

Fatigue fracture processes in polystyrene

M. D. SKIBO, R. W. HERTZBERG, J. A. MANSON

Materials Research Center, Lehigh University, Bethlehem, Pennsylvania, USA

Fatigue crack growth characteristics in polystyrene were studied as a function of stress intensity factor range and cyclic frequency. Precracked single edge notched and compact-tension type specimens made from commercially available polystyrene sheet (mol. wt. = 2.7×10^5) were cycled under constant load at frequencies of 0.1, 1, 10 and 100 Hz, producing growth rates ranging from 4×10^{-7} to 4×10^{-3} cm/cycle. For a given stress intensity level, fatigue crack growth rates were found to decrease with increasing frequency, the effect being strongest at high stress intensity values. The variable frequency sensitivity of this polymer over the test range studied was explained in terms of a variable creep component. The macroscopic appearance of the fracture surface showed two distinct regions. At low stress intensity values, a highly reflective, mirror-like surface was observed which transformed to a rougher, cloudy surface structure with increasing stress intensity level. Raising the test frequency shifted the transition between these areas to higher values of stress intensity. The microscopic appearance of the mirror region revealed evidence of crack propagation through a single craze while the appearance of the rough region indicated crack growth through many crazes, all nominally normal to the applied stress axis. Electron fractographic examination of the mirror region revealed many parallel bands perpendicular to the direction of crack growth, each formed by a discontinuous crack growth process as a result of many fatigue cycles. The size of these bands was found to be consistent with the dimension of the crack tip plastic zone as computed by the Dugdale model. At high stress intensity levels a new set of parallel markings was found in the cloudy region which corresponded to the incremental crack extension for an individual loading cycle.

1. Introduction

The introduction of polymers as replacements for metallic components is becoming of increasing commercial importance. Since many of these parts are subjected to a wide range of loading conditions from simple structural support to complex cyclic loading, it is important that the mechanical response of a polymer be known over similar loading conditions. For many polymers this has primarily been done for static loading conditions, with the cyclic response of most polymers receiving little attention.

To study the phenomenon of fatigue in polymers, a wide variety of laboratory fatigue tests and

specimen geometries are available which allow control of certain external variables. At one extreme, test methods can be devised to examine the thermal fatigue response of a polymer in which the test specimen fails as a result of hysteretic heating. Alternately, the mechanical fatigue behaviour of the polymer can be studied by the initiation and growth of a crack within a specimen. Furthermore, the crack propagation regime can be examined exclusively by precracking the test specimen. The latter approach has received increased attention recently [1, 2] and was adapted in this investigation.

A useful parameter to characterize the fatigue

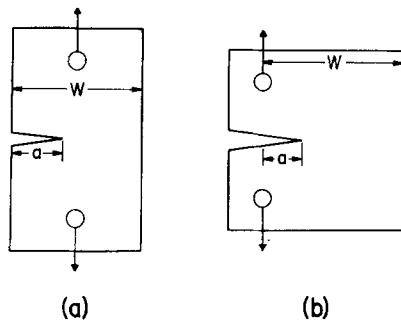


Figure 1 Specimen configurations used to generate fatigue crack propagation data in this study: (a) single edge notched specimen (SEN); (b) compact tension specimen (CT).

crack propagation (FCP) response in notched specimens is the stress intensity factor [1]. Derived from principles of fracture mechanics, the stress intensity factor describes the stress conditions at the tip of the advancing crack and is defined by

$$K = Y\sigma\sqrt{a} \quad (1)$$

where K is the stress intensity factor, Y the correction for specimen geometry, σ the stress, and a the crack length.

Fatigue crack propagation data are generated by cycling notched samples such as the two shown in Fig. 1 within a constant load range and recording crack growth over the corresponding number of fatigue cycles. Under fatigue conditions K will also vary over a range defined by $\Delta K = K_{\max} - K_{\min}$. As the crack length, a , increases, ΔK will also increase, enabling the measurement of the rate of crack growth per cycle, da/dn , over a large range of ΔK . Emphasizing the strong relationship between ΔK and da/dn , Fig. 2 shows crack growth rates over a range of ΔK values for several polymers having different molecular structures and mechanical properties [1, 2]. It has been shown [3], that such a relationship between da/dn and ΔK could be best represented by a simple equation of the form:

$$\frac{da}{dn} = A\Delta K^m \quad (2)$$

where A and m are constants for a given polymer.

Using this relationship between da/dn and ΔK , it is possible to analyse the FCP response of a polymer as a function of various external variables, one of the most important being the test frequency. A recent review of the effect of frequency on FCP in polymers has shown a large variation in response among different polymers [2]. For example, poly-

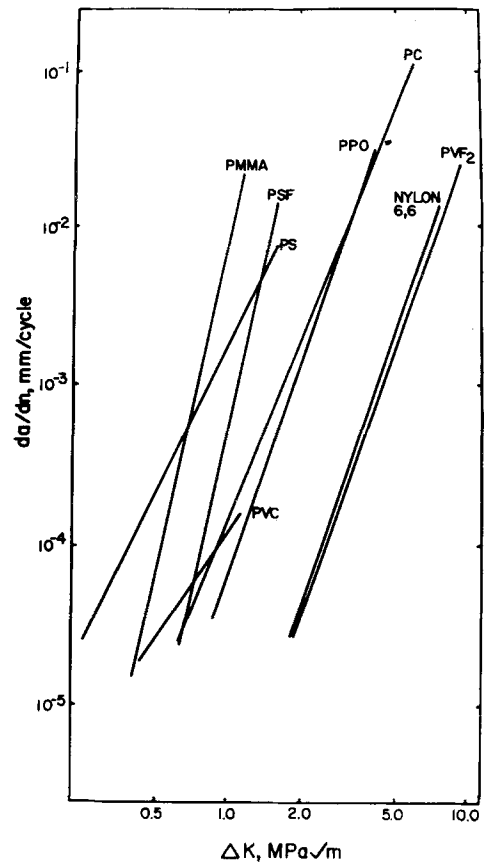


Figure 2 Relationship between crack growth rate per cycle in several polymers as a function of stress intensity factor range ΔK .

