proposed to write, instead of equation (2):

$$\mathbf{W = W_a \cdot g(M_c) \cdot f(v)} \qquad (7)$$

They justified the static dissipation factor, $g(M_c)$, by analogy with the argument of Lake and Thomas[20] to explain the tearing energy, T_0, of rubbers: in order to break one polymer chain one has to extend all the bonds forming the chain to near their rupture limit.

For weakly cross-linked rubber, if U_χ is the bond energy of one link in a polymer chain, ν the density of links by surface unit, and N the number of links in a chain, $T_0 = N.\nu.U_\chi$. They said that for a separation test where the fracture is cohesive, the fracture energy W could be expressed in the same way, $g(M_c)$ being the number of C—C bonds between two crosslinks. They supposed further that the dissipation factor is the same whether the fracture was adhesive or cohesive and that W_a was either the adhesion energy or the cohesive energy in each respective case, as Andrews also did.[2] However, this explanation cannot account for values higher than $100\,\mathrm{J\,m^{-2}}$ and we showed in section 2.2 that values as high as $6000\,\mathrm{J\,m^{-2}}$ could be measured.

We suggest that the higher magnification observed has two reasons:

1) Upon tearing, all polymer chains are firmly maintained in the grip of the pulling machine and extended, whereas in the peeling or the cleavage of adhesives joints, the polymer network is extended only through these chains that are linked to the substrate surface.
2) In commercial adhesives many chains are interacting whether in crystalline domains or through cross-links. In those cases, one substrate-adhesive bond pulls a large number of chains (Fig. 7). This provides a further amplification factor: G_0 is equal to the number, $N = N \cdot n_c$, of extended links in all of the n_c chains which are stressed, times the density, ν, of chains bound to the substrate, times U_χ:

$$G_0 = N \cdot \nu \cdot U_\chi$$

Compared with the value of T_0, G_0 is n_c times higher.

This also explains why G_0 depends upon W_a. The density, ν, of chains bound to the substrate is proportional to W_a. If the substrate-adhesive bond has the energy U_c, the adhesion energy is $W_a = \nu \cdot U_c$; thus, $G_0 = N \cdot W_a \cdot U_\chi / U_c$. Setting $g_0 = N \cdot U_\chi / U_c$, one finds that G_0 depends on W_a and $G_0 = g_0 \cdot W_a$, as shown by experiments. In the case of low adherence due to Van der Waals interactions, where the fracture is interfacial, $g_0 = 1$ as there is no chain extension.

The static toughness, G_0, is the product of the density of chains bound to the substrate times the number of links stressed upon pulling one attached chain and that number will depend on the way that the polymer breaks. This is the reason why the amplification factor is much higher for semi-crystalline polymers or for foamed pressure sensitive adhesives, than may be expected from the simple extension of one polymeric chain.

