Methods for Introducing Pre-cracks in Fracture Mechanics Testing of Polymers

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Abstract. Fracture mechanics testing of polymers requires the introduction of some sort of pre-crack in the standard specimen. Since the classic work by Lu *et al.* [J. Mater. Sci. Vol. 26 (1991) p. 881] it is recognized that pre-cracking introduces some sort of damage in the polymer matrix, which may or may not affect the testing results depending on the events that occur upon first loading. The key property to be required for a pre-cracking method, therefore, is reproducibility. This is investigated in the present work by fracture toughness testing using SE(B) samples produced from PMMA and MDPE (the later in cryogenic conditions) with two methods of pre-crack introduction (tapping on a razor blade and gentle pressing a razor blade using a standard device). The results show that the method of pre-crack introduction influences the K_{Ic} values obtained when testing PMMA, with the more conservative value obtained for pre-cracks obtained by tapping on a razor blade. For the case of MDPE the average results are statistically equivalent, although, again, tapping on a razor blade results in more conservative values. Using this method of pre-crack introduction it is concluded that K_{Ic} values for the two resins are 1.11 MPa $m^{1/2}$ for PMMA and 3.42 MPa $m^{1/2}$ for MDPE (under cryogenic conditions).

Introduction

The use of polymers in structural applications where structural integrity is critical is crescent in the industry. It is expected, therefore, that the demand for fracture mechanics parameters like plane strain fracture toughness will increase in the near future. Fracture mechanics testing is knowingly time-consuming and requires tight control over specimen geometry. A particular characteristic of these tests is the need to introduce a pre-crack, to simulate a natural flaw in the material. In the case of metals and alloys it is well recognized that fatigue pre-cracking produces the most reliable results and this is incorporated in the Standard Test Methods (*e.g.* ASTM E 399-90). In the case of polymers, however, this is less evident. Most Standard Test Methods (STM) and Test Protocols for polymers suggest using some sort of sharp knife (like razor blades) to introduce pre-cracks, method widely adopted by different authors.

The ESIS test protocol for plane strain fracture toughness testing of polymers, for example, suggest introducing a natural crack by tapping on a new razor blade placed in the notch, if this is not possible (the case of tough polymers), the protocol allows sliding or sawing a new razor blade across the notch [1]. ASTM D 5045-99 adopts the same procedure, but specifically discard pressing the blade, due to the danger of introducing residual stresses at the crack tip. ASTM D 6068-96 (J-R curve determination), on the other hand, allows sliding the blade, but also permits alternative methods as fatigue pre-cracking or pressing. Lu *et al*. [2], in their careful study of Slow Crack Growth tests for PE, recommend introducing the pre-crack by gently pressing the razor blade in a controlled manner (*i.e.* using a specific device). More recently Dapp and Rimnac [3], compared several methods of pre-crack introduction for obtaining J-R curves in UHMWPE and concluded that fatigue testing produces the worst results, presumably due to the extensive damage introduced in the

polymer matrix. They recommend pressing the blade as pre-cracking method. The ideal procedure for pre-crack introduction in fracture toughness testing of polymers, thus, is considerably less consensual than in the case of metals and alloys and these divergences probably increases the variability of results obtained in different laboratories.

The aim of the present work is to compare two pre-crack introduction procedures applied to the plane strain fracture toughness testing of two polymers with distinct properties: Poly(methyl metacrylate) – PMMA – as an example of a brittle polymer (which allows the introduction of a natural crack) and medium density polyethylene – MDPE – as an example of a tough polymer (which does not allow the introduction of a natural crack). Although pre-cracking is made at room temperature, the tests for the later resin will be conducted under cryogenic conditions, in order to obtain valid K_{Ic} values. The two procedures here investigated are: (1) tapping on a new razor blade and (2) gently pressing a new razor blade in the machined notch.

Experimental

Materials. Two resins were selected for this study[1]:

- Acrygel PMMA general purpose resin, furnished by Resarbras da Bahia SA (Unigel Group), density 1180 kg m^{-3}, fracture strength, σ_f = 69 MPa [4], melt flow index, MFI (10 min) = 2.5 g , estimated glass transition temperature, $T_g > 100^oC$.
- MDPE 8818 unpigmented resin, designed for water pipe extrusion, furnished by PBBPolysur (Dow Latin America), density 940 kg m^{-3}, yield strength[2], σ_y = 15.5 MPa [5,6], MFI (190^oC/10kg/10 min) = 0.77g, estimated glass transition temperature, $T_g < -50^oC$.

