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Thickness dependence of work of fracture parameters of an amorphous copolyester

József Karger-Kocsis*
Institut für Verbundwerkstoffe GmbH, Universität Kaiserslautern, Pf. 3049, D-67653 Kaiserslautern, Germany

and Tibor Czigány
Institute of Machine Design, Faculty of Mechanical Engineering, Technical University of Budapest, H-1111 Budapest, Hungary

and Eric J. Moskala
Eastman Chemical Co., Research Laboratories, PO Box 1972, Kingsport, TN 37662-5150, USA
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The fracture toughness of an amorphous copolyester (COP) of different sheet thickness (0.5, 3 and 6 mm) was determined by the essential work of fracture (EWF) concept using tensile-loaded deeply double-edge notched (DDEN-T) specimens. It was shown that this COP meets the basic requirement of the EWF concept, viz. full ligament yielding (marked by a load drop in the load–displacement curve) prior to the crack growth, in the thickness range studied. Based on the well-resolved yielding both the specific essential (w_e) and non-essential work of fracture (w_p) were split in the contributing terms related to yielding (w_I), and necking and fracture (w_{II}). It was found that w_e is likely to be independent on the thickness range when plane stress conditions prevail, and thus represents a material parameter. This finding is at odds with previous results suggesting that w_e is thickness dependent. The development and size of the plastic zone were studied by light microscopy and infra-red thermography. The latter technique overestimated the plastic zone in thicker sheets by not differentiating between pure and diffuse yielding. © 1997 Elsevier Science Ltd.

(Keywords: amorphous copolyester; essential work; fracture mechanics)

INTRODUCTION

The assessment of fracture toughness of ductile polymers by concepts of the nonlinear or plastic fracture mechanics is currently of great interest. The most widely used approaches of the latter are the *J*-integral, proposed by Eshelby and Rice (refs 1 and 2, and refs within) and the essential work of fracture (EWF) theory, credited to Broberg[2–4].

The EWF concept differentiates between the essential (W_e, required to fracture the polymer in its process zone) and non-essential or plastic work (w_p, consumed by various deformation mechanisms in the plastic zone; see *Figure 1*). The total work of fracture (W_f) is composed of the two above terms as follows:

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

Taking into consideration that W_e is surface related, whereas W_p is volume related, W_f can be given by the related specific work terms (i.e. w_e and w_p, respectively):

$$W_f = w_e \cdot lt + \beta w_p l^2 \cdot t \qquad (2)$$

$$w_f = \frac{W_f}{lt} = w_e + \beta w_p l \qquad (3)$$

where l is the ligament length, t is the thickness of the specimen and β is a shape factor related to the form of the plastic zone. The beauty of this approach is that according to equation (3) the specific essential work of fracture (w_e) can be easily determined by reading the ordinate intercept of the linear plot w_f vs l. Recall that by using the multiple specimen technique for determination of the critical value of the *J*-integral (J_{Ic}) it is essential to assess the crack advance[1,5] which is very often not a simple task.

Results published on w_e do not confirm uniequivocally that w_e is a material parameter. The fact that w_e is independent of specimen geometry, reported by several groups[2,6–8], is a necessary but not a sufficient prerequisite that w_e is a material parameter. Vu-Khanh[9] found recently that w_e depends on the specimen geometry and concluded that no clear distinction between the process and plastic zone can be made, so that the toughness cannot be characterized by a single material parameter. In order to show how controversial this issue is, the authors refer to the w_e data of Hashemi[7,10], showing that w_e is also thickness dependent in a film thickness range where plane stress conditions should always be accommodated. How can this discrepancy be explained? Can w_e be considered as a material parameter? It is not obvious why the extrapolation of w_f vs l data pairs, achieved on specimens of different thickness, should give

* To whom correspondence should be addressed

various intercepts (i.e. w_e values)[6,11], when the specimens fracture in plane stress. The authors believe that the aforementioned discrepancies are due to the fact that the most crucial precondition of the EWF approach, viz. full ligament yielding before the crack advances, was not met in the earlier works. It was shown[12,13] that this precondition can be met by using amorphous copolyesters of high ductility.

The objective of this study was to investigate the thickness effect on the EWF response of an amorphous copolyester (COP), that fully yields prior to crack growth. A further aim of this work was to establish whether or not the plastic work (w_p) and the related shape factor (cf. equation (3)) depend on the specimen thickness as suggested by Saleemi and Nairn[14].

