Determination of the fracture toughness of polymeric films by the essential work method

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Complete ligament yielding before crack advance is one of the criteria in the essential work method. It is found that this criterion also holds when the essential work method is used to obtain the fracture toughness of polymeric films. This is done by using two specimen geometries to test each of two nylons. The criterion is then used to find the fracture toughness of three polyethylenes, a cast, amorphous poly(ethylene terephthalate) and two kaptons. In addition, the effects of specimen width and strain rate on the specific essential work of fracture w_e are studied.

(Keywords: fracture toughness; polymeric films; essential work method)

INTRODUCTION

The characterization of high toughness and low yield strength materials is a difficult matter and many problems remain. The recent successes in applying the J integral method to metals[1] have generated interest in using the same approach for polymers[2–4]. Some progress has been made and an international standard for using J tests on polymers is on its way to completion[5].

However, the draft standard emphasizes finding a toughness value in plane strain which is independent of the geometry and dimensions of the specimen tested. This imposes a restriction on the minimum thickness of the specimens used. Since many tough materials used in industry are in thin-film form, testing such thick samples will give a lower toughness value than that actually encountered. This is due to the transition in the stress state from plane strain to plane stress, which results in a higher toughness value. In addition, the specimen geometries (i.e. compact tension (CT) and single-edged notch bending (SENB)) recommended in the draft standard are difficult if not impossible to use in film form and there is no practical method of characterizing the fracture toughness of polymeric films.

The J integral method is based on the existence of a parameter J which can be evaluated either via Rice's line integral[6] on a path surrounding the crack tip

$$J=\oint_{\Gamma} W\mathrm{d}y-T_i\frac{\partial u_i}{\partial x}\mathrm{d}s$$

where $W=\int\sigma_{ij}\,\mathrm{d}\varepsilon_{ij}$, T_i is the outward traction vector on ds, u_i is the displacement vector at ds, ds is the increment of the contour path, σ_{ij} is the stress component and ε_{ij} is the strain component, or from the potential energy interpretation of the above integral[7]

$$J=-\frac{\mathrm{d}U}{\mathrm{d}A}$$

where U is the potential energy and A is the fracture area.

By contrast, the essential work method is based on Borberg's suggestion[8,9] that the non-elastic region at the crack tip should be divided into an end region, where the fracture process takes place, and an outer region, where screening plastic deformation is required to accommodate the large strains in the end region. Although the two methods approach the fracture problem differently, it has been shown that the resulting critical fracture parameters are compatible[10]. Since the J integral method is an established method[1] for testing the fracture toughness of ductile materials, its association with the essential work method would suggest that the latter can be used to characterize the fracture toughness of ductile materials.

The essential work method has been applied successfully to both metals[11–15] and polymers[10,16–19] in thin-sheet form. The single-edged notch tension (SENT) and double-edged notch tension (DENT) specimen geometries used in the method are convenient geometries for polymeric films. The maximum ligament length used to extrapolate the plot of specific work to fracture w_f against ligament length L to zero L is limited to the size of the calculated plastic zone[11]. In some cases, this implies that the maximum L must be less than one third of the specimen width. The aim of this paper is to verify the applicability of the essential work method to characterize the fracture toughness of polymeric films. The effects of specimen width and strain rate on w_e are also investigated.

THEORY

The essential work method divides the input energy into

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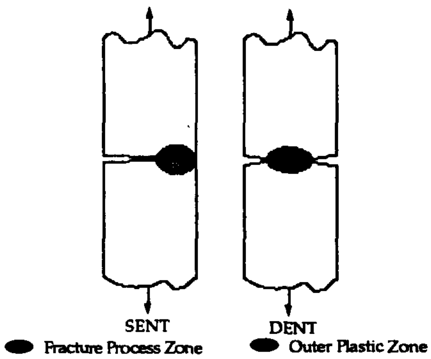

Figure 1 Schematic diagrams showing the fracture process zones and outer plastic zones of SENT and DENT specimens

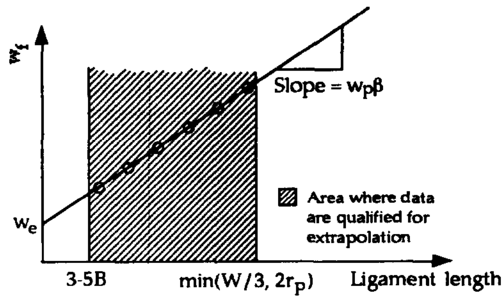

Figure 2 Plot of specific energy to fracture w_f against ligament length L

two parts, namely the essential work and the non-essential work

$$W_f = W_e + W_p \quad (1)$$

where W_f is the energy to fracture, W_e is the essential work of fracture and W_p is the non-essential work of fracture. The former will account for the necking and fracture of the material in the fracture process zone, while the latter is the plastic deformation in the outer plastic zone, which for metals will involve shear and for polymers will involve microvoiding and shear. *Figure 1* shows the fracture process zones and outer plastic zones for two specimen geometries, namely SENT and DENT.

