# Impact Specific Essential Work of Fracture of Compatibilized Polyamide-6 (PA6)/Poly(phenylene ether) (PPE) Blends

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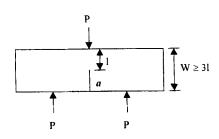
The essential work of fracture (EWF) method has aroused great interest and has been used to characterize the fracture toughness for a range of ductile metals, polymers and composites. In the plastics industry, for purposes of practical design and ranking of candidate materials, it is important to evaluate the impact essential work of fracture at high-rate testing of polymers and polymer blends. In this paper, the EWF method has been utilized to determine the high-rate specific essential fracture work,  $w_e$ , for elastomer-modified PA6/PPE/SMA (50/50/5) blends by notched Charpy tests. It is found that  $w_e$  increases with testing temperature and elastomer content for a given specimen thickness. Morphologically, there are two failure mechanisms: shear yielding and pullout of second phase dispersed particles. Shear yielding is dominant in ductile fracture, whereas particle pullout is predominant in brittle fracture.

# INTRODUCTION

The assessment of fracture toughness of ductile polymers by nonlinear or plastic fracture mechanics is currently of great interest. The J-integral technique has been widely adopted to overcome the inadequacy of linear elastic fracture mechanics (LEFM) that the formation of a large plastic zone prior to crack initiation violates the limit of small scale yielding, a necessary condition for the validity of LEFM. Although theoretical analysis (1-3) and experimental procedures have been standardized (4, 5) for J-integral evaluation, some aspects of the method still remain controversial for ductile polymers. For example, the nature of the standardized experimental procedure for J-R curve construction restricts the application of the J-integral method to static loading tests only. Its extension to determine the high-rate impact fracture toughness is difficult. Moreover, to obtain the planestrain J-integral value, the size requirements make the application of the J-integral method to polymeric thin specimens or films impossible. Despite many important proposals to overcome these problems, most are focused on modifications of J-integral evaluation for static loading tests (6–13). Hence, the development of a new and different experimental technique for high-rate fracture toughness characterization is needed.

Methods of measuring the impact resistance of polymers have always been of great interest. Many ductile polymeric materials can become brittle under impact loading conditions, so it is essential to determine the impact resistance of these materials when designing plastic parts. The most widely used methods to characterize impact fracture toughness of polymeric materials are the notched Charpy or Izod impact tests because of their convenience, simplicity and ready acceptance by plastics manufacturers and end users. In these tests, analytical methods based on fracture mechanics have been developed with the

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plastic zone

plastic zone

precrack

(a) deeply single edge-notched specimen (DSEN)

(b) a fractured specimen (fully yielded)

Fig. 1. Schematics of (a) DSEN geometry and (b) a fractured sample.

objective of providing a quantitative description of the impact fracture process. Though these notched impact-resistance tests are inherently flawed since a basic material property cannot be determined, they are considered the most severe fracture toughness tests. Besides the dynamic effect in impact tests, the mode of fracture involved could result in complication of the fracture energies measured. Thus, there are interesting and practical benefits to determine better fracture characterization parameters with these simple impact tests.

The essential work of fracture (EWF) method for determining the specific essential work of fracture  $w_e$  of a ductile polymeric material in plane-stress is now generally accepted by many researchers. It was recently proposed as a European Structural Integrity Society (ESIS) Test Protocol for Essential Work of Fracture under quasi-static loading conditions. There have been considerable interest and debate on the impact EWF method and testing methodology since Wu, Mai and Cotterell (14) extended the EWF concept to impact fracture of ductile polymer blends several years ago. Martinatti and Ricco (15) showed that it is valid to apply the EWF method to evaluate the high-rate plane-strain fracture toughness of polypropylene-based materials. Vu-Khanh (16), however, has argued that the EWF method is invalid by carrying out some tests and also using data of other investigators to show that  $\beta w_n$  is a negative quantity, which is physically meaningless.

To clarify the applications and usefulness of the EWF method to ductile polymer blends, this study intends to test its validity in determining the specific essential fracture work of PA6/PPE/SMA/G1651 blends using Charpy impact tests at different temperature. The fracture surfaces of the specimens are also investigated by scanning electron microscopy (SEM) and the relationship between the depth of the plastic deformation zone and the ligament length is investigated. This paper should be read in conjunction with other related studies concerned about the impact essential work of fracture measurements of ductile polymers (17–19).

