ASSESSMENT OF METHODS TO DETERMINE FRACTURE TOUGHNESS OF POLYMERS IN THE DUCTILE-TO-BRITTLE TRANSITION REGION

R.G. Cocco[1], P.M. Frontini[2] and J.E. Perez Ipi□a[1]
[1]GMF/LPM/CONICET, Universidad Nacional del Comahue, Bs. As. 1400, Neuquén, Arg.
[2]División Polímeros INTEMA/CONICET, Juan B. Justo 4302, Mar del Plata, Arg.

ABSTRACT

The fracture behaviour of materials in the ductile-to-brittle region is neither completely brittle nor entirely ductile. Besides, scatter in toughness results has been reported in polypropylene and nylon. At the moment there is no general agreement on the methodology to determine the fracture toughness in the transition region. In this work an assessment of different proposed methods based on LEFM, EPFM and statistical approach was carried out over two materials: polypropylene homopolymer (PPH) and a blend of PPH containing 20 wt.% of elastomeric polyolefin (PPH/POes). The methods analysed were Fernando- Williams method, plastic zone corrected LEFM proposed by Gerin et al., G_{ST} /G_{INST} method by V-Khanh and De Charentay, JR curve method by Santarelli et al., and a statistical approach proposed by the authors in a previous work. The results of this analysis indicate that the Fernando-Williams and Plastic zone corrected LEFM methods, based on LEFM, tended to underestimate the fracture toughness, being very conservative. On the other side, JR method may overestimate the toughness, as in PPH/POes blend case. The G_{ST} /G_{INST} and Statistical methods appear to be the most adequate to characterise the fracture toughness of PPH and PPH/POes blend. The values of the characteristic fracture toughness found by both methods were slightly smaller than the minimum determined experimentally and proved very close between themselves.

1 INTRODUCTION

Linear elastic fracture mechanics (*LEF*M) has been extensively applied to determine the fracture toughness of polymers in the region of brittle behaviour, known as *lower shelf*; this is at high strain rate or low temperature test conditions, by means of K_{IC} and G_{IC} parameters [1,2]. The linear elastic *stress intensity factor* K_I does not describe the stress atmosphere near the crack tip in the region of ductile behaviour or *upper shelf*, and it is necessary to make use of the elasto-plastic methodology (*EPMF*) applying either *CTOD* or *J* integral, being the latter more often applied to polymers[2,3,4]. Within the ductile-to-brittle transition region, non-linear load-displacement records are always present [5,6]. This non-linearity may be attributed in some cases to the generation of a plastic zone that cannot be considered small [5,6], or can include also some stable crack growth before fracture [7,8]. As a regime entirely ductile is not developed, JR curves can not be determined as they are in the upper shelf. On the other hand, different authors reported a considerable scatter in the toughness results of nylon, polypropylene and its blends with different rubbers[7,8,9,10].

Several methods have been proposed to determine a characteristic toughness value, although there is no general agreement on which is the best methodology to be employed. Polymers operating in the ductile-to-brittle transition region present uncertainty regarding their toughness value which can lead to an unexpected failure, risking the structural integrity of the component.

This work aims to compare fracture toughness values obtained by applying those methods proposed by different researchers as Fernando and Williams [5], Vu-Khanh and De Charentenay

[7], Santarelli and Frontini [10], and Grein and co-workers[6]. To this purpose, polypropylene homopolymer (PPH) and a polypropylene-elastomeric polyolefin blend (PPH/POes 20%wt) were examined within ductile-to-brittle transition region. In a previous work [11] the authors proposed a statistical treatment adapted from metal approaches where toughness lower bound determination was emphasised. This method was also included in this comparative analysis.

2 EXPERIMENTAL

Experiments were carried out on a commercial grade polypropylene homopolymer (PPH, Cuyolen NX1100) and a blend of PPH containing 20 wt.% of elastomeric polyolefin (POes, ENGAGE 8100 from Dow Chemilcals). Both materials were provided as pellets by Petroquímica Cuyo SAIC. Pellets were compression moulded in a hydraulic press into 20mm x 15mm x 6 mm plates at 200 ^{0}C and 3.7 MPa.