(methylmethacrylate) (PMMA) was shown to exhibit a three-fold decrease in FCP rate per decade increase in test frequency while growth rates in polycarbonate (PC) and most crystalline polymers were insensitive to frequency. It is believed that the characteristic frequency dependence of a given polymer depends on a number of factors, two of the most important being strain-rate sensitivity and creep. In some polymers, increasing the strain-rate by increasing the test frequency produces a corresponding increase in yield strength and modulus which may be responsible for the observed decrease in da/dn . In polymers that creep rapidly under load, crack growth by creep may be considerable with time and might be responsible for higher da/dn at low frequencies. A third factor believed to influence the frequency sensitivity of a polymer is the existence of polymer crazing. A considerable frequency sensitivity is found in those polymers which can form crazes easily [2].

While crack growth data are very useful in

characterizing the fatigue response of a polymer, much additional insight may be gained by an analysis of the micro-mechanisms of crack growth. This can best be accomplished by a study of the fatigue fracture surface. The use of fractography in polymers is not new since many detailed studies have been performed under monotonic loading conditions which identified the important role of crazing in the static fracture process [4–7]; however, fractographic studies of fatigue fracture are generally sparse. A common feature of the fatigue fracture surface of those polymers studied has been the presence of distinct parallel bands oriented perpendicular to the direction of crack growth. In PMMA and PC, each band was found [1, 8, 9] to be related to a single load cycle while in other polymers, PVC [1, 10, 11] and polystyrene-polyurethane (PS-PU) [12], these bands clearly were formed over hundreds of fatigue cycles and by a different mechanism than the former. Other researchers [13, 14] have reported bands on the fracture surface of unnotched PS samples; however, without accurate growth rate data, it is not known whether the bands were formed as a result of 1 or 1000 cycles. This problem will be discussed further in this paper.

The polymeric material chosen for this analysis of fatigue fracture was polystyrene (PS). The mechanical properties of pure polystyrene are typical of glassy polymers; the modulus and strength are high and the toughness low, indicating nearly elastic behaviour over the test temperature range.

Most previous fatigue studies [13, 14] on PS have been performed on unnotched specimens although some FCP data covering a small frequency range have been reported [1]. From Fig. 2, the FCP behaviour of PS compares poorly with respect to many other polymeric materials.

It is the purpose of this paper to describe the effect of frequency on the FCP behaviour of PS and, based on a study of the micromorphology of the fracture surface, to propose mechanisms for fatigue fracture.

2. Experimental procedure

The commercial polystyrene used in this study was obtained from Cadillac Plastics in the form of a sheet 6.35 mm thick. The molecular weight determined through viscosity measurements was 2.7×10^5 . All specimens used in the generation of fatigue data in this paper were oriented in the same direc-

tion in the sheet. Additional specimens tested normal to this direction revealed no anisotropy in FCP behaviour. Test samples were either single edge notched (SEN) (Fig. 1a) or compact tension (CT) (Fig. 1b) for which the stress intensity range ΔK is given by [15]

$$\Delta K = \frac{Y\Delta P\sqrt{a}}{BW}$$

where ΔP is the load range, B the specimen thickness, W the specimen width, a the crack length, and

$$Y = 1.99 - 0.041(a/w) + 18.70(a/w)^2 - 38.48(a/w)^3 + 53.85(a/w)^4 \text{ for SEN}$$

$$Y = 29.6 - 185.5(a/w) + 655.7(a/w)^2 - 1017(a/w)^3 + 638.9(a/w)^4 \text{ for CT.}$$

All fatigue tests were performed on an electro-hydraulic closed loop testing machine covering a test frequency range of 0.1 to 100 Hz. Crack growth rates were monitored with the aid of a travelling microscope in increments of approximately 0.25 mm. Unless specified differently, all fatigue tests were run in laboratory air.

Fracture studies were conducted with an optical metallograph, a Philips EM300 transmission electron microscope (TEM) and an ETEC scanning electron microscope (SEM). Specimens prepared for use in the SEM were gold coated. Replicas for TEM observation were made by first replicating the fracture surface with a 10% solution of poly(acrylic acid) in water. After allowing sufficient time for drying, the plastic replica was stripped from the specimen, chromium shadowed, and carbon coated. Finally, the poly(acrylic acid) was dissolved leaving the shadowed carbon film ready to be mounted for viewing.

3. Experimental results and discussion

To better understand the overall effect of frequency on FCP behaviour in PS, it was necessary to obtain crack growth rates over a wide range of test frequency and ΔK . From Fig. 3, the data base developed during the course of this investigation may be seen to span a four decade change in growth rates obtained over a three decade range of test frequency. To the authors' knowledge, these data may well represent the most extensive range of fatigue crack propagation data available for any one polymer. Over an intermediate growth rate range (1.3×10^{-5} to 2.5×10^{-4} mm cycle), the slopes of

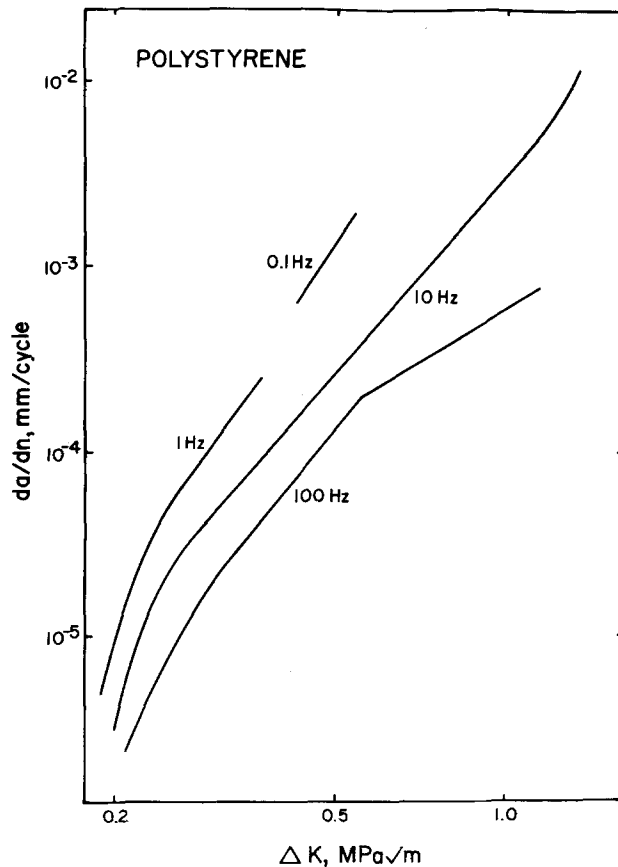


Figure 3 Effect of cyclic frequency on fatigue crack propagation in polystyrene.

