Time-Dependent Fracture Toughness Measure for Polyethylene

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SYNOPSIS

Because of their extraordinary cracking resistance, medium-density polyethylenes (MDPEs) do not fail in a brittle manner when tested with conventional fracture toughness procedures, but brittle fractures and J_{1c} values can be obtained for these materials by utilizing fatigue loading. However, because brittle fracture in polyethylene is a result of low stresses (energy) applied over long periods of time, and since J_{1c} neglects time, J_{1c} is incapable of differentiating MDPEs on the basis of their fracture resistance. Thus, the power to fracture method, which incorporates both energy and time, has been developed. During increments of crack growth, the product of the potential energy and the number of elapsed cycles is used to calculate the power to fracture. Within limits that assure a similar failure mechanism, the power to fracture for a particular resin is constant despite varied fatigue conditions. The power to fracture is capable of differentiating between resins on the basis of their brittle cracking resistance. © 1994 John Wiley & Sons, Inc.

INTRODUCTION

Load-bearing polymeric components are subject to a host of stresses that may vary in intensity and frequency over the lifetime of the structure. These varied stresses are applied for long periods of time and may initiate small brittle cracks from surface scratches or internal flaws. Eventually, these small cracks will propagate through the structure and produce a failure. These brittle cracks are characterized by very small damage zones at the crack tip and macroscopically flat fracture surfaces.

A material that displays small-scale yielding can be tested for resistance to such brittle (plane strain) crack propagation by using the American Society of Testing and Materials (ASTM) standard procedure E813—J_{1c}, A Measure of Fracture Toughness.[1] This method uses thick monotonically loaded specimens to determine the value of the J integral at crack initiation, J_{1c}. However, it is possible that differences in thickness between the test specimen and the actual structure may induce morphology variances, which may cause the fracture resistance of the test specimen to diverge from that of the component. In addition, some tough resins do not initiate a *brittle* crack under the conditions of ASTM E813.

This study discusses a method that overcomes both of these difficulties. Using fatigue, a test has been designed that permits the determination of J_{1c} from brittle crack propagation in thin specimens of tough medium-density polyethylene (MDPE). Yet, because J_{1c} neglects time, it was unable to detect differences in cracking resistance between different polyethylene resins. Thus, the power method of quantifying fracture toughness, which incorporates both energy and time, the factors inherent to brittle cracking of polyethylene, is proposed.

EXPERIMENTAL

Four-millimeter thick plaques were compression molded from four MDPE copolymer pipe resins and a high-density polyethylene (HDPE) homopolymer. While maintaining pressure on the mold, the platens were water cooled from 190 to 30°C at an average rate of 10°C/min. Single-edge notch tension spec-

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imens of 145 × 20 × 4 mm, with a 5-mm deep-pressed razor notch at midheight, were cut from the plaques.

These specimens were fatigue tested at ambient temperature on an MTS servohydraulic machine using a load control sinusoidal waveform. During testing of the method, experiments were conducted using σ_{max} values of 6.0, 6.7, and 7.5 MPa. Some tests were conducted using a frequency of 0.5 Hz, while others used a 1-Hz cycle. The 6.7-MPa level, which is about 30% of the material yield stress, was used to compare different resins. Stress levels near this percentage of the yield stress have previously been shown to produce brittle failures in reasonable testing times.[2]

Load–displacement hysteresis loops were recorded on an $x - y$ plotter, and crack length measurements were made using a traveling optical microscope.

RESULTS AND DISCUSSION

J_{1c} of Polyethylene

The fracture toughness of a polymer can sometimes be quantified by following the ASTM standard procedure E813.[1] This method involves monotonically loading thick specimens and approximating J by,[3]

$$J = \frac{2A}{Bb} \tag{1}$$

where A is the area under the load–displacement curve, B is the specimen thickness, and b is the uncracked ligament width.