#### **EXPERIMENTAL WORK**

## **Materials**

Polyamide-6 (PA6), trademark 1010C2, was obtained from Mitsubishi Kasei Co. Ltd. (Japan). Poly(phenylene

ether) (PPE), trademark HPP-820, with intrinsic viscosity of 0.4 dl/g was supplied by General Electrical Co. (USA). The compatibilizer, poly(styrene-co-maleic anhydride), trademark Dylark 232, with 8% MA and Mw =  $2 \times 10^5$ , was supplied by Arco Chemical Co. (USA). The elastomer, G1651, SEBS copolymer with styrene end-block, is a product of Shell Chemical Co. (USA).

#### **Experimental Procedure**

All blends were prepared in the CAMT at Sydney University on a co-rotating 30.85 mm twin-screw extruder (L/D = 43.5, ZSK-30, Werner & Pfleiderer Co., USA) with a rotational speed of 250 rpm. The barrel temperatures were set at 210 to 290°C. Three-millimeter-thick specimen plaques were prepared on a Boy-22S injection molding machine (L/D = 18). The injection molding temperature was between 270 to 290°C. Deeply single-edge-notched (DSEN) bend specimens with dimensions 83(S)  $\times$  17.5(W)  $\times$  3(t) mm were cut from these injection-molded plaques. A notch was introduced on the mid-length of one side using a guillotine-like apparatus with a fresh razor blade driven by a screw with 1 mm pitch. Pre-cracks with the required length (4  $\sim$  16 mm) could be easily made. To avoid possible plastic deformation at the crack tip, the razor should be always fresh and the pushing speed as slow as practical. Fracture mechanics evaluation was carried out on pre-cracked DSEN specimens with different notch depth, Fig. 1a, at different temperature using a Zwick 5102 Charpy impact tester with an impact velocity of 3 m/s. Fracture surfaces of specimens were studied with a Philips XL-30 scanning electron microscope (SEM).

## **Total Impact Work of Fracture Measurements**

Fracture toughness evaluation was carried out on pre-cracked SEN specimens with varying notch depth utilizing a Charpy impact tester at different temperature. The impact tester was equipped with a specimen holder that allowed three-point bending to be carried out. The impact fracture energies U were read directly from the scale on the machine. These values were then corrected for the kinetic energy  $K_E$  according to the methods described in (14, 19) to give  $W_f$  for each ligament length at a given temperature.

#### **RESULTS AND DISCUSSION**

#### **Background of Theory**

The essential work of fracture (EWF) concept was originally suggested by Broberg (20, 21) and later developed by Cotterell, Mai and their co-workers (22-27). This concept proposes that when a cracked ductile solid, such as a toughened polymer blend, is being loaded, the fracture process and the plastic deformation take place in two different regions: the inner fracture process zone and the outer plastic zone. During crack propagation, a large fraction of the total fracture work is dissipated in the plastic deformation zone; this fracton is not directly related with the fracture process. Only the work dissipated in the inner fracture process zone is a material constant. Hence, the total work is composed of the two components: essential work of fracture  $(W_e)$  and non-essential work of fracture  $(W_p)$  as follows:

$$W_f = W_e + W_p. \tag{1}$$

Theoretically, the specific essential work of mode I fracture can be defined as (25, 26):

$$W_e = d \int_0^{\varepsilon_n} \overline{\sigma} \ \overline{d\varepsilon} + \int_{\varepsilon,d}^{\delta_{RC}} \sigma(\Delta_1) d \Delta_1$$
 (2)

where d is the fracture process zone width which is approximately the same as the specimen thickness t.  $\overline{\sigma}$  and  $\overline{\epsilon}$  are true stress and true strain, respectively.  $\overline{\epsilon}_n$  and  $\epsilon_n$  are true and engineering necking strains.  $\sigma$  and  $\Delta_1$  are the stress and crack tip opening displacement within the fracture process zone.  $\delta_{IC}$  is the mode I critical value of  $\Delta_I$ . The first term of  $Eq\ 2$  represents the plastic flow work to form a neck and the second term is the additional work required to tear the neck to initiate fracture propagation.

