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ASSESSMENT OF IMPACT FRACTURE TOUGHNESS OF PP–ELASTOMERIC POLYOLEFIN BLENDS

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ABSTRACT

The present investigation is concerned with evaluation of the impact fracture toughness of novel blends based on commercial polypropylene homopolymer (PPH) and elastomeric polyolefin (POEs). At room temperature and high load rate, PPH behaves in a brittle manner while the blends exhibited semi-brittle behavior as judged from the nonlinearity in load deflection curves and fracture surface appearance. The challenge of determining reliable toughness values was faced by applying different approaches available in the literature based on fracture mechanics concepts, including corrected linear elastic fracture mechanics (LEFM), equivalent energy concept, and non-LEFM. The fracture toughness data of the blends appeared to be widely scattered in accord with the samples being in the ductile–brittle transition region. In order to provide a consistent description of the entire range of sample behavior, the statistical weakest link model was also applied to the data.

Key Words: Polypropylene-polypropylene-elastomeric polyolefin blends; Non-linear fracture; Weakest link model; Ductile-brittle transition

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INTRODUCTION

The great attraction in plastics as engineering materials is the way in which their properties can be tailored to a specific need. The lack of toughness at high testing rate and room temperature, combined with high notch sensitivity, exhibited by PP homopolymer hamper its high performance applications. As its brittleness was explained in terms of the vicinity of its T_g to room temperature, the usual way to achieve better impact properties for PP homopolymer is by adding elastomers. Blending polypropylene with rubber particles allows lowering the brittle-to-ductile transition temperature with a corresponding increase in toughness.

Fracture properties of several polypropylene–rubber blends, such as PP modified with EPR or EPDM, have been extensively studied due to their technological value.[1–6] However, recent developments of commercial metallocene catalyzed thermoplastic polyolefin elastomers have been suggested to create new frontiers for polypropylene modification.[7] Although a recent article has evaluated the impact performance of these novel blends by using standard tests,[8] the fracture and deformation behaviors of polypropylene homopolymer (PPH) toughened by metallocene catalyzed polyethylene elastomer are still unknown.

In this article, blends based on commercial PPH and elastomeric metallocene catalyzed polyolefin (POEs) are investigated under impact loading in the 0–30% range.

As is well known, the use of fracture mechanics theory allows description of the toughness of a material by parameters which are true material properties. However, the toughness determination in the framework of fracture mechanics theory is conditioned to the behavior displayed by the material itself. Up to date, very few procedures have been standardized[9–12] in the polymer field. Linear elastic fracture mechanics (LEFM) has been widely applied to the evaluation of the fracture behavior of brittle polymers when the size of the plastic zone is much smaller than the in-plane specimen dimensions and the initiation of unstable fracture can be accurately described by K_{IC} or G_{IC} parameters. The most widely accepted method to determine the high rate fracture toughness (around 1 msec $^{-1}$) for linear-elastic polymeric materials behavior is the G_{IC} methodology[13–15] since it avoids the need of determining the Young's modulus (E) at a reliable test rate. On the other hand, for tough polymers, elastic-plastic fracture mechanics (EPFM) concepts, such as the J-Integral, have been introduced[16–18] inspite of the inherent difficulties of J methodology in dynamic loading conditions.

Since the role of elastomer modification is to promote crack tip plasticity, nonlinearity in the force displacement curves is inevitable and fully linear elastic behavior is generally not observed in toughened blends.[12,18–20] Currently a suitable methodology to determine fracture toughness in the semi-brittle regime for polymers does not exist. The direct application of LEFM or EPFM under the

latter situation is not possible since the required conditions are only partially met.

Fracture mechanics approaches, which implies the use of instrumented test curves and which take into account the characteristic deviations from extreme behaviors, were tried in order to analyze experimental data obtained for these novel blends. The mentioned methodologies are: Corrected LEFM, equivalent energy concept and the non-LEFM. The different fracture toughness parameters calculated are compared and their pertinence is also discussed.

EXPERIMENTAL

Experiments were conducted on blends based on commercial PPH (Cuyolen NX1100, from Petroquímica Cuyo SAIC) and elastomeric polyolefin (POEs) (ENGAGE 8100 from Dow Chemicals) up to 30 %wt. Blends prepared in a twin screw extruder were kindly provided by Petroquímica Cuyo SAIC in the form of pellets.

Pellets were compression molded into 8 mm thick plaques at 200°C. Plaques were then annealed for 1 hr at 120°C in order to release residual thermal stresses generated during molding. The morphology of the PPH–POEs blends has been determined elsewhere, they form immiscible phase separated blends with the spherical elastomeric inclusions randomly dispersed in the neat PPH matrix.[8,21,22]

Bars for fracture experiments were cut from the compression molded plaques and then machined to reach the final dimensions and improve edge surface finishing. Sharp notches were introduced by scalpel-sliding a razor blade having an on-edge tip radius of 0.13 μm.

