

## Critical Energy Analysis of Fatigue Brittle-To-Ductile Transition in Polyethylene Gas Pipe Materials

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**Abstract:** Plastic pipes used for gas and water industries are retaining more and more attention through the multitude of studies that are concerning various behavioral aspects. Recent statistics indicate that more than 90% of newly installed piping gas systems throughout the world are made of Polyethylene (PE) because of its ease of installation and relatively low cost. Today, it is well established from laboratory work that extruded plastic pipes fail in a ductile manner as applied loads are sufficiently high and failure zone is characterized by large deformations around the damaged area. Despite such favorable ductility, PE pipes are also found to undergo brittle-like fracture when subjected to low stresses for long service periods. Such conditions usually favor Slow Crack Growth (SCG) fracture mode and as a result, constant load tests exhibit two general crack propagation mechanisms, ductile failure which is dominated by large scale homogeneous deformations in the bulk and brittle failure that starts at stress concentration points. This study is aimed to investigate fatigue brittle-to-ductile transition in Polyethylene pipes through a correlation between crack growth rate and the amount of irreversible work expended on viscoelastic processes in the bulk. Fatigue crack propagation tests carried out at ambient temperature show that two important damage mechanisms are competing while crack is running. These are brittle and ductile failure mechanisms. The proposed method is based on the measurement of two fatigue parameters: the rate of surface crack growth, obtained at different loads levels and the rate of irreversible work which is calculated from fatigue instantaneous hysteresis loops. The obtained correlations, for maximum fatigue loads between 20 and 35 % of the yield stress, show average critical energies of 211 and 695 J/m<sup>2</sup> respectively for brittle and ductile regimes.

**Key words:** Polyethylene, crack propagation, BDT, critical energy

### INTRODUCTION

Distribution networks for natural gas and water supplies in cities are basically made of plastic pipes with diameters reaching 250 mm and more depending on the pressure rate. These polymers are continuing to be the subject of many studies that highlight various behavioral aspects in terms of service lifetime<sup>[1]</sup>, mechanical characterization and structure relationship<sup>[2]</sup>, loading modes<sup>[3]</sup>, residual stresses<sup>[4]</sup> failure mechanisms<sup>[5]</sup> and environmental effects<sup>[6]</sup>. The design of thermoplastic pipes is achieved through the "Rate Process Method for Projecting Performance of Polyethylene Piping Components" which is standardized in ASTM D-2837 and D-2513. The calculation is based on a 3 coefficients-equation linking lifetime (time-to-failure), hoop stress and testing temperature. Newly installed piping gas systems in the world are exclusively made of PE because of its ease of installation, relatively low cost and long-term reliability against environmental aggressive agents. Today, it is well established from laboratory work that extruded plastic

pipes fail in a ductile manner as applied loads are sufficiently high and the failure zone is characterized by large deformations around the damaged area. Despite such favorable important ductility, PE piping and fitting materials are also found to undergo brittle-like fracture when they are subjected to low stresses for long service periods combined to given temperatures above the ambient. Such conditions usually favor Slow Crack Growth (SCG) fracture mode in Polyethylene pipes. As a result, constant load tests exhibit two general crack propagation mechanisms, ductile failure which is dominated by large scale homogeneous deformations in the bulk and brittle failure that starts at stress concentration points and propagates slowly preceded by a craze zone containing transformed material. As a general rule, failure curves are well described by the following relationship:

$$t_f = C\sigma^{-n} \quad (1)$$

where C is a positive constant representing stress level at which the material fails at unit time (also termed as the

load capacity parameter). The exponent  $n$  is a damage evolution index and is in the range ( $2.5 \leq n \leq 4.5$ ) and ( $20 \leq n \leq 27$ ), respectively for brittle and ductile regimes and the transition stress is roughly equal to half yield stress<sup>[7]</sup>. In a study of PE homopolymer failure under plane strain conditions over a range of temperatures and speeds, it was found that at a given temperature brittle-like behavior has been observed at high and low speeds while ductile failure characterized intermediate speeds<sup>[8]</sup>. An increase in temperature shifted brittle-ductile-brittle transitions to higher speeds because of a competition between 3 rival mechanisms, brittle failure by disentanglement, ductile shear yielding and brittle failure by chain scission<sup>[9]</sup>. Using Charpy impact test, Brittle-Ductile Transition Temperatures (BDTT) of PE were investigated with different degrees of crystallinity. A rise in the cooling plate temperature for MDPE and HDPE brought out a fall in BDTT and an increase in impact energy<sup>[10]</sup>. With decreasing degrees of entanglement density, PE exhibited a slightly decreasing BDTT<sup>[11]</sup>. Short term failure in PE is accomplished through fibril breakdown within the central part of the craze because of overall yielding whereas long term failure occurred by an accumulation brittle damage as micro voids at fibril roots and film joints<sup>[7]</sup>.

