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The influence of loading history on fatigue in engineering plastics

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Smooth bar rotating beam fatigue tests were conducted on acetal homopolymer (Delrin) and poly(methyl methacrylate) (PMMA) resins. Thermal failures due to hysteretic heating were encountered at high bending stress levels. Stable fatigue crack propagation (FCP) occurred in both materials at low stress levels. Interrupted fatigue tests of Delrin were performed by first load cycling until the surface temperature reached 65° C, then cooling to room temperature, and immediately recycling to 65° C. As expected, this intermittent testing procedure resulted in much longer fatigue lifetimes when compared to uninterrupted cyclic loading. Furthermore, each successive loading sequence required a shorter time to reach 65° C. It is suspected that this effect is related to cyclic-induced permanent changes in the material's viscoelastic damping response. Fractographic analysis of the Delrin samples revealed a duplex structure consisting of an annular surface region of microvoids combined with a flat, featureless central zone. The fracture surfaces of the PMMA samples were flat and generally nondescript, except at low bending stresses where some evidence of stable crack advance was seen.

1. Introduction

Fatigue failure in polymeric materials can occur either by thermal softening and melting due to massive hysteretic heating, or by fatigue crack initiation and stable crack propagation to fracture [1, 2]. Regarding the former, polymers typically exhibit high-energy damping characteristics and generally low thermal conductivity [3, 4]. As a result, hysteretic energy is generated within the sample during cyclic loading. This hysteretic energy is dissipated mainly in the form of heat, and can cause the temperature of the specimen to increase during cyclic loading, with associated loss of stiffness. The magnitude of this temperature increase is a function of the applied stress (σ), the test frequency (f), the material's inherent loss compliance (J''), temperature (T) and heat transfer characteristics of the material. For example, Ferry [5, 6] noted that the rate of energy dissipation, $\dot{E}$, could be given by

$$\dot{E} = \pi \cdot f \cdot J''(f, T)\sigma^2 \quad (1)$$

The Ferry equation can also be rewritten and expressed in terms of the temperature rise per unit time

$$\Delta\dot{T} = \pi \cdot f \cdot J''(f, T) \cdot \sigma^2/\varrho \cdot c_p \quad (2)$$

where c_p is the heat capacity and ϱ the density.

During fatigue testing at low applied stresses and/or low frequencies, the temperature rise is small, with sample temperature tending to stabilize at some intermediate level. In this situation, the generation of hysteretic heat is essentially balanced by heat dissipation to the surrounding environment. Under these isothermal conditions, it is common to see specimen failure occur as a result of the initiation and growth of a fatigue crack [2]. In sharp contrast, when tests are conducted under load control at high stress and frequency levels, the temperature of the sample does not stabilize but, rather, continues to rise with associated loss in section modulus as J'' increases. As the modulus falls, larger specimen deflections are generated for the stresses applied. It follows from Equation 2 that the rate of temperature rise increases with J'' and that an auto-accelerating temperature rise occurs in the sample when the specimen temperature approaches an internal damping peak [5, 7, 8].

This unique influence of J'' on thermal heating of polymers during cyclic loading was demonstrated by O'Toole *et al.* [8] with their work on PMMA, polytetrafluoroethylene (teflon) and Nylon-6. While these three materials are structurally dissimilar and possess different mechanical properties, thermal failure occurred in each material at temperature levels corresponding to their respective glass transition temperatures (T_g), in association with a rapid increase in J'' [5].

The extent of hysteretic heating during fatigue cycling of polymers is also dependent on the test method itself, which determines the stress distribution in the specimen. For example, a sample tested in rotating bending experiences the maximum bending stress at its outer surface, with the stress decaying to zero as the neutral axis is approached [1, 9]. As a result, the material at the sample interior heats more slowly than does the surface zone. As a result, specimen life is extended beyond that associated with uniform loading of the cross-section as in an axial "push–pull" test, where greater amounts of heating are expected [10]. Furthermore, the lower-stressed

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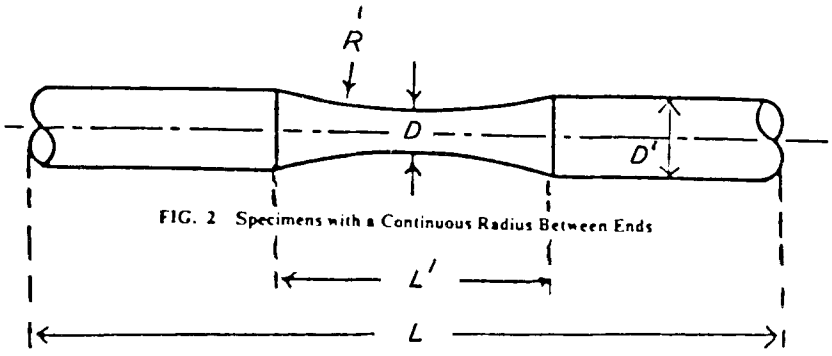


