Effect of test conditions on the essential work of fracture in polyethylene terephthalate film

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The tear resistance of polyethylene terephthalate film is characterized by the essential work of fracture method in mode I as a function of test speed and temperature. Attempts to extrapolate tearing resistance found by the method of essential work to commercial slitting processes are discussed. Limitations of the essential work of fracture method with regards to specimen size are evaluated. Based on the findings modifications to the test protocol are suggested.

1. Introduction

The cutting or slitting behavior of polymer films has tremendous practical importance for processing materials into manageable shapes and quantities. Thus far, most research on cutting (slitting) films has focused on commercial process parameters: knife angle, film speed, film tension, blade sharpness, etc. [1–4]. Accurate reproduction of a commercial process in the laboratory setting is cumbersome, and a combinatorial approach to finding the best process variables is time consuming. Although optimization of commercial process variables has great value to industry, it does not directly lead to an improved scientific understanding of the mechanisms that control a film cutting process, or how their characteristics are, in turn, related to the tear resistance of a film.

In the lab the common methods used to study tear in polymers include: a non-linear fracture mechanics approach (the "J-integral"), ASTM standards (D1004 and D1922), and the "essential work of fracture" (EWF) method [5–8]. In the case of films, the J-integral approach is not as useful since it is best suited for a plane strain condition and therefore underestimates the crack propagation energy under plane stress. ASTM standard D1004 is designed to look at the energy required to initiate a tear, but not to propagate a preexisting one and therefore is not applicable to commercial slitting. ASTM standard D1922 is designed to measure the energy required to propagate a tear but it is well known that the results from the experiments do not always correlate well with behavior in commercial processes. The essential work of fracture method has been demonstrated to be applicable on a variety of films under different conditions in plane stress.

This work examines the effect of test speed and temperature on the essential work of fracture tearing experiments of PET film. Correlations of the data with that from literature are considered. In addition, limitations of the method of essential work (MEW) were evaluated with regard to specimen size.

2. Theory

The essential work of fracture method is based on Cotterell and Reddel's application of an idea suggested by Broberg [9, 10]. For a tensile experiment of a precracked specimen, the total energy measured, W_t, can be separated into its essential work, W_e, and inessential work, W_i components:

$$W_t = W_e + W_i \quad (1)$$

The essential work is defined as the work in the fracture process zone, directly in front of the crack tip, that causes crack propagation. The essential work is assumed to be a material property for a given film thickness. The inessential work, or the plastic work, is the energy dissipated in the yielding processes over the entire plastic zone and is dependent on the geometry tested. The specific work of fracture can then be examined by normalizing the energies by their respective areas (using the ligament length, l, and thickness, t, variables). In the case of the inessential work, an additional parameter β is introduced as a shape factor related to the form of the plastic zone. The result is the following equation:

$$w_f = W_f/lt = w_e + \beta w_i l \quad (2)$$

In Equation 2 the specific essential work of fracture (per unit area), w_e, and the specific inessential work (per unit volume), βw_i, are found by plotting the specific work of fracture (w_f) versus ligament length.

There are requirements on the ligament sizes that may be used. The ligament must be at least larger than 3–5 times the thickness of the sample to ensure the test is performed in plane stress. Additionally, the ligament must be smaller than one-third the overall width of the sample, ω, and smaller than the plastic zone size, $2r_p$ to ensure that the ligament has fully yielded prior to crack propagation. The plastic zone is typically approximated by:

$$2r_p = (\pi E w_e)/8\sigma_y^2 \quad (3)$$

where E and σ_y are the modulus and yield stress, respectively, found in a traditional tensile test. Therefore, the overall limitations on the ligament, l, are as follows:

$$3 - 5t < l < (\omega/3 \text{ or } 2r_p) \tag{4}$$

3. Experimental procedures

3.1. Materials

Biaxially oriented polyethylene terephthalate (PET) film was purchased from McMasterCarr in 25 foot rolls and used as received. The film is 0.1 mm thick and has a tensile strength of 19.3 MPa.

