

# Deformation rate dependence of the essential and non-essential work of fracture parameters in an amorphous copolyester

József Karger-Kocsis<sup>a,\*</sup>, Tibor Czigány<sup>b</sup> and Eric J. Moskala<sup>c</sup>

<sup>a</sup>Institut für Verbundwerkstoffe GmbH, Universität Kaiserslautern, Pf. 3049, Kaiserslautern, D-67653, Germany

<sup>b</sup>Institute of Machine Design, Faculty of Mechanical Engineering, Technical University of Budapest, Budapest, H-1111, Hungary

<sup>c</sup>Eastman Chemical Co., Research Laboratories, PO Box 1972, Kingsport, TN 37662-5150, USA

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The plane stress fracture toughness of an amorphous copolyester (aCOP) was determined at ambient temperature as a function of the deformation rate ( $v = 1, 10$  and  $100 \text{ mm min}^{-1}$ ) by the essential work of fracture (EWF) concept using tensile-loaded deeply double-edge notched (DDEN-T) specimens. It was established that both specific essential ( $w_e$ ) and non-essential or plastic work of fracture ( $w_p$ ) are composed terms linked to yielding (subscript  $y$ ) and necking (subscript  $n$ ), respectively. The essential terms, i.e.  $w_{e,y}$  and  $w_{e,n}$ , did not change with increasing  $v$ . This indicates that by increasing  $v$  no alteration in the initial plane stress conditions occurred. The slopes of regression lines fitted for the  $w_y$  versus ligament (i.e.  $\beta' w_{p,y}$ ) and  $w_n$  versus ligament ( $\beta'' w_{p,n}$ ) data tended to increase with increasing  $v$ . The overall shape parameter of the plastic zone ( $\beta$ ) slightly decreased with the deformation rate. The plastic zone was formed by cold-drawing and not via true plastic deformation as evidenced by annealing-induced (just beyond  $T_g$ ) shape recovery. © 1998 Elsevier Science Ltd. All rights reserved.

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## INTRODUCTION

The essential work of fracture (EWF) theory, credited to Broberg<sup>1</sup>, is gaining in acceptance for the toughness description of ductile polymers. EWF is preferred because the tests are easy to perform and data reduction is simple<sup>2</sup>, especially compared to the  $J$ -integral technique<sup>2,3</sup>. The EWF approach makes a difference between the essential ( $W_e$ ) and non-essential or plastic work ( $W_p$ ). The former term represents the energy required to fracture the polymer in its process zone and is surface-related ( $lt$ , see later).  $W_p$ , in contrast, is a volume-related term ( $l^2t$ ) and represents the energy consumed by various deformation mechanisms in the surrounding outer plastic zone). The total work of fracture ( $W_f$ ) is composed of the two above parameters and can be given by the related specific work terms:

$$W_f = w_e lt + \beta w_p l^2 t \quad (1)$$

$$w_f = \frac{W_f}{lt} = w_e + \beta w_p l \quad (2)$$

where  $l$  is the ligament length,  $t$  is the thickness of the specimen and  $\beta$  is a shape factor linked to the form of the plastic zone. The  $\beta w_p$  term is approaching 0 when the plastic zone is confined to the necking process zone only. It is assumed that  $\beta$  does not change with  $l$ . This criterion is met when the load–displacement ( $F$ – $x$ ) curves at various

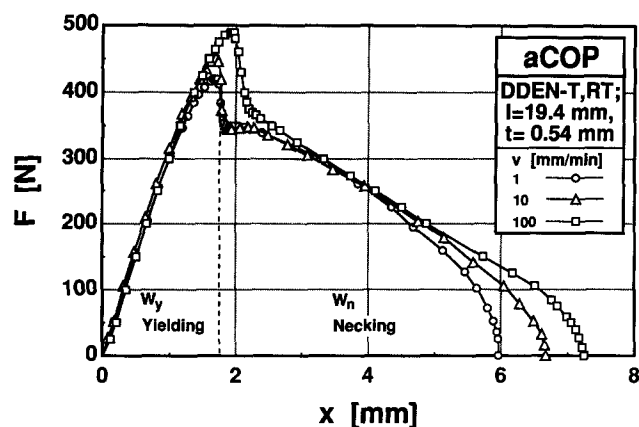
ligament are similar to one another (i.e. they ‘coalesce’ by a linear transformation procedure).