It is assumed for metals[11] and polymers[18] that the plastic zone area is proportional to the ligament length squared, and if it is assumed that the essential work is proportional to the ligament area, then equation (1) can be rewritten as

$$W_f = w_e LB + w_p \beta B L^2 \quad (2)$$

where B is the thickness, L is the ligament length, w_e is the specific essential work of fracture, w_p is the specific non-essential work of fracture and β is the shape factor. Hence

$$w_f = w_e + w_p \beta L \quad (3)$$

where $w_f = W_f/LB$. Therefore, if a set of specimens with different ligament lengths is tested and W_f divided by the ligament area is plotted against L, as shown in *Figure 2*, a linear relationship can be obtained. If L is extrapolated back to zero, the specific essential work of fracture w_e, which is assumed to be a material property, can be found.

Two restrictions are applicable to the essential work method. The first restricts the maximum ligament length used for extrapolation[18] to one third of the specimen width or the plastic zone size, whichever is the lower

$$L < \min(W/3, 2r_p)$$

where W is the specimen width and $2r_p$ is the plastic zone size. There are two reasons for imposing this restriction. Firstly, it restricts the plastic deformation to the ligament area, thus preventing the deformation from spreading to the lateral boundary of the specimen. Secondly, it ensures complete yielding of the ligament before the crack starts to grow. The second restriction to the essential work method is that the ligament length used for extrapolation should be longer than three to five times the specimen thickness[11]. This avoids the plane stress–plane strain transition region where the theory breaks down.

MATERIALS

Two types of nylon (Zytel ST801 and Zytel ST901), three types of polyethylenes (PE(LL0209AA), PE(LL7909AA) and PE(HD6070FY)), two types of polyimide (kapton V and kapton H) and a cast, amorphous poly(ethylene terephthalate) (PET) were used in this investigation. Both nylons are rubber toughened and hygroscopic. Zytel ST801 is crystalline and Zytel ST901 is amorphous, and the mechanical properties of both change with water content. They were stored in airtight packaging and the air seal was only broken just prior to testing. Therefore, the recorded mechanical properties were the as-moulded values. Both PE(LL0209AA) and PE(LL7909AA) are blow-mould grades of linear, low density polyethylene. PE(HD6070FY) is a cast, high density polyethylene. The two kaptons are polyimides and are almost identical, the main difference being that the V-type kapton has been heat treated to improve dimensional stability. The poly(ethylene terephthalate) is a cast, amorphous material.

Both the yield stresses and Young's moduli of the above materials were obtained by following ASTM D882-88[20]. Grip separation was taken as the extension except for the nylons, where clip gauges were used to monitor the extension of the strip. A summary of the yield stresses and Young's moduli, with the corresponding specimen thicknesses at a 10 mm min^{-1} crosshead speed, for all the above materials is given in *Table 1*.

It should be noted that for the kaptons the yield stress was taken at a 0.2% strain offset, whereas for the other materials it was taken as the maximum stress measured.

EXPERIMENTAL

SENT and DENT specimens of different sizes were used. The notches were made perpendicular to and at the midgauge length, using a razor blade with a tip radius

Table 1

Material	Yield stress σ_y (MPa)	Young's modulus E (GPa)	Specimen thickness B (mm)
Zytel ST901	59	2.1	1.6
Zytel ST801	47	2.0	1.6
PE(LL7909AA)	14	0.4	0.038
PE(LL0209AA)	10	0.2	0.038
PE(HD6070FY)	17	0.6	0.035
Kapton VN200	48.1	2.9	0.05
Kapton HN200	44.1	3.1	0.05
Cast, amorphous PET	40.0	2.3	0.18

