INFLUENCE OF THE SPECIMEN NOTCHING ON THE ESSENTIAL WORK OF FRACTURE OF DUCTILE POLYMER FILMS

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1. Introduction

Films and thin sheets of polymers, polymer blends and polymer composites are used in a wide variety of applications like packaging, agriculture, coating, and paints market segment. Their toughness is often a basic requisite to meet some industry needs.

The linear elastic fracture mechanics (LEFM) approach is used to study fractures occurring at nominal stresses well below the material yield stress. The dissipated energy is confined in a small area near the crack tip, and the fracture is brittle. The LEFM approach is not applicable when the plasticity around the crack tip becomes too large, in those cases the elastic plastic fracture mechanics (EPFM) apply. When the crack propagation occurs through a highly deformed and yielded material then the post-yield fracture mechanics (PYFM) can also be applied and the essential work of fracture (EWF) is a suitable method.

The EWF characterises the plane stress toughness of ductile polymer films in mode I, basically using the double edge notched tension (DENT) configuration.

The EWF method has become very popular and is increasingly used due to the apparent simple specimen preparation and the easy testing.

Many works have been published on the EWF method applied to polymers with the aim of determining their fracture properties. There is an excellent review [1] of the EWF with an extensive summary of published works on polymers.

The EWF experimental method was developed by Cotterell and Mai [2,3] following ~~the~~ Broberg's theoretical idea [4,5]. According to the theory, the value that represents the toughness, namely the specific essential work of fracture (w_e) is a material parameter, only if the ligament fully yields before the onset of crack propagation and is independent of the specimen geometry.

There is a European Structural Integrity Society (ESIS) test protocol [6] for the EWF method, and several round-robin exercises [6-7] have been performed under the guidance of the ESIS technical committee 4 (TC4).

In spite of the apparent simplicity of the test, some aspects of the validity of this technique remain controversial; there are intricate details that seem to play an important role in the repeatability and reproducibility of the EWF test. This problem has been and is still debated, and these questions indicate that the EWF procedure is not yet sufficiently defined to be considered as a standard.

Some of the aspects of the test validity are related to the specimen preparation, particularly the notching technique, and they should be studied more deeply.

The main aim of this work is to contribute to a better understanding and the meaning of some of the controversial factors involved in the EWF test.

Specimen manufacturing and specimen alignment in the test machine grips have been studied in detail in this work.

Particularly, the effect of different quality notches on the shape of the experimental stress-displacement curves and how these shapes are directly related to the EWF parameters w_e and βw_p, are analysed in detail.

Furthermore, there is a limited understanding of the types of polymers in which the EWF test may be applicable. The EWF approach has been successfully applied to amorphous and semicrystalline polymers that undergo necking before crack propagation. However, there is some controversy of the applicability of the EWF test to multiphasic polymers which can have other deformation micromechanisms. This is the reason why a rubber-toughened, an ethylene-propylene block copolymer (EPBC), was selected for its application in this EWF work.

2. EWF approach

2.1. EWF concept

The EWF theory is based on the hypothesis that the total energy W_f involved in the ductile fracture of a precracked specimen can be separated in two terms.

$$W_f = W_e + W_p \tag{1}$$

where W_e, the essential work of fracture, accounts for the energy necessary to generate new crack surfaces while W_p is called the plastic work or the non-essential work of fracture and includes all the other components of energy dissipated in the fracture process.

The EWF concept establishes that the process zone can be divided into an inner process zone (IPZ) where the fracture process actually occurs and an outer process zone (OPZ) (Fig. 1). Thus, W_e is proportional to the IPZ area while W_p is proportional to the volume of the OPZ. Using these considerations, Eq. (1) can be rewritten in specific terms as

$$w_f = \frac{W_f}{l_o.t} = w_e + \beta w_p.l_o \tag{2}$$

where l_o is the original ligament length, t is the specimen thickness and β is a factor related to the shape of the OPZ.

Eq. (2) can be assessed by performing a series of tests on specimens with different original ligament lengths where load vs displacement (Fig. 2) is registered.

The total energy dissipated is given by

$$W_f = \int_0^{d_r} P.dd \tag{3}$$

where d_r is the displacement at complete failure.

Thus, according to Eq. (2), the total energy per unit area w_f can be plotted (Fig. 3) as a function of the l_o.

From the best-fitting regression analysis, the intercept at the origin w_e and the slope βw_p can be determined. References [1,8] reviewed the EWF approach.

2.2. Key requirements

In the EWF analysis the following key assumptions are made:

a) The ligament length is fully yielded prior to the onset of crack propagation.

This requirement ensures that the fracture mechanism is the same irrespective of the ligament length and w_e is ~~in~~ an inherent material parameter. However, this key assumption is rarely accomplished and in most of the published articles this requirement is not satisfied and thus the w_e values become an apparent toughness only useful for comparison purposes. In a DENT specimen, the ligament length will be completely yielded prior the onset of crack propagation if it is less than twice the size of the plastic zone radius, r_p, in plane stress conditions.

