NOTCH EFFECT ON THE FRACTURE OF A POLYMERIC FILM

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ABSTRACT

The fracture behavior of a bio-based thermoplastic copolyester film has been studied by combining the essential work of fracture, the J-integral, and the crack tip opening displacement characterization methods on double edge notched tension specimens, in an attempt to provide a better understanding of the details that play an important role in the repeatability and the reproducibility of the essential work of fracture test, with particular attention to the effect of the quality of the notches generated by two different notch sharpening techniques. Specifically, the femtosecond pulsed laser ablation and the classic razor blade sliding techniques have been applied. The equivalence, reported and discussed in the literature, between the specific essential work of fracture and the J value at crack initiation, as well as their relationship with the crack tip opening displacement, have been confirmed. It is also identified the parabolic shape of the stress-displacement curves, which modeled relates the specific non-essential work of fracture with the stress at initiation and the extension ratio during the crack growth.

Keywords: Essential work of fracture, J-integral, crack tip opening displacement, femtolaser ablation, notch mechanics, bio-based copolyester

1. Introduction

Films of polymers are used in a wide variety of applications. Their toughness is often a basic requirement to meet some applications.

The Linear Elastic Fracture Mechanics (LEFM) approach is used to study fractures occurring at nominal stresses well below the material's yield stress. The main assumption of LEFM is that the dissipated energy is confined to a small area near the crack tip (small scale yielding), and that brittle fracture occurs without extensive plastic deformation. LEFM characterizes toughness using the critical stress intensity factor (K_{Ic}), and the energy released per unit area of crack growth (G_{Ic}), at fracture initiation. There is a direct relationship between both parameters.

The LEFM approach is not applicable when the plasticity around the crack tip becomes too large; in those cases Elastic Plastic Fracture Mechanics (EPFM) applies and the crack tip opening displacement (CTOD) and J-integral are appropriate methods to characterize fracture.

Based on the deformation theory of plasticity, Cherepanov [1] and Rice [2] developed a new fracture parameter, the J-integral. This theoretical concept represents an energy contour path integral that is independent of the path around the crack, parameter which characterizes fracture under elastic-plastic and fully plastic conditions. Begley and Landes [3] applied the J-integral principle and developed a measurement of the fracture toughness which represents the energy required to initiate crack growth, which can be expressed as

$$J = -\frac{1}{t}\left(\frac{dU}{da}\right)_{d=const} \qquad \text{Eq. (1)}$$

where U is the external work done up to a given constant displacement, d, and a is the crack length.

Another important concept, first proposed by Wells in 1963 [4], is the CTOD. This method is used to determine a fracture parameter for ductile materials. It essentially measures the resistance of the material to the propagation of a crack. A direct relationship between the J-integral and CTOD concepts exists.

When the crack propagates through a highly deformed and yielded material, then Post-Yield Fracture Mechanics (PYFM) can also be applied and the Essential Work of Fracture (EWF) is a suitable method for characterizing fracture. For ductile polymers where crack propagation occurs through a fully yielded ligament, the EWF, the CTOD, and the J-integral methods are commonly used. The EWF method is becoming more widely used to characterize the plane stress toughness of ductile polymer films in mode I, primarily using the double edge notched tension (DENT) configuration. The widespread use of the EWF technique is due to the apparent ease with which DENT specimens may be prepared and tested.

It has been experimentally found that w_e, the specific energy just up to crack initiation, is equivalent to the J-integral [5-8] value at crack initiation in plane stress, J_o, when the complete ligament yielding of the sample precedes the onset of crack initiation. The specific essential work of fracture, w_e, becomes an inherent material parameter only if the ligament fully yields before the crack initiation. However, this condition is rarely met, in most publications it is not satisfied, and w_e is an apparent toughness that is only valid for comparison purposes.

There is a European Structural Integrity Society (ESIS) test protocol [9] for the EWF method, and several round-robin exercises [9,10] 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 still is, a topic of much debate, and these concerns indicate that EWF procedure is not yet sufficiently defined to be considered as a standard.

The DENT geometry is the most appropriate for mode I testing because the transverse stresses between the notches are tensile and therefore buckling problems are avoided [11].

Some of the aspects of the test and specimen validity are related to the specimen manufacture, particularly the notch quality [5,6,12], and they are studied here in more depth. Two sets of DENT specimens were prepared. In one set the notches were sharpened using the femtosecond pulsed laser ablation technique (femtolaser), and the second set was sharpened using the classical razor blade sliding technique. Both sharpening techniques are able to generate notches with different quality.

A combination of several methods for fracture characterization is used, in an attempt to provide a better understanding of the EWF approach for characterizing the fracture toughness of thin ductile polymer films.

All of these methods are utilized to characterize the fracture behavior of a bio-based copolyester film.

2. The EWF approach

2.1. The EWF concept

The EWF approach was firstly proposed by Cotterell and Reddell [13] after Broberg's work on stable crack growth was published [14-16]. It is based on the hypothesis that the total energy involved in the ductile fracture of a pre-cracked specimen (W_f) can be separated into two terms.

$$W_f = W_e + W_p \qquad \text{Eq. (2)}$$

where W_e, the essential work of fracture, represents the energy required for the creation of two new surfaces during the crack propagation, whereas the second term, W_p, is called the plastic work or the non-essential work of fracture and collects all other sources of energy produced throughout the fracture process (plastic deformation around the crack, heat dissipation, etc.). The term W_e is considered to be proportional to the area of the Inner Process Zone (IPZ) while W_p is proportional to the volume of the Outer Plastic Zone (OPZ). These zones are schematized in Figure 1 for a DENT specimen.