 $W_e$  is surface-related and proportional to ligament length, l = (W - a), for a given specimen thickness t, whereas  $W_p$  is volume-related and proportional to  $l^2$ . Thus,  $W_f$  can be given by the specific related work terms ( $w_e$  and  $w_p$ ):

$$W_f = w_e lt + \beta w_p = l^2 t \tag{3}$$

$$w_f = W_f/lt = w_e + \beta w_p l \tag{4}$$

where  $\beta$  is a shape factor for the plastic deformation zone. Hence, it can be seen from Eq 4 that there is a linear relationship between  $w_f$  and l as long as  $\beta$  remains constant. The applicability of the EWF method depends on the following conditions being satisfied (17, 19): (a) The full ligament should yield prior to fracture initiation. (b) There should be geometric similarity between specimens of different ligament length during crack growth. (c) The essential work of fracture dissipated in the inner fracture process zone is proportional to l and the non-essential work of fracture dissipated in the outer plastic deformation zone is proportional to  $l^2$ , irrespective of the shape of the plastic zone. For ligament length significantly longer than

the specimen thickness, a ductile polymer blend will always be in a state of pure plane-stress. As the ligament length is reduced to values comparable to the specimen thickness, the stress state will become mixed mode having both plane-stress and plane-strain characteristics. To avoid mixed-mode fracture, the essential work of fracture experiments must be restricted to ligament lengths greater than 3 ~ 5 times the specimen thickness (22-24, 28). In the present study, the ligament lengths were less than 5t and hence are in the plane-strain/plane-stress regime. As shown by Wu, Mai and Cotterell (14), a linear relationship given by Eq 4 may still hold in this region if the conditions described for plane-stress above are obeyed. The intercept will then give a mixed mode specific essential work of fracture value,  $w_e$ . For this to be a true planestrain fracture toughness, then the specimen thickness t must satisfy the condition that

$$t \ge 25 \ w_e/\sigma_u \tag{5}$$

where  $\sigma_y$  is yield stress. Saleemi and Nairn (28) have given a different methodology to evaluate the plane strain  $w_e$  value from the  $w_f$  experimental data in the mixed-mode region by assuming  $\beta w_p$  to be invariant with t and hence the same as for plane stress deformation. However, this methodology cannot be adopted in the present study due to the limited range of ligament length investigated.

#### **High-Rate Impact Fracture Toughness**

Figure 2 shows the effect of temperature on the corrected impact energies of the elastomer-modified PA6/PPE/SMA (50/50/5) blends with a 14 mm ligament length. The corrected impact energies  $(U-K_E)$  of all blends increase with increasing temperature and elastomer content. The fracture behavior is brittle up to 80°C for the blend with 15 phr elastomer, up to 60°C for the blend with 20 phr elastomer and at 20°C for the blend with 25 phr elastomer. The blend with 30 phr elastomer shows a ductile fracture mode in the temperature range tested. For blends with brittle fracture behavior, their toughness values were evaluated by LEFM (by plotting  $W_f$  vs  $tW\Phi$ ) in accordance with Williams' analysis (29); the results are listed in Table 1 where applicable. Clearly, the strain energy release rates (G<sub>c</sub>) increase with temperature and elastomer content. A check on the thickness requirement for plane strain shows that this is not satisfied. Hence, the Gc values obtained are really mixed-mode toughness values. For blends that failed in a ductile mode, the EWF method was used to determine the high-rate specific essential work of fracture according to Eq 4. Typical plots are given for the PA6/PPE/SMA/G1651 (50/50/5/30) blends at four different temperatures in Fig. 3. Least squares lines are drawn through the data below 15 mm ligament length. That is, l < 5t. The specific essential works of fracture  $(w_e)$  of all other ductile blends that have met the required conditions for the EWF method are summarized in Table 1. It is noted that we also increases with elastomer content and