For a linear plastic zone

$$2r_p = \frac{\pi}{8}\left(\frac{Ew_e}{{\sigma_y}^2}\right) \tag{4}$$

and for a circular plastic zone

$$2r_p = \frac{Ew_e}{\pi{\sigma_y}^2} \tag{5}$$

where E is the elastic modulus, σ_y is the uniaxial yield stress, and w_e is the specific essential work of fracture.

Although a ligament length less than twice the plastic zone radius is a reasonable size criterion, it appears to be too restrictive considering the evidence encountered in amorphous copolyesters [1,9].

b) Fracture is under plane stress conditions.

There are constraints on the ligament length to assure a pure plane stress state on the specimens.

Arbitrarily based on experiences, it has been suggested a minimum ligament length between 3 and 5 times the specimen thickness [1,6]. This leads to specimens too small to be tested and handled. Then, a practical lower limit of 5 mm is accepted when preparing DENT specimens [7] of polymer blends, which have a thickness less than 1 mm.

The upper limit requires the full-ligament yielding before crack initiation. Hence, l_o has to be less than twice the plastic zone radius in DENT specimens. Another upper limit is given by the following relationship

$$l_o \leq \frac{W}{3} \tag{6}$$

where W is the specimen width. This last condition is necessary to prevent edge effects.

c) Good quality notches.

Identical and repetitive sharp notches without plastic deformation in front of the crack tip. This requirement guarantees self-similar load-displacement, stress-displacement, and ligament length-displacement curves for tested specimens with different ligament lengths. Ideally, the sharpened notches should be sharp enough to avoid that sharper notches result in a significantly lower value of w_e. However, the smallest reached notch tip radius was of about 1 µm.

It has been experimentally demonstrated the great influence of the notches on the EWF results, Clutton [6] and Williams and Rink [7] presented results of round robin test performed within the Technical Committee 4 of the ESIS where it was clear from the results that lower values of w_e are obtained with the sharper notches, but βw_p, the slope of the EWF plot, showed no dependence on the notch sharpness.

There are several methods used in the literature for notching film specimens, and each one generates notches with different quality that may even depend on the operator.

The femtosecond laser ablation technique (femtolaser) is a non-contact method for sharpening the pre-notches. Setting up properly is a complex matter in order to avoid thermal damage at the notch root. The application of this technique has allowed obtaining [9-11] repetitive notches with negligible thermal damage, without plastic deformation, and very sharp tip radius of about 1 µm. A limitation of the femtolaser technique is the availability of the equipment and the cost per notch.

The most popular way used to sharpen notches is the razor blade sliding technique. In this technique, the notches are sharpened by drawing a fresh razor blade across the pre-notch tip. It is advisable to do it in a single pass so that the notch follows the same track avoiding bifurcations. It has also been employed a scalpel instead of a razor blade.

Unintentional introduction of residual stresses and/or the development of a plastic zone ahead of the notch tip can be easily induced by the compressive component of the sideway sliding force. The higher ~~is~~ the yield stress of the ductile polymer, the lesser will be the extent of the plastic deformation ahead of the notch tip.

Cooling specimens below its glass transition temperature prior to sliding can be helpful.

The razor blade sliding is a manual procedure which regulary results in different sharpened notches. These differences can be in the notch tip radius and/or the extent of the plastic deformation [6].

In a set of DENT specimens, it is possible to have three different specimen~~s~~ populations which show a distinct behaviour among them and they are responsible for the non-similarity and crossing curves in the load-displacement, stress-displacement, and ligament length-displacement plots. These include specimens with two different notches, specimens with two equal notches and negligible plastic deformation, and specimens with two equal notches but both notches having noticeable plastic deformation [9].

When the specimen has two different notches, the crack initiation in both notches does not begin at the same time. Instead, when the two notches are equal, the crack initiation happens at the same time (simultaneously). Comparing specimens with and without plastic deformation at the notch root [10,12] but having the same notch tip radius it is noticed that the head of the stress-displacement curves have a different shape, which for the specimens without plastic deformation leads to a lower value for the crack tip opening displacement, that is, a lower w_e value too. However, the tails of these curves are independent of the notches and therefore show the same slope βw_p in the EWF plots.

Clearly, the EWF results are not only affected by the notch sharpness but also by the plastic deformation ahead of the notch tip.

Other procedures have been tried unsuccessfully for generating notches which include scissors cut, die-punch, and scalpel cuts generated starting from the notch tip. These techniques seem to generate plastic deformation ahead of the notch tip and provide higher w_e values that the razor blade sliding method [6] or scalpel sliding [13].

2.3 Other aspects and general considerations

Specimen

When w_e is an inherent material parameter, then it should be independent of the specimen geometry. Mai and Cotterell [14] verified this first by using different specimen geometries.

The double edge notched tension (DENT) geometry is the most appropriate for mode I testing because the transverse stresses between the notches are tensile and buckling problems are avoided [15].