Fracture characterisation was carried out on three point-bend specimens, cut from the compression moulded plates. Specimen dimensions were: thickness B = 6mm, width W = 2B = 12mm, span $S = 4W$ =48mm. The crack length to width ratio was approximately a/W = 0.5 to one set of 53 specimens of each material. Other small set of PPH of 14 specimens included a/W ratios of 0.3; 0.5; 0.7 and 0.8. -30 ^{0}C data were extracted from literature [12]. Sharp notches were introduced by sliding a razor blade having an on-edge tip radius of 13μm. Tests were performed on an Amsler screw machine HFP 1478 model with displacement control, at room temperature and 20 mm/min crosshead rate. Test conditions were selected to ensure a ductile-to-brittle transition behaviour.

3 DATA ANALYSIS METHODS

Fernando and Williams [5] found that the polypropylene homopolymer (PPH) toughness in the ductile-to-brittle transition region, evaluated in terms of K_I, depends on the specimen's thickness. They obtained apparent toughness values at low temperature (-60 ^{0}C) , K'_{IC}, for specimens with different thickness and found a linear relation between K'_{IC} and 1/B, where B is the specimen thickness. Then, they calculated a minimum value of toughness, K_{c1}, by extrapolating the K'_{IC} values to that corresponding to infinite-thickness. They proposed this minimum toughness value as the fracture toughness in plane strain, K_{c1}, and also stated that it remained constant as the temperature increased in the transition region.

Vu-Khanh and De Charentenay [7] worked with materials in which fracture initiated in a stable manner, and at some point becomes unstable. They assumed that the variation in G_C during the stable propagation is linear, and that the mean value is G_{STmean}. Then, the energy absorbed during this period of propagation, U_{ST}, can be expressed as the mean value, G_{STmean}, times the area of stable crack growth. On the other hand, if the fracture energy at the instability, G_{INST}, is assumed to be a material constant, the energy released during the unstable propagation, U_{INST} , can be written as a function of G_{INST}. The total energy absorbed by the specimen equals the energies consumed during both crack propagation periods:

$$U_{Tot} = G_{STmean} \cdot A_1 + G_{INST} \cdot BW\phi_1 \tag{1}$$

where A_1 is the area of stable crack, φ_1 is the calibration factor corresponding to the new crack length, $a_1 = a_0 + a_{st}$, B is the thickness and W is the width of the specimen. The total energy, U_{Tot}, experimentally is obtained as the area under the load-displacement record. Then, by plotting (U/A_1) vs. $(BW\varphi /A_1)$, G_{STmean} can be obtained from the y-axis interception and G_{INST} from the slope of this straight line.

Santarelli et al.[10] reported some scatter in the toughness of PP homopolymer and a variable

amount of stable crack growth, Δa, before unstable propagation. Despite some scatter, their results showed that the measured distance from the initiation site to the original crack tip correlated very closely to the measured fracture toughness. Then, they plotted the data on a J-R curve, proposing the engineering initiation *J* value
as a fracture lower bound value.

Grein and co-workers [6] proposed to characterise the fracture behaviour by using LEFM to which an experimental determination of plastic zone correction was added. Experimentally, K_I is obtained from

$$K_I = f(a/W)\, F_{Max} \Big/ B\sqrt{W} \tag{2}$$

The independence of the K values from the crack length, a, guarantees the applicability of LEFM. In the case of brittle fracture, K_{IC} can be calculated by expressing F_{Max}. vs. (B%W)/f(a/W) for different crack lengths. The straight line described by the experimental points passes through the origin and its slope yields K_{IC}. In the case of non-brittle mode failure, the data depict a straight line which cuts they-axis at a negative value. In order to correct this deviation they proposed forcing this straight line to pass through the origin by applying Irwin effective crack length (a_{eff} = a + rp) in calculations, being rp the plastic radii. For this, they used an iterative procedure in eqn. (2), replacing f(a/W) by f((a+rp)/W) until this 'pass through the origin' requirement was fulfilled. Then, they obtained the effective toughness, K_{eff}, from the slope of this straight line. They stated this K_{eff} at the maximum load as a geometry-independent quantity that can be used to characterise the material toughness.

