DETERMINATION OF THE J-R CURVES OF ETHYLENE-PROPYLENE BLOCK COPOLYMERS BY MEANS OF DIFFERENT J-INTEGRAL METHODOLOGIES

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ABSTRACT

A single specimen method as the normalization method has been applied to two different ethylene-propylene block copolymers with distinct structural parameters with the aim of analyzing the influence of a methodological parameter as the crack tip constraint factor, m, on the J-R curves. R-curves obtained via multiple specimen method have been taken as references. The results reveal that the crack tip constraint factor is strongly dependent on the material´s properties and dimensions. The best accuracy of the J-R curves of the copolymer with a large molecular weight ~ 820 kg/mol, is attained for values of m equal to 1.5 and 1.25 for the specimens´ dimensions related to 6.35 and 9 mm thick samples, respectively. Meanwhile, the PP block copolymer, with molecular weight ~ 300 kg/mol, showed the best fit between the J-R curves obtained experimentally with those determined via normalization by setting m=1, in accordance with the m values reported in the literature.

KEYWORDS: J-R curves, normalization method, crack tip contraint factor, multiple specimen, ethylene-propylene block copolymers.

1. INTRODUCTION

When Wells [1] attempted to measure K_{IC} values in a number of structural steels, he found that these materials were too tough to be characterized by Linear Elastic Fracture Mechanics (LEFM) and that the crack faces had moved apart prior to fracture; plastic deformation blunted an initially sharp crack. The degree of crack blunting increased in proportion to the toughness of the material [2-3]. These observations led to propose the opening at the crack tip (CTOD) as a measure of fracture toughness. Under LEFM conditions, there is a relationship between CTOD and the fracture toughness, K_I, and the energy release rate, G, given by:

$$\delta = \frac{K_I^2}{m\sigma_{YS}E} = \frac{G}{m\sigma_{YS}} \qquad (1)$$

where E is the Young´s modulus, σ_{YS} is the yield stress and m is a dimensionless constant that depends on stress state and materials properties, named as crack tip constraint factor.

For ductile polymers, J-integral is the method to determine the fracture toughness. The fracture toughness at crack initiation, J_{IC}, is measured by a crack resistance curve, J-R curve, where J is plotted versus the ductile crack extension, Δa. Since J = G for linear elastic material behaviour, under elastic-plastic conditions equation (1) becomes in:

$$J = m\sigma_{YS}\delta \qquad (2)$$

The resistance curve is divided into three stages [3]. During the initial stage, the crack is essentially stationary and the finite slope of R-curve is caused by blunting and described by [4]:

$$J = 2m\sigma_{YS}\Delta a \qquad (3)$$

The crack starts to grow in stage 2. A rising R-curve occurs, being J-integral only dependent on the crack extension and thus, J-R curve is a material property at this step. Finally in stage 3, well beyond the initial blunted tip, a steady-state condition is reached, where the local stresses and strains are independent of the extent of crack growth. During the steady-state crack growth, a plastic zone of constant size sweeps through

the material, leaving a plastic wake. Therefore, the R-curve is flat; J does not increase with crack extension, provided the material properties do not vary with position.

For the J-R curves construction of polymers, ASTM [5] and ESIS [6] recommend the multiple specimen method. This methodology, though straightforward and effective, is time and material intensive, as at least a minimum of seven specimens are to be tested to generate the R-curve. For that reason, indirect methods have been developed to obtain J-R curves with fewer specimens and, thus, less time requirements. The single specimen methods are based on the load separation criterion [7], and offer an easy and effective alternative approach to obtain J-R curves. Among the single specimen methods, the normalization method and the load separation parameter method have been successfully applied to polymeric materials [8-11].