EXPERIMENTAL

Materials

COP sheets with various thickness (*ca.* 0.5, 3 and 6 mm) were supplied by Eastman (Kingsport, TN). This COP was synthesized from dimethyl terephthalate (DMT) and two diols: ethylene glycol (EG) and 1,4-cyclohexane dimethanol (CHDM). The content of the latter monomer was 68 mol%. The inherent viscosity ($IV = 0.705\,dl\,g^{-1}$) of this COP was determined in a solution (0.5 g per 100 ml) of a 60/40 phenol/tetrachloroethane mixture at 25°C. *Figure 2* shows the results of dynamic mechanical thermal analysis (d.m.t.a.) taken under sinusoidal tensile loading at 10 Hz frequency at a heating rate of $0.5°C\,min^{-1}$ by using an Eplexor 150N device (Gabo Qualimeter, Ahlden, Germany). The d.m.t.a. spectrum in form of dynamic storage modulus (E') and mechanical loss factor (tgδ) as a function of temperature (T) shows two distinct relaxation peaks: the α-relaxation or glass transition temperature (T_g) is at about 100°C, whereas the less pronounced β-relaxation at $T = -50°C$. A peculiar feature of this COP is to undergo strain-induced crystallization, being favoured by the strain-controlled d.m.t.a. measurement in this case. The crystallization-induced 'hardening' can well be resolved in *Figure 2* beyond $T = 140°C$.

Mechanical testing

All mechanical tests reported here were performed at room temperature (*RT*) and $v = 1\,mm\,min^{-1}$ crosshead speed on a Zwick 1445 universal testing machine. Tensile *E*-modulus (E), yield strength (σ_y), elongation at yield (ϵ_y), ultimate tensile strength (σ_b) and ultimate tensile elongation (ϵ_b) were determined by using dumbbell specimens (No. 3 according to DIN 53 455) and the results are listed in *Table 1*. For the direct determination of the specific plastic work (w_p) the same tensile specimen was used. w_p was computed by dividing the total fracture work (beneath the related force–elongation curve) with the volume of the fully yielded region of the dumbbell[15].

For the EWF study double deeply edge-notched tensile (DDEN-T) specimens with a width of 35 and overall length of 100 mm (clamped length 70 mm; *cf Figure 1*) were selected. The free ligament length (l) was set in the range from 5 to 20 mm. At every ligament at least three specimens were investigated. Data reduction (cf. equations (2) and (3)) followed the recommendations of the ESIS TC-4 group[15].

The non-essential or plastic work (w_p) was also derived indirectly by assessing the shape of the plastic zone (cf. *Figure 1*) using light microscopy (LM) and infra-red thermography (IT, thermocamera from Hughes, Portland, OR)[12,13,16]. Viewing of the plastic zone by LM occurred after fracture of the DDEN-T specimens. On the other hand, IT frames were taken continuously by a videotape during loading of the specimens. IT served to map the relative temperature rise in the ligament region which was assigned to the shape of the plastic zone.

The failure of the specimens was viewed in LM and scanning electron microscopy (SEM, Jeol 5400) after gold coating.

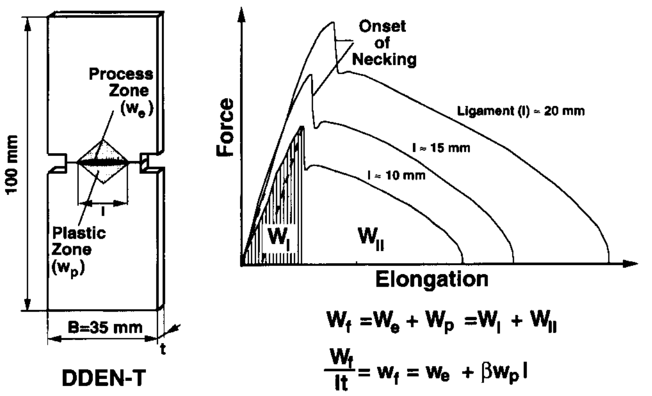


Figure 1 Designation and size of the specimens used along with F–x traces characteristic for COP

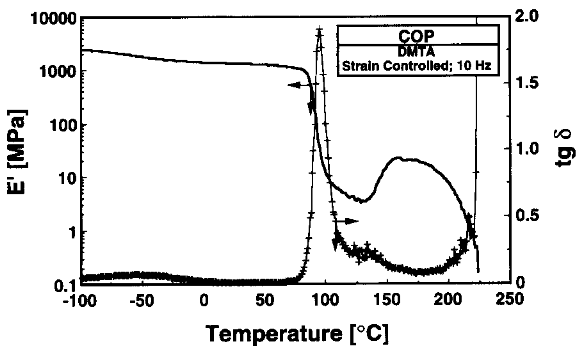


Figure 2 D.m.t.a. spectrum (E' and tgδ vs T) of the COP polymer

Table 1 Basic mechanical characteristics of the COP studied

Thickness (mm)	E modulus (GPa)	σ_y (MPa)	ϵ_y (%)	σ_b (MPa)	ϵ_b (%)	w_p ($MJ\,m^{-3}$)
0.54	1.98	44	2.9	58	≈190	≈80
3.07	1.85	48	3.5	58	≈230	≈104
6.05	1.70	47	3.9	57	≈235	≈105