Impact testing was carried out using a Fractovis Ceast Falling weight type machine. The spurious contributions to the measured energy due to machine compliance and specimen indentation were corrected following usual practices[11,12] at 0.5 m/sec.

Pre-cracked specimens were tested in three point bending at room temperature and at 1 m/sec. The specimen thickness, B, and the span to depth ratio, S/W, were always kept equal to $W/2$ and 4, respectively.

The original crack length, a, and the stress whitened zone length r_p were physically measured from the fracture surface using a profile projector with a magnification of 20 ×. Brittle or semi-brittle fracture propagation regimes were judged by the shape of the load–displacement curves and confirmed from the fracture surface appearance examined by optical microscopy. Fracture surfaces were also analyzed by SEM.

Elastic modulus of the materials at high strain rate was estimated using unnotched bend specimens according to the procedure proposed by Grellmann et al.[23] Yield strength was evaluated according to the normalized uniaxial test at low deformation rate.[24]

DATA ANALYSIS

Energy Release Rate, G_{IC} Determination

ISO/DIS 17281 Standard[11] states that for brittle behavior, a basically linear relationship exists between the fracture energy, U; and the energy calibration factor, ϕ. This relationship allows, by testing specimens having a crack depth ranging from 0.3 to 0.7 (a/W), to calculate the critical strain energy release rate G_{IC} for unstable fracture from the slope of the U vs. the product of the specimen dimensions and the energy calibration function ($BW\phi$) plot. U is the energy absorbed by the specimen during fracture, B and W are the specimen thickness and width, respectively, and the calibration factor, ϕ, depends on the length of crack size of the sample (a_0).

In the case of limited plasticity, the corrected elastic fracture mechanics concept may be applied.[25] It basically consists in replacing a_0 by an effective crack length $a_{eff} = a_0 + r_p$, then calculating G_{IC} following the normal elastic procedure. For the latter calculations, r_p was taken to be the white halo denoted on the post mortem fracture surfaces without distinguishing between sub-critical crack growth and true plastic deformation zone.

For G_{IC} determination, U was taken as the energy absorbed up to the maximum load F_{max} as shown schematically in Fig. 1a.

Stress Intensity Factor, K^{IC}_{max} Determination

Alternatively, the stress intensity factor K_{IC} was calculated according to the standardized procedure,[19,11] via the following equations:

$$K_{IC} = \frac{F}{B\sqrt{W}} f\left(\frac{a}{W}\right) \tag{1}$$

with

$$f\left(\frac{a}{W}\right) = \frac{3\frac{S}{W}\sqrt{\frac{a}{W}}}{2\left(1+2\frac{a}{W}\right)\left(1-\frac{a}{W}\right)^{3/2}} \times \left\{1.99 - \frac{a}{W}\left(1-\frac{a}{W}\right)\left[2.15 - 3.93\left(\frac{a}{W}\right) + 2.7\left(\frac{a}{W}\right)^2\right]\right\} \tag{2}$$

for single edge notched bend specimens SE(B), where F is the load, W is the specimen width, and S is the support span. Through this paper F was taken as the maximum of the load displacement trace (see Fig. 1); thus the toughness parameter obtained is K^{IC}_{max} which may defer from the real critical parameter K_{IC}. This approach was selected since it had been proven to provide a consistent description of the entire range of sample behavior in other similar systems.[19]

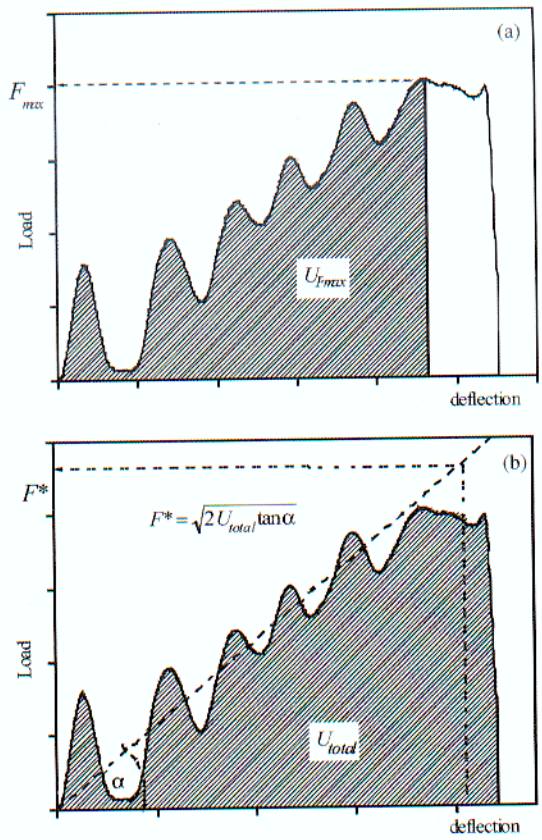

Figure 1. Example of energy and load determination used in the applied methodologies. (a) Linear elastic methods and (b) equivalent energy method.

