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Application of the essential work of fracture method in ranking the performance in service of high-density polyethylene resins employed in pressure pipes

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Abstract High-density polyethylene resins have increasingly been used in the production of pipes for water- and gas-pressurized distribution systems and are expected to remain in service for several years, but they eventually fail prematurely by creep fracture. Usual standard methods used to rank resins in terms of their resistance to fracture are expensive and non-practical for quality control purposes, justifying the search for alternative methods. Essential work of fracture (EWF) method provides a relatively simple procedure to characterize the fracture behavior of ductile polymers, such as polyethylene resins. In the present work, six resins were analyzed using the EWF methodology. The results show that the plastic work dissipation factor, βw_p, is the most reliable parameter to evaluate the performance. Attention must be given to specimen preparation that might result in excessive dispersion in the results, especially for the essential work of fracture w_e.

Introduction

High-density polyethylene (HDPE) is a semicrystalline thermoplastic polymer, ductile at room temperature, which is used for production of extruded pipes for water- and gas-distribution systems. In spite of offering significant advantages over competitors and of its increasing usage, this material is susceptible to long-term creep fracture via a stable crack growth (SCG) mechanism [1]. HDPE pipes, therefore, possess limited service lifetimes, estimated to be at least 50 years [2, 3]. In the industrial practice, however, brittle-like failure is occasionally observed to occur at shorter times [4], causing severe economic and environmental losses by leakage in the systems. These questions led to great effort of petrochemical industries in developing tougher resins and also to a large scientific and technological interest in identifying mechanisms and criteria for fracture and durability [5].

The usual practice to access the quality of basic resin for production of pressure pipes and to estimate the long-term fracture behavior of these components is to submit them to long-term hydrostatic strength (LTHS) tests [6] that result in bi-logarithm curves known in the praxis as "regression curves" [7]. These curves allow to classify different resins in classes of equal minimum required strength (MRS) as defined by the ISO 12162 standard [8]. Examples of "regression curves" are shown in Fig. 1. Note that several data points require tests with durations up to 10,000 h. These tests are thus expensive, unfit for quality control purposes and may be also criticized with respect their apparent direct transferability to design (see [7]). Therefore, alternative methods were suggested in the literature, some of them try to simplify and abbreviate the whole procedure [7], while others are based on accelerating the fracture by means of the use of notched specimens or adding surfactant agents in the water [9, 10].

Classical Fracture Mechanics methods, on the other hand, offer promising alternative techniques to the problem, especially by focusing into the crack propagation regime instead of the whole fracture process [4]. Linear elastic fracture mechanics (LEFM) parameters, like the stress intensity factor (K) or the crack extension force (G), however, have limited applicability due to the pronounced plastic behavior of the material, leading to excessive crack tip blunting effects, and also to the SCG nature of crack propagation [11–14].

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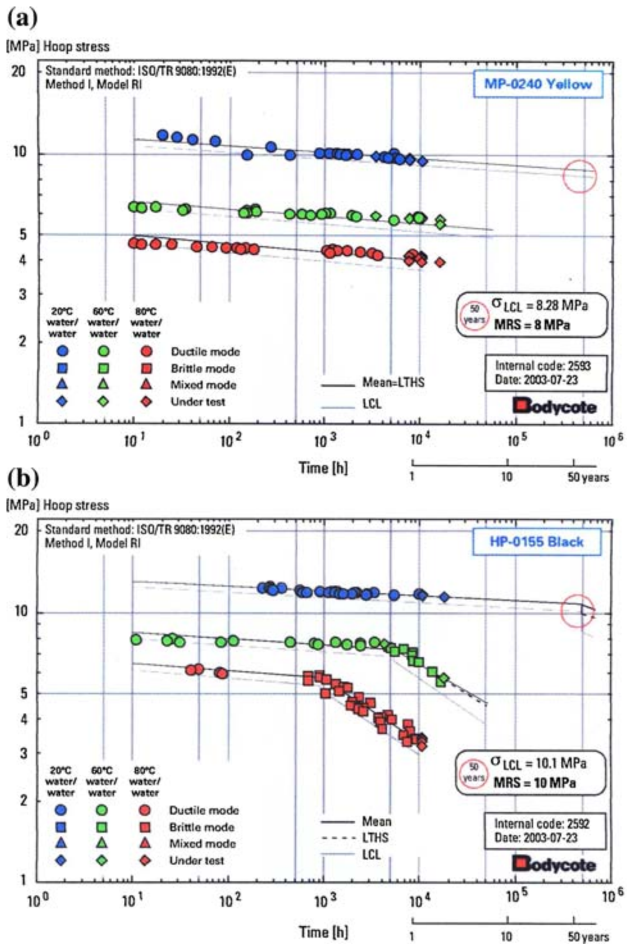