In a previous work [11], a statistical treatment of the scatter in toughness results evaluated in terms of *J* -integral was proposed. A weakest-link [13] model was assumed. This theory states: 1) fracture toughness is variable, differing throughout the given material and particularly along the specimen crack front, 2) the fracture toughness of any specimen is governed by the point or region having the lowest toughness along the crack front. The experimental results were fitted by a three-parameter Weibull model (3P-W) given by the following expression:

$$F(J) = 1 - \exp\left[-\left(J - J_0/B - J_0\right)^m\right] \tag{3}$$

where F(*J*) is the cumulative probability and can be calculated by means of an estimator, *'J'* represents the toughness value, *'B'* is the scale parameter, *'m'* is called the shape parameter, and *'J_0'* is the threshold toughness parameter independent of size. In this way a minimum toughness value is obtained implying that there is null probability of failure for driving forces lower than *'J_0'*.

4 RESULTS

The toughness values varied between 4.0 and 6.5 KJ/m^2 for PPH, while the PPH/POes blend displayed a larger variation: from 8.0 to 40.0 KJ/m^2.

In Figures 1 a and b the graphics corresponding to G_{ST} /G_{INST} method for PPH and PPH/POes respectively, the values for G_{ST} and G_{INST} are also shown.

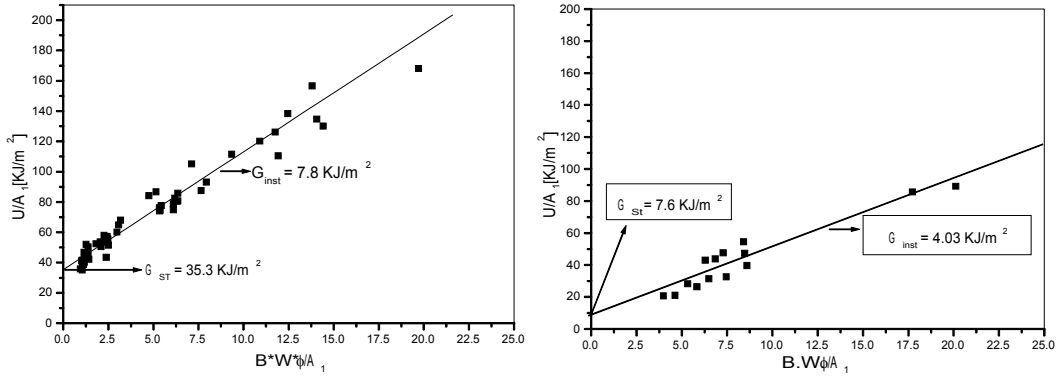

Figure 1: (U/A_1) vs. ($BW\varphi /A_1$) graphics a) PPH, b) PPH/POes blend

J-R curves for PPH and PPH/POes blend can be seen in Figure 2 a and b. The initiation *J* values resulting from the interception of J-R curve and the blunting line are also shown.

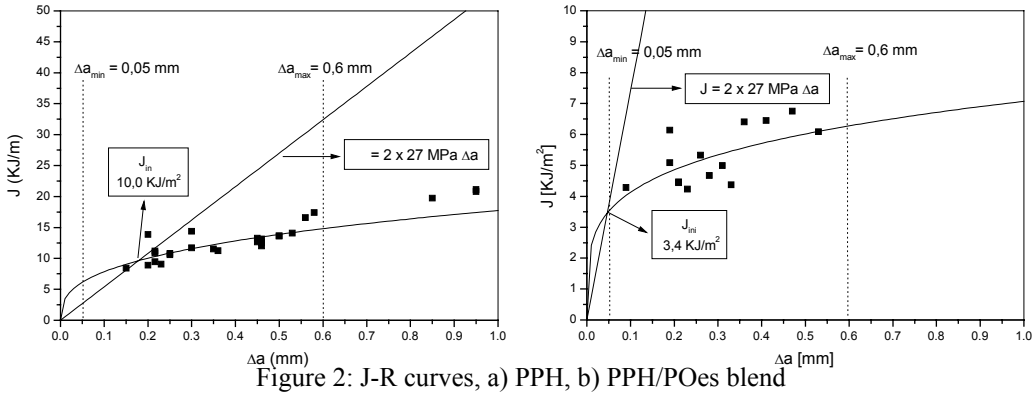

Figure 2: J-R curves, a) PPH, b) PPH/POes blend

Figure 3 shows the plastic zone correction method for PPH. The dashed regression line corresponds to those values without plastic zone correction and the filled one to those corrected.