As has been previously mentioned, the first section of the J-R curve is dominated by blunting which is described by equation (2). From this equation, the crack tip constraint factor, m, attains much significance. This parameter is closely related to the stress state at the crack tip, which is described by the Q-stress [3] and usually takes a value of 1 for Ramberg-Osgood materials [4], but it is very sensitive to the material type, the loading conditions and geometry of specimens. For polymers, the m value can range between 0.5 and 2 [9]. In particular, m=1 has been reported for polypropylene (PP) based materials [9-10] and m=2 for ultra high molecular weight polyethylene (UHMWPE) materials [11].

In this work, it is analyzed the applicability of the normalization method in different ethylene-propylene block copolymers, paying special attention to the m values which provide the best J-R curves when compared with the classical multiple-specimen method, attending to the structural properties of the polymers under study.

2. THE NORMALIZATION METHOD

The objective of any single specimen method including the normalization method is to obtain accurate crack length predictions using the load (P)-displacement (δ) data alone. The instructions given by ASTM E1820-06 [4] were taken as a guide.

The first step for the determination of the J-R curve is an optical crack-length measurement of the initial, a_o, and final, a_f, crack lengths. Subsequently, each value of the load P_i up to, but not including P_{max} is normalized using the following expression:

$$P_{Ni} = \frac{P_i}{WB\left[\frac{W - a_{bi}}{W}\right]^{\eta_{pl}}} \tag{4}$$

where W is the specimen width, B is the specimen thickness and $\eta_{pl} = 2$ for three point bending specimens (SENB). a_{bi} is the blunting corrected crack length given by:

$$a_{bi} = a_o + \frac{J_i}{2m\sigma_{YS}} \tag{5}$$

$$J_i = \frac{K_i^2\left(1-\upsilon^2\right)}{E} + J_{pli} \tag{6}$$

where K_i is the stress intensity factor, E is the Young´s modulus, ν is the Poisson ratio and J_{pl} is the plastic part of the J-integral [4].

Each corresponding load line displacement, δ_I, is normalized to give a normalized plastic displacement:

$$\delta'_{pli} = \frac{\delta_{pli}}{W} = \frac{\delta_i - P_i C_i}{W} \tag{7}$$

where C_i is the specimen elastic load line compliance, based on the crack length a_{bi}.

In this manner, data points up to maximum force are normalized. In order to obtain the final point, the same equations are employed, but instead of the initial crack length, the final crack length is used. The normalized plastic displacement values above 0.001 up to maximum force, excluding P_{max} value itself, and the points obtained with the use of the final crack length are used for the normalization function fit. The normalization function can be analytically expressed:

$$P_N = \frac{a + b\delta'_{pl} + c\delta'^2_{pl}}{d + \delta'_{pl}} \tag{8}$$

where a, b, c and d are searched fitting coefficients. When the fitting parameters are determined, an iterative procedure is further applied to force all P_{Ni} data to lie on the fitted curve by a_i adjustment. When the crack lengths are determined, the J-R curve can be then calculated and the critical J-integral value can be evaluated.

3. MATERIALS AND EXPERIMENTAL PROCEDURE

The materials studied were two commercial grade ethylene-propylene block copolymers, EPBC1 and EPBC5, supplied by Repsol in form of pellets. The bulk specimens for fracture characterization as well as the tensile specimens were prepared by injection molding. The basic characteristics such as the ethylene content, determined from Nuclear Magnetic Resonance (NMR), the molecular weight, M_W, obtained by Gel Permeation Chromatography (GPC) and the glass transition

temperatures corresponding to the elastomeric particles, T_g EPR, embedded in the propylene matrix, T_g PP, measured via Dynamic Mechanical Thermal Analysis (DMTA) are collected in Table 1.

The mechanical properties such as the Young´s modulus, E, and the yield stress, σ_{YS}, were measured via tensile tests at cross-head speeds of 1 mm/min for the ISO-527 bulk injected tensile samples. The elastic modulus values were of 1.38 ± 0.06 and 1.54 ± 0.03 GPa while the values of yield stress were of 26.1 ± 0.6 and 23.9 ± 0.5 MPa for EPBC1 and EPBC5, respectively.

