

Determination of the impact fracture parameters in ductile polymers

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This paper presents a new model for the impact fracture characterization of ductile polymers. The model takes into account the energy dissipated in plastic deformation during the crack propagation period. Two material parameters are used in the model: the fracture energy at initiation and an equivalent tearing modulus which represents the variation of the fracture energy as the crack grows. The method was used to measure the impact fracture toughness of a toughened nylon 66 and of a polycarbonate/polyethylene blend. Measurements were made in impact as well as at a low loading rate. The material fracture toughness was also measured by the J -integral method in nonlinear fracture mechanics at a low loading rate. The fracture energy at initiation determined from the proposed model is in good agreement with that of the J -integral method. Inconsistency and the negative intercept which result from the conventional method of analysis are also discussed in terms of the two material parameters.

(Keywords: ductile polymers; impact fracture parameters; nylon 66; blend)

INTRODUCTION

In spite of its importance, the characterization of impact fracture of plastics is still a controversial subject. The most common tests used in impact are the Charpy and Izod types. In these tests, analysis methods based on fracture mechanics have been developed with the objective of providing a quantitative description of the impact fracture process. However, several problems remain. Besides the dynamic effect in impact tests¹, the mode of fracture involved could result in a contradiction in the values of fracture energy measured.

Under impact, polymers can break in very distinct manners. Several modes of fracture have been identified depending on the amount of plastic deformation at the crack tip and the stability of crack propagation. For each mode, an appropriate analysis must be used to determine the impact fracture energy of the material.

Brittle fracture occurs when the strain energy stored in the sample up to the point of fracture is much larger than the energy dissipated in the creation of the two fracture surfaces. In this case the crack grows in an unstable manner. The energy absorbed by the specimen to fracture is that which is elastically stored in the sample. A method has been proposed for this mode of fracture to determine the fracture energy at crack initiation²⁻⁴.

When the plastic zone at the crack tip becomes significant, the fracture has a semi-brittle character and a plastic zone correction of the method is suggested⁵.

When the polymer is more ductile, stable crack propagation occurs before unstable fracture. For this semi-ductile fracture mode, a method using two material parameters has been proposed⁶ to characterize the impact resistance of the polymer. The method allows the determination of an average value of the fracture energy in the stable crack propagation stage and the fracture energy at instability.

The development of copolymers and polymer blends

has resulted in materials exhibiting more and more ductile behaviour in impact. When the material is ductile, unstable fracture does not occur. The crack propagation is generally completely stable. For this mode of fracture the measurement of fracture energy is still controversial. The current method is based on the assumption of constant fracture energy during the fracture process. The energy absorbed by the sample is considered as proportional to the fracture surface. The impact fracture energy is therefore determined from the slope of the plot of absorbed energy against fracture surface.

For many ductile plastics, this method often gives abnormally high values of impact fracture energy. Furthermore, an inconsistent negative intercept of the absorbed energy *versus* surface plot is generally observed.

The purpose of this paper is to analyse the inconsistency of the current method used to measure the impact fracture energy of ductile polymers. Measurements were made on toughened nylon 66 and a polycarbonate/polyethylene blend. A new method is proposed for the characterization of impact fracture in ductile polymers. The method allows the determination of fracture energy at crack initiation. Measurements were also performed at a low loading rate and compared with the contour J -integral method.

EXPERIMENTAL

Materials and sample preparation

The toughened nylon 66 was obtained from Du Pont (Zytel ST-801) in granular form. The granules were dried in a dry nitrogen atmosphere at 90°C for 16 h before moulding. Both 3 and 6 mm thick plates of 30 by 90 mm were injection moulded. Three-point-bend samples of 12 by 90 mm were made by machining away sides of the moulded plates.