the growth rate curves for different frequencies are approximately equal to 2.6. In this range crack growth rates decrease by a factor of 2.2 for every ten-fold increase in frequency. However, below this range the data converge (i.e. FCP rates become less sensitive to test frequency) while above this range frequency has an increasingly important role in determining crack growth rates. This fatigue behaviour is common to several polymers sensitive to frequency and was postulated by the authors [2] to be related to the existence of a variable creep component. It has been reasoned that fatigue crack growth rates may be considered to be the sum of a pure fatigue component da/dn_{fat} and a pure viscous creep component da/dt_{cr} where

$$da/dn_{tot} = da/dn_{fat} + da/dt_{cr}$$

At low ΔK where da/dt is small,

$$da/dn_{tot} \approx da/dn_{fat}$$

which accounts for the low frequency sensitivity in this test range. At high ΔK where da/dt is large, the latter component represents a greater portion

of da/dn_{tot} , thus explaining the increased frequency sensitivity. At intermediate levels of ΔK , da/dn_{fat} and da/dt_{cr} are presumed to be of comparable magnitude, thereby accounting for the simple displacement of the crack growth rate curves with changing test frequency.

While the creep-fatigue superposition model does provide a plausible explanation for the observed frequency behaviour, it is important to recognize a potential effect of the crack tip environment on crack growth which, if present, would account for the observed FCP response found in PS. Although most fatigue tests were performed in laboratory air, a preliminary comparison of growth rates was made with tests performed in a high purity nitrogen environment. No significant difference in growth rates between the two conditions was observed leading us to believe that environmental factors were not responsible for the observed frequency sensitivity of this material.

3.1. Fracture mode transition

An examination of the fatigue fracture surface of

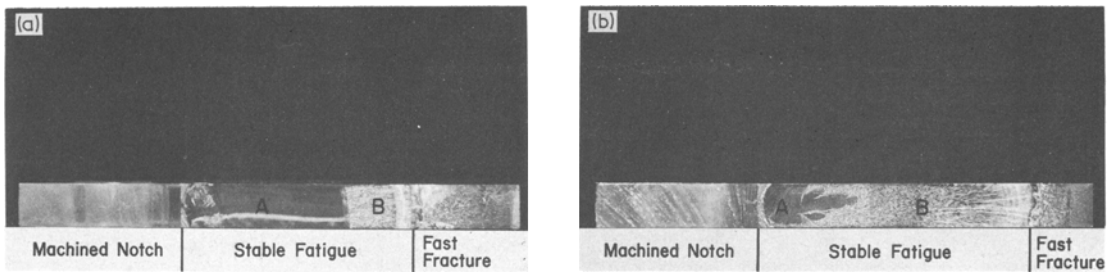


Figure 4 Macroscopic view of fracture surface of polystyrene showing “mirror” (A) to “mist” (B) transition at (a) 100 Hz and (b) 10 Hz.

PS revealed a striking transformation in macro-morphology (Fig. 4). As previously observed in PS [13, 14, 16, 17] and other glassy materials, the fracture surface changed from a smooth, mirror-like surface to misty appearance at test frequencies of 1, 10 and 100 Hz. When the fatigue crack was viewed in profile (Fig. 5), it became possible to identify the essential difference between the mirror and mist regions. Recognizing that crack growth in polystyrene is always preceded by a craze, the smooth appearance of the crack in the mirror region was found to reflect crack growth through a single craze. At higher ΔK levels, more energy is available for craze formation thereby causing the development of multiple crazing on either side of the crack plane. Crack growth through these craze bundles was found to produce a rough misty surface with the crack jumping from one craze to another. The mechanism for craze bundle initiation is uncertain; however, in all cases it occurred initially at the surface of the fatigue specimen as shown in Fig. 4 and progressed inward toward the mid-plane of the sample with increasing ΔK until the transition to mist was completed. A possible explanation for this behaviour might be related to the presence of imperfections and scratches on the surface of the specimen which act as initiation sites for craze bundling. The presence of residual tensile stresses

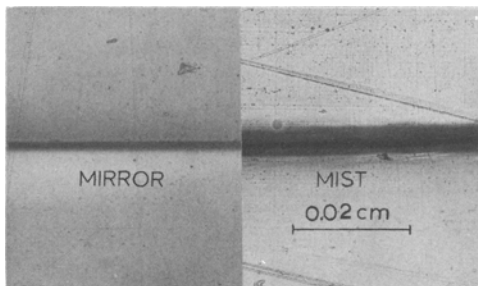


Figure 5 Comparison of the fatigue crack profile in polystyrene for “mirror” and “mist” regions. Note craze bundling in “mist” region.

at the surface of the specimen could also contribute to craze bundle formation. (No attempt was made in this investigation to identify such residual stresses.)