The DENT geometry is shown in Fig. 1, where W is the specimen width, t is the specimen thickness, L is the specimen length, and Z is the distance between clamps. Most work in the literature has been performed using this geometry. Two logical aspects to take into account are that the notches have to be collinear and that the specimen has to be clamped perpendicular to the collinear notches.

There is experimental evidence that, when W and Z are more than twice the largest ligament value, the w_e value does not change [7,16-17]. On the other hand, when Z is more than twice W, the specimens can get wavy in their own plane during the test [16] and thus modify the stress distribution in the ligament zone. Values of Z smaller than W seem to be far away from the infinite plate case, being the result probably influenced by the cross-hedges proximity to the fracture region.

Material

All polymers which fulfill the key assumptions established by the EWF approach can be successfully tested; nevertheless, they can exhibit considerable differences among them during testing.

In single phase homopolymers and copolymers, full ligament yielding must show a load and stress drop (necking) in the related load-displacement and stress-displacement curves [1,9,15], but this is not the case for multiphasic polymers, as rubber toughened polymers, where other deformation micromechanisms take place, as multiple shear yielding or multiple crazing.

The EWF parameters can be affected, as well as the other mechanical properties, by the microstructure, degree of crystallinity, chain orientation, molecular weight, additives such as plasticisers, among others.

Maximum stress

Hill [18-19] has shown, for rigid perfectly plastic materials in the DENT geometry under plane stress conditions, that no stress can exceed the value of 1.15 σ_y. This value rises until 2.97 σ_y for pure plane strain state condition. Where σ_y is the uniaxial tensile stress.

Thus, the maximum stress registered during the DENT tests, σ_{max}, has to be comprised between σ_y and 1.15 σ_y and its value is equal for all ligaments, when there is pure plane stress conditions.

Theoretically, at lower values of l_o, the specimen can be in a mixed state of stress which increases σ_{max}, and so the Hill criterion could be employed to determine the lower limit of the ligament length. Nevertheless, in practice, the experimental scatter in the σ_{max} values difficulties the application of this criterion.

Clutton [6] suggested another criterion which does not use the yield stress but uses the mean of the maximum stresses (σ_{max}) as follows

$$0.9\sigma_{max} < \overline{\sigma}_{max} < 1.1\sigma_{max} \qquad (7)$$

The Clutton criterion only removes data where errors in dimensional measurements or loads exist though these load errors can be undetected if the same load cell is used for all tests [7,20].

Based on our experience, the limits of the Clutton criterion might be reduced to be comprised between ± 5% of σ_{max}.

Specimen thickness

It seems that in non-oriented amorphous polymers of thicknesses lower than 1 mm, the w_e and βw_p values remained unchanged. The small differences encountered in [21-22] are within the statistical error.

In the case of semicrystalline thermoplastics, differences in the EWF parameters can be found [16] but are attributed to variations in the crystallinity of the samples.

Test speed and temperature

The selected test speed is, in principle, somewhat arbitrary. Although, the magnitude of the mechanical properties varies not only with the temperature but also with the test speed as a consequence of the viscoelastic nature of the polymers.

As can be expected, Increasing test speed causes an increase in σ_{max} and a decrease in d_r, which together contribute to a drop in w_e and βw_p values [16-17,22] but it is slightly at the conventional test speeds.

Raising the temperature has the same effect as decreasing the test speed. Thus, while σ_{max} decreases and d_r increases, both values of w_e and βw_p increase [17,21,23-24].

Displacement measurements

The displacement has been usually measured from the cross-head, but it has been also measured using video extensometers [25] or digital image correlation systems [9,11].

There is experimental evidence that the w_e value is insensitive to changes of the gauge length chosen for displacement measurements. Nonetheless, the slope βw_p of the EWF plots increases slightly with incrementing the gauge length [10,25]. This slight increase in the βw_p value has been attributed [10] to a higher contribution of the viscoelastic energy when the distance between the OPZ zone and the gauge marks is larger.

3. Experimental details

3.1 Material

Polypropylene requires improved toughness at both room and low temperatures to fulfill some industry requirements . This study has been conducted on an ethylene-propylene block copolymer (EPBC).

This copolymer has been synthesised through the spheripol process using a Ziegler-Natta catalyst of the fourth generation in two steps. The microstructure consists [26] of elastomeric ethylene-propylene particles embedded in the polypropylene matrix. The presence of different phases leads to different glass transition temperatures, one associated with the elastomeric particles ($T_{g,EPR}$), and the other one with the polypropylene matrix ($T_{g,PP}$).

The mechanism responsible for the toughness reinforcement is the formation of shear bands around the elastomeric particles which absorbs most of the deformation energy. This mechanism is always accompanied by the cavitation (void formation) of the elastomeric particles.

Table 1 summarises the main characteristics of this EPBC grade. The ethylene content and the isotacticity index were measured via Nuclear Magnetic Resonance (NMR), the average molecular mass in number (M_n) and weight (M_w) were determined by Gel

Permeation Chromatography (GPC). The T_g's were revealed by dynamic mechanical thermal analysis (DMTA).