Table 1. Basic properties of the copolymers under study

	Ethylene content (%)	M_W (kg/mol)	T_g PP (ºC)	T_g EPR (ºC)
EPBC1	6.9	816	10.57	10.41
EPBC5	8.5	353	-45.85	-49.30

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Experimental data used to generate J-R curves using a multiple specimen approach were obtained following the guidelines described in [5, 6]. Single edge notched bend specimens (SENB) were used for fracture characterization with 6.35 mm and 9 mm in thickness, being the overall dimensions of 6.35x12.7x55 mm and 9x18x80 mm, respectively. In all the specimens, an initial straight-through slot with a length to width ratio of 0.5 and terminating in a V-notch with 0.2 mm in root radius was mechanized. The notch was sharpened by sliding a razor blade across the notch to achieve a total crack depth of ~ 7 mm and ~ 9.9 mm for the 6.35 mm and 9 mm thick specimens, respectively. A minimum of seven specimens for each material and specimen dimensions were performed at room temperature at a crosshead speed of 1 mm/min using an electromechanical testing machine (MTS RF/100) with a load cell of ± 5 kN. A three point bend fixture was used with a span to width ratio of 4. One unnotched specimen was tested to correct the indentation produced by the support on the specimen. The J integral was calculated using:

$$J = \frac{2U}{B(W - a_O)} \tag{9}$$

To measure crack extension, Δa, the tested specimens were fracture at high loading rate after soaking in liquid nitrogen. The initial and final stable crack lengths were measured physically from the broken surfaces via light microscopy (Leica DMR). The resulting J-crack growth resistance curves were fitted to a power law $J=C\cdot\Delta a^N$, with $N\leq 1$.

J-R curves were also determined using the single specimen normalization method [4], focusing on the influence of the crack tip constraint factor on the predicted J-R curves by setting m to 1, 1.5 and 2. A computational procedure was attained to obtain J-R curves via normalization method with the help of Matlab 7.0.4 software.

For the computation of J_{IC} values, the guidelines described by Hale et al. [6] have been followed, where this critical value has been replaced by a pseudo-initiation value $J_{0.2}$, which defines crack resistance at 0.2 mm of the total crack growth. The size requirements for plane strain J_{IC} are given by:

$$B, a, W - a > 25\frac{J_{IC}}{\sigma_{YS}} \tag{10}$$

4. RESULTS AND DISCUSSION

Figure 1 and 2 show the J-R curves predictions based on the normalization method with a value of m=1 together with the J-R curves obtained experimentally for EPBC1 and EPBC5, respectively. Two samples were used to generate the J-R curve from the normalization method for each material and specimen dimensions and the power law fit is also included for every case. As can be observed, independently of the EPBC5 thickness, there is a good agreement between the J-R curves of EPBC5 determined via normalization with that obtained via multiple specimen when the constraint parameter is equal to 1 (Figure 2). This contrasts with the comparison realized on EPBC1 (Figure 1). The predicted normalized curves stand further from the experimental ones, especially in the 6.35 mm thick specimens (Figure 1a), and even some of them cannot be fitted to a power law with $N\leq 1$. The well reported m=1 for PP block copolymers [9-10] results inaccurate for EPBC1.

The poor concordance in case of EPBC1 responds to its different structural properties with regard to EPBC5 (Table 1), especially the molecular weight. The former presents molecular weights almost three times higher than the latter. Those large values are roughly comparable to those shown by UHMWPE. For UHMWPE materials, m has been accurately determined and a value of m=2 can be generally used [11]. This finding suggests that it may be a strong effect of the crack tip constraint factor, m, for EPBC1. That is the reason why the normalization method was applied to EPBC1 by setting m=1, 1.5 and 2. The results are gathered in Figure 3 and 4 for EPBC1 with 6.35 mm and 9 mm in thickness, respectively. As can be seen, the best fit is achieved when using a value of m=1.5 for the 6.35 mm thick EPBC1 specimens (Figures 3a and 3b),