The specific essential work ( $w_e$ ) is considered as a material parameter under plane stress conditions at least for a given thickness. It was shown by adopting the EWF method for a suitable model polymer, that  $w_e$  is a composite parameter under plane stress in which both instantaneous yielding and subsequent necking processes are involved<sup>4,5</sup>. According to equation (2)  $w_e$  should be independent on the specimen configuration (type, thickness) and even on the deformation rate at a given temperature if plane-stress is accommodated in the specimens. In the literature there are many examples stating that the specimen geometry does not affect the value of  $w_e$  (e.g. Refs. 6–9). In our previous article<sup>4</sup> it was shown that  $w_e$  is, in fact, thickness independent under the aforementioned conditions. In respect to the loading rate dependence of the work of fracture parameters, the state of knowledge is more controversial. Based on results achieved on polyimide films it was found that the effect of testing speed ( $v$ ) is minimal for both  $w_e$  and  $\beta w_p$ , at least for the range  $v = 5$ – $20 \text{ mm min}^{-1}$ <sup>7</sup>. For a filled, biaxial oriented polyethylene terephthalate (PET) film it was reported<sup>10</sup> that  $w_e$  does not depend, whereas  $\beta w_p$  (more exactly the  $w_p$  term itself) does depend on  $v$  (set for 1 and  $20 \text{ mm min}^{-1}$ , respectively). Antoniazzi *et al.*<sup>11</sup> studied the fracture behavior of pure and filled polyethylene (PE) and concluded that the plastic work data became smaller with increasing  $v$ . In a recent study aimed at the toughness determination of polybutylene terephthalate/polycarbonate (PBT/PC) blends<sup>12</sup>, the author claimed that

\* To whom correspondence should be addressed.

**Table 1** Basic mechanical properties of the aCOP sheet (thickness, 0.54 mm) as a function of the deformation rate

Deformation rate ( $\nu$ ) (mm min <sup>-1</sup> )	E modulus (GPa)	$\sigma_y$ (MPa)	$\epsilon_y$ (%)
1	1.70	39.2	≈ 3.0
10	1.81	40.8	≈ 3.5
100	1.90	44.9	≈ 4.0



**Figure 1** Comparison of the  $F$ - $x$  curves of DDEN-T specimens at the same ligament length at various deformation rates for aCOP

$\nu$  (in the range of 2–50 mm min<sup>-1</sup>) does not influence  $w_e$ , but strongly affects the  $\beta w_p$  term. Hashemi suspected, furthermore, that it is the shape parameter ( $\beta$ ) which is likely changing with  $\nu$ <sup>12</sup>.

The authors of this contribution believe that the effect of test speed can be clarified by using amorphous copolyesters (aCOP) as model materials for the EWF studies. They undergo full ligament yielding prior to onset of crack growth, which allows us to determine the contributing terms

of  $w_e$ , viz. required for yielding and subsequent necking, respectively<sup>4,5</sup>. Further, following the development of the plastic zone in situ (during loading via infrared thermography, IT) or analyzing its size post-mortem (by viewing in light microscopy, LM), the shape parameter ( $\beta$ ) can be unequivocally deduced and thus its effect, if any, quantified. This paper was devoted to the above aspects by studying the EWF response of an aCOP on deeply double edge-notched specimens (DDEN-T) at tensile loading in a deformation rate range covering two decades ( $\nu = 1, 10$  and 100 mm min<sup>-1</sup>).