The polycarbonate/polyethylene (PC/PE) blend was

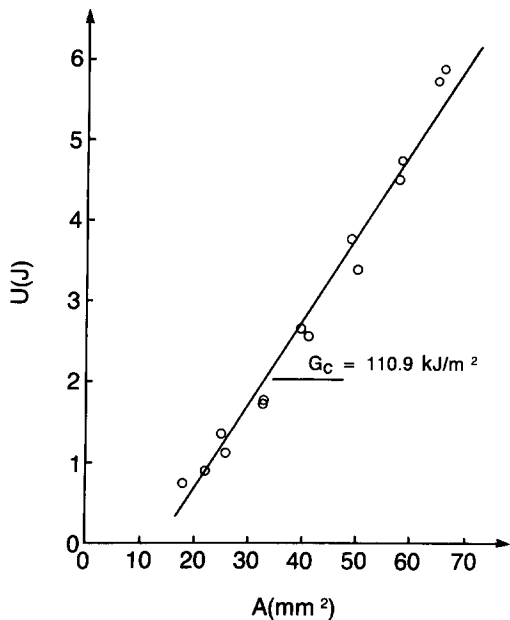


Figure 1 Absorbed energy versus fracture surface for toughened nylon 66 (correlation coefficient = 0.9915)

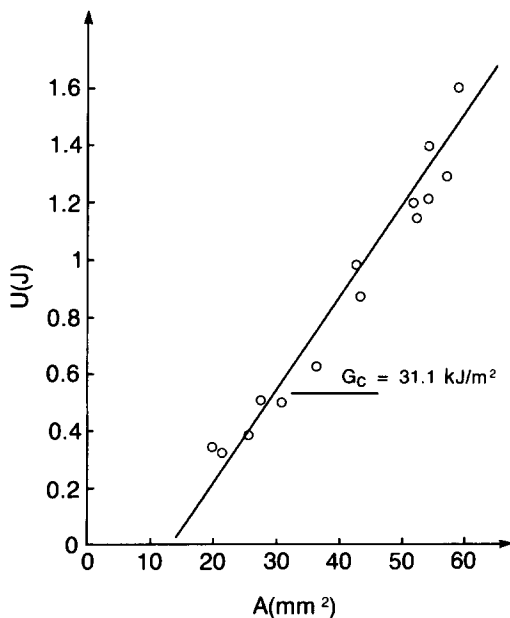


Figure 2 Absorbed energy versus fracture surface for PC/PE blend (correlation coefficient = 0.9788)

obtained from the VAMAS working party on polymer blends. The blend contained 75 vol % of a polycarbonate from General Electric Co. (Lexan RL), and 25 vol % of a linear low density polyethylene from Esso Chemicals Canada (Escarene LL). Samples of 12 by 90 mm were cut out from a 5 mm thick extruded sheet. The samples were cut parallel to the extrusion direction.

The length of the initial crack created in the specimens was varied between 20 and 80 % of the specimen width. A pre-notch was first made by a saw cut and the final crack was created by forcing a razor blade into the specimen jig.

Testing procedures

Impact tests were performed on a Dynatup instrumented impact tester, Model 600. The impact speed was 3 m s^{-1} . The energy absorbed by the specimen to

fracture as well as the load-time trace were automatically provided by the impact machine.

The three-point-bending tests at low loading rate were performed on a standard testing machine (Instron, Model 1125). The cross-head speed was 10 mm min^{-1} . The energy required to break the sample was determined from the area under the load-deflection diagram.

All the tests were carried out at ambient temperature (23°C) and 50 % relative humidity.

RESULTS AND DISCUSSION

Figures 1 and 2 show the plots of energy absorbed by the specimen to fracture, U , as a function of the fracture surface, A , for the cases of toughened nylon 66 (6 mm thick samples) and PC/PE blend, respectively. As often observed in ductile polymers, these plots show an inconsistent negative intercept. A close examination of these curves also revealed that dU/dA tended to increase with increasing A . This can lead to an erroneous conclusion about the mode of fracture involved. As demonstrated previously for unstable crack propagation^{2,3}, the energy absorbed by the sample is that stored elastically up to the point of fracture. The elastic strain energy was shown to be related to the material's fracture energy via a ubiquitous factor^{2,7} or a calibration factor^{3,4}. For three-point-bend samples, these factors are not a linear function of the fracture surface. For instance, for the calibration factor ϕ (refs 3, 4), $d\phi/da$ increases with increasing fracture surface (a is the crack length). The plot of U against A should therefore show an increasing slope with increasing A . In this case, the fracture energy is obtained by plotting U against ϕ (refs 3, 4). Figures 3 and 4 show such plots for the toughened nylon 66 and PC/PE blend, respectively. The U versus $BD\phi$ plots (where B is the thickness and D is the width of the samples) give slightly better correlation coefficients than the previous plots. This could result in spurious values of the fracture energy. Indeed, as discussed previously⁶, ductile fracture generally occurs with stable crack propagation and with a

