Fatigue of viscoelastic polymers:
2. Fractography

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The results of a fractographic study of fatigue failure in viscoelastic polymers is presented. Tests were conducted on a spherulitic low-density polyethylene in reversed loading using non-symmetrical deformation conditions. The microfractographic features have been found to depend on the deformation programme, the temperature of the test and the position on the fracture surface. The latter has been related to the different stages of growth revealed by plotting crack-growth propagation data. As well as interspherulitic fracture, other mechanisms that are sometimes prevalent involve cold-drawing, (fibrillation), and/or lamellar reorientation. The appearance of fatigue fracture surfaces of a non-spherulitic low-density polyethylene, a high-density polyethylene and a plasticized poly(vinyl chloride) corroborate the structural interpretations proposed to account for the observations with the spherulitic low-density polyethylene.

INTRODUCTION

The use of fractography in the post-failure analysis of metals is well established. There is a growing trend to develop this approach for polymers but it is clear that reliable diagnostic interpretation of fractographs awaits the documentation of tests conducted over a wide range of strictly controlled conditions. The results of early studies can be found in the book by Andrews[1] since when notable contributions have been made by Hull and coworkers, (ref 2 and refs therein), Jacoby[3,4], Doyle[5,6] and Kusy and Turner[7–9]. These studies[2-9] were all conducted on glassy polymers (polystyrene, poly(methyl methacrylate) and polycarbonate), and only the work by Jacoby includes examination of fatigue fracture. The review by Manson and Hertzberg[10] contains a substantial section on fatigue fractography, but there is very little presented relating to fatigue of non-glassy polymers. It is in this area that the work reported here belongs.

Fatigue tests have been conducted on thermoplastics under strictly controlled conditions and inspection of many of the fracture surfaces performed by scanning electron microscopy. Most of our attention has been concentrated on a spherulitic low-density polyethylene, (LDPE), as directed by the crack propagation investigation described elsewhere[11–14], but for comparison the programme was expanded to include fatigue fractographic studies of a non-spherulitic LDPE, a high-density polyethylene, (HDPE), and a non-crystalline, non-glassy polymer, plasticized poly(vinyl chloride), (PPVC).

EXPERIMENTAL

Materials and test-piece fabrication

Test pieces were cut into strips from sheet or, in the case of one grade of LDPE, compression-moulded into parallel-sided dumb-bells by a routine described elsewhere[11,13], that involved a final re-melting and slow-cooling stage under light pressure designed to give a good surface finish and an absence of internal moulding stresses or voiding. Designations and sources are listed in *Table 1* together with some relevant specifications.

Table 1

Designation	Source	Specifications				Form	Method of fabrication
		M_w	M_n	density (kg/m^3)	*MFI*		
LDPE A	ICI: WJG 11			918	2.0	Granules	Compression-moulded into sheet, stamped into dumbbells and re-moulded in close-fitting moulds
LDPE B	G. H. Bloore Ltd			922		Extruded sheet 0.96 mm thick	Cut into parallel strips
HDPE 1	RAPRA	$\sim 10^5$	$\sim 10^4$	960	2.5	Granules	Compression-moulded into sheet, 1.6 mm thick. Cut into parallel strips
PPVC	Stanley Smith & Co.	$\sim 10^5$	$\sim 4.5 \times 10^4$			Extruded sheet, 1.08 mm thick	Cut into parallel strips

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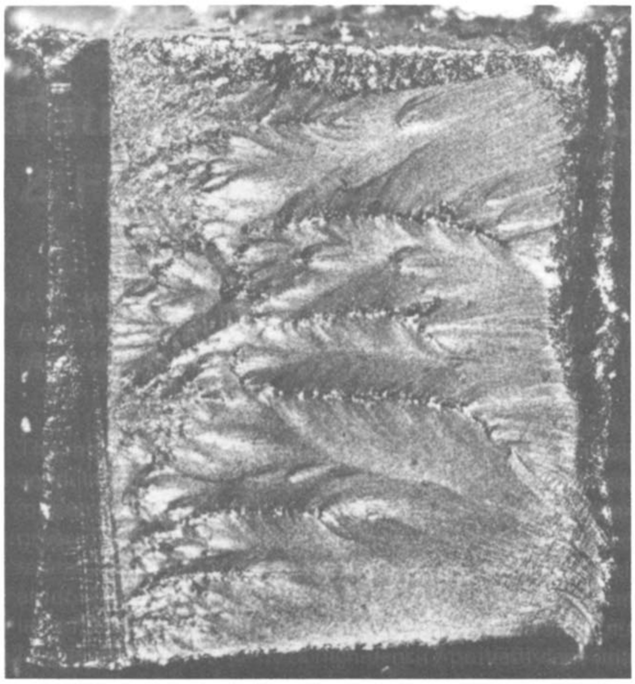

Figure 1 Optical micrograph of LDPE A fatigue fracture surface, (~3 x 3 mm). $\epsilon_T = 0.034$; $r = -0.1$; $N_f = 500$

Specimens cut from sheet were 10 or 15 mm wide with a gauge length of 25 mm, and oriented parallel to the extrusion direction where applicable. Dumb-bell specimens were approximately 3 mm wide, 3 to 3.5 mm thick, and had a gauge length of 10 mm. A central edge notch was made by inserting a razor blade prior to each test.

Fatigue testing

Cyclic loading experiments were conducted at 0.25 Hz on a machine designed and built for this programme[11]. A major objective of the work was to examine the influence of the compressive component of reversed loading programmes on fatigue crack propagation, and the mean strain and strain amplitude were altered independently.