Equivalent Energy Method, K_{IC}^{E} Determination

The equivalent energy method,[26] developed by Witt[27] in order to extend LEFM to metals where yielding has occurred prior to reaching maximum load, has also been applied to polymers.[28] Briefly, this concept states that an equivalent amount of energy would have been necessary to reach maximum load had the specimen been thick enough to avoid general yielding prior to fracture (that is, had the specimen been thick enough for plane-strain conditions to have existed). This methodology (ASTM E992-84) requires the experimental estimation of a pseudoelastic load F^* which is related to the deformation energy U and the initial slope, $\tan\alpha$, of load–displacement plots through Eq. (3) which then replaces F in Eq. (1) (Fig. 1b).

$$F^* = \sqrt{2U\tan\alpha} \tag{3}$$

Nonlinear Elastic Fracture Mechanics, J_c Determination

The J_c parameter[29,30] is applicable to characterize quasi-brittle failure behavior (quasi linear load–displacement curves with sharp load drop at the point of fracture). The J- integral was evaluated at the instability load point (Eq. (4)), by calculating the fracture energy required to produce cleavage behavior.

$$J_c = \frac{2U_{tot}}{B(W-a)} \tag{4}$$

Although ASTM E813-87 and ASTM E1152-87 apply only to ductile fracture, more recent standards permit J testing of materials that fail by cleavage.[29] It has been shown that J_c can be applied to polymers if the critical J values are independent of specimen size.[31]

Weakest Link Theory, Weibull Function

A three-parameter Weibull cumulative frequency distribution[32,33] was chosen to fit the distribution of J_c values.

$$P_f = 1 - \exp\left[-\left(\frac{J_c - J_{min}}{\theta_J - J_{min}}\right)^b\right] \tag{5}$$

$$P_f = \frac{i - 0.3}{N + 0.4} \tag{6}$$

where P_f is the cumulative probability of failure, N the number of tests, and i the rank corresponding to each N value. The three fitting constants are the scale parameter, θ_J (J_c equals θ_J when P_f equals 0.632), the Weibull slope b which is the shape of the distribution and is a measure of the relative scatter, and, J_{min}, the lower-bound toughness value that defines a lower limiting toughness for specimens of infinite thickness. The three fitting parameters were simultaneously determined by minimum squares. The toughness density function, Pd_f, can then be obtained from the model parameters by analytical derivation of P_f with respect to J_c. Then the most frequent toughness value (median value) can be inferred from the maximum of the toughness density function.[33]

Comparison of Parameters

For comparative purposes, it is preferable to convert J_c and G_{IC} to equivalent K_{IC} values through the following relationship[28,34]:

$$K_{IC}^G = \sqrt{G_{IC}E^*} \tag{7}$$

$$K_{IC}^J = \sqrt{J_cE^*}$$

where E^* is the E for the plane stress state, E^* is $E/(1 - \nu^2)$ for the plane strain state, and ν is Poisson's ratio.

Size Requirements Conditions

In order to check the geometry independency of the toughness parameters, the size requirements for the different methodologies were evaluated.

The size requirement for a valid plane strain fracture toughness K value (K_{IC}) is given by Ref. [9]:

$$B, (W - a_0), \quad a > \alpha\left(\frac{K}{\sigma_y}\right)^2 \tag{8}$$

where σ_y is the yield stress and α has an empirical value of 2.5. However, it has recently been shown that this empirical value (obtained from metal results) overestimates the size requirements for polymers[28].

The size requirement for a J_{Ic} is given by:

$$B, (W - a_0), \quad a > \beta\left(\frac{J}{\sigma_y}\right) \tag{9}$$

where β has a value of 25. Likewise, it was recently found that β is somewhat higher for polymeric materials[35] and also dependent on the material properties:[28]

$$\beta = 224J^{-0.94} \quad \text{with } J \text{ in N/mm} \tag{10}$$

In both cases the criteria adopted yield conservative size values since the yield stress was evaluated at low strain rate representing a lower bound value.

RESULTS AND DISCUSSION

Phenomenology

Striking differences in fracture behavior were observed between neat polypropylene and POEs modified blends. Typical load–time curves of materials obtained during instrumented impact tests are given in Fig. 2. Superimposed oscillations of the signal are due to the well-known dynamics effects.[14] This diagram shows a transition in the mechanical behavior. The transition occurs from pure elastic (PPH) to elastic–plastic material behavior with predominantly unstable crack growth (PPH/POEs blends).

