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Essential work of fracture: application for polymers showing ductile-to-brittle transition during fracture

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Abstract

The essential work of fracture (EWF) fails for the toughness determination of polymers showing a decrease of the specific work of fracture, as a function of the specimen ligament. This type of behaviour was observed for poly(butylene terephthalate) (PBT) and its core/shell rubber modified blend (PBT/CS). It was found that this peculiar behaviour is due to a ductile-to-brittle transition (DBT) in the crack propagation phase. Experimental data were corrected by considering only the ductile-fractured specimen area. When a non linear function of the type $y=a+bx^{-1}$ was applied for the corrected specific work of fracture and ligament data, the specific essential work of fracture (w_e) could be deduced. The latter being an inherent material toughness parameter was compared with the critical J-integral (J_c) values and a good correlation was found between them.

Introduction

In the last years there has been a lot of discussion and dispute over the applicability of the essential work of fracture (EWF) method. One of the most controversial issues the EWF confronts is that some polymers and composites exhibit a reduction in the total work of fracture with increasing ligament. Several authors like Mamat et.al (1) and Vu-Khanh (2-3) have reported on this issue, on the examples of toughened polyamide-66, high impact polystyrene and polyamide-6/ABS blends, respectively. The cited authors have observed that with increasing ligament length in single-edge notched specimens, the specific fracture energy (w_f) decreases under impact conditions. This resulted in a negative slope of the w_f plots against ligament size (l). As a result, it was impossible to obtain valid crack initiation parameters (viz. specific essential work of fracture, w_e) from these plots. Based on these facts, the authors concluded that the EWF method is inapropriate for the fracture toughness assessment of these materials. However none of them tried to explain or comment the actual mechanism which drove to such a behaviour. Similar problems have been reported by Wu (4). He was however succesful in correcting the experimental results using a non linear correction method, by taking in consideration the specimen kinetic energy. In other cases specimen inhomogenities or violations of the

The aim of this work was to study the rationale behind the fracture toughness decrease with increasing ligament in poly(butylene terephthalate) (PBT) blends. Efforts were undertaken to extract valid results irrespective to the singularities observed in the experimental data. The working hypothesis was that the negative slope of the w_f vs 1 plots is owing to a ductile to brittle transition (DBT), assosiated with a change in the plane stress/plane strain conditions.

Experimentals

In order to elucidate the effects of DBT on the fracture response, both most significant methods of elasto-plastic fracture mechanics were used: the EWF and the J-integral approaches. In this way it would be also possible to compare the applicability of both these approaches for the characterization of toughened polymers. Furtheron, crack initiation data (i.e. w_e) obtained by the EWF-correction method proposed would be easy to confirm, since both critical crack initiation parameters (i.e. J_c and w_e) should be identical.

Essential Work of Fracture

According to the EWF theory (6-8), a distinction is being made between a process zone or process plane where the actual crack runs, and a plastic zone, which surrounds the process zone. Consequently, the total work required to fracture a pre-cracked specimen can also be divided in two parts associated with each of the two zones mentioned above. It can be written therefore:

$$W_f = W_e + W_p \qquad [1]$$

where W_f is the total fracture work, W_e the work spent for the crack advance in the crack plane and thus for the generation of new surfaces and is W_p the energy consumed in the plastic zone. Thus, W_e is related with a 2-D plane and is therefore a function of area (lt) whereas W_p is dissipated in a 3-D plastic zone and can be thus considered a function of volume (l^2t), where: t=specimen thickness, l=ligament. Equation 1 can be also expressed by the specific terms:

$$w_f = w_e + ßw_p l \qquad [2]$$

where: $w_f = W_f/lt$, $w_p = W_p/l^2t$, and ß is a geometry factor associated with the shape of the plastic zone.

According to Equation 2, the work of fracture is a linear function of the ligament size. w_e can be determined from the interception of the linear regression line, fitted to the w_f vs l graphs, with the y-axis. It should be mentioned here, that W_f can be determined by calculating the integral of force over displacement from the tensile tests performed on deeply double edge notched tensile (DDEN-T, cf. Figure 1) specimens of increasing ligaments. An important prerequisite of the plane stress EWF approach (9), is that crack propagates only after the ligament has been fully yielded. It was observed that this requirement was not met in the materials tested.

