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Notchology–the effect of the notching method on the slow crack growth failure in a tough polyethylene

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The effect of various notching methods on the life time of slow crack failure at 80 °C for a tough gas pipe grade of polyethylene was investigated. The standard notching procedure involved pressing an ordinary razor blade into the single-edge notched tensile specimen at a rate of 50 μm min^{-1} at room temperature. The other notching methods involved using a sharper razor blade, cooling in liquid nitrogen, pre-cracking by fatigue, slicing with a scalpel, or using a rotary cutter. The standard procedure gave a life time of 28 000 min under a stress of 2.4 MPa; the sharper blade, cooling in liquid nitrogen, and pre-cracking by fatigue gave an equivalent life time. Slicing with a scalpel or a rotary cutter provided a much longer life time.

Introduction

Initiating the fracture process by the introduction of a well defined notch is the standard method for investigating the fracture behaviour of solids. The intent of the notching procedure is to make a very sharp and a reproducible notch. In the case of ductile materials, the damage introduced by the notching procedure may have a significant effect on the observed fracture and possibly mask the important material parameters that determine the fracture process. The method of notching is especially important in the case of a soft ductile material such as polyethylene. Polyethylene exhibits two distinct modes of brittle fracture. The fast fracture mode is favoured by high stress, fast strain rate and a low temperature. The slow crack growth mode is favoured by low stress and by temperatures in the range above room temperature. As in all cases of brittle fracture, the stress intensity is the most important parameter for both fracture modes; thus, the sharpness of the notch is a key factor.

In this paper, the effect of various notching procedures on the slow crack growth mode of fracture has been investigated. The research is important for the following reasons.

(1) Disagreements between laboratories may stem from differences in notching.

(2) A standard notching procedure for evaluating the slow crack growth of PE has yet to be established so that current specifications for this material are inadequate.

(3) An understanding of the effect of the notching method leads to a better understanding of the fracture process that produces slow crack growth.

The polymer being studied is TR418, a resin that is widely used in gas piping systems and whose resistance to slow crack growth is the critical property that determines its use. Since it takes about 20 years to produce brittle failure in TR418 at room temperature, experiments were conducted at 80 °C where brittle failure can be obtained in about 2 weeks. Also, the most common test in industry for evaluating the fracture behaviour of tough pipe resins is conducted at 80 °C in order to obtain results in a useful period of time. Tests at a higher temperature are not advisable because appreciable morphological changes occur in the material about 80 °C. Even 80 °C is not the most desirable temperature for completely avoiding morphological change during the test, but it represents a compromise in order to produce a slow crack growth in a reasonable period of time.

Much work has been done by Brown and coworkers [1–7] on the slow crack growth of polyethylenes using a razor blade to produce the notch. A notching procedure was developed which yields excellent reproducibility. A major purpose of this investigation is to determine whether there are other notching procedures which will cause PE to fracture in a shorter time than the procedure which has been developed by Brown and coworkers. The other procedures which are presented in this paper are used or were suggested by other laboratories in the world where the slow crack growth behaviour of polyethylene is also being investigated. The results of this study show that the notching procedure used by Brown and coworkers, notably pressing a fresh razor blade into the PE at the rate of 50 μm min^{-1} at room temperature produces

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fracture at a rate which is equal to or faster than any other method which has yet to come to our attention and also gives very reproducible data.

2. Experimental procedure

All of the experiments, except one, involved TR418 which is a pipe resin that was manufactured by the Phillips Chemical Company around 1986. It is an ethylene–hexene copolymer with about 4.6 n butyl branches per 1000C. $M_n = 15\,000$ and $M_w = 170\,000$ and density = 0.938. All specimens were made from 10 mm thick compression moulded plaques which were cooled slowly from 185 °C. The geometry of the single-edge tensile specimen is shown in Fig. 1. The width of the specimen and the 1 mm deep side grooves were chosen so that the fracture was almost completely plane strain. The depth of the notch was chosen so that it was as deep as possible and at the same time, the damage zone at the root of the notch was small compared to the remaining thickness of the specimen during most of its life time.

The stress on all specimens was 2.4 MPa and the temperature was 80 °C. Extensive experiments showed that 2.4 MPa is the maximum stress that will produce pure brittle behaviour at 80 °C and thus produce brittle fracture in the shortest possible time. The depth of all notches was 3.5 mm. The notch opening was measured as a function of time by looking into the notch with an optical microscope which had a filar eyepiece (Fig. 2).

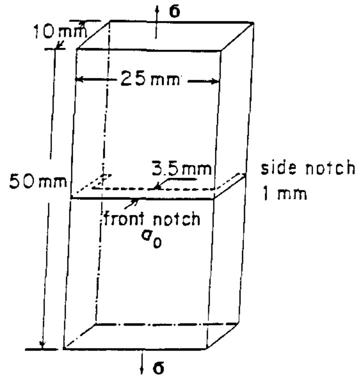

Figure 1 Geometry of specimen.