Similar to the crack growth rate test results, this macroscopic texture transition was found also to be sensitive to both ΔK and frequency, as shown in Fig. 6. At 100 Hz the transformation occurred abruptly at a ΔK of approximately 0.53 $\text{MPa}\sqrt{\text{m}}$, (Fig. 4a) while at 10 Hz and below, the

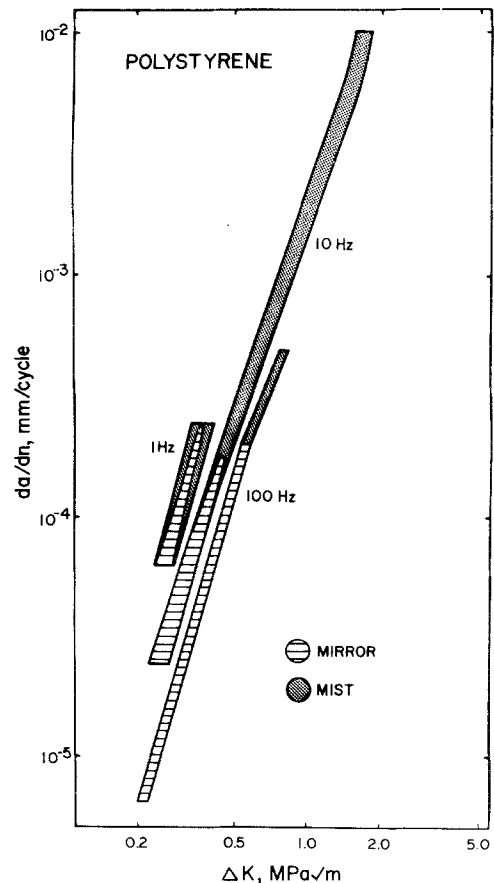


Figure 6 Effect of cyclic frequency on the stress intensity at which the “mirror” – “mist” transition occurs.

transition occurred over a large range of ΔK and terminated at $0.42 \text{ MPa}\sqrt{\text{m}}$. Since the initiation and growth of crazes is time dependent, the rapid loading associated with testing at 100 Hz might have been sufficient to suppress the initiation and growth of multiple crazing, thereby contributing to the sudden mirror-mist transition. Below 10 Hz the rate of loading is most likely slow enough to permit the initiation and growth of craze bundling at a lower ΔK thereby allowing for a more gradual texture transition. This behaviour was further analysed by running a fatigue test at a frequency of 1 Hz and allowing the mirror to mist region to come to completion at $\Delta K = 0.42 \text{ MPa}\sqrt{\text{m}}$. At this point the frequency was immediately changed to 100 Hz. It was found that the fracture surface remained misty in appearance even though the mirror morphology would have been stable to an even higher ΔK had the test been begun at 100 Hz. Only by decreasing the stress intensity level below the equilibrium transition ΔK for 1 and 10 Hz ($0.42 \text{ MPa}\sqrt{\text{m}}$) was it possible to reverse the transformation and obtain a mirror morphology. It is believed that by lowering the ΔK range, the plastic zone dimension ahead of the crack is reduced so that single craze growth could be stabilized again. Once the crack propagates through the existing craze bundles, the mirror appearance returns.

It is interesting to note that the texture transition does have an effect on the observed fatigue crack growth rate. From Fig. 6 it is seen that this transition causes an abrupt decrease in the slope of the 100 Hz growth rate curve from 2.6 to 2, an indication of a decrease in sensitivity of growth rates to ΔK . A possible explanation for this behaviour is that the growth of craze bundles ahead

of the crack requires more energy than for a single craze; therefore, less energy is available to drive the crack.

3.2. Fatigue fracture surface analysis

A fractographic study of PS proved to be particularly rewarding, although care was required when using the TEM so as not to misinterpret certain artifacts produced during the replication procedure (Appendix). In the mirror region at low values of ΔK at 10 and 100 Hz it was possible to view many parallel bands oriented perpendicular to the direction of growth (Fig. 7) which gradually increased in size with ΔK as shown in Fig. 8. A

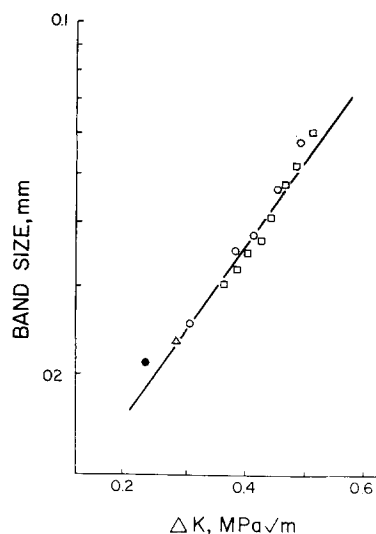


Figure 8 Increase in size of discontinuous crack growth bands with stress intensity.

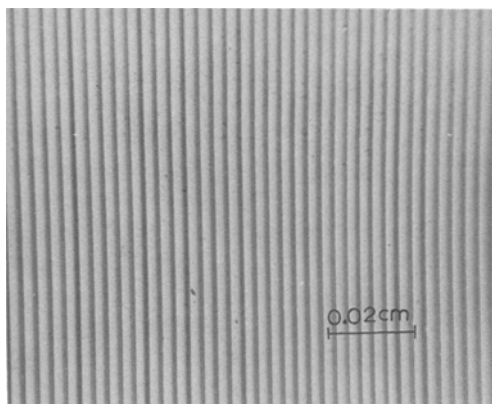


Figure 7 Discontinuous crack growth bands in the "mirror" region of the fatigue fracture surface of polystyrene.

comparison of the size of these bands and the growth rate per cycle from macroscopic growth data revealed that each band was not formed during a single load excursion, but was formed instead over many cycles ranging from 1400 to 600 cycles per band. Similar bands formed as a result of many loading cycles were observed previously in PVC [10, 11], PS-PU [12], PMMA, PC and polysulfone [18].

From Fig. 9, the morphology of each band is found to contain a gradient of void sizes ranging from $2 \mu\text{m}$ to $< 0.1 \mu\text{m}$ in the direction of crack growth. In a study of bands of similar structure in PVC, Hertzberg and Manson [11] demonstrated these bands to be formed by a discontinuous

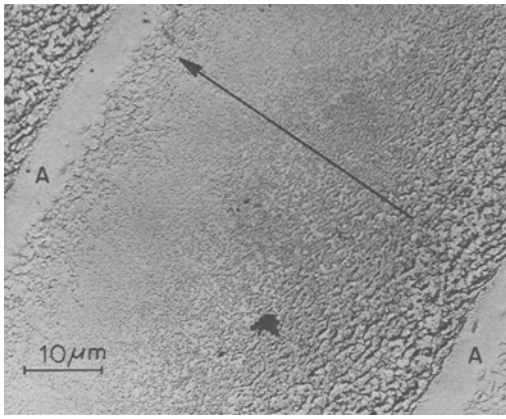


Figure 9 Transmission electron micrograph revealing morphology of discontinuous crack growth bands. Note decrease in void size within one growth band in direction of crack propagation (arrow). Stretch zones associated with crack arrest are seen at A.

growth process shown schematically in Fig. 10. They concluded that under cyclic loading conditions, a single craze would grow to a limiting size. With further cycling the craze would weaken and allow the crack to propagate rapidly to the craze tip. Such rapid fracture would occur by a void coalescence mechanism with the void size distribution reflecting the internal structure of the craze just prior to crack extension. Blunting, represented by the distinct stretch zone following each band (see region A in Fig. 9), and crack arrest then would occur when the crack encountered homogeneous uncrazed material.