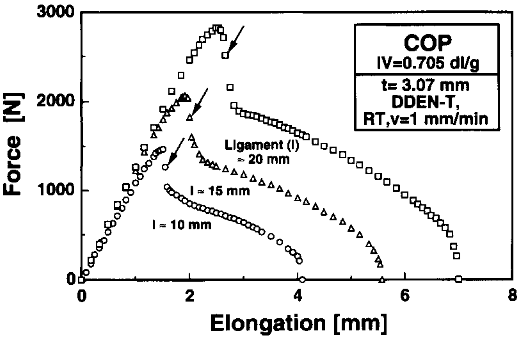
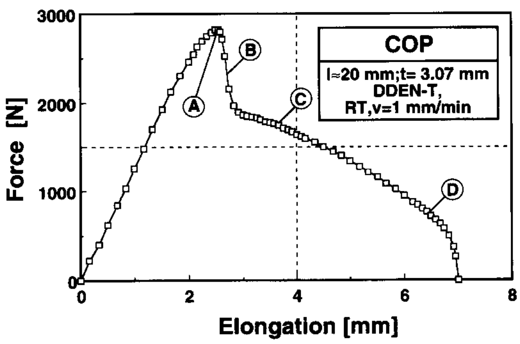


Figure 3 Comparison of the F–x curves of DDEN-T specimens at different ligament lengths for COP with a thickness of $t \approx 3$ mm



a

RESULTS AND DISCUSSION

Essential work of fracture

Figure 3 depicts the load–displacement (F–x) curves of DDENT-T specimens at various ligaments for the COP at $t \approx 3$ mm. It is very striking that the F–x curves at different ligament length are similar to one another. It should be borne in mind that this is the first evidence that the basic requirements of the EWF theory are met. Furthermore, the F–x curves exhibit a load drop (arrow indicates) which 'marks' where yielding is completed and the onset of necking takes place. At this point the whole ligament yields instantaneously. This is evidenced by IT pictures, as shown later. The full ligament yielding is followed by necking to the final fracture of the DDEN-T specimens.

IT frames taken during loading (cf. *Figures 4* and *5*) demonstrate, in fact, the full ligament yielding at the load drop. One can corroborate again that COP is the ideal polymer for the EWF test[12,13]. The temperature rises due to the instantaneous yielding up to about 7°C in the process zone of the specimens. The serial IT pictures in *Figures 4b* and *5b* show also the development of the plastic zone with the loading (the temperature in the ligament area can easily be determined by comparison with the scale of the probeye. The IT frames before final fracture of the DDENT specimens hint for the development of an elliptical plastic zone (cf. pictures D in *Figures*