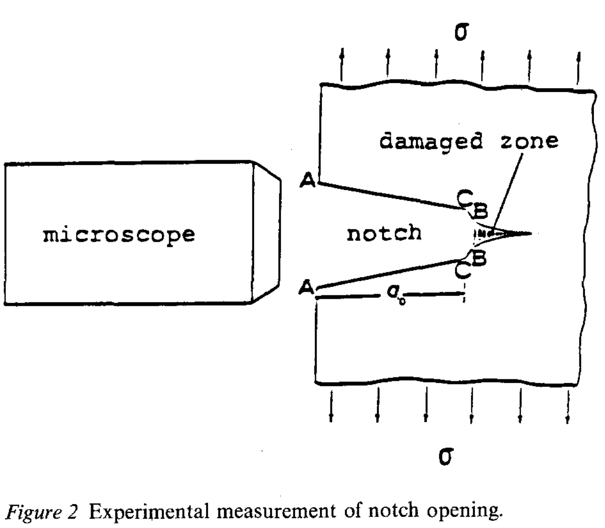

Figure 2 Experimental measurement of notch opening.

3. Results

3.1. Standard notching procedure

In the standard procedure a fresh ordinary commercial single-edge razor blade was pressed into the specimen at a rate of 50 µm min^{-1} at room temperature. The depth of the notch was 3.50 ± 0.05 mm. A fresh blade was used for each notch.

The times to failure for six tests ranged from 20 061 to 33 015 min with an average value of 28 100 min ± 15%. If the average value of two tests with another notching method is more than 30% different than 28 100 min and if the results from these two tests do not differ by more than 30%, then the notching method is considered to be significantly different from the standard procedure.

The curves of notch opening displacement against time for the standard procedure is shown in Fig. 3. The curve AA is the notch opening at the surface of the specimen; the curve CC is the notch opening at the root of the notch where the tip of the razor blade was, and BB is the base of the craze (see Fig. 2). The craze forms immediately after the specimen is loaded. It grows very slowly until fracture starts by the rupture of the fibrils at the base of the craze. Fig. 3 shows that immediately after the loading the thickness of the craze, BB, was 100 µm. Fracture of the fibrils was first observed after 19 000 min when the thickness, BB, was 350 µm. Subsequently the notch opening rate accelerates as the crack grows. Generally the time to initiate fracture is about 60% of the time to failure. The microscopic development of the craze and subsequent crack is shown in Fig. 4.

3.2. A sharper razor blade

A more expensive and sharper razor blade called Gem/Star was used under the standard notching conditions. The average value of the time to failure was 29 000 min. The sharper razor blade gave the same results as the ordinary blade.

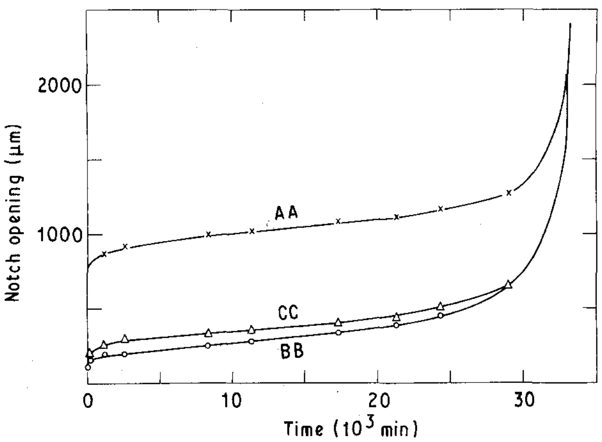

Figure 3 Notch opening displacement plotted against time at 80 °C, 2.4 MPa (× AA, ○ BB, △ CC).