Using the Dugdale [19] plastic strip model [20–22].

$$r_y \approx \frac{\pi K^2}{8 \sigma_{ys}^2},$$

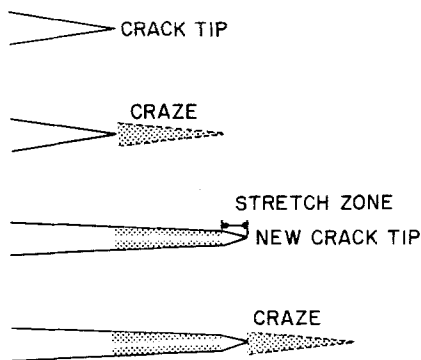


Figure 10 Schematic representation of discontinuous crack growth.

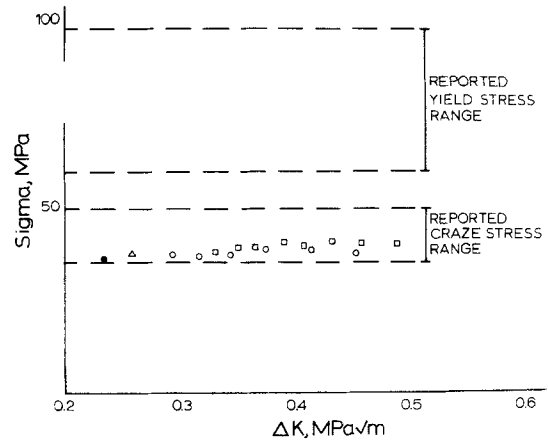


Figure 11 Relationship between inferred yield stress from discontinuous growth band measurements and stress intensity range. Also shown are yield and craze stress values for polystyrene as reported in the literature [13, 23, 24].

the yield strength σ_{ys} can be computed for different K levels by equating the plastic zone size to the width of the fracture surface bands.

Assuming that the limiting length of the craze formed ahead of the crack during fatigue crack growth is accurately represented by the size of these bands on the fracture surface, the change in craze length with ΔK may be recorded by measuring the band size over a range of crack lengths. This was done in the preparation of Fig. 8. One may also assume that the craze size represents the extent of the plastic deformation zone ahead of the crack (i.e. the plastic zone size, r_y). It is then possible to speculate that the band size is equivalent to r_y from which one can infer an effective yield strength for PS. A similar analysis was employed by Elinck *et al.* [10] with some success for the case of PVC.

Fig. 11 shows the inferred yield strength calculated from band size measurements taken from several different fatigue specimens over a range of ΔK values. Also shown in Fig. 11 are the experimentally determined ranges for the craze stress and yield stress which were taken from the literature [13, 23, 24]. It is important to note that all data fall within the boundaries of the craze stress range implying that the stress controlling the formation of the plastic zone ahead of the crack is the craze stress and not the nominal yield stress. Since a craze is always observed to precede a crack, this appears to be a logical conclusion.

At higher ΔK , though still in the mirror region, the discontinuous growth bands changed abruptly

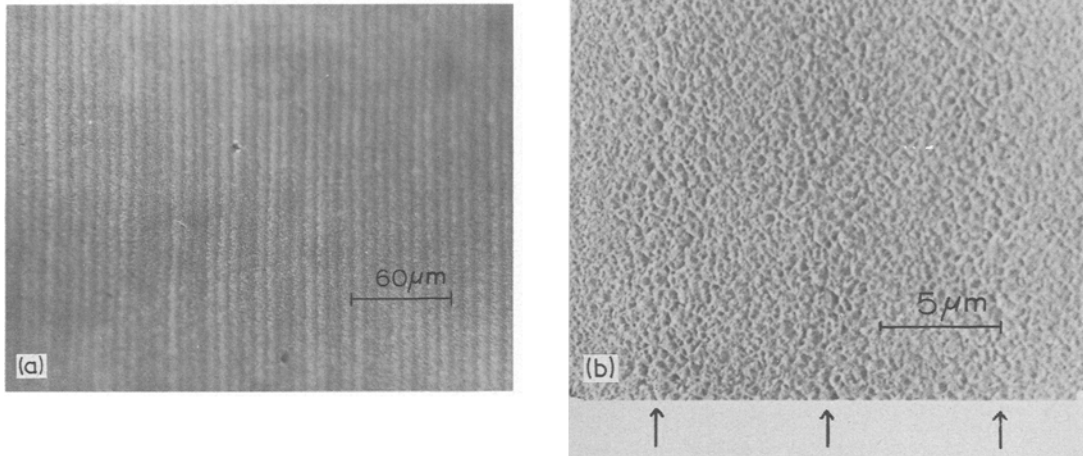


Figure 12 (a) Constant size parallel bands in the “mirror” region of the fatigue fracture surface. (b) Moderate change in void size is responsible for bands being observed. Band width given by distance between arrows.