3.2. Tear experiments

Double edge notched tensile (DENT) specimens 22, 45, 110 or 200 mm wide by 50, 100, 250, or 450 mm long were tested in mode *I* using an Instron™ 5800. Samples were notched with a razor blade to achieve ligaments ranging in size from 10 to 50% of their width. DENT samples were mounted in *T*-shaped grips to accommodate their width. ASTM D638 type *I* tensile bars were die-cut from the film to determine tensile properties. Samples were tested at speeds of 1, 10, or 100 mm/min. Samples were also tested over a range of temperatures in a Thermcraft™ furnace at −20, 0, 20 or 40°C. The furnace and grips were equilibrated for one hour prior to sample introduction. After each sample was mounted in the grips, it was equilibrated with the furnace temperature for at least 15 min before testing. A minimum of 10 samples was run for each set of DENT experiments.

Images of the DENT specimens were taken *In-situ* using a Panasonic™ CCD camera. Images were taken with crossed polarizers to view birefringence patterns. Additional images were used to determine strains from a grid stamped onto the film.

4. Results and discussion

Figs 1a–c and 2a–d are plots of specific work of fracture versus ligament length for varying temperatures and rates. Figs 1a-c document the effect of temperature on specific work of fracture at constant rates. Figs 2a–d contain the effect of rate on specific work of fracture at a given temperature. All relevant data used to plot Figs 1a–c and 2a–d are given in Table I.

The experiments presented by Figs 1a–c were performed at constant speeds of 1, 10 or 100 mm/min, respectively, with temperature varying from −20 to 40°C. In Fig. 1a, for a rate of 1 mm/min, the essential work, component, w_e, is shown to increase with temperature. The inessential parameter, βw_i, also increases with temperature. For experiments with a constant rate of 10 mm/min, shown in Fig. 1b, the essential work increases from −20 to 0°C, and then decreases significantly at 20°C. It is then followed by another increase at 40°C. The inessential parameter increases with temperature. In Fig. 1c, for a test speed of 100 mm/min, the data does not show any significant changes in the essential work component. However, the inessential work component does increase with temperature, but not as dramatically as the samples tested at the lower rates.

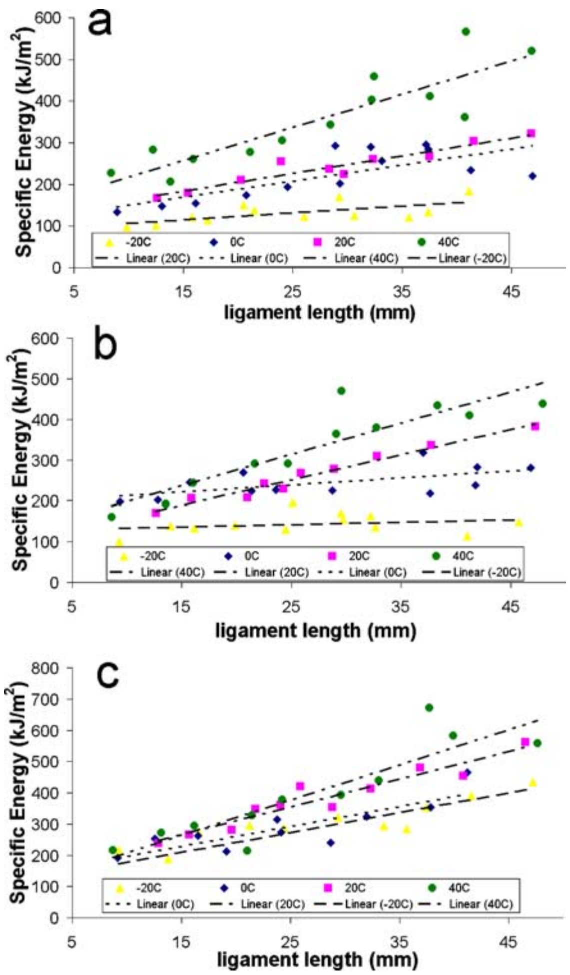

Figure 1 (a)–(c). Specific Energy vs. Ligament Length for constant test speeds of 1, 10, or 100 mm/min, respectively, and varying temperature.