Fractography

Fracture surfaces were aluminium-coated in an evaporation chamber prior to light-microscope examination in order to improve contrast, and the photomicrographs presented here are all bright-field images recorded using normal incidence illumination. Specimens were usually coated with gold/palladium for scanning electron microscope (*SEM*) observation in a Jeol JXA-50A instrument. A fairly low accelerating potential of 10 kV with absorbed current of 10^{-11}A were used to minimize radiation damage. In the case of LDPE significantly higher electron energies and intensities were found to promote damage which usually took the form of fissures lying perpendicular to the direction of crack propagation[15,16]. Under the irradiation conditions used for the part of the work described here no visible changes were detected during prolonged observation, that was conducted exclusively in the secondary electron image mode, (*SEI*).

RESULTS

The results will be presented for each material in turn. Tests are characterized in terms of the maximum strain (ϵ_t) and minimum strain (ϵ_c) by the total strain amplitude $\epsilon_T = \epsilon_t - \epsilon_c$ and the strain ratio $r = \epsilon_c/\epsilon_t$. In the studies reported here ϵ_t is always tensile and positive while ϵ_c represents a compressive strain and is therefore negative. Hence r will be negative and $\epsilon_T = \epsilon_t + |\epsilon_c|$. The number of cycles to failure is signified by N_f where appropriate.

Low-density polyethylene

Fatigue tests on LDPE A at room temperature. The appearance of the fracture surface was found to depend markedly on the strain ratio, r. *Figure 1* shows the appearance obtained with moderate tensile strains ($0.02 \leqslant \epsilon_t \leqslant 0.04$), and with the strain amplitude in tension exceeding that in compression, ($r > -1$). The initial slow crack growth region[14] has a rough fibrous appearance, shown in more detail in *Figure 2a*, that gives way gradually to a region containing parabolic markings, *Figure 2b*, and finally there is a region which displays both river markings in the direction of the crack and striations perpendicular to it, *Figure 2c*.

When the compressive strain exceeded that in tension, ($|\epsilon_c| > \epsilon_t$, $r < -1$), crack growth was fairly slow and the fracture surface has a rather rough appearance, *Figure 3*, giving the impression of a fibrous mode of fracture, (see *Figure 2a*), followed by damage to the drawn protrusions during subsequent compressive loadings. Conversely, when the deformation was predominantly tensile, crack growth was fairly rapid and the whole fracture surface has river lines and striations[13]. The appearance is similar to that obtained using tensile-only cyclic loading, and the 'microstriations' reported before[15] are in evidence again[13]. Since our previous publication[15] further tests have been conducted that indicate that the coincidence of the average increment of crack growth per cycle and the striation period was fortuitous, and an interpretation for these features must be sought that does not require that they correspond to successive crack front positions. Using a range of test conditions average cyclic crack-growth increments were found that were both much higher, ($\geqslant$8 μm/cycle), and much lower, ($\leqslant$0.1 μm/cycle), than the average microstriation spacing that remained unchanged at ~0.5 μm. An example is shown in *Figure 4* for which the average crack-growth rate was approximately 5.5 μm/cycle.

Tests at temperatures in the range 285–308K showed essentially similar features to those described here for room temperature (293–298K) testing.

Fatigue tests on LDPE A at 261K. There is considerable disagreement over the value of T_g for polyethylene, with quoted values ranging from slightly below 261K down to 180K. It is usually observed that significant changes in mechanical behaviour begin to occur around the former temperature, and the fatigue fracture behaviour is no exception. *Figure 5* shows a fracture surface obtained at low tensile strain and contains a brittle fracture region that probably corresponds to plane strain conditions. At high magnification this has a fibrous appearance, *Figure 6*, contrasting with the ductile region which is similar to that shown in *Figure 4c*[13]. At higher strains the fracture surfaces resemble those obtained at higher temperatures[13]. Although this might suggest that the deformational heat generated near to the crack tip may cause a significant local rise in temperature, it is more likely that the similarity is a consequence of the strain-rate effect discussed below whereby it is proposed that reduced ductility at the higher strain rates associated with faster crack propagation rates accounts for the microstriations. Apparently further reduction in duc-