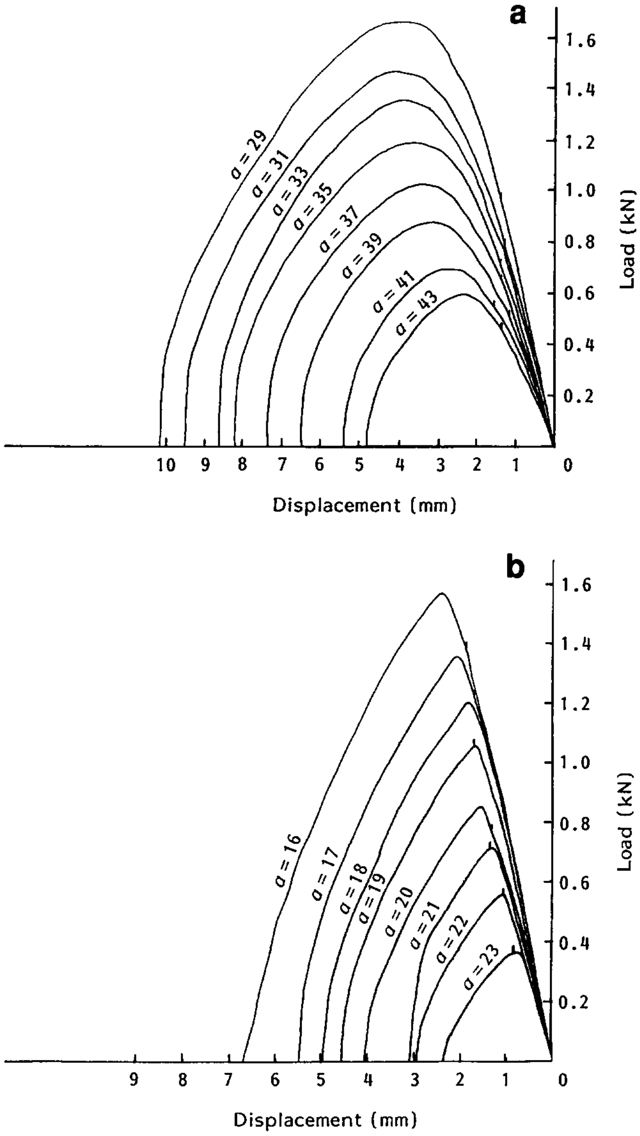

Figure 3 Load–displacement plots for Zytel ST801: (a) SENT geometry; (b) DENT geometry

of 0.01 mm. To obtain sharp crack tips, the razor blade was cut into the specimen. Plates with slots in their midplane were used to guide the blade. For the nylons and kaptons, antibuckling guides were used during loading to prevent out of plane deformation of the films owing to their low lateral stiffness. Such guides were not used for the polyethylenes because they easily became charged with static electricity during handling. If guides had been used, the films would have adhered to the guides and a high frictional force would have arisen during loading to distort the load–displacement results. The specimens were held in grips with sandpaper attached to the gripping surfaces.

Owing to the higher thickness of the nylon specimens, two modifications were made to the above experimental procedures. Firstly, a starter notch was generated by a hacksaw before the razor cut. Secondly, a rough gripping surface was used instead of the sandpaper gripping surfaces.

The grips were attached to an Instron screw-driven machine and the tests were carried out at room temperature.

RESULTS AND DISCUSSION

Nylons (Zytel ST801 and Zytel ST901)

Figures 3a and *3b* show the load–displacement diagrams for Zytel ST801 SENT and DENT specimens, respectively, and *Figures 4a* and *4b* show the corresponding diagrams for Zytel ST901. The thickness, width and gauge length for all the nylon specimens were 1.6 mm, 50 mm and 100 mm, respectively.

Figures 5 to *8* show that if the maximum ligament length used to extrapolate back to zero ligament length is gradually reduced, both the specific essential work of fracture and the specific non-essential work of fracture can be expressed as a function of ligament length *L*.

Figure 7a shows that w_e for the Zytel ST801 SENT specimens decreases with decreasing *L* down to 16 mm and then remains constant, while w_e for the DENT specimens is constant for all *L*. The w_e for the DENT and SENT specimens are equal for *L* less than 16 mm. Similarly, *Figure 7b* shows that $w_p\beta$ for the Zytel ST801 SENT specimens increases with decreasing *L* down to