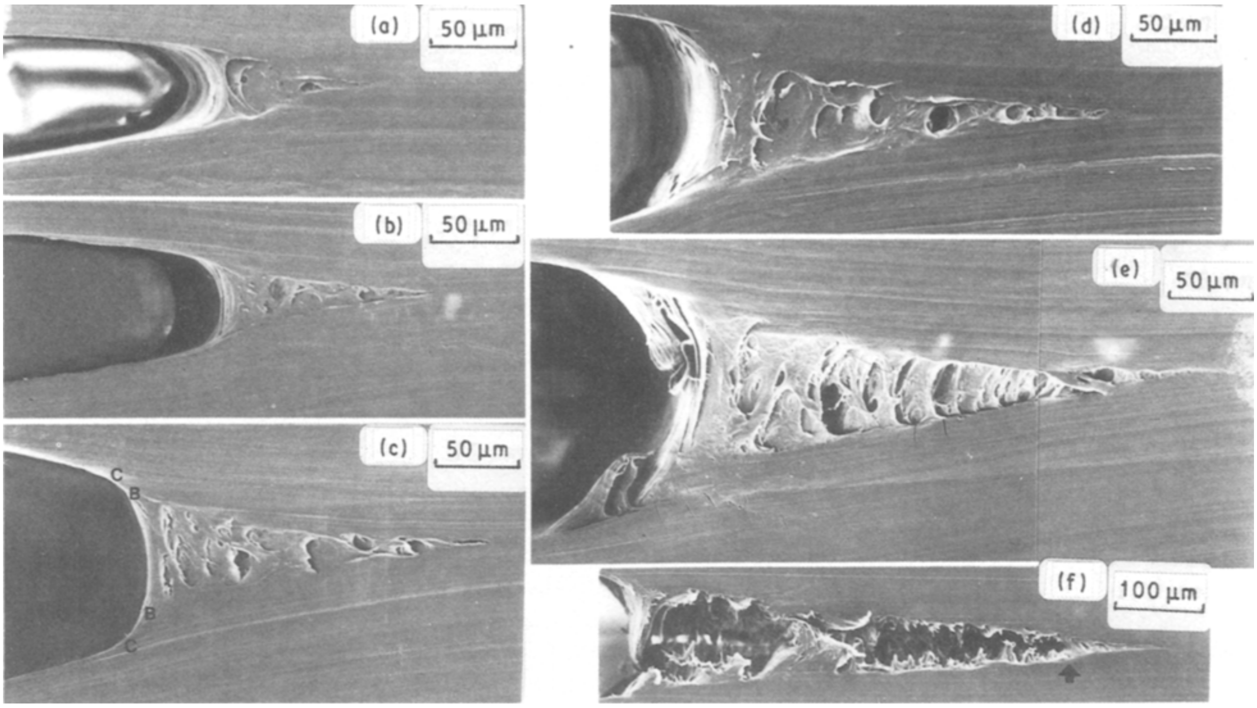

Figure 4 Successive changes in the zone of damage with time where (a) is shortly after loading and (e) is the beginning of fracture.

3.3. Cutting the front fibrils

An attempt was made to accelerate the time to failure by cutting the front fibrils of the craze with a razor blade. The specimen was loaded for 3000 min at which time the craze thickness BB, was 250 µm. The results are shown in Fig. 5. After cutting fibrils, it took 33 500 min for one specimen to fail. Another specimen took 31 200 min to fail after the front fibrils were cut. The cutting of the front fibrils did not change the time to failure significantly. Fig. 5 also shows micrographs of front fibrils before and after they were cut.

One reason for cutting the front fibrils was based on the microscopic observation that fracture first initiates at the fibrils that are immediately behind the front fibrils. This indicated that the front fibrils were somewhat stronger than the other fibrils. Cutting the front fibrils did not, however, make a significant change in the time to failure. Another reason for cutting the front fibrils was to determine whether the fibrils near the very bottom of the notch were influenced by the damage produced by the razor blade. These results showed the same incubation time for crack growth occurred after the original front fibrils were eliminated.

3.4. Cooling in liquid nitrogen

It has been suggested by various investigators that embrittling the polymer by lowering its temperature would allow the razor blade to make a sharper notch. The specimen was first notched at room temperature to a depth of 3.20 mm. The specimen was then cooled with liquid nitrogen and notched an additional 0.30 mm. This specimen failed after 38 000 min.

Other specimens were notched at room temperature to a depth of 3.50 mm and then were loaded in liquid nitrogen for 5 min in order to produce a craze at the root of the notch. Such a craze is shown in Fig. 6. This craze length was varied by varying the stress in liquid nitrogen. One specimen was loaded under 18.2 MPa

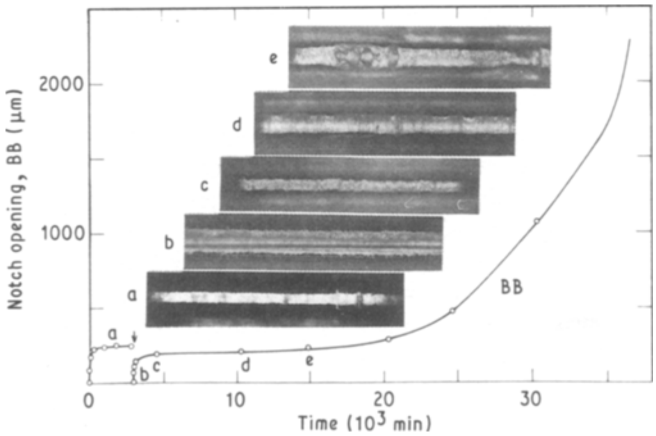

Figure 5 Notch opening plotted against time. After the time indicated by the arrow, the front fibrils of the damaged zone were cut with a razor blade. a to e refer to the micrographs of the notch opening at various times; (a) is just before cutting the fibrils and (b) is shortly afterwards. The central light zone in (b) is BB.