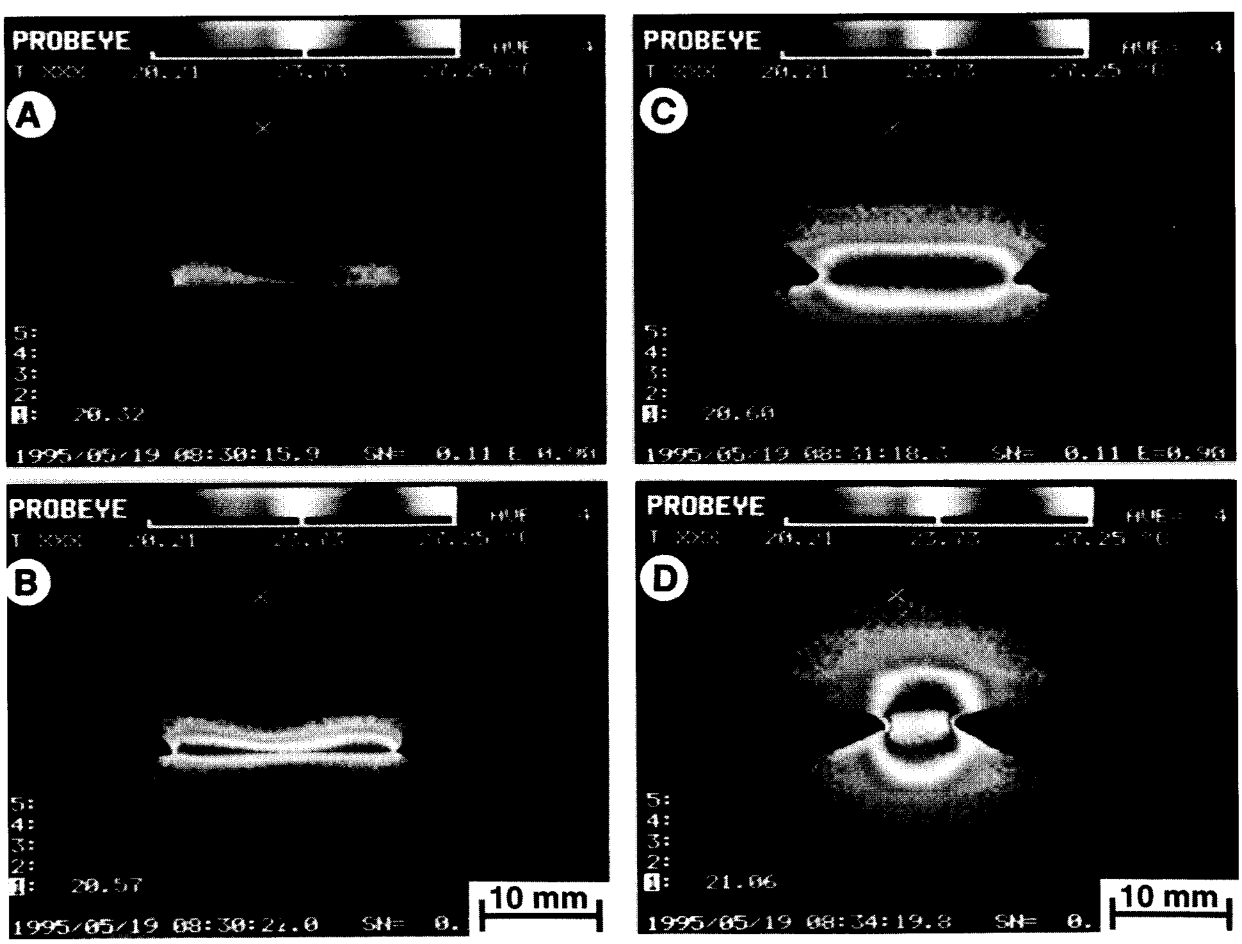


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Figure 4 F–x curve of a DDEN-T specimen of COP with $l = 20$ mm and $t = 3.07$ mm (a), along with the serial IT frames taken during loading (b). Note: taking position of the IT frames is indicated in the F–x curve in (a)

4b and *5b*). This was at odds with LM observations supporting the development of a shallow diamond-like plastic zone (*Figure 6*). This difference will be discussed later.

Figure 7 depicts the w_f vs l curves for the DDENT specimens of COP at various thickness. w_f was computed from the area beneath the F–x curves registered, as shown in *Figure 1*. According to equation (3), the essential work of fracture (w_e) was read from the intercept of the linear regression line with the ordinate and tabulated (*Table 2*). *Figure 7* shows that w_e is practically independent on the specimen thickness. This is the first experimental evidence that w_e does not depend on the thickness when the EWF concept is applied for the proper materials[12,13]. Considering the 95% confidence limits of the w_e data (recommended e.g. in ref. 15), which are listed in *Table 2*, the claim that w_e is thickness independent may not strictly hold. An open question in this respect is, however, whether or not the large confidence limits' range can be reduced by increasing the number of the specimens tested.

Furthermore, the finding that the slope of the regression lines, viz. βw_p, increases with the specimen thickness, seems to corroborate the suggestions of Saleemi and Nairn[14].

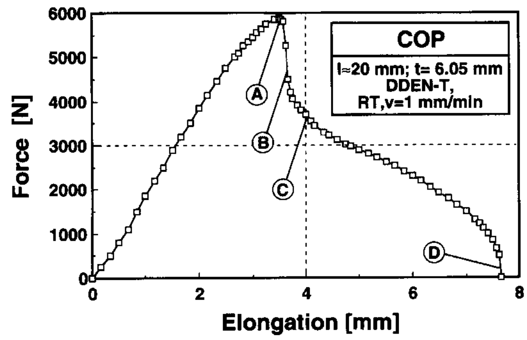
Before starting with further discussions on w_e, let us consider the usual size criteria of the EWF tests. The validity range of the EWF for the DDEN-T specimen configuration is generally given by refs 2, 7, 8, 12 and 14.

$$(3-5)t \leq l \leq \min\left(\frac{B}{3} \text{ or } 2r_p\right) \qquad (4)$$

where B is the width of the specimen (35 mm; cf. *Figure 1*) and $2r_p$ is the size of the plastic zone:

$$2r_p = \frac{1}{\pi} \cdot \frac{E w_e}{\sigma_y^2} \qquad (5)$$

The latter calculated by inserting the following average mechanical data: $E = 1.9\,\text{GPa}$, $w_e = 37\,\text{kJ}\,\text{m}^{-2}$ and $\sigma_y = 46\,\text{MPa}$, resulted in $2r_p \approx 11\,\text{mm}$. Though this size is very close to the alternative width criterion (i.e.



