Influence of crack sharpness on the fracture toughness of epoxy resins

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Abstract Notch sharpening of polymeric fracture samples is still an unsolved problem. The existing methodologies for the evaluation of the fracture toughness of polymers recommend the use of contact techniques such as razor sharpening which, in some cases, can induce high scatter in toughness results or even overestimated values.

The present work analyzes the effect of crack sharpness on the fracture toughness of epoxy resins. Three-point bending tests were carried out on specimens with a wide range of tip radii, going from the size of natural cracks, ~ 0.1 μm, to 2 mm. The behavior of the fracture toughness versus the crack tip radius has been modeled using a two criterion elastic model, a length and a stress, to estimate the overestimated toughness values with the crack tip radius and the effect of blunting arising from plastic deformation.

Keywords fracture toughness, crack sharpness, epoxy resins

1. Introduction

The influence of crack sharpness on the fracture toughness values of polymeric materials has recently caught the attention of the scientific community. Several works have appeared in the literature highlighting the role of the notch sharpening technique on the fracture parameters of either semi crystalline heterogeneous polymeric materials such as ethylene-propylene block copolymers [1-3] or amorphous polymers such as polycarbonate [4]. In all these studies, the values of the fracture toughness obtained using different approaches were determined from samples sharpened via two procedures: a non contact technique based on the femtosecond laser ablation and the traditional contact technique using a razor blade. Independently of the material under study, the results always followed the same trend. The fracture toughness determined from the samples sharpened via the femtosecond laser ablation technique showed lower values than those obtained from the specimens sharpened via the traditional razor blade technique. The reason is that the femtosecond pulsed laser ablation is characterized by very rapid creation of vapor and plasma phases, negligible heat conduction and the absence of liquid phase [5-6]. Thus, this technique can remove the material of the notch tip by ablating it with almost no heat dissipation, preventing melting and thermal deformations of the surrounding area. However, the specimens sharpened using the razor blade always showed damage ahead of the crack tip in form of plastic deformation which seems to be the reason of the higher fracture toughness values.

All these discoveries have aroused some questions concerning the well established criteria of the quality of the notch. Both ESIS (European Structural Integrity Society) [7-9] and ASTM (American Society for Testing and Materials) [10-11] indicate that when a natural crack cannot be generated in polymers by either fatigue cracking, due to hysteretic heating, or by tapping on a new razor blade placed in the notch, the sharpening can be achieved by sliding a new razor blade into the root of the machined notch. The resulting crack tip radius must be lower than 20 μm.

In the light of the previous results, the aim of the present work is to investigate more deeply the effect of the crack tip radius on the fracture parameters of a brittle polymer such as an epoxy resin. Special attention will be paid to the reasons of the overestimated values in the fracture toughness with the crack tip radius and the influence of blunting will be analyzed using an elastic model modified for blunt cracks [12].

2. Theoretical background

Kinloch and Williams [13] showed that in brittle polymers such as epoxy resins, the localized plastic deformation that occurs at the crack tip prior to crack propagation is a controlling factor and thus emphasising the role of crack tip blunting at the instant of fracture. A model based on the elastic solution for the stresses around the tip of an elliptical hole when subjected to a remote tension, σ, normal to the semi-major axis, a, is used to analyze the fracture toughness of specimens with blunt cracks [12]. During loading a specimen with an initial crack tip radius, R_0, the plastic deformation occurring at the crack tip front with length r_C and stress σ_C induces further crack tip blunting resulting in an increase in the crack tip radius, R_C (Figure 1).