For polymers that are too tough to initiate a brittle crack under monotonic loading, or when excessively thick specimens are required by the ASTM method, it may be beneficial to use the fatigue method of determining J_{1c}.[4] The fatigue method employs the exact energy definition of the J integral,[5]

$$J = \frac{-d\mathrm{PE}}{daB} \tag{2}$$

This definition states that J is proportional to the change in the potential energy ($-d$PE) with crack growth (da), where the potential energy is the negative of the area above the load-displacement curve.[5]

In order to compare the fatigue and ASTM method of determining J_{1c}, it was essential to study a material that produced brittle fracture in both tests. Since brittle fracture is not obtained in MDPE with the ASTM test, HDPE specimens were employed, and the equivalence of the two fracture toughness test methods was established.[4]

Even with very thick specimens, for some tough polymers, brittle cracks cannot be initiated using the monotonic loading method (ASTM). For example, Crist and Carr[6] have conducted J integral studies on thick compact-tension specimens of tough MDPE resins. Under the conditions specified by ASTM E813, these materials separate by a tearing process that is accompanied by a large damage zone; they do not *crack* under monotonic load. Since we are testing for resistance to plane strain brittle failure, a test method should be used that produces brittle fractures. The fatigue test can produce brittle failures in these tough materials, as well as provide J_{1c} values.

Fatigue lifetimes and past experience have shown medium-density copolymers to be much more resistant to fracture than high-density homopolymers. Yet, Table I shows that a higher J_{1c} value was obtained for the HDPE than for the MDPEs. In addition, independent tests have shown differences in crack propagation resistance within these MDPEs. These differences are not manifest by the J_{1c} values, which, due to experimental uncertainty (±0.5 kJ/m^2), are all approximately equivalent. A brief literature survey of polymeric ASTM J_{1c} testing confirms the difficulty of using J_{1c} to differentiate materials. This sampling shows most polymeric J_{1c} values are in the 1–10 kJ/m^2 range.[7–18] In some cases, lab-to-lab variability of these J_{1c} values can be extremely wide.[17,18] Thus, using J_{1c} by itself to separate polymers of varying fracture toughness may be difficult.

Time and Energy

As stated earlier, if polymeric structures develop brittle cracks, they are usually a result of the structure being exposed to a variety of low stresses applied

Table I Results of Fatigue Crack Propagation Tests

Material	J_{1c} (kJ/m^2)	Power (GPa/s)	N_p (Cycles)
HDPE-homopolymer	2.2	0.2	6,000
Ethylene/hexene 1	1.0	3.2	50,000
Ethylene/methylpentene	1.7	4.5	110,000
Ethylene/hexene 2	1.6	6.8	150,000
Ethylene/butene	1.6	8.0	230,000

N_p = number of cycles during crack propagation.

over a long time period. During this time, damage zone growth processes occur even prior to crack initiation and during crack arrest. This time-dependent damage growth consumes energy and affects the overall fracture process. However, J_{1c} only quantifies the change in potential energy associated with crack growth. Without crack growth there cannot be a J_{1c}, yet there can be time-dependent damage accumulation and energy dissipation. Thus, a more sensible, and hopefully more sensitive, measure of fracture toughness will sum this energy dissipation, which occurs over time.

The need to incorporate time in fracture toughness tests of viscoelastic materials has been displayed before. During a crack layer analysis of two different MDPE pipes, it was demonstrated that both pipes develop nearly equivalent amounts of damage.[2] From the crack layer theory, J is equivalent to the size of this damage (R) multiplied by a material constant, the specific enthalpy of damage (γ^*). Despite the fact that the lifetimes of the two pipes differed by a factor of 8, their specific enthalpies of damage only differed by 20%; J did not give a good indication of the difference in material toughness. This shortcoming was caused by differences in the damage accumulation rate. Under identical conditions, damage evolution, and hence the subsequent crack growth, was much slower in the tough specimen than it was in the more fracture prone specimen. This observation underscores the need to incorporate rate effects in polymeric fracture tests.