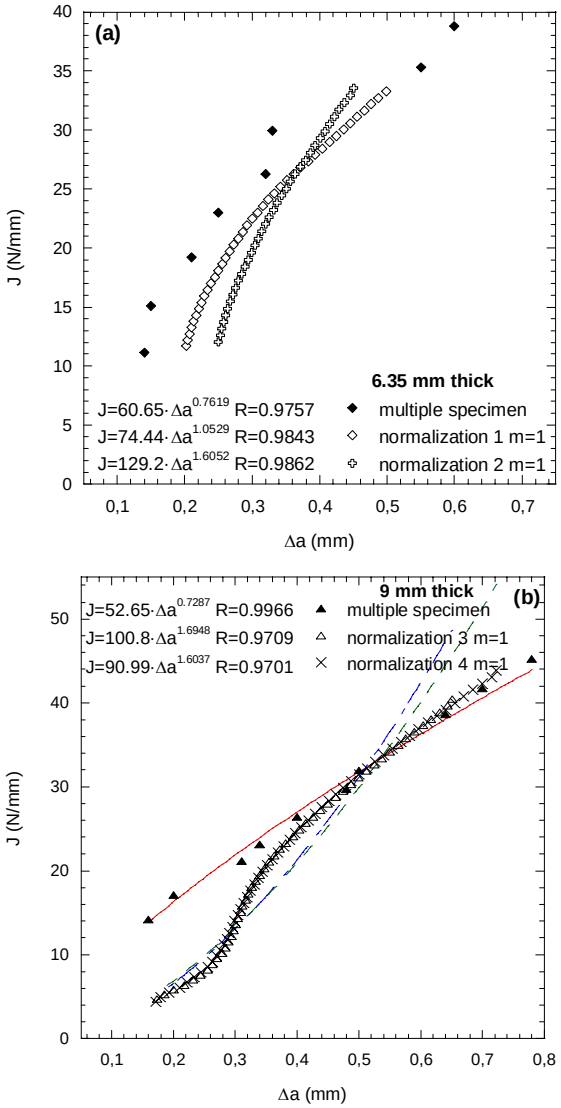

Figure 1. J-R curves of EPBC1 obtained from the multiple specimen method and from the normalization method with m=1 for (a) 6.35 mm and (b) 9 mm thick specimens.

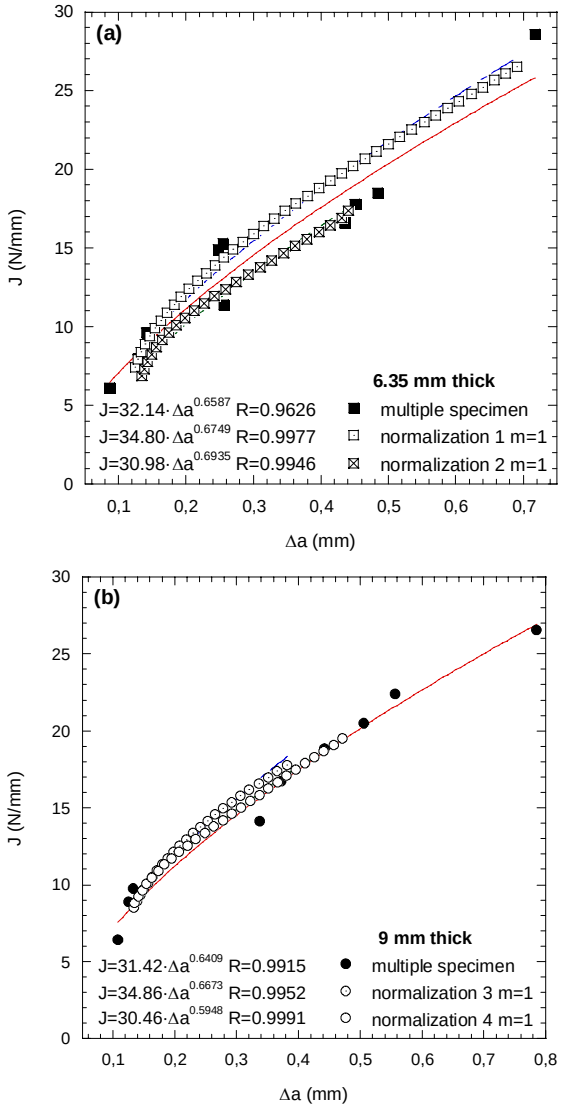

Figure 2. J-R curves of EPBC5 obtained from the multiple specimen method and from the normalization method with m=1 for (a) 6.35 mm and (b) 9 mm thick specimens.