to another series of parallel bands shown in Fig. 12. These markings were also found to be oriented perpendicular to the direction of growth. They are similar to the discontinuous growth bands in that they are formed over many load cycles (140 to 35 per band); however, unlike the discontinuous growth bands, these bands have a relatively constant size of 6 to 7 μm over a range of growth rates from 5×10^{-5} to 2×10^{-4} mm/cycle. At higher magnification (Fig. 12b) these bands are seen to be composed primarily of small voids whose small variation in size, 0.6 to 0.2 μm , is responsible for their periodic lineage. The mechanism for the formation of these bands is uncertain at this time and the analysis further complicated by their presence beyond the end of the mirror region and beneath the rough but transparent surface of the mist region. This indicates that these bands represent more than just a surface feature. A possible explanation for the surface morphology of the constant band size region is suggested from the study of Beahan *et al.* [25] who examined craze morphology in thin films and bulk specimens of polystyrene. They observed in thin films that craze width and length increased uniformly with stress to a point above which a mid-rib plane within the craze would develop. This plane was found to contain an array of relatively uniform voids separated by fine fibrils. Similar behaviour was expected for crazes in bulk specimens but lack of resolution of the details of the craze surface prevented verifi-

cation of this fact. Assuming this mechanism to be active in bulk PS specimens, especially under cyclic load conditions, one might expect that as the crazes grow longer and thicker with ΔK in the discontinuous growth region, a craze thickness will be reached at which this weak mid-rib can form. This weakened region would then form an easy path for crack propagation thereby ending discontinuous crack growth. The uniform voids of the midrib would account for the nearly constant void size observed in the constant band size and lend support to this hypothesis. Another fractographic feature found only in the vicinity of the constant size bands was a third series of bands. While the exact mechanism for the formation of these bands was not identified conclusively, they were found to be oriented at $\pm 45^\circ$ to the direction of growth and increased in number with ΔK (Fig. 13a). They emanated from the mist asperities at the sides of the fracture surface and involved a shear displacement process (Fig. 13b). In a macroscopic sense, these bands differ from those observed by others [26–28] which were found to extend throughout the entire specimen under conditions of gross yielding. In the current study, the shear bands were contained within the crack tip plastic zone which extended below the fracture surface only to a maximum depth of about 50 μm .

Continuation of our study of the fracture surface into the mist region revealed the presence of fine voids similar to those associated with the

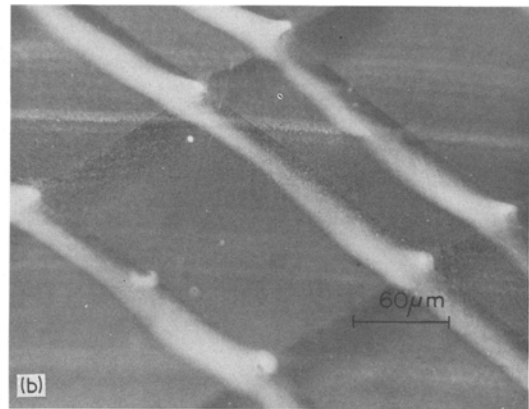
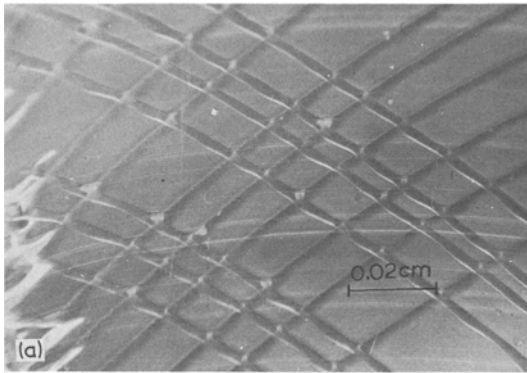


Figure 13 Bands formed oriented 45° to the growth direction in the “mirror” region on the fatigue fracture surface. (a) Note initiation of bands from “mist”, region at left. (b) Evidence of shear deformation.

constant size bands found in the mirror region. In other areas larger voids as shown in Fig. 14 were noted. Also seen quite frequently but only in the mist region were long ridges parallel to the direction of crack growth (Fig. 15). These ridges which contain a number of ledges represent the intersection of the crack plane with one or more crazes in craze bundles found only in the mist region. The ledge height was measured over a range of ΔK levels (0.66 to 1.00 $\text{MPa}\sqrt{\text{m}}$) and found to be about $6\ \mu\text{m}$. Since the walls between the ledges contained highly oriented fibrillar material, it is concluded that the craze thickness in this ΔK range is constant and equal to about $6\ \mu\text{m}$. Also Kambour [29] previously found the craze thickness in PS to reach a limiting size of about $6\ \mu\text{m}$.

At higher ΔK levels in the mist region (above growth rates of $9 \times 10^{-4}\ \text{mm/cycle}$) another set of parallel bands oriented perpendicular to the direc-

tion of growth were visible. From Fig. 16, these bands appear as shallow undulations with a substructure comprised of fine voids, $0.5\ \mu\text{m}$ in diameter, and similar to those found elsewhere on the fracture surface. The size of these bands increased with ΔK and were in good agreement with the macroscopic growth rates corresponding to the same ΔK conditions (Fig. 17). This indicates a 1:1 correlation between the bands and the loading cycles, proving that these bands are classical fatigue striations.

Thus, with the identification of the fatigue striation, a total of three types of parallel bands are found on the fracture surface of PS, each formed by a different mechanism. Consequently, without accurate growth rate data it is impossible to safely

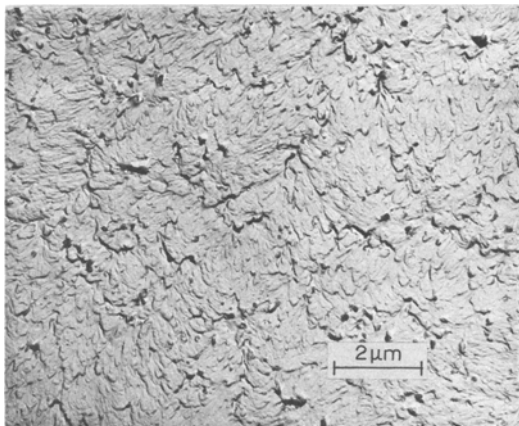


Figure 14 Transmission electron micrograph revealing layer void clusters found occasionally in “mist” region.

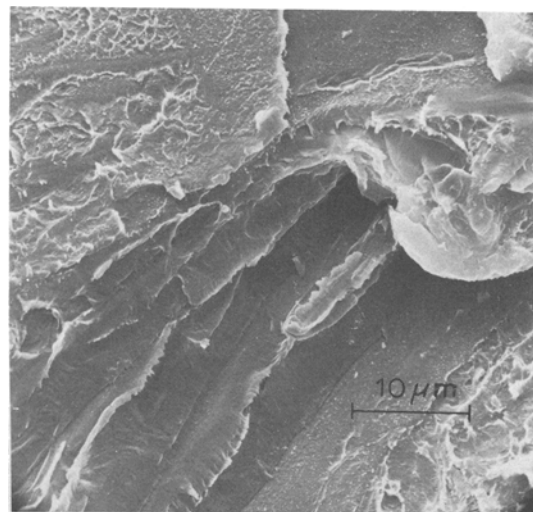


Figure 15 Ridge wall found in “mist” region. Note multiple levels of oriented material in this scanning electron micrograph.