Rate effects have been accounted for in creep crack growth of metal alloys by employing the energy rate line integral, C^*. This parameter is a measure of the stress–strain rate fields at the crack tip and is analogous to the J integral. Like the J integral, simplified methods to estimate C^* have been developed to circumvent the rigorous data reduction scheme originally required to calculate this parameter.[19,20] C^* shows a logarithmic relation to the creep crack growth rate and the displacement rate. While some authors have shown geometry independence of C^*,[21,22] others have noted that in more ductile materials, or for geometries that offer less crack tip plastic constraint, a load and geometry dependence exists as a result of large-scale specimen creep.[23,24] Although the energy rate line integral does characterize the creep crack growth rate in many materials, it seems incapable of differentiating between materials on the basis of chemistry or microstructure.[22,25] Like C^*, the power to fracture is measured in units of energy/area × time. Both measures, C^* for creep and the power to fracture in fatigue, are attempts to quantify the rate of energy dissipation at the crack tip.

Power to Fracture

From the above discussion, it seems clear that a quality fracture toughness test should produce brittle failures in tough materials and yield a time-inclusive measure of toughness that is capable of differentiating resins on the basis of their fracture resistance. In answer to these requirements, the power to fracture technique has been developed. This method is a fatigue-based test, because of the ease of producing brittle fatigue fractures in tough materials and thin specimens. Time and energy are measured during crack growth and combined to produce the power to fracture, which is a summation of the energy evolution that occurred during the lifetime of the crack.

If the power to fracture is to meet the criteria listed above, it must be independent of the rate of fatigue crack propagation. This rate is determined by factors such as the maximum stress applied during the fatigue cycle, as well as the frequency of the cycle. Thus, to check the rate independence of this new fracture toughness method, the power to fracture of one MDPE resin was tested under four different fatigue conditions. A plot of the number of fatigue cycles versus crack length (Fig. 1) shows the effect of the varied conditions on the kinetics of crack propagation. The four experiments plotted in Figure 1 were conducted using three different maximum stress levels, two unequal frequencies, and various notch depths. Obviously, these major differences create very different crack propagation kinetics.

The extremely different fatigue conditions of the four experiments also resulted in different load versus displacement hysteresis loops. Since higher maximum stress levels result in larger hysteresis loops, large values of the negative potential energy per cycle (area above the loading curve of the load–displacement hysteresis loop) were obtained for the experiments run at higher stresses (Fig. 2).

Figure 2 also shows no difference in negative potential energy between the experiments run at the same load level but two different frequencies. The low frequencies (0.5 and 1 Hz) were used to minimize crack tip heating effects and thus maintain the same failure mechanism under both conditions.

Since it is conceivable to break the entire crack propagation into shorter crack growth increments, it is possible to determine the number of cycles elapsed (ΔN) as the crack grows from a_1 to a_2 (Fig. 1). (In this investigation the increments were chosen

