The relationship of the initiation stage to the rate of slow crack growth in linear polyethylene

XICI LU*, NORMAN BROWN
Department of Materials Science and Engineering and Laboratory for Research on Structure of Matter, University of Pennsylvania, Philadelphia, Pennsylvania 19104, USA

The rate of notch opening was observed in single-edge notched tension specimens of linear high-density polyethylene under plane strain conditions as a function of applied stress and notch depth. The initial rate of notch opening was $22K^{4.3}$ μm min^{-1} (where units of K are MN m$^{-3/2}$). The initial rate of notch opening was constant until the opening at its root was 25 to 30 μm for all stresses and notch depths. The accelerating part of the notch opening against time curve and the time to failure could be predicted from the initial rate of notch opening. It is concluded that the same mechanism governs the initiation stage and the subsequent crack growth rate.

1. Introduction

This paper is concerned with the initiation of long-time brittle fracture of polyethylene (PE). The initiation is important because the initiation stage consumes a significant portion of the total time to failure. This paper is restricted to the case of plane strain fracture which is generally applicable to an engineering structure such as a gas pipe. In order to study the initiation stage, it is necessary to observe the interior of the specimen because the surface growth rate is different from that of the interior. In this investigation the initiation was observed by looking into the opening of the initial surface notch from which the damage zone emanates. The dependence of the initial rate of notch opening on stress and on the depth of the original notch was determined, and it was found that there was a good correlation between the stress intensity and the subsequent crack growth rate.

A practical consequence of this research is the finding that the time for ultimate failure can be predicted from short-time data based on the initiation process.

2. Experimental procedure

The PE was the same unpigmented commercial resin used in previous investigations of crack growth [1–3]. It was Marlex 6006 which was supplied and compression moulded by the Phillips Chemical Co, (Oklahoma) in accordance with ASTM D1928 and cooled at 15° C min^{-1}. The moulded plaques were 4 mm thick with melt index 0.75, M_n = 19 600 and M_w = 130 000 and a density of 0.964 g cm^{-3}. The PE had a yield point of 25 MPa at 300 K and at a strain rate of 0.02 min^{-1}.

The specimens were notched with a razor blade to a depth of 0.47 to 0.15 mm. As described in a previous paper [2] the notching was carefully controlled by using a fresh razor blade for each notch and using a notching speed of 0.05 mm min^{-1}. The notching technique produced negligible damage as checked by our microscopic methods.

The tensile specimens were 4 mm thick, 18 mm wide and the distance between the grips were 25 mm. The single shallow surface notch ran across the width of the specimen (Fig. 1).

Measurements of the notch opening were made at 80 × magnification using a filar eyepiece. The opening of the notch at the surface of the specimen and its root, the crack opening displacement (COD) (Fig. 2a), were measured as a function of time. The error in the measurement of the opening of the notch was about ± 3 μm.

The specimen was exposed to a constant load in a beam-loading device with a 5 : 1 lever ratio at a temperature of 30 ± 0.5° C. The applied stress was kept below one-half the yield point because previous work [2] had shown that stresses above one-half the yield point produce balloon-shaped deformation zones instead of the planar zone of damage which is characteristic of long-time brittle failure in PE.

Fibrils were photographed with the optical microscope while the specimen was under load. However, the best pictures of the fibrils were obtained with the SEM by opening the notch in a special jig and reproducing the notch opening that existed during the test.

3. Results

An optical view of the bottom of the notch during its measurement is shown in Fig. 2b and a corresponding view with the SEM is shown in Fig. 2c. Fig. 3 shows the notch openings at the surface of the specimen and at the root of the notch against time, along with the overall creep in the specimen. Several features of the notch-opening curves are to be noted: (i) there is an instantaneous value at t = 0; (ii) the initial rate of opening is constant followed by an acceleration to

*Visiting Scholar from the University of Science and Technology of China, Hefei, China.

0022–2461/86 \$03.00 + .12

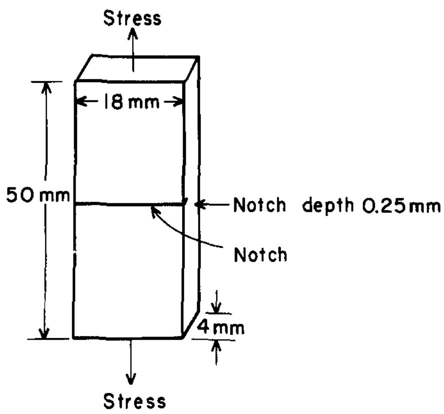

Figure 1 Single-surface notched tensile specimen.

ultimate failure, and (iii) the difference between the notch opening at the surface and the COD changes slowly during most of the lifetime of the specimen. The scatter in the measurement of the initial rate of notch opening was $\pm 30\%$ for duplicate conditions.