Specimens. Fracture toughness single-edge notched bending samples – SE(B) – were machined out of compression molded plates produced according to ASTM D4703-03 at 190^oC and 20MPa mold closure pressure. The MDPE plates were produced using a flash mold with machined cavity with final plate dimensions 180 x 180 x 14 mm. The PMMA plates were produced using a flash picture-frame mold with final plate dimensions 180 x 180 x 6 mm. All plates were found to be free of internal flaws, like pores and cracks and were homogeneous under visual inspection. Figure 1 schematically shows the specimen dimensions. The actual dimensions for the PMMA and MDPE specimens are given in Table 1. Particular care was taken in notch machining, which was made using single-point flycutter made of a steel cutting disk, with an edge sharpened to 45^o, mounted in a standard drilling machine operating at 3600 rpm.

Figure 2 shows an example of notch root produced using this procedure. It is obvious that other dimensions may play a role in this region (*e. g.* the notch root radius), but it was also observed that the main attribute of the notch root is to direct the razor blade during pre-crack introduction, avoiding its bending. These dimensions were, therefore, not explicitly specified, assuming that the adopted procedure allows to obtain reproducible notches in the different samples.

Pre-cracking methods. The two pre-crack introduction procedures here investigated are:

1. Tapping on a new razor blade placed in the machined notch and
2. Gently pressing a new razor blade place in the machined notch

In order to allow for reproducible results in both procedures they were conducted using a specifically designed device, used to hold the blade in place and keep it parallel to the borders of the machined notch (Figure 3).

In the fist procedure, use was made of a Goldsmith hammer to perform the tapping. The procedures for introducing the pre-crack in PMMA and MDPE are distinct. In PMMA it was observed that the controlled introduction of a natural pre-crack is easy. The procedure starts with

[1] Main physical properties are taken from the manufacturer´s catalog, except when otherwise specified.
[2] Measured according to ASTM D638, using Type IV tensile specimens produced from compression molded plates. The test was conducted at 25 ± 2°C and 5 mm min^{-1} crosshead displacement rate.

tapping on the device until the natural crack is nucleated, then the tapping is carefully continued in order to direct the crack front to be perpendicular to the specimen surface and to grow approximately to the pre-defined length. In the case of MDPE, tapping is constantly performed until the razor blade penetrates a given depth (defined by a specimen holder) into the polymer matrix.

The second procedure was conducted by placing the device in the electro-mechanical universal testing machine also used for the tests. The crosshead is lowered at constant rate (0.01 mm min^{-1} for PMMA and 0.05 mm min^{-1} for MDPE) to a predefined penetration. In the case of MDPE the penetration is set to 2mm, which is also the targeted pre-crack length, while in the case of PMMA the penetration is smaller (typically 0.8 mm), defined by the operator´s experience. It was observed that the risk of obtaining a fully broken PMMA specimen using this procedure is great (roughly 10%).

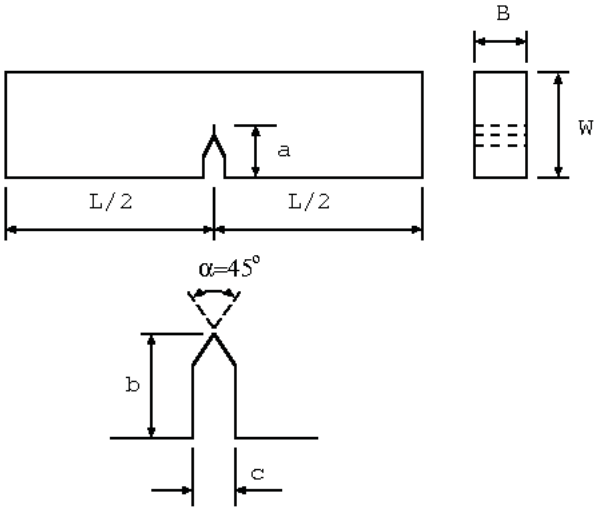


Fig. 1 – Schematic representation of the SE(B) specimen dimensions.