Fig. 1 Sample "regression curves" for two of the investigated resins: (**a**) MP0240 and (**b**) HP0155

Elastic–plastic fracture mechanics (EPFM) concepts, especially *J*-Integral and essential work of fracture (EWF), emerge as more appropriate parameters for assessment of fracture characteristics of high density polyethylene [15].

The EWF approach becomes attractive particularly due to its simplicity and ease of implementation [16–18]. The EWF method is usually applied in characterizing the fracture behavior of materials in the thin-film or thin-sheet form [18, 19], like those used in industrial packaging [20]. In this case, it is a fairly justified hypothesis to assume that the material works under a plane stress state. Assuming that the pressure pipe's wall is approximately under plane stress state during operation (i.e. supposing that the pipe can be approximately considered as a thin wall pipe) [21], it can be very instructive and useful to evaluate the fracture resistance of materials employed in its production through EWF parameters. The main objective of this work is to investigate the ability of the EWF method to evaluate the performance in fracture of polyethylene-based materials used in gas and water distribution systems, suitable for industrial application in ranking the performance of the resins.

Essential work of fracture

The inspiration of the EWF method is usually credited to the ideas of K. B. Broberg about SCG [22–24]. This author defines an *end-region* around the crack tip as a region of material instability were the unit separation processes occur. The end-region is intrinsically influenced by the discontinuous character of the matter, in a similar sense as the region at the crack-tip in brittle linear elastic materials, where the forces of cohesion have maximum intensity, as discussed by Barenblatt [25]. The unique characteristic of the end-region, according to Broberg, is that, under fairly general conditions its state is autonomous. The fracture process, therefore, will be controlled only by the energy flow to the end-region [24]. This is trivially realized in the case of unstable crack growth in linear elastic materials in the form of the Griffith instability criterium [23, 25].

In elasto-plastic materials and, particularly, in the case of incipient SCG this energy flow equals the energy flow to the *plastic zone*, and becomes smaller as SCG progresses due to an increasing screening action of the growing plastic zone [24]. The implication of these considerations to the EWF method is that the energy flow to the end-region, a material property, may be determined if, by some means, the contribution of this screening action of the plastic zone can be separated from the total work of fracture. This becomes evident in the recent attempts extension the EWF methodology to measure the fracture resistance of polymers in tearing mode (Mode III loading) [26, 27].

The principle of the EWF method, thus, is that the energy related to the fracture of an elastic–plastic material can be separated into two components: the essential work of fracture (related to the energy flow to the end-region) and the non-essential work of fracture (related to the screening action of the plastic zone). The first part, as already discussed, is a material property under rather general conditions. The second part, however, is controlled by the geometry of the body, by the stress distribution and by the crack length [15], and is related to the development of an *outer-region*, usually identified with the plastic zone. This region surrounds the end-region and is necessary to accommodate the large strains there observed [11, 28]. In the EWF praxis this separation is achieved by testing standard specimens with varying ligament lengths. Under rather general conditions the plastic zones formed in these specimens are self-similar, and, therefore their sizes (≡ volume) scale with the ligament length.

The basic concept was first applied in metals fracture by Cotterell and Reddel [11] and extended to ductile polymers by several researchers [5, 12–20, 28–31], including in the characterization of tearing fracture [26, 27] and of impact fracture toughness [32]. Basic principles of the method are widely described in the literature, thus only a