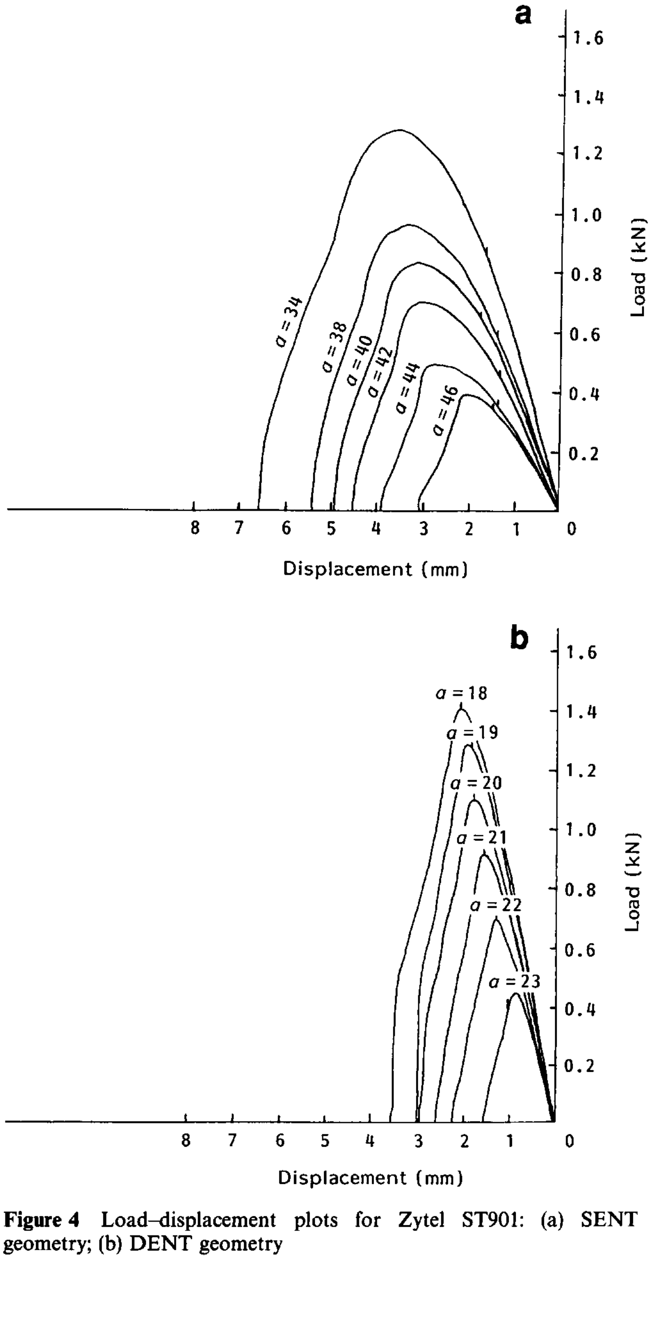

Figure 4 Load–displacement plots for Zytel ST901: (a) SENT geometry; (b) DENT geometry

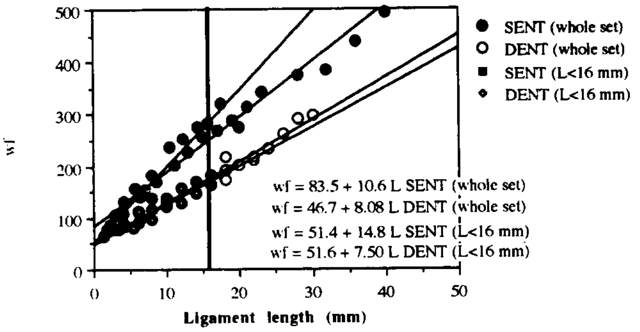

Figure 5 Comparison of different interpolation ranges on w_f (kJ m^{-2}) against L for Zytel ST801 SENT and DENT specimens (gauge length = 100 mm, W = 50 mm, B = 1.6 mm) tested at a 10 mm min^{-1} crosshead speed

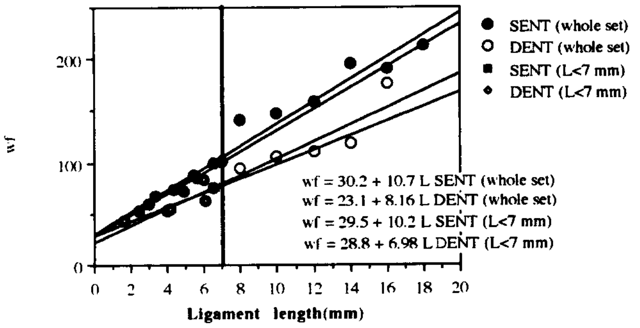

Figure 6 Comparison of different interpolation ranges on w_f (kJ m^{-2}) against L for Zytel ST901 SENT and DENT specimens (gauge length = 100 mm, W = 50 mm, B = 1.6 mm) tested at a 10 mm min^{-1} crosshead speed

16 mm and then remains constant, while for the DENT specimens it stays constant for all L.

The SENT specimen results indicate that the linear relationship between w_f and L, as postulated in the essential work theory, breaks down when L is greater than 16 mm

$$w_f = 51.4 + 14.8L \quad \text{for SENT}$$

$$w_f = 51.6 + 7.50L \quad \text{for DENT}$$

The same conclusion can be drawn from the results for Zytel ST901, which show that the linear relationship between w_f and L breaks down when L is greater than 7 mm

$$w_f = 29.5 + 10.2L \quad \text{for SENT}$$

$$w_f = 28.8 + 6.98L \quad \text{for DENT}$$

This is illustrated in *Figures 8a* and *8b*.

If the maximum ligament length used for extrapolation $\hat{L}$ is compared with the calculated plastic zone $2r_p$ for contained yielding[21], where

$$2r_p = \frac{Ew_e}{\pi\sigma_y^2}$$

then it can be seen in *Table 2* that $2r_p$ is approximately equal to $\hat{L}$ for both materials. This means that L must be completely yielded before the crack advances. Cotterell and Reddel[11] have also suggested a similar restriction on $\hat{L}$ for metals.

The initiation loads were recorded by marking the load–displacement curves (*Figures 3* and *4*) at the points where the cracks started to grow, as observed via a microscope. It was noted that for both nylons, the initiation load was below the maximum load for small crack lengths and moved towards the maximum load as the crack length increased, as shown in *Figures 3* and *4*.