J-Integral

J-integral analysis is preferred when the toughness assessment of tough polymers and related blends is concerned. Traditional experimetal techniques of the linear elastic fracture mechanics, are inapplicable due to specimen geometry restrictions for such systems. According to Rice (10), J-integral can be considered as the difference of the potential energy between two loaded identical bodies with slightly different crack lengths systems. According to Rice (10), J-integral can be considered as the difference of the potential energy between two loaded identical bodies with slightly different crack lengths i.e. $J = \frac{1}{B}\frac{dU}{d\alpha}$ [3] , where: B: body thickness , U=total potential energy and α = crack length. According to Sumpter and Turner (11) Eq. (3) can be expanded and rewritten as:

$$J = J_e + J_p \ [4] \text{ or , } J = \frac{\eta_e U_e}{B(W-\alpha)} + \frac{\eta_p U_p}{B(W-\alpha)} \ [5]$$

where: J_e, J_p are the elastic and plastic components of the total energy, and η_e, η_p are the elastic and plastic geometry factors, respectively. In the case of compact tension (CT) specimens, where W-α represents the net unnotched specimen section, Eq. 5 is simplified to:

$$J = \frac{\eta}{B}\frac{U}{(W-\alpha)} \qquad [6]$$

where: η=2+0.522(1-α/W). U can be determined from the area under the load vs displacement curve up to the point corresponding to the test termination. The procedure for the critical crack initiation value of J-integral was standardized in ASTM E-813. Several different versions of this protocol exist; the most important are ASTM-81 and -89, where J_C is determined by the intersection of the linear regression or the power law fitted to the experimental J vs $\Delta\alpha$ data, with the crack tip blunting line $2\sigma_y\Delta\alpha$ respectively. According to the ESIS recommendation (12), the critical J-value for a 0.2mm crack advance, $J_{0.2}$,should also be considered as an initiation parameter. The authors do not wish to analyse the experimental techniques or the evaluation of the J-integral data, the interested reader is addressed to the related testing protocols.

Materials and specimen preparation

Plaques of pure PBT and a PBT blend with core/shell (CS) particles dispersed in a styrene/acrylonitril (SAN) matrix (overall modifier content: 20wt%) were involved in this study. Material for the experimental work was provided by the BASF AG. (Ludwigshafen , Germany) in form of injection molded quadratic plates of 180^*180^*4 mm^3 (see Fig. 1). DDEN-T; (dimensions: 40^*80^*4 mm^3) and compact tension (CT); (dimensions: 60^*60^*4 mm^3) specimens for EWF and J-integral tests respectively, were cut by a rotating disk table-saw. Both types of specimens were precracked using a band saw and a fine notch was introduced afterwards by tapping with a fresh razor blade. All specimens were of the LT type according to the ASTM E-616 prescriptions (cf. Figure 1).

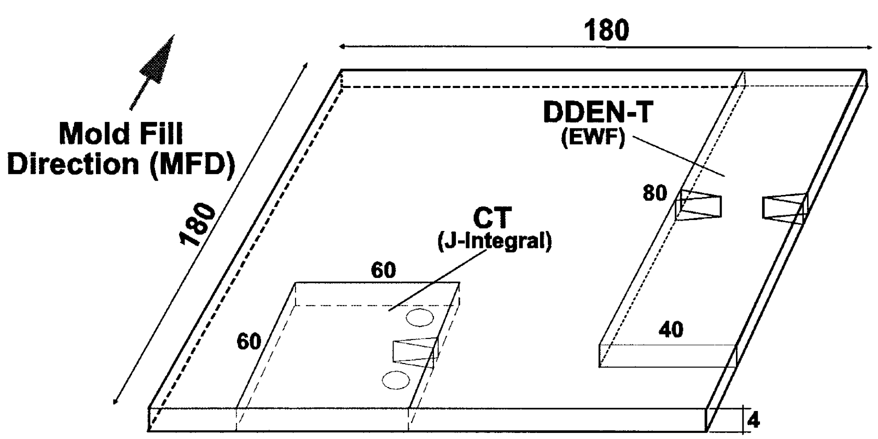

Figure 1. Configuration and dimensions of the specimens used (all dimensions are in mm)