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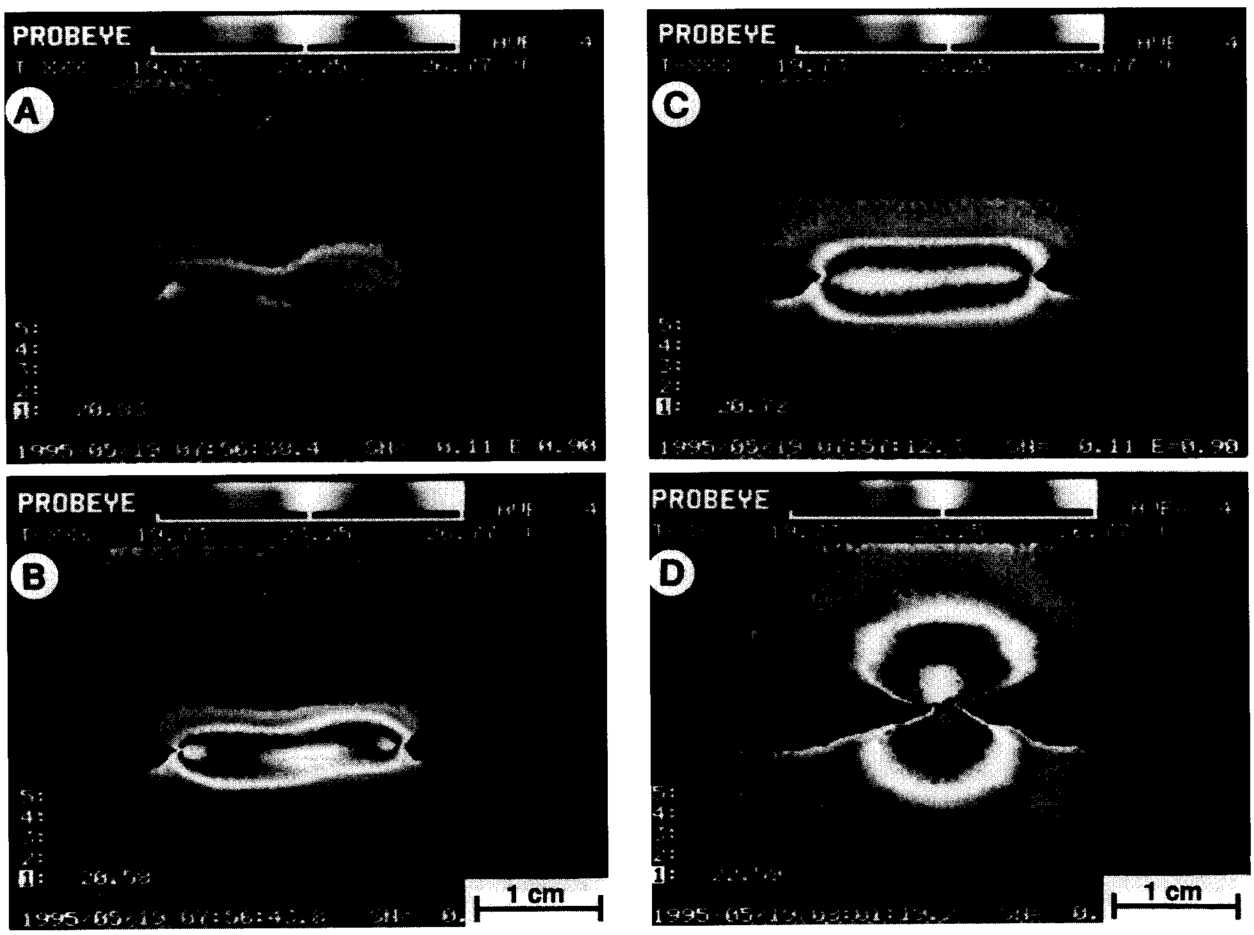

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Figure 5 F–x curve of a DDEN-T specimen of COP with $l = 20\,\text{mm}$ and $t = 6.05\,\text{mm}$ (a), along with the serial IT frames taken during loading (b). Note: taking position of the IT frames is indicated in the F–x curve in (a)

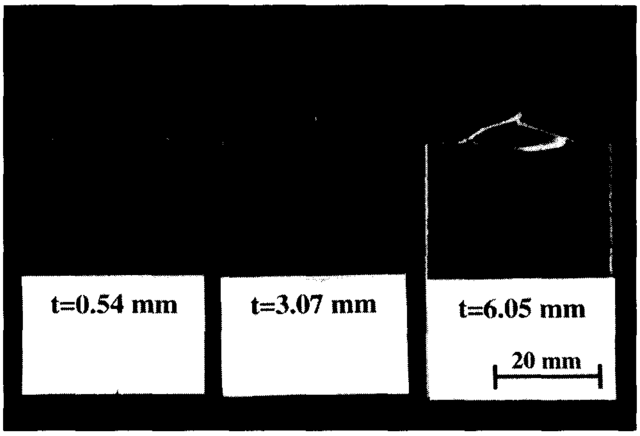

Figure 6 LM pictures taken on broken DDEN-T specimens of the same ligament length ($l \approx 15$ mm) at different sheet thickness

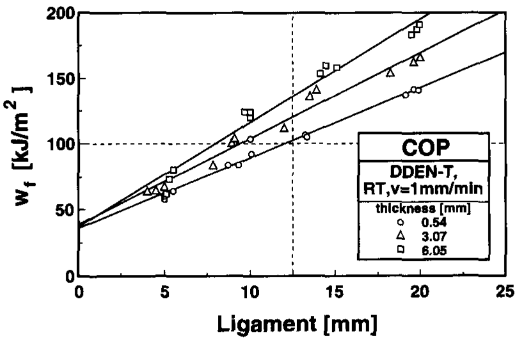

Figure 7 Total specific work of fracture (w_f) vs ligament length (l) for the COPS at various sheet thickness (t)