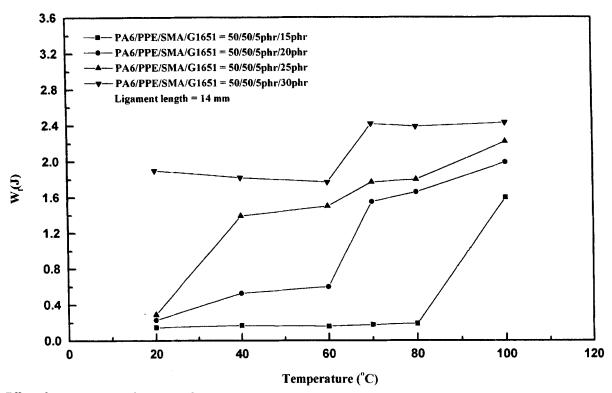


Fig. 2. Effect of temperature on the corrected impact energy absorption ( $W_f$ ) of elastomer-modified PA6/PPE/SMA (50/50/5) blends.

temperature for a given sheet thickness. Again, these  $w_e$  values measured do not satisfy the thickness required for plane strain as determined from Eq 5. They should be regarded as mixed-mode specific essential works for the blends with a sheet thickness of 3 mm.

For those blends whose ligaments are not fully yielded, that is, blends with 25 phr elastomer at 20°C and 20 phr elastomer at 40°C, their fracture behavior is ductile tearing with plastic flow confined to a small circular region ahead of the notch tip, followed by fast

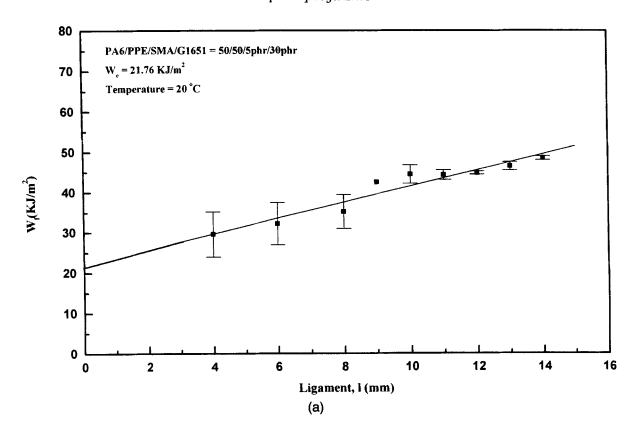
Table 1. Summary of Impact Fracture Toughness Analyzed by Different Methods.

Composition	w <sub>e</sub> (kJ/m²)²	$\beta w_p  (MJ/m^3)$	$G_c(kJ/m^2)^b$
20°C			
PA6/PPE/SMA/G1651 = 50/50/5/15	<b>油油油油</b>	***	4.73
PA6/PPE/SMA/G1651 = 50/50/5/20	***	****	10.58
PA6/PPE/SMA/G1651 = 50/50/5/25	****	****	****
PA6/PPE/SMA/G1651 = 50/50/5/30	21.76	1.99	****
40°C			
PA6/PPE/SMA/G1651 = 50/50/5/15	****	****	17.80
PA6/PPE/SMA/G1651 = 50/50/5/20	****	****	****
PA6/PPE/SMA/G1651 = 50/50/5/25	18.42	1.40	****
PA6/PPE/SMA/G1651 = 50/50/5/30	22.25	2.35	****
70°C			
PA6/PPE/SMA/G1651 = 50/50/5/15	****	****	8.61
PA6/PPE/SMA/G1651 = 50/50/5/20	18.53	1.80	****
PA6/PPE/SMA/G1651 = 50/50/5/25	20.74	1.78	****
PA6/PPE/SMA/G1651 = 50/50/5/30	22.97	2.54	****
100°C			
PA6/PPE/SMA/G1651 = 50/50/5/15	20.00	1.30	****
PA6/PPE/SMA/G1651 = 50/50/5/20	22.00	1.62	****
PA6/PPE/SMA/G1651 = 50/50/5/25	24.95	1.53	****
PA6/PPE/SMA/G1651 = 50/50/5/30	26.26	2.13	****

 $w_t = w_e + \beta w_e$ 

 $<sup>{}^{</sup>b}W_{t} = G_{c}W\Phi t.$ 

<sup>\*\*\*\*\*</sup> Method of analysis inapplicable.