The raw material in form of pellets was kindly supplied by Repsol. Films with thickness of 0.5 mm were cast-extruded from the pellets. The melt flow index (MFI) of both the pellets and the films were determined following the ISO standard 1133 at 230°C/2.1 kg. The results showed in table 1 indicate almost no polymer degradation during the extrusion process.

3.2 Specimen preparation

Two kinds of specimens were prepared; dumbbell shaped specimens for uniaxial tensile tests and DENT specimens for EWF tests. All specimens were prepared and tested in the machine direction (MD) of the cast-extruded EPBC film.

Dumbbell shaped specimens were obtained in a cutting press with the shape and dimensions of Type IV specimen as defined in ASTM standard D 638.

The DENT specimens used to perform the EWF tests were obtained by cutting rectangular coupons 60 mm wide x 90 mm long (Fig. 1). To minimise the plastic deformation at the pre-notch root, the pre-notching on the specimens coupons were obtained by applying a saw over a sandwich formed by the EPBC specimen coupon compressed between two skin layers of a 1 mm thick coupons of polymethyl methacrylate (PMMA).

3.3 Notch sharpening

Table 2 presents a summary of the ten sets of specimens that were prepared. Two sets of specimens were pre-notched to obtain samples with ligaments separated 1 mm comprised between 5 and 20 mm.

The pre-notches in the first set of specimens were sharpened by the femtolaser ablation technique (reference SF) using optimised operation conditions [10] to avoid thermal damage.

In the second set of specimens, the pre-notches were sharpened by the traditional method of sliding a fresh razor blade (reference SG) across the pre-notch tip in only one pass to follow the same track and thus avoid bifurcations. It was intended to apply the minimum compressive force during the sideway sliding, in order to minimise both the plastic deformation and the volume material accumulation at the notch tip.

The EWF approach was applied on these two sets of specimens.

Other eight sets of specimens were also prepared. In each set, all specimens had the same ligament length, l_o = 11 mm.

Six sets were prepared with different notches, as follows:

Set 3 -S: Only pre-notched specimens

Set 4 -SD: Pre-notched + additional deformation of the notch root

Set 5 -SG: Pre-notches + sliding fresh razor blade

Set 6 -SGN: Pre-notched + sliding fresh razor blade in liquid nitrogen

Set 7 -BG: Pre-notched with scalpel + sliding fresh razor blade

Set 8 -SF: Pre-notched + femtolaser

Basically, these six sets of specimens were tested to analyse the effect of different notches on the shape of the stress-displacement curves.

The remaining two sets of specimens were prepared as the second set of specimens, but in one (set 9, DR) of these two last sets the notches were intentionally not collinear, and in the other one (set 10, AT) the specimens were tilted in the clamped zone.

During testing, all notches were collinear except in the ninth set, and all specimens were in perfect alignment within the grips except in the tenth set where the specimens were tilted 5 degrees on the grips.

The original ligament length, l_o, of the specimens were measured before and after testing using an optical microscope.

3.4. Test conditions

All tests were carried out at room temperature (23 °C ± 1~~ºC~~) using a crosshead speed of 2 mm/min on a universal testing machine (Galdabini Sun 2500, Italy) equipped with a 1 kN load cell.

Uniaxial tensile tests were performed on the dumbbell shaped specimens.

The DENT specimens were loaded in tension until rupture. The displacement was measured in one-camera video extensometer (Mintron OS-65D CCD, Taiwan), selecting for each specimen a video extensometer length mark equal to its own original ligament length.. The initial distance ~~Z~~ between grips, Z, was 60 mm.

Except when indicated, all specimens were properly placed between grips.

4. Results and discussion

4.1 Tensile test

Uniaxial tensile tests performed on the 5 dumbbell specimens provided a yield stress σ_y = 20.60 ± 0.20 MPa and an elastic modulus E = 1.03 ± 0.01 GPa.

4.2 EWF of femtolaser and razor blade sharpened specimens

16 specimens of the set 1, sharpened ~~by~~ using a femtolaser, were prepared. The notches of 3 specimens were observed in both an optical microscope (OM) and using a scanning electron microscope (SEM); therefore, 13 specimens were tested.

All notches observed through the microscopy techniques turned out to be virtually identical with a notch tip radius of 1 µm and a sharpening depth of 480 µm. In the micrographs shown in Fig. 4, it is also possible to see the absence of plastic deformation and the negligible thermal damage at the notch root.

19 specimens of the set 2, sharpened by a razor blade, were prepared. In this case, the notches have been observed using OM. In Fig. 5 can be seen how these specimens showed plastic deformation at the notch root. 16 specimens with the most similar plastic deformation were chosen, and 3 of them were rejected. Other 3 specimens were also observed by SEM. The SEM micrographs, as the one shown in Fig. 5, revealed volume accumulation of plastically deformed material at the notch root. For these specimens, the notch tip radius was of 1 μm and a sharpening depth between 500 and 550 μm. In this case, 13 specimens have been tested.

Both sharpening techniques produced similar notch tip radii and sharpening depths. The main difference between both procedures is the plastic deformation in front of the notch tip, which is absent in the femtolaser sharpened specimens but is observed in the micrographs of the razor blade sharpened specimens.