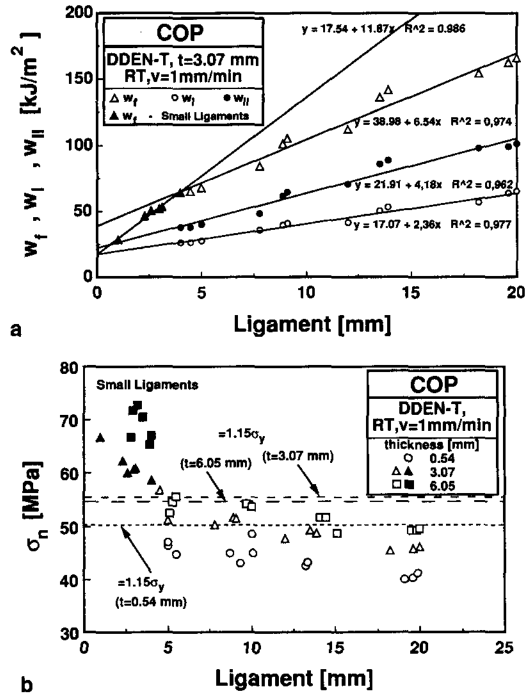

Figure 8 (a) Total specific work of fracture (w_f) and its contributing terms (w_I and w_{II}) vs ligament length (l) for the DDEN-T specimens of COP with $t \approx 3$ mm thickness. Note: this figure also contains data achieved on specimens with a ligament below the lower threshold value. (b) $\sigma_n - l$ curves of the DDEN-T specimens of various thickness. Notes: this figure contains also data achieved on specimens with a ligament below the lower threshold value. The $1.15\sigma_y$ values are 50.6, 55.2 and 54.1 for sheet thickness of 0.54, 3.07 and 6.05 mm, respectively

Table 2 Essential work (w_e), its contributing terms (w_{eI} and w_{eII}) and the related correlation coefficients (R^2) for the COP studied

Thickness (mm)	w_e with 95% confidence limits ($kJ\,m^{-2}$)	w_{eI} ($kJ\,m^{-2}$)	R^2	w_{eII} ($kJ\,m^{-2}$)	R^2
0.54	36.11 ± 9.50	14.52	0.949	21.59	0.974
3.07	38.98 ± 12.30	17.07	0.977	21.91	0.962
6.05	37.28 ± 15.90	26.59	0.934	10.69	0.981

$B/3 \approx 12$ mm), none of the data represents the upper ligament limit. Based on the self-similarity of the F–x curves in the ligament range up to $l = 20$ mm, one can claim that both above exclusion criteria are too conservative for this COP studied. The same comment holds also for the lower ligament threshold value. A transition from plane stress to plane strain could not be observed for the thinnest sheet. Limiting threshold $l \approx 4$ mm was found for the sheet thickness $t \approx 3$ mm (*Figure 8a*). An analogous low exclusion ligament (comparable with the thickness) could be concluded for the COP with $t \approx 6$ mm, as well.

Very useful information on the constraint effects is given by the plot of the nett section stress (σ_n, defined as F_{max}/lt, where F_{max} is the peak load) vs l, and the relationship of σ_n to σ_y[15]. The $\sigma_n - 1$ curve in *Figure 8b* shows that σ_n slightly decreases with increasing l, as expected. According to ref. 15 the σ_n values should lie below $\sigma_n = 1.15\sigma_y$ when plane stress is accommodated. *Figure 8b* corroborates that this essential prerequisite was met. A further very important aspect is that the constraint in specimens with short ligaments (designated by full symbols in *Figure 8b*), which were considered for the estimation of the plane strain w_e value (see later), exhibit higher σ_n values than the $1.15\sigma_y$ threshold.

Based on the load drop in the F–x curves of COP, it is possible to distinguish between the specific work of fracture required for yielding (w_I) and that for necking and subsequent fracture (w_{II}), cf. *Figure 1*. This is demonstrated by the example of specimens of ≈ 3 mm thickness (*Figure 8a*). By extrapolation of the w_f vs l data below the lower threshold ligament, the plane strain essential work of fracture ($w_{I,e}$) can be derived. Its value ($w_{I,e} \approx 17\,kJ\,m^{-2}$) agrees well with that of w_{eI} at $t = 3.07$ mm thickness (*Table 2*). Recall that w_{eI} data are very similar for the sheet thicknesses 0.5 and 3 mm (cf. *Table 2*). Further investigations are needed, however, to state whether or not w_{eI} is identical with $w_{I,e}$. It is worth noting that the splitting of the F–x curves as shown in *Figure 1* is not necessarily the right way, since the onset of crack initiation could also be assigned to F_{max}. In this case, the surface beyond the F–x curve (w_i, where i stands for initiation) is considerably lower than w_I, since the hypothetical deloading line is parallel to the initial slope of the F–x curve (cf. *Figure 1*). w_i should be independent of the ligament length[2,17], and it is supposed that its value agrees with w_{eI} (and thus possibly with $w_{I,e}$).

The reason why w_{eI} is much higher at $t \approx 6$ mm (cf. *Table 2*) is due to the fact that the F–x curves at this sheet thickness are not strictly self-similar. The relative proportion of w_I is higher at lower ligament lengths. This results in a peculiar and unexpected feature: w_f data lie higher at ligament lengths below the lower threshold value (*Figure 9*). This makes the determination of $w_{I,e}$ via extrapolation impossible. For this feature a change in the plastic flow constraint in the COP should be responsible, that makes the initial notch line curved (*Figure 10*).

