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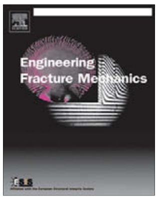

On the essential work of fracture of linear low-density-polyethylene. I. Precision of the testing method

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ABSTRACT

The precision (i.e. the repeatability) of the essential work of fracture (EWF) method in determining the fracture parameters of a highly extendible linear low-density-polyethylene film is investigated. In order to minimize any interference from external variables, a random data collection procedure is adopted to extract, from a large data set, various EWF samples with sizes ranging from 11 to 150 data points. Two different notching procedures have been considered, involving different tools (scalpel or razor blade) and cutting methodologies.

The notching procedure has only a marginal influence in terms of the correlation coefficient of the linear regression and standard error on the specific essential work of fracture (w_e). However, the mean of w_e values is markedly affected by the notching procedure, being its influence on the specific non-essential work of fracture (βw_p) parameter relatively lower. The dispersion of the w_e and βw_p data around their mean values decreases as the sample size increases, with a trend clearly affected by the notching procedure.

1. Introduction

The essential work of fracture (EWF) method is currently the only test able to furnish a fundamental fracture parameter for highly ductile materials under plane-stress conditions [1]. Even if Chen and Wu recently made an attempt to understand the underlying physics of the essential work of fracture at a molecular level [2], the method mostly relies on the empirical assumption that in some ductile tearing failures, the total energy consumed could be partitioned into the work involved in creating new fracture surfaces (i.e. the specific essential work of fracture w_e) and that involved in the plastic deformation of the region surrounding the ligament, which is non-essential and likely to be geometry dependent [1,3,4]. Over the last 20 years, the EWF method has been widely adopted for the evaluation of fracture toughness of polymer films and sheets [1]. Due to its relative simplicity, the EWF method has been used by several authors in order to assess the effect of testing variables such as temperature [5–10] and strain rate [7,11–15] on the fracture toughness of polymer films and sheets. The effects of the molar mass [16–18], the chain orientation [10,19,20], the crystallinity [21,22], and the aging (physical, hygrothermal or UV) [23–26] on the plane-stress fracture toughness of polymeric materials have been also investigated. Moreover, the EWF method has been frequently employed for evaluating the fracture toughness of polymers toughened by rubber particles [20,27–31] or containing various additives, fillers and reinforcing agents [32–36]. The compositional dependence of the fracture toughness of polymer blends has been also investigated by the EWF method [8,37,38]. More recently, the fracture toughness of polymer nanocomposites has been experimentally evaluated by the essential work of fracture method [39–46].

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Nomenclature

CV	coefficient of variation
DDENT	deeply double-edge-notched tension
h	specimens gage length
H	specimens total length
L	specimens ligament length
$\bar{L}$	mean of ligament length values
LLDPE	linear low-density-polyethylene
n	number of samples of a given size
N	sample size (number of specimens in a sample)
R^2	correlation coefficient
S	standard error of w_e (see Eq. (7))
S_{11}; S_{12}; S_{22}	sums of squared residuals (see Eq. (4))
SD	standard deviation
t	specimens thickness
w_f	specific total work of fracture
$\bar{w}_f$	mean of specific total work of fracture values
w_e	essential specific work of fracture (see Eqs. (2) and (5))
W	specimens width
W_f	total work of fracture
W_e	essential total work of fracture
W_p	non-essential total work of fracture
β	shape factor for the plastic zone
βw_p	non-essential specific work of fracture (see Eqs. (2) and (5))
σ_{max}	maximum stress on the ligament
$\bar{\sigma}_{max}$	mean of maximum stress on the ligament
σ_y	yield stress

Since 1992 the technical committee four (TC4) of the European Structural Integrity Society (ESIS) is operating to reach a standardization of the EWF method, conducting several round-robin tests on an evolving protocol [1,47,48]. Nevertheless, an international standard for the EWF method is not available yet. Still unresolved issues of the EWF method are the determination of its precision (i.e. reproducibility or repeatability) [49] and an assessment of the role played by the sample size, notching procedure [50,51], viscoelastic effects and accurate evaluation of the displacements involved [52].

Aim of the present work is to furnish a contribute for the assessment of (i) the precision (the repeatability in particular) and (ii) the role played by the notching method on the results of the EWF testing procedure.

2. Experimental

2.1. Material

The material used in the present study is a linear low-density-polyethylene (LLDPE) produced by Polimeri Europa S.p.A. (Italy) using Ziegler–Natta catalysis and hexene as a comonomer. A film with a nominal thickness of 50 μm was obtained by cast film extrusion. The most relevant physical and mechanical properties of the material are summarized in Table 1, along with details on experimental conditions and the ASTM standard involved.

