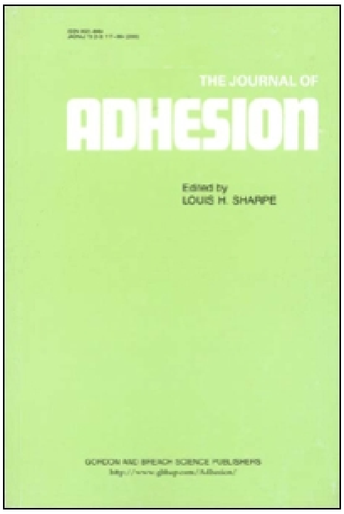

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The Fracture Behaviour of a Rubber-Modified Epoxy Under Impact Fatigue

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The fracture behaviour of a rubber-modified epoxy adhesive under impact fatigue has been investigated. An instrumented-impact test has been employed to apply multiple, "repeated-blow," impacts to pre-cracked three-point-bend specimens at sub-critical energy levels. A fracture mechanics approach has been adopted to assess the effects of the impact-fatigue damage on the epoxy material.

KEY WORDS Crack growth; fatigue; impact; linear-elastic fracture mechanics; rubber-modified epoxy adhesives.

INTRODUCTION

An important aspect of the mechanical properties of materials and structures is their resistance to fatigue failure. This includes the problem of the repeated impact loading of a material when the applied energy levels are below those necessary to cause fracture in a single impact; *i.e.* when "repeated-blow" sub-critical impact loads are applied. There has been very little work published[1] on the impact-fatigue behaviour of polymers, although there is an increasing use of polymers, including adhesives, in applications where their performance against repeated impact loading is of vital importance; for example the use of adhesives in bonding train- and car-door panels where the adhesive joints must endure repeated impacts during the normal operation of the doors.

The aim of the present paper is to extend the work which we have already published[2–4] on the impact behaviour of adhesives and adhesive joints in order to assess the impact fatigue resistance of a rubber-toughened adhesive. The specimens contained sharp pre-cracks and a linear-elastic fracture-mechanics (LEFM) approach has been adopted to ascertain the effects of repeated sub-critical impact loads on the toughness and lifetime of the epoxy material.

EXPERIMENTAL

Materials and specimen preparation

The epoxy adhesive examined in this study was a model material based on a multiphase, rubber-toughened epoxy polymer. The epoxy resin was derived from the reaction of bisphenol A and epichlorohydrin, and was largely composed of the diglycidyl ether of bisphenol A (DGEBA). The rubber used for toughening the resin was a carboxyl-terminated random copolymer of butadiene and acrylonitrile (CTBN: at a concentration of 15 parts per hunderd of resin (phr)). The curing agent was piperidine, at a concentration of 5 phr, and the curing schedule was 16 hours at 120°C. Full details of the preparation of this formulation have been given in previous publications[3,4] and sheets of the rubber-toughened epoxy, 10 mm in thickness, were cast employing a metal mould.

Bars were machined from the cast sheets and were 80 mm × 12 mm × 10 mm in size. Edge cracks of different lengths were inserted in the specimens by carefully machining sharp notches of about 12 μm tip radius into one of the 10 mm × 80 mm faces, see Figure 1. Previous work[3] has shown that, for these relatively tough polymers, this method of crack insertion results in a crack tip of sufficient sharpness to give true minimum values of the fracture parameters.

Test method

An instrumented machine was used to apply impact loads to the single-edge cracked three-point bend specimens, and a full description of the equipment has been previously reported.[3,4] Briefly, the machine consists of a pendulum striker which is instrumented and is allowed to impact against the specimen, on the reverse face to that of containing the crack. The impact velocity, and applied energy level, may be varied by changing the initial angle of the striker. In the present system, a maximum impact velocity of 3.7 m/s, resulting in an energy of 15 J, could be obtained. The three-point bend specimen was placed on the shoulders of the vice and the instrumented striker allowed to impact upon it at the lowest point of its swing. The strain-gauge transducer on the tip of the striker was