## EXPERIMENTAL

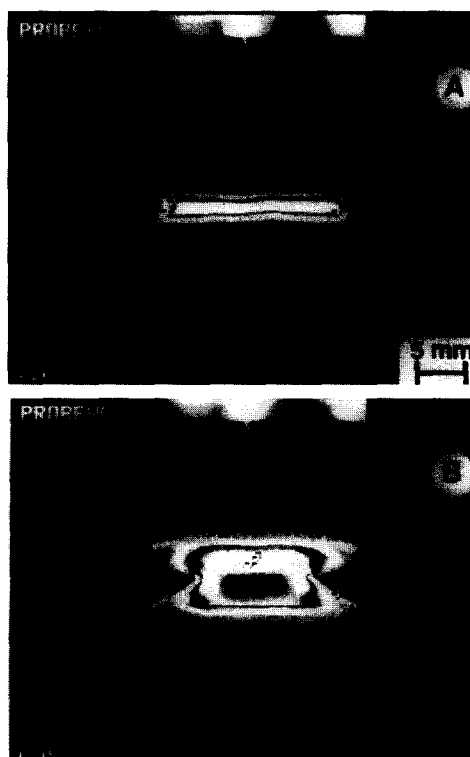
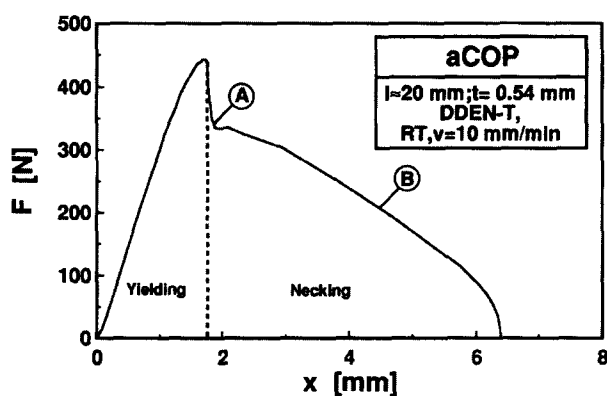
### Materials

Amorphous copolyester (aCOP) sheets with a thickness of ca. 0.5 mm, supplied by the Eastman Chemical Co. (Kingsport, TN, USA), were used for preparation of the DDEN-T specimens. This aCOP of an intrinsic viscosity (IV) of 0.705 dl g<sup>-1</sup> was synthesized from dimethyl terephthalate, ethylene glycol and 1,4-cyclohexane dimethanol. The content of the latter monomer was 68 mol%.

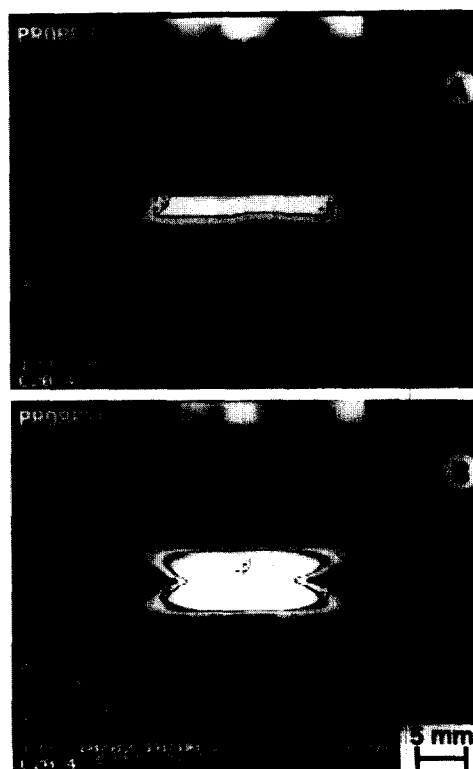
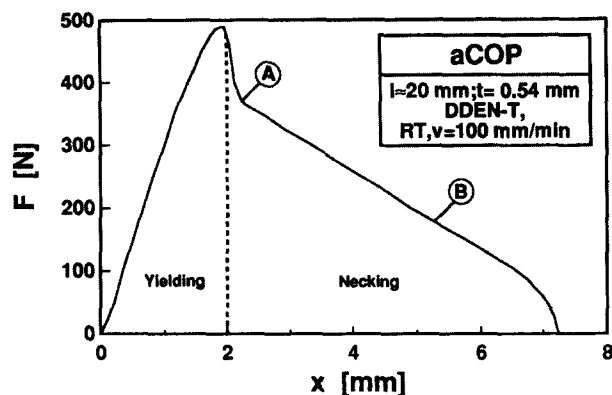
### Testing

All mechanical tests reported here were performed at room temperature (RT) on a Zwick 1445 universal testing machine. Tensile E-modulus ( $E$ ) and yield strength ( $\sigma_y$ ) and strain ( $\epsilon_y$ ) were determined as a function of  $\nu$  by using dumbbells (No. 3 according to DIN 53 455) and the results are listed in Table 1. It is noteworthy that the dumbbells failed after necking induced by coarse shear band formation<sup>5</sup> in the whole deformation rate range selected.