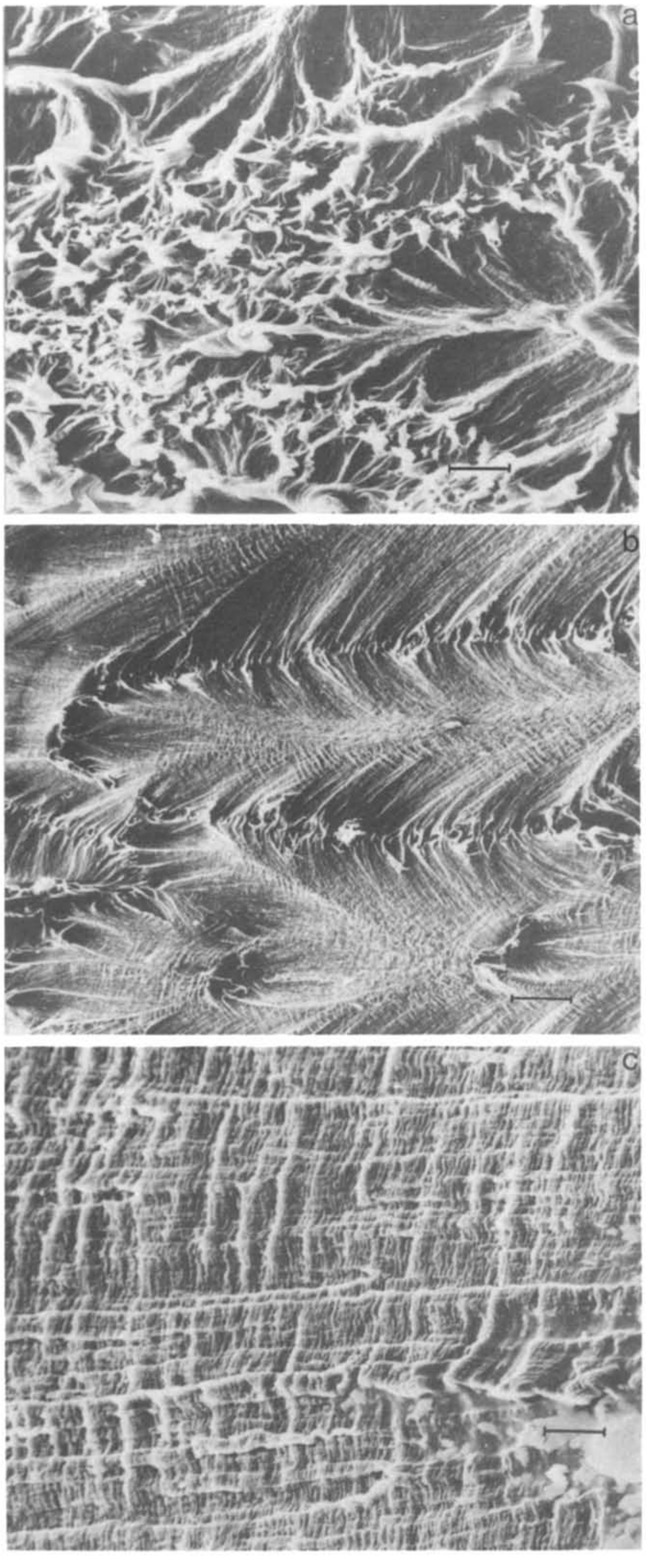

Figure 2 *SEI* of (a) rough fibrous region, (scale bar = 10 μm), (b) parabolic markings (scale bar = 0.1 mm) and (c) smooth striae region (scale bar = 10 μm) of the surface shown in *Figure 1*. Crack propagation direction: left to right

tility produced by lowering the temperature does not further modify the fracture mechanisms.

A fracture surface obtained under unidirectional tensile loading at this temperature showed characteristic brittle fracture features with a mirror region, corresponding to slow initial crack propagation, followed by the arrest zone, and, finally, the fast fracture area[13].

Fatigue tests on LDPE B at room temperature. High tensile strain cyclic loading tests produced fracture surfaces with distinct fatigue striations with large enough spacings to enable quite rigorous confirmation that each striation corresponded to an increment of crack advance and that one is obtained each load cycle, *Figure 7.* Fatigue striations of much smaller period were obtained at lower strain amplitudes *Figure 8*, and the surface was found to have a spongy appearance at high magnification, *Figure 9.* This morphology may be determined by the initial morphology of this class of specimen, which seems to be non-spherulitic; no spherulites could be found when examining thin sections optically, nor was it possible to obtain the characteristic 'clover leaf' (H_V) pattern of spherulitic polymers with a polarized laser beam plus crossed analyser. Essentially similar observations were made using reversed loading conditions.

Fatigue tests on HDPE at room temperature. Stress whitening was found to occur at the crack tip prior to crack growth with HDPE as with LDPE. The fracture surfaces showed a fibrous type of failure in which the fibrillation was far more extensive and developed to a far greater degree than in any of the other types of material examined in this study (*Figure 10*). Microfibrils with diameters as small as the governing instrumental resolution (several tens of nano-

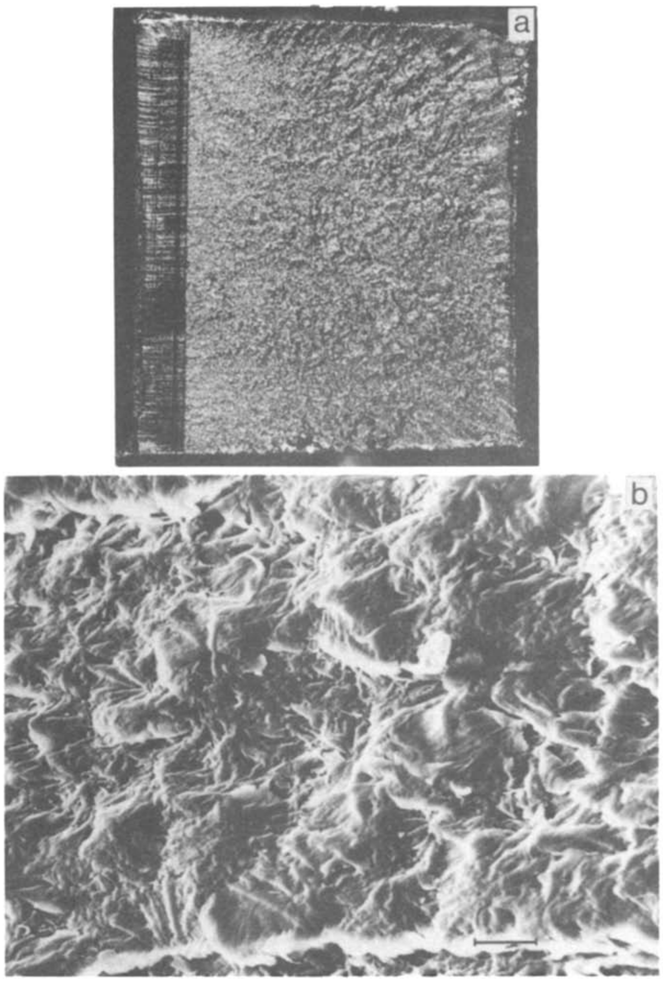

Figure 3 Fatigue fracture surface of LDPE A from a test with a high negative strain ratio, ϵ_T = 0.035, r = −1.33; N_f = 4300. (a) Optical micrograph (~3 x 3 mm); (b) *SEI* (scale bar = 10 μm). Crack propagation direction; left to right