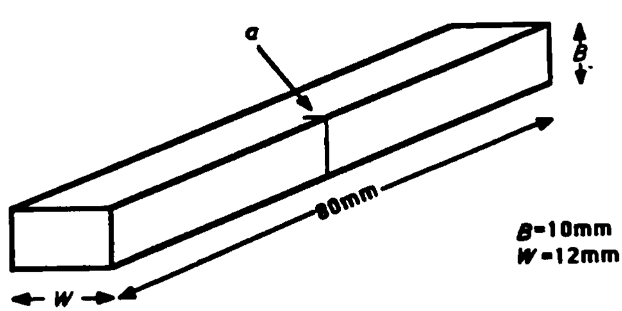

FIGURE 1 The single-edge crack three-point bend specimen.

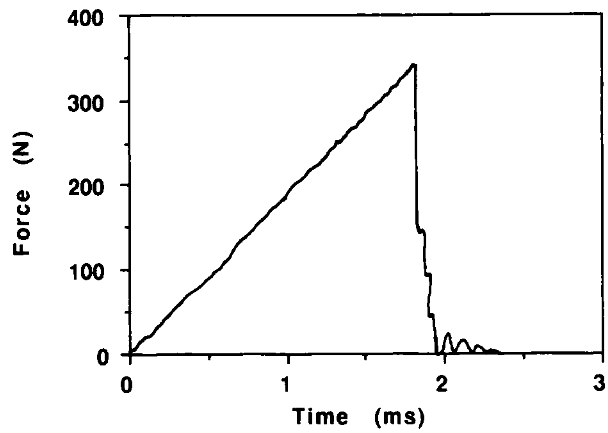

FIGURE 2 Typical force *versus* time trace for a "single-blow" impact test.

connected to a transient recorder and, via a prior static calibration, the impact force *versus* time signal could be obtained. The transducers were of the semi-conductor strain-gauge type. The memory module of the transient recorder received the signal, converted it into a digital form, and sorted the data. These data were then transferred to an Apple microcomputer, through a computer module on the transient recorder, and were analysed to give plots of force, energy, displacement and striker velocity against time, as well as the load *versus* displacement trace.

The basic toughness of the epoxy adhesive was determined by undertaking "single-blow" impact tests; *i.e.* where the specimens were fractured by a single blow of the striker. In these tests the specimens were struck by the pendulum striker with an initial angle of 20°, resulting in an impact velocity of 0.66 m/s and an impact energy of 479 mJ. This energy was higher than that needed to fracture the specimens with even the smallest crack length, which was about 0.5 mm. A typical force *versus* time trace obtained is shown in Figure 2. The energy lost by the striker during the fracture event may be readily attained by integration of the area under the force *versus* displacement trace, and this is shown as a function of time in Figure 3. At these low velocities previous work[4] has shown that the onset of crack growth is associated with the maximum force recorded by the striker and that dynamic effects may be ignored. Hence, the energy lost by the striker may be equated to the stored elastic energy in the specimen at crack initiation.

In the "repeated-blow" impact tests each specimen was struck by the pendulum with an applied energy of 44 mJ at a frequency of about 1 Hz until complete fracture of the specimen was observed. This energy level was selected since it was just slightly smaller than that needed to cause fracture in a single impact with the longest crack length of about 5 mm was employed. Obviously, depending upon the length of the initial crack, the above energy represented varying percentages of the energy needed for fracture in a single blow. For example, for a specimen containing a 5 mm crack it represented about 91% of the energy required to

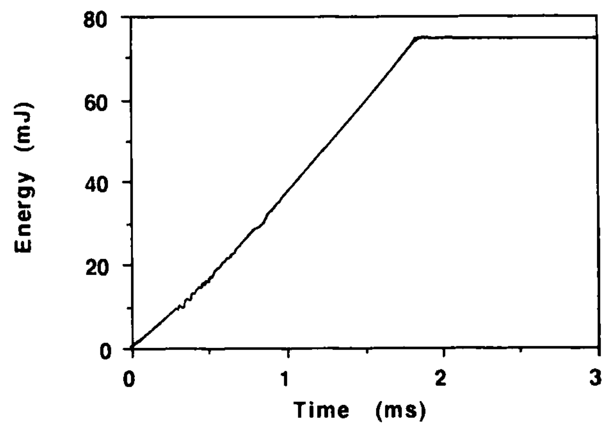

FIGURE 3 Typical energy *versus* time trace for a "single-blow" impact test.