Rewriting Eq. (2) using specific terms, i.e., dividing all terms by the area of the ligament cross-section, the following expression can be obtained:

$$w_f = \frac{W_f}{l_o.t} = \frac{W_e}{l_o.t} + \frac{W_p}{l_o.t} = w_e + \beta w_p.l_o \qquad \text{Eq. (3)}$$

where t is the specimen thickness, l_o is the original ligament length and β is a factor that depends on the shape of the OPZ.

It is possible to assess Eq. (3) by performing a series of tests on specimens with different ligament lengths and plotting the specific total work of fracture values, w_f, as a function of their ligament lengths. A simple regression analysis of this plot shows that the specific essential work of fracture, w_e, and βw_p, are the intercept for a zero ligament length and the slope of the linear regression line, respectively. The specific non-essential work of fracture is $\beta w_p.l_o$.

The EWF technique was originally developed for metals [17] and later extended to polymers by Mai [18]. A more detailed description of the procedure can be found elsewhere [19].

The value which represents the toughness, namely, w_e, is an inherent material parameter only if the ligament fully yields before the onset of crack propagation and is independent of the specimen geometry, as verified first by Mai and Cotterell [20].

2.2. Key requirements

In the EWF analysis, the following three basic 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 that w_e is an inherent material parameter. However, this key assumption is rarely realized, even 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 to the onset of crack propagation if it is less than twice the size of the plastic zone radius, r_p, under plane stress conditions [21]:

for a linear plastic zone

$$2r_p = \frac{\pi}{8}\left(\frac{Ew_e}{{\sigma_y}^2}\right) \qquad \text{Eq. (4)}$$

and for a circular plastic zone

$$2r_p = \frac{Ew_e}{\pi{\sigma_y}^2} \qquad \text{Eq. (5)}$$

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

Although having a ligament length that is 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 [6,22].

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 [5,6,19].

b) Fracture occurs under plane stress conditions.

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

Hill [23,24] has demonstrated 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 to 2.97 σ_y in a pure plane strain state, where σ_y is the uniaxial tensile yield stress. Thus, the maximum stress registered during the DENT test, σ_{max}, has to be maintained between σ_y and 1.15 σ_y, and its value is equal for all the ligaments, when there are pure plane stress conditions.

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

Clutton [9] suggested another criterion, which does not use the yield stress but uses the mean of the maximum stresses (σ_{max}) as follows

$$0.9\sigma_{max} < \bar{\sigma}_{max} < 1.1\sigma_{max} \qquad \text{Eq. (6)}$$

The Clutton criterion only removes data where an error in dimensional measurement or load exists, although these load errors can be undetected if the same load cell is used for all tests [10,25].

The polymer films have a thickness of less than 1 mm. A practical lower limit of 5 mm [10] in the ligament length has been accepted when preparing DENT specimens, which facilitates handling of specimens. The upper limit in the ligament requires full-ligament yielding before crack propagation. Hence, l_o has to be less than twice the plastic zone radius in DENT specimens.

Another upper limit is given by the relationship:

$$l_o \leq \frac{W}{3} \qquad \text{Eq. (7)}$$

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

c) Good quality notches.

Good quality entails identical and consistently sharp notches without plastic deformation in front of the notch tip. This requirement guarantees self-similar load-displacement and ligament length-displacement curves for the tested specimens [5,6].

The notch sharpening is of critical importance in obtaining good results. There is evidence [10] that the larger notch tip radius, the higher the w_e values. Thus, the notches should be sharpened as much as possible, so that an even sharper tip radius will not affect significantly the value of w_e. However, the smallest notch tip radius that was reached was about 1 µm.

It has also been demonstrated [12,26,27] that the larger plastic deformation in front of the notch tip, the higher the w_e values.

Several methods can be found in the literature that are used for sharpening notches in polymer films. Further, it appears that the notch quality is dependent on the technique used, and even on the skill of the operator.

The femtosecond pulsed laser ablation technique (femtolaser) is a non-contact method useful for sharpening the pre-notches. This powerful technique can be used to micromachine practically any material, with negligible thermal damage to the surface surrounding the ablated areas [28,29]. With proper set-up, the femtolaser ablation technique produces consistent notches that have negligible thermal damage, no plastic deformation, and a notch tip radius of approximately 1 µm [5,6,12]. A limitation of the femtolaser technique is the high cost per notch.

The most popular method 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 this in a single pass so that the notch follows the same track avoiding bifurcations. Very small notch tip radius of around 1 µm can be manufactured.

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 sideways sliding force. The higher the yield stress of the ductile polymer, the smaller the extent of the plastic deformation ahead of the notch tip.

Cooling specimens below their glass transition temperature (T_g) prior to the application of the razor blade sliding technique can be helpful.

The razor blade sliding is a manual procedure, which regularly results in differently sharpened notches. These differences can be in the notch tip radius and/or the extent of the plastic deformation [9].

In a set of DENT specimens, it is possible to have three different specimen populations that show distinct behaviors from one another, which are responsible for the non-similarity and crossing of the 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 both of which have noticeable plastic deformation [6].