Table 2. Critical values of J-integral for EPBC1 and EPBC5 determined through different methods and crack tip blunting behaviours.

Sample		EPBC1		EPBC5	
Thickness (mm)	m	6.35	9	6.35	9
Multiple Specimen		17.8	16.3	11.1	11.2
Normalization	1	-	-	[1] 11.7	[3] 11.9
				[2] 10.2	[4] 11.7
	1.25	-	[3] 14.8	-	-
			[4] 14.6		
	1.5	[1] 19.0	[3] 18.9	-	-
		[2] 18.1	[4] 18.7		
	2	[1] 21.4	[3] 21.6	-	-
		[2] 21.3	[4] 23.0		

- *J-R curve does not fit $J=C\cdot\Delta a^N$, with $N\leq 1$*

while for the 9 mm thick EPBC1 specimens (Figures 4a and 4b), the best accuracy is attained with m=1.25.

The critical fracture toughness values, J_{IC}, obtained considering the different methods and as a function of the crack tip constraint factor, m, in case of the normalization method are collected in Table 2. None of the J_{IC} parameters verifies the size criterion specified in equation (10), so all the values are not in plane strain state. For EPBC5, the results obtained via multiple specimen method are in good agreement with those determined with the normalization method using a value of m=1 for the two analyzed specimens´ configurations. In such PP block copolymer, it has been proved that the established value of m=1 [8-9] works accurately for EPBC5. On the other hand, for the other PP block copolymer, EPBC1, with a molecular weight almost three times higher than EPBC5, it has been evidenced the dependence of the crack tip constraint factor on the

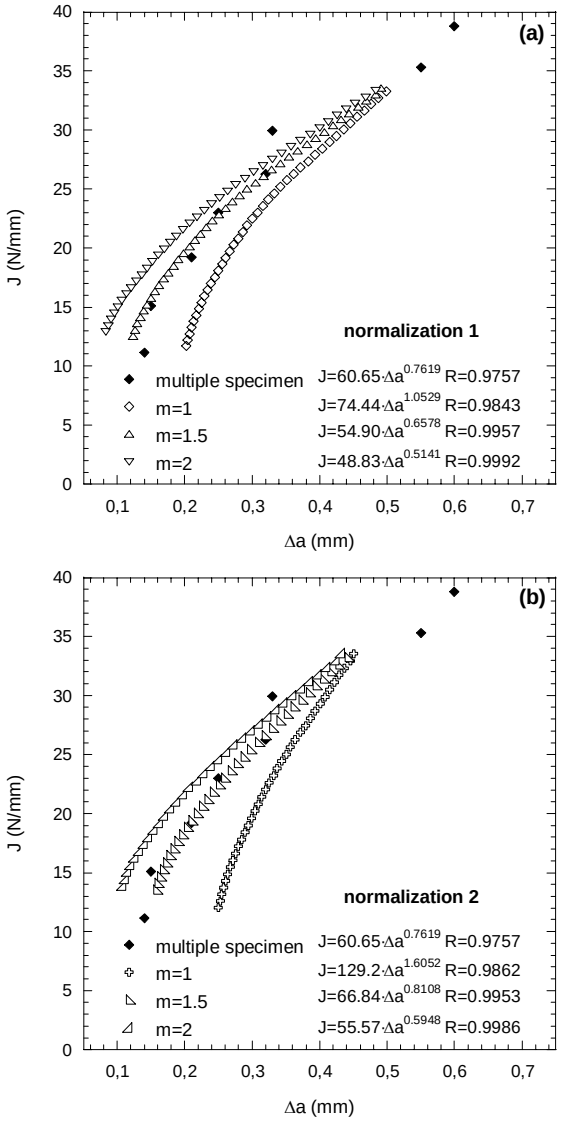

Figure 3. Influence of the crack tip constraint factor, m, on the J-R curves of EPBC1 with 6.35 mm in thickness: (a) sample normalization 1 and (b) sample normalization 2.