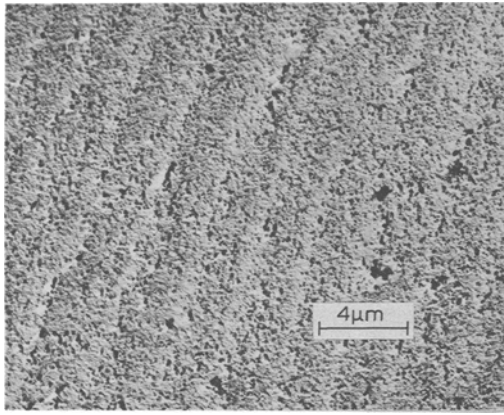


Figure 16 Parallel fatigue striations found in the “mist” region corresponding to incremental advance of crack after one load excursion. Transmission electron micrograph.

relate “growth band spacings” measured on PS fracture surfaces to the advance of the crack during one load cycle. From the present study, a growth band in PS could represent crack extension after one load cycle or after over 1000 cycles. It is highly probable that Havlicek *et al.* [14] and Rabinowitz *et al.* [13] misinterpreted the bands in the mirror region of their unnotched fatigue specimens as being true fatigue striations which occur after one loading cycle. Rather, these bands appear to be the type related to the plastic zone dimension. As a result, the propagation portion of the fatigue life

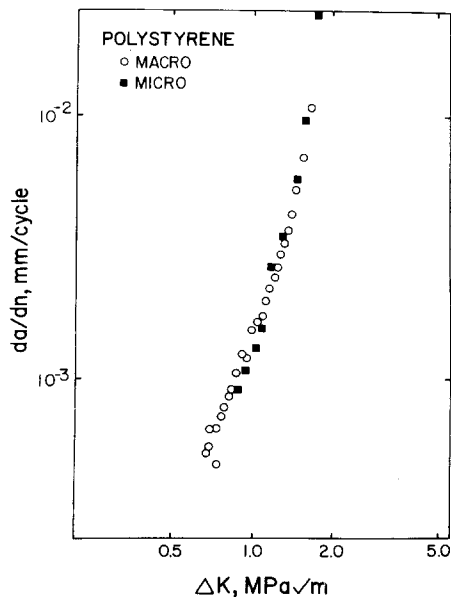


Figure 17 Comparison of macroscopic growth rates and striation measurements.

reported in these investigations was probably as much as 1000 times greater than originally computed. This confusion encountered in interpreting fatigue behaviour underscores some of the shortcomings of using unnotched fatigue specimens. In addition to the inability to measure crack growth rates in these specimens, it is also impossible to separate crack initiation from crack growth; hence, much valuable information is lost.

4. Conclusion

Increasing test frequency served to decrease crack growth rates, an effect which becomes stronger with increasing ΔK and which can be rationalized by the creep and fatigue superposition model. PS undergoes a fracture mode transition from mirror to mist morphology reflecting a transformation from FCP through a single craze to FCP through craze bundles, respectively. This transition can be partially suppressed by increased frequency until a higher ΔK is reached. The effect of this transition on FCP response is only observed at 100 Hz where a decrease in the slope of the growth rate curve indicates a decrease in sensitivity of crack growth to ΔK . From observations of bands formed over hundreds of fatigue cycles in the mirror region, it is apparent that discontinuous crack growth is the favoured mechanism for FCP at low values of ΔK . The size of these bands were shown to be equivalent to the plastic zone dimension, assuming the effective yield strength ahead of the crack to be the stress for crazing. At high ΔK levels, continuous crack growth is noted by the presence of fatigue striations on the fracture surface with one striation corresponding to one loading cycle.

Appendix. Possible artifacts results from replicated fracture surfaces

One frustrating aspect of the fractographic study of PS involved the danger of misinterpreting artifacts as true fracture markings on fracture surfaces prepared by replication. The use of 10% poly(acrylic acid) in water as a replicating agent is considered chemically harmless to most polymers; however, SEM fractographs of several previously replicated and non-replicated fracture surfaces revealed strong differences, indicating evidence of physical damage in the replicated specimens. For example, Fig. 18a and b are SEM fractographs of the mist region at low ΔK of a specimen which had been replicated twice showing large holes where material had obviously been ripped out.

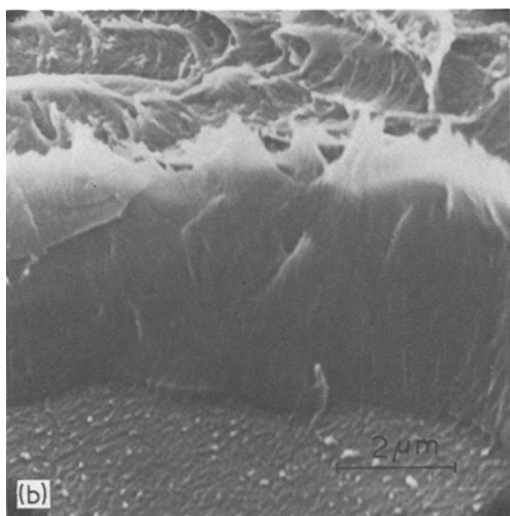
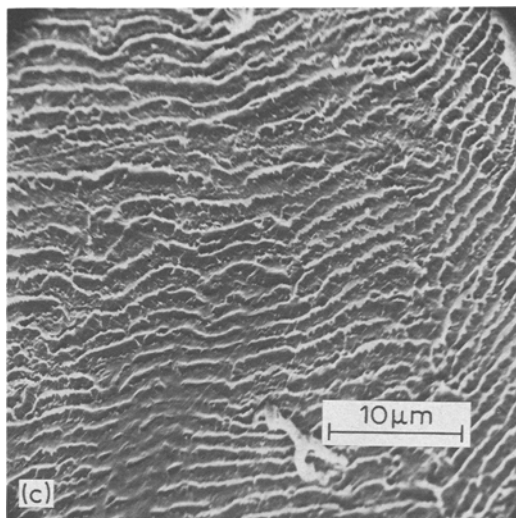
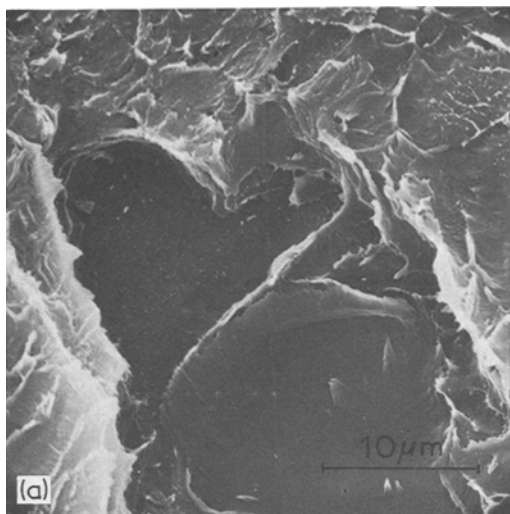