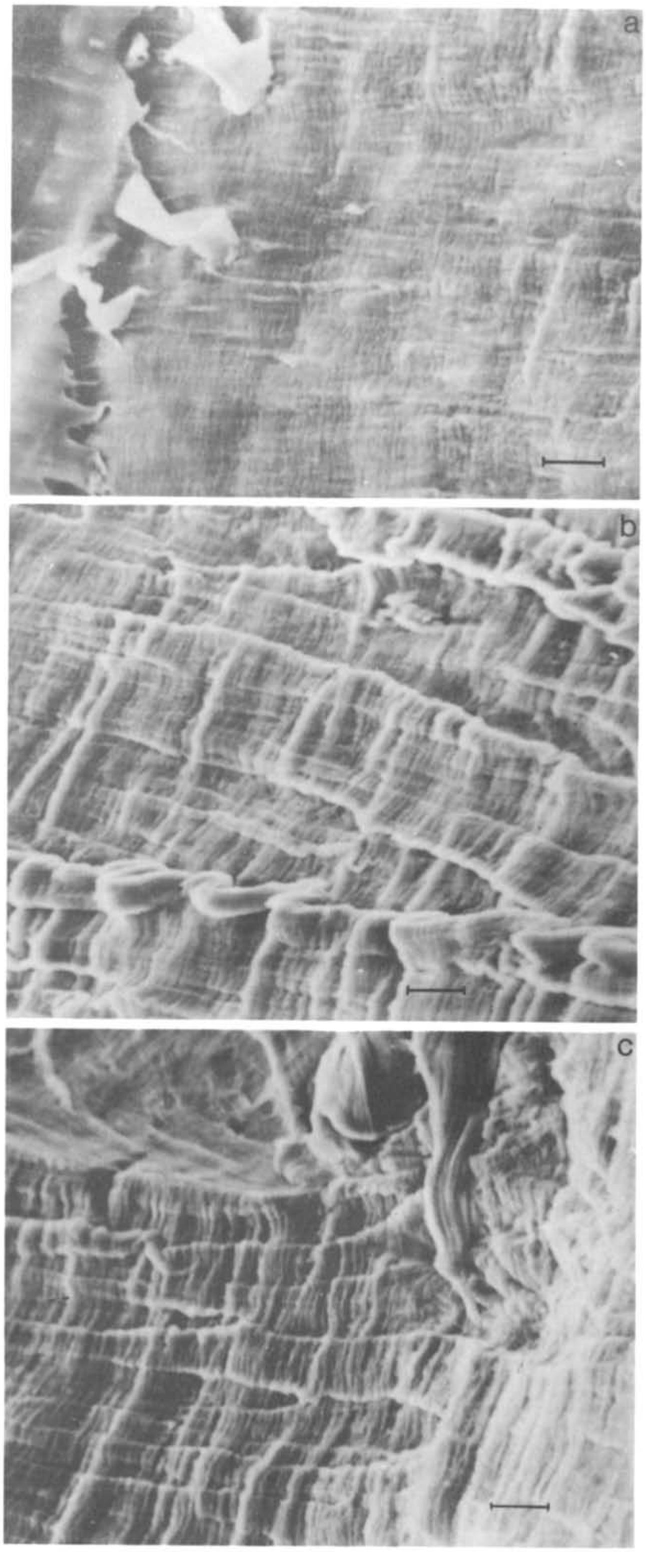

Figure 4 *SEI* showing microstriations (a) near the razor notch; (b) nnear the centre of the specimen; (c) near the completion of failure. (Scale bars: 10 μm). ϵ_T 0.065; $r = -0.3$; $N_f = 460$. Crack propagation direction: left to right

meters at instrumental magnifications small enough to prevent excessive damage) were common, comparable to the well-developed microfibrils produced, (using a different grade of HDPE), by cold-drawing at elevated temperature[17]. The fractured fibril remnants observed on the fatigue fracture surfaces were rarely greater than 3 μm in length, however, compared to continuous microfibrils apparently measuring several mm in cold-drawn specimens.

Fatigue tests on PPVC at room temperature. Fatigue striations were obtained under high tensile strain cycling and again shown to correspond to cyclic incremental crack advance. An example is shown in *Figure 11,* for which average crack growth rates measured at various stages during the test by the normal procedure, (following the intersection of the crack front with the surface of the specimen with an optical microscope), are compared with values measured from the fractographs in *Table 2.*

At low crack growth rates (~0.2 μm/cycle) the apparent spacing exceeds the average crack advance per cycle. It is impossible to decide whether this is a consequence of intermittent crack growth or due to the difficulty experienced in recognizing each striation with certainty at such small spacings.

Many of the fracture surfaces of PPVC contained ribbon-like features somewhat similar to those seen by Bird *et al.*[18] and attributed by them to the main crack overtaking a secondary crack. In the example presented in *Figure 11* the ribbons have a wrinkled appearance as if they had been held under tension to produce some drawing, then released. The introduction of a compressive component did not produce any marked changes in the overall appearance of the fracture surfaces.