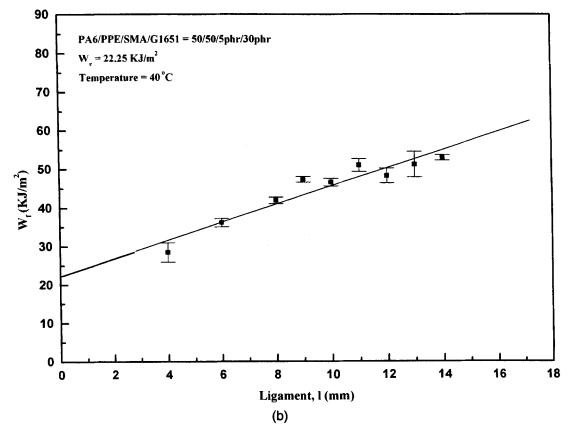
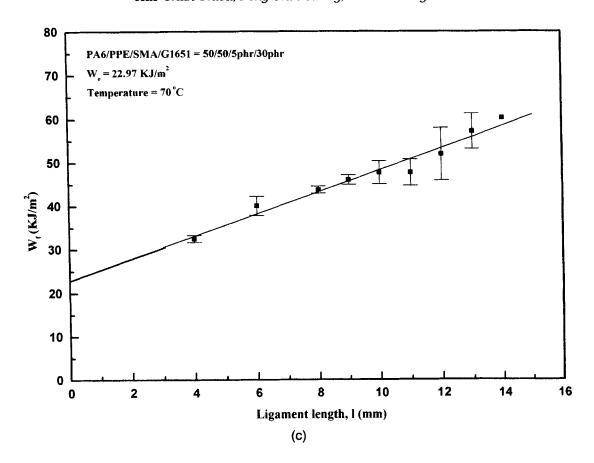


Fig. 3. Plots of corrected impact energy absorption  $(W_f)$  against ligament l for elastomer-modified PA6/PPE/SMA (50/50/5) blends at different temperature. (a)  $20^{\circ}$ C, (b)  $40^{\circ}$ C, (c)  $70^{\circ}$ C, (d)  $100^{\circ}$ C.



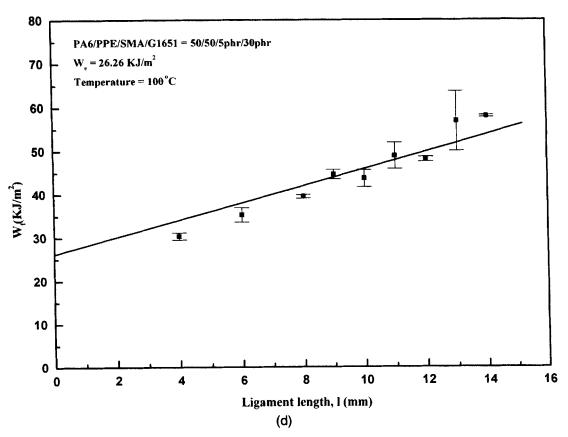
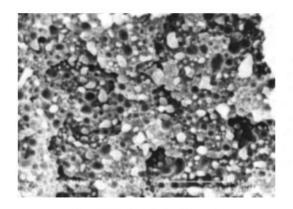
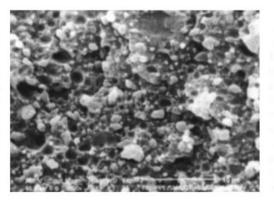


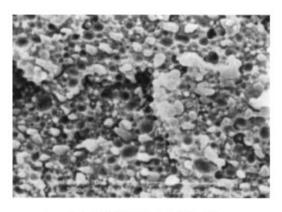
Fig. 3. Continued.



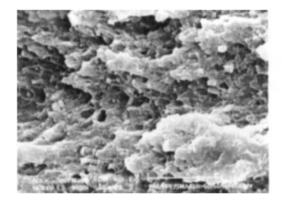
(a) PA6/PPE/SMA/G1651 = 50/50/5phr/15phr Brittle (3,500X)



(b) PA6/PPE/SMA/G1651 = 50/50/5phr/20phr Brittle (3,500X)



(c) PA6/PPE/SMA/G1651 = 50/50/5phr/25phr Brittle (3,500X)



(d) PA6/PPE/SMA/G1651 = 50/50/5phr/30phr Ductile (3,500X)

Fig. 4. SEM micrographs of fracture surfaces by Charpy impact tests at  $20^{\circ}\text{C}$  .