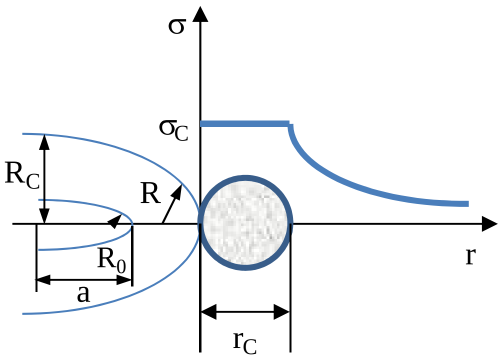

Figure 1. Crack tip front at the instant of fracture of specimens with blunt cracks

The stress σ_C at a distance r_C from the notch tip may be written in terms of the tip radius at the instant of fracture, R, and the remote applied tension σ as [14]:

$$\frac{\sigma_C}{\sigma} = \frac{2\sqrt{a}\left(R + r_C\right)}{\left(R + 2r_C\right)^{3/2}} \tag{1}$$

For a perfectly sharp crack, R=0, we have:

$$\frac{\sigma_C}{\sigma} = \sqrt{\frac{a}{2r_C}} \tag{2}$$

and thus, the fracture toughness of a sharp crack, K_{IC}, can be expressed as a function of either the remote applied tension σ and the crack length a or the near field tension at the crack tip σ_C and length factor at the crack front through:

$$K_{IC} = \sigma\sqrt{\pi a} = \sigma_C \sqrt{2\pi r_C} \tag{3}$$

For blunt cracks, the high level of stress developed at the crack tip implies the use of an apparent fracture toughness, K_b, that is, the fracture toughness of an equivalent sharp crack specimen with a stress distribution at the instant of fracture identical to that of a specimen with a blunt crack. This apparent fracture toughness, K_b, is related to the fracture toughness of the material, K_{IC}, via:

$$\frac{K_b}{K_{IC}} = \frac{\left(1+\frac{R}{2r_C}\right)^{3/2}}{\left(1+\frac{R}{r_C}\right)} \tag{4}$$

Since we are using the elastic field, the contained yielding may be modelled using a line zone with a stress identical to the yield stress, σ_Y, giving a crack opening displacement, δ:

$$\delta = \frac{K_b^2}{E\sigma_Y} \tag{5}$$

For a crack of original radius R_0, the plastic deformation characterized by δ will further blunt the tip and assuming smooth blunting, then:

$$R = R_0 + R_C = R_0 + \frac{\delta}{2} \tag{6}$$

So, the increase in the crack tip radius at the instant of fracture is equal to half the crack opening displacement. On the other hand, the crack tip radius at the instant of fracture, R, can be also expressed as function of the R_0 and R_C through the expression:

$$\sqrt{\frac{R}{r_C}} = \sqrt{\frac{R_0}{r_C}} + \sqrt{\frac{R_C}{r_C}}\left(\frac{K_b}{K_{IC}}\right) \tag{7}$$

Thus, the fracture for blunt cracks is going to be defined in terms of σ_C and r_C, which is also compatible for sharp cracks. For blunt cracks, it is the comparison of R and the length factor r_C which determines the influence of the crack tip radius.

The apparent fracture toughness, K_b, can be obtained from the experimental fracture toughness values, K_I, through the equation:

$$K_b = \frac{2}{\pi}\hat{K}\left[\ln\left(\sec\left(\frac{\pi}{2}\frac{K_I}{\hat{K}}\right)\right)^2\right]^{1/2} \tag{8}$$

where $\hat{K}$ is the plastic fracture toughness, that is, a plastic collapse condition of the uncracked specimen ligament (the width effect [13]) is to be taken into account as the stress level at failure increases with the initial crack tip radius, R_0. $\hat{K}$ has been calculated following the guidelines given in [12].