The instantaneous value of the COD, δ_0, is about equal to that predicted from the Dugdale model [4] where

$$\delta_0 = K^2/\sigma_y E^* \quad (1)$$

Here K is the stress intensity, σ_y is the yield point, and for plane strain $E^* = E/(1 - \nu^2)$ where ν is Poisson's ratio; $E = 1$ GPa, $\sigma_y = 25$ MPa and $\nu = 0.35$. K is given [5] by

$$K = 1.12\pi^{1/2}\sigma a_0^{1/2} \quad (2)$$

where σ is stress and a_0 the initial notch depth.

Knowing the difference between the opening at the surface and at the root of the notch and the depth of the notch, the notch angle can be calculated. From Fig. 3, the notch angle is initially 7° and then increases to about 7.5° at 200 min and then to 9° after three-fourths of the life of the specimen. Thus, during most of the time the notch surfaces remain nearly parallel to themselves. The initial notch angle was calculated for all the experiments and most angles were about 10°. There was a variation from about 7 to 15° which did not correlate with stress or notch depth.

In these experiments the maximum applied stress was less than one-half the yield point so that the creep rate was always decreasing and the total overall creep strain was less than about 6% at failure. The creep strain for general yield was about 12%.

The opening at the root of the original notch, δ, against time is shown in Fig. 4 for various applied stresses for a constant notch depth of about 0.24 mm. It is seen that the instantaneous value of δ increases with stress as expected from the Dugdale model. The initial slope also increases with stress as expected. The beginning of the acceleration stage occurs at about the same COD, which was about 25 to 30 μm.

Fig. 5 shows δ against time for different notch depths and a constant stress of 10 MPa. Again the critical value of COD, δ_c, at which the curves begin to accelerate is equal to 25 to 30 μm. This value of δ_c was the same for the range of stress, 8 to 12 MPa, used in this investigation and for notch depths ranging from 0.15 to 0.47 mm.

The data were analysed on the basis of the assumption that the initial slope of the δ against time curve, $\dot{\delta}_0$, depended on stress and the initial notch depth, a_0, as follows:

$$\dot{\delta}_0 = C\sigma^n a_0^m \quad (3)$$

where C, n and m are constants. It was found that the average value of n was 4.4 and the average value of m was 1.9. The correlation coefficients for the linear plots of $\log \dot{\delta}_0$ against $\log \sigma$ with a_0 held constant, and $\log \dot{\delta}_0$ against $\log a_0$ with σ held constant, were about 0.95 (Figs. 6 and 7). Finally all the data were plotted as $\log \dot{\delta}_0$ against $\log K$ (Fig. 8), where for single-edge notch tension the resulting relationship is

$$\dot{\delta}_0 = 22K^{4.3}\,\mu\text{m min}^{-1} \quad (4)$$

with a correlation coefficient of 0.94 for the log–log plot; K has units of MN m$^{-3/2}$.