Material	B [mm]	W [mm]	L [mm]	b [mm]	c [mm]
PMMA	6.5	13	60	4	1.6
MDPE	14	28	130	12	1.25

Tab. 1 – Dimensions of the SE(B) specimens.

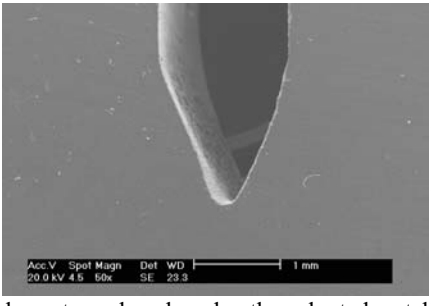


Fig. 2 – Example of notch root produced under the adopted notch machining methodology. PMMA specimen, gold plated. Secondary electron image.

Testing conditions. The fracture toughness tests were conducted in an electromechanical universal testing machine under displacement control using three-point bending configuration. The support span was set to 52 mm in the case of PMMA and 113 mm in the case of MDPE (= 4W in both cases). Load was measured using a previously calibrated 20000 N load cell and the crosshead displacement rate was set to 10 mm min^{-1}. Test procedure and result analysis followed the ESIS Test Protocol [1] except, of course, for the pre-crack introduction method. Following this Test Protocol, three specimens were tested for each condition.

The MDPE specimens were tested after being inserted for about 2 h in liquid nitrogen. The actual test temperature was not controlled, but it was estimated to be lower than the glass transition temperature of the polymer, such that it fractures as a brittle polymer during the testing. This was confirmed by the fact that the load displacement curves obtained in this case are markedly linear and that the produced fracture surfaces show brittle aspect. The test temperature for PMMA specimens was controlled and set to 25 ± 2 ^{o}C. The size of the pre-crack was determined *post-mortem* using a light optical traveling microscope.


(a)


(b)

Fig. 3 – Methods of pre-crack introduction showing the use of the device exemplified for the case of MDPE specimens. (a) – Tapping on a razor blade inserted in the machined notch and (b) – Gently pressing the razor blade in the machined notch. MDPE specimens are shown for improved visualization.

Results and discussion

Figure 4 shows a PMMA specimen with a natural pre-crack produced by tapping a razor blade in the machined notch. The second procedure produces the similar results when applied to PMMA, so only one case is shown here.

Fig. 4 – PMMA specimen with natural pre-crack produced by tapping on a razor blade. Oblique ilumination was used to evidence the pre-crack.

Figure 5 shows the trace of the pre-crack plane produced by tapping on a razor blade inserted in the machined notch in the surface of one of the PMMA specimens. Again, as already mentioned above, the results are similar for both procedures so that only this case is shown here. It is interesting to note that some kind of damage in the polymer matrix is visible below the pre-crack tip (including what appears to be voids). The nature of this damage was not determined in the present work.

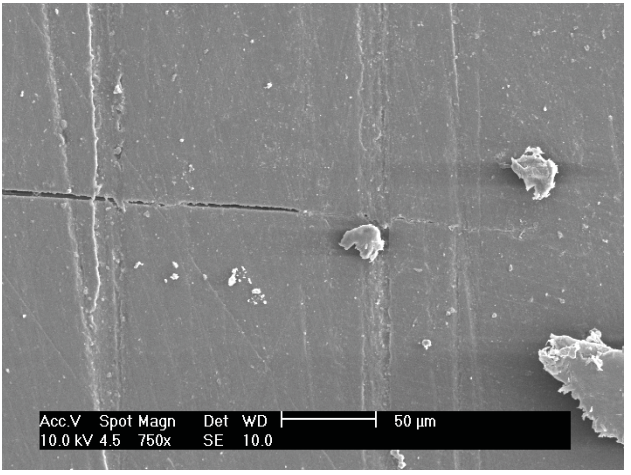
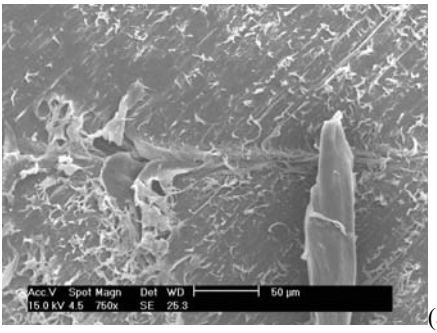


Fig. 5 – Trace of the pre-crack plane in a PMMA specimen surface. This particular pre-crack was produced by tapping on a razor blade inserted in the machined notch, but it is also characteristic of the pre-cracks produced by tapping the razor blade. Secondary electron image of the gold coated sample.