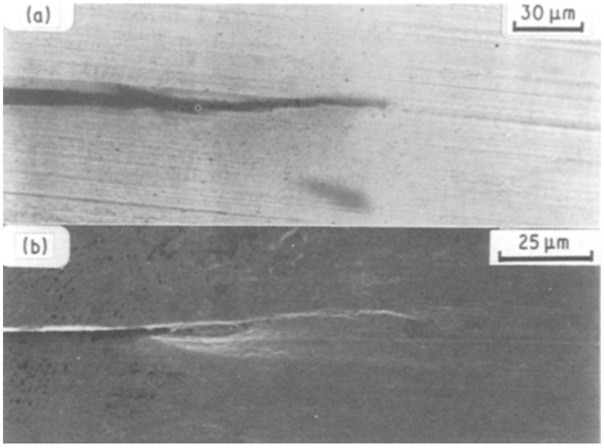

Figure 6 Damage zone of a standard notched speciment after it was loaded for 5 min at a stress of 18.2 MPa in liquid nitrogen (a) is an SEM micrograph and (b) an optical micrograph.

and another under 25.4 MPa and the respective failure times under the standard conditions at 80 °C were 38 000 and 33 000 min. It is important to note that the craze that is produced upon the initial loading at 80 °C is much larger than the crazes that were produced in the liquid nitrogen. Thus, the 80 °C craze (Fig. 4a), in essence, overshadowed the liquid nitrogen craze.

3.5. Pre-cracking by fatigue

In the case of metals it is common practice to sharpen a machined notch by producing a fatigue crack. In the case of polyethylene the fatigue process produces failure many times faster than under a constant load. In this experiment the specimens were given the standard 3.50 mm notch and then were fatigued at 0.5 Hz and a stress of ± 3 MPa and at 42 °C. One specimen was given 14 280 cycles and the other 15 840 cycles in order to initiate fracture in the front fibrils of the craze. The former specimen failed in 30 105 min and the latter in 38 800 min. The curves of notch opening against time for the specimens precracked by fatigue was about the same as those with the standard razor blade notch. It is important to note that the notch opening after fatiguing was about 18 µm whereas the initial notch opening in the 80 °C test was about 100 µm. Once again, the 80 °C craze overshadowed the damage zone that was introduced during the fatigue precracking process.

3.6. Slicing with a surgical blade

A fresh scalpel was used on each of the following specimens. The blade was mounted on a milling machine at an angle of 30° to the surface of the specimen. The specimen was moved past the blade at the minimum speed of the machine, 14 mm min^{-1}, so that a 3.5 mm notch was sliced into the specimen. The results of three tests gave failure times of 52 798, 70 000 and 77 280 min, all of which are significantly greater than for the standard procedure.

The above test was modified by first making a 2.5 mm deep notch with a thin rotating saw. Then the remaining 1.00 mm of depth was made by the slicing procedure described above. The time to failure was 64 780 min.

The procedure was modified by having the scalpel make a 90° angle to the surface of the specimen. The failure times for two tests were 52 300 and 68 000 min. The curves of notch opening against time for the razor blade and scalpel notches are shown in Fig. 7. Slicing with a scalpel causes the life time to be about twice that for the standard procedure.

3.7. Rotary cutter

The following procedure was introduced by Marshall for testing polyethylene pipes and is described in detail by Laurence and Summer [8]. It is now used by British Gas in order to decrease the failure time of the standard 80 °C pressure tests on gas pipes and is part of their acceptance procedure. The cutter is 3″ (1″ = 2.54 cm) in diameter with 32 teeth and the teeth form a 60° angle. The cutter is mounted in a milling machine and rotates at 700 r.p.m. The specimen is fed into the cutter in the same direction that the teeth are moving at a speed of 150 mm min^{-1}. For two specimens the 3.5 mm notch was made in a single pass. The failure time for one specimen was 200 000 min and the other specimen did not initiate fracture after 47 300 min.

The above procedure was modified by increasing the cutter notch with a razor blade using the standard procedure. In one case the 3.5 mm cutter notch was increased by 0.125 mm with the razor blade. This specimen has yet to initiate crack growth after 110 000 min. In two other specimens the cutter notch was increased by 0.25 mm with the razor blade. Their times to failure were 45 230 and 66 000. These results indicate that in order to remove the effects of the cutter notch it is necessary to cut through the bottom of the cutter notch by more than 0.25 mm with a razor blade. The curves of notch opening against time for the standard razor blade notch and the various cutter notches are shown in Fig. 8.

The microscopic details of the cutter notch are seen in Figs 9c and 9d. The morphological structure of the cutter notch is completely different from that for the razor blade. The notch is much blunter. The damage zone at the very base of the notch appears to consist of an oriented structure which apparently resists the growth of a crack. Beyond the bottom of the notch the material is severely deformed to a depth of at least 200 µm.

