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Fatigue crack propagation in polyacetal

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The fatigue crack propagation characteristics of a typical commercial homopolymer and copolymer polyacetal were determined. These materials were found to be the most fatigue resistant plastics examined to date, thus confirming the generally high fatigue resistance of all crystalline polymers. A discontinuous fatigue cracking process was identified at all test frequencies in the acetal copolymer and at high frequencies in the homopolymer, while continuous crack propagation occurred at low test frequencies in the homopolymer. The discrete advance increments of the crack in the discontinuous mode were equal to the dimension of the prevailing crack-tip plastic zone. On a more local scale, the crack path is seen to be mainly trans-spherulitic in nature.

1. Introduction

In recent years a considerable body of data has been generated regarding the fatigue crack propagation (FCP) characteristics of both amorphous and crystalline polymers [1]. In all reported cases, crystalline polymers proved, as a class, to be most resistant to the propagation of fatigue cracks. For example, at a given level of stress intensity factor, ΔK, nylon 66 and polyvinylidene fluoride (PVDF) exhibited crack growth rates several orders of magnitude lower than observed for amorphous polymers such as polystyrene, polymethyl methacrylate, polysulfone, and polyvinyl chloride. Previously [2], attempts were made to determine whether or not this generalization of superior FCP resistance in crystalline polymers applied to polyacetal (PA). However, existing test procedures proved to be inadequate for the FCP characterization of these materials; it was not possible to generate stable fatigue cracks from machined notch roots prior to failure of the entire specimen. Since others had previously reported (for the case of unnotched samples) that PA was extremely fatigue resistant [3], additional attempts using an improved notching technique were made to examine the FCP behaviour. In this paper, results of this new study are described and discussed.

2. Experimental procedure

In initial attempts to obtain stable FCP data, cyclic tests were conducted at a load range thought sufficient to grow a crack from a machined notch root. After failure to detect any noticeable crack development after many thousands of cycles, the load range was increased arbitrarily by 10%. After another futile period of cycling, the load range was increased once again. Unfortunately, after this sequence had been repeated several times, the sample fractured catastrophically upon loading to the next level without generating a stable fatigue crack.

It was thought that if the starting notch root could be made sharper, it would be possible to initiate a crack more readily, and to follow subsequent crack growth. The following technique was developed and shown to achieve this objective. A sharp scalpel was placed at the notch root and drawn back and forth *while the specimen was being fatigue-cycled.* This simple procedure produced fatigue cracks readily. Since most of the testing was conducted at test frequencies of from 10 to 50 Hz, the crack initiation stage was completed in every sample within a few minutes. This simple procedure has since been employed to develop sharp notches in brittle, densely crosslinked epoxy resins and to enable FCP data to be generated in these as well as less brittle polymeric materials [4].

Two commercial polyacetal resins, Delrin and Celcon, were examined in this study. While both polymers have the $-CH_2-O$ group as the repeating unit, they are not otherwise identical [5]. Thus, Delrin consists of the repeat units mentioned, with the ends of the molecules reacted with a chain-capping reagent in order to enhance thermal stability. In Celcon, on the other hand, thermal stability is achieved by copolymerization with other monomers such as ethylene oxide.

All fatigue tests were conducted with compact tensile-type specimens. The stress intensity factor range for this configuration is [6].

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

where $\Delta K = K_{max} - K_{min}$

$Y = 29.6 - 185.5\,(a/W) + 655.7\,(a/W)^2 - 1017\,(a/W)^3 + 638.9\,(a/W)^4$

ΔP = load range

a = specimen length

B = specimen thickness

W = specimen width.

Fatigue tests were conducted in a 9 kN MTS electrohydraulic closed-loop testing machine at cyclic (sinusoidal) frequencies of 10, 50, and 100 Hz. The ratio of minimum to maximum loads was held constant at 0.1 for all tests. Crack advance was measured in increments of approximately 0.25 mm with an optical travelling microscope. The fracture toughness, K_c, for each sample was estimated by noting the final K_{max} value prior to unstable fracture. All specimens were tested in a laboratory air environment at room temperature.