Figure 9 shows the maximum net section stress σ_{nm}, i.e. the maximum load divided by the original ligament length and thickness, as a function of ligament length for

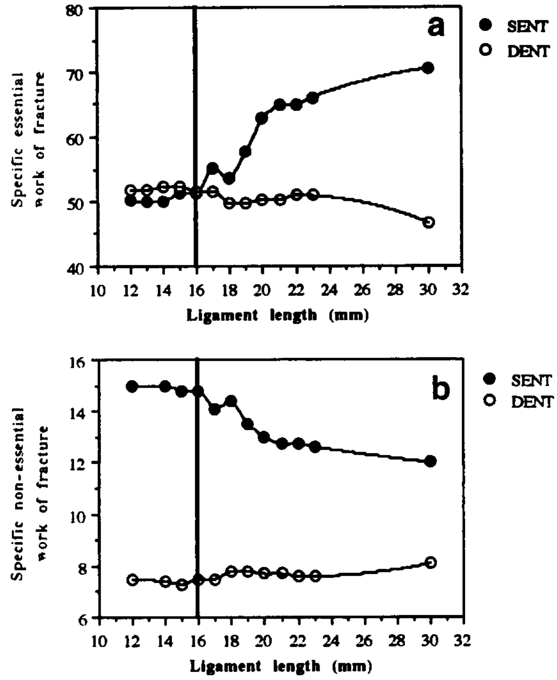

Figure 7 (a) Specific essential work of fracture w_e (kJ m^{-2}) and (b) specific non-essential work of fracture w_p (MJ m^{-3}) *versus* ligament length for Zytel ST801 SENT and DENT specimens tested at a 10 mm min^{-1} crosshead speed

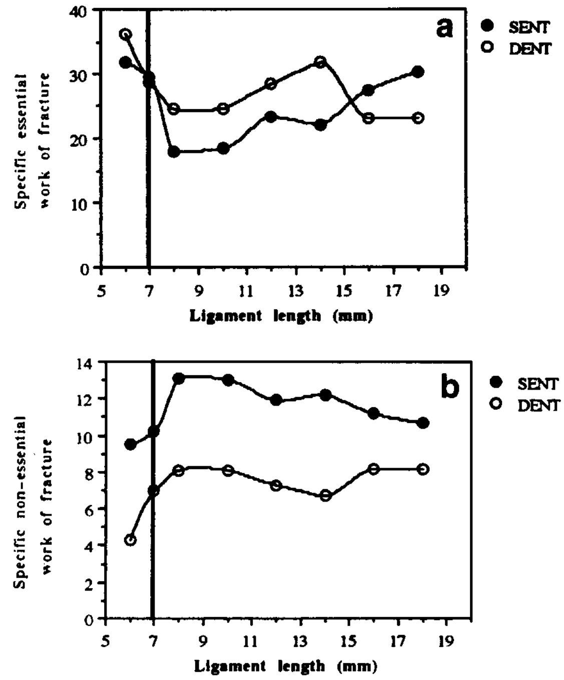

Figure 8 (a) Specific essential work of fracture w_e (kJ m^{-2}) and (b) specific non-essential work of fracture w_p (MJ m^{-3}) *versus* ligament length for Zytel ST901 SENT and DENT specimens tested at a 10 mm min^{-1} crosshead speed

Table 2

Material	$\hat{L}$ (mm)	E (GPa)	σ_y (MPa)	w_e (kJ m^{-2})	$2r_p$ (mm)	$2r_p/\hat{L}$
Zytel ST801	16	2.0	47	51	14.7	0.92
Zytel ST901	7	2.1	59	29	5.6	0.8

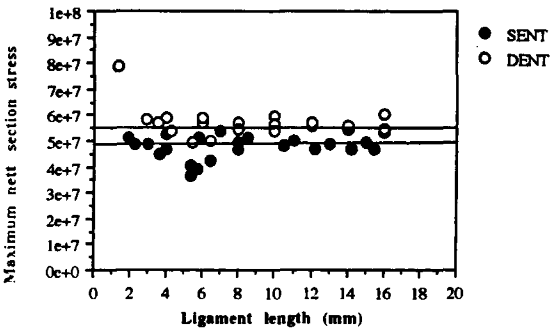

Figure 9 Maximum net section stress (Pa) *versus* ligament length for Zytel ST801 SENT and DENT specimens

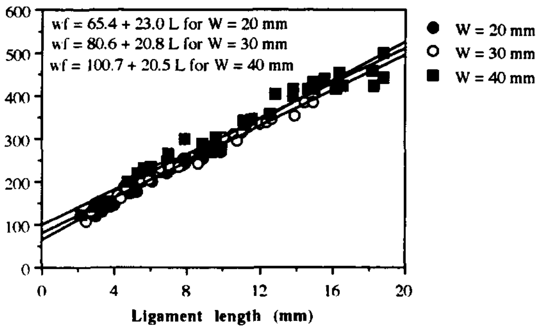

Figure 10 Plots of specific energy to fracture w_f (kJ m^{-2}) against ligament length L for PE(LL0209AA) SENT specimens (gauge length = 100 mm, W=20–40 mm, B=0.038 mm; whole set)

both specimen geometries of Zytel ST801. Note that the net section stress at the maximum load is equal to the yield stress for the specimen geometry concerned. The yield stresses of the two specimen geometries are different because the notch constraint effect elevates the DENT yield stress to 1.15 times the uniaxial yield stress[22], which is the yield stress for SENT.