Results and discussion

Elastoplastic analysis

Suprisingly, the wf vs l diagramms of both pure PBT and PBT/CS(20wt%) blend, did not deliver the expected scenario: an increasing work of fracture (w_f) with increasing ligament size (7-9,13-14). Instead, a rather large experimental scatter was observed along with a negative trend in the w_f data with increasing ligament was observed (see Figure 2). It is noteworthy, that the load-displacement curves of the DDEN-T specimens should be self-similar. This was not the case for our materials however, due to the DBT effect. Load-displacement curves showed a self-similarity in the pre-maximum load range which was missing in the post maximum stage due to the DBT. It is a first hint that the traditional way of extracting the w_e parameter cannot be applied in this case. Also, the non-essential work of fracture cannot be negative in any way(cf. Eq. 2). Recall, the latter term represents dissipated energy in the plastic zone, so the non-essential work of fracture is always positive. Note that the lowest threshold is zero, if no plastic zone develops during fracture of the specimen.

But what is the actual reason for the negative slope of the w_f vs l data in Figure 2 ? As known from theory, w_e data is surface dependent, therefore the fracture surface must logically provide traces of this peculiar fracture response. Indeed, this was the case. By examining the fracture surface of a DDEN-T specimen of the PBT/CS blend, very distinctive markings of differentiating fracture mechanisms along the ligament were found. Such a surface, sputtered with a Pt/Pd alloy in order to enhance visibility is presented in Figure 3. Observe the central highly reflective smooth region, which is typical for brittle fracture. In contrast, the darker areas ahead of the razor blade-induced notches represent a fibrillated ductile-fracture plane (15). Based on Figure 3, a DBT in the fracture mode can be confirmed. This DBT effect, observed before (5 and references therein), for the case of PP homopolymers, has up-to-today been identified as a sign of non-applicability for the EWF method.

Moreover, it is very appropriate to suppose that the brittle part of the fracture surface has very little or no contribution to the total work of fracture of the specimen at least in this case. That is, because the crack requires very small amount of energy to propagate when it becomes unstable. This implies the possibility to correct the EWF data by subtracting the brittle area from the total fracture plane. Thus, if $\boldsymbol{l}$ is the initial ligament and $\boldsymbol{l_B}$ is the width of the brittlely fractured ligament path, then the effective ligament corresponding to the EWF will be: $l^{corr} = l - l_B$ [7]

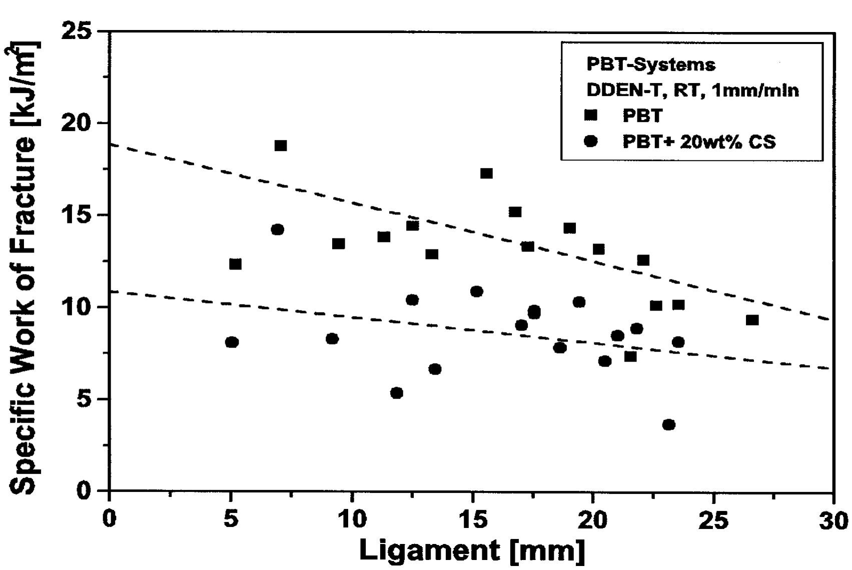

Figure 2. ***w_f vs l plots for the PBT systems studied***

l_B can be easily determined by using a stereomicroscope with a build-in measuring scale. By multiplying with specimen thickness t:

$$l^{corr}t = lt - l_Bt \ [8], \text{ or } A^{corr} = A - A_B \ [9]$$

where: A_B is the brittle fracture area and A^{corr} the surface corresponding to the ductile fracture (prerequisite of the EWF), respectively. Obviously, by using Equation 9 the specific work of fracture can be recalculated as:

$$W_f^{corr} = W_f / A^{corr} \ [10]$$

By using Equations 8 and 10 the corrected work of fracture can be plotted against the effective ligament. The $\mathbf{w_f^{corr}}$- $\boldsymbol{l^{corr}}$ graphs obtained are shown in Figure 4 for both PBT and PBT/CS. A clearly non-linear reduction of the fracture energy with increasing ligament can be confirmed. The trend appears to be asymptotic. Indeed, a function of the type $\boldsymbol{F(x)=C+Dx^{-1}}$ [11] can be fitted to the experimental data with satisfactory results (cf. Figures 4a and 4b, for the PBT matrix and the PBT/CS blend respectively).

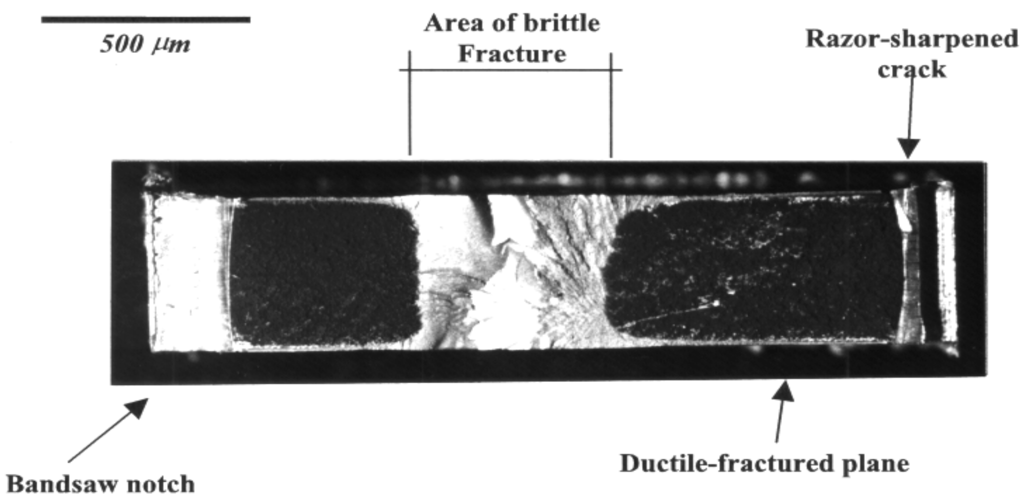

Figure 3. Fracture surface of a PBT/20wt% CS, DDEN-T specimen showing DBT transition.