At very low tensile strains the fatigure crack was found to propagate at an angle of approximately 45° to the stress axis. In order to inspect the fracture surface in this region at an early stage in its development, free from the surface deformation that occurs during the later stages of a reversed loading fatigue test run to failure, during which rubbing together of the opposite faces must inevitably occur, the following procedure was employed. A specimen was tested in the normal way until the development of the 45° crack was observed, then the test was terminated, the specimen removed,

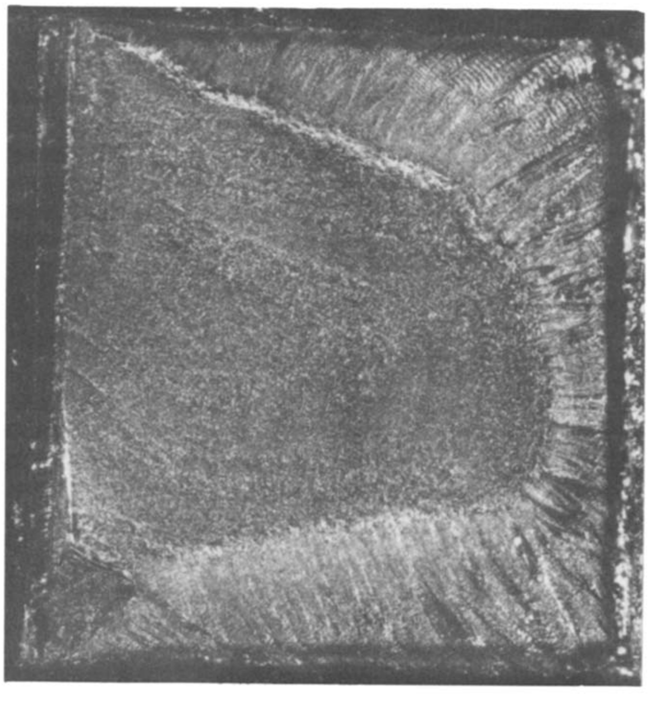

Figure 5 Fatigue fracture surface on LDPE A tested at 261K. (~3 x 3 mm). $\epsilon_T = 0.022$; $r = -0.15$. Crack propagation direction: left to righ

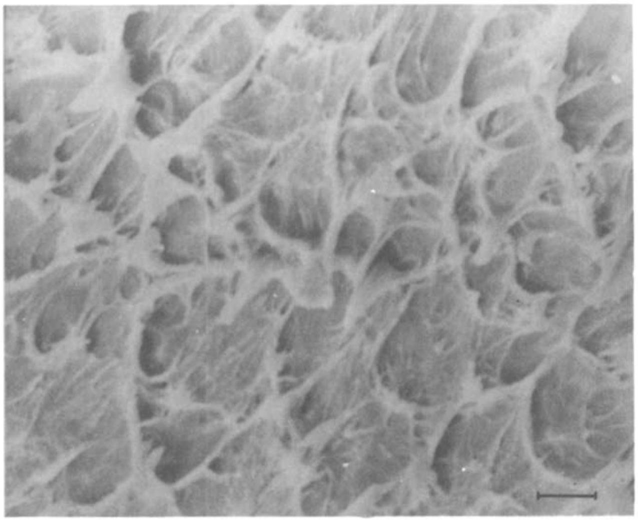

Figure 6 *SEI* obtained from the central plane strain (brittle) region in *Figure 5.* (Scale bar: 10 μm).

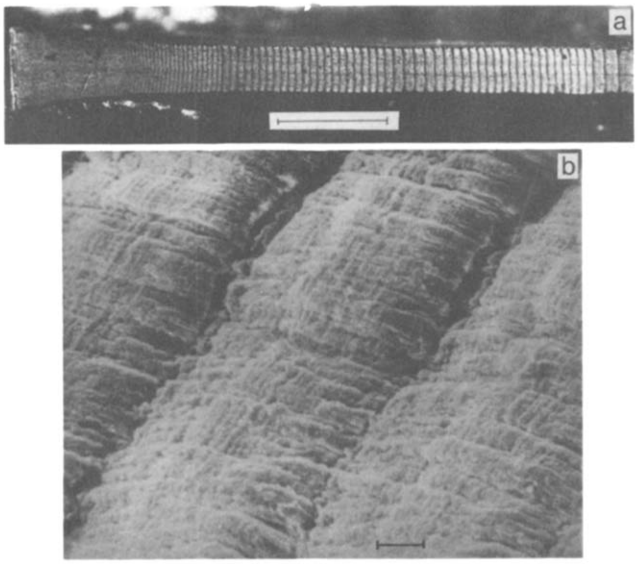

Figure 7 Fatigue fracture surface of LDPE B: (a) optical micrograph showing fatigue striations, (scale bar = 1 mm); (b) *SEI* showing striation detail and microstriations; (Scale bar: 10 μm). $\epsilon_T = 0.108$; $r = 0$. Crack propagation direction: left to right

placed in liquid nitrogen, and fractured. The consequent fast fracture was easily distinguished from the fatigue region, which had a spongy appearance[13].