Figure 1 is a typical representation of experimental stress versus time-to-failure obtained from sustained pressure tests for different pipe materials<sup>[12]</sup>. The transition from brittle to ductile failure can be observed as the slope of a given curve changes (Knee-type curve). It becomes clear that extrapolating linearly such data to estimate the long-term stress (30 or 50 years) is not a realistic approach.

The objective of this research to study Fatigue Brittle-to-Ductile Transition (FBDT) in Polyethylene pipes through an eventual correlation between crack growth rate and the amount of irreversible work expended on viscoelastic processes in the bulk. The proposed method is based on the measurement of two fatigue parameters, the rate of surface crack growth, obtained at different loads levels, which is expressed as

$$\left( \frac{1}{t_0} \frac{da}{dN} \right)$$

where  $t_0$  is the initial thickness and the rate of irreversible work

$$\left( \frac{dW_i}{dN} \right)$$

which is calculated from fatigue instantaneous hysteresis loops. In order to validate these results with brittle and ductile failures, microscopy is used to analyze the extent of associated damage zone ahead of the crack-tip.

**Experimental procedures:** The pipes were extruded from a non-pigmented Phillips Petroleum Co. TR418 resin. The weight-average molecular weight is 170000 and the density is 939 kg m<sup>-3</sup>. The MFI is in the range 0.4 to 1.5 and the degree of crystallinity is 67%±0.03<sup>[13]</sup>. The minimum pipe wall thickness is 11 mm and the average outside diameter is 115 mm. Because of the fact that the specific state of residual stress and micro-structural gradients have important effects on the rate of crack growth, 28 mm wide rings are cut from the pipe and then C-shaped specimens are machined as described in ASTM E-399. Fig. 2 shows the specimen together with its clevis grips. Special steel sleeves were designed to reinforce the loading lines of the specimen and to avoid high friction between the metallic pin and the polymer. The distance  $S$  separating the loading holes is 25 mm. A 2.5 mm deep notch was introduced longitudinally at the pipe bore using a razor blade and a special press.

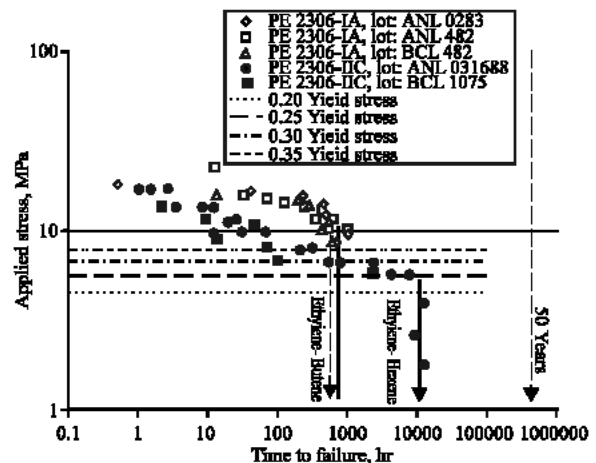


Fig. 1: Stress versus time-to-failure under sustained pressure for 50 mm OD pipes (PE2306-IIC: 2 lots) and (PE2306-IA: 3 lots). Horizontal discontinuous lines show the fatigue testing levels in this study

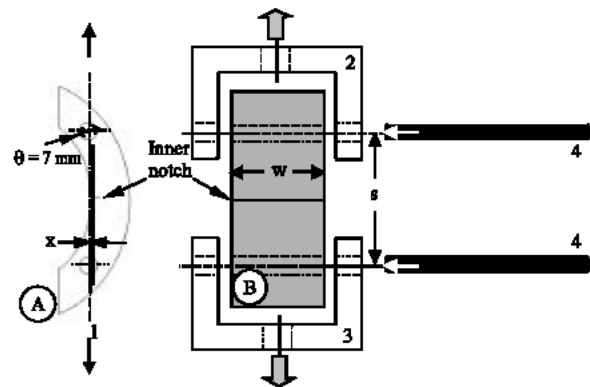


Fig. 2: Schematics of side (A) and front (B) views of Polyethylene C-shaped specimen with its gripping metallic machined fixtures (2 and 3) and loading pins (4).