Fig. 2 shows the experimental load-displacement curves recorded for the set of femtolaser specimens. Fig. 6 shows the stress-displacement curves for the femtolaser specimens obtained by dividing the load in Fig. 2 by the original area of the cross section of the ligament length. The value of maximum average stress, σ_{max} = 20.63 ± 0.40 MPa, is equal to σ_y and its standard deviation is less~~er~~ than 5%.

Proceeding in the same way, Fig. 7 shows the stress-displacement curves obtained for the razor blade sharpened specimens. In this case σ_{max} = 20.57 ± 0.39 MPa which is also equal to σ_y and its standard deviation is also lesser than 5%.

The specific fracture energy has been found by integrating the curves shown in Figs. 6 and 7, and plotting the results as a function of the original ligament length as seen in Fig. 3. In these plots, the intercept at the origin is w_e and the slope of the regression line is βw_p. The w_e values are 36.90 and 54.00 kJ/m^2 for the femtolaser and razor blade sharpened specimens, respectively. Both sets have the same slope.

Applying Eq. (4) resulted $2r_p$ = 28 mm, and thus all specimens had fully yielded ligaments and under plane stress conditions. The full ligament yielding before crack initiation was also verified by visual inspection of the video extensometer registered frames shown in Figs. 4 and 5 for the femtolaser and the razor blade sharpened specimens, respectively.

Hashemi and O'Brien [27] applied to DENT specimens a method for obtaining the critical crack tip opening displacement $CTOD_c$ (CTOD value at the onset of crack propagation). The following linear relationship exists under plane stress conditions

$$d_r = CTOD_c + \alpha . l_o \tag{8}$$

where d_r is the displacement at rupture and α ~~is~~ the propagation contribution to the displacement.

In Fig. 8 is plotted the displacement at rupture as a function of the ligament length for the femtolaser and razor blade sets of specimens. The best linear regression line was drawn to each set of data. The intercept values at zero ligament length or $CTOD_c$ are 1.96 and 2.70 mm for the femtolaser and razor blade sets of specimens, respectively.

The hatched area represented under the σ-d curves in Figs. 6 and 7 is equal to the w_e values of 36.9 and 54.0 kJ/m^2 for the femtolaser and razor blade sharpened specimens, respectively. A set of overlapping curves (heads) can be observed in the low displacement range up to a fairly well recognizable value d_i from which the curves (tails) start to diverge. These hatched areas begin at d = 0 and finish when the displacements are $d_i = 1.97 \pm 0.05$ mm for the femtolaser specimens and $d_i = 2.84 \pm 0.06$ mm for the razor blade specimens. These values agree very well with their corresponding $CTOD_c$ values found when Eq. (8) was applied. This is, at d_i crack propagation is initiated and when the displacements are larger than d_i there is crack propagation and the curves depart from each other.

The crack initiation stress can be identified as the stress value at displacement initiation d_i or $CTOD_c$. In Figs. 6 and 7 these values are $\sigma_i = 18.90 \pm 0.37$ MPa and $\sigma_i = 18.82 \pm 0.26$ MPa for the femtolaser and razor blade specimens, respectively. Both sets have practically the same σ_i value.

In Figs. 4 and 5 are represented some video extensometer recorded frames for specimens having ligament lengths equal to 11 mm. A very careful observation on these frames makes possible to recognise the onset of crack initiation in both sets of specimens which, in addition, have the same σ_i values, aforementioned.

The hatched area represents the energy per surface unit required to create two new surfaces in a cracked body submitted to an external load, it is then a specific energy up to crack initiation, and so w_e corresponds to an initiation-specific energy.

An identical behaviour has been previously found in other polymer films [9-10], where w_e has also been experimentally identified having the same value that J_o, the J-integral value at the onset of crack initiation.

The following relationship [9-10]

$$w_e = \sigma_i . CTOD_c \tag{9}$$

is successfully accomplished for both sets of specimens, independently of the sharpening method.

In Fig. 9 are represented the stress-displacement curves for specimens with l_o = 17 mm, sharpened by femtolaser and razor blade. It is clear that both specimens have equal σ_{max} and σ_i values, but the $CTOD_c$ and d_r of the razor blade sharpened specimen are larger than the femtolaser sharpened specimen, resulting a higher w_e value for the razor blade sharpened set of specimens.

Another way to represent the data obtained with these two sharpening methods can be performed by shifting the stress-displacement curves of the same ligament length (Fig. 9) along the axis of displacement so that the rupture displacement d_r coincides, as shown in Fig. 10. Doing so, the displacement values at the onset of crack initiation d_i fall in the same place, and the tails overlap. With such representation, furthermore, it can be deduced that the propagation energy is the same for both sharpening methods, and then the slope βw_p is the same for both sets of specimens.