However, the initiation load for small crack lengths is less than the maximum load, and this implies that the net section stress at initiation for small crack lengths is below the yield stress and the ligament is not completely yielded at initiation.

Since the initiation point moves towards the maximum load as the crack length increases, provided that the crack is small enough the ligament will be completely yielded. Because complete ligament yielding is one of the assumptions in the essential work method, the restriction on the ligament length used for extrapolation is obviously needed. This conclusion supplements the previous results.

It is concluded that $\hat{L}$ is subject to a restriction, namely $\hat{L} < Ew_e/\pi\sigma_y^2$. As long as this condition is met, the essential work method can be applied to obtain the fracture toughness w_e of a material. The following results illustrate how the w_e values of some ductile films can be obtained, and also show the effects of the specimen dimensions and test speed on w_e.

Polyethylenes (PE(LL0209AA), PE(LL7909AA) and PE(HD6070FY))

Figure 10 shows that for PE(LL0209AA) SENT specimens w_e increases when the specimen width W increases

$$w_f = 65.4 + 23.0L \quad \text{for } W = 20\,\text{mm}$$
$$w_f = 80.6 + 20.8L \quad \text{for } W = 30\,\text{mm}$$
$$w_f = 100.7 + 20.5L \quad \text{for } W = 40\,\text{mm}$$

and $w_p\beta$ decreases with increasing W. However, if $\hat{L}$ is reduced to 7 mm, as shown in *Figure 11*, then w_e becomes independent of W

$$w_f = 47.3 + 27.1L \quad \text{for } W = 20\,\text{mm}$$
$$w_f = 51.2 + 27.0L \quad \text{for } W = 30\,\text{mm}$$
$$w_f = 50.0 + 31.0L \quad \text{for } W = 40\,\text{mm}$$

If this w_e is used to evaluate the limit for $\hat{L}$, namely $Ew_e/\pi\sigma_y^2$, it gives $\hat{L}$=31.8 mm. This means that the ligament is completely yielded before the crack starts to grow, and all the data for each width can be used for extrapolation.

Figure 12 shows the load–displacement diagram for PE(LL0209AA), which indicates that the crack initiates at maximum load. Also, the maximum net section stress is equal to the uniaxial yield stress for all ligament lengths, as shown in *Figure 13*, which implies that all the ligaments are completely yielded. Therefore, the $\hat{L}$ used in *Figure 11* must be due to a factor other than the yielding requirement.

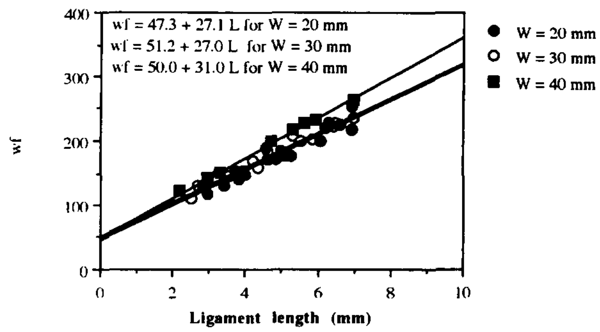

Figure 11 Plots of specific energy to fracture w_f (kJ m^{-2}) against ligament length L for PE(LL0209AA) SENT specimens (gauge length = 100 mm, W=20–40 mm, B=0.038 mm; L<7 mm)

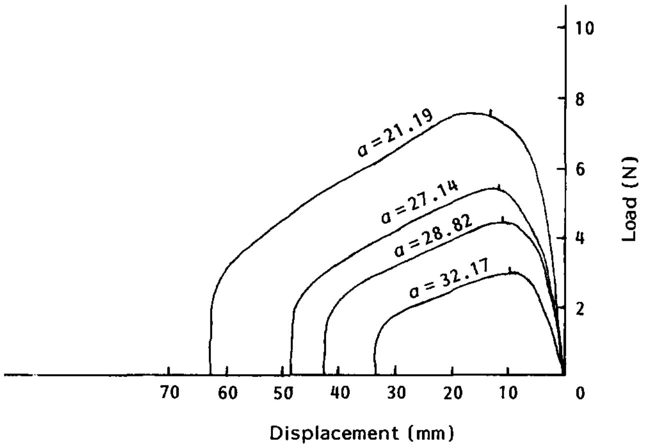

Figure 12 Load–displacement plot for PE(LL0209AA) (w=40 mm)

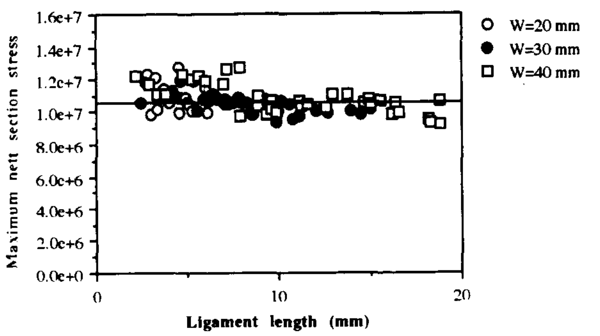

Figure 13 Maximum net section stress (Pa) *versus* ligament length for PE(LL0209AA) SENT specimens