Finally, combining equations (4) and (7), we have

$$K_I \approx K_{IC}\left[\frac{1}{2\left(1-\sqrt{\frac{R_C}{8r_C}}\right)}+\frac{\frac{R_0}{8r_C}}{\left(1-\sqrt{\frac{R_C}{8r_C}}\right)^2}\right]^{1/2} \quad (9)$$

For $\frac{R_0}{8r_C}$ too many greater than one, equation (9) reduces to:

$$K_I \rightarrow K_{IC}\sqrt{\frac{R_0}{8r_C}} \quad (10)$$

Replacing K_{IC} by the equation (3), equation (10) may be written by

$$K_I \rightarrow \sigma_C \frac{\sqrt{\pi}}{2}\sqrt{R_0} \quad (11)$$

From this analysis, it is suggested that K_I will be larger, i.e. ductile behaviour, and the criterion for this condition can be written as

$$\frac{\pi}{8}\frac{\sigma_C^2}{E\sigma_Y}=1 \quad (12)$$

where E is the Young´s modulus of the material.

3. Experimental procedure

The material under study was a commercial diglycidyl ether of bisphenol A (DGEBA) epoxy resin with a Young´s modulus of 2.65 GPa and a yield stress of 85 MPa. The chosen configuration for the fracture toughness tests was the Single Edge Notch Bend specimen (SENB) with overall dimensions of 6x12x54 mm^3. The initial crack length after sharpening to width ratio was 0.5.

All the fracture specimens presented an initial V-machined notch. Different notches were introduced in the materials which can be mainly divided into two groups: samples with sharp cracks and those with blunt notches. The sharp cracks were obtained by two procedures: the traditional contact technique for brittle polymers as tapping and a non-contact technique as the use of a femtosecond pulsed laser ablation or femtolaser. For the contact procedure, a light hammer was used to tap a razor blade placed into the root of the machined notch till a natural crack was attained. The natural crack was inserted with the razor blade at room temperature and frozen at liquid nitrogen temperature. In case of the non-contact technique, the sharpening of the notch was produced through a Femtolaser [5-6], using a commercial Ti:sapphire oscillator (Tsunami, Spectra Physics) plus a regenerative amplifier system (Spitfire, Spectra Physics) based on chirped pulse amplification (CPA) technique. For the epoxy, 120-fs pulses at 395 nm with a repetition rate of 1 kHz were utilized. The scanning speed was 130 μm/s and 4 passes were carried out with pulse energy of 0.008 mJ. The sharp length inserted by the femtolaser was of 500 μm.

For the blunt notched samples, different techniques were used to achieve different crack tip radii which can be divided into three groups depending on the dimensions. To introduce notches with 0.01, 0.02, 0.05, 0.07 and 0.1 mm in diameter, razor blades produced at the Imperial College with different crack tip radii were employed. The razor blade was slid gently across the root of the notch trying to remove the material without pressing.

To introduce notches bigger in size, that is, with diameters of 0.4, 0.6, 0.8, 1, 1.5, 2, 2.5 and 3 mm, a drilling technique was employed using drills with different radii mounted on a high speed drilling machine.

Finally specimens with up to 4 and 7 mm in notch diameters were analyzed and for the insertion of such huge notches a milling machine was used.

Prior to testing, the surfaces of every single specimen were analyzed via optical microscopes and scanning electron microscopy to check the crack tip front and measure the crack tip radius. The samples which presented any type of damage in form of microcracks or areas with different colour or texture were discarded.

The tests were carried out in an electromechanical testing machine (Instron 3365) at room temperature under displacement control at a cross-head rate of 10 mm/min. A three point bending fixture was used with a loading span four times de width. A load cell of ± 1 kN and an extensometer were used to measure the load and load displacement line, respectively. The methodology employed to obtain the fracture toughness and the energy release rate was the ESIS TC4 protocol entitled "K_C and G_C at slow speeds for polymers" [7].

After the tests, the morphology of the fracture surfaces was inspected via optical microscopes.