FIG. 2 Specimens with a Continuous Radius Between Ends

Figure 1 Rotating beam fatigue test specimen. Specimens machined according to ASTM E466-82. D = reduced section diameter = 0.25 in. (6.35 mm), D' = grip section diameter = 0.50 in. (12.70 mm), L = overall specimen length = 4.0 in. (101.60 mm), L' = reduced section length = 1.50 in. (38.10 mm), R = reduced section radius = 2.5 in. (63.50 mm).

material at the interior of the bend sample will, at least initially, act as a heat sink and speed heat dissipation from the hotter surface regions. This influence of test procedure on cyclic life was reported by Crawford and Benham [9], for the case of polyacetal; samples cycled under uniaxial loading conditions underwent thermal melting at lower applied stresses and at lower cyclic frequencies than those encountered in rotating bend samples.

It should be noted that thermal failure can be avoided if cyclic loading is periodically interrupted to allow the specimen to cool. In this manner, cyclic lifetimes may be greatly enhanced relative to those associated with uninterrupted tests at the same applied stress range and frequency [8, 10]. The ability to extend fatigue life through the introduction of rest periods contradicts traditional cumulative damage theories, such as that proposed by Miner [11]. It should be noted, however, that such theories are based on the linear accumulation of mechanical damage and do not recognize the duality of failure processes in viscoelastic polymers that exhibit either thermally or mechanically induced structural breakdown.

The objective of this investigation was to study the influence of intermittent loading on fatigue failure in amorphous PMMA and semi-crystalline polyoxymethylene polymer (Delrin). Particular emphasis was given to an assessment of changes in specimen temperature during cycling after periodic cooling periods.

2. Experimental procedures

Smooth bar rotating beam fatigue tests were conducted on an acetal homopolymer (Delrin "500") and PMMA to assess the susceptibility of these two materials to hysteretic heating and thermal melting during fatigue cycling. Delrin and PMMA rods (1.25 cm diameter) were machined according to ASTM E466-82 [12] into an "hourglass" configuration with a final reduced section diameter of 0.63 cm (Fig. 1). During fabrication, care was taken to ensure that the reduced area was free of machining defects.

A modified R.R. Moore rotating beam test machine (model RBF-200), capable of applying bending moments up to 35 N m (200 in. lb) was utilized to apply the desired bending stresses. A static, dead-load bending moment was applied to one end of the specimen, with the other end of the sample being gripped and rotated at a frequency of 50 Hz. As the sample was rotated, the reduced section experienced alternating tensile and compressive stresses with the sample's outer surface being subjected to the maximum stress level.

The R.R. Moore machine was designed to test metallic materials which experience much lower deflections than the plastic samples under the action of the bending moment. To use this machine to test these more compliant polymeric materials, test machine modifications were necessary to allow for greater static deflections and to maintain a constant stress condition in the specimen's reduced section. The surface temperature of the test specimens was monitored as a function of time (and total cycle count) with the use of a Barnes Radiometric Infrared microscope (Model RM-2B) [13].

A Delrin sample was also cycled intermittently at a bending stress of 63 MPa until a surface temperature of 65° C was obtained. The specimen was then allowed to cool until the surface temperature reached ambient, and then immediately recycled to 65° C. This iterative process of cyclic-induced heating and surface cooling during "rest" periods was continued until an asymptote in the heating time was reached. After reaching this asymptotic value of heat-up time, the sample was allowed to cool overnight to ensure equilibration of the entire cross-section at ambient temperature. The iterative heating and surface cooling procedure described above was then resumed the next day.

The fracture surfaces of selected PMMA and Delrin samples were examined in an ETEC Autoscan SEM in order to correlate the macroscopic failure events to specific microscopic fracture mechanisms.

3. Results and discussion

3.1. Fatigue testing

The stress–cyclic lifetime data (Table I) for both PMMA and Delrin are presented in Fig. 2. It is evident that Delrin shows superior fatigue life to that of PMMA at all applied stress levels. Figs 3 and 4 reveal the surface temperature against time response of Delrin and PMMA to cyclic loading (50 Hz) at various applied bending stresses. For both Delrin and PMMA, thermal failures occurred at high stress levels, with stable crack propagation occurring at lower stress levels.