Figs 2a–d show results from constant temperatures of −20, 0, 20 or 40°C with test speeds of 1, 10 and 100 mm/min. For the experiment performed at −20°C, Fig. 2a, there is no consistent trend in either the essential or inessential parameters. The essential work increases from 1 to 10 mm/min and then decreases from 10 to 100 mm/min. The inessential work fluctuates in an opposite manner with a decrease from 1 to 10 mm/min followed by an increase from 10 to 100 mm/min. In Fig. 2b, the 0°C-condition, again, no consistent behavior is found. The essential component increases from 1 to 10 mm/min and is followed by a decrease from 10 to 100 mm/min. The inessential components fluctuate inversely to the essential components. In Fig. 2c, the 20°C experiments, the essential component decreases from 1 to 10 mm/min and then increases from 10 to 100 mm/min. The inessential component, however,

TABLE I Essential and inessential work of fracture parameters

	−20°C	0°C	20°C	40°C
	Essential work of fracture w_e (kJ/m^2)			
1 mm/min	85.90	110.62	119.88	137.66
10 mm/min	128.13	189.59	95.21	113.27
100 mm/min	114.89	132.35	133.02	137.05
	Inessential work of fracture βw_i (kJ/m^3)			
1 mm/min	1.63	3.30	4.28	7.21
10 mm/min	.59	1.59	6.23	7.56
100 mm/min	6.34	6.38	8.90	9.51

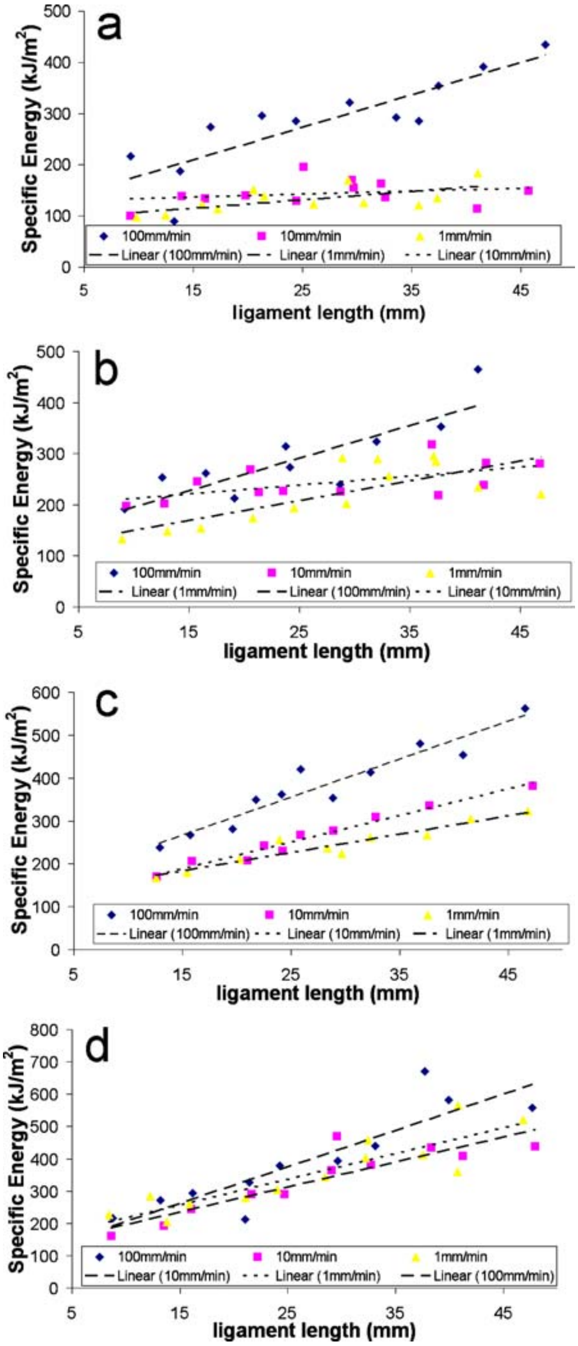

Figure 2 (a)–(d). Specific Energy vs. Ligament Length for constant temperature of −20, 0, 20, or 40°C, respectively, and varying test speed.

increases with rate. For the experiments conducted at 40°C, shown in Fig. 2d, the values for both the essential and inessential components are fairly constant, with a slight decrease at a rate of 10 mm/min in the essential component and a slight increase with rate in the inessential component.