Table 1
Basic properties of the investigated LLDPE film.

Property (units)	Conditions – ASTM standard	Mean value	Standard deviation
Density (g/cm^3)	23 °C – ASTM D1505	0.9178	
Melt flow index (g/10′)	190 °C/2.16 kg – ASTM D1238	2.55	
Elmendorf tear resistance (N/mm)	Direction MD – ASTM D1922	199	16
	Direction TD – ASTM D1922	214	10
Yield strength (MPa)	Direction MD – ASTM D882	8.6	0.3
	Direction TD – ASTM D882	8.5	0.2
Stress at break (MPa)	Direction MD – ASTM D882	29.3	3.2
	Direction TD – ASTM D882	27.9	3.2
Strain at break (%)	Direction MD – ASTM D882	492	33
	Direction TD – ASTM D882	600	55
Secant modulus @ 1% strain (MPa)	Direction MD – ASTM D882	185	5
	Direction TD – ASTM D882	180	6

2.2. Essential work of fracture method

The EWF approach assumes that the total work of fracture, W_f, is dissipated into two separate processes:

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

where W_e is the energy consumed in the so-called "fracture process zone" to effectively create the new fracture surfaces, and W_p is the energy dissipated in the "outer plastic region", a more diffuse zone where energy is prevalently causing plastic deformation. By assuming that W_e is proportional to the cross-sectional area of the ligament, and W_p is proportional to the volume of the outer plastic region, the following specific terms can be defined:

$$w_e = \frac{W_e}{tL} \quad \text{and} \quad w_p = \frac{W_p}{\beta t L^2} \tag{2}$$

where t is the specimen thickness, L is the ligament length and β is a plastic zone shape factor depending on the geometry of the specimen and the crack. By combining Eq. (2) with Eq. (1) the following relationship can be obtained:

$$w_f = \frac{W_f}{tL} = w_e + \beta w_p L \tag{3}$$

where the terms w_f, w_e and βw_p are called specific total, essential and non-essential work of fracture values, respectively. EWF tests were performed on deeply double-edge-notched tension (DDENT) specimens whose dimensions are reported in Fig. 1. The ligament L was varied between 5 and 15 mm. All the EWF tests were performed by an Instron universal testing machine model 4400R, at room temperature and at a cross-head speed of 100 mm/min.

Two different notching procedures were adopted. According to the notching method #1, the specimens were laid on a flat glass substrate and notched using a scalpel (Suzhou Kyuan Medical Apparatus Co. Ltd., China). As evidenced in Fig. 2a, metal templates (of various ligament lengths) were utilized, and the cuts were generated starting from the notch tip. For every 15 specimens, the scalpel was substituted with a new one. For the notching method #2, a cutting device was realized that allows one to firmly sandwich the specimens between two rigid supports, one of them consisting of a metal template (see Fig. 2b). A razor blade (Wilkinson Classic) was then used to generate notches with a sliding mode, changing it every four specimens with a fresh one. The morphology of the produced notches was observed by a scanning electron microscope (SEM) Zeiss model DSM 960. A total number of 300 specimens were tested: half of them (i.e. 150 specimens) were prepared according to notching method #1, and the remaining 150 were notched following method #2. The ligament lengths distribution of the specimens is reported in Table 2. The ligament length, L, and the thickness, t, were measured on each specimens with an optical microscope and a digital micrometer, respectively.

2.3. Data reduction and analysis

A number (n) of samples consisting of N specimens were generated by randomly extracting the data points (represented by the load–displacement curves and the corresponding ligament lengths) from the entire experimental data set. In order to minimize any interference from uncontrolled extraneous variables (such as temperature variations, sharpness of the notching tools, fluctuations of power supply, etc.), samples were generated through a randomization procedure [53] implemented in a Microsoft® Excel® worksheet. Table 2 summarizes the number and the composition of the generated samples, in terms of their ligament lengths distribution. The samples were then analyzed according to the EWF data reduction scheme in order to evaluate both w_e and βw_p parameters. According to the currently available ESIS-TC4 test protocol [1,47], a stress criterion was preliminarily applied to each sample in order (i) to ensure greater likelihood of fracture occurring under plane-stress conditions and (ii) to remove data where fracture has occurred prior to full ligament yielding. This check is based on the

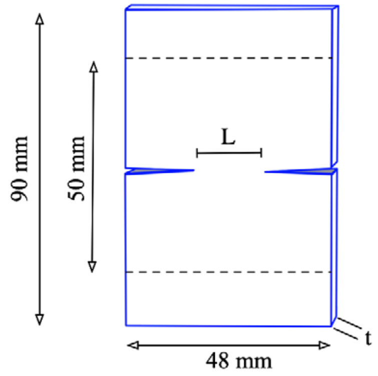

Fig. 1. Schematic of the DDENT specimens used for the EWF tests.