The effect of hysteretic heating was very pronounced in the Delrin specimens that were tested at bending stresses above 54 MPa; these specimens underwent rapid heating, especially at higher stress levels. For Delrin, the "changeover" stress level separating thermal from mechanical failure appears to be in the range of 54 MPa. It is also interesting to note

TABLE I Cycles to break at 50 Hz

Material	Specimen I.D.	Applied bending moment (J) (in. lb)	Maximum bending stress (MPa) (10^3 p.s.i.)	Cycles at failure, N_f (10^3)	Maximum temperature (°C)
PMMA (Polymethyl-methacrylate)	P-10*	1.13 (10)	45.0 (6.53)	2.25	36.0
	P-8*	0.91 (8)	36.0 (5.22)	5.00	43.5
	P-7*	0.79 (7)	31.5 (4.57)	15.00	42.0
	P-6†	0.68 (6)	27.0 (3.92)	114.00	35.0
	P-5†	0.57 (5)	22.5 (3.26)	480.00	28.0
	P-4‡	0.46 (4)	18.0 (2.61)	>3150.00	22.0
Delrin (Polyacetal)	D-16*	1.81 (16.0)	72.0 (10.44)	3.70	125.0
	D-14*	1.58 (14)	63.0 (9.14)	8.75	108.0
	D-13*	1.47 (13)	58.5 (8.48)	19.00	130.0
	D-12c*	1.36 (12)	54.0 (7.83)	96.50	125.0
	D-12a‡	1.36 (12)	54.0 (7.83)	>4300.00	54.0
	D-10‡	1.13 (10)	45.0 (6.53)	>3300.00	29.0

*Sample showed temperature "runaway" with failure by thermal melting.
†Sample showed temperature stabilization prior to fracture.
‡Sample showed temperature stabilization and did not break.

that the Delrin samples that did undergo thermal melting reached a maximum temperature in the range 100 to 130° C, corresponding to the T_g peak (glass transition temperature) for this material [14]. This correspondence of thermal melting to major damping peaks has been reported elsewhere for PTFE and PVC [8, 10], with thermal failure occurring at or near the glass transition in temperature for these materials.

The fatigue response in PMMA was influenced by thermal heating at stress levels in excess of 31 MPa with temperature increases of 20° C seen prior to fracture. However, brittle fracture of the test specimens (as evinced by limited deformation) precluded the temperature from rising into the glass transition temperature region (105° C) [2] for this material. Low applied bending stresses resulted in only modest temperature increases, with specimen temperature stabilizing after only a 5 to 10° C increase. After achieving steady state temperatures, fracture occurred as a result of fatigue crack initiation and propagation (Fig. 4). It should also be noted that at sufficiently low stresses (18 MPa), no temperature increase was observed and the specimen did not fail, even after 5 million cycles.

3.2. Interrupted fatigue testing

The fatigue lifetime in Delrin was much improved when periodic rest periods were introduced during cyclic loading (Fig. 2). It is evident from Fig. 5 that each successive cycling increment during the same loading sequence required less time to reach the target temperature of 65° C. Furthermore, after each overnight cooling segment, the initial time to reach 65° C upon reloading decreased significantly, which indicates that permanent changes may well have occurred in the material's viscoelastic state and/or microstructure. (Note that all external test parameters were kept constant – recall Equation 2.) Studies are currently planned to illuminate this point further. Alternatively, the decreased time to reach 65° C may have been caused by the accumulation of mechanical damage within the test sample as a result of repeated cycling (see below); in this context, the effective stress within the sample would have been greater