The length of the damaged zone was viewed with

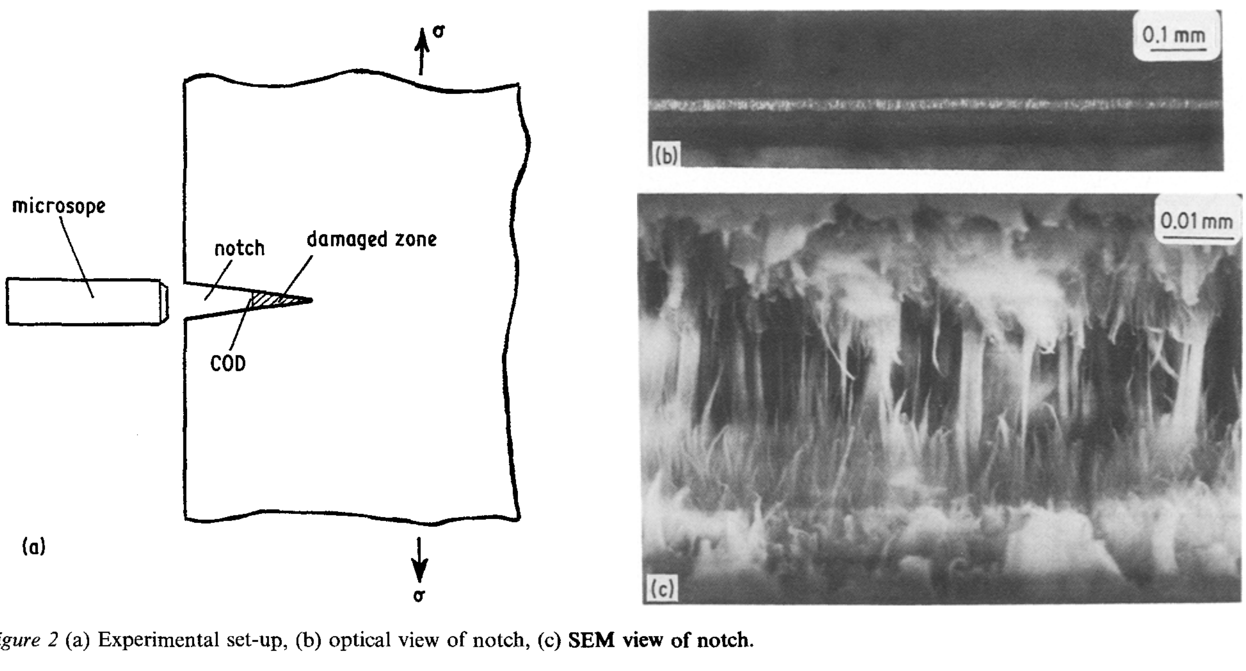

Figure 2 (a) Experimental set-up, (b) optical view of notch, (c) **SEM view of notch.**

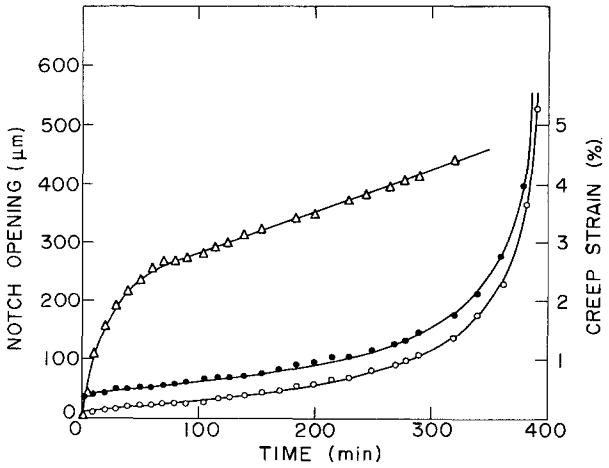

Figure 3 (△) Overall creep strain, (●) notch opening at the surface and (○) notch opening at root, plotted against time. a_0 = 0.246 mm, stress = 10 MPa, temperature = 30° C.

the SEM after slicing open the interior of the specimen as shown in Fig. 9. It was found that the length of the damage zone could be predicted from the calculated value of the notch angle based on data such as those in Fig. 3. The leading tip of the damaged zone is close to the intersection of the planes of the notch surface.

As the notch opened, its root was viewed optically as a function of time. It was found that fibrils began to fracture during the linear region of the δ against t curve. Complete fracture at the root of the original notch was usually observed about the time that the δ against t curve began to accelerate.

It is seen from Fig. 9b that the first evidence of damage consists of microcrazes. The fibrils within these microcrazes are much finer than the coarse fibrillation that is produced after the microcrazes interact. The fine cracks perpendicular to the notch