DISCUSSION

Fracture in semicrystalline polymers

It is well-known that crazing precedes fracture in the vast majority of failures in glassy polymers. Even in fast fracture conditions a craze appears to be present just ahead of the crack, often travelling at the same speed, and we are unaware of any evidence to indicate the certain absence of craze participation in fracture of these materials. The detection of crazes in non-glassy polymers is harder to accomplish than with the transparent glassy polymers, though the existence of crazes in these materials can hardly be doubted[19–21]. The well-known microfibrillation of many semicrystalline polymers, notably linear polyethylene and polypropylene, produced on cold-drawing has much in common with crazing. When conditions are suitable for cold-drawing the microfibrillation can be very well-developed, resulting in complete separation of the microfibrils[17]. The differences between a cold-drawn region and a craze are therefore mainly in their macroscopic dimensions and many semicrystalline polymers must consequently be regarded as intrinsically suited to craze formation. Crazes have been observed in polypropylene deformed at various temperatures[21–24]. On the other hand semicrystalline polymers have available to them many deformation mechanisms that are not present in glassy polymers, so that however universal the craze-prior-to-fracture rule is found to be in glassy polymers, this result need not be expected to extrapolate to semicrystalline polymers. Nevertheless, we consider crazing to be an appropriate description of the kind of deformation we have observed to occur during fracture and will adopt this term in the discussion of our results.

We envisage craze development to occur in the zone ahead of the crack tip, promoted by the attendant stress concentration, just as in glassy polymers. (Although our tests have been conducted on notched specimens it is expected

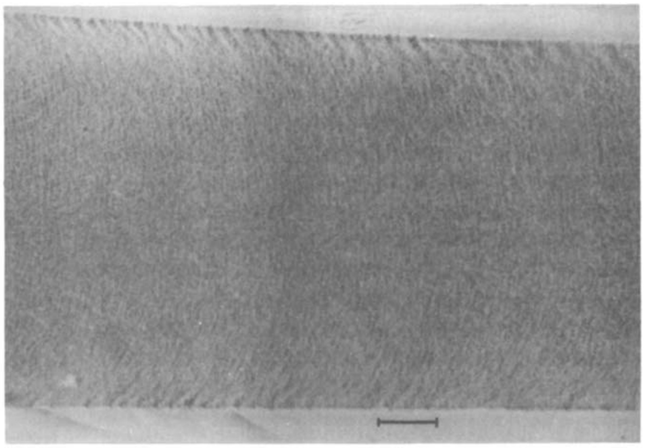

Figure 8 Fatigue striations on LDPE B at low crack growth rate, (*SEI*: scale bar = 0.1 mm). $\epsilon_T = 0.042$; $r = 0$. Crack propagation direction: left to right

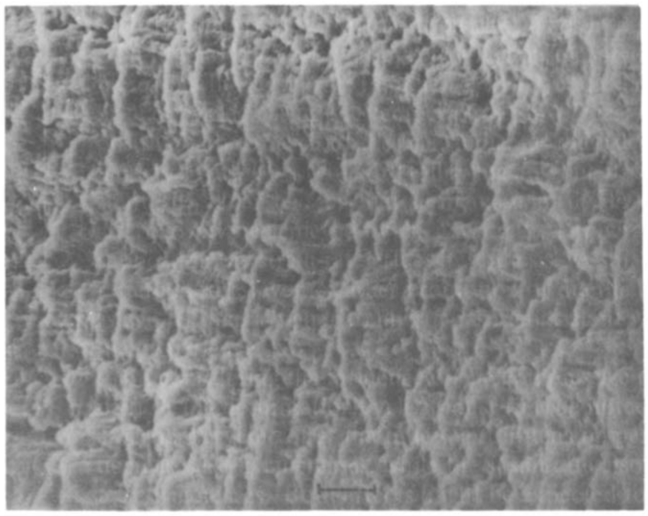

Figure 9 *SEI* showing part of *Figure 13* at higher magnification. (Scale bar: 10 μm)

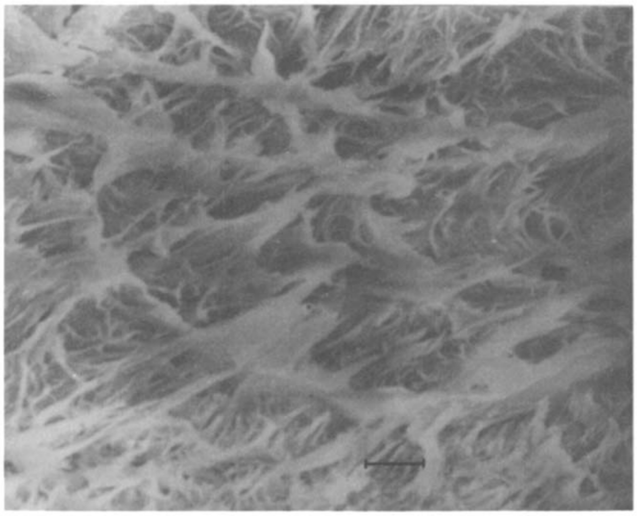

Figure 10 *SEI* of HDPE fatigue fractured in tension. (Scale bar = 10 μm). $\epsilon_T = 0.03$; $r = 0$. Crack propagation direction: left to right

that the intrinsic heterogeneity of the semicrystalline polymer, (see below), would be able to promote the nucleation of crazes[25], given that other deformation mechanisms could not intervene, so that an unnotched specimen may well fail in the same manner through the presence of inherent flaws). A stress-whitened region is normally seen ahead of the crack and is easily visible in the semicrystalline polymers we have tested, (LDPE, HDPE and polypropylene), and we associate this with the drawing and voiding that constitutes the craze. The thickness of the craze will depend on the conditions of fracture. When conditions favour cold-drawing the crystal lamellae may break up to form microfibrils, as happens over a large area in the case of (macroscopic) cold drawing. In cold-drawn macroscopic samples failure eventually occurs in the drawn section, away from the neck, and so it seems is the case in semicrystalline crazes too. Fibrillation is well-developed and fracture appears to be located at the midplane of the craze, as tends to be the case with slow fracture through crazes in glassy polymers[26,27].