3.8. Microstructure of the various notches

The microstructures of the damaged zones from the razor blade notch, the scalpel, and the cutter after exposure to 2.4 MPa at 80 °C for a short time are shown in Fig. 9. The razor blade notch is the sharpest and its damage zone contains fibrillated structure at the root of the notch. The damage zone from the scalpel contains a non-fibrillated region of damage at the root of the notch with fibrillation starting further away. The cutter notch is very blunt and its damage

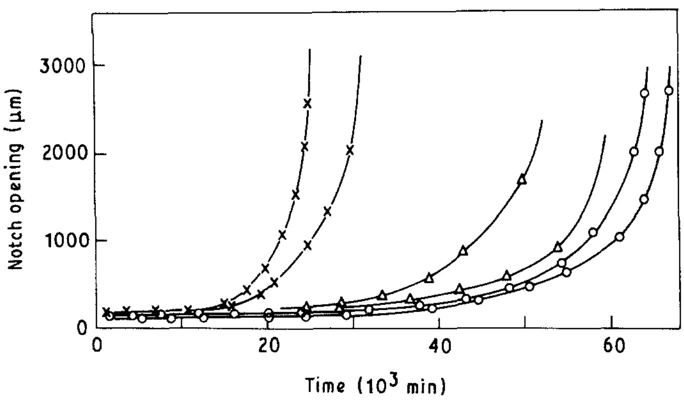

Figure 7 Notch opening against time curves of a standard razor notch (× , $a_0 = 3.5$ mm) as compared to notches made by slicing with a scalpel (△ 90°, $a_0 = 3.5$ mm, ○ 30° $a_0 = 3.5$ mm). 80 °C, 2.4 MPa.

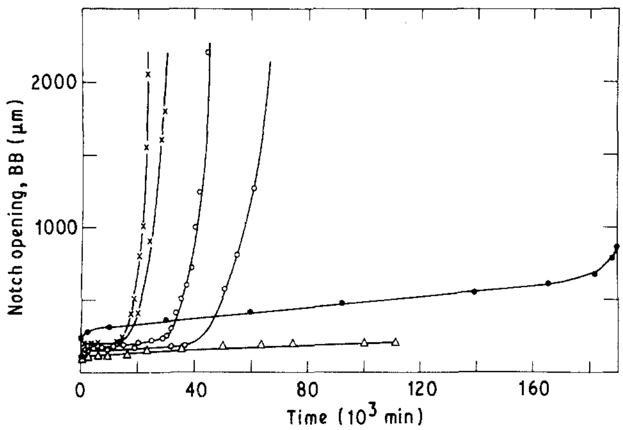

Figure 8 Notch opening versus time curves of standard razor blade notch (×) as compared to notches made with the rotary cutter. (○ rotary cutter plus 0.25 mm razor notch, ● rotary cutter, △ rotary cutter plus 0.125 mm razor notch).

zone is entirely different from the craze that was initiated by the razor blade notch. Prior to loading the cutter notch, the thick zone of deformation, about 200 µm deep, was produced by the cutter at the bottom of the notch.

3.9. Difference between razor blade and cutter notch in fatigue test

A fatigue test was run at 42 °C, 0.5 Hz, square wave, and ± 5.5 MPa. One specimen was given the standard razor blade notch and the other the rotary cutter notch. Cycles to failure for the former were 3000 and 8764 for the latter.

3.10. Difference between razor blade and cutter notch for another pipe grade resin

Another pipe grade resin, an ethylene–octene copolymer, was tested. Two tests with the rotary cutter notch gave life times of 8528 and 8890 min. Three tests with a razor blade notch gave life times of 480, 496, and 581 min. The razor blade notch produces failure 16 times faster in this material than does the rotary cutter.

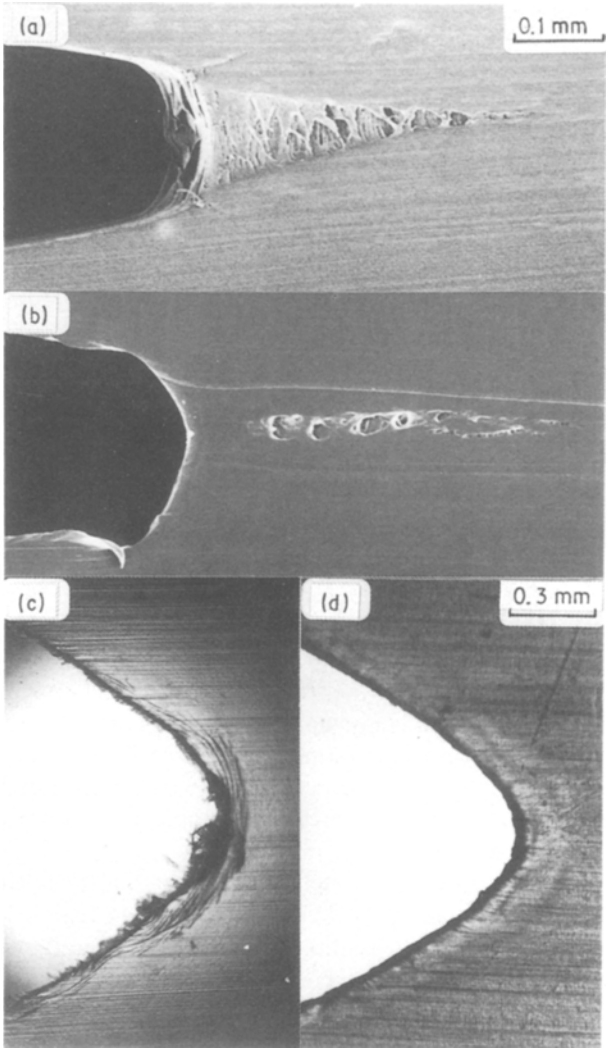

Figure 9 SEM micrographs of damage zones (a) standard razor notch after 7571 min of loading; (b) scalpel notch sliced at 90° angle after 8000 min of loading; (c) rotary cutter notch after 47 300 min of loading; (d) is a transmission optical micrograph of the damage zone shown in (c).