When the specimen has two different notches, crack initiation does not begin at the same time in both notches. Conversely, when the two notches are equal, the crack initiation happens at the same time (simultaneously). When comparing specimens with and without plastic deformation at the notch root [5,6] but with the same notch tip radius, it is noticed that the heads of the stress-displacement curves have different shapes. Specimens without plastic deformation yield a lower value for CTOD, that is, a lower w_e value. However, the tails of theses 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 for generating notches have been tried unsuccessfully, which include cuts made by scissors or die-punch and scalpel cuts generated starting from the notch tip. These techniques generate greater plastic deformation ahead of the notch tip and result in higher w_e values than does the razor blade sliding method [9] or scalpel sliding method [27].

3. Experimental details

3.1. Material

This study has been conducted on an amorphous thermoplastic copolyester, ECOZEN (SKYGREEN chemicals, Korea), produced by the polymerization of terephthalic acid, ethylene glycol, 1.4-cyclohexanedimethanol, and a bio-based monomer that is derived from renewable resources [30].

The material was kindly supplied by Nudec S.A (Spain) in the form of films with a thickness of 0.4 mm. The copolyester is slightly hygroscopic and the film had the water absorption at saturation.

Differential scanning calorimetry measurements (DSC) were conducted on a Q2000 equipment from TA instruments. The samples were heated at a scanning rate of 10 ºC/min. The DSC test revealed a T_g at 101 ºC at midpoint.

3.2. Specimen manufacture

Two kinds of specimens were prepared: dumbbell shaped and DENT specimens to perform uniaxial tensile testing and plane stress fracture toughness (EPFM and PYFM) testing, respectively. All specimens were prepared with the longitudinal axis in the machine direction of the cast-extruded film.

3.2.1. Dumbbell shaped specimens

Eleven dumbbell shaped specimens were prepared from the 0.4 mm thick films using a cutting press, with the shape and dimensions of a Type V specimen in accordance with the ISO standard 527-3.

3.2.2. DENT specimens

The DENT geometry is shown in Figure 1, where *W*, *t*, *L*, and *Z* correspond to the specimen width, specimen thickness, specimen length, and the distance between clamps, respectively. Most published research has been performed using this geometry. There is experimental evidence that when *W* and *Z* are more than twice the largest ligament value, the w_e values do not change [10,31,32]. However, when *Z* is more than twice *W*, the specimens can become wavy in their own plane during the test [31] and thus modify the stress distribution in the ligament zone. Values of *Z* that are smaller than *W* seem to be far from the infinite plate case, where the result is probably influenced by the close proximity of the cross-heads to the fracture region.

Taking these pieces of evidence into consideration, the DENT specimens were obtained by cutting rectangular coupons from the 0.4 mm thick films with the dimensions shown in Figure 1. In order to minimize the plastic deformation at the notch root, a sandwich was formed by compressing a copolyester coupon between two layers of 1-mm-thick polymethyl methacrylate coupon, and was subsequently pre-notched with a saw. In this way, pre-notched specimens with different original ligament lengths (l_o) were obtained.

The notches must be collinear and placed opposite of one another at the midpoint of *L* [12].

The notch sharpening of the DENT specimens was carried out using two different methods, the non-contact femtosecond pulsed laser ablation technique and the manual razor blade sliding technique. This resulted in two sets of DENT specimens, femtolaser and razor blade, on which to perform the plane stress fracture toughness testing.

In one set of 10 specimens, the machined pre-notch was extended by the application of the femtolaser with the same set-up and other variables, as previously defined for polyethylene terephthalate (PET) [5] and polyethylene terephthalate glycol-modified (PETG) [6].

A second set of 22 specimens was sharpened manually by sliding a new razor blade across the pre-notch in a single pass, as the accepted method to sharpen notches in polymer films.

Prior to testing, one surface of each DENT and dumbbell shaped specimens was adequately spray painted in order to achieve a speckle pattern. The original ligament length of each specimen was measured after testing in an optical microscope.

3.3. Test details

All tests were carried out at 23 ± 1°C. The DENT and dumbbell shaped specimens were tested on a Zwick servo-hydraulic testing machine, which was fitted with a two-camera digital image correlation (DIC) system and Aramis software (GOM, Germany).

In general, DIC is based on the principle of comparing speckle patterns on the surface of the deformed and the undeformed samples, or between any two deformation states. Surfaces with high contrast are required to avoid image distortion and therefore inaccurate data. Hence, before testing, one surface of each specimen was covered with a thin white coating before being sprayed in order to obtain black points, as required by the DIC system.

3.3.1. Uniaxial tensile testing

Uniaxial tensile tests were performed on the dumbbell shaped specimens until complete failure at a cross-head speed of 1 mm/min, in accordance with the guidelines provided by the ISO standard 527. The load was registered by the load cell, and the deformation and displacements were measured by the DIC system.

3.3.2. DENT tests

The DENT specimens were tested in mode I until complete failure at a cross-head speed of 1 mm/min. The DENT specimens were tested with the tensile direction perpendicular to the collinear notches. The specimens were properly mounted on the grips with an initial distance between clamps of Z = 60 mm (Figure 1).

DIC system images obtained during the tests were recorded for all the specimens. These images were also analyzed with the Aramis software to measure the displacement and the ligament length evolution throughout the test. There is experimental evidence [24] that w_e is insensitive to changes in the gauge length chosen for displacement measurements. Nonetheless, the slope βw_p of the EWF plot and the rupture displacement (d_r) increase slightly with increasing gauge length [26]. If the viscoelastic energy and displacement outside the plastic zone are recovered upon unloading, then d_r and W_f would be expected to be equal. The elastic contribution is always recovered, but if the recovery of other displacements or energies (as the viscous contribution) does not happen before specimen rupture, then βw_p and d_r will increase with increasing gauge length. The displacement d was measured with two reference points very near to each other, but outside the largest OPZ, which corresponds to the largest original ligament length specimen. The distance between these two reference points was always the same for all specimens of a given set.