For the EWF study double deeply edge-notched tensile (DDEN-T) specimens with a width of 35 and overall length



**Figure 2**  $F$ - $x$  curve of a DDEN-T specimen of aCOP with  $l \approx 20$  mm along with the serial IT frames taken during loading with  $\nu = 10$  mm min<sup>-1</sup>. Note: The IT frames were recorded at the positions indicated in the  $F$ - $x$  curve



**Figure 3**  $F$ - $x$  curve of a DDEN-T specimen of aCOP with  $l \approx 20$  mm along with the serial IT frames taken during loading with  $v = 100$  mm  $\text{min}^{-1}$ . See Figure 2 for note

**Table 2** Essential work ( $w_e$ ) and slope of the regression lines ( $\beta w_p$ ) along with the related correlation coefficients ( $R^2$ ) as a function of the deformation rate

$v$ (mm $\text{min}^{-1}$ )	$w_e$ (kJ $\text{m}^{-2}$ )	$\beta w_p$ (MJ $\text{m}^{-3}$ )	$R^2$
1	35.55	5.43	0.993
10	33.95	5.76	0.983
100	33.37	6.59	0.966

of 100 mm (clamped length 70 mm) were selected. The free ligament length ( $l$ ) was set in the range from  $l = 5$ –30 mm. At every ligament at least three specimens were investigated. Data reduction (cf. Equation (2)) followed the recommendations of the ESIS TC-4 group<sup>14</sup>.

The non-essential or plastic work ( $w_p$ ) was calculated in the knowledge of the shape parameter of the plastic zone. The latter was defined by light microscopic (LM) and infrared thermographic (IT; thermocamera of Hughes, Portland, OR, USA) inspection as disclosed in our previous works<sup>4,5</sup>. Further details to the experimental section can also be taken from the above references.

## RESULTS AND DISCUSSIONS

### Effects of deformation rate

Figure 1 depicts the  $F$ - $x$  curves of DDEN-T specimens of aCOP at the same ligament for various deformation rates. The load decay in the  $F$ - $x$  curves is obvious. Here the yielding process is completed and necking accompanied with crack growth start. At this load drop the whole ligament yields instantaneously, as evidenced by IT pictures (see Figures 2 and 3). Comparing the IT frames in Figures 2 and 3 one can see that by increasing  $v$  the temperatures in the process and plastic zones also increase. According to the cursor points, put in the view field of the IT maps for

indication of the local temperature, a temperature rise of about 4<sup>5</sup>, 7 (Figure 2) and 15°C (Figure 3) can be established for  $v = 1$ , 10 and 100 mm  $\text{min}^{-1}$ , respectively. A further important observation from the videotaped IT failure sequence is that neither the shape nor the size of the plastic zone seem to change with increasing  $v$ . In Figure 1 one can recognize that both  $w_y$  and  $w_n$  terms, related to yielding and necking ended by fracture, are increasing with increasing  $v$ .

Figure 4 depicts the  $w_f$  versus  $l$  curves for the DDEN-T specimens of aCOP at various values of  $v$ . The specific work of fracture ( $w_f$ ), being the sum of  $w_y$  and  $w_n$ , was computed from the area beneath the  $F$ - $x$  curves monitored (see Figure 1). According to equation (2), the essential work of fracture ( $w_e$ ) was obtained from the intercept of the linear regression line with the ordinate (see Table 2). Figure 4 shows that  $w_e$  does not change practically with  $v$ . This can also be claimed if  $w_e$  is determined by considering the data-related 95% confidence limits, recommended by the EWF testing protocol<sup>14</sup>, which of course augments the scatter range. Figure 4 provides experimental evidence that  $w_e$  does not depend on the  $v$  when the EWF concept is applied to a suitable polymer.

The next important finding is that the slope of the regression lines, e.g.  $\beta w_p$ , increases with increasing  $v$ . Attention will be paid next to whether this increase in the slope depends on a change in the plastic zone (shape, size) or on the related deformation mechanisms.