Figure 6 shows the trace of the pre-crack planes produced by both methods in the surface of MDPE specimens. Contrary to what was observed in the case of the PMMA specimens, the two methods produce radically different pre-crack morphologies. It is evident that pressing the blade introduces less damage in the matrix, in agreement with the conclusions of Lu *et al.* [2].



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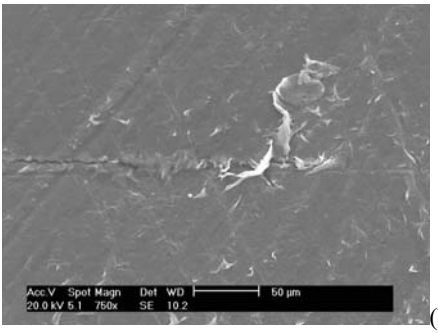
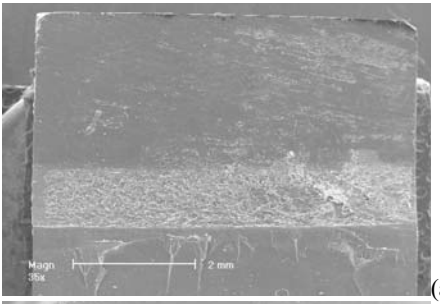


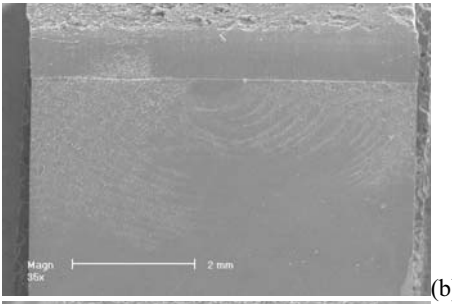
(b)

Fig. 6- Trace of the pre-crack planes in two MDPE specimen surfaces. (a) pre-crack introduced by tapping on a razer blade and (b) pre-crack introduced by gently pressing a razor blade in the machined notch. Secondary electron image, gold coated surfaces.

Figure 7 shows the fracture surfaces obtained for the broken specimens. The four images are characteristic of brittle fracture surfaces, as expected. The PMMA samples show less distinct details in the fracture surfaces, but the case in which the pre-crack is introduced by pressing the razor blade shows concentric lines that suggests that the crack nucleated near the center of the pre-crack tip line. In the case of the MDPE sample this is more clear, and both fracture surfaces present well developed "mirror" and "hackle" regions, indicating that the unstable crack nucleated near the center of the crack-tip line. In all cases it is quite obvious that the position of the pre-crack tip can be precisely measured from the observation of the fracture surfaces.



(a)



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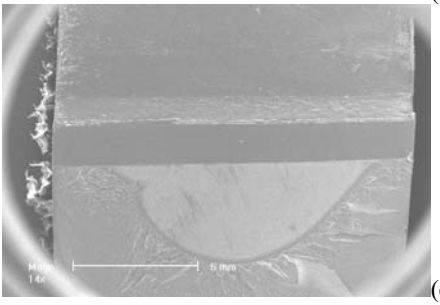

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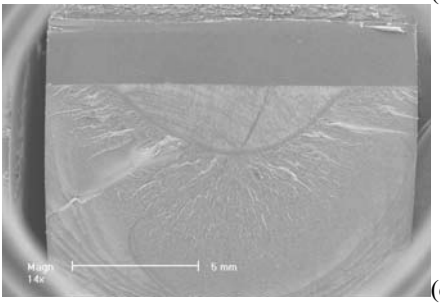

(d)

Figure 7 – Fracture surfaces of broken specimens. (a) PMMA with pre-crack introduced by tapping on a razor blade, (b) PMMA with pre-crack introducing by gently pressing a razor blade, (c) MDPE with pre-crack introduced by tapping on a razor blade, (d) MDPE with pre-crack introducing by gently pressing a razor blade. Secondary electron images, gold coated samples.