3. Experimental results

The fatigue crack propagation test results for Celcon are given in Fig. 1a. For two reasons, the FCP data covered a relatively narrow range of ΔK. First, the relatively low K_c value of the material (about 2.5 MPa m$^{1/2}$) precluded tests at higher ΔK levels. Secondly, tests conducted at ΔK levels lower than reported here would have been unduly long and costly, even at 50 Hz.

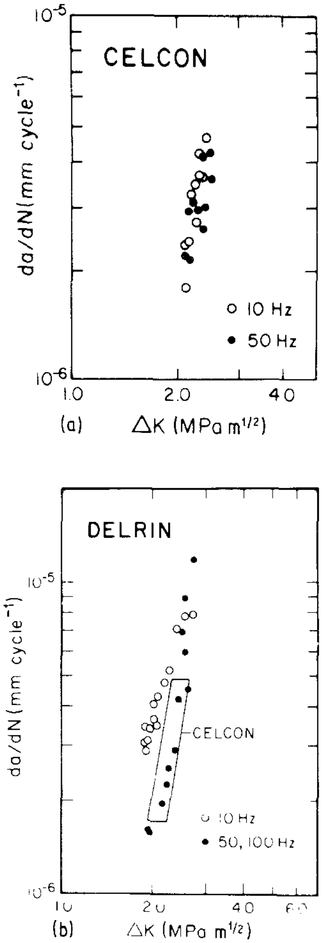

Figure 1 Fatigue crack growth rates as a function of frequency in (a) Celcon and (b) Delrin. Note excellent agreement between Celcon data band and high frequency Delrin data.

In previous studies with crystalline polymers essentially no sensitivity of FCP to frequency was shown by polymers such as nylon 6,6 and PVDF [7]. Similarly, no change in crack growth rate was detected when the test frequency in Celcon was changed from 10 to 50 Hz. Of particular interest was the fact that both samples possessed coarse bands on the fracture surface which were oriented parallel to the advancing fatigue crack front (Fig. 2). The significance of these bands will be discussed at greater length in the next section.

The fatigue test results for Delrin are shown in Fig. 1b. Apart from the fact that the fracture toughness of Delrin proved to be slightly higher (about 2.8 MPa m$^{1/2}$) than that found for the case of Celcon (as is the case with impact strengths [5]) the major difference in the response of this

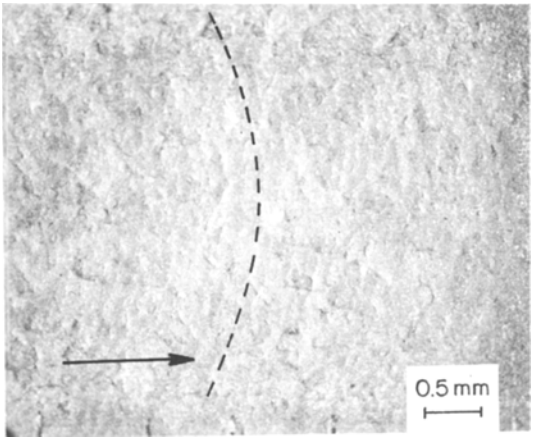

Figure 2 Fractograph of Delrin (50, 100 Hz) revealing rough macroscopic growth bands. Dotted line delineates contour of band. Direction of crack growth indicated by arrow.

material was a frequency-induced change in the slope of the $da/dN - \Delta K$ plot. When Delrin was tested at 50 and 100 Hz, the crack growth behaviour was almost identical to that of Celcon. (Note the data band for Celcon.) It is interesting to note that the fracture surfaces of these Delrin samples also possessed markings oriented parallel to the advancing crack front. For the sample tested at 10 Hz, the slope of the $da/dN - \Delta K$ was reduced. In contrast to the cases of 50 and 100 Hz, however, no parallel fracture markings were found.

4. Discussion

When the present test results for polyacetal are compared with previous results for nylon 6, 6 and PVDF, it is seen that for a given ΔK value PA is, indeed, the most fatigue resistant crystalline polymer examined thus far (Fig. 3a). In fact, if these test results are normalized with respect to the modulus of elasticity [8] and compared with metal alloys for engineering applications, it is seen that the crystalline polymers as a group are more fatigue resistant than the alloys (Fig. 3b). The concomitant high fatigue crack propagation resistance and low fracture toughness of PA is puzzling, however, in the light of previous findings by Manson and Hertzberg [1]. They reported that as fracture toughness increased in a wide range of both amorphous and crystalline polymers, a larger value of ΔK was necessary to propagate a fatigue crack at a given velocity. This empirical correlation has been confirmed for other polymers by the authors [4, 9], and by Martin and Gerberich [10]. The lack of confirmation of this relationship for PA is not understood at this time.