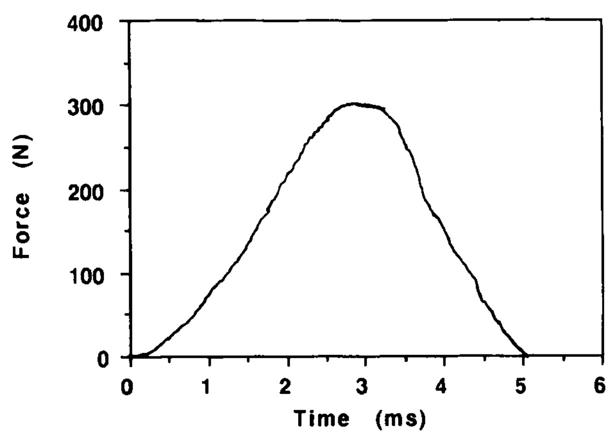

FIGURE 4 Typical force *versus* time trace for "repeated-blow" impact test.

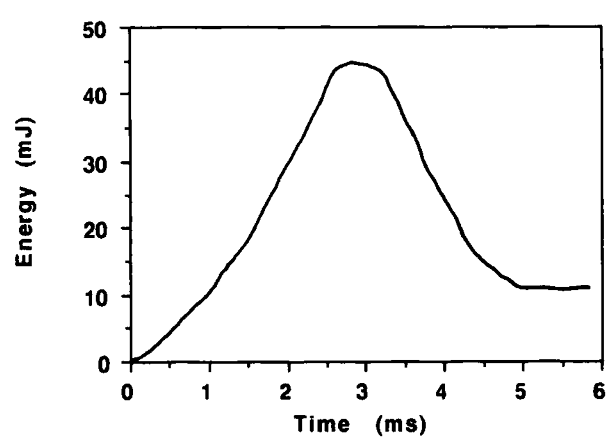

FIGURE 5 Typical energy *versus* time trace for "repeated-blow" impact test.

fracture in a single blow, but in the case of a specimen containing a 1 mm crack it only represented 27% of the energy for fracture in a single blow. For the repeated-blow impact tests typical force *versus* time and energy *versus* time curves are shown in Figures 4 and 5, respectively. After the specimen had failed, the fracture surfaces were examined using both optical and scanning electron microscopy.

THEORETICAL

The critical strain-energy release rate, or fracture energy, G_{Ic} is given from linear-elastic fracture-mechanics analysis by:[5]

$$G_{Ic} = \frac{U_c}{BW\phi} \tag{1}$$

where U_c is the stored elastic energy in the specimen at the onset of crack growth, B is the thickness of the specimen and W is the width of the specimen. The term ϕ is a dimensionless geometry factor such that:[5]

$$\phi = \frac{C}{dC/d(a/W)} \tag{2}$$

where C is the compliance of the specimen and is defined by the displacement/load. The value of ϕ may be evaluated either from measuring the compliance as a function of crack length or, more readily, from published tables of the value of ϕ as a function of a/W and L/W, where L is the length or span of the test specimen between the support points.