Then, by using the registered loads, displacements, and ligament lengths, two sets of data can be plotted for each specimen. Specifically, the stress is plotted as a function of the displacement, and the ligament length as a function of the displacement.

4. Results and discussion

The DENT test generates two different kinds of plots. Specifically, the stress-displacement and the ligament length-displacement plots for each tested specimen have been obtained. The same results can be used to perform EWF, CTOD, and J-integral analyses.

4.1. Deformation behavior

σ_y, E, and ν of the ECOZEN material were measured from the dumbbell shaped specimens.

The tensile stress-strain curves, represented in Figure 2, show an extensive amount of plastic deformation. A yield point (maximum load) can be seen where necking starts, followed by a sudden load drop where the neck develops. Afterwards, there is a drawing of the necked region (cold drawing), during which the tensile deformation continues at a constant stress (engineering flow stress, σ_{fs}), followed by a stress drop when the specimen ruptures.

The results obtained from the test of 11 specimens were: E = 1.91 ± 0.02 GPa, σ_y = 44.48 ± 0.42 MPa, σ_{fs} = 33.77 ± 0.44 MPa, and ν = 0.44 ± 0.01.

4.2. DENT tests

4.2.1. Femtolaser sharpened DENT specimens

Four notches in two specimens were observed by scanning electron microscopy (SEM). The SEM samples had to be cut; hence, they could not be tested. All of the femtolaser sharpened notches observed through the SEM turned out to be virtually identical, as shown in Figure 3, with a notch tip radius of less than 1 µm and a sharpening depth of 1 mm. It was also observed that there was no plastic deformation and negligible thermal damage ahead of the notch tip.

The load cell of a servo-hydraulic testing machine was used to measure the applied load, while the displacements were simultaneously measured by the DIC system. The registered load can be divided by the area of the cross-section of the ligament length to give stress. As such, the stress-displacement curves are represented in Figure 4, for each one of the 8 tested specimens.

The images collected from each specimen by the DIC system in the DENT tests were analyzed to measure the ligament lengths, l, and their associated displacements. These data are represented in Figure 5.

w_f can be calculated by integrating each curve represented in Figure 4. In Figure 6, the w_f values are represented as a function of their corresponding l_o. A regression analysis of this plot shows that w_e = 27.85 KJ/m^2 and βw_p = 5.91 MJ/m^3 with a coefficient of determination R^2 = 0.968.

The hatched area under the stress-displacement curves in Figure 4 is equal to w_e = 27.85 KJ/m^2. In this figure, a set of overlapping curves (heads) can be observed in the low displacement range up to a fairly well recognizable displacement value, d_i, from which the curves (tails) start to diverge. The hatched area begins at d = 0 and continues until the displacement d_i, where the crack initiation begins. When the displacements are larger than d_i there is crack propagation and the curves diverge from each other. From the analysis of Figure 4, the crack initiation displacement d_i = 0.80 ± 0.01 mm and the crack initiation stress σ_i = 34.82 ± 1.18 MPa are identified. The hatched area represents the specific energy up to crack initiation, and then w_e corresponds to an initiation specific energy. In other words, it represents the energy per surface unit area required to create two new surfaces in a cracked body. An identical behavior was previously found in other polymer films, including PET [5], PETG [6], and ethylene-propylene block copolymer (EPBC) [8]. Curve overlapping occurs in the uniaxial tensile tests (Figure 2) if the distance between the displacement reference points is constant for all the specimens and yielding occurs between the above mentioned reference points. In the DENT tests, the displacements are measured using as a reference two points located close to the initial sharpened notch, and such that the OPZ zone is contained between the two points. The distance between these reference points is taken to be constant for all specimens. In this way, the elastic and viscoelastic contributions due to the bulk polymer located outside the fracture process are negligible. It is important to remark that the heads of the registered stress-displacement curves (Figure 4) overlap and closely resemble those of the uniaxial tensile tests displayed in Figure 2, including the decrease in stress, characteristic of necking.

The average of the maximum stresses, σ_{max} = 42.48 ± 0.46 MPa is practically equal to σ_y. The Hill and Clutton criteria are satisfied. The crack initiation stress σ_i = 34.82 ± 1.18 MPa is almost equal to σ_{fs}.

The sequence of events leading to the fracture of the DENT specimens, as observed in the frames shown in Figure 4, encompasses the opening and blunting of the notch tip with the yielding and necking of the ligament area (heads), followed by crack initiation and propagation until complete fracture (tails).

Prior to the onset of crack initiation, there is an increment of the crack length Δa_b due to blunting (Figure 1), where l_i is the remaining portion of the ligament length yet to be fractured. Thus

$$l_i = l_o - \Delta a_b \qquad \text{Eq. (8)}$$

When representing l_i as a function of l_o a linear dependence arises. The intercept is located at $-\Delta a_b$ and the slope is equal to 1. The l_i values taken from Figure 5 and their corresponding l_o, are plotted in Figure 7. The regression line in Figure 7 provides a slope of 1, as predicted, and Δa_b= 1.40 mm.

Even following a careful observation of the frames stored by the DIC system, we were not able to distinguish the exact frame in which blunting finishes and crack initiation begins. The recorded frames shown in Figure 4 correspond to the tested specimen with the largest original ligament length (l_o = 14.66 mm). The second frame, which corresponds to d_i, clearly shows the full-ligament yielding and necking before the onset of the crack initiation. The plastic zone is wedge-shaped and narrow, which is consistent with a linear plastic zone. These observations were similar for all specimens.