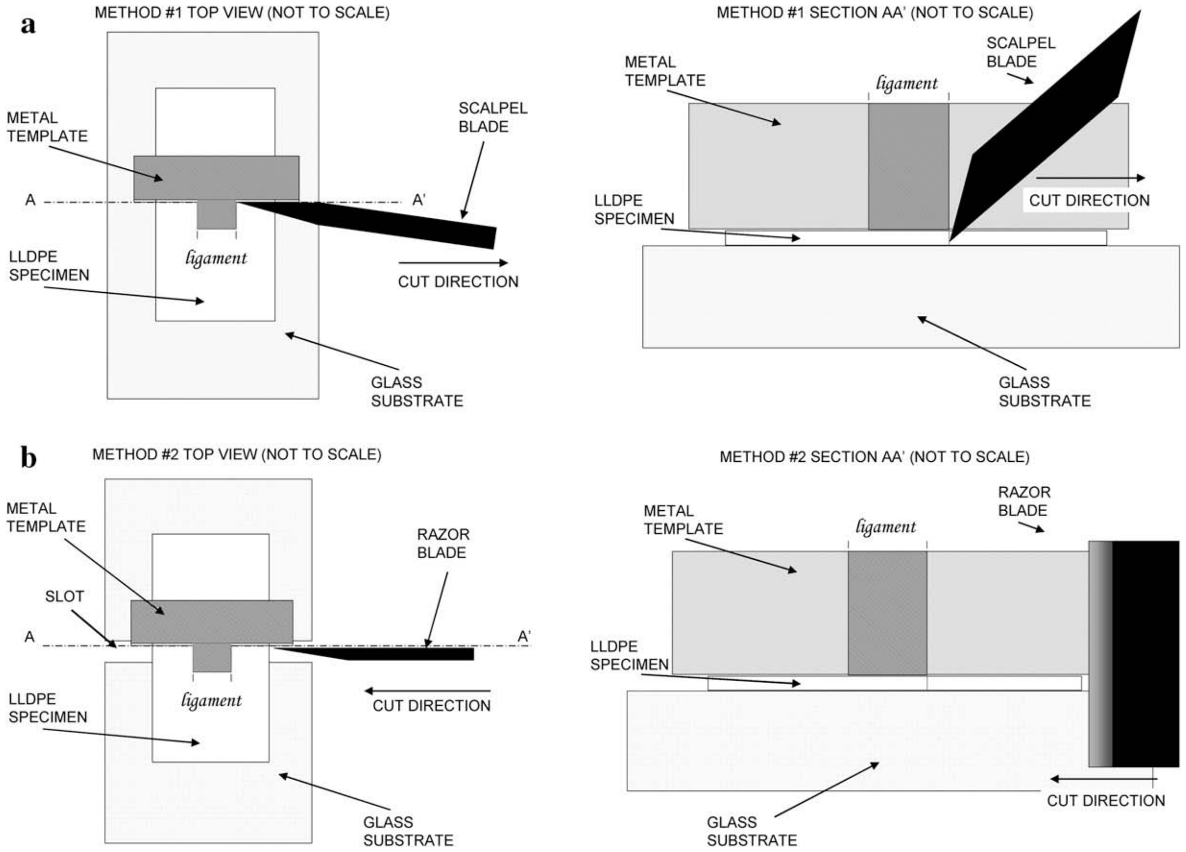

Fig. 2. Schematic representation of cutting methods #1 (a) and #2 (b).

Table 2
Ligament lengths distribution of the specimens within each sample.

L (mm)	Sample size, N (total number of specimens in the sample)													
	11	22	25	33	36	44	47	55	58	66	69	75	110	150
	Number of tested samples, n													
	13	6	6	4	4	3	3	2	2	2	2	2	1	1
	Number of specimens with ligament, L													
5	1	2	3	3	4	4	5	5	6	6	7	9	10	17
6	1	2	2	3	3	4	4	5	5	6	6	6	10	11
7	1	2	2	3	3	4	4	5	5	6	6	6	10	13
8	1	2	2	3	3	4	4	5	5	6	6	6	10	13
9	1	2	2	3	3	4	4	5	5	6	6	6	10	12
10	1	2	3	3	4	4	5	5	6	6	7	9	10	16
11	1	2	2	3	3	4	4	5	5	6	6	6	10	13
12	1	2	2	3	3	4	4	5	5	6	6	6	10	13
13	1	2	2	3	3	4	4	5	5	6	6	6	10	13
14	1	2	2	3	3	4	4	5	5	6	6	6	10	13
15	1	2	3	3	4	4	5	5	6	6	7	9	10	16