3.11. Microscopic details of the razor blade notch

Fig. 10b shows a side view of the microscopic details of the razor blade. The radius of curvature of the tip, t, is about 2 µm. The tip, w, forms an angle of about 60° and is about 10 µm long. The major surface of the blade, n, forms an angle of about 12°. Fig. 10a shows the tip view of the razor blade. A side view of the notch

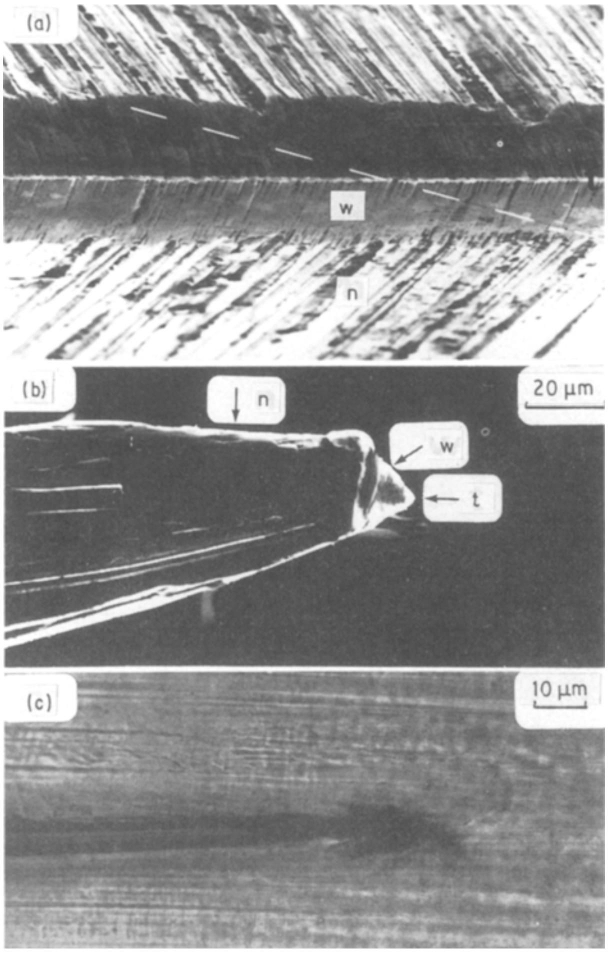

Figure 10 (a) SEM micrograph of razor blade as viewed toward the tip; (b) side view of razor blade; (c) damage zone from razor blade after notching.

is shown in Fig. 10c after the razor blade was removed and the notch was not opened. The dark arrow head shaped region is the damage produced by the razor blade and mostly corresponds to the area of the notch in contact with the surface w and the corner between surfaces w and n of the razor blade.

Fig. 11 shows the notch region after the specimen had been fractured at 80 °C. The scratched region N was not altered by loading the specimen. The boundary between N and L corresponds to the boundary between n and w in Fig. 10b. The region L corresponds to the arrowhead-shaped deformation zone in Fig. 10c and corresponds to the inner surface of the notch where it was in contact with the surface w (Fig. 10b) of the razor blade. Immediately upon fully loading the specimen at 80 °C the compressed arrowhead-shaped region adjacent to the razor blade separated from the rest of the material and fractured.

Also upon loading, the craze formed with a film at the base of the craze. Region S in Fig. 11 is this film after the specimen fractured. This film is evident in Fig. 4. The fibrillated region F in Fig. 11 was produced when the craze fractured. The direct observations of the dynamics of the fracture process with the microscope showed that the very initial fracture of the craze occurred immediately behind the front film S. Figs 4, 5, 10, and 11 in conjunction with the optical observations of the notch during the entire test lead to the following pictures of the structure in the neighbourhood of the notch and the fracture process.