4. Results and discussion

4.1. Experimental results

Figure 2 shows the experimental fracture toughness values, K_I, as a function of the initial crack tip radius, R_0. As expected, the fracture toughness increases with the notch tip radius. The fracture toughness of the specimens with a natural crack obtained by tapping presented the lowest fracture toughness values, ~0.5 $MPa \cdot m^{1/2}$, and of course the smallest crack tip radii, of 0.2 μm. It is worth mentioning that a small increase in the crack tip radius implies a huge increase in the fracture toughness. For example, even the specimens with sharp cracks sharpened via femtolaser presented values of the fracture toughness almost twice the value obtained from specimens with a natural crack, as the crack tip radius of the former was 0.8 μm, four times bigger.

The rapid increase of the fracture toughness with the crack tip radius is due to blunting as the fractographic analysis reveals. There is a marked change in the morphology of the fracture surfaces as the crack tip radius increases. Figure 3 shows a characteristic fracture surface of a specimen with a sharp crack. As shown, the fracture surface is plain and smooth without any remarkable characteristic.

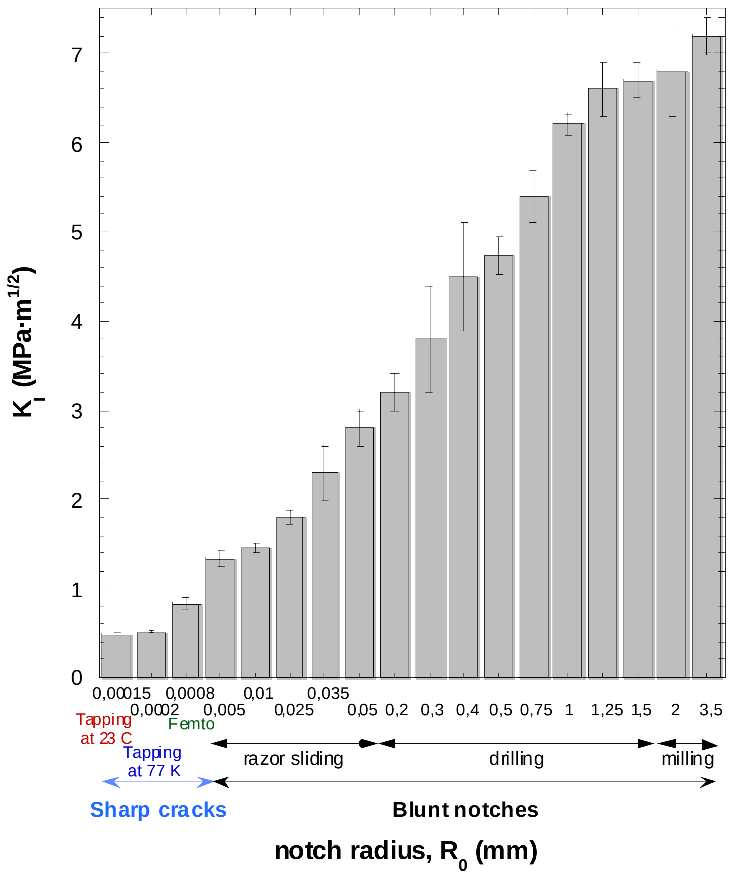

Figure 2. Experimental fracture toughness, K_I, versus the original initial crack tip radius, R_0.

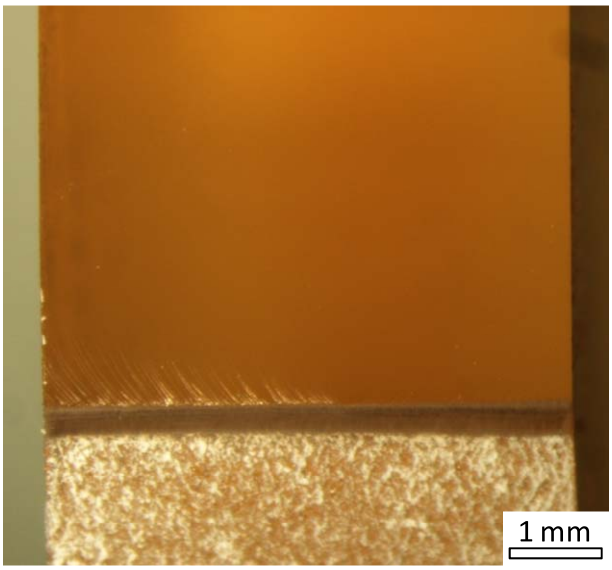

Figure 3. Characteristic fracture surface of a specimen with a sharp crack (specimen sharpened using a femtolaser)