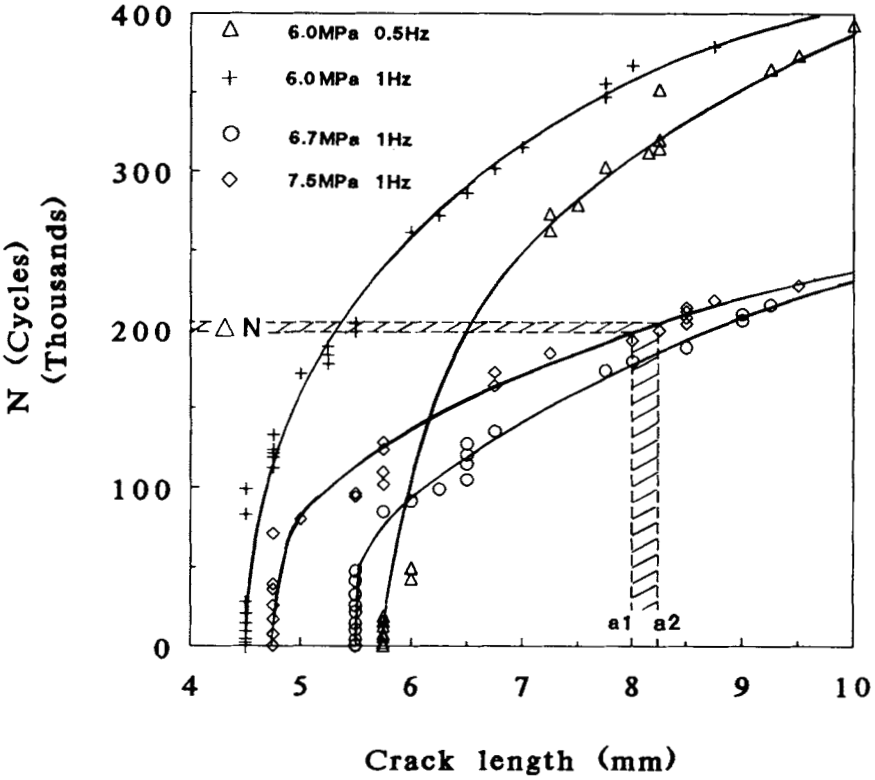

Figure 1 Number of cycles vs. crack length for four different test conditions. ΔN is the number of cycles required for the crack to propagate from a_1 to a_2.

to be 0.25 mm long: $a_1 + 0.25$ mm $= a_2$.) In a similar manner, the average of the negative potential energy per cycle ($-PE_{avg}$) during that crack growth period can be determined (Fig. 2). The power to fracture is then calculated for each increment of crack growth by multiplying $-PE_{avg}$ by ΔN and by the fatigue frequency of the test, while dividing by the length of the crack growth increment (0.25 mm) and the specimen width (4 mm). Despite the variety of fatigue conditions, Figure 3 shows that the evolution of the power to fracture is relatively constant. All four conditions reduce to a single narrow "power band."

This power band was only calculated in the brittle crack propagation region. The ductile contribution to fracture, which is proportional to the roughness of the fracture surface, increases as the crack propagates (Fig. 4). This gradual brittle to ductile transition occurs near a crack length of 10 mm ($a/w = 0.5$). This investigation is an attempt to quantify brittle fracture. Since after such a transition the failure process is one of ductile tearing, rather than brittle fracture, the region beyond a 10-mm crack length has not been included in the analysis.

Using the Power to Fracture to Differentiate MDPEs

Within our chosen test conditions, which were selected to maintain the same failure mechanism, the power to fracture has been shown to be independent of several testing variables (Fig. 3). This independence may imply that the power to fracture is a measure of the intrinsic fracture resistance of the material. In order to examine if the power to fracture is indicative of a material's resistance to fracture, the power analysis has been applied to four different MDPEs and a HDPE tested under the same condition ($\sigma_{max} = 6.7$ MPa, $\nu = 1$ Hz). Figure 5 shows that material differentiation is possible with the power analysis. The resins that are most resistant to brittle cracking display the steepest power curves.

Rather than report the entire curve, it would be advantageous to report a single fracture toughness value for each material. That value was chosen as the power to fracture at a 5-mm crack length. This value, the power, as well as J_{1c} and the number of cycles during crack propagation are reported in Table I. The most fracture-resistant materials are also