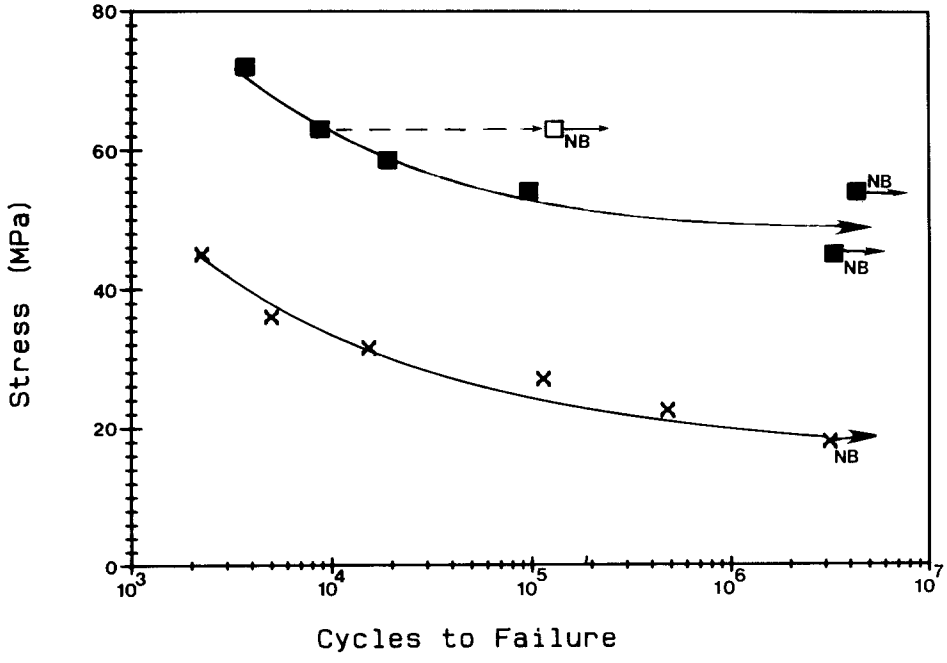


Figure 2 S–N curves for (□, ■) Delrin 500 and (×) PMMA (50 Hz). (□) Interrupted cycling of Delrin, NB, no break.

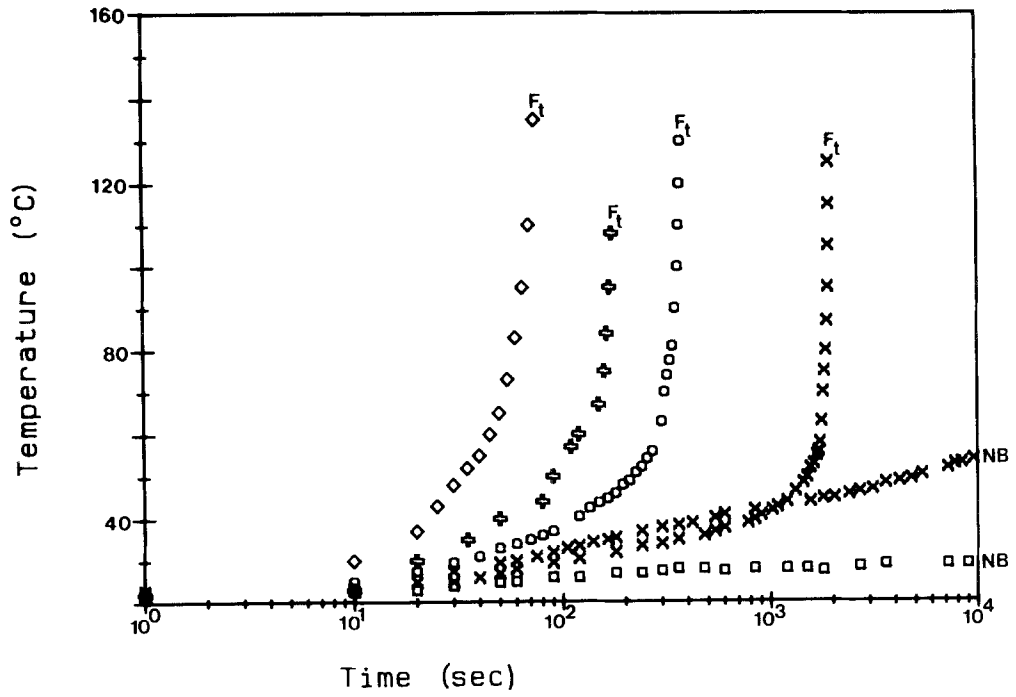


Figure 3 Specimen temperature plotted against cycling time for Delrin, at (◇) 72.0, (+) 63.0, (○) 58.5, (×) 54.0, (□) 45.0 MPa. NB, no break; F_t, failure by melting; F_m, failure by FCP.

than that computed, based on nominal specimen dimensions.

3.3. Fractography

At all bending stress levels, the Delrin samples displayed a fracture surface consisting of an annular ring of microvoids near the outer surface of the samples, and a flat, featureless central plateau region (Figs 6a and b). These two regions reflect the temperature distribution within the sample. At the outer surface, intense heating developed and resulted in the formation of many microvoids. These microvoids coalesced in conjunction with rapid heating of the unbroken ligaments in this surface zone. In effect, the specimen then took on the configuration of a circumferentially "notched" round bar which contributed to rapid (and brittle) fracture of the core region in this notch-sensitive resin. Furthermore, when the magnitude of the applied stress decreased from 58.5 to 54.0 MPa, the depth of the microvoid zone increased from 900 to 1200 μm (Figs 6c and d). This is to be expected because the material should be capable of supporting a larger "crack" at lower stress levels.