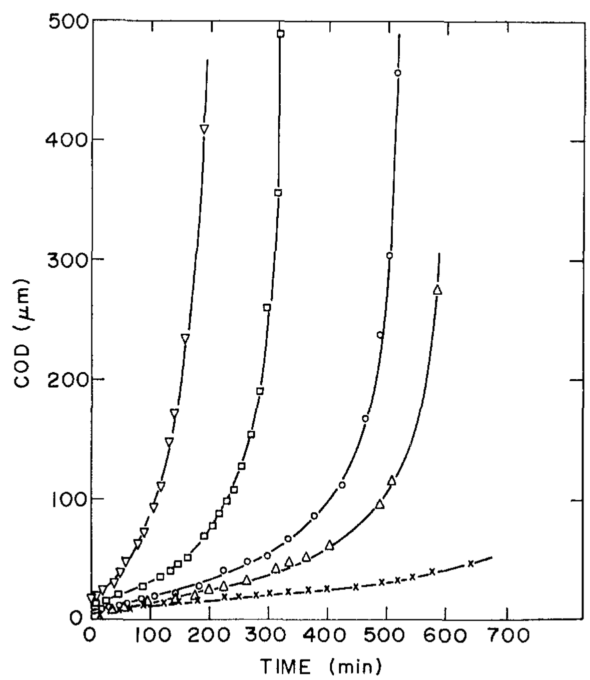

Figure 4 Opening of initial notch at the root, COD, against time for various stresses at a nearly constant notch depth of 0.24 mm at 30° C. Values of stress and a_0 as follows: (×) 8 MPa, 0.228 mm; (△) 9 MPa, 0.253 mm; (○) 10 MPa, 0.242 mm; (□) 11 MPa, 0.240 mm; (▽) 12 MPa, 0.241 mm.

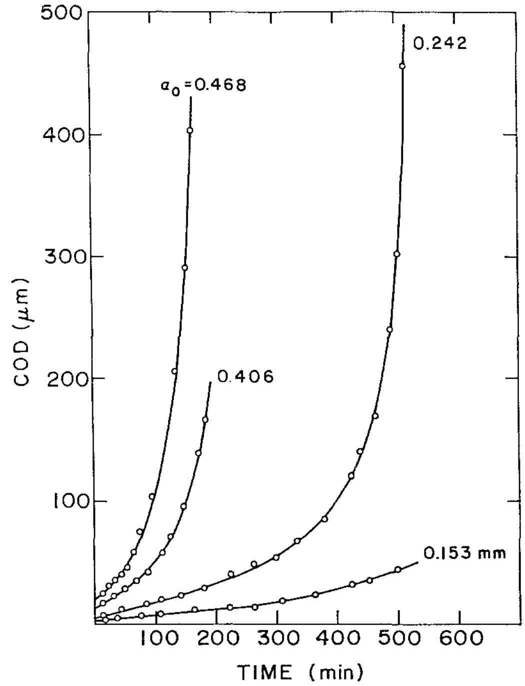

Figure 5 Same as Fig. 4 except for various notch depths and a constant stress of 10 MPa.

are artefacts; they are cracks in the metal coating from heating by the SEM beam. Further microstructural details concerning the transformation from microcrazing to coarse fibrillation will be presented in another paper.

4. Discussion

4.1. Geometry of damaged zone

The damaged zone was found to have a nearly triangular shape. The length of the damaged zone, Δa,

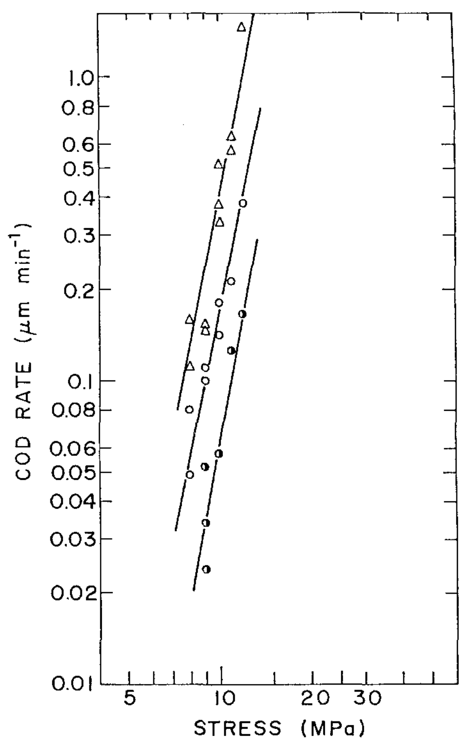

Figure 6 Initial notch opening rate $\dot{\delta}_0$ against stress for constant notch depth = 0.24 mm. a_0 = (◑) 0.15 mm, (○) 0.24 mm, (△) 0.40 mm.

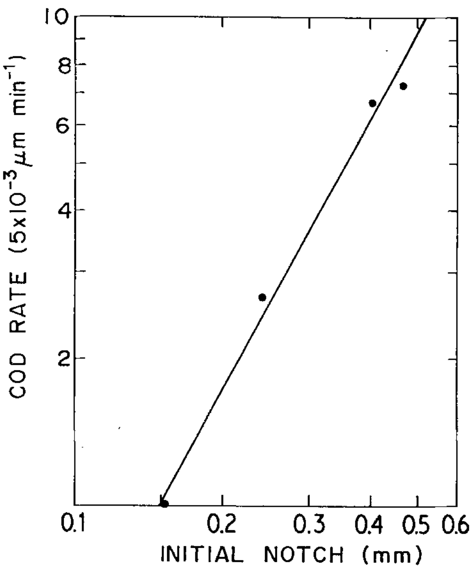

Figure 7 δ_0 against notch depth for constant stress = 10 MPa. $\delta_0 = 1.6a_0^{1.81}\ \mu\text{m min}^{-1}$.