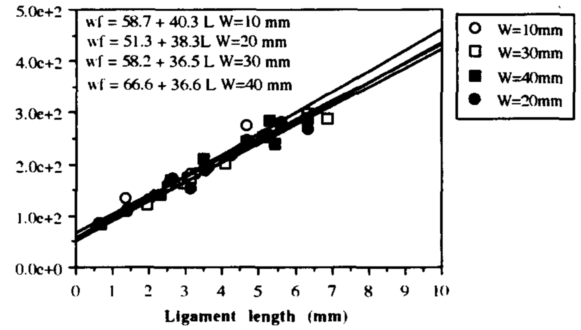

Figure 14 Specific energy to fracture w_f ($kJ\,m^{-2}$) *versus* ligament length L for PE(LL7909AA) SENT specimens (gauge length = 100 mm, W = 10–40 mm, B = 0.038 mm)

One plausible explanation for the variations in w_e with W is distortion of the load–displacement results by out of plane deformation, which occurs at random and is more significant at small crack lengths. This is confirmed by the scattering of data at larger ligament lengths, as shown in *Figure 10*. The reason for not using a guide to suppress out of plane deformation was given earlier. Apparently, the load–displacement results will be distorted whether or not a guide is used. It is suggested that a suitable lubricant be used to alleviate the frictional force between the guide and the film surface, thus minimizing the distortion to the load–displacement results when guides are used.

The essential work method was also applied to study the variations in w_e with specimen width for PE(LL7909AA). Since the load–displacement results will be distorted whether or not a guide is used, it was decided not to use a guide and scattering in the data was allowed for when interpreting the results.

After accounting for scattering, *Figure 14* shows that specimen width has no effect on the specific essential work of fracture for SENT specimens

$$w_f = 58.7 + 40.3L \quad \text{for } W = 10\,\text{mm}$$

$$w_f = 51.3 + 38.3L \quad \text{for } W = 20\,\text{mm}$$

$$w_f = 58.2 + 36.5L \quad \text{for } W = 30\,\text{mm}$$

$$w_f = 66.6 + 36.6L \quad \text{for } W = 40\,\text{mm}$$

The maximum ligament length for extrapolation $\hat{L}$, calculated using a w_e of 60 $kJ\,m^{-2}$, is 39.0 mm. Therefore, the ligament is completely yielded before the crack advances.

The w_e for PE(HD6070FY) was obtained by testing SENT specimens of different ligament lengths L. As shown in *Figure 15*, w_e is 61 $kJ\,m^{-2}$ and the limit for $\hat{L}$ is 40.3 mm. Therefore, the w_e is valid and is the fracture toughness of the material. From the figure it can be seen that scattering of the data is more significant at larger L. However, note also that the extrapolation is not very sensitive to a reduction in $\hat{L}$.

Kaptons (VN200 and HN200)

The specimen thickness, width and gauge length for both kaptons were 0.05 mm, 20 mm and 100 mm, respectively.

Figure 16 shows that the w_e of kapton VN200 for both SENT and DENT specimens are the same, and the specific non-essential work of fracture $w_p\beta$ for the SENT specimens is greater than that for the DENT specimens

$$w_f = 39.5 + 3.7L \quad \text{for DENT}$$

$$w_f = 40.4 + 1.9L \quad \text{for SENT}$$

The calculated maximum ligament length for extrapolation $\hat{L}$ for this material is 16.0 mm, and this indicates that the w_e obtained from the test is valid.

The effects of the test speed on both w_e and $w_p\beta$ are minimal, and these results are illustrated in *Figure 17*, where

$$w_f = 39.4 + 1.7L \quad \text{for } 2\,\text{mm min}^{-1}$$

$$w_f = 43.0 + 1.9L \quad \text{for } 5\,\text{mm min}^{-1}$$

$$w_f = 40.2 + 1.2L \quad \text{for } 10\,\text{mm min}^{-1}$$

$$w_f = 41.4 + 2.1L \quad \text{for } 20\,\text{mm min}^{-1}$$

Figure 18 shows that the w_e of kapton HN200 is also

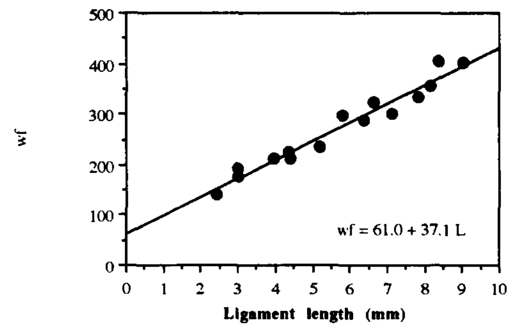

Figure 15 Specific energy to fracture w_f ($kJ\,m^{-2}$) *versus* ligament length L for PE(HD6070FY) SENT specimens (gauge length = 100 mm, W = 20 mm, B = 0.035 mm)