### Validation of the EWF tests

Before starting with the discussions of the work of fracture, it is reasonable to check the validity of the tests. First let us focus on the size criteria. Test results on DDEN-T specimens are usually accepted as valid, if the ligament range set is between the following limits<sup>2,7-16</sup>

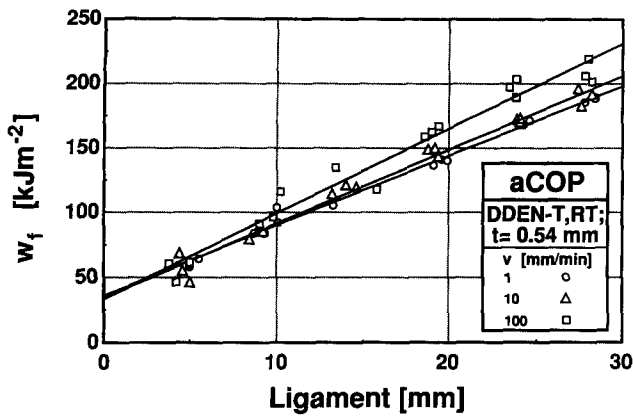


Figure 4 Total specific work of fracture ( $w_f$ ) versus ligament length ( $l$ ) for the aCOP as a function of the deformation rate ( $v$ )

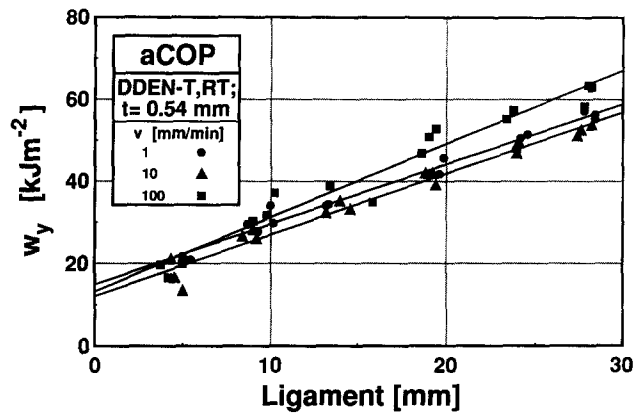


Figure 7  $w_y$  versus  $l$  data for the DDEN-T specimens of aCOP at various deformation rates

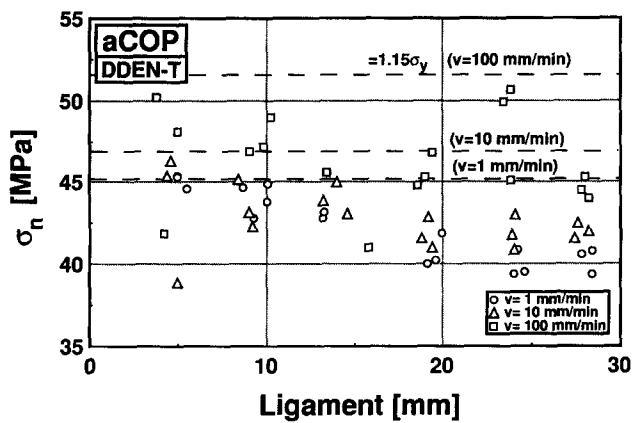


Figure 5  $\sigma_n$  versus  $l$  curves of the DDEN-T specimens at various deformation rates

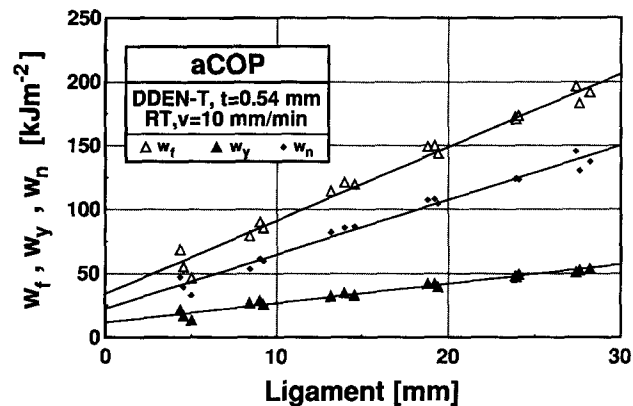


Figure 6 Total specific work of fracture ( $w_f$ ) and its contributing terms ( $w_y$  and  $w_n$ ) versus ligament length ( $l$ ) for the DDEN-T specimens of aCOP at  $v = 10 \text{ mm min}^{-1}$

$$(3 - 5)l \leq 1 \leq \min \left( \frac{B}{3} \text{ or } 2r_p \right) \quad (3)$$