Contrary to other viscoelastic polymer properties such as modulus and yield stress, the data shown in Figs 1 and 2 does not follow a typical time-temperature response. To determine whether the data's inconsistencies were associated with the MEW treatment or the material used in the experiments, uniaxial tensile experiments were performed on PET film. These experiments were conducted at the same conditions as those for the MEW studies, varying rate (1, 10, 100 mm/min) and temperature (−20, 0, 20, 40°C). Table II summarizes the tensile test results of the PET film. This clearly

TABLE II Uniaxial tensile response of PET film

	−20°C	0°C	20°C	40°C
		Yield stress (MPa)		
1 mm/min	100	84	77	60
10 mm/min	100	87	83	70
100 mm/min	120	107	90	80
		Yield strain (mm/mm)		
1 mm/min	0.05	0.05	0.06	0.03
10 mm/min	0.05	0.05	0.06	0.05
100 mm/min	0.05	0.05	0.06	0.05
		Modulus (GPa)		
1 mm/min	2.21	2.02	2.1	2.06
10 mm/min	2.12	2.02	2.1	1.93
100 mm/min	2.5	2.2	2.1	1.96

shows typical temperature and rate dependence with the yield stress increasing with increasing rate and decreasing with increasing temperature. The yield strain was found to be fairly invariant over the test conditions. After reviewing these results, it was determined that the anomalies in the MEW data in Figs 1 and 2 could not be related to the material.

The yield data in Table II was also plotted using an Eyring-type thermally activated model, as shown in Fig. 3 [11].

$$\frac{\tau_{yo}^{oct}}{T} = \frac{E}{T\upsilon} + \frac{2.303R}{\upsilon} \log \frac{\dot{\gamma}}{\Gamma} \quad (5)$$

where the octahedral yield stress (τ_{yo}^{oct}) is normalized by the test temperature (T) and plotted versus the log of the strain rate ($\dot{\gamma}$). R is the gas constant, E is the activation energy, υ is the activation volume, and Γ is a prefactor. The parallel lines in Fig. 3 allow the activation energy and volume to be determined from the slope and intercept. The activation energy and activation volume were found to be 139.3 kJ/mol and 5.66e-6 m^3/mol, respectively.

It is interesting to note that the uniaxial yield response of the film follows a typically thermally activated process, while the essential work of fracture does not. The interrelationships between a materials yield and fracture behavior is certainly more well-defined in brittle materials. In fact, the increase in a materials toughness is directly (and inversely) related to the materials yield stress. In this case, either the interrelationships between

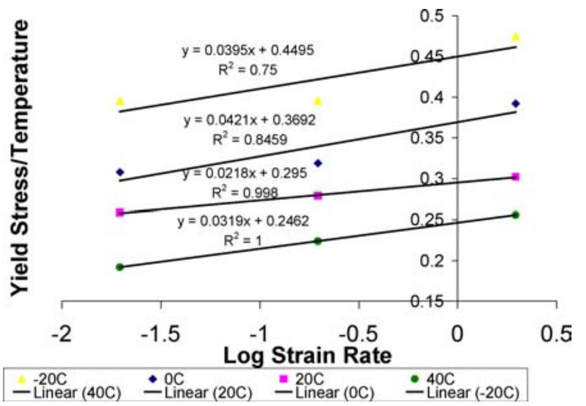

Figure 3 Yield stress normalized by temperature vs. logarithm of strain rate for the uniaxial tensile response of PET film.

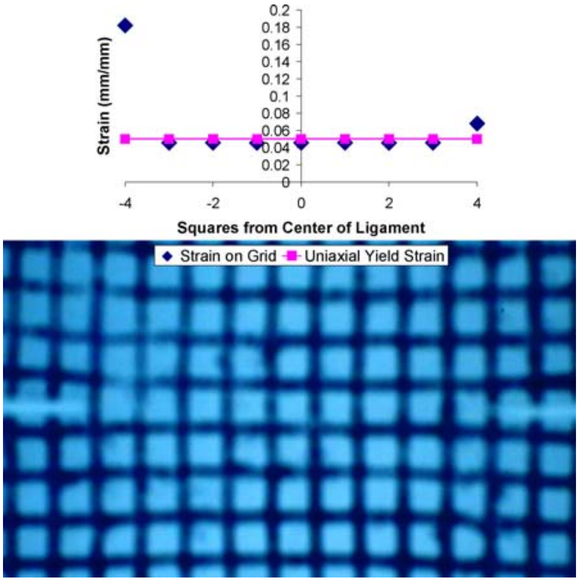

Figure 4 Image of the grid stamped on a 200 mm × 450 mm sample taken at maximum load. Plot of ε_{11} vs. distance to determine yielding in the image.