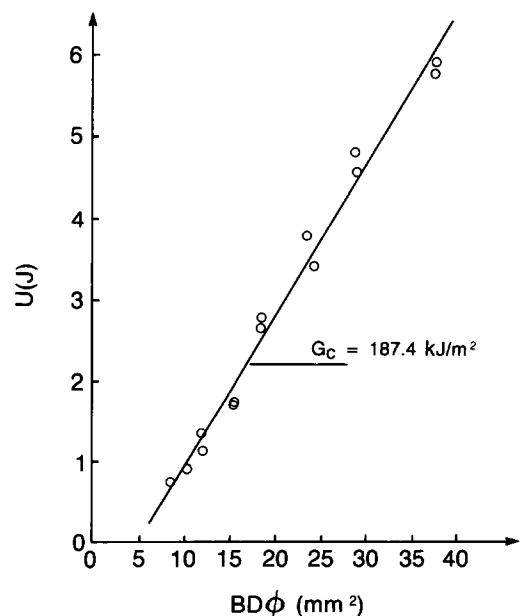


Figure 3 Absorbed energy as a function of $BD\phi$ for toughened nylon 66 (correlation coefficient = 0.9941)

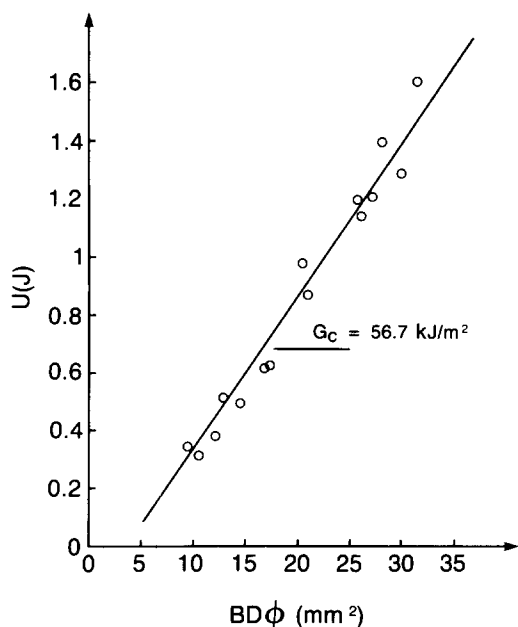


Figure 4 Absorbed energy as a function of $BD\phi$ for PC/PE blend (correlation coefficient = 0.9875)

continuous supply of energy from the hammer to the sample. The energy absorbed by the sample to break is therefore not related to the elastic strain energy.

Before pursuing the discussion, it is worth noting that two techniques could be used to distinguish between stable and unstable fractures. The first technique consists in observing the movement of the sample after fracture. In unstable crack propagation, the energy absorbed by the sample is that stored elastically. Only part of this elastic strain energy is dissipated in the fracture surfaces. The remaining part is transformed into kinetic energy. As a consequence, the sample generally tends to break completely into two halves and fly away after fracture. In contrast to this behaviour, ductile fracture occurs with stable crack propagation and with a continuous supply of energy from the hammer to the specimen. After the fracture, the two halves remain attached together by a thin ligament and are pushed away by the hammer with a much lower velocity. The second technique is based on the observation of the fracture surface. For many polymers the fracture surface of an unstable fracture is typical. With the naked eye, the surface appears rough and often shows a branching effect as opposed to that of stable fracture, where the surface exhibits a whitening effect or could become bright, reflecting light, due to craze formation⁸.

From Figures 1–4, it can be seen that both the U versus A and U versus $BD\phi$ plots exhibit an inconsistent negative intercept for these materials. Furthermore, from the slope of these curves, abnormally high values of G_c are obtained. For example, the Irwin model expresses the plastic zone ahead of the crack tip in a plane strain situation as:

$$r_p = \frac{1}{6\pi(1-\nu^2)} \frac{G_c E}{\sigma_y^2} \quad (1)$$

where G_c is the fracture energy, E is the elastic modulus, σ_y is the yield stress and ν is the Poisson ratio. For toughened nylon 66, with a yield stress of about 40 MPa, an elastic modulus of about 2000 MPa and $\nu \approx 0.3$, from the

value of fracture energy of 110.9 kJ m^{-2} (Figure 1), the plastic zone ahead of the crack tip would be about 8 mm. This is not plausible given the dimensions of the specimen. Examination of specimens in which the hammer was stopped before complete fracture also revealed that the plastic zone in front of the crack tip was much smaller, as shown in Figure 5.