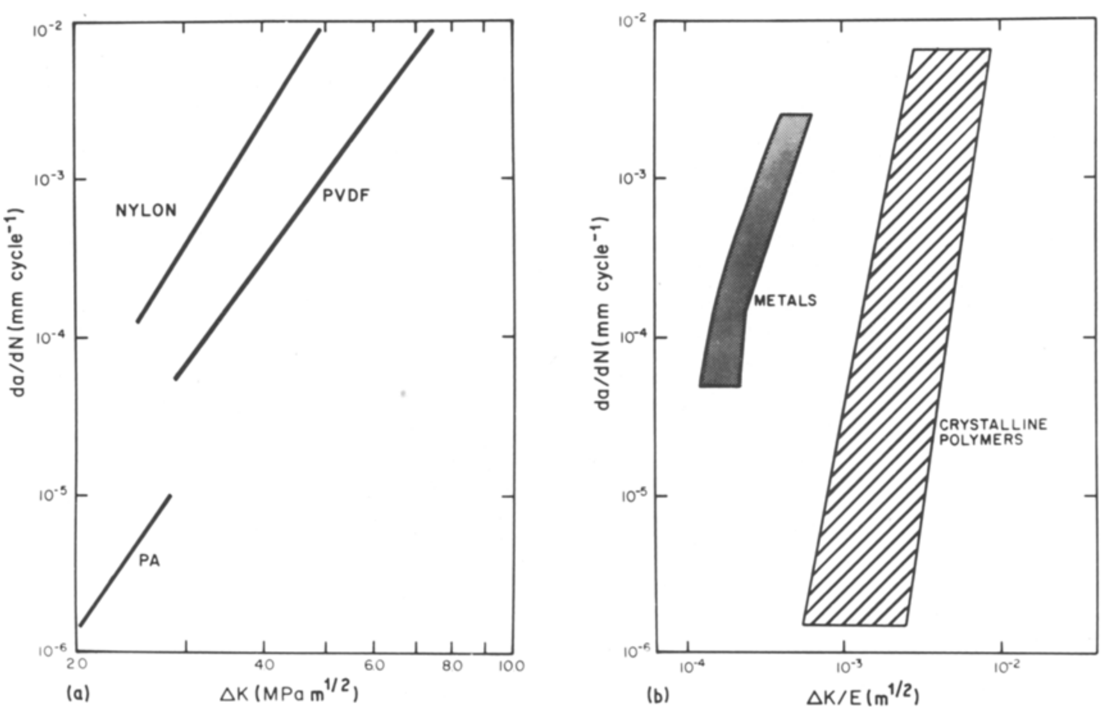

Figure 3 (a) Comparison of fatigue crack growth rates at 10 Hz in Nylon 6, 6, PVDF and Polyacetal (Delrin). (b) Comparison of crack growth rates in metals and crystalline polymers as a function of normalized stress intensity factor, $\Delta K/E$.

It would appear from the test results that the frequency-induced shift in the slope of the $da/dN-\Delta K$ plot in Delrin was related to a change in the mechanism of fatigue crack advance, as evidenced by the presence or absence of the parallel fracture markings. Phenomenologically, this is consistant with similar findings in polystyrene [11]. Here too, the slope of the crack growth rate plot was higher when a series of macroscopic bands (about 20 to 70 μm in width) was detected at high test frequencies; the slope was reduced at lower test frequencies where the parallel bands were absent.