The main outcome of this treatise on EWF is that the plane stress essential work of fracture is possibly thickness-independent and consists of two constituents which are related to yielding (w_{eI}) and necking including fracture (w_{eII}), respectively[12,13]. Preliminary results indicate that w_{eI} may be identical or closely matched to the plane strain essential work of fracture ($w_{I,e}$).

Non-essential (or plastic) work of fracture

w_p was determined by direct and indirect methods. In the former case the plastic work dissipated per unit volume in uniaxial tensile loaded dumbbells was considered (cf. *Table 1*[15]). Interestingly, this w_p value was lower for the thinnest sheet than for the thicker ones (*Table 1*), for which no explanation can be given by the authors. Recall that the slope of the w_f vs l lines is proportional with w_p (i.e. βw_p). The proportionality constant is the shape parameter (β) of the plastic zone.

The indirect estimation of β occurred by viewing the form of the necked region of the broken specimens by LM (cf. *Figure 6*). An alternative indirect way was to consider the IT heat maps taken during loading of the DDEN-T specimens[12,13,16]. The shape of the plastic zone was approached by a shallow diamond for which

$$\beta = \frac{1}{2}\frac{h}{l} \qquad (6)$$

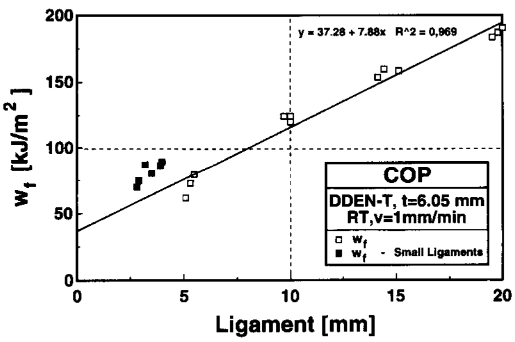

Figure 9 w_f vs l data for the DDEN-T specimens of COP with $t \approx 6$ mm thickness

Figure 10 Deformation of the notch plane (arrow indicates) in COP of $t \approx 6$ mm at a ligament laying below the lower threshold value ($l \approx 4$ mm)

holds[15], where h is the overall height of the plastic zone (cf. *Figure 1*). A very good agreement between the direct and indirect methods (i.e. LM and IT) was found for the shape parameter of the plastic zone of the thinnest sheet[13]. Furthermore, the agreement between the direct method and LM is good for the thicker sheets (*Figure 11*). However, the related β-values, derived by IT, lay considerably higher (*Table 3*). IT frames in *Figures 4b* and *5b* (cf. picture D in both cases) hint for an elliptical plastic zone rather than for a diamond-like one. The reason why IT overestimates β can be attributed to the fact that this technique cannot differentiate between pure and diffuse yielding. Diffuse yielding occurs at the border of the plastic zone toward the bulk, which can well be resolved by LM (cf. *Figure 6*). The effect of this diffuse yielding is negligible in such a highly ductile polymer as this COP, when w_p is determined by the direct tensile method. On the other hand, this diffuse yielding zone acts as a hot-spot, and thus affects the overall shape of the plastic zone during heat mapping considerably. Thus caution should be exercised when IT is applied for the determination of the plastic zone of thicker specimens.

Based on the results listed in *Table 3* it can be stated that β increases with sheet thickness, as suggested by Saleemi and Nairn[14].

Failure mode

Figure 2 shows that the COP used may undergo crystallization in the molten state (hot crystallization). Such polymers usually show strain-induced crystallization as well. It was found by differential scanning calorimetry d.s.c. that necking was not accompanied with strain-induced crystallization in the COP during the EWF test[13]. This phenomenon could also be excluded by Fourier transform infra-red investigations. Spectra taken from the bulk and necked region of the DDEN-T specimens did not indicate shifts in the absorption bands being related to conformational changes due to crystallization. The SEM pictures in *Figure 12* demonstrate the highly ductile failure of the DDEN-T specimens of the COP used.

CONCLUSIONS

The plane stress ductile fracture behaviour of an amorphous COP was studied by the EWF method using DDEN-T specimens cut from sheets of *ca* 0.5, 3 and 6 mm thickness. Based on this study the following

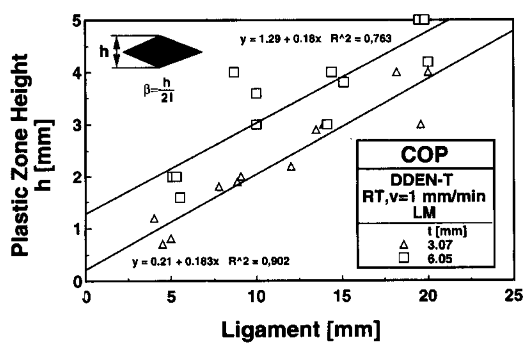

Figure 11 Total height of the plastic zone (h) determined by LM technique vs ligament length (l) for DDEN-T specimens of COP with $t \approx 3$ and 6 mm, respectively