w_e and σ_y are more complex, or the assumptions made in evaluating w_e were not valid. Thus, the method of essential work was then carefully analyzed for validity. One requirement is a fully yielded ligament prior to crack propagation. To investigate if this requirement was met in the DENT experiments, the strains in the 11 and 22 directions were measured by image analysis of a grid stamped on the sample prior to loading. Fig. 4 is an image of a DENT specimen at maximum load prior to crack propagation. The strain in the 11 direction was found to be about at the uniaxial yield strain found earlier, approximately 0.05. The image analysis also showed a slight strain in the 22 direction, indicating biaxial load. For the level of accuracy of this experiment it is reasonable to assume that the ligaments are fully yielded prior to crack propagation. Additionally, due to the biaxial load, it would be expected that the yield strains measured in a uniaxial test would overestimate the actual yield strain in the DENT tests.

It is also interesting to note that the strains did not extend much beyond the yield point. A reason for this may be that the material undergoes substantial strain hardening immediately after yield. The uniaxial stress-strain curve, shown in Fig. 5, supports this hypothesis since strain hardening is observed immediately after yielding. Nonetheless, the analysis suggests that the material has yielded and therefore the requirement for ligament yielding prior to crack propagation has been met.

The findings presented herein do not correlate well with pre-existing literature. Wing-Mai *et al.* found no change in the essential or inessential parameters when testing PET at 20, 40 and 60°C at a constant speed of 1 mm/min [12]. Karger-Kocsis *et al.* have studied PET at rates of 1 and 20 mm/min, and separately 1, 10, and 100 mm/min at room temperature and found no significant changes in the essential work components [13, 14]. However, Karger-Kocsis *et al.* did find in both cases that the inessential component was rate sensitive. Additional authors have also studied temperature and rate effects for other materials including:

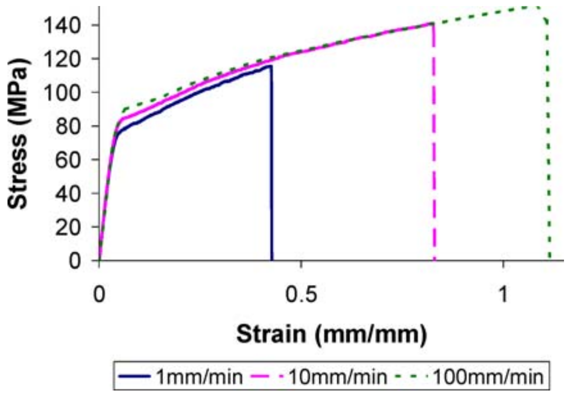

Figure 5 Stress-strain curves for uniaxial tensile specimens tested at 1, 10 or 100 mm/min.

polyimide, polyethylene, polybutylterephthalate, polycarbonate and polypropylene and have found that rate and temperature have little to no effect on the essential work component [15–18].

It was postulated that the discrepancies between the data presented here and the aforementioned studies in the literature was linked to differences in sample size. In the method, the only important aspect ratio is thought to be between the ligament and sample width. The overall size of the samples is not considered to have an influence on the method of essential work results. Most published data using this method was obtained from samples ranging in width from 30–50 mm. After the experiments were performed, it was noted that although the aspect ratio of width to length was similar to those in literature, our overall sample size was more than double that of others.

A new set of experiments was performed in which the MEW was applied to a range of sample sizes. The aspect ratios of width to length and ligament to width of all samples were kept the same and consistent with the literature. The size of the samples tested ranged from 22 to 250 mm wide and 50 to 450 mm long. All samples were tested at 20°C and 10 mm/min. Figs 6a–d are plots of the specific work of fracture versus ligament length for the four size samples. For each sample size, the curve produced in Fig. 6 is linear and would seem to indicate that the method is functioning correctly. However, by comparing the values of essential and inessential work contained in Table III for the different sizes, it is apparent that the values vary widely. For increasing sample size the essential work steadily increases from about 25 to 155 kJ/m^2 while the inessential work decreases from 12 to 4 kJ/m^3. These findings are surprising because they show that the essential work can be strongly influenced by the size of the sample used in the test.