Tension-tension crack propagation experiments were carried out on a MTS servo-hydraulic testing machine. The maximum load was set at 20, 25, 30 and 35% of  $\sigma_y$  while the minimum-to-maximum load ratio (R) was chosen to be 0.1 to avoid creeping effects that occurred at higher R ratios. The cycling frequency was kept constant at 0.5 Hz to reduce hysteric heating, which, in turn, might cause structural changes in the material. An X-Y plotter was used to record load-displacement relationships at selected crack lengths while testing.

## RESULTS AND DISCUSSION

It is known that brittle and ductile regimes co-existed during fatigue crack propagation and both evolved continuously towards final material tearing. Also, the lower the fatigue stress levels, the higher the brittle contributions. This is confirmed in brittle-to-ductile testing under sustained pressure and also from service failure observations as the known brittle-like failure usually occurs at low stresses for longer durations. The brittle regime is usually characterized by fatigue bands, which indicate discontinuous crack propagation<sup>[5-7]</sup>. Fig. 3 represents experimentally measured energy release rates (ERR) at different load levels from load-displacement curves using the relation:

$$J_1 = -\frac{1}{t_0} \frac{dP}{da} \quad (2)$$

where  $(dP/da)$  is the change in potential energy per change in crack length. The behavior of the ERR curves is similar for varying applied mean stress, as they show an increase in energy with crack length. This state is predicted since released energy is basically spent on

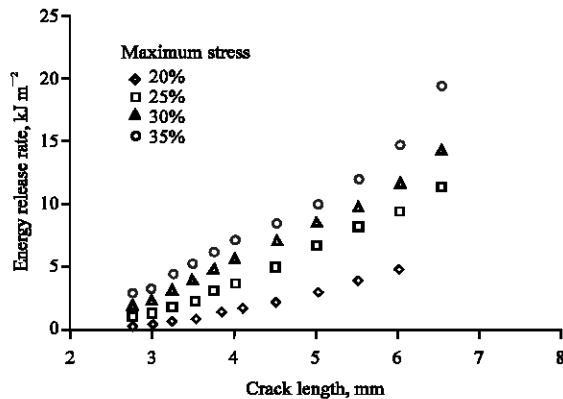


Fig. 3: Experimentally measured energy release rate from load-displacement curves as a function of crack length at different load levels

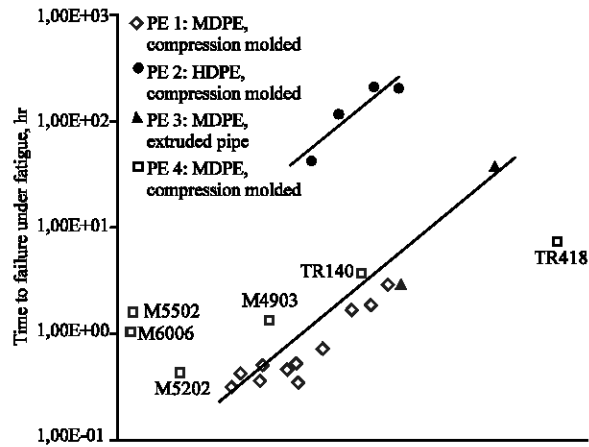


Fig. 4: Relationship between failure times under creep and fatigue modes for different resins<sup>[5,14,15]</sup>

Table 1: Comparison of initiation, brittle and ductile lifetimes as a function of applied Stress level. \* Tests stopped and fractured in liquid nitrogen

Pipe material	Stress (%)	N initiation (10 <sup>3</sup> cycles)	N brittle (10 <sup>3</sup> cycles)	N ductile (10 <sup>3</sup> cycles)	N total (10 <sup>3</sup> cycles)
PE2306-IIC	20	251	347	>22*	>620*
	25	140	65	35	230
	30	90	50	66	206
	35	75	40	35	150
PE2306-IA	25	10	6	15	30

crack propagation. The measurement is achieved using load-displacement curves for different crack lengths. Compared to elastic energy, this measurement is more representative as it includes all other irreversible processes due to crack incursion. Since ASTM tests are based on creep data, it is interesting to compare it with fatigue data. Figure 4 illustrates such relation between failure times from both creep and fatigues modes. The data were gathered from literature and a proportional relation is found to characterize both modes. This is an important result which states that fatigue tests can also be used to draw conclusion on pipe life under acting pressure<sup>[5,14,15]</sup>. Table 1 summarizes the initiation periods compared to total number of cycles. For lower stress level, as expected initiation takes longer times which is the same trend followed by brittle propagation. For the ductile regime, the 20% condition had to be each time failed in liquid nitrogen, as the durations are too long. Roughly, the trend is maintained especially for the limiting conditions. When comparing to another piping material (PE2306-IA), the same decomposition can be made and since it is known from service that this material is of a lower performance, it is observed that the performance is very poor.