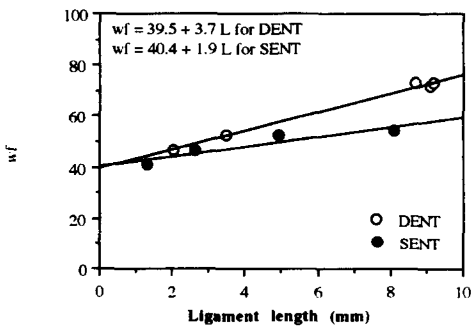

Figure 16 Comparison of the specific essential work ($kJ\,m^{-2}$) results for kapton VN200 SENT and DENT specimens (gauge length = 100 mm, W = 30 mm, B = 0.05 mm)

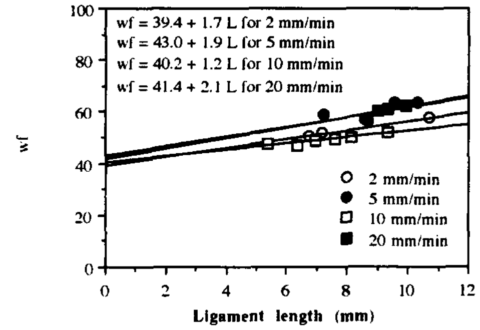

Figure 17 Plots of specific energy to fracture w_f (kJ m^{-2}) against ligament length L at different strain rates for kapton VN200 SENT specimens (gauge length = 100 mm, W = 20 mm, B = 0.05 mm)

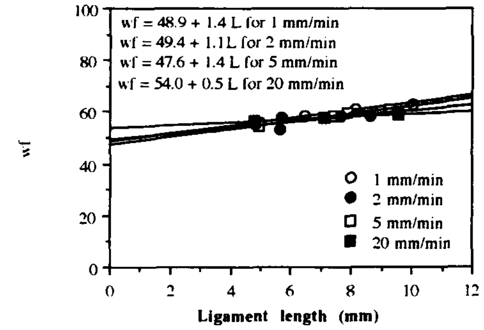

Figure 18 Plots of specific energy to fracture w_f (kJ m^{-2}) against ligament length L at different strain rates for kapton HN200 SENT specimens (gauge length = 100 mm, W = 20 mm, B = 0.05 mm)

insensitive to test speed, where

$$w_f = 48.9 + 1.4L \quad \text{for} \quad 1\,\text{mm min}^{-1}$$
$$w_f = 49.4 + 1.1L \quad \text{for} \quad 2\,\text{mm min}^{-1}$$
$$w_f = 47.6 + 1.4L \quad \text{for} \quad 5\,\text{mm min}^{-1}$$
$$w_f = 54.0 + 0.5L \quad \text{for} \quad 20\,\text{mm min}^{-1}$$

The calculated $\hat{L}$ for this material is 25.4 mm, and this indicates that the w_e obtained from the test is valid.

Poly(ethylene terephthalate)

An additional specimen geometry, deep centre-notched tension (DCNT), was used to find the w_e of this material. The gauge length and thickness were 100 mm and 0.18 mm, respectively. The width of the DENT and DCNT specimens was 40 mm, while that of the SENT specimens was 20 mm.

Figure 19 shows that the three specimen geometries give approximately the same w_e

$$w_f = 53.6 + 12.3L \quad \text{for DENT}$$
$$w_f = 56.2 + 12.7L \quad \text{for DCNT}$$
$$w_f = 51.7 + 29.3L \quad \text{for SENT}$$

and the calculated $\hat{L}$ is 24.3 mm, which indicates that the w_e values obtained are valid.

CONCLUSION

It has been shown that the essential work method can

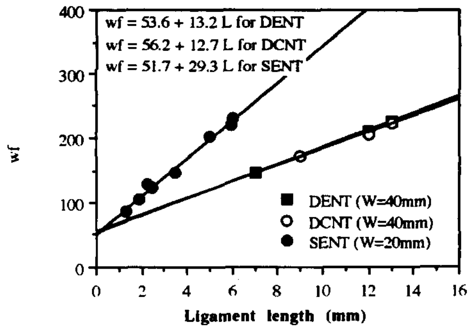

Figure 19 Comparison of the specific essential work (kJ m^{-2}) results from SENT, DENT and DCNT specimens of cast, amorphous PET (gauge length = 100 mm, B = 0.18 mm)

be used to obtain the fracture toughness of polymeric films. The validity of the specific essential work of fracture w_e obtained from testing was established by satisfying the criterion that the maximum ligament length used for extrapolation $\hat{L}$ should be less than $Ew_e/\pi\sigma_y^2$. This was accomplished by imposing an upper limit on $\hat{L}$ It has also been shown that, after allowing for experimental scattering, w_e is not sensitive to specimen width or test speed for the materials and ranges tested.

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