RESULTS AND DISCUSSIONS

"Single-blow" impact tests

As described above, the measured force *versus* time relations were used to obtain the stored elastic energy, U_c, at the onset of growth crack length and values of U_c are plotted against the respective values of $BW\phi$ in Figure 6. From Eq. (1) a linear relationship passing through the origin would be expected if the value of G_{Ic} is independent of crack length, as is indeed observed. The data in Figure 6 yield a value of G_{Ic} of 1.57 kJ/m^2, and this value is in excellent agreement with previously reported values.[4]

"Repeated-blow" impact tests

As described above, in these tests a specimen, containing a given initial crack length, was subjected to repeated blows at a frequency of about 1 Hz. The applied energy level was approximately 44 mJ and, as illustrated in Figure 5, part

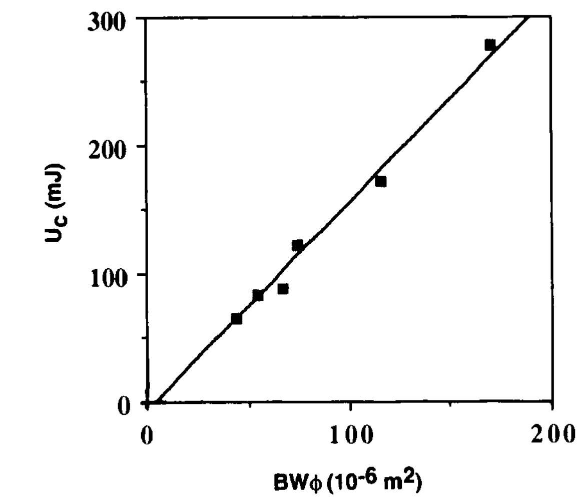

FIGURE 6 Stored elastic energy, U_c, *versus* $BW\phi$ for the "single-blow" impact tests.

TABLE I
Data for impact fatigue tests

Inital crack length (mm)	Applied energy level, as a percentage of that needed to cause failure in a single blow	Number of impacts to failure
1.11	27%	3747
2.04	36%	907
3.00	51%	393
4.00	36%	190
5.07	91%	37

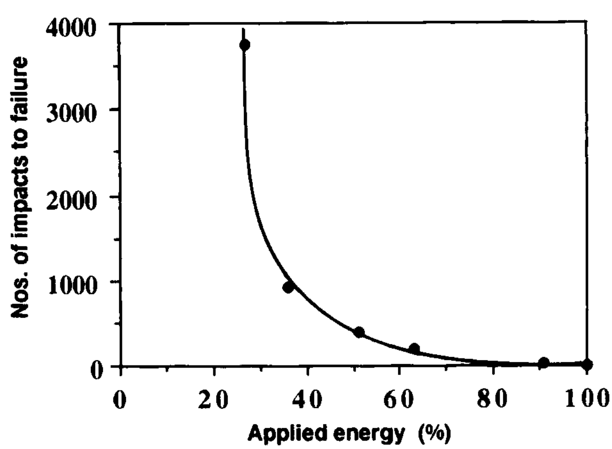

FIGURE 7 Number of repeated blows required to cause fracture as a function of the applied impact energy, where the applied energy is expressed as a percentage of the energy needed for fracture in a single blow.

of this energy was absorbed irreversibly by the specimen and could, therefore, contribute towards some form of damage accumulation mechanism. In Figure 5 about 11 mJ of energy was absorbed irreversibly by the specimen and this represents 25% of the applied energy level. It was of interest to note that during the "repeated-blow" tests this percentage remained virtually constant, being independent of crack length and of number of impacts applied. (Apart from the final blow to cause failure, when the absorbed energy level was virtually 100% of the applied level, see Table II.)

The number of such repeated blows needed to cause complete fracture of the specimen was recorded and is shown in Table I, together with values of the initial crack length and the applied energy (*i.e.* 44 mJ) expressed as a percentage of the energy needed to cause fracture in a single blow. Figure 7 shows the number of blows needed to fracture as a function of the applied energy, the latter again being expressed as a percentage of the energy to cause fracture in a single blow for the particular crack length of interest.