The stress-displacement curves of specimens sharpened by femtolaser and razor blade with the same l_o overlap, (Fig. 9) from the displacement d = 0 to the displacement point with coordinates at (σ_{max},$d_{\sigma max}$). The distance between $d_{\sigma max}$ and d_i is larger (Fig. 10) for the razor blade sharpened specimen; meanwhile the tails of the curves comprised between d_i and d_r also overlap (Fig.10). The same trends have been observed in other polymer films [8-11] when both, femtolaser and razor blade sharpened specimens were tested.

The tails of the stress-displacement curves (Figs. 6 and 7) can be normalized as is shown in Figs 11 and 12 for the femtolaser and razor blade sharpened specimens, respectively. All the normalized curves for the same sharpening overlap. Although Figure 11, which corresponds to the specimens sharpened by a razor blade, shows 2 specimens with a small discrepancy when overlapping, this is probably due to small differences in the notches compared to the other specimens. Figs. 11 and 12 will also overlap if a single graph of both is made, confirming that both sets of specimens have the same propagation behaviour.

Considering a set of specimens with the same crack tip radius but containing some specimens with and others without plastic deformation at the crack root, after tested, all specimens would have the same σ_{max} and σ_i but different d_i, resulting in crossing curves in the load-displacement and stress-displacement plots.

4.3 Shape of the stress-displacements curves for different notches

It is clear that the shape of the stress-displacement curves is influenced by both, the notch tip radius and the plastic deformation at the notch root.

From the analysis of the set 1 and 2 corresponding to the femtolaser and razor blade sharpened specimens, respectively, it has been noticed the direct relationship between the shape and size of the stress-displacement curve and the EWF results.

Different notched sets containing a large number of specimens with repetitive notches are very difficult, practically impossible even, to be obtained.

Sets with a small number of specimens, with repetitive notches, and with the same original ligament length are less difficult to manufacture.

Then the shape and size of the stress-displacement curves obtained with these short sets of differently notched specimens do make suitable to analyze their EWF behaviour.

So as to investigate this influence has been prepared the specimen sets from 3 to 8 in table 1 which have the same ligament length (l_o = 11 mm) but different kinds of sharpened notches.

The third set was pre-notched forming saw cut slots and its main characteristics are summarised in Fig. 13. In here, the optical micrograph of the notch shows relatively small plastic deformation at the notch root. The crack initiation, indicated with arrows, can be estimated from a careful observation of the video extensometer registered frames. The corresponding point to this frame is signalized by a small circle on the stress-displacement curve, also represented in Fig. 13. The stress-displacement curves of the 4 tested specimens were averaged and the resulting curve S is depicted in Fig. 14.

In the fourth set of specimens, the pre-notches forming saw cut slots were intentionally deformed plastically, resulting in a large radius of curvature, as can be observed in the optical micrograph showed in Fig. 15. In this figure is also represented the stress-displacement curve and the circle traced over this curve indicates the point of crack initiation which was estimated visually on the recorded frame marked by arrows. The average curve SD of the 4 tested specimens is shown in Fig. 14.

The fifth set of specimens was sharpened as the second set, and in Fig. 5 are summarised the results for l_o = 11mm. 3 specimens were tested, and the mean stress-displacement curve SG is shown in Fig. 14. The initiation point of propagation marked by a circle on the stress-displacement curve (Fig. 5) which in turn corresponds to the frame containing arrows is coincident with the d_i and σ_i values found on the set 2 of specimens.

The sixth set of specimens was sharpened by the classic method of sliding a fresh razor blade across the pre-notch tip, but in this case, the sharpening was carried out over frozen specimens by liquid nitrogen. Fig. 16 summarises the results obtained. The optical micrograph shows less plastic deformation at the notch root and lower sharpening depth extension than the obtained by just using the razor blade at room temperature. The reduction in plastic deformation is expected because the sharpening occurs when the EPBC is below its glass transition temperature, T_g. The SEM micrograph provides a radius of curvature in the notch tip of about 1 μm, similar to the value found in the SF and SG sets of specimens. The circle on the stress-displacement curve indicates the initiation point of propagation found by visual inspection (arrows) on the registered frames. 3 specimens were tested, and the average stress-displacement curve SGN is represented in Fig. 14.

The seventh set of specimens was pre-notched with a scalpel and sharpened by sliding a fresh razor blade on the notch root. The results are summarised in Fig. 17. Here in the optical micrograph can be observed a large plastic deformation at the notch root which is also observed in the SEM micrograph as some volume accumulation of plastically deformed material at the notch root. The mean stress-displacement curve BG of 3 tested specimens is represented in Fig. 14.

The eighth set of specimens was sharpened by femtolaser as the set 1 of specimens analysed before. The summarised results for l_o = 11 mm are shown in Fig. 4 with the circle, frames, and arrows having the same meaning as in the other sets. 3 specimens were tested and the average stress-displacement curve SF is also depicted in Fig. 14.

The average of the stress-displacement curves has been performed to minimise the differences caused by the very slight differences among the original ligament lengths, and the plastic deformation in front of the crack tip. Many specimens were made, but only those whose ligament lengths were between 10.9 and 11.1 mm, and with similar plastic deformation in front of the notch root as observed by the optical microscope were tested. At least 3 specimens have been tested.