In order to study the FBDT, it is interesting to use an energetic approach proposed by N. W. Klingbeil for ductile solids failed under fatigue<sup>[16]</sup>. This study is

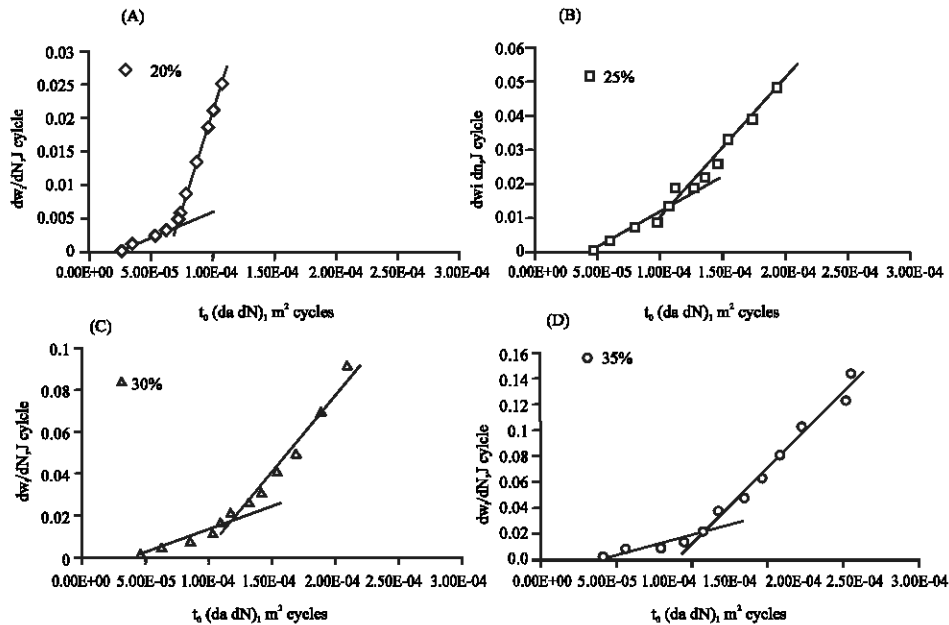


Fig. 5: Rate of irreversible work spend on deformations as a function of corresponding fatigue crack growth rate at (a) 20, (b) 25, (c) 30 and (d) 35% of applied stress

concerned with a new theory of fatigue crack growth in ductile solids based on the total plastic energy dissipation per cycle ahead of the crack tip. The FCG is explicitly given in terms of the total plastic dissipation per cycle and the plane strain fracture toughness of the material from the equation:

$$\frac{1}{t_0} \frac{da}{dN} = \frac{1}{G_c} \frac{dW}{dN} \quad (3)$$

where  $G_c$  is the critical energy release rate. In this instance, typical plots of the rate of irreversible plastic work as a function the crack propagation rate are constructed. These plots for 20, 25, 30 and 35% of yield stress are shown in Fig. 5. It is observed that both damage mechanisms are well separated and each slope would represent an independent resistance to fatigue crack propagation. The equivalent load levels are also indicated in Fig. 1 using horizontal dashed lines. The computed values of toughness are summarized in Table 1. It found that the brittle regime critical energy release rate so calculated lays between 97.7 and 264 J/m² and these values are quite small compared to the measured energy rates from potential energy evolution (Fig. 3). On the other hand, the ductile energies varied from 589.5 to 987.8 J/m². The ductile regime energy is important as it considers a lot of energy spent on damage and high deformation. There is a consistent evolution of calculated  $G_c$  as this value is much higher when ductility becomes important.