Applying Eq. (4) results in $2r_p$ = 10.64 mm, which is the theoretical maximum ligament length, but specimens with larger values of l_o have been used in this work. Although Eq. (4) is a reasonable size criterion, it appears to be too restrictive considering the visual evidence observed in the second frame of Figure 4, and as indicated by the ligament necking and the stress drop, which happen before d_i is reached. Similar behaviors have been noted in other thermoplastic copolyesters [6,9,19,22]. Unfortunately, the strain fields at the point of crack initiation cannot be studied. The large deformations developed in the blunted crack-tip region lead to such distortion of the speckle pattern that they cannot be analyzed by the Aramis software.

The key assumptions of the EWF analysis were thoroughly satisfied, including very sharp notches without plastic deformation, specimens in a plane state of stress, and ligaments completely yielded before the onset of crack initiation. Thus, w_e represents and inherent material property.

Hashemi and O'Brien [33] applied to DENT specimens a method for obtaining the critical crack tip opening displacement $CTOD_c$ (CTOD value at the onset of crack propagation). Under plane stress conditions the following linear relationship exists

$$d_r = CTOD_c + \alpha . l_o \qquad \text{Eq. (9)}$$

where d_r is the displacement at rupture and α is the extension or displacement ratio from crack initiation until rupture.

In Figure 8, d_r is plotted as a function of l_o. A simple linear regression analysis of this plot shows that the $CTOD_c$ value is the intercept at zero ligament length, 0.85 mm. This $CTOD_c$ value, as can be expected, agrees very well with the d_i value previously found. Using the Δa_b and $CTOD_c$ values makes it possible to normalize the ligament length-displacement curves shown in Figure 5 for the purpose of comparing the crack propagation of the DENT specimens. The normalized curves are represented in Figure 9. A complete overlap of the curves for all specimens can be observed, indicating the same propagation behavior, which is independent of l_o.

The crack propagation can also be analyzed. When the ligament length is completely yielded and necked, the following relationship applies:

$$P = t\,(W - a) \cdot \sigma_i \qquad \text{Eq. (10)}$$

where P is the load, and a the crack length. The derivative yields

$$\frac{dP}{da} = -t\,\sigma_i \qquad \text{Eq. (11)}$$

Thus, a linear dependence of the load on a is predicted for the propagation region (tails).

According to Figure 1, a can be represented by

$$a = W - l \qquad \text{Eq. (12)}$$

Thus, after combining Figures 4 and 5 with Eq. (12), the load is represented as a function of a for the tails in Figure 10. Here, a linear dependence can be observed except at higher a-values, close to the complete fracture where the slope and hence σ_i increases, probably as a consequence of orientation hardening. From the slope, it is found that σ_i = 34.37 MPa, coinciding with the previously found value of σ_i = 34.82 ± 1.18 MPa from Figure 4.

There are several approaches to evaluating the J-integral, but for the sake of simplicity, the Begley and Landes experimental method [3] is used here because it can be adequately applied to the previous DENT test results.

This experimental method requires two steps. The first step consists of the graphical representation of the energy divided by the specimen thickness as a function of the crack length when the displacement is fixed. This method consists of taking a constant displacement value d, in Figure 5, where only one l-value is found for each specimen. Then, using Eq. (12), each l value is transformed into a crack length value. The energy is found through numerical integration of the load over the constant displacement value *d* in Figure 4. This same procedure is applied to a new set of constant displacements adequately chosen. This graphical representation is shown in Figure 11. The points belonging to the same displacement show linearity and can be modeled by a straight line. Following Eq. (1), the slope of the regression lines are –J.

The second step consists of the representation of the J values as a function of the displacement as shown in Figure 12. Following Hodgkinson and Williams [34], the total energy U for a non-work hardening full yielding of the ligament is

$$U = P \cdot d \qquad \text{Eq. (13)}$$

which, combined with Eq. (10), results in

$$U = t\,(W - a)\sigma_i \cdot d \qquad \text{Eq. (14)}$$

That is, *U/t* is a linear function of the crack length at a constant fixed displacement, and this is verified in Figure 11.

Equation 1 merged with Eq. (14) gives

$$J = -\frac{1}{t}\left(\frac{dU}{da}\right)_{d=const} = \sigma_i \cdot d \qquad \text{Eq. (15)}$$

From the slopes of the regression lines in Figure 11 and the slope in Figure 12, Eq. (15) is verified. From the slope in Figure 12, it is found that σ_i = 38.97 MPa, a value that matches reasonably well with the σ_i values obtained before.

The J value at crack initiation J_o is obtained by introducing the $CTOD_c$ value into the J plot, as shown in Figure 12. The resulting value J_o = 28.00 KJ/m^2 coincides with w_e = 27.85 kJ/m^2. This result confirms the equivalence between w_e and J_o when the ligament length is fully yielded prior to the onset of crack initiation.

The Shih analysis [35] shows that there is a unique relationship between J_o and CTOD$_c$. For a non-hardening material in plane stress and assuming that the stress in the plastic zone is σ_i, then

$$J_o = \sigma_i \cdot CTOD_c \qquad \text{Eq. (16)}$$

In the present study this relationship is completely fulfilled. Moreover, our analysis shows a complete relation between w_e, J_o, and $CTOD_c$.