Temperature dependence

At room temperature semicrystalline polyethylene is above its glass transition temperature and owes its strength and stiffness to the crystalline phase. Deformation will occur principally in the amorphous phase, but its form will be largely determined by the presence of the crystals, for example interlamellar slip has been identified as an important mechanism. In addition crystalline deformation mechanisms (*c*-axis slip, twinning phase transformation) all occur at moderate strains. At lower temperatures deformation within the amorphous phase will be inhibited and the material can be expected to behave more like a glassy polymer. Despite the fact that the crystal phase plays such a prominent role in fibrillation, this process requires the cooperation of the amorphous phase and can doubtless be prevented from occurring if motion in this region is sufficiently inhibited. This conclusion is particularly unavoidable if the chain-folded model for bulk-crystallized lamellae is rejected, in concord with growing opinion, (see a recent summary by Flory and Yoon[28]).

Crack propagation in the rough fibrous region

In the rough fibrous region the crack advances fairly slowly. The exact locations of the crack front and of the craze front will be largely determined by the local stress concentrations and the local drawability of the material. In the case of a semicrystalline polymer there are two relevant levels of structural heterogeneity. The deformability of the crystal lamellae and the intervening amorphous material will be very different, causing microscopic heterogeneity with a period of the order of 20 nm, while in uniaxial stress conditions the local orientation of the lamellae with respect to the stress axis will be important, so that a second level of heterogeneity corresponds to the three-dimensional arrangement of lamellae and therefore to the spherulite size. In the spherulitic LDPE specimens used in the investigation reported here the spherulite size was 2–10 μm. It is therefore of note that in the rough fibrous region the drawing appears to reflect a

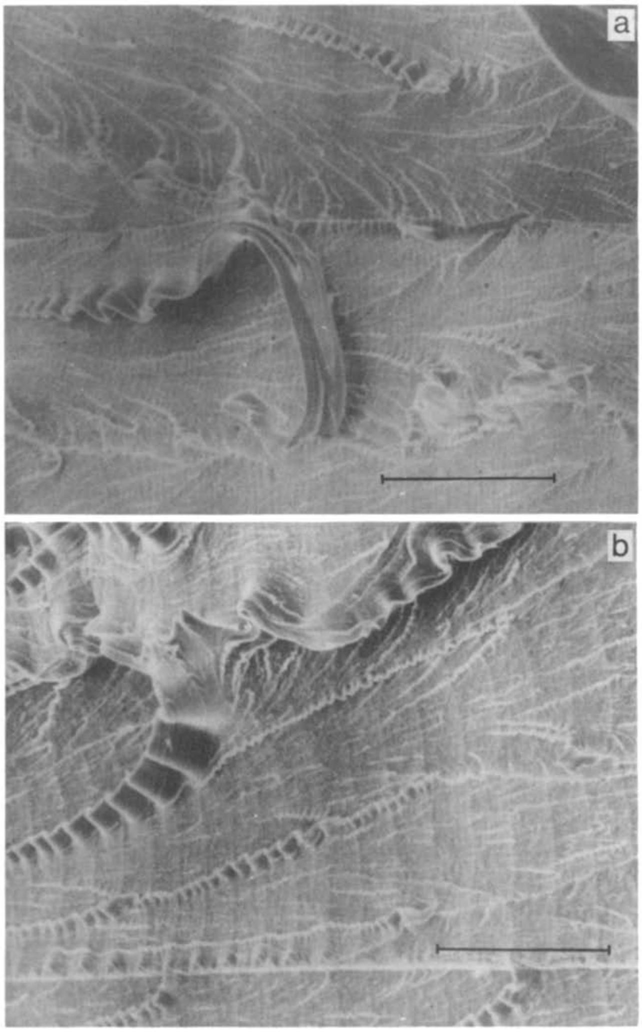

Figure 11 *SEI* of PPVC fatigue fracture surface at different positions (a) *c/w* = 0.10; (b) *c/w* = 0.67; *w* is the width of the specimen (scale bars: 0.1 mm). $\epsilon_T = 0.218$; $r = 0$. Crack propagation direction: left to right

Table 2

c/w	dc/dN (μm/cycle)	Striation spacing (micron)
0.10	4.4	4.0
0.67	26	20
0.92	60	60

domain structure with the same dimensions. The drawability of the lamellae will clearly be dependent on the orientation relative to the stress axis and this probably provides the connection between spherulite size and the fracture surface pattern. If this is the case then it would seem that the majority of the deformation that precedes fracture is concentrated in a relatively small fraction of each of the participating spherulites and fracture can be regarded as being interspherulitic.