evaluation of a net section stress $\sigma_{\max} = P_{\max}/Lt$, where $P_{\max}$ is the maximum peak load. For all the data, an average value for $\sigma_{\max}$, denoted by $\bar{\sigma}_{\max}$, was determined, and the specimens for which $\sigma_{\max} < 0.9\ \bar{\sigma}_{\max}$ or $\sigma_{\max} > 1.1\bar{\sigma}_{\max}$ were rejected. For each sample, the total energy to failure (W_f) of valid specimens was calculated from the load–displacement traces, its specific value (w_f) was computed according to Eq. (3), and the data plotted against L. A least square regression line was then performed in terms of the following sums of squared residuals [1]:

$$S_{11} = \sum_{j=1}^{N} (w_{f_j} - \bar{w}_f)^2, \quad S_{22} = \sum_{j=1}^{N} (L_j - \bar{L})^2, \quad S_{12} = \sum_{j=1}^{N} (w_{f_j} - \bar{w}_f)(L_j - \bar{L}) \tag{4}$$

where $\bar{L}$ and $\bar{w}_f$ are the mean values of the ligament length and of the specific total work of fracture, respectively.

Thus,

$$w_e = \bar{w}_f - \bar{L}\frac{S_{12}}{S_{22}} \quad \text{and} \quad \beta w_p = \frac{S_{12}}{S_{22}} \tag{5}$$

The correlation coefficient R^2 of the linear regression and the standard error S on w_e can be computed as follows [1]:

$$R^2 = \frac{S_{12}^2}{S_{11}S_{22}} \tag{6}$$

$$S = \sqrt{\left(\frac{1}{N} + \frac{\bar{L}^2}{S_{22}}\right)\frac{1}{(N-2)}\left(S_{11} - \frac{S_{12}^2}{S_{21}}\right)} \tag{7}$$

3. Results and discussion

Scanning electron micrographs of the region near the notch tip are reported in Fig. 3. When notching procedure #1 is applied, some plastic deformation can be noticed on the crack boundaries, while a better quality of the notch obtained by method #2 can be clearly observed.

Examples of the load–displacement curves of specimens with various ligament lengths are reported in Fig. 4. First of all, it is important to notice that the self-similarity between the load and displacement curves is maintained for all the ligament lengths. It can be also noticed that the load–displacement curves of the specimens prepared according to notching method #1 display maximum load values systematically higher than those obtained for specimens notched according to method #2. A confirmation of this visual observation can be obtained by plotting the maximum neat stress, $\sigma_{\max}$, conventionally defined as the ratio of the maximum load to the ligament cross sectional area, as a function of the ligament length, as reported in Fig. 5. The mean value $\bar{\sigma}_{\max}$ of the maximum neat stress is 11.4 ± 0.4 MPa for specimens notched according to method #1, and it significantly decreases to 10.9 ± 0.4 MPa for those notched according to method #2. It is worthwhile to note that the $\sigma_{\max}$ values are in the range $0.9–1.1\bar{\sigma}_{\max}$, which is one of the validity criteria of the current ESIS-TC4 protocol on the

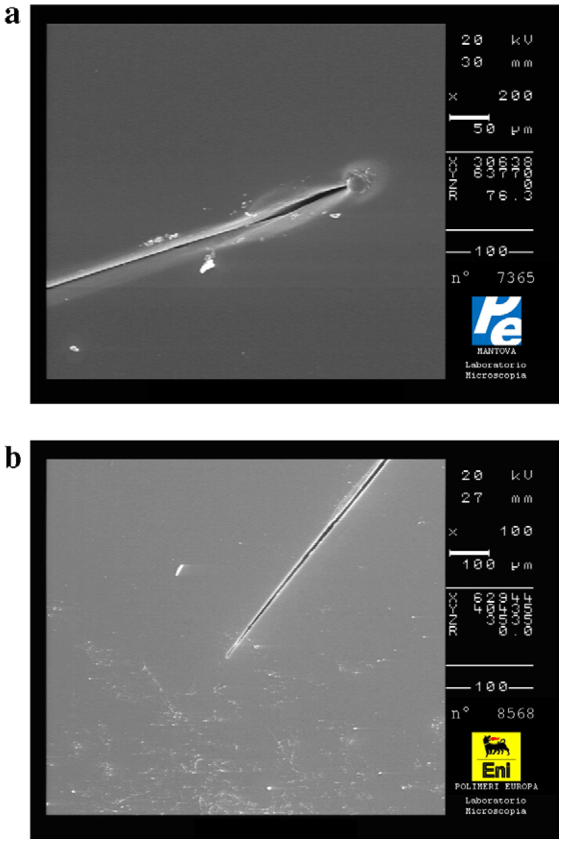

Fig. 3. Scanning electron micrographs of the region near the notch tip as obtained with notching methods #1 (picture a) and #2 (picture b).