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

$$2r_p = \frac{1}{\pi} \frac{E w_e}{\sigma_y^2} \quad (4)$$

$2r_p$  calculated by inserting the data included in Table 1 resulted in values of ca. 10–12.5 mm. Although this range and the alternative width criterion (i.e.  $B/3 \approx 12 \text{ mm}$ ) are

similar, neither represents the upper ligament exclusion limit. Based on the self-similarity of the  $F-x$  curves in the ligament range up to  $l = 30 \text{ mm}$ , it is obvious that the upper threshold values in equation (3) strongly underestimate the reality. Due to problems related to notching and assessment of the free ligament, the lower threshold value could not be confirmed—the minimum ligament set was at about 5 mm.

A further validity check is linked to the constraint effects. In DDEN-T specimens under plane stress the net section stress ( $\sigma_n$ , defined as  $F_{\max}/lt$ , where  $F_{\max}$  is the peak load) should lie below  $1.15\sigma_y$ , where  $\sigma_y$  is the yield stress of the material, for the ligament range studied<sup>14,16</sup>. The  $\sigma_n$  versus  $l$  curves in Figure 5 show that this criterion holds for the whole  $v$  range studied. It is worth noting that  $\sigma_n$  decreases monotonically with increasing  $l$  at  $v = 1 \text{ mm min}^{-1}$  (the usual case), but this tendency disappears with increasing  $v$  (see the related data scatter in Figure 5). It can thus be stated that  $v$  did not affect the original plane stress condition of the specimens.

#### Essential work of fracture and constituting terms

Based on the load drop in the  $F-x$  curves of COP, the specific work of fracture required for yielding ( $w_y$ ) can be distinguished from the specific work of fracture required for necking and subsequent fracture ( $w_n$ ); cf. Figure 1. This splitting is demonstrated on the example of the  $w_f$  versus  $l$  results achieved at  $v = 10 \text{ mm min}^{-1}$  (Figure 6). Accordingly, equation (2) can be rewritten by considering the yielding- and necking-related terms:

$$w_f = w_e + \beta w_p l = w_y + w_n \quad (5)$$

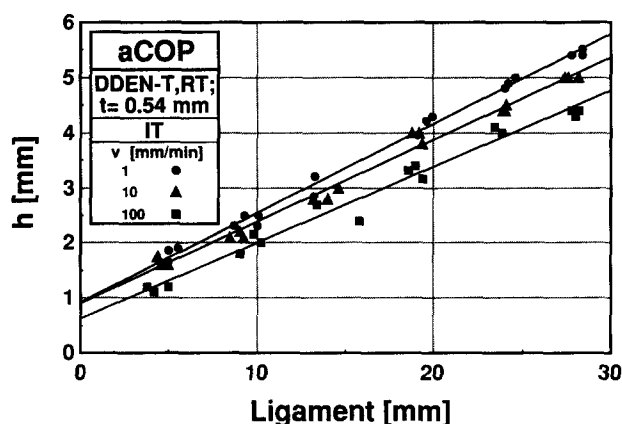
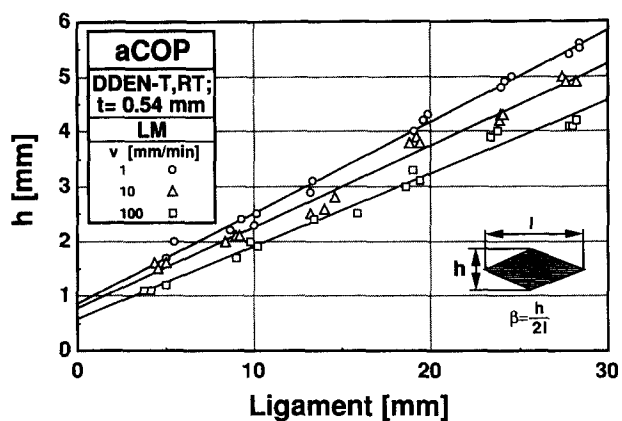
$$w_y = w_{e,y} + \beta' w_{p,y} l \quad (6)$$

$$w_n = w_{e,n} + \beta'' w_{p,n} l \quad (7)$$

Figure 6 provides experimental evidence that the plane stress essential work of fracture is itself a composite term. It was argued in our previous paper<sup>4</sup> that because of a change toward plane strain (induced by increasing thickness) the necking contribution diminishes and thus  $w_e$  may become a single material parameter. The results of the plots  $w_y$  versus  $l$  as a function of  $v$  are shown in Figure 7, and the outcome is summarized in Table 3. One can see that the  $w_{e,y}$  values are practically constant. Table 3 contains also the necking contribution to  $w_e$ , viz.  $w_{e,n}$ , which does not change with  $v$  either. These data were obtained by plotting  $w_n$  versus  $l$ , analogously to Figure 7. The fact that