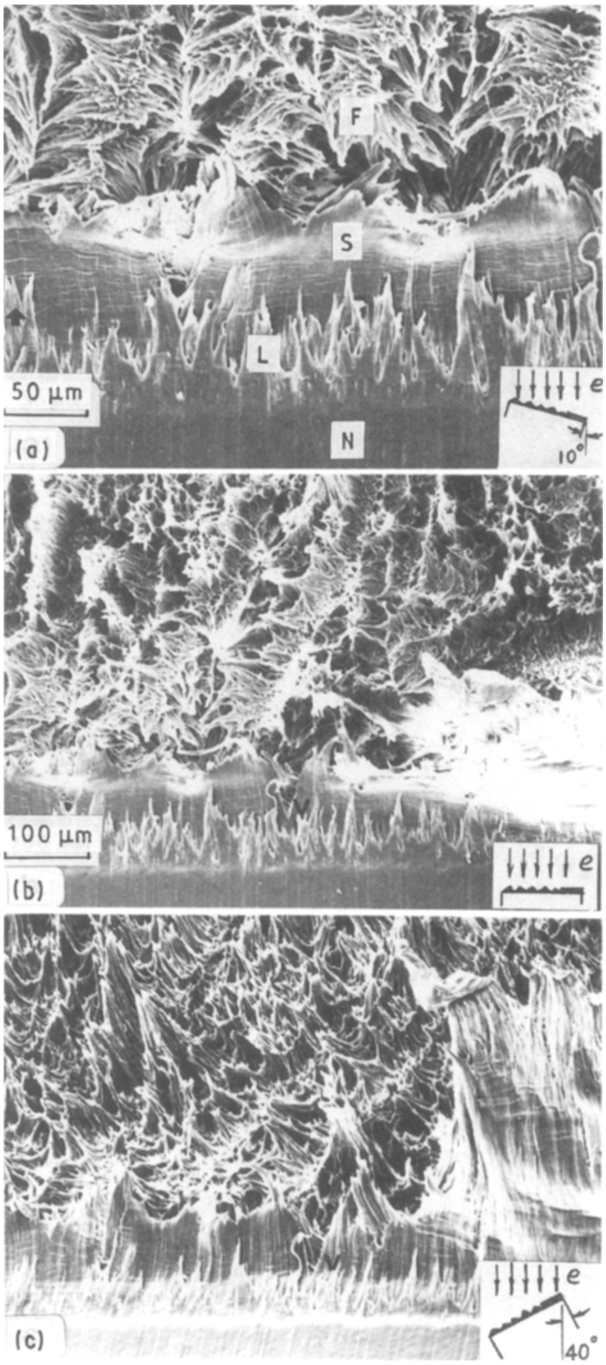

Figure 11 SEM micrographs of fractured surface with different tilts in the microscope. N is surface of the notch as scratched by the razor blade. L are broken fibrils which formed immediately after loading. S is the film that forms at the base of the craze after loading. F are the fibrils of the craze that fractured during slow crack growth. (b) and (c) are the same as (a) for different angles of tilt in the SEM.

Fig. 12a illustrates the razor blade embedded in the specimen with a thin, intensely deformed damage zone which surrounds the surface w of the razor blade. Fig. 12b shows the collapse of this damaged zone when the razor blade is removed. Fig. 12c shows the damaged zone when the specimen is partly but not completely loaded at 80 °C. The film CC corresponds to the thin zone of damage that was in contact with the razor blade. The film BB is the front part of the

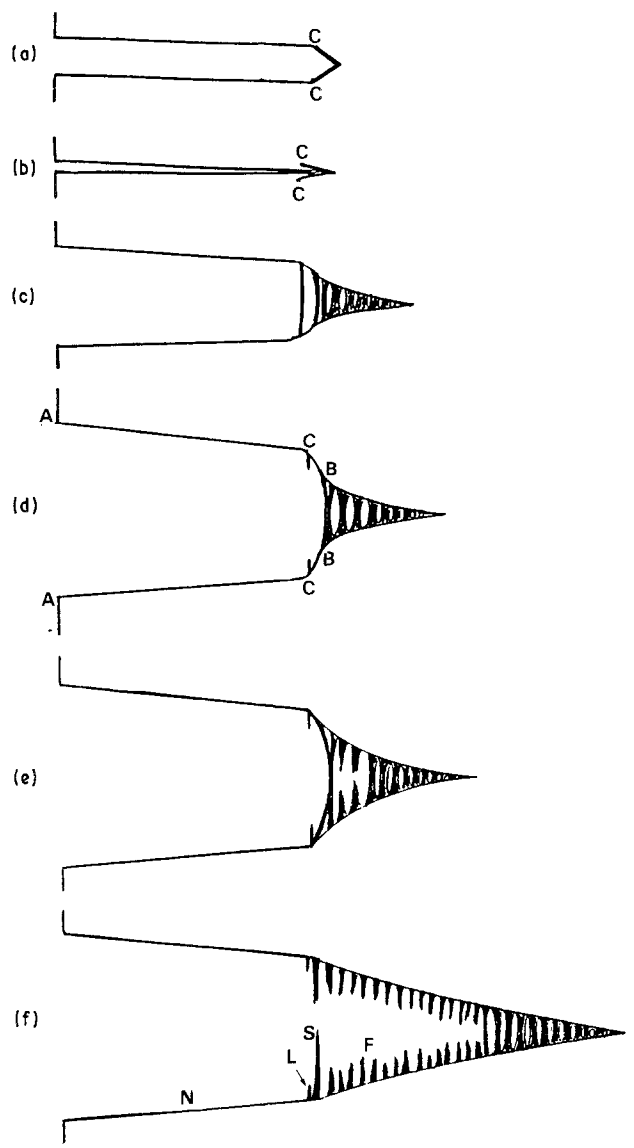

Figure 12 Schematics of the failure process.
(a) the damaged layer that forms at the interface of the razor blade and the polymer matrix. (b) as (a) after the razor blade was removed. (c) damage zone before the load is fully applied. (d) damage zone immediately after full loading. (e) damage zone at initiation of crack growth. (f) damage zone during crack growth.

craze that forms during loading. The remaining fibrils of the craze follow behind BB. When the specimen is fully loaded the film CC ruptures instantly as shown in Fig. 12d and the size of the craze is about that predicted by the Dugdale theory.