Note: Contrast enhanced by sputtering with Pt/Pd

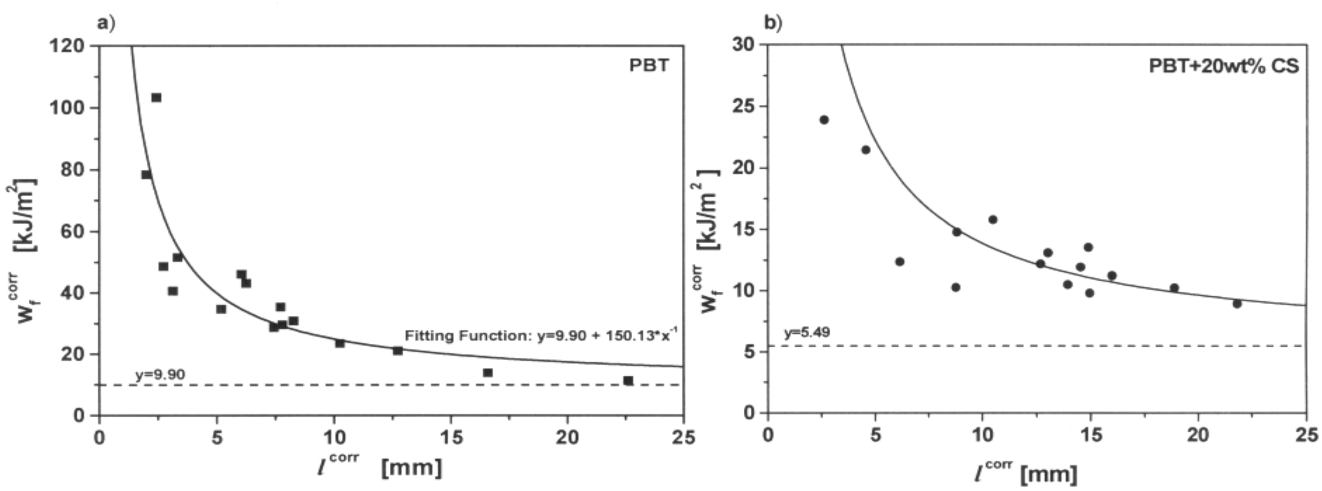

Figure 4. $\boldsymbol{w_f^{corr}}$ ***vs*** $\boldsymbol{l^{corr}}$ ***curves for the DDEN-T specimens of a). PBT and b). PBT/20wt% CS***

The curve fits in Fig. 4 confirm that data tend to an asymptotic value for the corrected specific work of fracture at large ligaments. A great advantage of this fit, is that the actual value of C represents per se the lowest threshold. Assuming that the asymptotically approached C represents the actual material parameter (i.e. C=$\mathbf{w_e}$) then it is de facto the crack initiation parameter. Results of the asymptotic fit are listed in Table 1. The authors are not aware of the physical meaning of parameter D in Equation 11 and further investigations are needed to determine it.

An analogue phenomenon to the above non linear EWF results has been stated by J.S.Wu et al. (4). He has observed a non linear increase in the specific work of fracture with increasing ligament, in the case of PBT/PC blends under impact (Charpy) testing. That behaviour was attributed to the effect of the specimen kinetic energy increase with increasing ligament under impact conditions. It is possible to make a comparison with Wu's discovery. In our case it is not the kinetic energy of the specimen that increases with increasing ligament but the kinetic energy of the crack tip. When the crack accelarates to a critical size it becomes unstable and DBT occurs. The following Figure 5 shows the actual growth of the brittle ligament region with increasing total specimen ligament.

J-integral tests delivered very important clues for the validity of the above mentioned approach. The primary data of the J-determination are given in Figure 6. Table 2 presents the plane strain critical crack initiation values (J_{IC}) as derived by the various protocols. w_e , delivered by the corrected and asymptotically fitted data, are also given for comparison purposes in Table 2. A very good agreement can be concluded, between all J_c values and the asymptotic EWF fit (C parameter), for the pure PBT. Data related to the PBT/CS blend are also not very different from each other. It is a fact that a mixed-mode (plane stress/plane strain) fracture was confronted in the case of the PBT/CS blends, which is likely responsible for the DBT effect.

Material:	**Parameter C [kJ/m²]**	**Parameter D [J/m]**
Pure PBT	**9.90**	**150.13**
PBT/CS(20wt%)	**5.49**	**77.85**