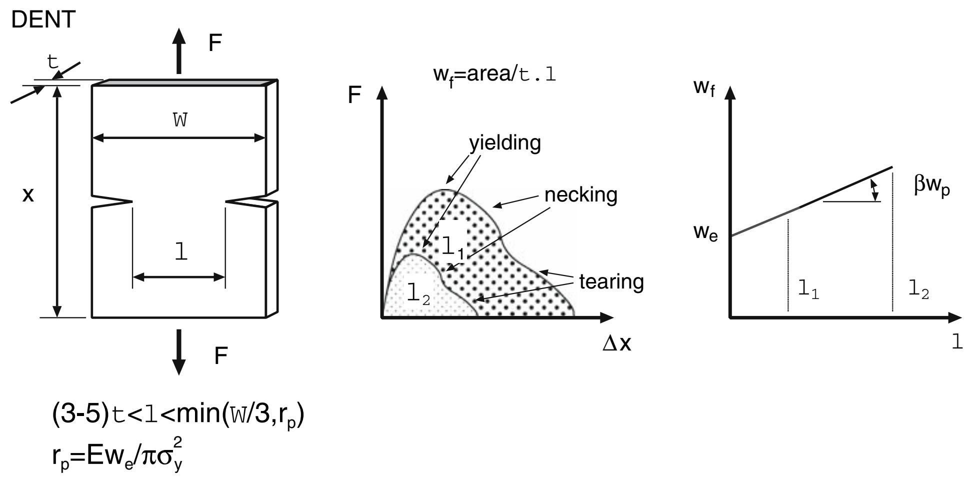

Fig. 2 Schematic representation of the EWF test with all relevant parameters. (**a**) Deep-double edge notched tension specimens (DENT), (**b**) Typical load–displacement curves obtained in the test and their dependencies on the ligament length and (**c**) representation of the evaluation of the essential (w_e) and non-essential (βw_p) parts of the specific work of fracture, r_p is an estimate of the plastic zone radius depending on E, the Young modulus, and σ_y, the yield stress of the material

brief description will be presented here. For more details the reader is directed to the works of Williams and Rink [18] and Clutton [19].

The EWF methodology requires to calculate the total work of fracture from load (F) × displacement (Δx) curves resulting from tests with several specimens with different ligament lengths. It is important to consider that in polymers both the yielding and post-yielding behaviors, as well as the fracture toughness itself, is highly dependent on strain rate and temperature [33]. The most common specimen geometry adopted in EWF tests (deeply double edge notch tensile—DENT)[34] and the general shape of the load–displacement curves are illustrated in Fig. 2.

Cotterell and Reddel [11] postulated that it is possible to separate the total fracture energy into one part spent along the fracture line and other that is spent in a volume of material surrounding the crack front. The first one is proportional to the fracture surface area and therefore to the length of ligament, whereas the second is proportional to the volume of plastic region. It has been observed for metals, as well as for plastics, that the volume of plastic region in tensile loading (Mode I) is proportional to the square of ligament length [28]. In this way, the total energy absorbed by the specimen, equivalent to the work spent in the fracture process, W_f, is given by:

$$W_f = w_e t\ell + \beta w_p t\ell^2 \quad (1)$$

where t is the film thickness, l is the ligament length of the sample and w_e and βw_p are, for the moment, material related constants. Normalizing Eq. 1 by the ligament area (tl) one obtains the specific work of fracture (w_f):

$$w_f \equiv \frac{W_f}{t\ell} = w_e + \beta w_p \ell \quad (2)$$

where w_e is now defined as the essential work of fracture (i.e. the work spent in the end-region), β is a shape factor related to the dimension of plastic zone normal to the crack line, and w_p is now defined as the non-essential part of the fracture energy, related to the plastic work dissipated per unit material volume [19]. Equation 2 implies that the specific work of fracture should scale linearly with the ligament length (l), hence the two parameters, w_e and βw_p may be determined by measuring the specific work of fracture for specimens with different ligament lengths. This is, in brief words, the principle of the EWF test. Actually this analysis is correct if only a plane stress state could be realized in the ligament area and if the whole ligament is fully yielded before start of SCG. This imposes geometric constraints to the extremal ligament lengths depending on the thickness of the specimen [12, 16, 17, 19, 29, 30, 35]. These geometric restrictions are also represented in Fig. 2.

According to Clutton [19], it is advisable to apply a stress-exclusion criterion to the results of EWF method such as to maximize the probability that the fracture has occurred under plane stress state and also to exclude data corresponding to fracture which occurred before full ligament yielding. This criterion consists in calculating a mean value (σ_m) of the maximum stress in the ligament, observed in all tests, and to exclude those results for which the maximum stress was higher than $1.1\sigma_m$ and those for which the maximum stress was lower than $0.90\sigma_m$. This is equivalent to state that a range of validity for the maximum stress around 10% of the mean stress is established.