In ductile fracture, it has been shown by J -integral measurements for many materials that the energy dissipated in the fracture process generally increases with crack extension^{9,10}. The increase in fracture energy with crack propagation has also been observed in impact fracture of semi-ductile polymers. It was found that the average fracture energy during the stable crack growth period in polyamide 11 was substantially lower than the fracture energy at the instability point⁶. The increase in fracture energy during crack extension is therefore most probably the cause for the increase in dU/dA with increasing A in the U versus A plots and the inconsistencies of the above results.

Taking G_R as the fracture energy and considering that G_R varies with crack extension, the energy dissipated in the fracture surface A becomes:

$$U = \int_A G_R dA \quad (2)$$

Since G_R is a function of A , U is no longer a linear function of A and the U versus A plot cannot be used to determine the fracture energy.

The increase in fracture energy with crack extension has often been characterized by the contour integral J_R , crack growth resistance curve^{9,10}. It has also been suggested that the rise in J_R occurs at a constant rate which is a material characteristic^{10,11}. Recent results of measurements on different specimen types¹² have shown that the tearing modulus

$$T = \frac{E}{\sigma_y^2} \frac{dJ_R}{da} \quad (3)$$

is apparently independent of specimen geometry.

Although the tearing modulus is only defined for small magnitudes of crack extension, the idea was extended to the case of stable impact fracture. Considering an equivalent tearing modulus, T_a , as a material constant,