The PMMA samples cycled at high bending stresses (45 to 31.5 MPa) showed no indications of stable crack growth with fast fracture presumably being dominated by crack initiation processes. These

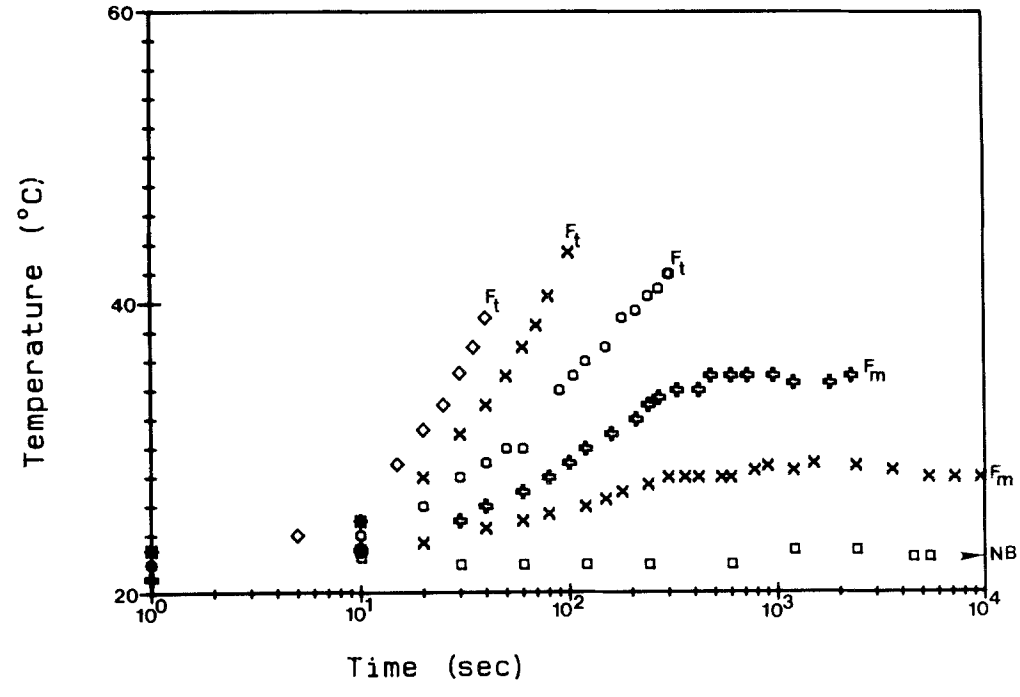


Figure 4 Specimen temperature plotted against cycling time for PMMA at (◇) 45.0, (×) F_t 36.0, (○) 31.5, (+) 27.0, (×) F_m 22.5, (□) 18.0 MPa. NB, no break; F_t, failure by melting; F_m, failure by FCP.

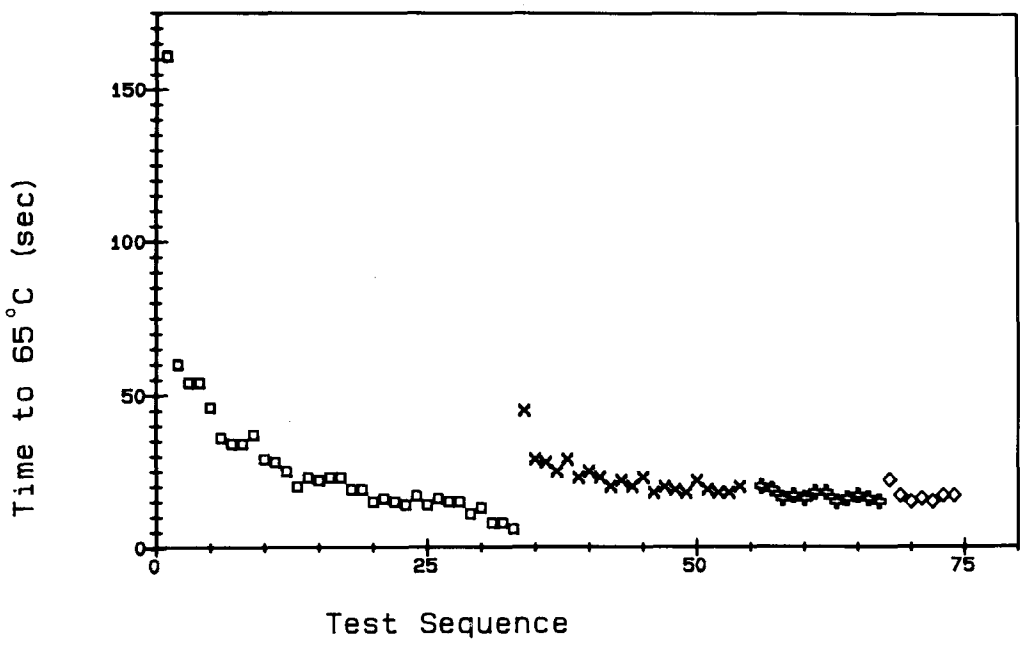


Figure 5 Time to reach 65° C plotted against number of loading sequences (Applied stress = 63.0 MPa) for Delrin at 50 Hz. Each symbol represent an individual loading sequence to 65° C. Similar symbols represent reheating after surface cooling to room temperatures. Different symbols represent reheating after overnight cooling of specimen.