Experimental

Materials

Six polyethylene resins, provided by four different suppliers[1] were selected for this investigation. Five out of

[1] Resin GM5010T2 was supplied by Ipiranga Petroquímica S.A, resin PC002-50R968 was supplied by Solvay Indupa do Brasil S.A., resin MDPE8818 was supplied by PBBPolisur S.A. (Dow Latin America) and resins MP0240, HP0155 and BS002 were supplied by Braskem S.A.

Table 1 Summary of the resins investigated in the present work

Resin	Class (ISO12162)	Density (kg m^{-3})	MFI (g)	LTHS/LCL (MPa)
GM5010T2	PE-80	954	0.53	9.9
PC002-50R968	PE-80	944	0.85	8.6
MDPE8818	PE-80	940	0.77	8.0
MP0240	PE-80	939	0.80	8.3
HP0155	PE-100	955	0.30	10.1
BS002	–	954	0.29	–

MFI = Melt flow index (190 °C/5 kg/10 min), LTHS/LCL = Lowest confidence limit for the long-term hydrostatic strength test (50 years/20 °C)

these six resins are specifically designed for pipe extrusion and used either in gas or in water distribution systems. The sixth resin, BS002, is designed primarily for blow molding applications and, therefore, no data on its "regression curve" is available. This resin was selected to check the behavior of a non pipe material in the test. The properties of the HDPE resins investigated in the present work are sumarized in Table 1.

Specimens and test conditions

About 0.200 mm thick films were produced by blown film extrusion in a laboratory-scale extruder, from c.a. 90 mm diameter balloons (die diameter: 60 mm; die gap opening: 0.8 mm; blow-up ratio: 1.5:1, barrel temperature: 190 °C, production rate: 5.8 kg/h).

Rectangular 130 mm length × 32 mm width strips were cut from the films such that the ligaments were parallel to the axial direction of the balloons (with one exception, see below). Following Clutton [19] five ligament classes were adopted: 6, 8, 10, 12, and 14 mm. Five to six specimens were produced for each ligament class, amounting 25–30 specimens for each material. For the case of resin GM5010T2 a special test was designed to test the influence of sample anisotropy on the EWF results. In this case specimens were extracted such that the ligament was perpendicular to the axial direction of the balloon.

These rectangular strips were used for the production of DENT specimens (see Fig. 2), via the introduction of symmetric pre-cracks at the middle of the strip length. These pre-cracks were introduced with an ordinary sharp steel scalpel per hand using a steel ruler as a guide. In order to achieve two notches directly opposite one another a line was drawn in the strip across its width prior to notching, as suggested by the European Structural Integrity Society (ESIS) protocol [19]. Specimen dimensions (thickness and ligament length) were measured with a caliper square with resolution of 0.01 mm.

The tests were conducted in tensile mode in a electromechanical testing machine operating with a 500N load cell under displacement control at constant crosshead speed of 5 mm min^{-1}. Testing temperature was controlled, corresponding to 25 ± 2 °C.

Results and discussion

Typical load (F)–displacement (Δx) curves obtained in the EWF tests are shown in Fig. 3. They are consistent with the expected behavior reported for similar resins in the literature [19].

Figure 4 shows an example of the procedure for evaluation of w_e and βw_p and of the application of the exclusion criterium for the for the particular case of resin MDPE8818. Results which fail to comply with the exclusion criterium are marked as excluded in Fig. 4a. Table 2 shows the results obtained in all EWF tests for the six resins here investigated.

Both the large confidence intervals observed for w_e and the relatively low values of r^2 in Table 2 reflect the large dispersion in the EWF results (see Fig. 4). One possible origin for this excessive dispersion is the rough method used for pre-crack introduction. It is interesting to observe, however, that SCG starts only after a large crack blunting is observed (of about 1 cm radius) in the course of the EWF tests. It is quite improbable, thus, that the sharpness of the pre-crack would be a reason for this dispersion, since very large local deformations must exist at the crack tip prior to onset of SCG. It is more probable that the damage introduced during sample pre-cracking would