Table 2 shows the the individual results of the fracture toughness tests for the PMMA and MDPE specimens with the two pre-crack introduction procedures. For all cases the relevant geometric measures of the samples are given as well as the average and standard deviation values. Adopting the measured fracture strength of PMMA and estimating the yield stress (> 60 MPa) at low temperatures using literature data [7] it is possible to show that all samples fulfill the geometric criteria for plane strain dominance, therefore the fracture toughness results are representative of K_{Ic} for both materials.

A simple statistical test can be applied to these average values and show that, within 95% confidence, the pre-crack introduction method affects the measured K_{Ic} values in the case of the PMMA specimens. The MDPE results show a larger dispersion and this does not allow to conclude that the pre-crack introduction procedures affect the measured fracture toughness values, but it is interesting to note that the fracture toughness values measured for samples with pre-crack introducing by tapping razor blades are lower than the ones in which the pre-cracks were introduced by pressing the razor blades.

PMMA, tapping on a razor blade in the machined notch, σ_f = 69 MPa								
Specimen	W [mm]	B [mm]	a [mm]	P_Q [N]	K_Q [MPa $m^{1/2}$]	$(K_Q/\sigma_f)^2/\pi$ [mm]	$<K_{Ic}>$ [MPa $m^{1/2}$]	Std. Dev.
1	13.0	6.7	5.82	104.0	1.24	0.103	1.11	0.14
2	13.1	6.6	5.68	99.0	1.14	0.087		
3	13.1	6.5	5.53	85.0	0.96	0.062		
PMMA, gently pressing a razor blade in the machined notch, σ_f = 69 MPa								
Specimen	W [mm]	B [mm]	a [mm]	P_Q [N]	K_Q [MPa $m^{1/2}$]	$(K_Q/\sigma_f)^2/\pi$	$<K_{Ic}>$ [MPa $m^{1/2}$]	Std. Dev.
1	13.2	6.5	4.42	194.2	1.73	0.2	1.81	0.14
2	13.0	6.5	4.92	195.2	1.97	0.26		
3	13.1	6.4	4.17	198.0	1.73	0.2		
MDPE, tapping on a razor blade in the machined notch, σ_y > 60 MPa								
Specimen	W [mm]	B [mm]	a [mm]	P_Q [N]	K_Q [MPa $m^{1/2}$]	$(K_Q/\sigma_y)^2/\pi$	$<K_{Ic}>$ [MPa $m^{1/2}$]	Std. Dev.
1	28.2	13.8	13.48	835.5	3.59	<1.13	3.42	0.42
2	28.0	13.9	13.67	846.3	3.74	<1.24		
3	28.1	13.8	12.88	727.7	2.95	<0.77		
PMMA, gently pressing a razor blade in the machined notch, σ_y > 60 MPa								
Specimen	W [mm]	B [mm]	a [mm]	P_Q [N]	K_Q [MPa $m^{1/2}$]	$(K_Q/\sigma_y)^2/\pi$	$<K_{Ic}>$ [MPa $m^{1/2}$]	Std. Dev.
1	28.0	14.1	14.63	1.178.8	5.72	<2.9	4.69	0.89
2	27.9	14.1	14.31	886.5	4.18	<1.54		
3	27.9	14.1	14.19	900.3	4.18	<1.54		

Table 2- Results from the fracture toughness tests.

Conclusions

Two different procedures adopted in the literature to introduce pre-cracks in fracture toughness specimens of polymers (by tapping on a razor blade inserted in the machined notch and by gently pressing a razor blade in the machined notch) were applied to two resins of different properties, PMMA, as an example of a brittle polymer, and MDPE, as an example of a tough polymer (at room temperature). It was observed that tapping on a razor blade apparently introduced more damage in

the polymer matrix of MDPE, but that no visible changes in pre-crack morphology are observed in the case of PMMA.

From the perspective of practicability, tapping on a razor blade resulted in better control over the production of the specimens (particularly in PMMA), while gently pressing the razor blade resulted in less damage. The analysis of test results showed that the pre-cracking method affect the value of fracture toughness in the case of PMMA with 95% confidence. The dispersion of values in the case of MDPE is larger and this does not allow to conclude that the pre-cracking method affects the measured value of fracture toughness, however, the values measured when the pre-crack was introduced by tapping on the razor blade are systematically lower than the alternative method.

The most conservative measured value of plane strain fracture toughness of PMMA was 1.11 MPa $m^{1/2}$ and that of MDPE (under cryogenic conditions) was 3.42 MPa $m^{1/2}$.

Acknowledgments

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