**Table 3** Yielding- (subscript y) and necking-related (subscript n) specific work of fracture parameters as a function of deformation rate

$v$ (mm min <sup>-1</sup> )	$w_{e,y}$ (kJ m <sup>-2</sup> )	$\beta'w_{p,y}$ (MJ m <sup>-3</sup> )	$R^2$	$w_{e,n}$ (kJ m <sup>-2</sup> )	$\beta''w_{p,n}$ (MJ m <sup>-3</sup> )	$R^2$
1	14.73	1.46	0.981	19.29	4.03	0.988
10	12.05	1.49	0.970	21.90	4.27	0.982
100	13.23	1.80	0.958	20.15	4.79	0.960


**Figure 8** Total height of the plastic zone ( $h$ ) determined by LM (a) and by IT (b) versus ligament length ( $l$ ) for DDEN-T specimens of aCOP at various deformation rates

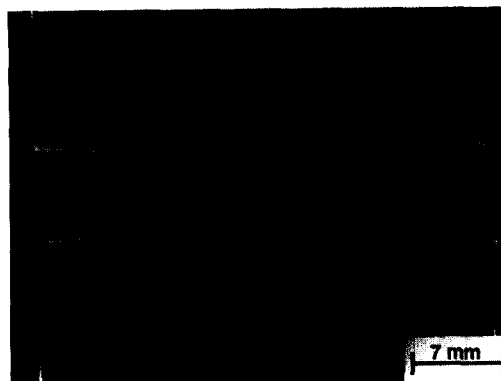
**Table 4** Plastic work-related terms and shape parameter of the plastic zone ( $\beta$ ) defined by different approaches

$v$ (mm min <sup>-1</sup> )	$\beta w_p$ (MJ m <sup>-3</sup> )	$\beta'w_{p,y}$ (MJ m <sup>-3</sup> )	$\beta''w_{p,n}$ (MJ m <sup>-3</sup> )	Shape parameter	
				$\beta$ (LM)	$\beta$ (IT)
1	5.43	1.46	4.03	0.082	0.081
10	5.76	1.49	4.27	0.074	0.075
100	6.59	1.80	4.79	0.066	0.069

both  $w_{e,y}$  and  $w_{e,n}$  are practically unaffected by  $v$  indirectly confirms that plane stress conditions are maintained in the tests. Note that change from plane stress toward plane strain condition should have resulted in a decrease in these terms.

#### Non-essential work of fracture and constituting terms

The shape parameter ( $\beta$ ) was estimated by two indirect methods: (a) viewing the form of the plastic zone of the broken specimens by LM (cf. Figure 8a) and (b) analyzing the IT heat maps taken on the specimens prior to their final separation (cf. Figure 8b,<sup>12,13</sup>). According to the LM results the shape of the plastic zone was approached by a shallow


**Figure 9** Macrophotograph of the ligament area ( $l \approx 25$  mm) of a DDEN-T specimen before annealing (upper side) and after annealing (lower side) just above  $T_g$  ( $T = 105^\circ\text{C}$ , 10 min)

diamond for which

$$\beta = \frac{h}{2l} \quad (8)$$

holds<sup>14</sup> (note that the surface of this diamond-shaped plastic zone is  $hl/2$ ; cf. Figure 8a), where  $h$  is the overall height of the plastic zone (cf. Figure 1). A very good agreement between the LM (Figure 8a) and IT results (Figure 8b) was found for  $\beta$  (see Table 4).  $\beta$  slightly decreased with increasing  $v$ . However, further investigations are needed to substantiate this tendency.