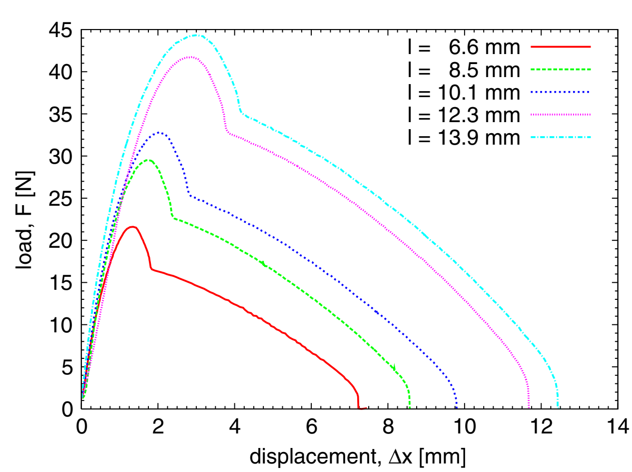

Fig. 3 Sample load–displacement curves obtained in the EWF tests. Resin GM5010T2—ligament perpendicular to the extrusion direction

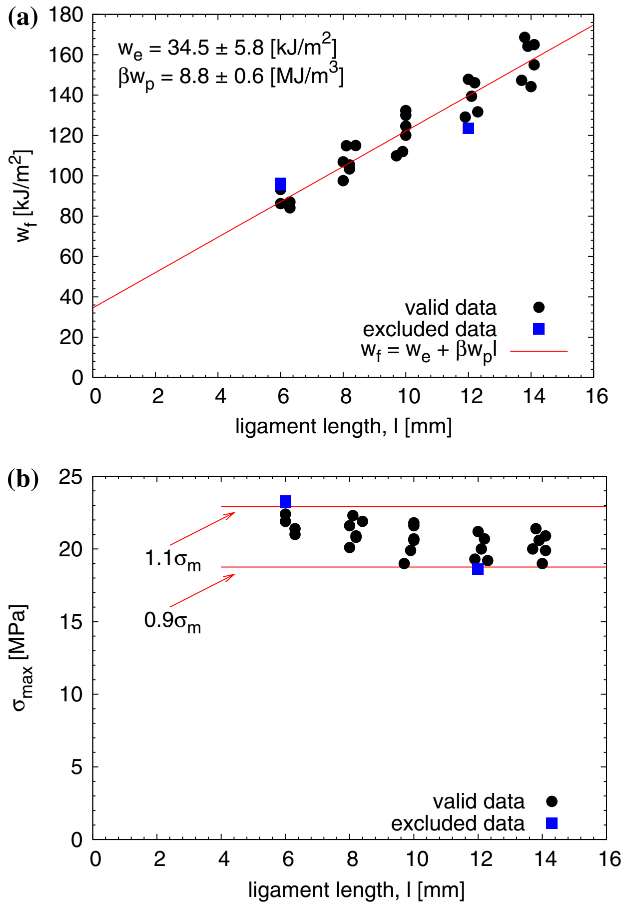

Fig. 4 Sample result from the EWF test: (**a**) evaluation of w_e and βw_p for resin MDPE8818 and (**b**) illustration of the application of the exclusion criterium suggested by Clutton [19]

significantly influence the formation of the plastic zone and then produce variations in its shape. This hypothesis is subject of an on-going research work by the present group.

According to Fayole and Verdu [5], the plastic-work dissipation factor, βw_p, is the indicated parameter to evaluate structural effects on toughness of polyethylene with regards to the quality of the resin for pipe application. This parameter depends on the test's strain rate and probably also on specimen geometry, but, as it can be seen in Table 2, it is less affected by the large dispersion in the data, resulting in significative differences among the behavior of the six resins. In other words, this parameter, in principle, is able to rank the resins.

The βw_p parameter is connected to the plasticity of material in the outer-region, and therefore correlates with the resin's ductility. We can make some inferences by comparing its values with qualitative aspects of the material's "regression curve." In this way, resin HP0155 has the lowest βw_p value among all investigated resins (6.2 ± 1.0 MJ m^{-3}), while resin MP0240 has the second largest value of βw_p (9.6 ± 1.4 MJ m^{-3}) of all investigated