Now, from the data shown in Figure 7, it might appear that there is a threshold value at about a 20% applied energy level below which impact fatigue does not occur; *i.e.* a minimum applied energy below which no damage accumulation occurs. However, plotting the "repeated-blow" impact data on a logarithmetic basis, as illustrated in Figure 8, clearly reveals a linear correlation, with a correlation coefficient of 0.99. This demonstrates that there is no experimental evidence from the present study for the existence of a minimum, or threshold, applied energy level below which impact fatigue failure is not observed. (Of course, the linear correlation shown in Figure 8 does not preclude the existence of such a threshold value if even lower energy levels were employed.)

The nature of the damage accumulation mechanism was studied using both optical and scanning electron microscopy. Examination of the fracture surfaces of the specimens revealed that those subjected to repeated-blow impact tests

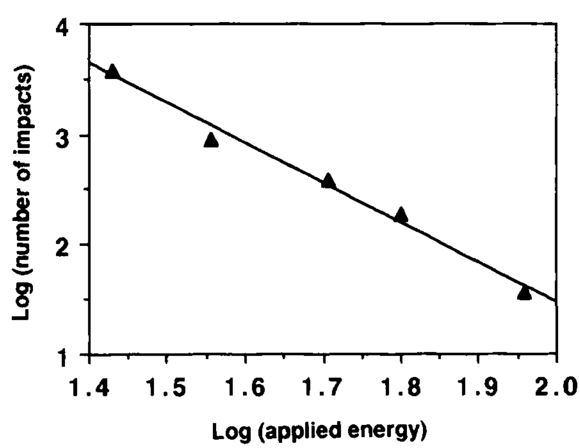

FIGURE 8 The "repeated-blow" impact data shown in Figure 7, but now plotted on a logarithmetic basis.

TABLE II
Crack length and fracture energy values for the impact fatigue tests

Initial crack length (mm)	Increment of crack growth, Δa (mm)	Crack length at fracture (mm)	Absorbed energy at fracture (mm)	G_{Ic} at fracture (kJ/m^2)
1.11	5.80	6.91	41.2	1.59
2.04	4.80	6.84	42.1	1.67
3.00	3.72	6.72	42.1	1.71
4.00	3.07	7.07	42.8	1.77
5.07	1.53	6.60	42.1	1.67

developed a relatively large stress-whitened zone ahead of the original crack tip. At final fracture the length of this zone varied with the length of the initial crack; it was longest when the initial crack was at its shortest and *vice versa*. Scanning electron microscopy indicated that this stress-whitened zone was the result of the original crack growing in a stable manner from the repeated blows the specimen received during the impact fatigue test. It was not possible to distinguish whether this sub-critical extension of the initial crack occurred in a continuous incremental fashion (*i.e.* a small amount of such growth occurring on every blow) or whether it occurred in discontinuous bursts after a given number of blows had been sustained.

The extent of this stable crack growth for the various specimens was measured and is shown in Table II. It should be noted that the total final crack length is virtually constant for all specimens. Now, the applied energy is constant at ≅44 mJ and for the final blow which causes complete fracture the absorbed elastic energy is essentially equivalent to the applied energy in all cases. Hence, the fracture energy, deduced from Eqs. (1) and (2), is also constant, within experimental error. This may be readily seen from the values of G_{Ic} stated in Table II, where the average value of G_{Ic} is $1.68 \pm 0.07\,\mathrm{kJ/m^2}$. This scatter is typical of fracture experiments and there is clearly no statistical dependence of the value of G_{Ic} upon the initial crack length employed. Further, this value of $1.68 \pm 0.07\,\mathrm{kJ/m^2}$ is the same as that needed to cause fracture under a single blow. Thus, it appears that the effect of repeated blows under impact loading at sub-critical energy levels is to cause the inital crack to grow until it reaches a size when the applied energy level is sufficient for the specimen to fail at the fracture energy associated with fracture by a single-blow.