In order to support the treatise on the non-essential work parameters, they are repeatedly listed in Table 4. Considering the absolute values of  $\beta'w_{p,y}$  and  $\beta''w_{p,n}$ , it becomes obvious that the plastic zone development is linked mostly to the necking process, as expected. Furthermore, the  $\beta'w_{p,y}$  term seems to correlate very well with  $\sigma_y\epsilon_y$  (cf. Table 1). It cannot be excluded either that  $\sigma_y\epsilon_y$  is even identical with  $\beta'w_{p,y}$ . Unfortunately, the exact determination of  $\epsilon_y$  becomes more problematic with increasing  $v$ . Further, the corresponding conditions of the tensile (yielding) tests on dumbbells and DDEN-T specimens are not specified yet. Nevertheless, if  $\beta'w_{p,y}$  is identical to or changes similarly to  $\sigma_y\epsilon_y$ , the  $\beta'$  parameter remains unaffected. The authors are, however, not aware how this  $\beta'$  term could be defined explicitly.

Regardless of whether the overall shape parameter  $\beta$  is decreasing or not,  $w_p$  is increasing with  $v$ , especially at  $v = 100$  mm min<sup>-1</sup> (cf. Table 4). How can this finding be explained? This aspect can be clarified by understanding the yielding and necking mechanisms. As demonstrated in Figures 2 and 3, a substantial temperature rise accompanies yielding. The following question arises: does the local temperature exceed the glass transition temperature ( $T_g$ )? If yes, an irreversible plastic deformation should take place. By keeping the broken DDEN-T specimen for few minutes just above the  $T_g$  ( $T_g$  of this aCOP is ca.  $90^\circ\text{C}$ <sup>4</sup>), the plastic zone diminished and the shape of the specimen was fully restored ('healed') even when fractured at  $v = 100$  mm min<sup>-1</sup> (Figure 9)<sup>13</sup>. This suggests that neither the  $T_g$  was reached nor a substantial change in the initial entanglement network structure occurred due to loading. Recall that the existence of the latter is the driving force for this memory effect<sup>17,18</sup>. The recovery of the initial shape of the DDEN-T specimens is the evidence that cold-drawing, and not true plastic flow, took place in the plastic zone of this aCOP. Based on this finding hardly any change in the  $w_e$  and  $w_p$  parameters can be expected. This prediction was

found as valid for the essential work terms, but is likely to fail for the non-essential parameters (especially when the data related to  $v = 100 \text{ mm min}^{-1}$  are considered, cf. Table 4). This can only be true if some morphological changes are triggered by increasing deformation rate. The precise mechanisms of the morphological changes, however, should not influence considerably the segmental recovery within the temporary physical network structure composed of chain entanglements. Therefore the following processes may be supposed to be at work: formation of small crystalline domains (recall that the aCOP studied is susceptible to strain-induced crystallization), overstretching of segments between the network knots without causing rupture, or the onset of multiple localized shear bands (provided that the molecular slippage involved can be recovered by segmental motions). Input to a deeper understanding of this issue is expected from modulated d.s.c. (m.d.s.c.) investigations, which are now in progress.

## CONCLUSIONS

The plane stress ductile fracture behavior of an amorphous copolyester (aCOP) sheet (thickness *ca.* 0.5 mm) was studied as a function of the deformation rate at ambient temperature by adopting the essential work of fracture (EWF) method on deeply double-edge notched tensile (DDEN-T) specimens. Based on this study the following conclusions may be drawn:

(i) The plane-stress specific essential work of fracture ( $w_e$ ) is independent of  $v$ , when the EWF approach is applied for an ideal polymer (undergoing full ligament yielding prior to the onset of crack growth), provided that no plane stress–plane strain transition takes place due the increase in the deformation rate. Based on a load drop marking an instantaneous yielding in the load-displacement curves of the DDEN-T specimens, the specific essential ( $w_e$ ) and plastic parts of work ( $w_p$ ) could be separated into terms related to yielding (subscript y) and necking (subscript n). The essential constituents of the related terms, i.e.  $w_{e,y}$  and  $w_{e,n}$  remained constant, whereas the plastic ones seemed to increase with increasing  $v$ .

(ii) The overall shape parameter ( $\beta$ ), estimated by

light microscopic (LM) and infrared thermographic (IT), decreased slightly with increasing  $v$ . Upon annealing above  $T_g$ , the plastic zone disappeared and the original shape of the specimen was restored. This suggests that the plastic zone, in which no true plastic deformation occurred, was developed by a cold-drawing process in which the entanglement network was well preserved.

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