In order to demonstrate the variations in the data, Fig. 7, a plot of the specific work of fracture versus lig-

TABLE III Essential and inessential work components for different sample sizes

Specimen size (mm)	w_e (kJ/m^2)	βw_p (kJ/m^3)
22 × 50	25.67	12.48
45 × 100	40.82	8.86
110 × 250	95.21	6.29
200 × 450	154.84	4.56

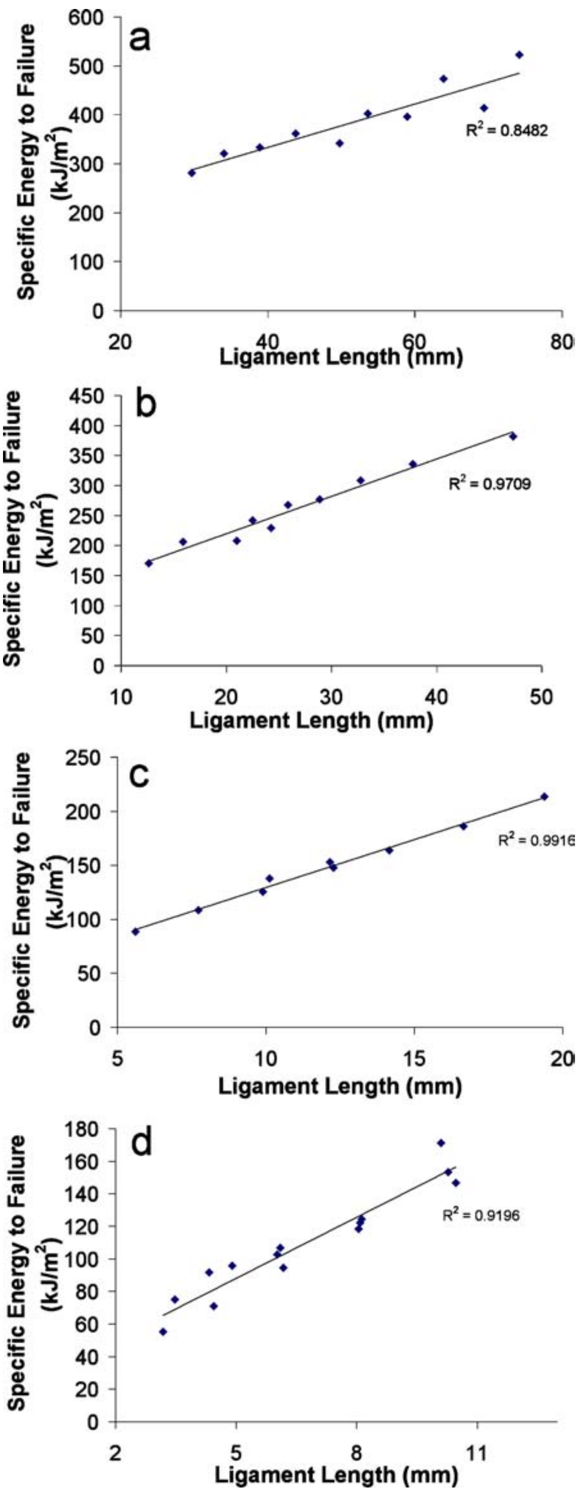

Figure 6 (a)–(d). Specific Energy to Fracture vs. Ligament Length for specimen sizes of: 200 × 450 mm, 110 × 250 mm, 45 × 100 mm, and 22 × 50 mm, respectively.

ament length, shows the data for all four sample sizes presented on one graph. The most common ligaments used in previous literature are labeled. It is clear from the curve that the ligament lengths tested in the literature are small enough such that major non-linear deviation is not present. Although each sample tested in this series of experiments satisfies the requirements of the method it is not possible to obtain consistent essential and inessential work values from samples with ligaments exceeding 20 mm. While there is currently no explicit requirement for the overall size or length of

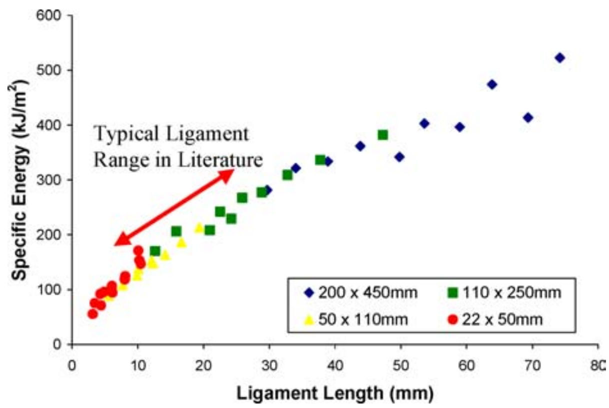

Figure 7 Specific Energy to Fracture vs. Ligament Length for all sample sizes tested.