These similar fracture markings and their presumable effect on the $da/dN-\Delta K$ relationship suggest that the crack growth kinetics for the two materials (PA and PS) exhibit some common features. In PS it was found that these macroscopic bands represented discontinuous crack growth increments [11]. That is, the crack was observed to remain dormant for several hundred loading cycles during which time a craze at the crack tip grew steadily, though at a decreasing rate. At some critical point, the crack was seen to advance abruptly to the craze tip and stop. The distance between the macroscopic bands (macrobands) was then taken to correspond to the increment of crack advance and to equal the length of the craze at the time of the transient instability. We suggest that the arrest markings found on some fracture surfaces of Delrin and Celcon also reflect the existence of discontinuous crack growth, though not necessarily involving the same micromechanism deduced for PS and other polymers [12, 13] (see below). Since the spacing between these bands was found to lie in a range of about 0.15 to 0.35 mm as compared with the typical increment over which crack extension data were collected (i.e., about 0.25 mm) one would expect to find more scatter in the crack velocity test results if the growth bands in PA corresponded to discontinuous crack extension. Indeed, it may be seen from Fig. 4 that considerable scatter was associated with the test results for the Delrin and Celcon samples that contained growth bands on the fracture surface while much less scatter was associated with the results from the Delrin sample tested at 10 Hz, where no bands were observed. Consequently, we conclude that the Celcon samples and the Delrin samples tested at high frequencies did experience discontinuous fatigue crack growth.

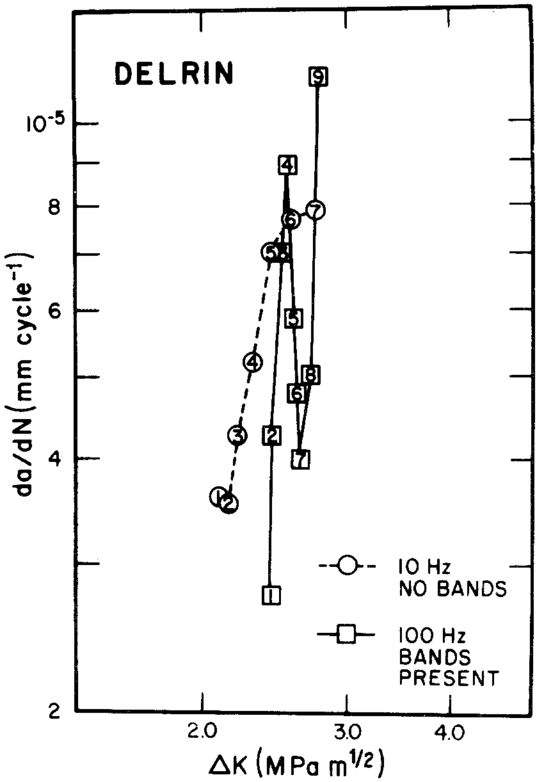

Figure 4 Evidence of discontinuous crack growth at high frequency in Delrin. Note large scatter at 100 Hz.

The macrobands were found to be very jagged in appearance and difficult to photograph. However, their sizes varied approximately with the second power of ΔK as has been the case for all other polymers that revealed these markings [11, 13, 14] (Fig. 5). Accordingly, if one assumes the size of the growth bands to be equal to the plastic zone size at the crack tip, it is possible to compute an inferred yield strength for the deformation process at the crack tip. From the Dugdale relationship [15], the inferred yield strengths of Delrin and Celcon are found to be about 100 and 90 MPa, respectively, in excellent agreement with reported values for these materials [16].

If the band size associated with discontinuous crack growth is divided by the average crack growth rate through this region, one may detemine the cyclic stability of the damage zone ahead of the crack. It was found for the case of PA that as many as 100 000 loading cycles were necessary to enable the crack to penetrate though the deformation zone. (Recall that this crack advance took place abruptly). Comparing this value with corresponding values for PMMA, PC,

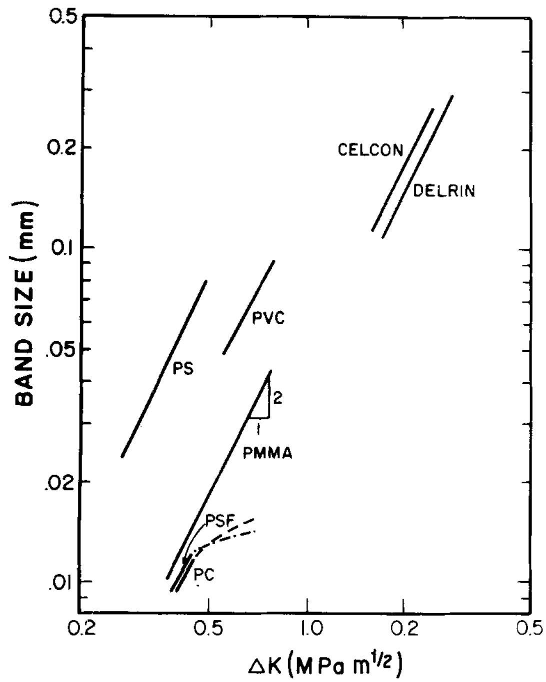

Figure 5 Dependence of fracture band size on ΔK for five glassy polymers and polyacetal.