Table 2 Results from the EWF tests for all resins here investigated

Resin	Orientation	w_e [kJ m^{-2}]	βw_p [MJ m^{-3}]	r^2
GM5010T2	⊥	32.4 ± 10.8	11.1 ± 1.0	0.931
MP0240	‖	20.9 ± 15.2	9.6 ± 1.4	0.933
MDPE8818	‖	34.5 ± 11.6	8.8 ± 1.2	0.937
GM5010T2	‖	31.6 ± 9.2	8.3 ± 0.9	0.929
BS002	‖	34.4 ± 12.1	8.1 ± 1.1	0.931
RIGIDEX PC002-50R968	‖	37.6 ± 15.4	7.3 ± 1.4	0.945
HP0155	‖	35.0 ± 10.4	6.2 ± 1.0	0.937

Error estimates represent the 95% confidence limits for the respective parameter, as obtained in the linear regression. Orientation of the ligament is defined relative to the main extrusion direction, and r^2 represents the square of Pearson's correlation coefficient for the linear regression

cases.[2] Comparing their "regression curves" (Fig. 1), we observe that resin HP0155 is more prone to the to brittle-like fracture compared to resin MP0240, as indicated by the observation of the brittle-to-ductile transition at 60 and 80 °C only in the former resin, while no transition is observed for the later. This happens in spite of HP0155 being classified as PE-100 due to its performance in LTHS tests at room temperature, which reinforces the critics expressed in [7] to the use of the "regression curves" as an evaluation tool for the in-service fracture behavior: in this case the more "brittle" resin would receive a high classification from the point-of-view of design. From the remaining resins, Rigidex PC002-50R968 shows a βw_p value of 7.3 ± 1.4 MJ m^{-3}, while GM5010T2, shows 8.3 ± 0.9 MJ m^{-3}, which suggests a better performance of the later resin in terms of the tendency to brittle-like fracture. This is consistent with their regression curves (see [7]), since the ductile-to-brittle transition is not observed for GM5010T2, even at 60 and 80 °C, while it is for Rigidex PC002-50R968. On the other hand, the higher βw_p value of the MP0240 Resin is consistent with its superior behavior in the ductile wing of the "regression curve" compared with GM5010T2 (i.e. the ductile wing of the "regression curve" for MP0240 shows higher fracture times, compared with that of GM5010T2). There is no high temperature data in the available "regression curve" of resin MDPE8818 and no "regression curve" is available at all for resin BS002, so comparisons for these two materials cannot be made on the same basis. However, assuming that this ranking is indeed indicative of the "quality" of the resin, MDPE8818 would present a comparable performance to GM5010T2, which is plausible, since both are commercial advanced resins. Surprisingly enough, BS002, which is not designed for pipe

[2] It is the largest value if we compare only the cases in which the ligament is parallel to the axial direction of the balloon.

extrusion, would present superior performances relative to both RIGIDEX PC002-50R968 and HP0155 according to this ranking. Testing this resin as candidate for water pipe extrusion (i.e. obtaining its "regression curve") would be an interesting exercise.

An additional test was made to verify the impact of the exclusion criteria in the determination of the EWF parameters. In the case of resin MDPE8818 (the data shown in Fig. 4), the inclusion of the non-valid data (two points) in the regression leads to the EWF parameters $w_e = 34.48 \pm 11.4$ kJ m^{-2} and $\beta\ w_p = 8.76 \pm 1.1$ MJ m^{-3}, i.e. representing only a minor change when compared with the values shown in Table 2. For the remaining resins this inclusion leads to varying degrees of change. It was observed that the tests which presented larger number of excluded points resulted also in a larger variation of the EWF parameters, notably for w_e. In all cases here presented, however, these changes are compatible with the dispersion of the data (i.e. the new parameters are withing the 95% confidence limits of the parameters shown in Table 2).

Processing imposes anisotropy to extruded profiles, so we should expect superior mechanical properties (particularly strength) in the main extrusion direction due preferred chain orientation. This was observed with results for resin GM5010T2, where the values of βw_p corresponding to perpendicularly oriented ligament is about 30% higher than the parallel ligament, which is consistent with an improved performance of the resin in this direction.

Williams and Rink [18], assuming that the ligament is fully yielded and that during crack advance the crack front assumes the shape of a semi-circle, deduced that:

$$\beta w_p = \frac{\sigma_y}{2} \tag{3}$$

where σ_y is the tensile yield stress of the resin. Table 3 shows the comparison between the values of $2\beta w_p$ and the yield stresses independently measured[3] for five out of the six resins [7].