The PPH samples fractured in an unstable brittle way. Load–time curves dropped to zero instantaneously upon reaching the maximum load at relatively short time levels (Fig. 2). Consistently, the fracture surface features were mirror-smooth without stress whitening (Fig. 3a).

On the other hand, the blends exhibited semi-brittle behavior.[19,28] The load increased nonlinearly and displayed a drastic drop at large time values

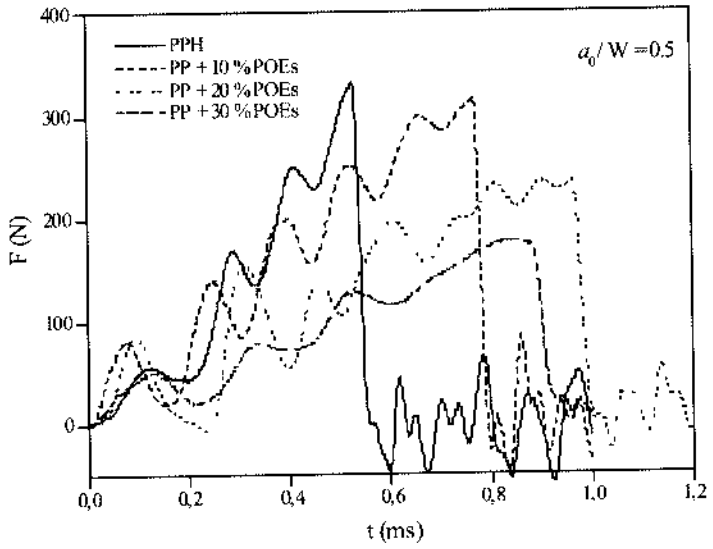

Figure 2. Typical load–time curves obtained at 1m/sec.

(Fig. 2) in coincidence with sample failure. The pattern exhibited by the modified materials may be caused by plasticity, sub-critical crack growth, or both phenomena. Post-mortem fracture surface analysis revealed two distinct zones: a rough and whitened zone developed ahead of the crack tip followed by a smooth mirrorlike zone (Fig. 3b). Neither the naked eye nor scanning electron microscopy (SEM) inspections allowed us to undoubtedly discern whether this damage zone corresponds to plastic deformation or stable crack growth (Fig. 4). Nevertheless, in some specimens it was possible to observe what was presumed to be a small amount of sub-critical crack growth, (Δa), especially for 20% POEs samples. Hence, if stable crack growth is present, it can be considered negligible compared with the starter crack and the damage zone length.

Fracture surface analysis revealed that the 20% POEs samples exhibited a large plastic zone developed at crack tip concomitantly with the maximum in the impact duration (Fig. 2). It clearly emerged that this composition has the largest tendency towards gaining stability in crack propagation.

Even though the load-line traces (Fig. 2) are similar to that of other PP–rubber blends in which stable propagation was reported,[28,36] completely stable behavior was not displayed by any of the tested specimens.

Quantitative Assessment

Impact fracture toughness values determined by the different approaches explained in the section "Data Analysis" are summarized in Tables 1 and 2.