On the other hand, Figure 4 displays the characteristic morphology of the fracture surface of a specimen with a blunt crack introduced via razor sliding. The surface is mainly plain and smooth as in the case of specimens with sharp cracks but some river markings appear close to the notch and become larger and more accentuated as the notch radius is higher.

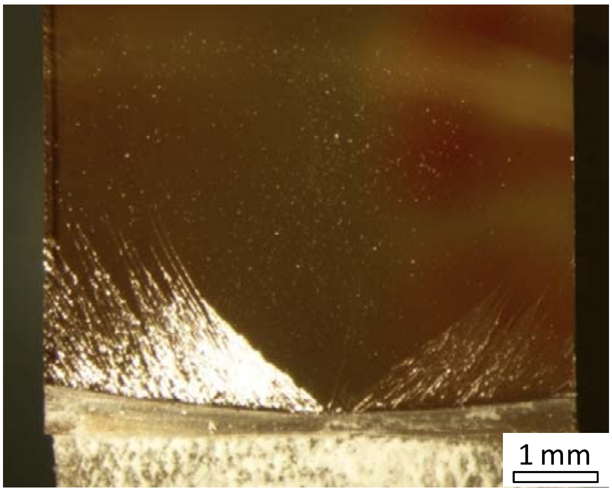

Figure 4. Characteristic fracture surface with a blunt notch introduced via razor sliding (initial crack tip radius of 25 μm)

The morphology of the fracture surfaces of the specimens with blunt notches inserted via drilling or milling changes drastically (Figure 5). The river markings are not longer limited to a tiny area close to the notch but they are spread all over the surface and the roughness is increased with the crack tip radius. Looking more closely to the surface, there is a more or less rounded region from where the unstable crack propagation seems to emerge. This type of morphology is caused by blunting.

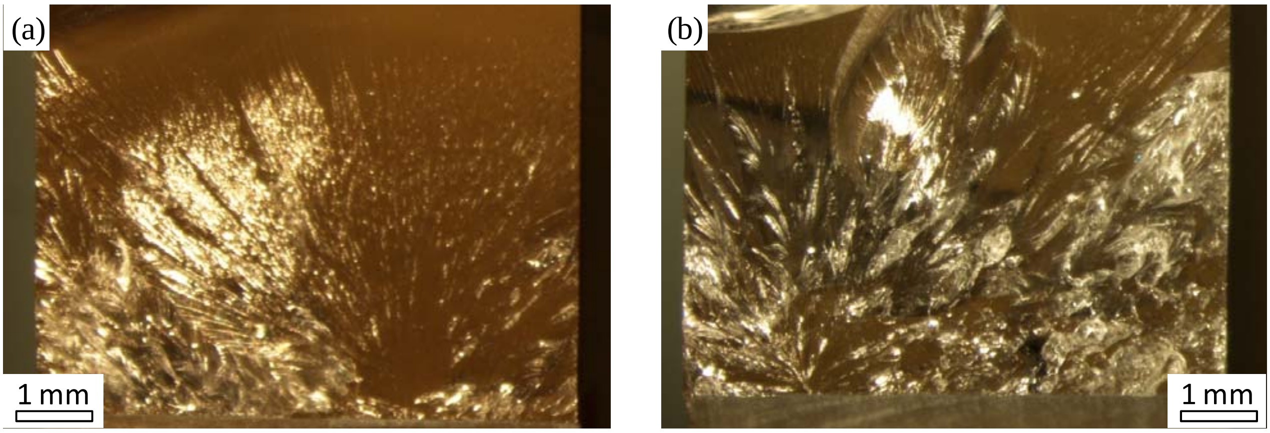

Figure 5. Fracture surfaces of the specimens with blunt notches inserted via (a) drilling (initial crack tip radius of 300 μm) and (b) milling (initial crack tip radius of 1.5 mm)