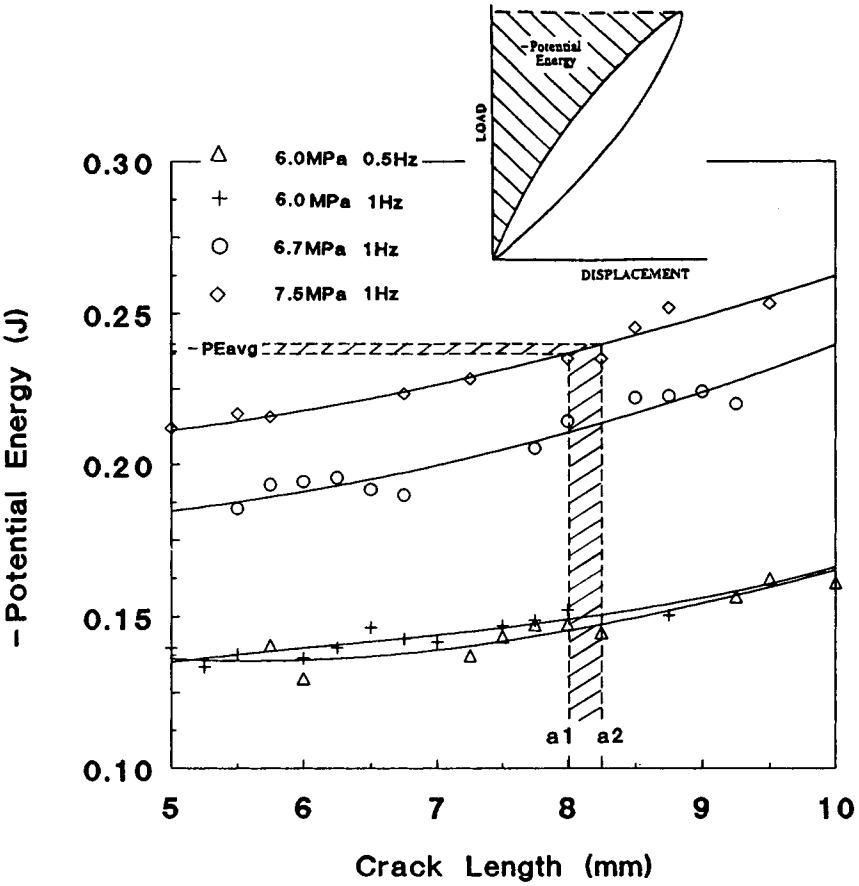

Figure 2 Potential energy vs. crack length for four different test conditions. The potential energy is proportional to the area above the loading curve of the load–displacement hysterisis loop. PE_{avg} is the average potential energy per cycle during crack propagation from a_1 to a_2.

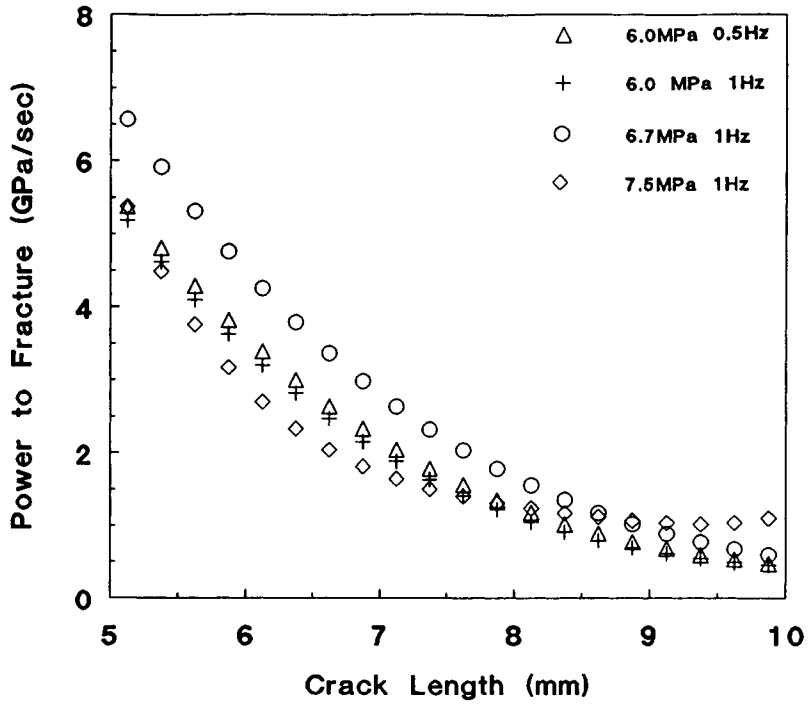

Figure 3 Power to fracture vs. crack length for four different test conditions.