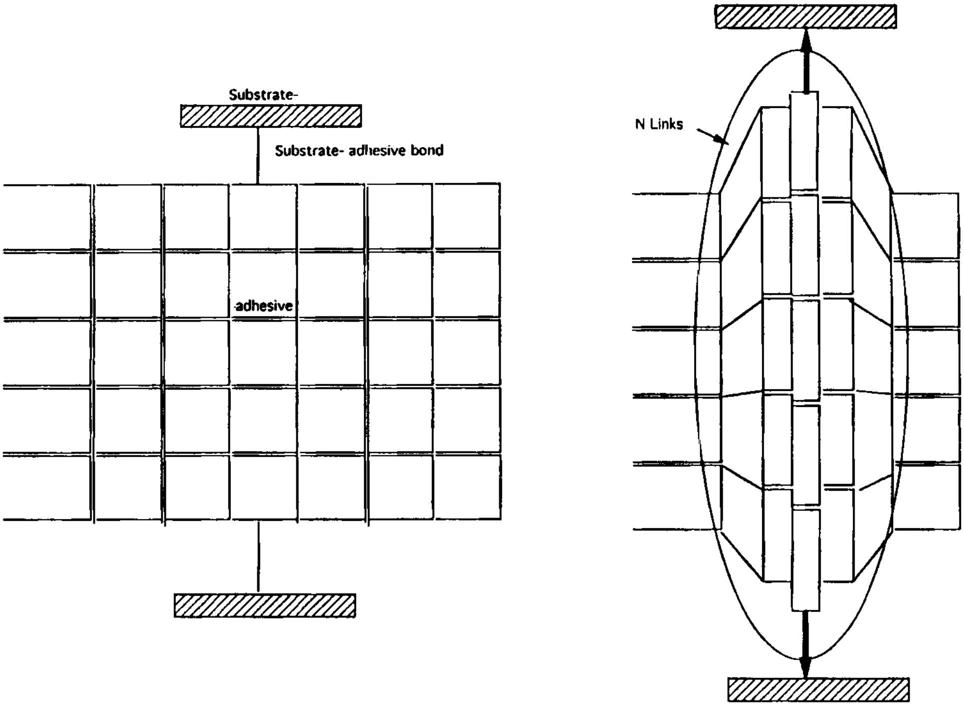


FIGURE 7 Schematic representation of one chain attached to the substrate from a cross-linked polymer network. Upon peeling or cleavage of an adhesive joint, one substrate adhesive bond pulls a large number of chains.

Experimentally, it is difficult to establish whether an increase of G_0 comes from an increase in the number of substrate-adhesive bonds or an increase in the number of extended links. In the case of pressure sensitive adhesives, or weakly cross-linked rubbers, the number of extended links is equal to the average number of links between two cross links. In the case of semi-crystalline hot-melts, or highly cross-linked structural adhesives, the number of extended links depends upon the mode of fracture. Depending upon whether the adhesive chains cavitate, fibrillate, craze or whatever different numbers of links are involved.

One may imagine that the maximum value of W_a is attained when the best surface treatments have been achieved and G_0 does not increase on further treatment. Then the fracture will be cohesive and its energy close to the tearing energy of the bulk adhesive.

The maximum number of bonds is one every $100\,\mathrm{A}^{\circ 2}$; taking $U_c = 320\,\mathrm{kJ.mole}^{-1}$ then $(W_a)_{\max} = 1\,\mathrm{J.m}^{-2}$. A value of $G_0 = 6000\,\mathrm{J.m}^{-2}$ means that one substrate-adhesive bond pulls 6000 links in the adhesive.

As the energy of adhesion depends upon the number and the nature of substrate-adhesive bonds which depend, in turn, on the surface treatment, the effective energy of adhesion is not always the thermodynamic value.

Inserting $G_0 = g_0 . W_a$ in equation (6), one gets

$$\mathbf{G} = \mathbf{g}_0 \cdot \mathbf{W}_a + \alpha \cdot \mathbf{K}_2 \cdot (\mathbf{v})^n \tag{8}$$

4 THE CASE OF CROSS-LINKED ADHESIVES

Slightly cross-linked polymers, such as those used in pressure sensitive or hot-melt adhesives, are highly viscous fluids and it is easy to understand that they dissipate energy through a viscous flow described by the consistency of the adhesive. The case of cross-linked polymers, however, is not so clear, although the following experiment indicates that their fracture behaviour is also correctly described by equation (8).

4.1 The Influence of Humidity on the Progression of a Crack in an Epoxy Adhesive

A joint made of two plates of stainless steel bonded with a one-component, CTBN-modified epoxy was cleaved as described above. Once the wedge is inserted, the increase in crack length may be measured during the first day. This allows the definition of an average crack speed giving the upper curve of Figure 8, describing crack propagation in indoor conditions. When the already-cracked assembly is placed in an humid atmosphere (40°C, 90% relative humidity) the static toughness decreases and the fracture propagation starts again. Its evolution is fairly slow, permitting one to follow it during two years. A plot of the fracture energy deduced from the fracture length *versus* the average speed gives the lower curve of Figure 8 which has the form expected from equation (7) for an adhesive having a static toughness of 8 J.m^{-2}.

5 IMPLICATION CONCERNING THE FATIGUE OF ADHESIVE BONDED JOINTS

It has been shown[21] that when an adhesive bonded joint is submitted to alternating stresses the fracture progresses a length, da, after dn cycles, as given by the Paris equation:

$$\mathbf{da/dn = C(G - G_0)^m} \tag{9}$$

Actually, that relationship may be deduced from equation (8). For a frequency, $f = \mathrm{dn/dt}$, one obtains from

$$\mathbf{G = G_0 + \alpha \cdot K_2 \cdot v^n}$$

and

$$v = \frac{da}{dt} = \frac{da}{dn} \times \frac{dn}{dt} = f \cdot \frac{da}{dn}$$

the equation

$$\frac{da}{dn} = \frac{1}{\alpha \cdot \mathbf{K}_2 \cdot f} (G - G_0)^m \tag{10}$$

is obtained which is equation (9), with $m = 1/n$ and $C = 1/\alpha \cdot \mathbf{K}_2 f$*

---

* Since the first version of this paper, I have become aware of the work of Kinloch and Osiyemi on the fatigue of adhesively-bonded joints (*J. Adhesion* **43**, 79–90 (1993)). Their data may, in the linear region, be interpreted using equation (10). As they worked at a frequency of 5 Hz, the value $\alpha \mathbf{K}_2 = 3.86 \times 10^{13}$ is obtained from their results. That is in the range expected for a glassy polymer.