The specific energy as a function of *a* for the femtolaser sharpened specimens is represented in Figure 13. The curves are obtained as follows. For each *a*-value a corresponding *l*-value is obtained [Eq. (12)]. For each *l*-value the corresponding displacement *d* can be found through Figure 4. Finally, the specific energy is obtained by the numerical integration of the curves shown in Figure 4 up to the above mentioned displacement *d*. This procedure is repeated for increasing *a*-values, and for each specimen.

In Figure 13, it can be observed that though there is not a unique curve, the termination points indicate a linear dependence on l_o. At the termination points, l_o is equivalent to Δa at complete fracture. The regression line is the same that was found in the EWF plot (Figure 6). If the increment of crack length just prior to crack initiation Δa_b is introduced in Figure 13, a coincidence between the specific initiation energy and w_e is again observed.

The $w_e = J_o = 27.85$ kJ/m^2 value for the ECOZEN is slightly lower than the value reported [6] for PETG (32 kJ/m^2), as can be expected, due to its chemical structure as a modified PETG.

4.2.2. Razor blade sliding sharpened specimens

In the second set of DENT specimens, the pre-notch was extended by the popular contact method of sliding a fresh razor blade across the pre-notch tip in a single pass. Eight notches from 4 specimens were observed by SEM. Figure 14(a,b) shows the micrographs of the two notches from the same specimen. In Figure 14a, a volume accumulation of plastically deformed material ahead of the notch tip induced by the compressive component of the sideways sliding force can be observed, whereas this feature is absent in Figure 14b. All of the analyzed notches were very sharp with a notch tip radius less than 1 μm, but with different sharpening depths, ranging between 100 μm and 250 μm. The razor blade sliding sharpening method generates notches of different quality, which are strongly dependent on the operator's skill. Thus, 3 kinds of specimens can be produced. Specifically, specimens with both notches as those in Figure 14a, specimens with both notches as those in Figure 14b, and finally specimens with one notch as that in Figure 14a and the other one as that in Figure 14b.

The remaining 18 specimens were tested in the same way as the femtolaser sharpened specimens. The obtained EWF parameters were $w_e = 35.86$ KJ/m^2 and $\beta w_p = 5.94$ MJ/m^3, with $R^2 = 0.960$.

Careful observation of the frames collected by the DIC system showed that in 5 specimens the two edge notches did not propagate at the same time and therefore were discarded. It has been demonstrated that in the specimens with both notches of different quality, they do not propagate simultaneously. The registered stress-displacement and ligament length-displacement curves of the remaining 13 specimens are shown in Figures 15a and 16, respectively.

For the 13 specimens, the EWF parameters were (Figure 17a) $w_e = 27.73$ KJ/m^2 and $\beta w_p = 6.40$ MJ/m^3, with $R^2 = 0.972$.

The hatched area in Figure 15a corresponds to $w_e = 27.73$ KJ/m^2 and, proceeding as before with the femtolaser sharpened specimens, it is found that $\sigma_{max} = 44.44 \pm 1.23$ MPa, $\sigma_i = 38.33 \pm 3.42$ MPa and $d_i = 0.77 \pm 0.02$ mm.

In Figure 15a, two different populations of curves can be observed. In the first one (9 specimens), the heads overlap, and d_i occurs after the necking of the ligament length. In the second one (4 specimens), the heads do not completely overlap, and d_i occurs before the complete necking of the ligament length, resulting in a large standard deviation for σ_i.

These two sets of curves can be explained by differences between the two populations in the plastic deformation ahead of the notch tip. As has been recently demonstrated [12], it occurs between specimens with and without plastic deformation ahead of the notch tip. As has been shown [12], when specimens with the same l_o and notch tip radius but with different levels of plastic deformation at the notch root are tested, the stress-displacement curves are equal in the range between d = 0 and the displacement corresponding to σ_{max} ($d_{\sigma max}$), but the distance between d_i and $d_{\sigma max}$ increases with the plastic deformation at the notch root. However, the tails are equal.

Once the population of the specimens with excessive plastic deformation in front of the notch tip was separated, the stress-displacement curves for the 9 specimens with small or negligible plastic deformation are presented in Figure 15b, and the corresponding EWF plot is shown in Figure 17b. The parameters obtained with the EWF method were w_e = 29.48 KJ/m^2 and βw_p = 6.19 MJ/m^3, with R^2 = 0.942. The w_e value (29.48 kJ/m^2) is represented by the hatched area in Figure 15b. The stress-displacement curves (heads) overlap just in this hatched area until a d_i value of 0.83 ± 0.02 mm. After this point, the curves (tails) start to diverge from one another. The σ_{max} = 44.22 ± 0.94 MPa and σ_i = 35.69 ± 0.68 MPa values are also found by analyzing this plot. The l_o values of these 9 specimens were in a plane stress state, completely yielded, and necked before crack initiation. The Hill and Clutton criteria were also satisfied. Finally, the key requirements of the EWF approach were fulfilled. Thus, w_e is an inherent material property.

The w_e, σ_{max}, σ_i, and d_i values of these 9 specimens agree reasonably well with the corresponding values of the set of femtolaser sharpened specimens presented before, which had no plastic deformation ahead of the notch tip. The l_i-l_o and d_r-l_o plots are presented for the 9 valid razor blade sharpened specimens in Figures 18 and 19, respectively. A simple linear regression analysis of both plots leads to Δa_b = 1.42 mm and $CTOD_c$ = 0.85 mm. As can be expected, the $CTOD_c$ value matches the value obtained from the femtolaser sharpened specimens.