and the COD, δ, are related by the equation

$$\delta = \alpha \Delta a \tag{5}$$

where α is the apex angle of the damaged zone. It is also observed from data such as in Fig. 3 that the notch angle, α_N, is given by

$$\tan(\alpha_N/2) = (\delta_s - \delta)/2a_0 \tag{6}$$

where δ_s is the opening of the notch at the surface. Thus, for example, from Fig. 3, α_N can be calculated and was found to be initially 7° and it increased slowly with time. In a previous investigation Δa was measured directly as shown in Fig. 10 under the same test conditions as used for Fig. 3. The corresponding values in time of δ and Δa are plotted against each other in Fig. 11, from which α is obtained from the initial slope of the curve and found to be 7°. The value of δ in Fig. 11 when $\Delta a = 0$ corresponds to the thickness of the tip of the razor blade which made the initial notch. Micrographs such as Fig. 9 show that α_N is somewhat less than α, but tends to approach it as Δa includes more of the crack than of the deformation zone. These results show that the length of the damaged zone can be obtained from measurements of the notch opening, without having to cut open each specimen after a given time as was done in previous investigations.

4.2. Kinetics of the initiation rate.

The initiation process for crack growth is exhibited by the initial part of the δ against t curves in Figs 3 to 5.

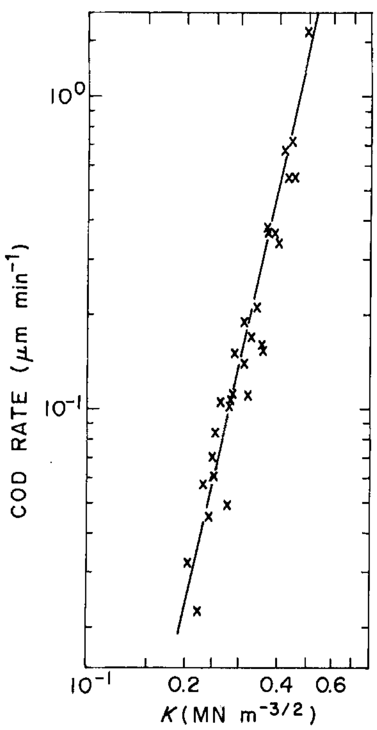

Figure 8 δ_0 against stress intensity.

The size of the instantaneous damage at $t = 0$ is closely predicted by the Dugdale model. The Dugdale model is independent of time, but it is of interest to determine how the shape of the time-dependent deformation zone such as in Fig. 9 compares with the Dugdale prediction. The thickness of the deformed zone at the root of the notch is given by Equation 1 and its length, Δa, is given by

$$\Delta a = \frac{\pi}{8}\frac{K^2}{\sigma_y^2} \tag{7}$$

$$\frac{\Delta a}{\delta} = \frac{\pi}{8}\frac{E}{(1 - \nu^2)\sigma_y} \tag{8}$$

Using previous values of E, ν and σ_y, $\Delta a/\delta = 17$ and the experimental value from Fig. 9 is 4. This difference may be caused by the stress distribution along the deformation zone not being constant as is assumed in the Dugdale model. From Equation 7 the initial values of Δa should be 29 μm, whereas after 10 min at 12 MPa the observed value was 62 μm. this increase in Δa could be interpreted in terms of a time-dependent yield point that after 10 min reduced to 68% of the initial yield point.

The zone of damage initially grows at a constant velocity. Fibril fracture is observed during the constant