During the test for about 0.6 of the time to failure little happens as shown by the change in notch opening (see Fig. 3). When fibril fracture is first observed, it occurs immediately behind the front film BB as shown in Fig. 12e. Shortly, thereafter the film BB breaks as shown in Fig. 12f when, as the crack advances, it is preceded by additional crazing. The front film BB is somewhat stronger than the fibrils immediately behind it. Prematurely cutting this front film with a razor blade (Section C of Results) did not, however, significantly change the time to fracture.

4. Discussion

The shortest failure time was produced by pressing an ordinary razor blade into the specimen at a speed of $50\,\mu\text{m}\,\text{min}^{-1}$ at room temperature. Using a sharper razor blade made no difference because the sharper blade probably has a more highly polished tip, but the damage of the layer should be about equivalent to that of the ordinary blade. Any differences would not matter because this damage layer, CC in Fig. 12c, breaks immediately upon loading and has no effect on the subsequent time to failure.

The fact that cutting the front film of the craze did not affect the failure time means that the fibrillated structure of the craze that was adjacent to the razor notch is no different from the structure that develops further away from the notch. After the fibrils were cut another film formed at the base of the craze. The base of the craze is a film rather than fibrils because the front film forms under plane stress conditions and the fibrils form under plane strain.

That liquid nitrogen notching and notching by fatigue had little effect is simply explained by the fact that the damage zone produced by these procedures was overwhelmed by the much larger craze that is produced at 80 °C immediately upon loading the specimen.

The scalpel produces an entirely different damage zone from the razor blade. As shown in Fig. 9b, an unfibrillated zone of damage is produced immediately below the surface of the notch. Fibrillation is observed at about 50 μm away from the bottom of the notch.

The rotary cutter produces an entirely different type of damage zone which is so strong that a time to failure of 200 000 min was observed as compared to 28 000 min for the razor blade notch. In addition, the sharpness of the rotary cutter notch is much less than that by the razor blade. In order to remove the effects of the rotary cutter it appears that the razor blade must be inserted to an additional depth of greater than 0.25 mm. The razor blade notch is generally more effective than that from the rotary cutter as evidenced by their comparative effect on fatigue life. Similarly, for the conventional hydrostatic pressure test used to qualify the performance of plastic pipe, it is expected that the razor blade notch will give an answer much faster than with a rotary cutter notch.

The standard test specimen developed by Brown and coworkers for evaluating various types of polyethylene is the result of several years of very empirical research. The damaged layer produced by the razor blade at a speed of $50\,\mu\text{m}\,\text{min}^{-1}$ does not interfere with the formation of the natural craze which is produced during the loading of the specimen and which governs the subsequent fracture process. Possibly a somewhat-faster notching rate would give the same results. Comparative testing was done with two independent laboratories, Tokyo and Osaka Gas using specimens of the same geometry and of the same material, and the Japanese razor blade notch was made at a much faster rate than $50\,\mu\text{m}\,\text{min}^{-1}$. This faster notching rate doubled the lifetime. An important advantage for using the slow rate of notching is in the control of the depth of the notch; thus, our data are very reproducible. Much of the scatter in our results is caused by temperature variations during the test and the fact that the variations in thickness of the starting plaque also are a factor. However, a ± 15% scatter in times to failure is considered to be excellent in the light of data reported in most other investigations.

The side grooves in the specimen are important because without side grooves the time to failure is about 30% longer. The side grooves essentially eliminate the component of plane stress fracture that occurs without them.

Our standard notching method has been compared with other methods based on an 80 °C slow crack growth test with gas pipe grades of polyethylene. It is not completely certain that our standard procedure will also give the shortest time for tests at other temperatures down to room temperature. Extensive microscopic examinations of the mechanism of slow crack growth from 80° to 23 °C and with a large number of polyethylenes indicate, however, that the fracture mechanism as illustrated in Figs 12d, 12e, and 12f is basic to all. Consequently, it seems that our standard razor blade notch will generally give the shortest failure time compared to the other notching methods that are presented in this paper.

It is important to recognize that for the rapid fracture process that occurs at low temperature, high strain rates and at high stresses, our standard procedure may not be better than other methods for reproducing the value of the fracture toughness. However, when the notching speed is low, the notch depth can be very well controlled and thus, the scatter in the test results will be decreased.

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