Finally, it is of interest to consider the rate of crack growth in the "repeated-blow" impact fatigue experiments. It was previously commented that it was not possible to distinguish whether this sub-critical extension of the initial crack occurred in a continuous incremental fashion or in discontinuous bursts after a given number of blows had been sustained. Nevertheless, the average increment of crack growth, $\Delta a/N$, per blow may be deduced from the data given in Tables I and II and this is plotted in Figure 9 against the value of the strain-energy release rate, G_I, based upon the value of the absorbed strain-energy associated with each blow (*i.e.* 11 mJ) and the initial crack length. (Obviously, using the initial crack length in Eqs. (1) and (2), as opposed to the current value

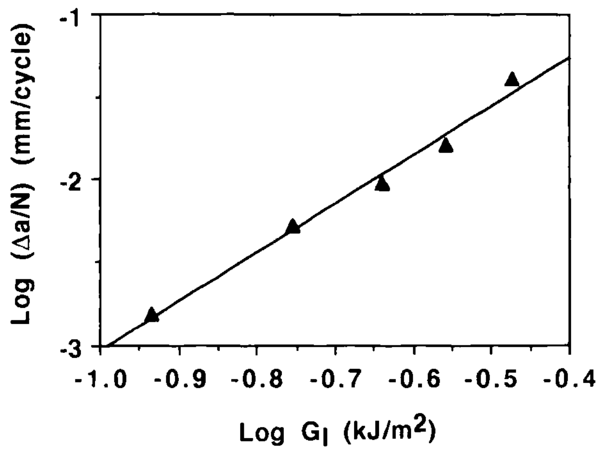

FIGURE 9 Logarithm of the average increment of crack growth per cycle, $\Delta a/N$ *versus* the logarithm of the strain-energy release rate, G_I, based upon the initial crack length.

of the crack length which applies as the crack grows during the impact-fatigue experiment, will result in Figure 9 representing a lower-bound relationship.) As may be seen, there is a linear correlation which suggests that a "Paris-Law" type equation, which is widely used with respect to dynamic-fatigue crack growth studies,[6] also describes the current impact-fatigue results. Hence, the data presented in Figure 9 may be described by:

$$\frac{\Delta a}{N} = AG_I^n \quad (3)$$

where the value of the constants A and n are -0.08 and 2.94, respectively.

CONCLUSIONS

The impact-fatigue behaviour of a rubber-toughened epoxy has been studied by determining the number of "repeated-blows," applied at a sub-critical energy level, that are needed to cause fracture of a single-edge cracked three-point bend specimen. The number of such repeated impacts that are necessary to cause fracture is a function of the applied energy level, when this energy is expressed as a percentage of the energy necessary to cause failure in a "single-blow"; the energy necessary to cause failure in a single-blow being, of course, a function of the initial crack length. There is no experimental evidence from the present study of a threshold value of the applied energy, below which impact fatigue failure would not be observed.

The damage accumulation mechanism appears to involve the growth of the initial crack until the length of the crack is such that the applied energy level is sufficient for the material to fail at the value of the fracture energy, G_{Ic}, needed to cause failure by a single-blow. For the present material this value of the fracture energy is about 1.5 to 1.6 kJ/m^2. Further, the crack growth rate during the impact-fatigue experiments may be described by a power-law equation, as typically employed in dynamic-fatigue studies, and in future work it is planned to use this observation as the basis for predicting the impact-fatigue lifetimes of bonded joints.

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References

1. B. N. Epstein and G. C. Adams, in *Toughened Plastics* (Conference Proc., Plastics and Rubber Institute, London, 1985), p. 10/1.
2. A. J. Kinloch and G. K. A. Kodokian, *J. Mater. Sci. Lettrs.* **6,** 653 (1987).
3. A. J. Kinloch and G. K. A. Kodokian, *J. Adhesion* **24,** 109 (1987).
4. A. J. Kinloch, G. K. A. Kodokian and M. B. Jamarani, *J. Mater. Sci.* **22,** 4111 (1987).
5. J. G. Williams, *Fracture Mechanics of Polymers* (Ellis Horwood, Chichester, 1984), p. 68.
6. A. J. Kinloch and R. J. Young, *Fracture Behaviour of Polymers* (Applied Science, London, 1983), p.204.