samples have fracture surfaces that are, in general, quite flat and nondescript, except for the presence of cleavage-like markings indicative of brittle fast fracture (Figs 7a and b). At low stress levels (22.5 and 27.0 MPa), however, clear indications were found for stable, sub-critical crack growth prior to fast fracture. Fig. 8a shows the fracture surface of the PMMA specimen that was cycled at a bending stress of 22.5 MPa. It is evident that the fracture process began at a surface discontinuity, and that crack growth occurred by stable fatigue crack propagation in association with the presence of fatigue striations. Fig. 8b shows a high magnification view of these markings and the measured spacing (5 to 7 μm) was in the range of other reported striation spacing measurements at similar K levels for PMMA [5]. These striations (S), and the associated ratchet lines (R) were observed to a depth of about 500 μm from the sample's surface, at which point the fracture surface assumed the featureless morphology representative of fast fracture.

Similar features were seen in the PMMA specimen that was cycled at a stress of 27 MPa (Fig. 8c). Again, there is evidence for the existence of stable, sub-critical crack growth prior to fast fracture (Fig. 8c). The morphology of these quasi-parallel fracture lines, however, is not consistent with that of the fatigue striations.

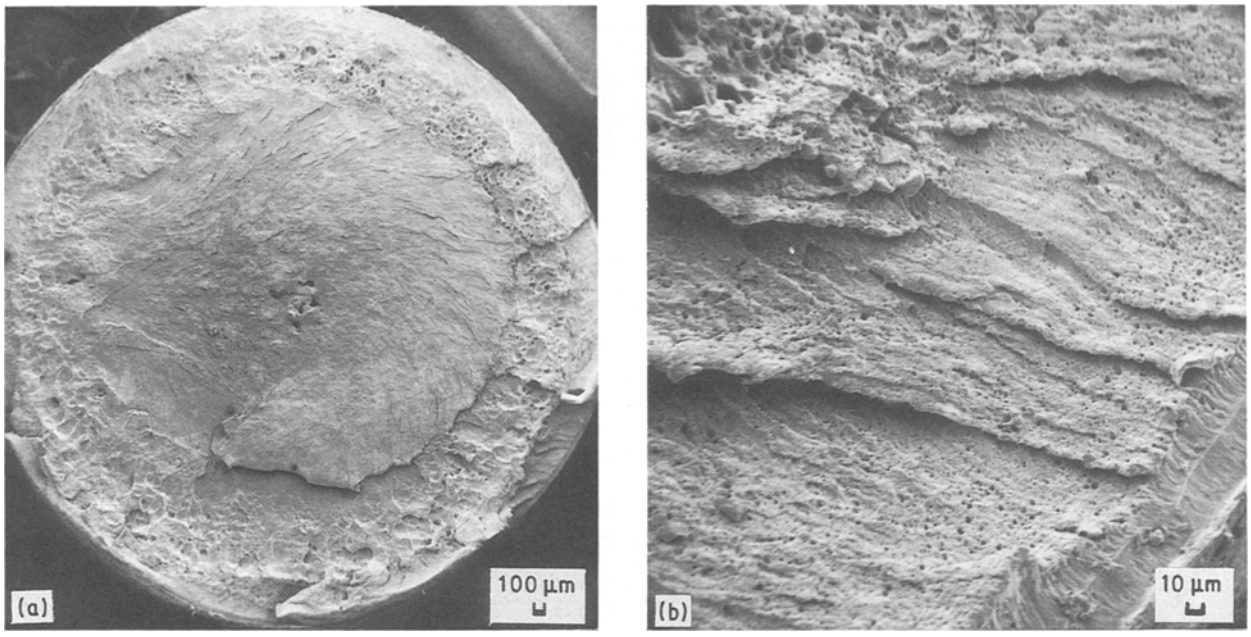
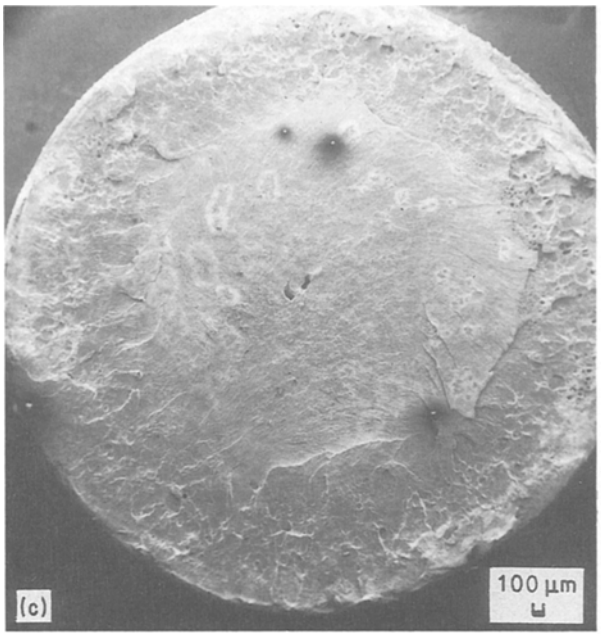