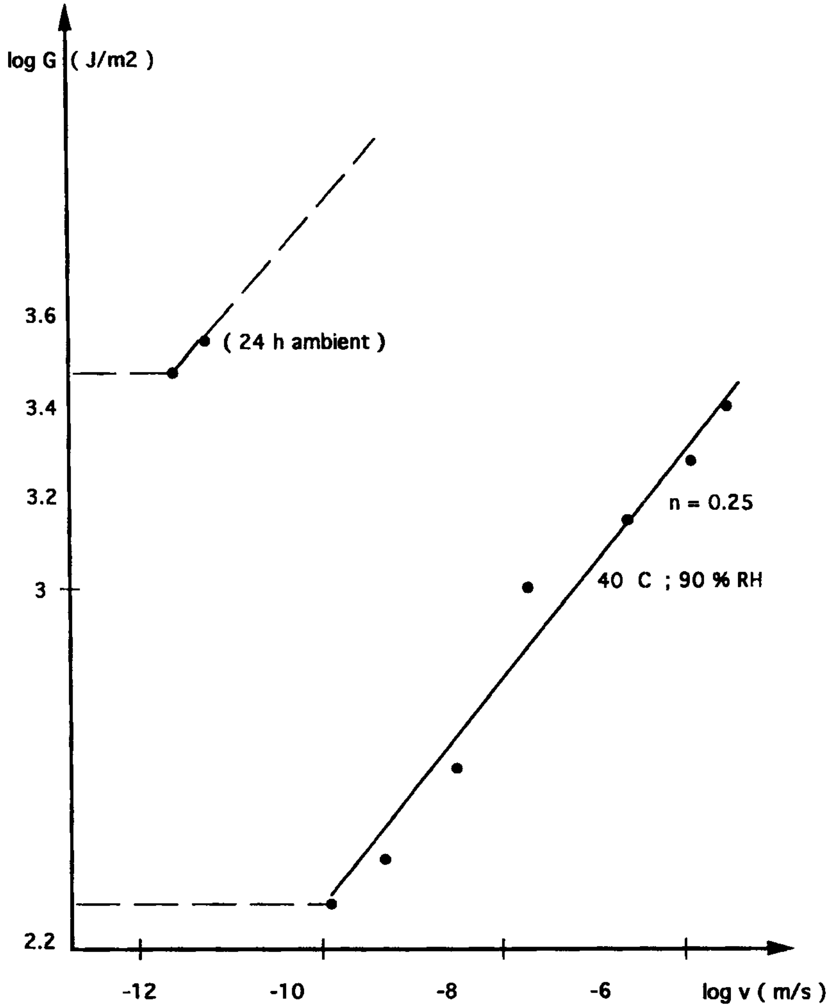


FIGURE 8 The evolution of the cleavage crack of an epoxy adhesive in humid atmosphere follows the variation expected for a slightly cross-linked adhesive.

The progression of a crack in a viscoelastic adhesive depends on frequency, temperature, energy of dissipation within the adhesive and the static toughness. At an applied stress for which G is below G_0, the fracture length does not increase.

The exponent of equation (8) is the inverse of that of the Paris law, equation (9).

CONCLUSION

The form of equation (8) is a generalisation of the previous works mentioned above. It accounts for most results obtained during the study of the fracture of adhesive joints either by peeling, cleavage or fatigue experiments. The low speed limit is the static toughness of the adhesive which results from some damage and which determines its endurance limit. It is proportional to the energy, W_a, of the substrate-adhesive bond. The amplification factor results from the extension of all the links of all the chains pulled out by one adhesive bond. The consistency of the adhesive is due to a non-Newtonian flow of viscoelastic adhesives, hence the factor $(a_T v)^n$ describing the speed and temperature dependence. In the case of cross-linked adhesives, its meaning is not as clear, although it accounts well for the behaviour of the bond in humid conditions. Because the coefficient, α, is proportional to the number of extended chains, dissipation is proportional to W_a, as supposed, for instance, in equation (2).

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