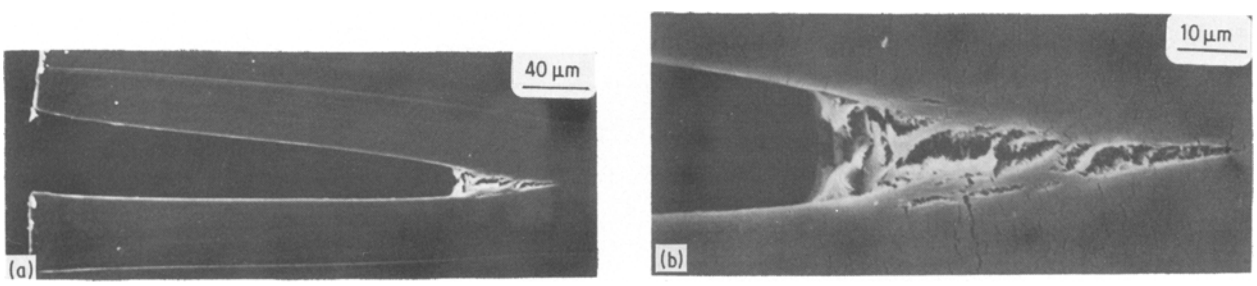

Figure 9 **(a), (b) Scanning electron micrographs of interior surface of specimen with same notch opening as existed during the test after 10 min at 12 MPa. The fine cracks perpendicular to the notch are cracks in the metal coating caused by beam heating in the SEM.**

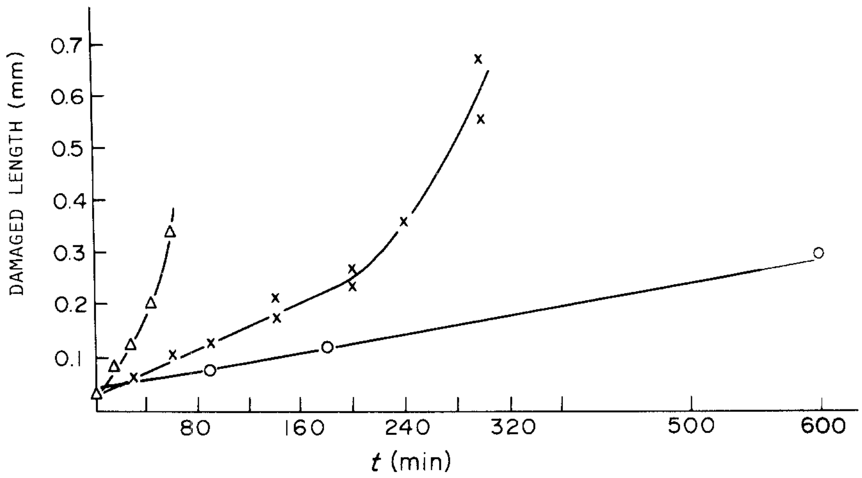

Figure 10 Length of damaged zone against time at various temperatures: (O) 25° C, (×) 30° C, (Δ) 40° C. Stress = 10 MPa, notch = 0.25 mm, K = 0.3 MPa m$^{1/2}$.

velocity part of the curve. Complete fracture at the root of the notch is observed about the time that the curve begins to accelerate. It is surprising that the initial part of the δ against t plot is linear in the light of the complex changes in morphology that take place and the fact that $1/\sigma_y$ does not change linearly with time.

The dependence of $\dot{\delta}_0$, the initial rate of damage, on stress and notch depth does not exactly agree with a simple K dependence, but the agreement is sufficiently close (Fig. 8) for most practical considerations. This K dependence may be limited to the specimen design of Fig. 1 under simple tension and for the ranges of stress and notch depths used in this investigation. Current research indicates that three-point bending with the same specimen design gives an appreciably lower rate of initiation than simple tension for the same value of K.

4.3. Relationship of initiation to crack growth

Why $\dot{\delta}_0$ should depend on $K^{4.3}$ is not clear. Chan and Williams [6] reported that the crack growth rate $\dot{a}$ varied as K^4 in the same PE as used in this investigation. Considering the combined scatter in the data of Chan and Williams and in ours, a difference in the exponent between their value of 4 and our value of 4.3 may not be significant.

From data such as Fig. 10, the initial rate of damage was plotted against $1/T$. An activation energy of 97 kJ mol^{-1} was obtained. Chan and Williams [6] measured an activation energy for crack growth rate of 107 kJ mol^{-1}. Both these values are practically the same. Thus, since the initial rate of damage and crack growth rate have the same K dependence and activation energy it is strongly suggested that both processess have the same underlying mechanism. Thus, it is expected that the curves in Figs. 4 and 5 which included the initiation stage and crack growth to failure can be described by a common equation as given below.