Figure 18 Scanning electron micrographs of previously replicated surfaces showing possible artifacts introduced by mechanical damage during the replication procedure. (a) Large holes showing highly oriented walls; (b) higher magnification of walls; (c) large areas of scalloped striations.

under monotonic test conditions [5, 6], they appear not to represent the cyclic fracture process examined in this investigation. Consequently, to prevent misinterpretations of the fracture surface morphology, many of the TEM fractographs were disregarded. We suggest discretion be used in analysing fracture surface detail by TEM study of two-stage replicas unless an SEM is available to corroborate the replica results. If at all possible fractography of polymers that craze should be done with an SEM.

Also visible in this same specimen were beautifully formed scalloped striations shown in Fig. 18c. None of these morphological features occurred in any other unreplicated specimens, even though all testing conditions were the same. Other examples of physical damage were visible in the region of constant size bands. After stripping the replica from the surface, many colourful fringes were visible. In a previous discussion, it was suggested that in this region fatigue crack growth occurs through a weak mid-rib of a craze. This leaves one-half of the craze on the fracture surface. Stripping the replica can loosen the craze at the craze matrix interface and create an air wedge of variable thickness. The colour fringes would then be formed by selective and destructive interference of light by the air wedge. While some of the features found on replicated surfaces (e.g. Fig. 18a) have been found

Acknowledgements

The authors wish to acknowledge the financial support of the Army Research Office – Durham Grant DAHC04 7460010. They are also appreciative of the assistance given by Dr Soojaa L. Kim in the determination of the molecular weight of PS.

References

1. J. A. MANSON and R. W. HERTZBERG, *CRC Rev. Mac. Sci.* **1** (1973) 433.
2. R. W. HERTZBERG, J. A. MANSON and M. D. SKIBO, *Polymer Eng. Sci.* **15** (1975) 252.
3. P. PARIS and F. ERDOGAN, *J. Basic Eng. Trans. ASME, Series D* **85** (1963) 528.
4. R. P. KAMBOUR, *Polymer Eng. Sci.* **8** (1968) 281.
5. D. HULL, *J. Mater. Sci.* **5** (1970) 357.
6. J. MURRAY and D. HULL, *Polymer* **10** (1969) 451.

7. *Idem*, *Polymer Letters* 8 (1970) 159.
8. G. H. JACOBY, ASTM STP 453 (1969) p. 147.
9. G. H. JACOBY and C. CRAMER, Off. of Nav. Res., A.F. Mat. Lab., Proj. NR064-470 (1967).
10. J. P. ELINCK, J. C. BAUWENS and G. HOMES, *Int. J. Frac. Mech.* 7 (1971) 227.
11. R. W. HERTZBERG and J. A. MANSON, *J. Mater. Sci.* 8 (1973) 1554.
12. J. S. HARRIS and I. M. WARD, *ibid* 8 (1973) 1655.
13. S. RABINOWITZ, A. R. KRAUSE and P. BEARDMORE, *ibid* 8 (1973) 11.
14. V. HAVLICEK and V. ZILVAR, *J. Macromol. Sci.* B5 (1971) 317.
15. W. F. BROWN, JUN. and J. E. SRAWLEY, ASTM STP 410 (1966).
16. A. D. MCMASTER and D. R. MORROW, *Polymer Eng. Sci.* 14 (1974) 801.
17. G. P. MARSHALL, L. E. CULVER and J. G. WILLIAMS, *Int. J. Fracture* 9 (1973) 295.
18. M. D. SKIBO, R. W. HERTZBERG and J. A. MANSON, to be published.
19. D. S. DUGDALE, *J. Mech. Phys. Solids* 8 (1960) 100.
20. N. J. MILLS, *Eng. Fract. Mech.* 6 (1974) 537.
21. R. D. R. GALES and N. J. MILLS, *Eng. Fract. Mech.* 6 (1974) 93.
22. H. F. BRINSON, *Proc. Soc. Exp. Stress Anal.* 27 (1970) 72.
23. R. N. HAWARD, B. M. MURPHY and E. F. T. WHITE, *J. Polymer Sci. A-2* 9 (1971) 801.
24. *Idem*, Proceedings of the Second International Conference on Fracture (Chapman and Hall, London, 1969) p. 519.
25. P. BEAHAN, M. BEVIS and D. HULL, *J. Mater. Sci.* 8 (1972) 162.
26. L. CAMWELL and D. HULL, *Phil. Mag. Ser. 8* 27 (1973) 1135.
27. A. S. ARGON, R. D. ANDREWS, J. A. GODRICK and W. WHITNEY, *JAP* 39 (1968) 1899.
28. T. E. BRADY and G. S. Y. YEH, *J. Mater. Sci.* 8 (1973) 1083.
29. R. P. KAMBOUR, *J. Polymer Sci. A-2* 4 (1966) 349.

Received 23 May and accepted 1 September 1975.