In Fig. 14 are represented the average stress-displacement curves corresponding to the six different types of notches with all curves shifted along the axis of displacement to show how, once the initiation point of propagation is reached, all curves have the same crack propagation.

All curves have equal values of σ_{max}. The visual assessment of σ_i also provides equal values. Then, we can consider the onset of crack initiation for these curves as being similar to the sets 1 and 2 of specimens. All curves overlap from $\sigma = 0$ to σ_{max} except for the case of the curve designated as S where the specimens were only pre-notched. In these curves, differences arise when we look the distance between displacements corresponding to σ_{max} and σ_i values. The larger distance between displacements provided larger values of CTOD and w_e. These differences increase with the radius of curvature and/or with the extent of the plastic deformation in front of the notch tip.

Imagine a set of specimens where there are various types of notches which had different crack tip radius and/or extent of plastic deformation, then the load-displacement and the stress-displacement plots will show a loss of self-similarity and/or crossing curves.

The smallest notch tip radius of curvature,1 μm, can be obtained with both femtolaser ablation [9-11] and razor blade sliding [6,9-11] sharpening techniques. The femtolaser ablation generates equal repetitive notches without plastic deformation in front of the notch tip [9-11].

In the EPBC, the EPR particles act as stress concentrators, and so the compressive component of the sliding force easily produces plastic deformation in front of the notch tip. There are always specimens with more or less different amount of plastic deformation which can be separated by observation in the optical microscope, but giving higher w_e values that the femtolaser set of specimens.

In single phase polymers, as amorphous PET [9] and PETG [11], has been possible to obtain specimens with and without plastic deformation at the notch tip when razor blade sliding was applied. Selecting razor blade specimens without plastic deformation at the notch tip lead to the same w_e value that the femtolaser sharpened specimens.

For ductile polymer films, the best and the most repetitive way to obtain notches with the highest quality is the femtolaser ablation technique.

4.4 Non-collinear notches

It has been tested 3 specimens with the notches sharpened by razor blade sliding, but in these specimens the notches were not collinear, they had a 1 mm separation.

In Fig. 18 is represented the average stress-displacement curve DR of 3 the specimens tested having non-collinear notches. The arrows placed in the frame represent the initiation points of crack propagation which also correspond to the small circles on the stress-displacement curve.

In Fig. 19 are represented the mean stress-displacement curve DR for the non-collinear notches and the mean stress-displacement curve SG which accounts for collinear notches obtained with the same notch sharpening procedure as the non-collinear notches. These curves show equal σ_{max} and σ_i values for both the collinear and non-collinear specimens, but, in this last one, the curve is slightly shifted to the right which implies larger d_i and thus higher w_e values than the curve DR.

4.5 Tilted specimens

The last set of specimens was notched by sliding a razor blade across the pre-notch tip as in set 2. The specimens had a ligament length of 11 mm, but the tests were performed with the specimens tilted 5 degrees on the grips.

Fig. 20 shows the stress-displacement curve and some registered frames of the tilted specimens. The arrows on these frames stand out the initiation points of notch propagation and are directly related to circles traced on the average stress-displacement curve.

It is noticed in this figure that the notches do not begin its propagation at the same time, that is, the notches do not propagate simultaneously. The non-simultaneous crack propagation was also observed [9] in non-tilted specimens and was attributed to differences in the quality of the two notches in the same specimen.

In Fig. 19 the average stress-displacement curve AT is represented to be compared with the stress-displacement curves SG and DR corresponding to collinear non-tilted and non-collinear non-tilted sets of specimens, respectively.

The tilted specimens and the non-collinear specimens have the stress-displacement curve slightly shifted to the right when both are compared to the SG specimens, but the shape of the curve in the propagation zone is different.

5. Conclusions

The specimens sharpened by femtolaser contain sharpened notches with a radius of curvature of 1 μm and negligible plastic deformation in front of the crack tip. All these specimens have equal repetitive notches that result in equal values for the displacement d_i at the onset of crack initiation. The d_i value coincides with the crack tip opening displacement $CTOD_c$ at crack initiation. All this leads to self-similar load-displacement and stress-displacement curves where the heads (from $d = 0$ to d_i) overlap. Integration of the stress-displacement curve between $d = 0$ and d_i gives the specific essential work of fracture w_e.

The razor blade sharpened specimens generated the same radius of curvature that the femtolaser sharpened specimens, but, in the first case, the specimens had some plastic deformation in front of the notch root. When testing selected razor blade sharpened specimens, with the same level of deformation in front of the crack tip, it has been obtained stress-displacement curves which follow the same trends than that ~~the~~

obtained with the femtolaser sharpened specimens. The main difference is the larger d_i value (equal to $CTOD_c$) for the razor blade sharpened specimens. It results in a higher w_e value for the razor blade ~~that~~ as compared to the femtolaser sharpened specimens.

When there are represented all normalized curves of the tails of the stress-displacement plots (values comprised between d_i and d_r) overlap, independently of both the original ligament length and the sharpening method, indicating the same propagation behaviour and equal βw_p values.