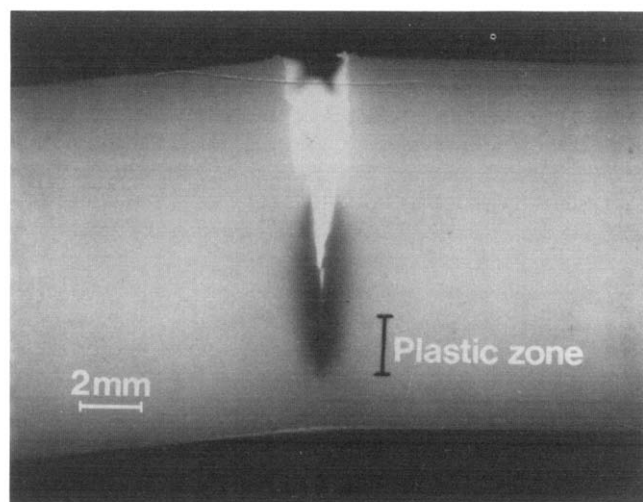


Figure 5 Plastic zone in 6 mm thick toughened nylon samples

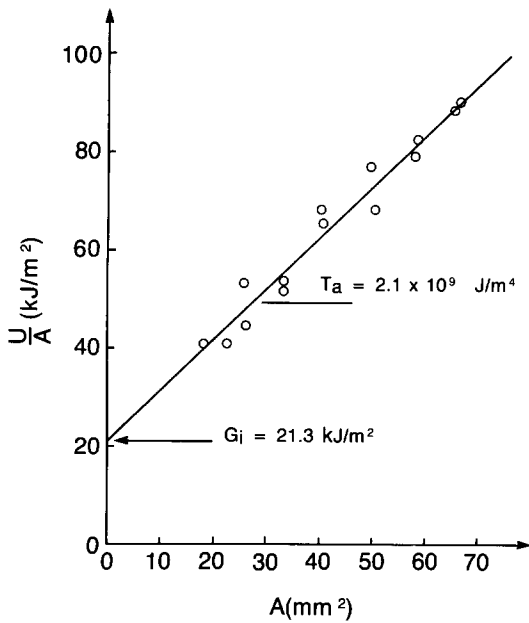


Figure 6 U/A as a function of A for toughened nylon 66

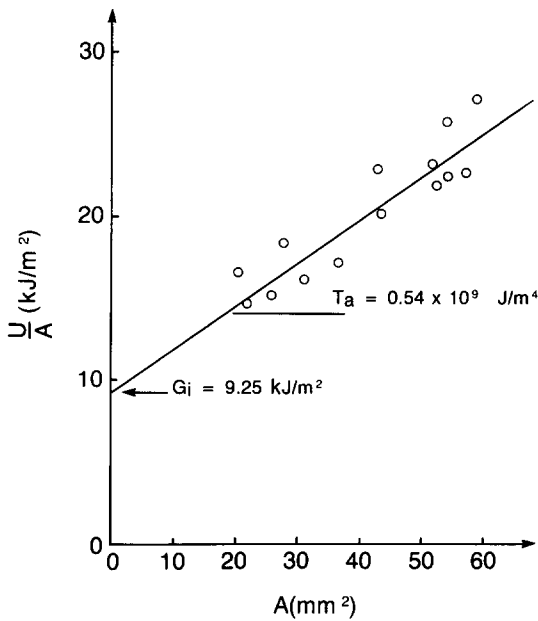


Figure 7 U/A as a function of A for PC/PE blend

representing the rate of increase in G_R with A , the fracture energy can be expressed as a function of the fracture surface, A , as:

$$G_R = G_i + T_a A \quad (4)$$

where G_i is the fracture energy at crack initiation. Assuming that the energy absorbed by the specimen is that dissipated in the fracture surface, the energy absorbed by the specimen becomes:

$$U = G_i A + \frac{1}{2} T_a A^2 \quad (5)$$

Figures 6 and 7 show the plots of U/A against A for the toughened nylon 66 and PC/PE blend, respectively. Surprisingly, a relatively good linear relationship between U/A and A was obtained. The results suggested that in spite of the large amount of crack growth involved, the equivalent tearing modulus described these ductile

impact fractures fairly well. From these curves, the extrapolation to $A=0$ gives an estimate of the value of fracture energy at crack initiation, G_i . In a way the technique is similar to that of the J_R -curve method. In fact, in the J_R -curve method, the initiation fracture energy is determined from the intersection between the crack growth resistance curve and the blunting line. The crack extension due to crack tip blunting could be considered similarly in the U/A versus A plot. However, the error is generally small. Moreover, the determination of yield stress in impact is too difficult to take into account the blunting effect.

From the values of G_i and T_a determined in Figures 6 and 7, the actual energy absorbed by the specimen to fracture can be plotted as a function of the fracture surface according to the above model. Figures 8 and 9 show the comparison between the proposed model for ductile fracture and the experimental measurements for the toughened nylon 66 and PC/PE blend, respectively. The results demonstrate that the proposed model is a good description of the energy absorbed by the specimen in ductile fracture. It also explains the inconsistency of the negative intercept in the current method. From the proposed method, much lower values of fracture energy at crack initiation are obtained. For toughened nylon 66, the value of G_i is also substantially lower than the impact fracture toughness measured by an instrumented impact test, reported previously¹³. To verify the result, measurements on 3 mm thick samples of toughened nylon 66 were made at a low loading rate. The J -integral technique¹⁴ was used to measure the fracture toughness at crack initiation. Different samples of the same initial crack length, $a_0/D=0.4$, were loaded to different values of load point deflection, leading to different magnitudes of crack extension, Δa , and then unloaded. The small amounts of crack growth were marked by a penetrant ink and the specimens were broken by impact at room temperature. The J_R values were approximated as:

$$J_R = \frac{2U}{B(D-a)} \quad (6)$$

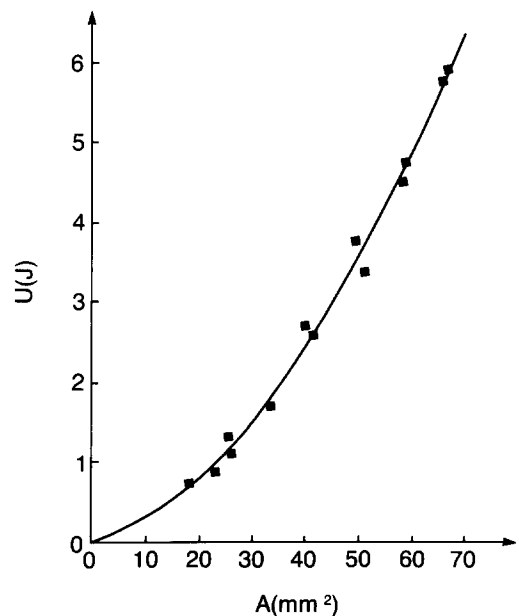


Figure 8 Absorbed energy as a function of fracture surface for toughened nylon 66: comparison between (—) the proposed model ($G_i=21.3 \text{ kJ m}^{-2}$, $T_a=2.1 \times 10^9 \text{ J m}^{-4}$) and (■) experimental data