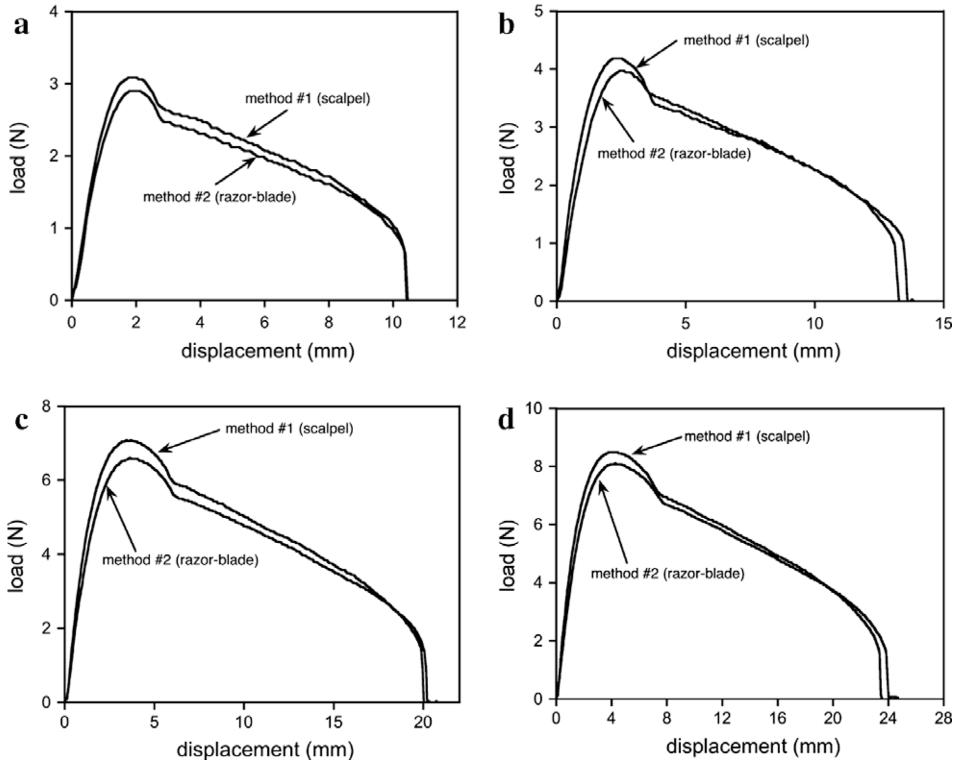

Fig. 4. Effect of notching method on the load–displacement curves of specimens with various ligament length: L = 5.1 mm (a), L = 7.1 mm (b), L = 12.2 mm (c) and L = 15.1 mm (d).

EWF approach [1]. Since the ligament is laterally constrained, the maximum neat stress values are expected to be $2/\sqrt{3}\sigma_y = 1.15\sigma_y$ [54], where the yield stress σ_y is determined in such a way that the time to peak load in the tensile test, i.e. time to yield, is roughly the same as the average time to peak load in the essential work tests. A line corresponding to $1.15\sigma_y$ is reported in the plots of Fig. 5, and it clearly emerges that the maximum neat stress values markedly deviate from the expected ones. A possible reason could be related to the marked anisotropy [55] of the mechanical behavior of the investigated materials [56].

Samples of various sizes, generated from the randomization procedure described in the experimental section, have been treated in accordance with the EWF data reduction scheme. As reported in Eq. (3), this implies a linear correlation between the total specific work of fracture values and the corresponding ligament lengths. An example of the procedure is reported in Fig. 6 for two samples of 11 specimens notched following method #1. The linearity of the data is satisfactory, being the correlation coefficient R^2 of the linear regression lines of the two data sets equal to 0.988 and 0.980, respectively. In fact, as recently suggested by Williams and Rink [1], values of $R^2 > 0.98$ are expected for an acceptable quality of the linear fit of EWF data. The effect of the sample size on the correlation coefficient of the linear regression lines of samples prepared according