Table 3 Non-essential or plastic work (w_p), its contributing terms (w_{pI} and w_{pII}) for the COP studied along with the shape parameter of the plastic zone (β) defined by different approaches

Thickness (mm)	βw_p ($MJ\,m^{-3}$)	βw_{pI} ($MJ\,m^{-3}$)	βw_{pII} ($MJ\,m^{-3}$)	Shape parameter β (tensile)	β (LM)	β (IT)
0.54	5.36	1.49	3.87	0.07	0.08	0.08
3.07	6.54	2.36	4.18	0.06	0.09	0.22
6.05	7.87	3.67	4.20	0.07	0.09	0.28

Note: the correlation coefficients related to w_p are indicated in *Table 2*

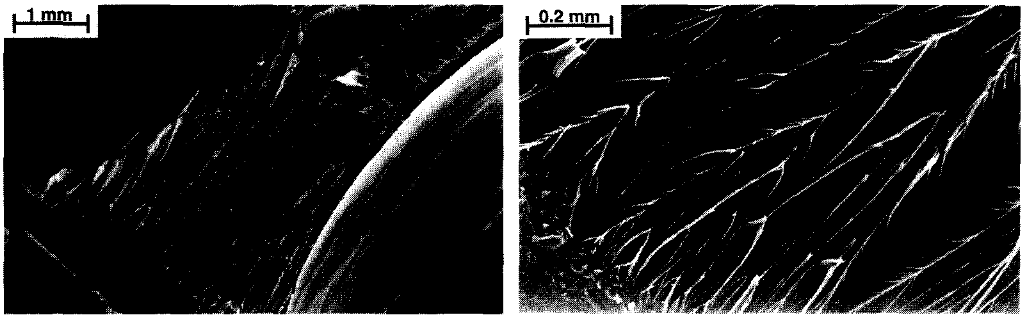

Figure 12 SEM pictures taken from the necked ligament area ($l \approx 10$ mm) of DDEN-T specimens of COP with $t \approx 6$ mm

conclusions may be drawn:

(i) The plane-stress specific essential work of fracture seems to be thickness-independent when the EWF approach is applied for an ideal polymer (undergoing full ligament yielding prior to the onset of crack growth), like the COP studied. Based on the load drop marking the sudden yielding in the load–displacement curves the specific essential and plastic parts of the work can be further split into terms related to yielding (w_{eI} and w_{pI}), and to subsequent necking and fracture (w_{eII} and w_{pII}), respectively. Further investigations are needed, however, to conclude if w_{eI} agrees with the plane strain essential work of fracture ($w_{I,e}$), and to elucidate whether or not the latter conforms with the fracture initiation (w_i) term.

(ii) For direct determination of the plastic work, uniaxial tensile tests on dumbbells seem to be most suitable. Among the indirect methods targeting the assessment of the shape parameter (β), LM investigations should be preferred, since by IT hardly any distinction can be made between pure and diffuse yielding in the plastic zone of thicker sheets.

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REFERENCES

1. Joyce, J. A., ed., *Elastic-Plastic Fracture Test Methods: The User's Experience–ASTM STP 1114*. Amer. Soc. Test. Mater., Philadelphia, 1991.
2. Atkins, A. G. and Mai, Y.-W., *Elastic and Plastic Fracture*. Ellis Horwood, Chichester, 1988.
3. Broberg, K. B., *Int. J. Fracture*, 1968, **4**, 11.
4. Broberg, K. B., *J. Mech. Phys. Solids*, 1975, **23**, 215.
5. Williams, J. G., *Fracture Mechanics of Polymers*. Ellis Horwood, Chichester, 1987.
6. Mai, Y.-W. and Powell , P., *J. Polym. Sci., Part B, Phys.*, 1991, **29**, 785.
7. Hashemi, S., *Plast. Rubb. Compos., Process. Appl.*, 1993, **20**, 229.
8. Chan, W. Y. F. and Williams, J. G., *Polymer*, 1994, **35**, 1666.
9. Vu-Khanh, T., *Theor. Appl. Fract. Mechanics*, 1994, **21**, 83.
10. Hashemi, S. , *J. Mater. Sci.*, 1993, **28**, 6178.
11. Mai, Y.-W., in *Proceedings of the International Symposium on Polymer Alloys and Composites*, Hong Kong, 9–11 Dec., 1992, ed. C. L. Choy and F. G. Shin. Hong Kong Polytechnic Press, Hong Kong, 1994, pp. 104–120.
12. Karger-Kocsis, J., *Polym. Bull.*, 1996, **37**, 119.
13. Karger-Kocsis, J., Czigány, T. and Moskala, E. J., *Polymer*, submitted.
14. Saleemi, A. S. and Nairn, J. A., *Polym. Eng. Sci.*, 1990, **30**, 211.
15. Gray, A., Testing Protocol for Essential Work of Fracture, ESIS TC-4 Group, 1993.
16. Karger-Kocsis, J. and Czigány, T., *Polymer*, 1996, **37**, 2433.
17. Mai, Y.-W. and Cotterell, B., *Int. J. Fracture*, 1986, **32**, 105.