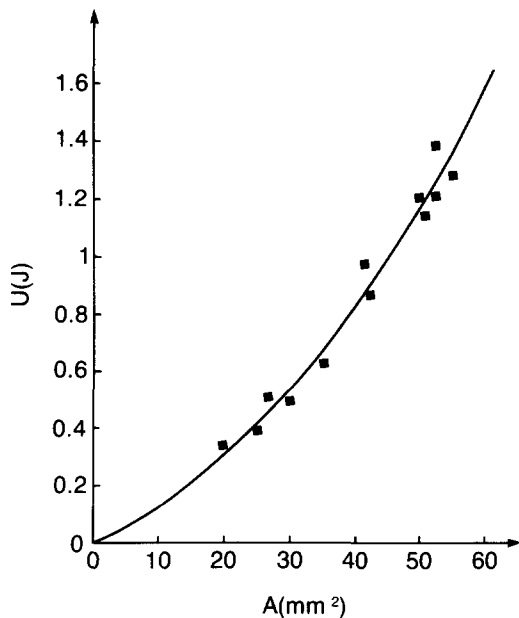


Figure 9 Absorbed energy as a function of fracture surface for PC/PE blend: comparison between (—) the proposed model ($G_i = 9.2 \text{ kJ m}^{-2}$, $T_a = 0.54 \times 10^9 \text{ J m}^{-4}$) and (■) experimental data

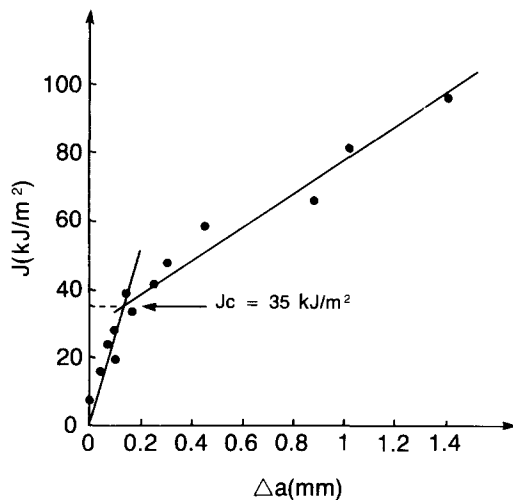


Figure 10 J -integral measurement for toughened nylon 66 at 10 mm min^{-1}

where B is the specimen thickness, D is the specimen width, a is the current crack length and U is the area of the load–deflection curve. *Figure 10* shows the plot of J_R as a function of crack extension. A clear crack extension due to blunting was observed. At crack initiation the slope of the J_R versus Δa line changed abruptly, permitting a straightforward determination of J_c . The J_c result was also found to be smaller than the impact fracture energy reported for toughened nylon 66¹³.

Complete fracture tests were then performed on samples of different initial crack lengths. The energy absorbed by the sample to fracture was measured from the area under the load–deflection diagram. *Figure 11* shows the U/A versus A plot. The intercept of the curve gives the value of G_i , which is in good agreement with the J_c value. Since the blunting effect is neglected in the U/A versus A plot, G_i is slightly lower than J_c . The results show that in this case the error is about 10%. It is worth noting

that, in impact, the yield stress is higher and the effect of blunting must be reduced, resulting in less error in the determination of fracture energy at crack initiation. The results suggest that the concept of tearing modulus is still valid for large magnitudes of crack extension.

Another question could be raised at this point regarding the effect of initial crack length on the apparent tearing modulus in the proposed model. In varying the initial crack length, the neutral axis in the uncracked ligament also varies. This could affect the spread of plasticity in front of the crack tip and alter the apparent modulus. To verify this effect, comparison was made between specimens containing initial crack lengths, a_0/D , of 0.2 and 0.6. The rise in crack growth resistance in these specimens was determined by the variation of the critical stress intensity factor during crack propagation. This factor was calculated using the calibration factor $f(a/D)$ for three-point-bend samples provided in Reference 15:

$$K_R = \frac{PS}{BD^{3/2}} f\left(\frac{a_{\text{eff}}}{D}\right) \quad (7)$$

where P is the load, S is the support span and a_{eff} is the effective crack length, which is the physical crack size augmented for the dimension of the plastic zone ahead of the crack tip. At each load level on the load–deflection diagram, the effective crack length, a_{eff} , was calculated from the instantaneous compliance, C , determined by a secant passing through the origin of the load–deflection curve, as discussed previously^{16,17}.