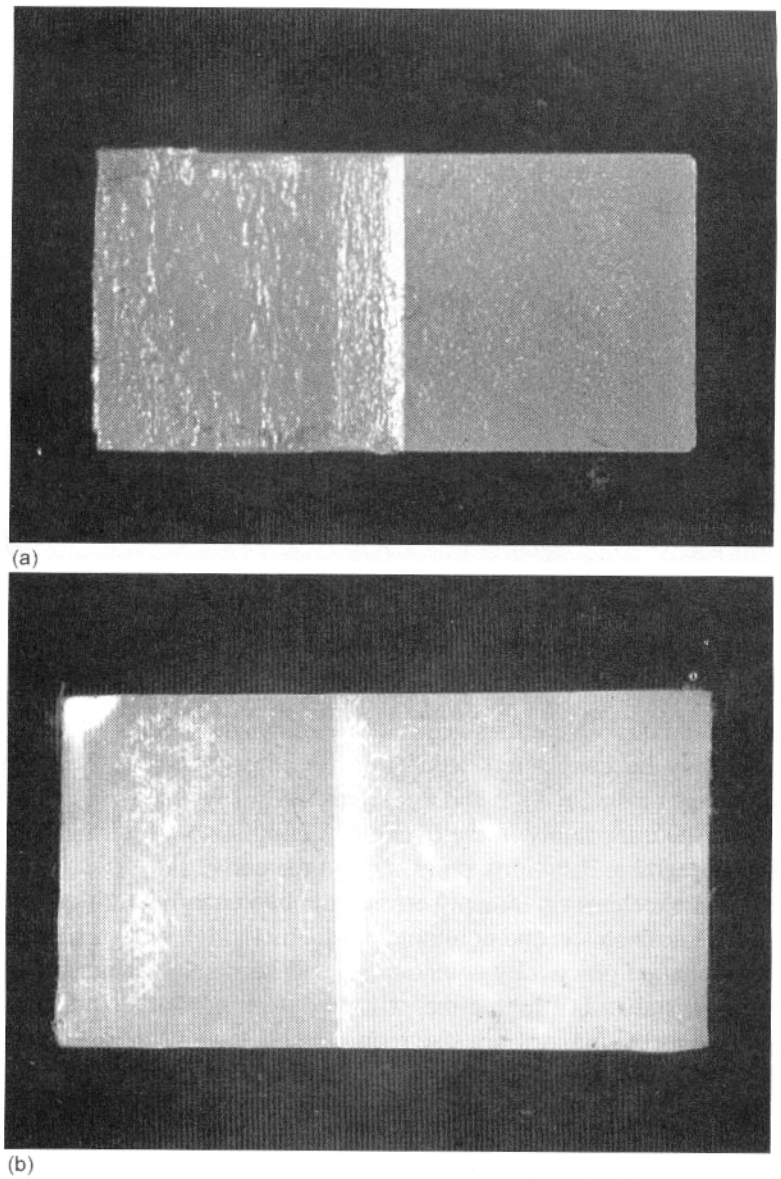

Figure 3. Fractographs of the fracture surfaces of (a) PPH, (b) PP + 10% POEs fractured at 1 m/sec.

Brittle Regime

The impact fracture behavior of PPH samples, which occurred in the elastic regime, seems to be well represented by the elastic fracture mechanics-based methodologies. The critical energy release rate (G_{IC}) plot is shown in Fig. 5 (filled squares and solid line). The linearity of the regression line judged by the

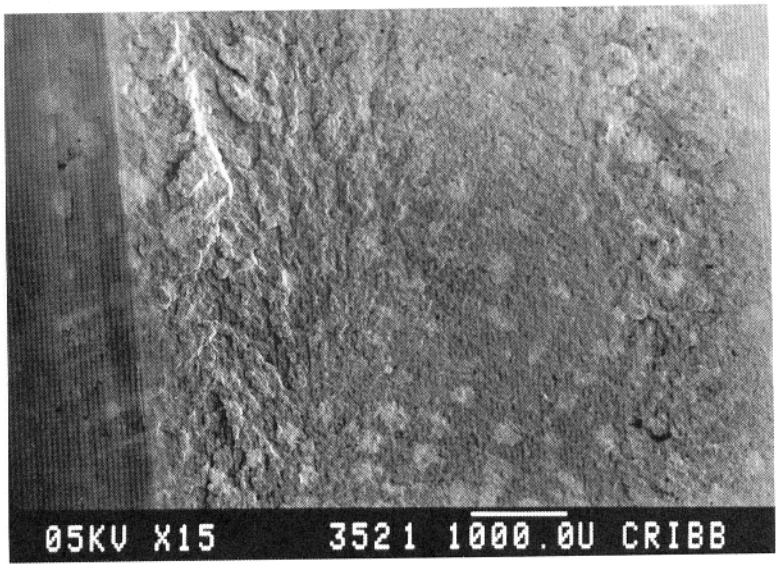

Figure 4. SEM micrographs of the fracture surface of PP + 10% POEs fractured at 1m/sec.

correlation factor value (see R^2 in Table 1) is quite good. The stress intensity factor (K_{IC}^{max}) and its 95% confidence limits are shown in Table 1. The scatter of the data points may be considered acceptable for impact testing. The differences obtained between average K_{IC}^{max} and the inferred K_{IC}^{G} [obtained via Eq. (7)] seems to arise from the uncertainty in the dynamic Young's modulus value.

The J-Integral at instability point, J_c, and the derived K^{J_c} [Eq. (7)] are given in Table 2. An excellent agreement between the two energetic critical parameters derived from linear-elastic and nonlinear elastic methodologies, respectively, was found. J_c results are practically equal to G_{IC} and hence K_{IC}^{G} coincides with K^{J_c}.

In the unstable brittle regime, all proposed methodologies are strictly applicable and as the plane strain size requirements have been met (see Table 3), real critical fracture toughness parameters could be determined.