The ligament length-displacement curves of the razor blade sharpened specimens shown in Figure 16 can be normalized for the propagation region. The normalized curves are represented in Figure 9 together with the normalized curves of the femtolaser sharpened specimens. A complete overlap of all the normalized propagation curves is observed in Figure 9.

The load-crack length curves for these 9 specimens are represented in Figure 20. From the slope and applying Eq. (11), it can be found that σ_i = 35.18 MPa, which is nearly equal to the value previously found for the femtolaser sharpened specimens.

As in the case of the femtolaser sharpened specimens, the Begley and Landes method is used to evaluate the J-integral. The two steps required for the application of this method lead to Figures 21 and 22. In Figure 21, it can be observed that U/t is a linear function of the crack length at a constant fixed displacement. From the slope of the regression line in Figure 22, applying Eq. (15), it is found that σ_i = 38.98 MPa, which practically matches the value calculated for the femtolaser specimens.

J_o is obtained by introducing the $CTOD_c$ value in the J-integral plot. This provides a value of J_o = 29.64 KJ/m^2 equal to w_e and coincident with the value obtained for the femtolaser sharpened specimens. Equation 25 is also satisfied.

4.2.3. Femtolaser versus razor blade

All of the femtolaser sharpened notches had very consistent, sharp notches without plastic deformation in front of the notch tip and non-crossing tails in the stress-displacement curves (Figure 4). The ligament length-displacement curves also had self-similarity (Figure 5).

The razor blade sharpened specimens had very sharp notches, as sharp as the femtolaser ones, but the different compressive component of the sliding force that is applied by the operator generates three different levels of plastic deformation ahead of the notch tip. There are specimens with negligible plastic deformation ahead of the notch tip, similar to the femtolaser specimens, which give equal results to the femtolaser. In other specimens, notches of the same specimen, but with different levels of plastic deformation cause non-simultaneous crack propagation and, in consequence, these specimens must be discarded. In this case, there are crossing tails in the stress-displacement curves (Figure 15a). Finally, specimens where both notches have the same high level of plastic deformation ahead of the notch tip cause that the shapes of the stress-displacement curves to be different when compared to specimens with no plastic deformation. These specimens also must be discarded.

Figure 23a represents the stress-displacement curves for the femtolaser l_o = 14. 66 mm and the razor blade l_o = 14.17 mm specimens. The razor blade sharpened specimen was 1 of the 4 non-valid specimens. In Figure 23b, the femtolaser curve is shifted to the right. Analyzing Figure 23(a,b), it is found that both specimens have practically the same σ_{max} and σ_i values. Nevertheless, the $CTOD_c$ and d_r values of the razor blade specimen are larger than those of the femtolaser specimen, resulting in a higher w_e value for the razor blade sharpened specimen. The same trends have been observed in other polymer films [4,5,10] between specimens with different levels of plastic deformation ahead of the notch tip. The shapes of the stress-displacement curves are influenced by the plastic deformation surrounding the crack tip, but the tails remain equal. Thus, βw_p and the propagation behavior are independent of the notch quality.

In Table I, the mean values and the coefficients of variation for the main EWF parameters, that is, w_e, βw_p, and d_i are represented. The coefficients of variation for the femtolaser sharpened specimens are lower in comparison to those of the razor blade sharpened specimens for all the main EWF parameters, indicating a lower degree of variation in the case of femtolaser sharpened specimens.

The stress-displacement curves in Figures 4 and 15b, allow the partition into “head” (or initiation) and “tail” (or crack propagation), as described previously: The shape of the “tail” can be approximated to be a half of a parabola, described by σ_i in the y-direction and (d_r-d_i) in the x-direction. Thus, the energy absorbed during the crack propagation per ligament section (U_p) can be estimated as:

$$U_p \cong \left(\frac{\sigma_i \cdot (dr - di) \cdot 2}{3}\right) \qquad \text{Eq. (17)}$$

The value of d_i can be obtained from either the stress-displacement plots (Figures 4 and 15b) or from the slope of the d_r-l_o plots (Figures 8 and 19). In any case, combining with Eq. (9), it results in

$$U_p \cong \left(\frac{\sigma_i \cdot \alpha \cdot l_o \cdot 2}{3}\right) \quad \text{Eq. (18)}$$

As w_e remains constant during crack propagation, the propagation energy per ligament length is a constant value, independent of the notch sharpening method. Then, considering the essential term w_e as the initiation energy, the propagation energy per ligament section is $W_p/(l_o \cdot t)$, giving the equivalence:

$$U_p = \beta \cdot w_p \cdot l_o \quad \text{Eq. (19)}$$

Substituting into Eq. (18) gives [7]

$$\beta \cdot w_p \cong \left(\frac{\sigma_i \cdot \alpha \cdot 2}{3}\right) \quad \text{Eq. (20)}$$

The parabola equation is given by

$$d = d_r + s\sigma^2 \quad \text{Eq. (21)}$$

where

$$s = \frac{d_i - d_r}{\sigma_i^2} \quad \text{Eq. (22)}$$

In Figure 23, the modeled parabola (dashed line) for the femtolaser sharpened specimen with l_o = 14.66 mm is also represented. It is observed that the parabolic shape fits well with the experimental stress-displacement curve in the propagation range (tails).

The stress-displacement curves were previously roughly approximated to be a quarter of an ellipse [7] during the crack growth (tails), which results in

$$\beta \cdot w_p \cong \left(\frac{\sigma_i \cdot \alpha \cdot \pi}{4}\right) \quad \text{Eq. (23)}$$

The βw_p, σ_i, and α values obtained for this bio-based copolyester, as well as the values obtained for other polymer films, PET [5], PETG [6], and three different commercial grades of EPBC [8,12,36], are presented in Table II along with the obtained βw_p values using the semi-parabola [Eq. (20)] and the quarter of an ellipse [Eq. (23)].