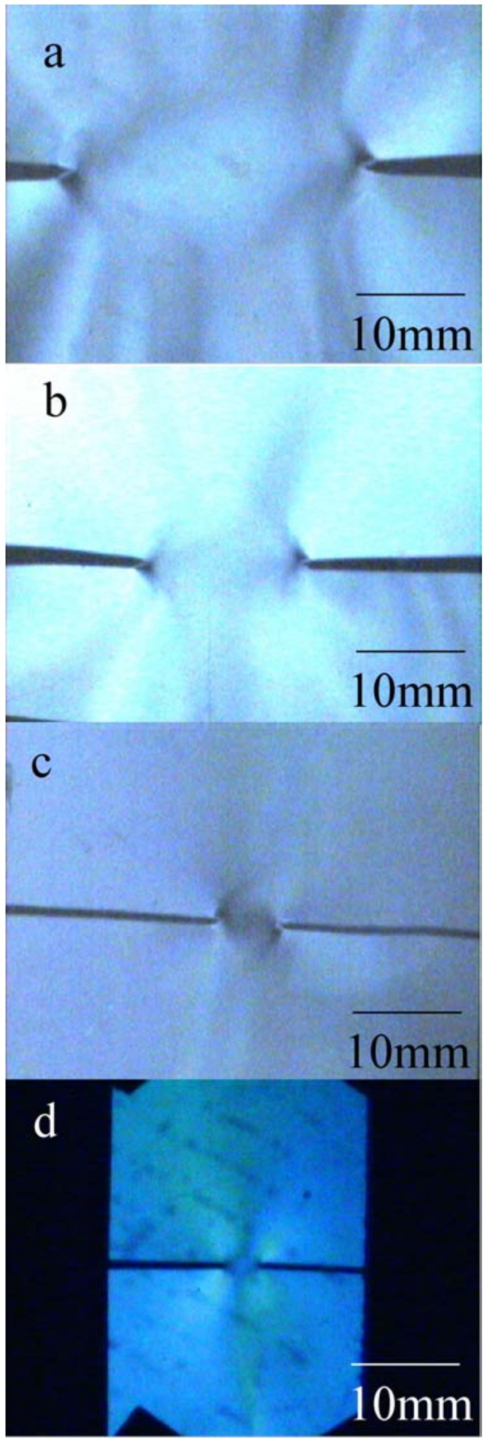

Figure 8

the samples, these experiments indicate that one may be needed. It is also important to note that this is not the first work to present variations with size. Although not addressed, the data published by Hashemi [19] contained non-linearity for both single-edge notched tensile (SENT) and DENT polyester film specimens at increasing ligaments. His experiments kept either a constant width to length ratio and increasing specimen size as found here or kept a constant length while varying the width.

One potential explanation for the non-linearity at increasing ligaments sizes relates to the amount of overlap in the region of influence in front of an approaching crack tip. It is proposed that for the method of essential work to generate reliable measurements in DENT specimens there must be full overlap of this region in front of the approaching crack tips. Figs 8a–d show birefringence images of the four sample sizes, taken with the same ligament to width ratio at maximum load. In the larger two specimens, Figs 8a–b, there is not complete overlap of the birefringence regions between the crack tips showing less effective crack interaction. The influence of a crack tip is a function of the crack length, geometry and loading condition. In all four samples the geometry and relative crack lengths are the same. It is also proposed that the radius of the region of influence in plane stress is defined by the crack tip radius, as opposed to the crack length. All cracks in the experiments previously outlined were made with similar razor blades therefore, it is reasonable to assume that all crack tips have the same radius. This result suggests that there may be further requirements needed for the ligament length when imposing MEW experiments. According to the hypothesis, that length would be determined by requirement of full interaction between the crack tips prior to crack propagation.