Table 1. Fracture Toughness Determined from Linear Elastic Fracture Mechanics Based Methodologies

	Elastic and Corrected Elastic			Equivalent Energy
Material	G_{IC} [N/mm] (R^2)	K_{Ic}^{G} [MPa m$^{1/2}$] Eq. (7)	K_{Ic}^{max} [MPa m$^{1/2}$] ($K \pm 2$sd)[a]	K_{Ic}^{E} [MPa m$^{1/2}$] ($K \pm 2$sd)[a]
PPH	1.04 (0.90)	1.48	1.72 ± 0.32	—
PP + 10% POEs	3.57 (0.79)	2.51	2.67 ± 0.46	2.51 ± 0.54
PP + 20% POEs	4.28 (0.73)	2.46	2.34 ± 0.56	2.31 ± 0.22
PP + 30% POEs	2.71 (0.83)	1.71	1.99 ± 0.48	1.83 ± 0.64

[a] 95% confidence limits for K.

Table 2. Fracture Toughness Determined from Non Linear Elastic Method and Weibull Model

	Weibull Analysis				
	Fitting Parameters			Toughness parameters	
Material	J_{min} [N/mm]	θ_J [N/mm]	b	$J_{c_{mean}}$ [N/mm]	$K^{J_c}_{mean}$ [MPa m$^{1/2}$]
PPH	—	—	—	1.08	1.51
PP + 10% POEs	3.02	4.20	1.28	3.37	2.43
PP + 20% POEs	2.41	4.24	1.52	3.30	2.16
PP + 30% POEs	1.65	3.12	3.14	2.73	1.72

Semibrittle Regime

As a first attempt to evaluate the toughness of the blends in the semibrittle transition region, the traditional corrected elastic method was tried. As can be observed in Fig. 5, the data appear widely scattered and a good linear fitting could not be obtained for any of the blends (see R^2 in Table 1). The inconsistency of the methodology arises from the excessive damage zone developed before crack propagation, which violates the model assumption.[14] Further, G_{IC}^{C} can not be considered a relevant fracture parameter in order to assess the fracture toughness of the studied blends.

The blend's fracture behaviors were then examined by the equivalent energy concept and compared with the linear elastic stress intensity factor. Consistent

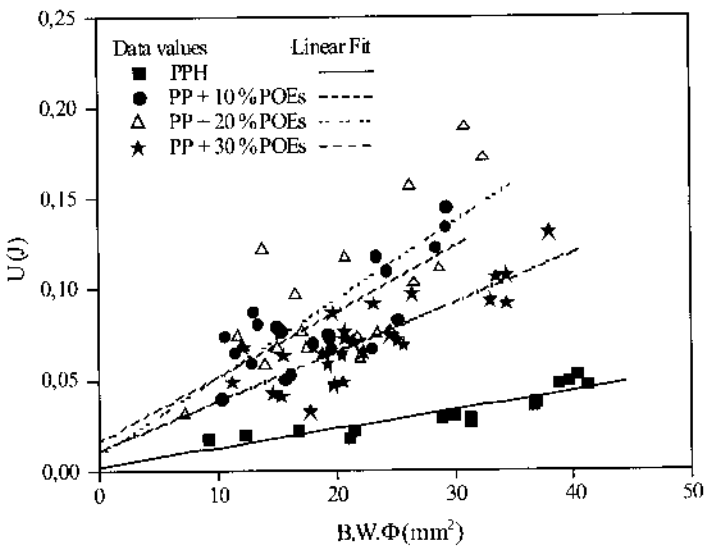

Figure 5. Determination of fracture parameters. Linear elastic (G_{IC}) and corrected linear elastic (G_{Ic}^{C}) methodologies.

Table 3. Plane Strain Size Requirements of Fracture Parameters

Material	σ_y (MPa)	Nominal B (mm)	Size Requirement $B \geq \ldots$ (mm) For K_{IC} (Eq. (8))	For J_c (Eq. (9))	For J_c (Eqs. (9) and (10))
PPH	37.1	8.5	7.5	0.7	6.1
PP + 10% POEs	33.2	7	21.0	2.5	7.3
PP + 20% POEs	29.3	7.5	18.7	2.8	8.2
PP + 30% POEs	25.3	7.5	23.8	2.7	9.4

with its definitions, the energy equivalent concept K_{IC}^{U} led to slightly lower values than K_{IC}^{max}, but both methodologies yield practically the same results. On the other hand, a closer look at the blends results revealed that the +95% confidence limits (see Table 1) are too wide and the scatter bands overlap. Size independency of parameters has not been met (see Table 3). Under this situation, an average toughness value lacks significance.