Table 2: Calculated fracture toughness from brittle-to-ductile transitions under fatigue testing with determination coefficients

Stress level (% $\sigma_y$ )	$G_c$ brittle (J/m²)	$R^2$ (brittle)	$G_c$ ductile (J/m²)	$R^2$ (ductile)
0.20	97.7	0.937	589.5	0.997
0.25	256.5	0.928	411.6	0.976
0.30	226.2	0.971	790.1	0.982
0.35	264.0	0.980	987.8	0.988

In order to confirm such approach, microscopy is used to identify the plastic zone ahead of the crack tip. Thin sections were obtained from interrupted fatigue crack propagation tests and observed using microscopy. The results are illustrated in Fig. 6. In the brittle regime, the plastic zone is very small and is associated with a main craze. The latter is made of yielded material and structural voids. Under polarized light, the extent of damaged material is confined to a narrow zone next to the crack plane. For the ductile regime, the damaged zone is much important and the craze are longer compared the brittle stage. The SEM examination illustrates the multiple crazes and the highly deformed material at the crack-tip. Around the damage zone, another part of the transformed material is highly affected plastically as its limits extend many folds until the specimen edge (Fig. 6).

This approach is interesting as it allows separating brittle from ductile contributions in a given FCP test. The use of microscopy enables to confirm the extents of each mechanism from fracture surface analysis and damage dissemination within Polyethylene. Compared to Long-Term Hydrostatic Strength (LTHS), FCP may be considered

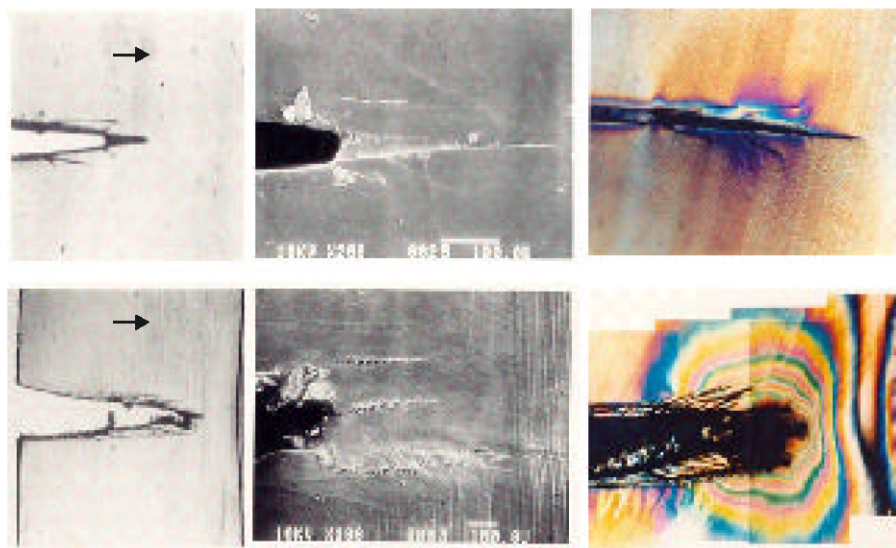


Fig. 6: Optical, SEM and polarized light photomicrographs of plastic zone shape ahead of the crack-tip in both brittle and ductile regimes under fatigue mode as obtained by diamond disc sectioning

as a more efficient approach since it helps produce BDT in much shorter times. A recent study showed that under creep testing, ductile failure is primarily driven by the yield stress of PE. The examination of ductile failure data at different temperatures also indicated that a systematic improvement in performance is observed with increasing temperature and this might be related to the progressive relaxation of internal stresses<sup>[17]</sup>.

### CONCLUSION

The results of the study allow making the following conclusions based on observations and the energetic analysis:

- The C-shaped specimen is a good candidate to study crack propagation in Polyethylene pipes.
- Crack propagation acceleration may be obtained using fatigue loadings with appropriate frequency to avoid hysteric heating of tested material.
- Brittle fracture can be reproduced from fatigue testing. The lower the applied mean stresses the higher the brittle contribution as stated from microscopic analysis.
- The fatigue brittle-to-ductile transition (FBDT) in semi-crystalline materials may be analyzed using irreversible work and the rate of crack propagation under fatigue to obtain the critical energy release rate for both damage mechanisms.
- Compared to the energy release rate obtained from potential energy measurements, the calculated critical energy rate is one order of magnitude smaller.

- For maximum fatigue loads between 20 and 35 % of the yield stress, the results show average critical energies of 211 J/m<sup>2</sup> and 695 J/m<sup>2</sup>, respectively for brittle and ductile regimes.

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