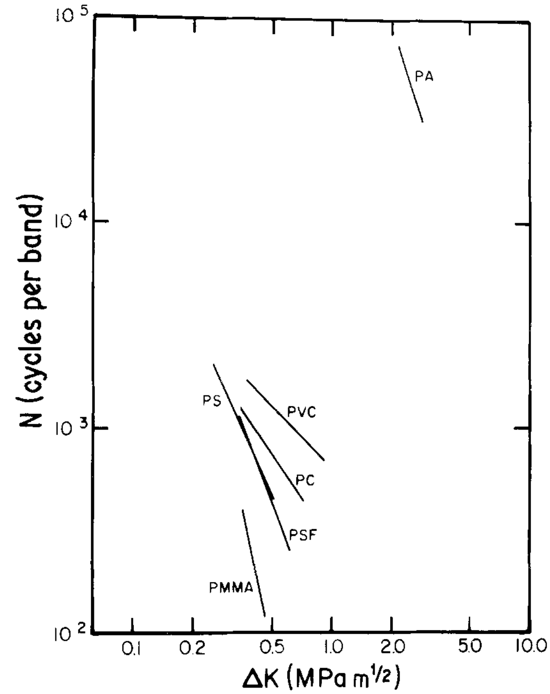

Figure 6 Effect of ΔK level on the number of cycles required for growth through a discontinuous growth band.

PVC, PS, and PSF [13], it is seen that the cyclic stability of the deformation zone in PA is several orders of magnitude greater than any of these materials (Fig. 6). This is consistent with the much greater fatigue crack propagation resistance of PA as compared to these other materials.

At this time, some uncertainties persist regarding the nature of FCP kinetics in PA. It is not clear why DG bands are found in Celcon when tested at 10 Hz while such bands do not appear in Delrin under the same test conditions. The micromechanism for the macroband formation in PA also remains unclear. Initial attempts to examine the details of the fracture surfaces of these materials in the SEM at 20 kV were of limited success since the sample surfaces became disfigured as a result of irradiation damage. The damage probably corresponds to transformation in the spherulite morphology under the electron beam. Greater success with fractographic viewing was achieved at an accelerating potential of 5 kV, though associated with some loss in resolution. The discontinuous growth bands appeared jagged with occasional 50 to 100 μm nodules and cavities dispersed randomly on the fracture surface. These nodules corresponded in size to the diameter of individual spherulites. While some secondary cracking was identified along spherulite boundaries, the advancing fatigue crack generally preferred a transpherulitic path similar to that found in polyethylene at low growth rates [17]. Both types of failure have been reported for brittle failure in Delrin (static loading) [18]. For example, the fractograph shown in Fig. 7a reflects the passage of the crack through the middle of the spherulite, thereby revealing its radial symmetry. This is compared with a companion SEM micrograph (Fig. 7b) which shows the same radial symmetry of a spherulite cut by the polishing plane.

Finally, occasional inclusions of unknown origin (Fig. 8) were noted in Delrin; these could possibly correspond to domains of a polymeric stabilizer such as a polyamide [5].

On the basis of these observations, we conclude that the discontinuous growth bands in PA differ significantly from those found in amorphous polymers [11–13]; no pattern of decreasing microvoid sizes across each band was observed. Consequently, while the phenomenology of the discontinuous crack growth process (i.e., the band size second power dependence on ΔK) appears to be the same for both amorphous and crystalline polymers, the micromechanisms are different.