Experimental data was then analyzed in terms of J_c and results are shown in a three parameters Weibull type plot in Fig. 6 [Eqs. (5) and (6)]. It can be observed that the data values appear widely scattered (see b parameter in Table 2) even though the size requirements were practically met (see Table 3). Some specimens, even if theoretically identical, failed with little plastic deformation while other specimens had large stress whitening zones prior to cleavage, as can be observed

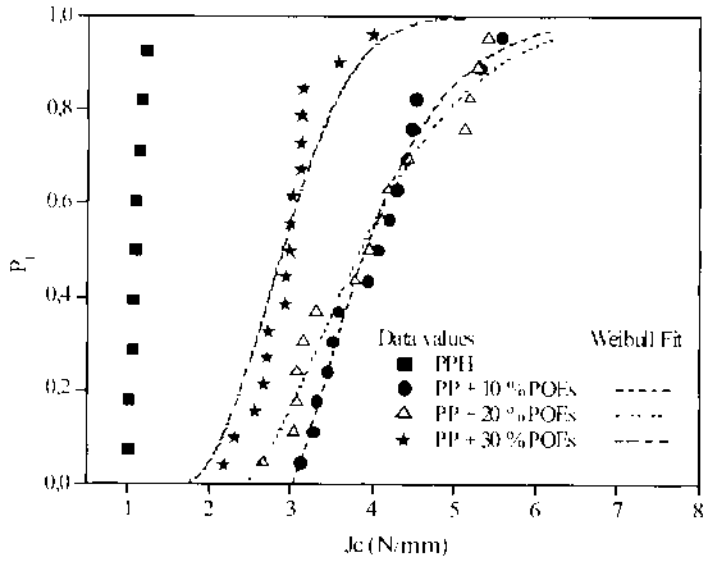

Figure 6. Data scatter of J_c values in terms of a three parameter Weibull model.

in the Weibull analysis given in Fig. 7 (the weakest link model was also applied to damage zone length values and density distributions derived).

The various methods predict the same material's ranking and the same trend for mean parameters (see Tables 1 and 2); however, the scatter bands corresponding to the parameters obtained for the different compositions overlapped. Hence, meaningful parameters cannot be obtained by simple averaging the values obtained from the repeated runs.

From the above described results, we can obtain a simple conclusion: independently of the approach used to assess the blend's fracture toughness, the data are widely scattered and seem to be better described by a distribution of values rather than a single value (see the toughness density functions in Fig. 8). The scatter is inherent to the material behavior since increasing the number of determinations could not eliminate it. A previous work carried out on a similar neat PP commercial grade under static conditions showed that the PP toughness values were also widely scattered and different levels of ductile tearing before cleavage were reached.[37] The present investigation clearly shows that dynamic loading suppressed PP homopolymer ductile tearing and the concomitant scatter, hence allowing use of traditional linear elastic methodologies with confidence. It is worth pointing out that the predicted toughness parameter value, $J_c = 1.08\,\mathrm{N/mm}$, is well below the lower-bound value found under static conditions, $J_{min} = 6\,\mathrm{N/mm}$.[37] On the other hand, elastomer modification improves the fracture behavior of PP shifting the ductile–brittle transition to a higher speed testing. Our findings are consistent with those of Gensler et al.[38] who also reported scattering in toughness measurements in similar blends associated with

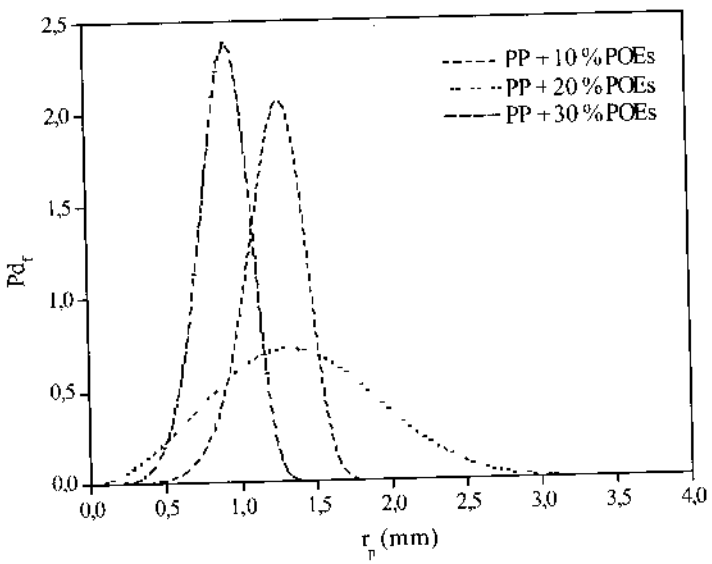

Figure 7. Variation of the distribution of damage zone length (r_p) at cleavage instability according to a two parameter Weibull model.