For specimens with different notches but equal ligament lengths, the shape of the stress-displacement curves is different although the crack initiation stress σ_i values are the same. The shape of the curves from the beginning ($d = 0$) until reaching σ_{max} is identical, and the σ_{max} values are equal, as well as the displacement value corresponding to σ_{max}. The crack initiation stress σ_i values are also independent of the notch, but it is not the case for the corresponding displacements d_i which increase with the radius of curvature at the notch root and with the plastic deformation in front of the notch tip. The area under the stress-displacement curve from $d = 0$ until d_i is greater when d_i is larger and, consequently, resulting higher w_e values. The range between the displacements, which correspond to σ_{max} and σ_i, increase when the radius of curvature of the notch root and/or the plastic deformation in front of the notch tip are larger. The tails of the stress-displacement curves lying between the displacement value at the onset of crack initiation d_i and the displacement at rupture d_r overlap, indicating that the propagation follows the same behaviour, i.e., it is independent of the notch, which results in equal values of the slope of the regression line βw_p in the EWF plots for sets of specimens with different notches. In sets of specimens containing notches of different quality, the load-displacement curves will not be self-similar and can cross each other. The stress-displacement curves will have different d_i's and do not overlap, between $d = 0$ and the different d_i's.

One millimeter non-collinear notches do not modify the σ_{max} and σ_i values obtained with the collinear specimens. The shape of the stress-displacement curve changes slightly, and the small increase in d_i gives a slight increase in the w_e value, too.

The specimen manufacture has crucial importance and plays a very important role on the accuracy, repeatability, and reproducibility of the results.

The incorrect alignment of the specimens on the grips (when the specimens are tilted) causes changes in the shape of the stress-displacement curve and the non-simultaneous initiation (at the same displacement and time) of the two notches of the same specimen.

The classical way to sharpen specimens by sliding a razor blade across the pre-notch can yield various types of notches that may depend on the operator and the ductility of the polymer, and then specimens with notches of different quality can be produced.

The femtolaser ablation technique allows obtaining equal repetitive notches with a radius of curvature at the notch root of 1 μm and without plastic deformation in front of the notch tip, and therefore it is a very suitable technique for the notch sharpening of ductile polymer films.

It is confirmed that a key requirement to obtain meaningful results is to have repetitive notches without plastic deformation in front of the notch root.

It is also confirmed that the specimens should have collinear notches, and that the correct alignment (without tilting) of the specimen on the grips of the testing machine is likewise required.

The EWF has been successfully applied on a rubber-toughened semicrystalline polymer (EPBC). This multiphase polymer has multiple shear yielding as a deformation mechanism which is different of polymers that undergo necking before the onset of crack propagation and where there is a consensus on the validity of the EWF test.

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FIGURE CAPTIONS

Figure 1. DENT specimen geometry.

Figure 2. Load-displacement curves for the femtolaser sharpened specimens. Specimen set 1.

Figure 3. EWF plots for the (▲) femtolaser and (●) razor blade sharpened specimens.

Figure 4. Summary of the femtolaser sharpened specimens (l_o = 11 mm). Specimen set 8.

Figure 5. Summary of the razor blade sharpened specimens (l_o = 11 mm). Specimen set 5.

Figure 6. Stress-displacement curves for the femtolaser sharpened specimens. Specimen set 1.

Figure 7. Stress-displacement curves for the razor blade sharpened specimens. Specimen set 2.

Figure 8. $CTOD_c$ determination for (▲) femtolaser and (●) razor blade sharpened specimens.

Figure 9. Stress-displacement curve for femtolaser and razor blade sharpened specimens (l_o = 17 mm).

Figure 10. Stress-displacement curves for the femtolaser and razor blade sharpened specimens shifted along the axis of displacement (l_o = 17 mm).

Figure 11. Normalized tails of the stress-displacement curves for femtolaser sharpened specimens. Specimen set 1.

Figure 12. Normalized tails of the stress-displacement curves for razor blade sharpened specimens. Specimen set 2.

Figure 13. Summary of pre-notched specimens (l_o = 11 mm). Specimen set 3.

Figure 14. Stress-displacement curves of specimens from set 3 until set 8 (l_o = 11 mm).

Figure 15. Summary of the deformed pre-notched specimens (l_o = 11 mm). Specimen set 4.

Figure 16. Summary of the liquid N_2 razor blade sharpened specimens (l_o = 11 mm). Specimen set 6.

Figure 17. Summary of the specimens pre-notched with a scalpel and sharpened by a razor blade (l_o = 11 mm). Specimen set 7.

Figure 18. Stress-displacement curve for specimens with non-collinear notches (l_o = 11 mm). Specimen set 9.

Figure 19. Stress-displacement curves for well-aligned collinear specimens (SG), well-aligned non-collinear specimens (DR), and collinear tilted specimens (AT).

Figure 20. Stress-displacement curve for tilted specimens (l_o = 11 mm). Specimen set 10.