4.2. Modelling

The model described in the theoretical background was applied to the experimental data presented here to find out the role of blunting on the high values of the fracture values with the increase in the crack tip radius through the parameters σ_C and r_C. Figure 6 shows the apparent fracture toughness, K_b, against the square root of the initial crack tip radius, $\sqrt{R_0}$. Each colour groups the fracture values of specimens with notches introduced via a specific notching technique. In general and independently of the notching procedure, the true fracture toughness increases monotonically with the crack tip radius although some slightly deviations are to be pointed out. First of all, the values obtained from specimens with blunt notches introduced by razor sliding slightly divert from the general trend. These values are higher than expected and the microscopic analysis of the virgin specimens reveals the reason (Figure 7). There is a tiny region ahead of the crack tip with different texture corresponding to some damage caused during the notching process (outlined with a red dotted line). Secondly, from $K_b \approx 7$ MPa·m$^{1/2}$, the increase in the true fracture toughness with the crack tip radius is not so marked and it seems that from there on the values are tending to a horizontal asymptote.

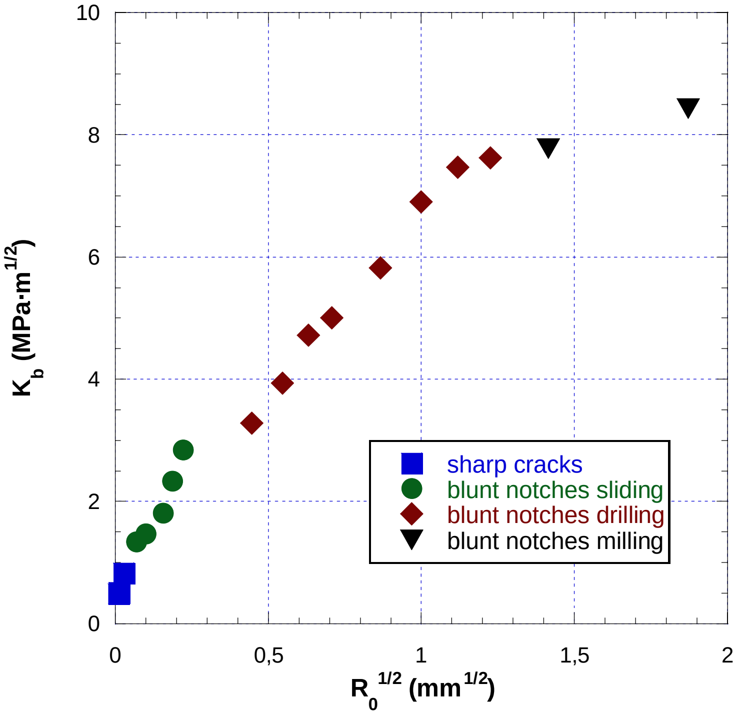

Figure 6. Apparent fracture toughness as a function of the initial crack tip radius

Figure 7. Crack tip front of a specimen with a notch introduced via razor sliding where some damage can be observed ahead of the crack front (initial crack tip radius of 35 µm)

Figure 8 shows K_b vs $R_0^{1/2}$ together with the fitting to equation (9) and table 1 gives the parameters of the model. The fracture toughness resulting from the fitting of the data to equation (9) gives a value of 0.89 MPa·m$^{1/2}$, which is higher than the experimental fracture toughness obtained from specimens with sharp cracks (0.49-0.83) MPa·m$^{1/2}$ but lower than the fracture toughness obtained from samples with blunt notches introduced via razor sliding (1.34-2.80) MPa·m$^{1/2}$ (Figure 2).