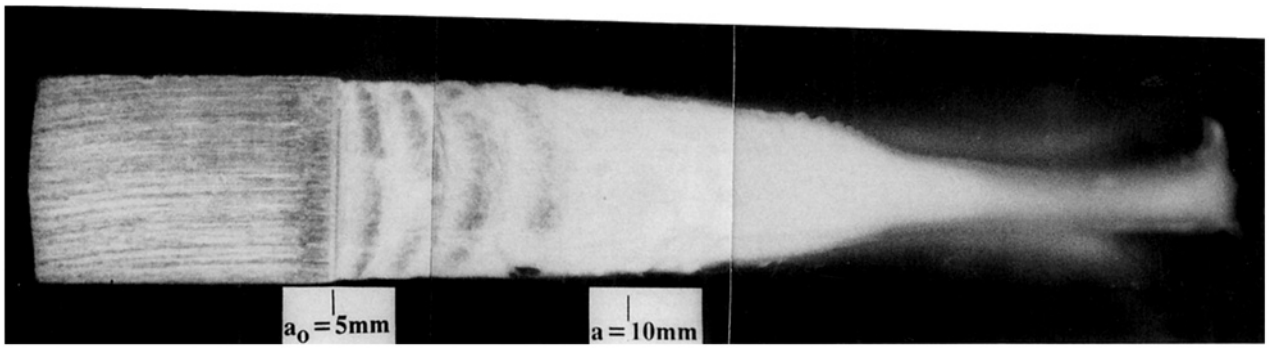

Figure 4 Fracture surface of a failed specimen showing the notch and the brittle and ductile failure regions.

the ones with the highest power values. Figure 5 also shows that, despite the variance in fatigue conditions for the testing of the eth/hexene 2 sample, the resulting power band is still narrow enough to permit material differentiation.

Of course, this method does have limitations. In each experiment discussed above the crack propagation mechanism was the same. Brittle discontinuous crack growth occurred up to a crack length of about 10 mm (a/w = 0.5), after which the failure