It must be considered that the ellipse approach [Eq. (23)] overestimates the area of the tails, thus giving higher βw_p values that the obtained by the slope of the EWF plot. However, the parabola approach [Eq. (20)] fits well with the experimental βw_p values for the six polymer films, indicating a parabolic shape for the tails of the experimentally measured stress-displacement curves. Eq. (20) relates the plastic term βw_p to the crack initiation stress σ_i and the extension ratio α at rupture.

The volume of material deformed just up to crack initiation, V_e, is given by

$$V_e = l_o . \mathrm{t} . d_i \quad \text{Eq. (24)}$$

Then, taking into account Eq. (16), it can be found that

$$\frac{W_e}{V_e} = \sigma_i \tag{25}$$

The volume V_p of material deformed during propagation is comprised between two parabolas of length l_o and height $(d_r - d_i)/2$. Then, using Eq. (20), it can be found that

$$V_p = 4.\frac{2}{3}.\frac{l_o}{2}.\frac{d_r - d_i}{2}.\mathrm{t} = \frac{2}{3}\alpha.{l_o}^2.\mathrm{t} \tag{26}$$

and

$$\frac{W_p}{V_p} = \frac{\beta.w_p.{l_o}^2.t}{V_p} = \sigma_i \tag{27}$$

Such that,

$$\beta w_p = \frac{2}{3}.\sigma_i = \frac{2}{3}.\frac{W_p}{V_p} \tag{28}$$

where β is equal to 2/3 and w_p is equal to (W_p/V_p). Therefore, βw_p is the propagation energy density.

5. Conclusions

The femtosecond pulsed laser ablation technique generates highly consistent, sharp notches, without plastic deformation in the region surrounding the crack tip. This sharpening technique is the most appropriate to sharpen notches of the most ductile polymer films, giving the lowest values for the fracture parameters. Limitations of this technique include the availability of the equipment and the high cost per notch.

Notch sharpening of polymer films using the razor blade sliding technique can generate notches with different quality, which depends on the material ductility and the compressive component of the sideways sliding force that is applied. Three different types of specimens can be obtained. Specimens with sharp notches and without plastic deformation near to the crack tip can be obtained, which yield fracture parameters equal to the femtolaser sharpened specimens. Generally, however, specimens with plastic deformation around the crack tip are obtained. When the two notches in the specimen have different levels of plastic deformation, the crack propagation from both notches does not happen at the same time. Specimens with sharp notches, but equal levels of plastic deformation ahead of both notches can be also obtained. These specimens result in stress-displacement curves with different shapes and higher w_e values than those of specimens without plastic deformation in front of the notch tip.

In DENT specimens, the crack propagation does not depend on the plastic deformation surrounding the crack tip, and equal values for βw_p will be obtained.

In polymer films, the variability of the w_e, J_o, and $CTOD_c$ values obtained in the same laboratory and especially between laboratories can be explained by differences in the quality of the notches that can be generated by different operators. This is especially significant when the notch sharpening is performed using the razor blade sliding technique.

The equivalence between w_e and J_o, as well as their relationship with the $CTOD_c$ is again confirmed.

The stress-displacement curves in the propagation range (tails) have a parabolic shape, which modeled results in βw_p being directly related with the stress at initiation σ_i and the extension or displacement ratio α.

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FIGURE CAPTIONS

Figure 1. Double edge notched tension geometry.

Figure 2. Uniaxial tensile stress-strain curves.

Figure 3. SEM micrograph for a femtolaser DENT specimen.

Figure 4. Registered stress-displacement curves for the femtolaser DENT specimens.

Figure 5. Registered ligament length-displacement curves for the femtolaser DENT specimens.

Figure 6. EWF plot for the femtolaser specimens.

Figure 7. Determination of Δa_b for the femtolaser specimens.

Figure 8. Determination of $CTOD_c$ for the femtolaser specimens.

Figure 9. Normalized propagation curves.

Figure 10. Load-crack length for the femtolaser specimens.

Figure 11. Input energy divided by thickness versus crack length at constant displacement for the femtolaser specimens.

Figure 12. J-integral plot for the femtolaser specimens.

Figure 13. Specific energy as a function of the crack length for the femtolaser specimens.

Figure 14. SEM micrographs of a razor blade sharpened specimen: a) Notch tip with plastic deformation, b) Notch tip without plastic deformation.

Figure 15. Stress-displacement curves for the razor blade sharpened specimens: a) Thirteen specimens, b) Nine selected specimens.

Figure 16. Ligament length-displacement curves for the razor blade sharpened specimens.

Figure 17. EWF plot for the razor blade sharpened specimens: a) Thirteen specimens, b) Nine selected specimens.

Figure 18. Determination of Δa_b for the nine selected razor blade sharpened specimens.

Figure 19. Determination of $CTOD_c$ for the nine selected razor blade sharpened specimens.

Figure 20. Load-crack length for the nine selected razor blade sharpened specimens.

Figure 21. Input energy divided by thickness versus crack length at constant displacement for the razor blade sharpened specimens.

Figure 22. J-integral plot for the nine selected razor blade sharpened specimens.

Figure 23. Femtolaser and razor blade sharpened specimens with $l_o \approx 14$ mm: a) Measured displacement, b) Displacement shifted to d_r.