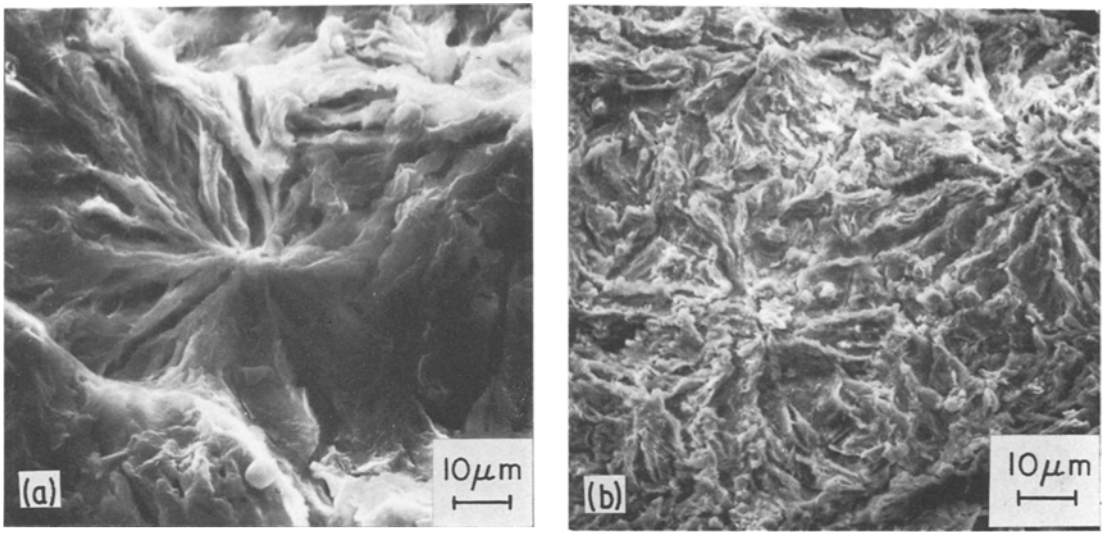

Figure 7 SEM micrographs of (a) fracture surface of Delrin showing internal structure of a spherulite and (b) polished and etched specimen showing the same radial symmetry.

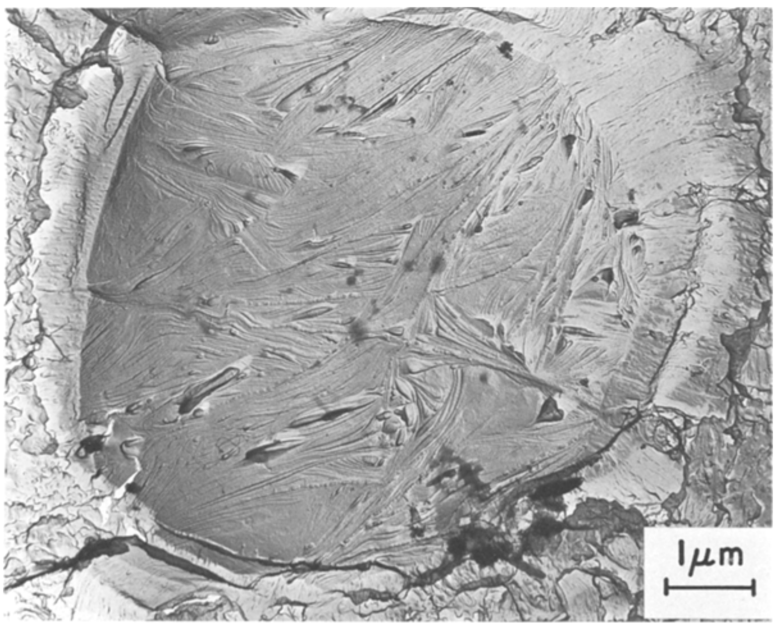

Figure 8 TEM micrograph showing nodular inclusion in Delrin.

5. Conclusions

It is possible to generate stable sub-critical fatigue-crack propagation in polyacetal. At any given value of ΔK, this material is found to be the most fatigue-resistant engineering plastic examined to date. A discontinuous crack growth process is found for all test conditions in Celcon but restricted to test frequencies of 50 and 100 Hz in Delrin. The increment of crack advance associated with this cracking mode is equal to the crack tip plastic zone dimensions. On a local scale, the crack is believed to propagate across spherulites but with some indication given for secondary cracking along spherulite boundaries.

Acknowledgements

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