Figure 6 Microfractographic appearance of polyacetal (Delrin). (a, b) Bending stress = 58.5 MPa. (a) Overview of fracture surface. (b) Fine microvoids near outer edge of specimen. Note the coarsening of the voids from the bottom right to upper left as depth into the sample increases. (c, d) Bending stress = 54.0 MPa. (c) Overview of fracture surface. Notice the similarity to (a). (d) Transition from fine to coarse microvoids. Arrow indicates crack growth direction.



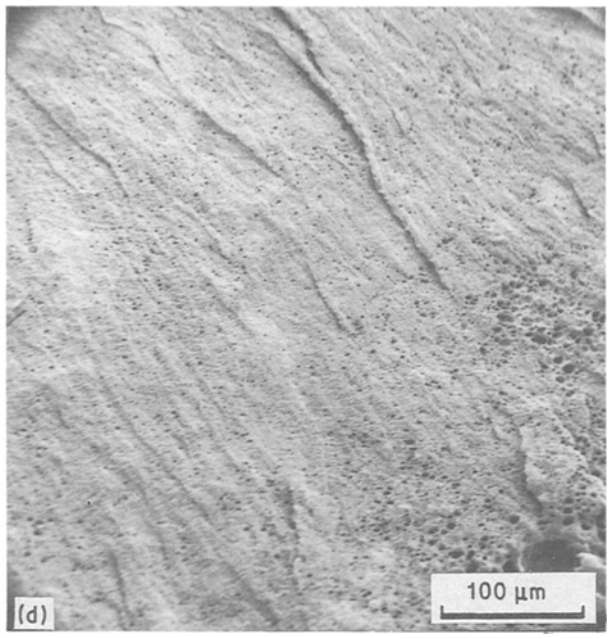


Figure 6 Continued

4. Conclusions

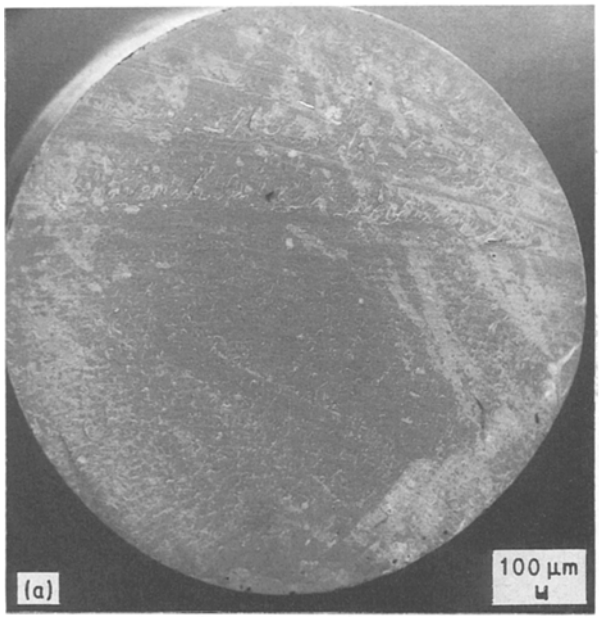
1. Delrin and PMMA experience fatigue failure by both thermal melting and stable crack initiation/propagation. The exact failure mechanism depends on the applied bending stress, with higher stresses favouring thermal failure. For a cyclic frequency of 50 Hz, the values of the "changeover" stress from thermal to mechanical failure are 27.0 MPa for PMMA and 54 MPa for Delrin.