5. Conclusions

Mode *I* tearing experiments were performed on PET film with in-site imaging of the crack tip during the tests. The method of essential was applied to analyze the dependence of the essential and inessential work components on test speed and temperature. The dependence of these components on both speed and temperature was found to be inconsistent with typical viscoelastic behavior such as yield. To determine whether these differences were due to the method of essential work treatment or the PET film properties, uniaxial tensile experiments were performed. In the uniaxial tensile tests, the material did follow the typical response for time and temperature shown by an Eyring-type thermally activated yield plot. Therefore the anomalous dependence of the essential and inessential work components on test speed and temperature were attributed to the method of essential work treatment.

The conditions for applying the method of essential work were examined. The method required that yielding occur in the ligament prior to crack propagation. *In-situ* image analysis confirmed yielding of the ligament prior to crack propagation. It was shown that the specimens tested in this study had width-to-length ratios similar to those found in literature, but were more than double in overall size. A series of experiments with a range of sample sizes, which maintained the aspect ratios of width to length and ligament to width, was conducted to investigate the effect of overall sample size on the essential and inessential work components. Sample size was shown to affect both values. It was shown that for small ligament lengths the method of essential work produced consistent results. However, using ligament lengths larger than approximately 20 mm led to significantly different essential and inessential work values. Previous data found in the literature was typically generated using specimens that were small enough such that ligaments rarely exceeded 20 mm.

Currently, the method of essential work makes no requirements on the maximum size of a sample. However, as shown by these experiments, the ligament length and overall size do have an effect on measured results. Additional constraints on the specimen or ligament size may be needed in order to produce consistent test results when applying the method of essential work. It is proposed that this size constraint be based around the idea that full interaction between crack tips is needed for DENT specimens when the method of essential work is to be applied.

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References

1. D. BOLLEN, J. DENEIR, E. AERNOUDT and W. MUYLLE, *J. Mater. Sci.* **24** (1989) 2957.
2. C. ARCONA and T. A. DOW, *ibid.* **31** (1996) 1327.
3. R. R. MEEHAN and S. J. BURNS, *Experim. Mech.* **38**(2) (1998) 103.
4. R. R. MEEHAN, J. KUMAR, M. EARL, E. SVENSON and S. J. BURNS, *J. Mater. Sci. Lett.* **18** (1999) 93.
5. Y.-W. MAI, B. COTTERELL, R. HORLYCK and G. VIGNA, *Poly. Engng. Sci.* **27**(11) (1987) 804.
6. D1004, *Amer. Soc. Test. Mater.* **08.01**.
7. D1922, *Amer. Soc. Test. Mater.* **08.02**.
8. Y.-W. MAI and J. WU, *Poly. Engng. Sci.* **36**(18) (1996) 2275.
9. B. COTTERELL and J. K. REDDEL, *Intern. J. Fract.* **13** (1977) 267.
10. K. B. BROBERG, *J. Mech. Phys. Solids* **23** (1975) 215.
11. I. M. WARD and D. W. HADLEY, An Introduction to the Mechanical Properties of Solid Polymers (1993).
12. J. S. S. WONG, D. FERRER-BALAS, R. K. Y. LI, Y.-W. MAI, M. L. MASPOCH and H.-J. SUE, *Acta Mater.* **51** (2003) 4929.
13. J. KARGER-KOCSIS, T. CZIGANY and E.J. MOSKALA, *Polymer* **39**(17) (1998) 3939.
14. J. KARGER-KOCSIS and T. CZIGANY, *ibid.* **37**(12) (1996) 2433.
15. S. HASHEMI, *J. Mater. Sci.* **35** (2000) 5851.
16. S. HASHEMI, *ibid.* **32** (1997) 1563.
17. W. Y. F. CHAN and J. G. WILLIAMS, *Polymer* **35** (1994) 1666.
18. D. FERRER-BALAS, M. L. MASPOCH, A. B. MARTINEZ, E. CHING, R. K. Y. LI, and Y.-W. MAI, *ibid.* **42** (2001) 2665.
19. S. HASHEMI, *Plast. Rubb. Comp. Process. Appl.* **20** (1993) 229.

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