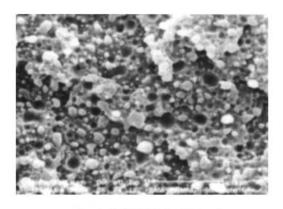
unstable fracture with a finite yield strip on either surface. The EWF method cannot be applied for these two blends as shown in *Table 1*. If *Eq 4* is forced to plot the corrected impact energies against the ligament length, a straight line with a negative slope will be obtained. This is because the energy absorption in the stress-whitened or plastically deformed zone is approximately independent of ligament length so that the specific total work of fracture decreases as the ligament length increases. Similar results supported by photographic evidence (of the stress-whitened zone in the ligament) have also been reported for a random PP copolymer subjected to impact testing in (17, 19).

It is interesting to note that  $\beta w_p$  generally increases with elastomer content for a given temperature. This indicates that the slope of the EWF plot may be regarded as a useful measure of the fracture resistance to crack growth and it reflects the compositional difference of these blends provided the same specimen geometry is used for comparison.

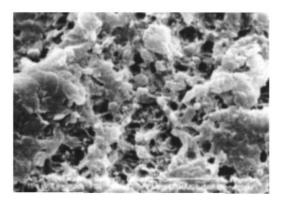
## **Morphologies of Fracture Surfaces**

Before discussing the morphology of the fracture surfaces, we need to explain the microstructure of the blends. In the PA6/PPE/SMA (50/50/5) blends, the continuous matrix is PA6 and the dispersed particles are PPE. Elastomer G1651 is not distinguishable from the fracture surfaces by SEM and it is embedded in the dispersed PPE phase since G1651 is a SEBS copolymer that is compatible with PPE but incompatible with the PA6 matrix. Fracture surfaces from the impact specimens at different temperatures and elastomer contents show three distinctly different features: brittle, semi-brittle and ductile. Figure 4 shows SEM micrographs of elastomer-modified PA6/PPE/SMA (50/50/5) blends fractured by Charpy impact tests at 20°C. Figures 4a and 4b present fast-fracture brittle surfaces where the dispersed PPE particles including G1651 elastomer particles are pulled out from the matrix. When shear yielding of the PA6 matrix slowly increases with elastomer content as shown in Fig. 4c, the dispersed PPE particle pullout mechanism becomes less dominant. Figure 4d shows the ductile fracture surface of the PA6/PPE/SMA/G1651 (50/50/5/30) blend where shear yielding becomes the dominant mechanism. SEM micrographs of the impact-fractured specimens at 40°C are illustrated in Fig. 5. Figure 5a shows a brittle fracture surface dominated by dispersed particle pullout mechanism in a blend with 15 phr elastomer. Figures 5b to d show the three regions of semi-brittle fracture, ductile-brittle transition, and ductile fracture, respectively, in a blend with 20 phr elastomer. In the ductile region it can be seen that both shear yielding and particle pullout co-exist. As the elastomer content is increased to 30 phr, Fig. 5e, fracture remains ductile but the extent of shear yielding on the fracture surface is more extensive when compared to Fig. 5d.

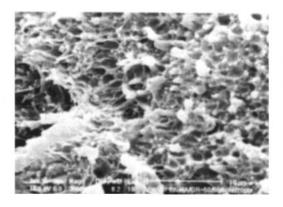
There also exists a ductile-brittle transition at testing temperature of 70°C for the PA6/PPE/SMA/S1651 (50/50/5/15) blend. As shown in Fig. 6a, brittle fracture is controlled by the particle pullout mechanism. Figure 6b shows the fracture surface profile including the regions of pre-crack, damage zone and fast crack growth for this blend for which the ligaments have not been fully yielded. There is a distinguishable ductilebrittle transition between the two regions where a short stable crack growth (damage zone) is followed by unstable fast crack propagation. A high magnification of the damage zone is shown in Fig. 6c. Though there are many micro-voids on the fracture surface, massive matrix tearing dominates its fracture behavior. Again as the elastomer contents are increased to 20 and 30 phr in the blends, ductile fracture surfaces become predominant as illustrated in Figs. 6d and 6e, where the ratio of shear yielding to particle pullout