Figure 12 shows the plot of K_R as a function of effective crack extension for toughened nylon 66 specimens containing different initial crack lengths. The result shows that the rise in K_R with Δa_{eff} is identical for $a_0/D = 0.2$ and 0.6, suggesting that the initial crack length has no effect on the rise in crack growth resistance with crack propagation. The consideration of apparent tearing modulus as a constant, regardless of initial crack length, is therefore a valid assumption in the proposed method.

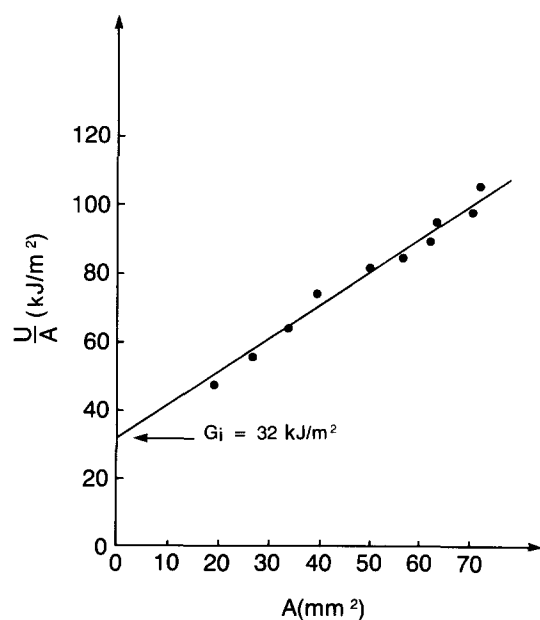


Figure 11 U/A as a function of A for toughened nylon 66 at 10 mm min^{-1}

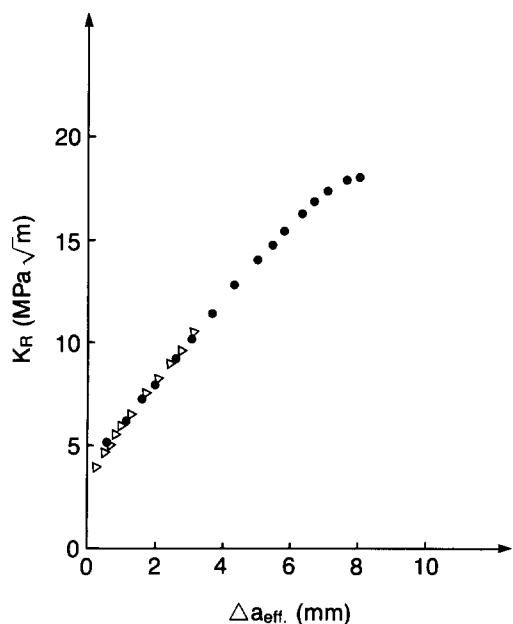


Figure 12 Critical stress intensity factor as a function of effective crack extension for toughened nylon 66: ●, $a_0/D=0.2$; ▽, $a_0/D=0.6$

SUMMARY AND CONCLUSION

The measurement of impact fracture energy in ductile polymers has been analysed. The inconsistencies of the current method have been shown to be related to a variation of fracture energy with crack propagation. A new method has been proposed for the characterization of impact fracture. This method is based on a constant rate of increase in crack growth resistance with crack extension. Two parameters can therefore be determined from the proposed model: a fracture energy at crack initiation and an equivalent tearing modulus. The model describes closely the variation of the energy absorbed by the specimen to fracture with the fracture surface. It demonstrates the cause of the abnormally high value of fracture energy and explains the inconsistent negative intercept of the current method.

Although the tearing modulus is only defined for small amounts of crack extension, the results showed that the concept could be extended to the case of large magnitudes of crack propagation. Measurements performed under a low loading rate demonstrated that the fracture energy at crack initiation is in good agreement with that measured by the J -integral technique. Comparison between specimens containing different initial crack lengths showed that the uncracked ligament has no effect on the equivalent tearing modulus.

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