We start with the observation by Chan and Williams [6] that the crack growth rate is given by

$$\dot{a} = C\sigma^4 a^2 \qquad (9)$$

where C is a constant for a given temperature, specimen geometry, loading condition and material. We now transform a to δ in accordance with the results of this investigation, to obtain

$$a = a_0 + \delta/\alpha \qquad (10)$$

$$\dot{a} = \dot{\delta}/\alpha \qquad (11)$$

Since we have observed that the rate of initiation of crack growth, $\dot{\delta}_0$, also follows Equation 9 we combine Equations 9, 10 and 11 to give

$$\frac{\dot{\delta}}{\dot{\delta}_0} = \left(\frac{a_0 + \delta/\alpha}{a_0 + \delta_c/\alpha}\right)^2 \qquad (12)$$

where δ_c is the value of δ at the beginning of the acceleration stage; it was found to be 25 to 30 μm for all stresses and notch depths used in this investigation. The values of $\dot{\delta}$, $\dot{\delta}_0$, a_0, α and δ were obtained from eleven curves such as those in Figs. 3 to 5, and the value 27 μm was used for δ_c. About eight values of $\dot{\delta}$ were calculated from each curve. Fig. 12 shows the log–log plot of Equation 12 using these data. The slope of the curve is 2 and all the data points fall on a single curve as predicted by Equation 12. This results very strongly supports the conclusion that the same mechanism controls the initiation stage and subsequent crack growth in this material.

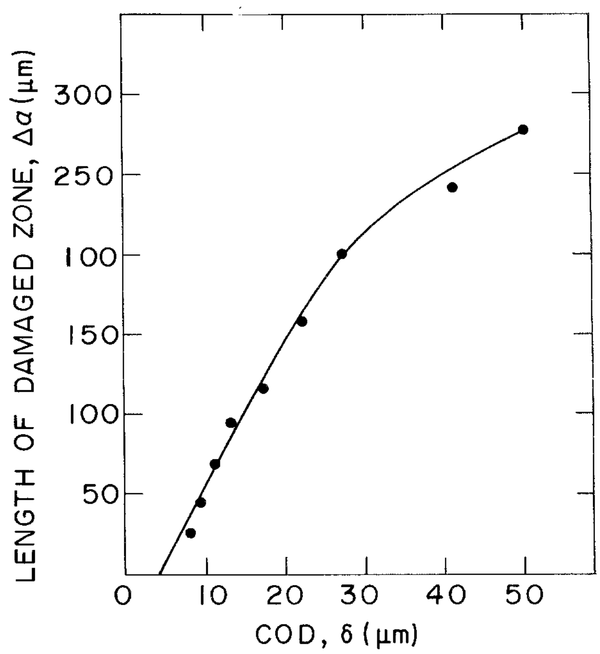

Figure 11 Length of damaged zone against opening at root of initial notch.

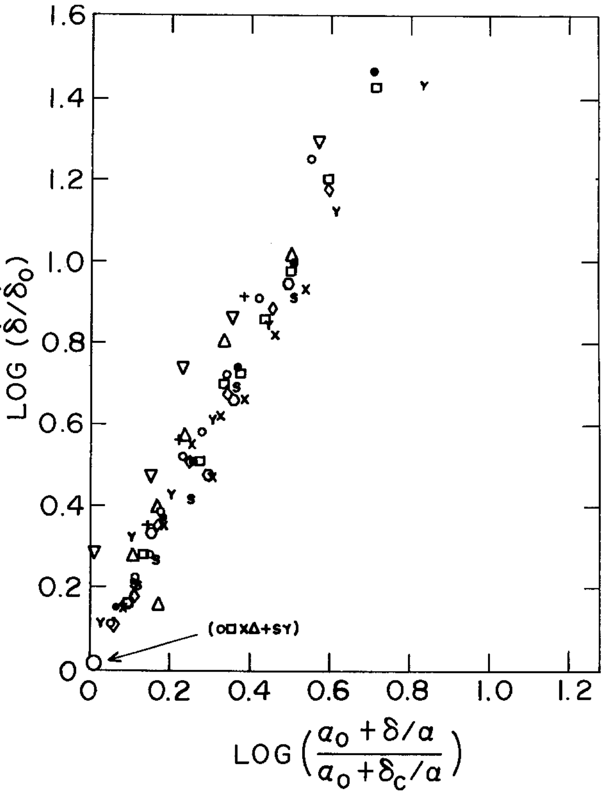

Figure 12 Plot of Equation 12. Values of stress and a_0 as follows: (●) 9 MPa, 253 μm; (o) 10 MPa, 242 μm; (□) 11 MPa, 240 μm; (×) 12 MPa, 241 μm; (△) 8 MPa, 416 μm; (▽) 9 MPa, 406 μm; (+) 10 MPa, 406 μm; (◯) 11 MPa, 407 μm; (S) 12 MPa, 419 μm; (◇) 11 MPa, 135 μm; (Y) 12 MPa, 143 μm.