When the temperature is lowered, the lamellae will have less freedom to reorient, due to the reduced mobility of the amorphous regions, and there will be less opportunity for them to take up the favoured drawing orientation under stress. It is therefore to be expected that drawing will be more concentrated into those regions which crystallized into the appropriate orientation, with less spreading into neighbouring regions which it is assumed can reorient under stress at higher temperatures. This is indeed confirmed on inspection of the micrographs, as the fracture surfaces obtained at 261K show a more clearly defined domain structure (*Figure 6*). Note that a similar appearance attaches to monotonic uniaxial tensile failure[13]. As is to be expected, drawability is reduced at the lower temperature and fracture occurs at a far less developed degree of drawing than at room temperature. We suppose that the lamellae already lying parallel to the stress axis will be the prime candidates for drawing, and that fibrillation is principally generated from such regions. Some reorientation of lamellae will doubtless occur just ahead of the crack tip, as discussed in the following section, and will be influenced by the stress concentrations associated both with the crack itself and with the fibrillation. In this way fibrillation may become fairly widespread. On the other hand cooperation of the adjacent amorphous phase is required, and in those areas in which complete fibrillation is not in evidence it is likely that failure must have occurred in the non-crystalline phase before the combination of lamellar reorientation and local stress could promote fibrillation of those parts which began in unfavoured orientations. Further to the point, the reorientation of lamellae in unfavoured orientations will at the same time largely relieve the stress upon them, so reducing the chance that the stress to promote drawing will be exceeded.

Large cyclic crack advances

Finally it is necessary to seek an explanation for the appearance of the fracture surface when the cyclic advance of the crack is fairly large, as in tests at high tensile strain and during the final stages of other tests. It is quite often the case that fatigue striations are formed, one per loading cycle, by mechanisms that are not difficult to understand; alternatives have been proposed and it is sometimes possible to obtain an indication as to which one pertains by detailed examination of both fracture surfaces[10]. The PPVC results appear to fall into this category. On the other hand with spherulitic LDPE cycled with high tensile strain a most striking feature is the pattern of microstriations lying parallel to the crack front[13,15]. In our early experiments using this type of loading the number of microstriations over the fracture surface was found to agree fairly closely with the number of cycles to failure[15] and it seemed likely that they corresponded to true fatigue striations, grown one per cycle. Later studies showed that this could not be so, however, as the pattern and period of the microstriations remained substantially the same throughout tests in which the measured crack propagation rate varied by a factor of more than ten. The important point to note is that microstriations with periods both much smaller and much larger than the local average cyclic crack front advance are seen, so that the discrepancy cannot be explained by invoking a crack arrest mechanism.

To the knowledge of the authors no reports have been made of striation periods smaller than the incremental crack advance on glassy polymers and it is therefore appropriate to seek a structural explanation to account for their appearance on semicrystalline fracture surfaces. The fact that the LDPE used was isotropic while the microstriations were extremely well oriented means that some interaction with the crack front or its attendant stress field must be involved. Microstriations are also found on fracture surfaces on non-spherulitic LDPE and HDPE, though are less prominent.

The size and arrangement of the microstriations is consistent with their identification as lamellae, either exposed ends or fractured sections, and we will now examine the implications of this proposal.

At small deformations the effect of an uniaxial stress on a spherulitic polymer is to rotate lamellae into orientations in which the lamellar surface becomes more nearly parallel to the stress axis[29–34]. It would therefore be expected that a certain amount of this type of re-ordering takes place in advance of the crack tip during the tensile parts of the load programme. Further modification to the local orientation of the lamellae will presumably take place under the influence of the stress concentration associated with the crack tip[35,36]. This will favour orientations in which the lamellar surface lies parallel to the advancing crack front. As a consequence fracture is now likely to occur across a nearly perpendicular section of the lamella, so accounting for the appearance of the microstriations. Alternatively fracture could take place in the amorphous material at the end of the lamella, giving rise to the same appearance.

Some microstriations are in evidence in fibrillated regions[13], and although their orientation is predominantly perpendicular to the crack propagation direction some distribution of orientation is seen, presumably caused by modifications to stress field direction corresponding to stress concentrations associated with the fibrils. The detection of microstriations is mostly prevented by the presence of fibrillation in such regions, however. When the crack advances rapidly the effective strain rate in the material ahead of the crack is much higher than with a slowly moving crack, and cold-drawing is consequently prevented, and the high degree of fibrillation lost.

As noted previously, any deformation in the crystalline lamellae that changes their shape or distribution will require the cooperation of the amorphous material, and it is probably significant that microstriations were not observed with specimens fractured at lower temperatures.

The river lines travelling in the direction of crack propagation are probably also determined by the microstructure. Their separation corresponds fairly well to the spherulite diameter and they probably correspond to interspherulitic paths at slightly different levels. Similarly the generally undulating appearance of the fracture surface when tracing paths in the direction of crack propagation may also correspond to interspherulitic boundaries, though plastic deformation prevents positive identification of the kind possible in a recent publication by Friedrich concerning fracture in polypropylene[37].

Two- and three-stage growth behaviour

The crack-growth characteristics for LDPE show two or three distinct stages[12,14]. The fracture surfaces produced by tensile-only testing displayed two distinct morphologies separated by a transition zone that coincided with the intermediate 'stage II' of crack growth[12]. A similar correlation was found in the present work for tests in which the compressive component of strain was small. The correspondence between the transition position on the crack growth characteristics and on the fracture surface was sometimes less exact; this may be connected with the bowed profile of the crack front and the square cross-section of the specimens used in the current investigation. The position of the transition became less easy to detect as the compressive component of the loading programme was increased. It was similarly found[14] that the transition between the different stages in the crack growth characteristics was less sharp than in the earlier study[12].

CONCLUSIONS

In the limited number of polymers investigated here the appearance of the fatigue fracture surface has been found to reflect to a large extent the microstructure and properties of the materials used. In a non-homogeneous polymer such as spherulitic LDPE several deformation mechanisms are available and the one which operates depends upon the temperature of testing and on the stress field near to the crack tip, and therefore on the extent of advancement of the crack. The fracture surface topography may be substantially modified by post-crack damage when a large compressive deformation component is included in the loading programme.

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