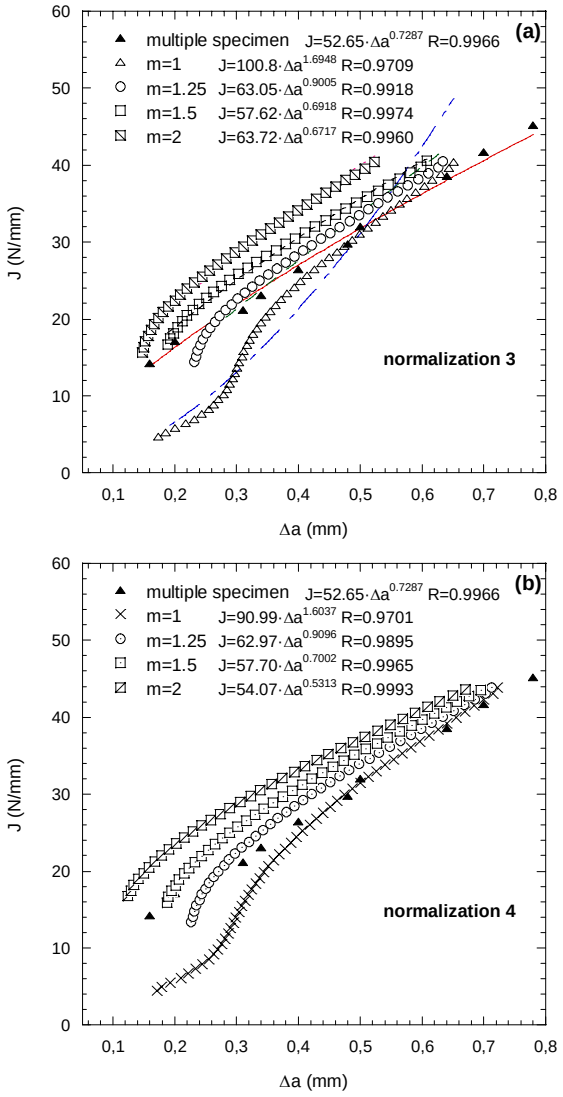

Figure 4. Influence of the crack tip constraint factor, m, on the J-R curves of EPBC1 with 9 mm in thickness: (a) sample normalization 3 and (b) sample normalization 4.

material properties and even geometry. Particularly, thebest fit between the multiple specimen J-R curves and those determined via normalization method are achieved by setting m to 1.5 and 1.25 for 6.35 and 9 mm thick samples, respectively.

5. CONCLUSIONS

A single specimen method as the normalization method, included in ASTM E1820-06, has been applied to two different ethylene-propylene block copolymers with distinct structural parameters, especially the molecular weight, with the aim of analyzing the influence of a methodological parameter as the crack tip constraint factor, m, on the applicability of this methodology. The J-R curves obtained via multiple specimen method have been taken as references. The results reveal that the crack tip constraint factor is strongly dependent on the material´s properties and dimensions. The best accuracy of the J-R curves of the copolymer with a huge molecular weight ~ 820 kg/mol, roughly that of UHMWPE, is attained for values of m equal to 1.5 and 1.25 for the specimens´ dimensions related to 6.35 and 9 mm thick samples, respectively. Meanwhile, the PP block copolymer, with molecular weight ~ 300 kg/mol, showed the best fit between the J-R curves obtained experimentally with those determined with the normalization method by setting m=1, in accordance with the m values reported in the literature.

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