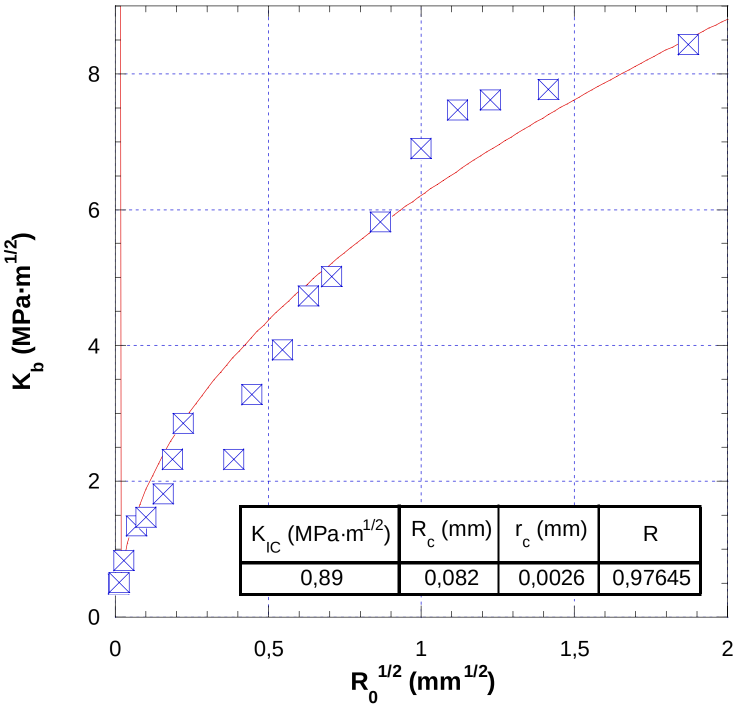

Figure 8. True fracture toughness versus the initial crack tip radius. Line is fitted from equation (9).

Concerning the parameters related to the fracture process, r_C and σ_C describe the critical length and the cohesive stress, respectively. The values of r_C= 2.6 μm and σ_C= 220 MPa are comparable to those found in the literature for similar epoxy resins [13]. It is interesting to note that the value of the plastic condition given by the equation (12) is far away from the unity, so the degree of ductility is relatively small. Finally, the value of the increase in the crack tip radius at the instant of fracture due to blunting, R_C, is 74 μm.

Table 1. Parameters of the model resulting from the data fitting to equation (9)

K_{IC} (MPa·m$^{1/2}$)	R_C (μm)	r_C (μm)	σ_C (MPa)	$\frac{\sigma_C}{\sigma_Y}$	$\frac{\pi}{8}\frac{\sigma_C^2}{E\sigma_Y}$
0.89	82	2.6	220	2.6	0.08

5. Conclusions

This work analyzes the influence of the crack tip radius on the fracture toughness on a brittle polymer such an epoxy resin. Different sharpening techniques were employed to attain different crack tip radii. To introduce sharp cracks, the traditional contact technique such as tapping a razor blade placed into the root of a machined notch and the more sophisticated non-contact procedure as the used of the femtosecond pulsed laser ablation were used. For the blunt notches, the procedures of razor sliding, drilling and milling were utilized to get different crack tip radii. The lowest values of the fracture toughness were obtained for the specimens with sharp cracks, in particular for those with cracks inserted via tapping. The fracture toughness increased rapidly with crack tip radius and the microscopic analysis of the fracture surface indicated that blunting was the reason of the steady increase.

A model based on the line zone model was applied to the experimental data to achieve a useful insight into the crack tip sharpness effects. The parameters obtained from the model denote that the degree of ductility reached during the fracture process is not large and the plastic length and stresses developed in the crack tip front before the unstable crack propagation are comparable to the critical length and cohesive stress.

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