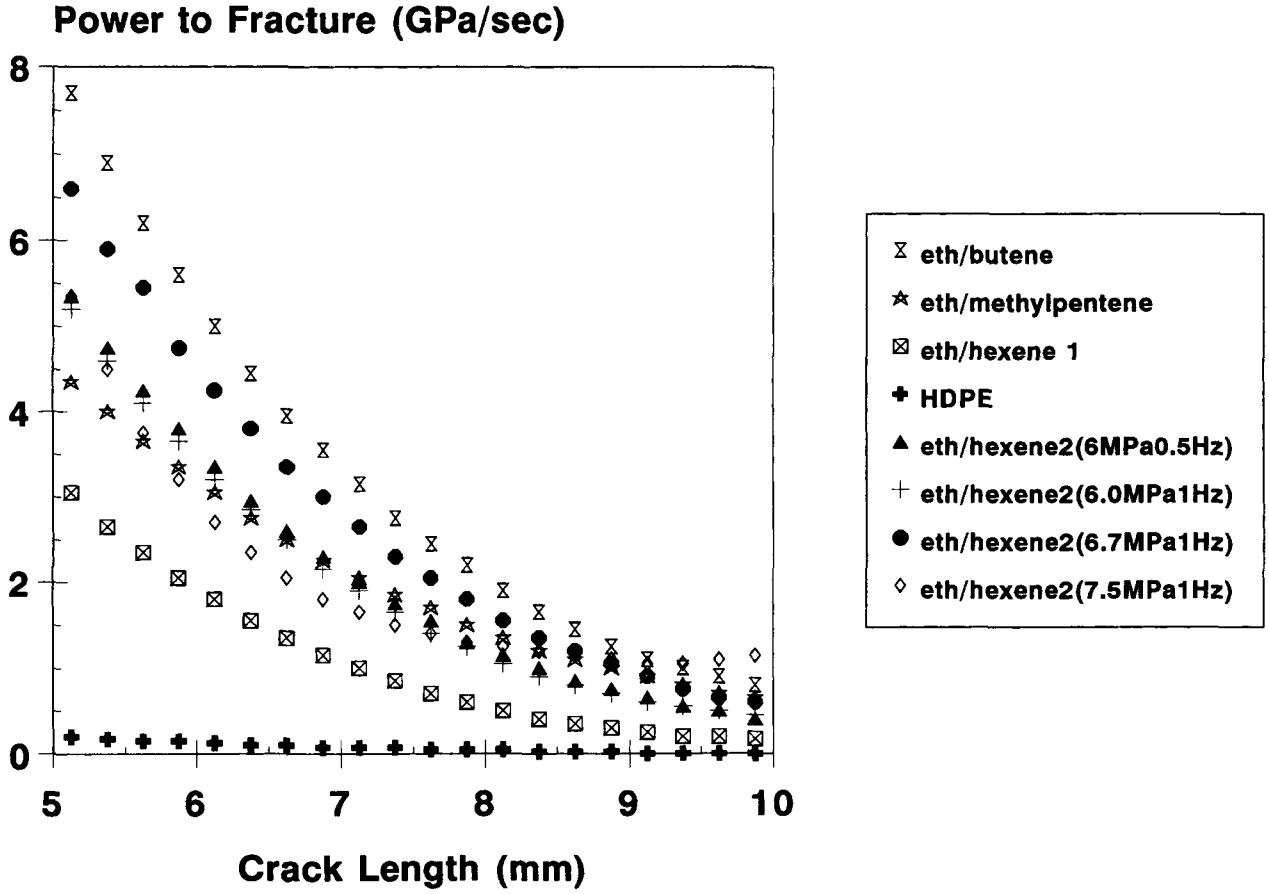

Figure 5 Power to fracture vs. crack length for five types of polyethylenes tested under the same condition, as well as for the one polyethylene tested under four different conditions (Fig. 3).

became ductile. In a preliminary experiment, the test was conducted at a very high stress (8.4 MPa). This condition produced mostly ductile crack propagation and a noticeably more curved power versus crack length behavior. Crack tip heating from very high frequencies may also alter the failure mechanism and hence the power analysis. Thus, the variability in fatigue conditions is limited to a region in which the same failure mechanism is present in each specimen. Obviously, the most appropriate loading conditions are dependent on the class of material being tested.

Although the development of this power to fracture concept is still in its fledgling state, the results are extremely promising. Brittle cracking in polyethylene is a result of the material being exposed to low stresses for long periods of time. Techniques that only measure energy associated with fracture (J_{1c}) neglect the rate of the fracture processes and thus are insufficient to distinguish differences in brittle crack propagation resistance between tough polyethylene resins. However, by incorporating both the time effect and the energy in a measure of the power to fracture, MDPE resins can be differentiated on the basis of their cracking resistance.

CONCLUSIONS

Using the fatigue method of determining J_{1c}, brittle cracks were propagated in tough MDPE pipe resins, which did not crack in a brittle manner when tested using the monotonically loaded bending method (ASTM). Material differentiation with J_{1c} was not possible because J_{1c} neglects the rate of processes leading to fracture (time). To overcome this impediment, a method to measure the power to fracture, which incorporates both energy and time has been developed. The power to fracture is proportional to the product of the average potential energy and the number of elapsed cycles per increment of crack growth. The resulting power curve is constant despite varied fatigue conditions, and it is capable of differentiating polyethylene resins on the basis of their brittle cracking resistance.

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