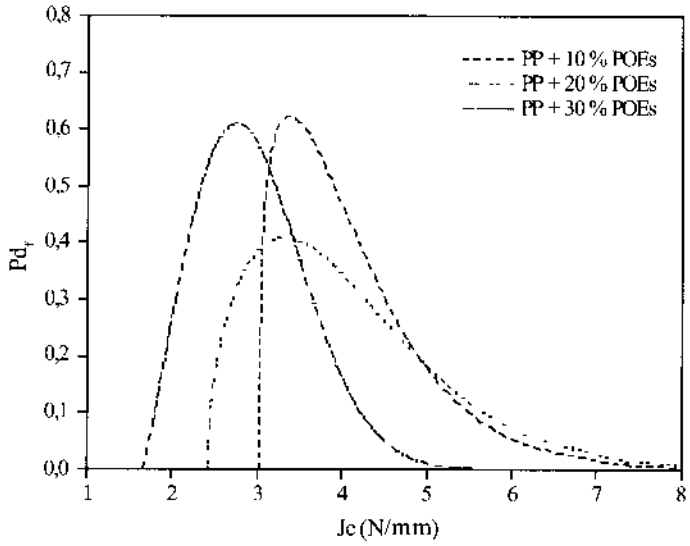

Figure 8. Toughness density functions derived from Weibull analysis: Prediction of median J_c values.

the competition between mechanisms that promote ductile tearing (shear yielding) and cleavage fracture (crazing)[139] under the testing conditions used.

Scatter in Toughness Values

The most developed approach for explaining scatter and size effects of fracture toughness has been the weakest link model, which has been applied to data for steels in the ductile–brittle transition region.[140] This theory assumes that small regions of very low toughness, called weakest links, are randomly distributed in the material. Failure occurs if at one of these weakest links the critical stress is reached. The fracture load depends on the location of the "weak link" in the volume ahead of the crack tip and on the critical stress of the individual weak link. This statistical model seems to fit J_c distribution quite well as can be observed from Fig. 6, and hence allows us to set a J_{min} parameter (see Table 2), which is a lower bound toughness in the ductile to brittle transition region. This J_{min} parameter is useful as a design parameter and also for comparative purposes.[141]

The best overall performance was displayed by the 10% elastomer blend. Further elastomer incorporation was less effective in toughening (Table 2). The larger scatter in the 20% elastomer blend originates from the large trend to stability (ductile tearing) before cleavage failure.[142] The nature of the impair next in properties shown by blends containing more than 20 %wt POEs will be further discussed in a future paper.

CONCLUDING REMARKS

A phenomenological study was carried out on metallocene-catalyzed elastomer modified PP blends in the range 0–30 %wt. Results show that impact toughness of PP homopolymer was greatly enhanced by the presence of the elastomer. The size independence of the parameters calculated was not directly verified since only one thickness of sample was assayed: however, results are strictly comparable.

The PP homopolymer behaved in an elastic way and fracture propagation occurred by cleavage. The toughness parameter could be easily derived from either current linear (G_{IC} and K_{IC}) and nonlinear (J_c) elastic fracture mechanics approaches. Hence, due to the simplicity of determination, the J_c parameter appears as an interesting alternative methodology to evaluate cleavage fracture under impact conditions.

The differences observed between K_{IC} values predicted from equivalent energy method and that calculated from the load field may arise from the uncertainty in predicting Young's modulus.

Blends of propylene homopolymer and POEs seem to behave according to a semi-brittle pattern when tested at room temperature and high loading rate, as judged from the nonlinearity in load deflection records and the appearance of the fracture surfaces. Relevant damage zones developed ahead of the crack tip prior to catastrophic crack propagation were revealed as a white halo in the fracture surface. It was difficult to distinguish if stress whitening was due to sub-critical crack growth, plastic deformation, or both effects combined. Hence the J–R curve, which was useful to determine a lower bound toughness in the case of PP static toughness determination,[37] cannot be used here due to the uncertainty in the measurement of crack advance. The LEFM methodologies, which were modified to take into account plasticity effects, were tried in order to assess the toughness parameters of the blends. Corrected elastic methodology could not be applied due to the very high nonlinearity of behavior. K_{IC}^{max}, K_{IC}^{E}, and J_c approaches were tried. Even if very good agreement between the mean fracture toughness parameters obtained from the methodologies were obtained, the results were widely scattered (measured by the standard deviation) and hence the average parameters lacked significance. Only the J_c parameter deduced from the nonlinear elastic approach, met size requirements. As the behavior shows very similar characteristics to those developed by steels in the ductile–brittle transition regime[40], the results were presented in the way of a Weibull plot according to the statistical weakest link model. Reliable lower bound toughness, J_{min}, seems to be obtained. Indeed, for comparative purposes, median values derived from the Weibull model can be very useful parameters. The toughness was optimal for 10% elastomer blend.

Further work is in progress in order to develop new approaches based on the weakest link philosophy to evaluate polymer fracture behavior in the ductile to brittle transition region. The work will aim to assess if data taken from small fracture mechanics-type specimens can be used to infer the fracture toughness performance in full-scale structures.

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