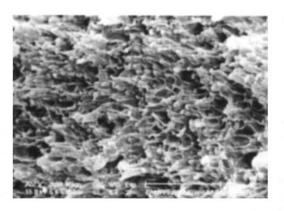
(a) PA6/PPE/SMA/G1651 = 50/50/5phr/15phr Brittle (3,500X)



(b) PA6/PPE/SMA/G1651 = 50/50/5phr/20phr Semi-Brittle (3,500X)

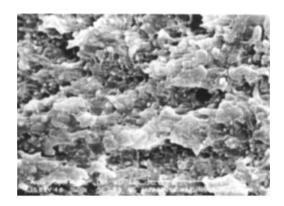


(c) PA6/PPE/SMA/G1651 = 50/50/5phr/20phr (3,500X) Ductile-Brittle transition



(d) PA6/PPE/SMA/G1651 = 50/50/5phr/20phr Ductile (3,500X)

Fig. 5. SEM micrographs of fracture surfaces by Charpy impact tests at 40°C.



(e) PA6/PPE/SMA/G1651 = 50/50/5phr/30phr Ductile (3,500X)

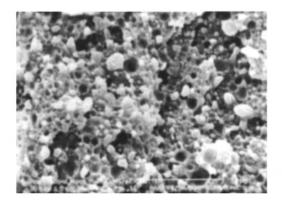
also increases. At 100°C all blends possess high impact energies and their fracture behavior is completely ductile. Figure 7 shows their fracture surfaces. Matrix shear yielding is dominant in these blends and dispersed particle pullout becomes insignificant and finally disappears as elastomer content increases to 30 phr. To sum up, these two fracture mechanisms, matrix shear yielding and particle pullout, compete with each other in the elastomer-modified PA6/PPE/SMA (50/50/5) blends. Shear yielding is pre-dominant in ductile fracture and particle pullout in brittle fracture. Impact fracture toughness can be evaluated by the EWF method to determine the specific essential work of fracture,  $w_e$ .

The depth of the outer plastic zone, D, as defined in Fig. 1b, is typified in Fig. 8a and b for the blends at

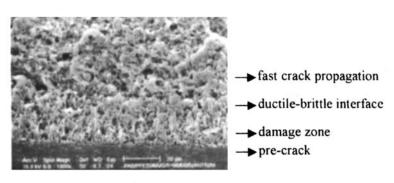
40°C and 100°C, respectively. There is an approximately linear relationship between D and the ligament length l hence supporting the postulate that the nonessential work is proportional to  $l^2$  in deriving Eq 4. (Similar plots of D vs. l are obtained for the blends at other test temperatures used in this study). It is also noted that the depth of the plastic deformation zone increases with temperature; but the effect of the elastomer content is not so easily discernible. However, these results confirm that high temperature and generally high elastomer content will impart high energy absorption to these blends during impact fracture.

#### **CONCLUSIONS**

There is a need to establish a standardized method to measure the impact fracture resistance of newer and tougher polymeric materials. The essential fracture work (EWF) method can be utilized to determine the high-rate specific essential work of fracture,  $w_e$ , for the elastomer-modified PA6/PPE/SMA (50/50/5) blends by Charpy notched impact tests if all requirements for the validity of the method are met. For a fully yielded specimen, the essential fracture work is proportional to the ligament length and the non-essential work of fracture to the square of the ligament length. It is found that  $w_e$  increases with testing temperature and elastomer content for a given sheet thickness. Morphologically, there are two fracture mechanisms, PA6 matrix shear yielding and pullout of second phase dispersed particles of PPE, that compete against each other. Shear yielding is dominant for ductile failure and particle pullout is typical of brittle fracture. Moreover, the depth of the plastic deformation zone increases linearly with ligament length for those blends that exhibit ductile failure, hence supporting the essential work of fracture method for impact toughness characterization.