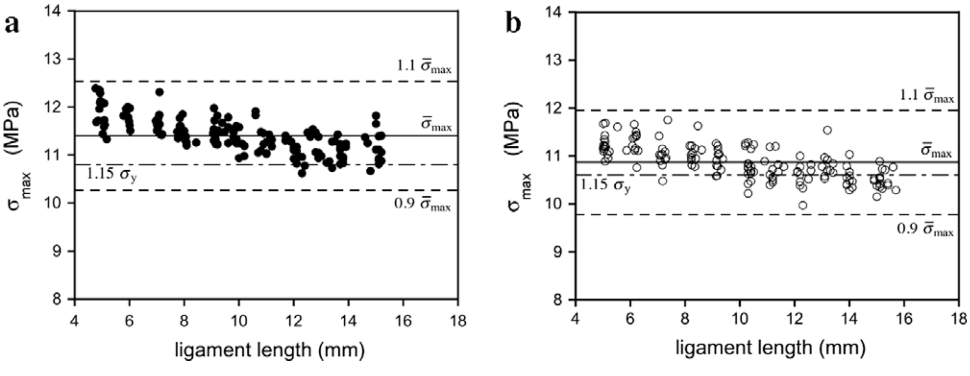

Fig. 5. Net section stress, σ_{max}, of the 300 tested specimens notched according to method #1 (a) and method #2 (b).

to notching methods #1 and #2 is summarized in Fig. 7, where the mean R^2 values are reported. It can be noticed that, independently of the adopted notching method, the linearity of the data is satisfactory (i.e. average $R^2 > 0.98$) for all the investigated sample size. Another parameter that can be used for assessing the quality of the data is the standard error S on the intercept w_e, since, in general, a value of $S < 0.1\ w_e$ is expected [1]. Fig. 8a summarizes the dependence of the mean S values on sample size for specimens prepared by both notching methods #1 and #2. First of all, it clearly emerges that the notching procedure does not practically affect the standard error on the intercept. The mean standard error decreases with the sample size to an apparent limiting value of about 1 kJ/m^2, with a trend decreasing as $1/\sqrt{N}$ (continuous line in the plot). The ratio of the mean value of the standard error to the mean value of the specific essential work of fracture is reported in Fig. 8b as a function of the sample size. Moreover, it can be noticed that an acceptable value of $S < 0.1\ w_e$ is always reached, even for the smallest samples consisting of 11 specimens. This result is in good agreement with the observation reported by Marchal et al. on a statistical procedure for improving the precision of the measurements of the essential work of fracture of thin sheets [49]. In particular, they measured w_e and the standard deviation on this value Δw_e for LLDPE sheets 290 μm in thickness using various sets of data chosen for ligaments uniformly distributed within the plane-stress region. Marchal and coworkers erroneously defined the ratio $\Delta w_e/w_e$ as a precision on the measurement of w_e, even if this parameter does not convey information on the reproducibility or repeatability of the method. Nevertheless, when plotted as a function of the sample size, this parameter shows a trend similar to that we report in Fig. 8b for the ratio of mean S over mean w_e.

The precision, i.e. the ability of a method to furnish similar results in repeated tests [57], can be now analyzed by examining the w_e values of various EWF tests repeated under the same condition. An example of two tests repeated under the same conditions is reported in Fig. 6, which gives a preliminary indication of the rather poor precision characterizing this method when a limited number of specimens (11 in this example) are tested. Fig. 9 summarizes all the w_e values obtained from samples of various sizes and notched according to both method #1 (Fig. 9a) and method #2 (Fig. 9b). At a first glance, it can be noticed that the dispersion of the w_e values diminishes as the sample size increases. A mean w_e value can now be estimated for any investigated sample size by simply taking the arithmetic average of the various specific essential work of fracture values obtained from the repeated tests (Fig. 10a). First of all, a marked influence of the notching procedure on the mean w_e value can be observed. In fact, mean w_e values are closely distributed around an average value equal to 38.8 kJ/m^2 or 35.9 kJ/m^2 for notching procedure #1 or #2, respectively. It is, therefore, quite evident that the quality of the initial notch plays an important role in determining the specific essential work of fracture values. Notching method

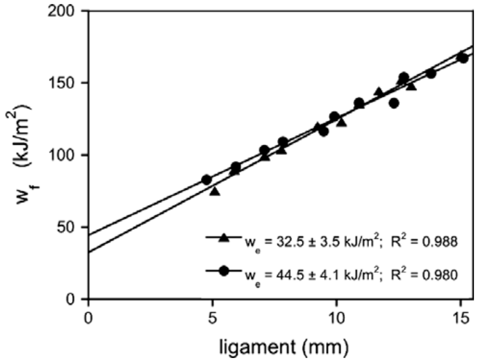

Fig. 6. Example of the variability between two EWF samples consisting of 11 specimens notched according to method #1.