4.4. Predicting failure

It is very useful to be able to predict ultimate fracture from short-time test data. Using Equation 12 as a basis, a simple predictive equation will be presented. A solution to Equation 12 is

$$t - t_c = \frac{(\alpha a_0 + \delta_c)^2}{\dot\delta_0}\left(\frac{1}{\alpha a_0 + \delta_c} - \frac{1}{\alpha a_0 + \delta}\right) \quad (13)$$

where $t = t_c$ when $\delta = \delta_c$ and $t_c = (\delta_c - \delta_0)/\dot\delta_0$. t_f, the time to failure, occurs when the length of the crack reaches the critical value, $(a_0 + \delta_f/\alpha)$, to produce rapid fracture as in the Griffith theory. Thus

$$t_f = \frac{\alpha a_0 + \delta_c}{\dot\delta_0}\left(1 - \frac{\alpha a_0 + \delta_c}{\alpha a_0 + \delta_f}\right) + \frac{\delta_c - \delta_0}{\dot\delta_0} \quad (14)$$

Based on the values for α, δ_c and $\dot\delta_0$ and estimating that $\delta_f \approx 1$ mm, t_f is approximated by

$$t_f \approx \frac{\alpha a_0 + \delta_c}{\dot\delta_0} \quad (15)$$

Table I shows how the predicted values for t_f (Equation 15) compare with the experimental observations. The agreement is within about ±30% and should be considered to be very good, since the scatter in the experimental values is also about ±30%.

5. Conclusions

1. The length of the damaged zone during the initiation of crack growth can be determined from measurements of the notch opening.
2. The notch angle is about 10° during the initiation stage and remains nearly constant as the damaged zone grows.
3. The COD against time curve is linear until the COD becomes 25 to 30 μm.
4. The initial rate of notch opening depends on stress and notch depth as $\dot\delta_0 = C\sigma^{4.4}a_0^{1.9}$.
5. The relationship with stress intensity is well approximated by $\dot\delta_0 = 22K^{4.3}$ μm min^{-1}.
6. The initiation stage and crack growth rate are based on the same underlying mechanism.
7. Time to ultimate failure can be predicted from experimental parameters which govern the initiation stage for crack growth.

Acknowledgements

The research was sponsored by the Department of Energy. Use was made of the Central Facilities of the Materials Research Laboratory at the University of Pennsylvania which are supported by the National Science Foundation. We appreciate the technical assistance of Mr N. T. Brown. Stimulation for the research was provided by the general research programme of the Gas Research Institute for the purpose of enhancing the utilization of polyethylene pipelines.

References

1. S. K. BHATTACHARYA and N. BROWN, *J. Mater. Sci.* **19** (1984) 2519.
2. *Idem, ibid.* **20** (1985) 2767.

TABLE I Comparison of theoretical and experimental times for failure*

a_0(μm)	σ(MPa)	α(deg)	$\dot\delta_0$(μm min^{-1})	t_f(theo)(min)	t_f(exp)(min)	t_f(theor)/t_f(exp)
253	9	7.2	0.11	560	600	0.93
242	10	12.3	0.13	610	515	1.17
240	11	10	0.27	260	310	0.84
241	12	11	0.47	160	195	0.80
135	11	12.9	0.092	630	615	1.02
143	12	12.2	0.148	390	510	0.76
412	8	9	0.146	640	440	1.46
386	9	9	0.142	620	350	1.77
406	10	8.8	0.33	270	240	1.13
407	11	8.8	0.59	150	150	1.01
419	12	10.2	1.11	90	100	0.92
						average = 1.07 ± 0.30

*$t_f = (\alpha a_0 + \delta_c)/\dot\delta_0$ where $\delta_c = 27$ μm, $\dot\delta_0$ = initial slope of δ against t, α = notch angle and a_0 = initial notch depth.

3. *Idem, ibid.* **20** (1985) 4553.
4. J. K. KNOTT, "Fundamentals of Fracture Mechanics" (Butterworths, London, 1973) Ch. 3.
5. P. C. PARIS and C. G. SIH, ASTM Technical Publication No. 381 (American Society for Testing and Materials, Philadelphia, 1965) p. 30.
6. M. K. V. CHAN and J. G. WILLIAMS, *Polymer* **24** (1983) 234.

Received 26 July
and accepted 18 September 1985