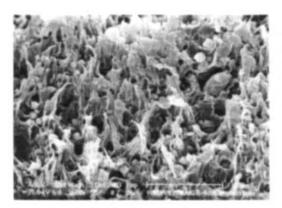


(a) PA6/PPE/SMA/G1651 = 50/50/5phr/15phr Brittle (3,500X)

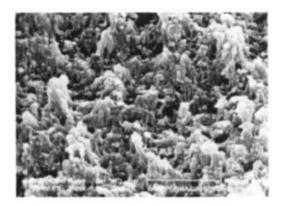


(b) PA6/PPE/SMA/G1651 = 50/50/5phr/15phr Brittle, precrack+damage zone (1,000X)

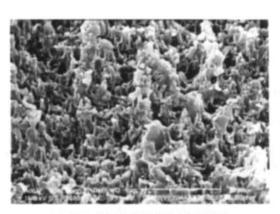
Fig. 6. SEM micrographs of fracture surfaces by Charpy impact tests at 70°C.



(c) PA6/PPE/SMA/G1651 = 50/50/5phr/15phr damage zone (3,500X)

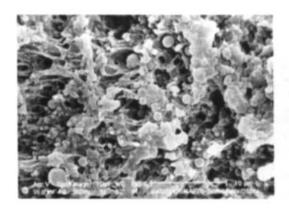


(d) PA6/PPE/SMA/G1651 = 50/50/5phr/20phr Ductile (3,500X)



(e) PA6/PPE/SMA/G1651 = 50/50/5phr/30phr Ductile (3,500X)

Fig. 6. Continued.

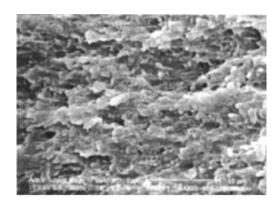


(a) PA6/PPE/SMA/G1651 = 50/50/5phr/15phr Ductile (3,500X)



(b) PA6/PPE/SMA/G1651 = 50/50/5phr/20phr Ductile (3,500X)

Fig. 7. SEM micrographs of fracture surfaces by Charpy impact tests at  $100^{\circ}$ C.



(c) PA6/PPE/SMA/G1651 = 50/50/5phr/30phr Ductile (3,500X)

#### **ACKNOWLEDGMENTS**

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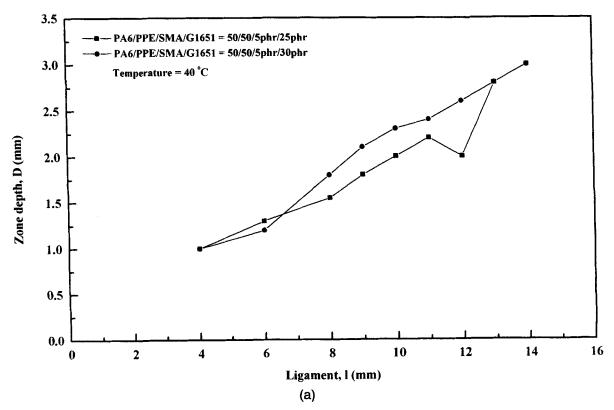


Fig. 8. Dependence of outer plastic zone depth, D, on ligament length, L, at (a)  $40^{\circ}$ C and (b)  $100^{\circ}$ C.

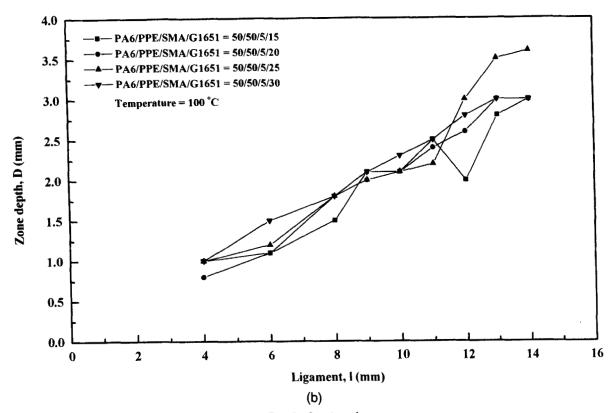


Fig. 8. Continued.

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