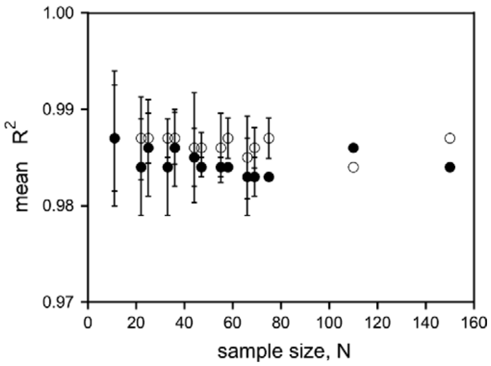

Fig. 7. Effect of sample size on the mean value of the correlation coefficient of the linear regression as obtained from specimens notched according to method #1 (●) and method #2 (○).

#2 produces notches with a lower tip radius and a lower plastic damage at the crack tip thus yielding to lower w_e values with respect to method #1. Marano and Rink recently reported on the notch sensitivity of the EWF approach [58]. In fact, they applied the EWF approach on a propylene–ethylene-copolymer film, testing specimens with various notch tip radii from 8 to 70 microns. The specific essential work of fracture values resulted to be independent of the notch tip radius for values lower than 50 micron and increasing for higher values of the notch tip radius. From our SEM observations (see Fig. 3), the notch tip radii of the specimens tested in the present work seem to be in any case lower than 20 micron, but still the different notching procedure effects the w_e parameter in a statistically significant manner.

The dispersion of the w_e data around a mean value has been quantified by computing a coefficient of variation, i.e. the ratio of the standard deviation to the mean value, which is reported in Fig. 10b as a function of the sample size. As expected, the dispersion of the w_e data monotonically decreases as the sample size increases. It is worthwhile to note that the quality

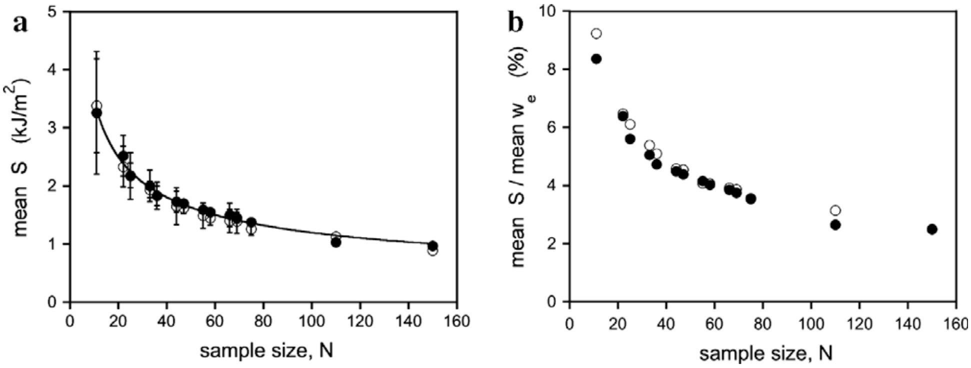

Fig. 8. Effect of sample size on (a) the mean value of the standard error S on w_e and (b) its ratio to the mean w_e value, as obtained from specimens notched according to method #1 (●) and method #2 (○).

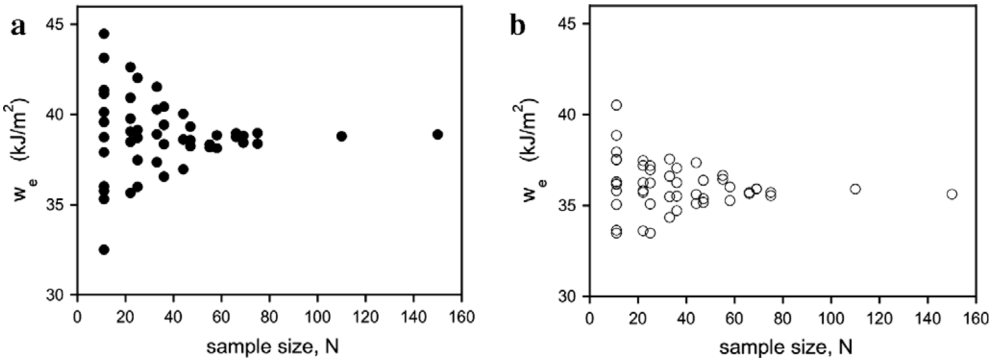

Fig. 9. Effect of sample size on the w_e values as obtained from specimens notched according to (a) method #1 and (b) method #2.