Table 1. Results of the asymptotic fit for PBT and PBT/CS(20wt%) materials

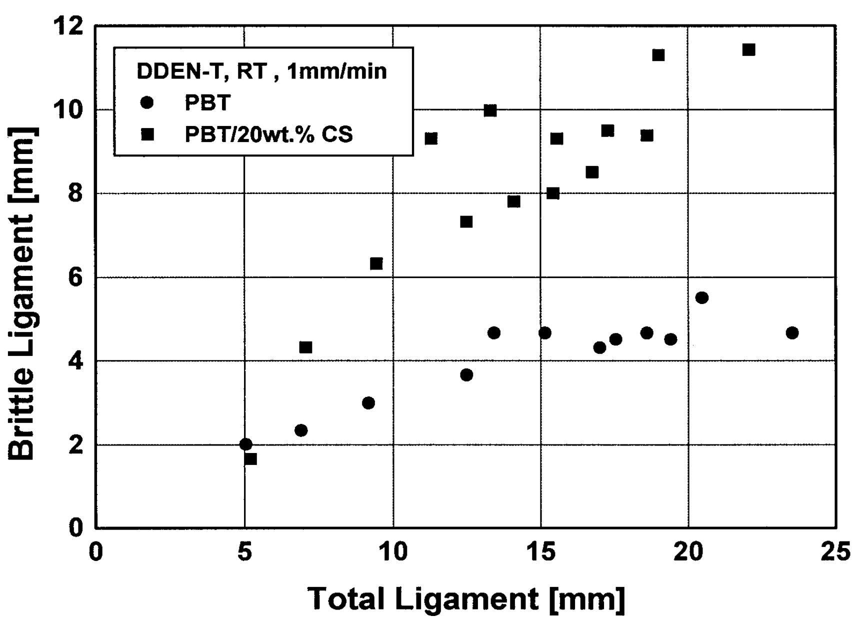

Figure 5. Brittle section as a function of the total specimen ligament

The extracted we data are confirmed by the critical plane strain J-integral values as given in Table 2 according to various protocols. The matching of the plane strain w_e and J_{IC} data has been proven earlier by Mai and Cotterell in their pioneering work (7).

However, the authors would like to state that as seen in Figure 4b, more experimental data were needed or specimens with larger ligaments (>30mm) to get a C= w_e, which is even closer, or identical to the J-integral data. It can be now concluded that the asymptotic fitting on the ligament-corrected EWF data provides a practical and likely a valid method for the evaluation of the plane strain specific essential work of fracture for polymers showing a ductile-to-brittle transition during fracture.

Conclusions

Based on the above performed analysis of the fracture behaviour of PBT and core/shell particle modified PBT (PBT/CS) the following conclusions can be drawn:

i- the decrease in the work of fracture with ligament size for the DDEN-T specimens is related to a ductile-to-brittle transition (DBT) in the crack propagation phase.

ii- A valid plane strain specific essential work of fracture parameter can be estimated by an asymptotic curve fitting method adopted for plots of the corrected fracture energy against the corrected specimen ligament. Ductile fractured ligament area is considered by this correction only.

iii- The specific work of fracture w_f , appears to be independent of ligament size at large ligaments. This implies that ductile fracture occurs without the formation of a plastic zone, so that the related critical value C= w_e is likely a material parameter, since plane strain conditions prevail.

Method:	EWF, C=w_e (asymptotic)	J_{IC} (ASTM 813-81)	J_{IC} (ASTM 813-89)	$J_{0.2}$ (ESIS)
Pure PBT	9.90	10.8	8.36	11.25
PBT/CS(20wt%)	5.49	2.18	1.42	2.51

Table 2. Results of the J-Integral analysis and comparison with EWF data

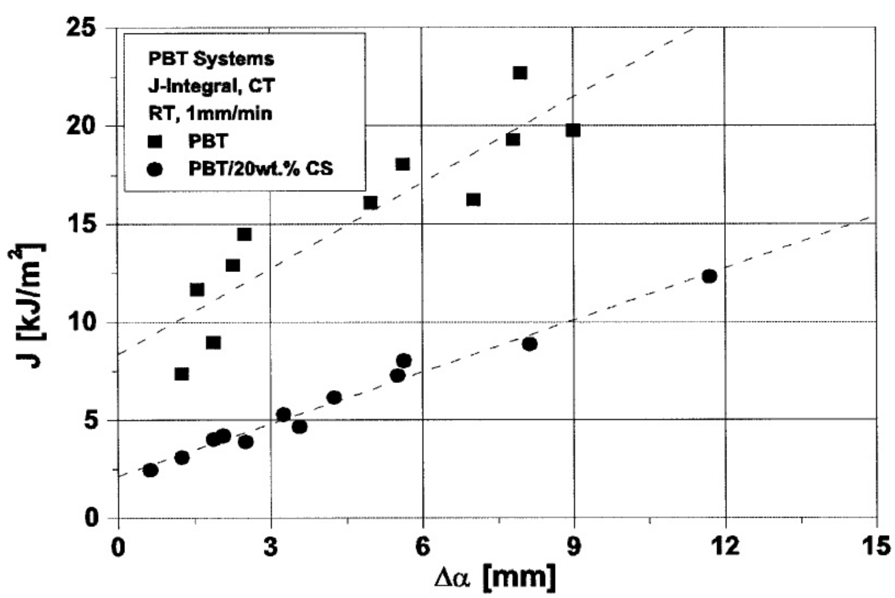

Figure 6. J-Integral evaluation of the PBT systems.

Acknowledgments

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