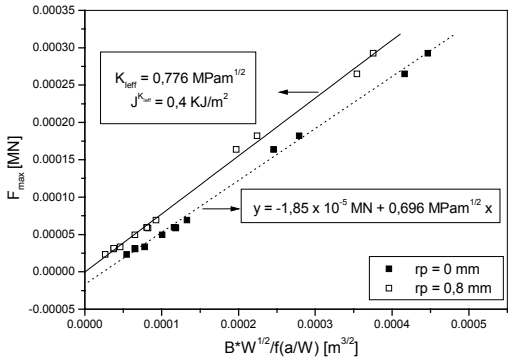

Figure 3: F_{Max} vs. (B%W)/f(a/W) graphic for PPH.

The fracture parameters obtained from the different methodologies are showed in Table 1. J_C mean values are also included.

Table 1: Toughness parameter values.

	J^K_{c1} [KJ/m^2]	G_{INST} [KJ/m^2]	J^K_{eff} [KJ/m^2]	J_{ini} J-R [KJ/m^2]	J_0 [KJ/m^2]	J_{Cmean} [KJ/m^2]
PPH	1.5 (-30 ^{0}C)	4.3	0.4	3.4	3.9	4.9
PPH/POes	2.7 (-30 ^{0}C)	7.8	----	10.0	7.8	21.3

All values were expressed in J units [KJ/m^2]. The LEFM values, K_I, were converted to J through the following relation:

$$J_I = K_I^2 / E \tag{7}$$

where E is the Young modulus at test temperature and rate.

The PPH and PPH/POes toughness values used in the Fernando and Williams method were obtained from literature [12], while the toughness lower bound values, J_0, used in the Statistical method were taken from a previous work [11].

5 CONCLUSIONS

It clearly emerges from Table 1, that the toughness values estimated by using the method of Fernando and Williams[5] at low temperature resulted very conservative, since both J^K_{1c} value (1.5 KJ/m^2 and 2.7 KJ/m^2 for PPH and PPH/POes, respectively) are much lower than the minimum experimental values of Jc determined under the experimental conditions.

PPH toughness value determined following the plastic zone correction method [6] (0.4 KJ/m^2) resulted even lower than that given by using the Fernando and Williams method [5] and, therefore, still more conservative.

The initiation *J* values determined from the J-R method were 3.4 KJ/m^2 for PPH and 10.0 KJ/m^2 for PPH/POes blend. In the case of PPH, J_{ini} was lower than the minimum experimental J_C value and hence, it appears adequate to characterise the toughness. However, for PPH/POes, J_{ini} value was higher than the minimum experimental J_C, resulting a non conservative alternative.

G_{INST} values obtained from the method proposed by Vu-Khanh and De Charentenay [8] were 4.0 KJ/m^2 for the PPH and 7.8 KJ/m^2 for the PPH/POes blend. These values were slightly lower than the minimum J_C obtained experimentally from large samples for both materials and, consequently, this method appears proper to characterise the fracture toughness of the PPH and PPH/POes.

Both toughness lower bound, J_0, 3.9 KJ/m^2 for PPH and 7.8 KJ/m^2 for PPH/POes blend from

the statistical approach [11], resulted slightly lower than the minimum experimental value of J_C for each material, suggesting that they are representative material toughness values. The J_0 values were in good agreement with the G_{INST} parameter.

As a final observation, it is worth mentioning that the mean J_C values in the transition region are much higher than the toughness parameters determined by the different methods analysed.

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