It is observed in Table 3 that a relatively good agreement with Eq. 3 is obtained. The deviations can be justified by the following arguments: let us consider crack blunting effects to fracture toughness, characterized by the ratio $\frac{K_{IB}}{K_{IC}}$, where K_{IB} is the stress intensity factor measured at onset of crack growth and K_{IC} is the lowest value of stress intensity factor for crack propagation (equivalent to the fracture toughness for an hypothetical sharp crack), as defined in [36]. For brittle (unstable) crack growth this ratio should be ≈ 1, being this the lowest bound to this ratio, since no significative crack tip blunting is expected in this case. In the

[3] Measured according to ASTM D 638, using type IV tensile specimens produced from compression molded plates. The test was conducted at 25 ± 2 °C and 5 mm min^{-1} crosshead displacement rate.

Table 3 Comparison between $2\beta w_p$ and σ_y for the resins here investigated

Resin	σ_y [7]	$2\beta w_p$
MP0240	15.7 ± 1.1	19.2 ± 2.8
MDPE8818	15.5 ± 0.8	17.6 ± 2.4
GM5010T2	17.8 ± 0.5	16.6 ± 1.8
RIGIDEX PC002-50R968	16.4 ± 1.0	16.2 ± 2.8
HP0155	18.7 ± 0.9	12.4 ± 2.0

Results are given in MPa, which is dimensionally equivalent to MJ m^{-3}

presence of a plastic zone at crack tip, however, blunting occurs and this this ratio tends to increase. As demonstrated by Kinloch and Williams [36] for the case of five epoxy resins, the ratio $\frac{K_{IB}}{K_{IC}}$ depends exponentially on $(\sigma_y)^{-1}$. It is reasonable to assume that the shape of crack tip front can deviate from semi-circle to a half-ellipse depending on the value of σ_y, as well. For more ductile materials the minor axis of half-ellipse would be parallel to the ligament, leading to a ratio between $2\beta w_p$ and σ_y higher than unity. Possibly this occurs with resin MP0240, and is compatible with the "regression curve" of material, where no brittle-to-ductile transition is observed, as already discussed (see Fig. 1a). On the other hand, less ductile materials should present the major axis aligned with the ligament direction (tending to a sharp crack), leading to a ratio between βw_p and σ_y lower than 0.5. This occurs with resin HP0155 and is confirmed by the presence of pronounced brittle-to-ductile transitions in the high temperature data of the "regression curve" (see Fig. 1b).

As previously stated, βw_p generally depends on sample geometry. As a consequence, the ranking discussed above may, in principle, also depend on DENT sample thickness and be quite different to what would be observed for the pipe in service. It can be argued, however, that toughness is expected to depend linearly on thinness provided the whole ligament is submitted to a plane stress state and as long as the material's microstructure remains the same. It can be expected also that, if the plastic zone shape is invariant, βw_p should not vary significantly with sample's thickness [12]. This statement has been experimentally verified in the case of AA6082-O aluminum samples with thickness varying from 1 mm to 6 mm [16]. Finally, even if the plastic zone shape changes with sample thickness, one should expect that the approximated proportionality between βw_p and σ_y predicted by Williams and Rink [18] and verified in the present work would warrant that no dramatic inversion of positions in the ranking should be expected when testing samples of the same materials with different thickness. As mentioned before, the pipe's wall is expected to work under a plane stress state [21], so the same arguments may apply to the material in the form of a pressure pipe.

Conclusions

EWF method is useful to rank polyethylene resins used in pressure pipes fabrication. The plastic work dissipation factor, $\beta\ w_p$, is the indicated parameter to do this differentiation: resins with higher values of βw_p are more ductile, and therefore, behave better in LTHS tests. This "ranking" allows a better classification of the resins compared with the usual methodology adopted by ISO 12162, in the sense that βw_p detects tendencies to brittleness which are present in the "regression curves", but which would not be identified using a MRS classification, as defined by this standard. There are evidences for a dependence of the EWF results in the pre-cracking method, in the form of an increased dispersion of the data. As discussed in the present work, it is more likely that this effect is a consequence of damage introduced during pre-cracking, rather than of introducing pre-cracks with insufficient sharpnesses. An approximate identity between material's yield stress and βw_p, as suggested by Williams and Rink [18], was confirmed for the present resins and it was argued that deviations from the perfect correlation could be justified by crack tip blunting effects, related to the material's plastic behavior.

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