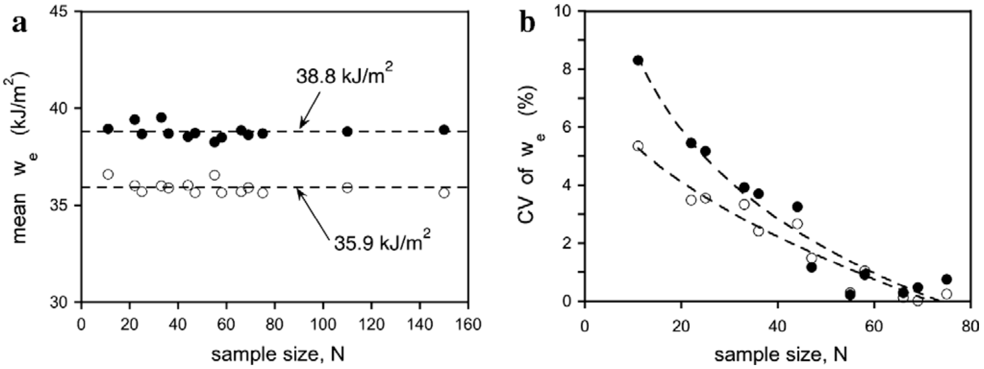

Fig. 10. Effect of sample size on (a) the mean w_e value and (b) the coefficient of variation of w_e, as obtained from specimens notched according to method #1 (●) and method #2 (○).

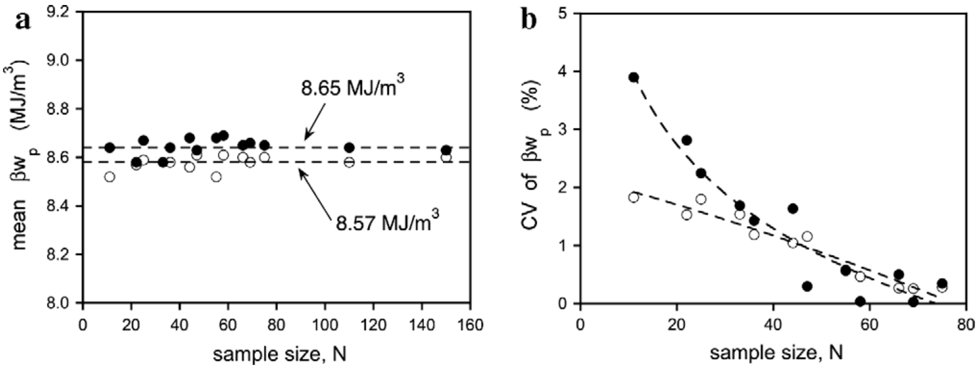

Fig. 11. Effect of sample size on (a) the mean βw_p value and (b) the coefficient of variation of βw_p, as obtained from specimens notched according to method #1 (●) and method #2 (○).

of the notch tip has an influence on the coefficient of variation. In fact, the data dispersion is generally lower when notching method #2 is adopted. For the typical sample size of 25 specimens, currently required by the current ESIS TC4 EWF testing protocol, dispersion values in the range from 3.6 to 5.2% of the mean w_e value can be estimated, depending on the notching method. Because all the experimental data have been collected in the same laboratory, by the same operator, using the same instruments and experimental conditions, and adopting a randomization procedure in grouping the data, it can be concluded that the observed dispersion of the w_e data cannot be ascribed to differences in the experimental conditions or data acquisition, and they can be considered as intrinsic of the EWF approach. As evidenced in Fig. 11, similar considerations could be drawn for as regards the influence of sample size and notching procedure on the mean value and correlation coefficient of the non-essential specific work of fracture component. Nevertheless, it can be observed that the difference between the mean βw_p values obtained using the two different notching methods is lower that 1%, while a difference of about 8% was observed between the corresponding mean w_e values.

4. Conclusions

In this work, an intra-laboratory assessment of the precision of the EWF parameters measured on a LLDPE film has been attempted, taking into particular consideration the effects of the notching procedure and of the sample size. The following conclusions can be drawn:

- the notching procedure has practically no influence on the correlation coefficient of the linear regression of the total specific work of fracture vs. ligament plots and on the standard error on the specific essential work of fracture;
- the standard error on the specific essential work of fracture decreases with the sample size, scaling as $1/\sqrt{N}$;
- even if the fracture is preceded by large plastic deformation, the mean w_e value of tests repeated under the same conditions is affected by the notching procedures, with lower values as the sharpness of the notch improves;
- the dispersion of the w_e data monotonically decreases as the sample size increases, with a trend affected by the notching procedure.

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