2. As the applied bending stress increases, the total number of cycles to failure during uninterrupted fatigue testing decreases as more hysteretic heating is observed. From the relative locations of the SN curves (Fig. 2), it is evident that Delrin is significantly more fatigue resistant than PMMA.

3. All Delrin samples experienced thermal melting in the temperature range 100 to 130° C, corresponding to the T_g damping peak in polyacetal; this finding confirms the relation between high values of J'' and the onset of thermal melting.

4. Interrupted fatigue tests conducted on Delrin at a bending stress of 63 MPa led to a significant improvement in fatigue life. No fracture was seen after 1.3 million cycles, which compares with thermal melting after 8700 cycles during uninterrupted testing at the same bending stress. However, the time needed to elevate the surface temperature of the sample to 65° C decreased with each successive recycling event. Such behaviour may be attributed to a cyclic-induced change in the material's viscoelastic state and/or microstructure.

5. Fractographic analysis of both PMMA and



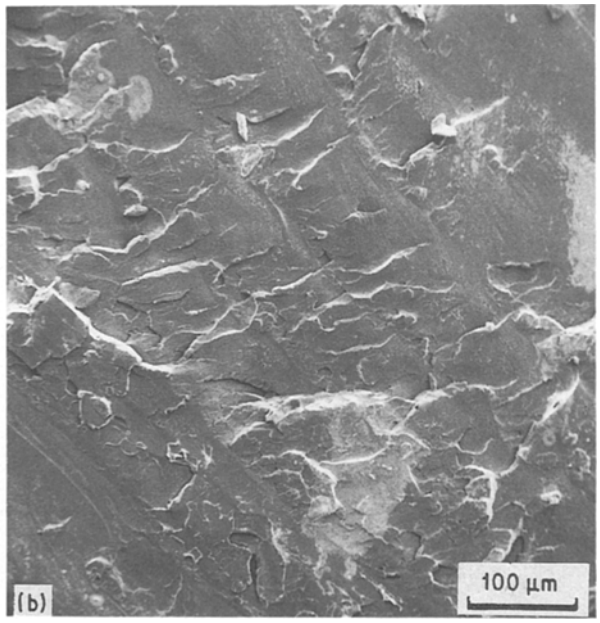
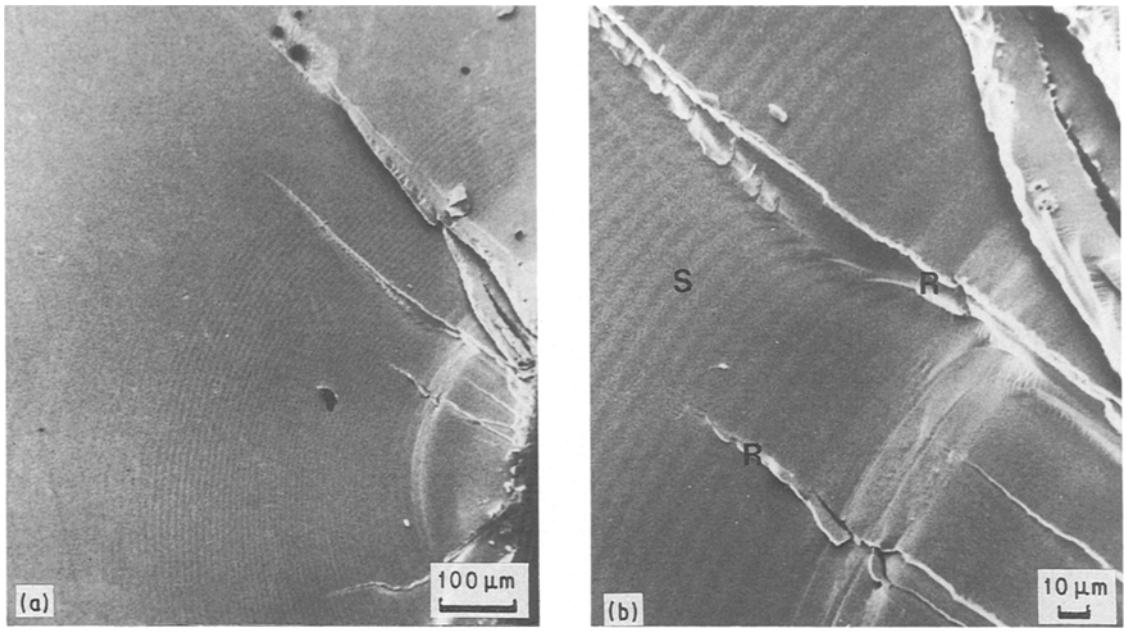


Figure 7 Microfractographic appearance of PMMA associated with unstable crack growth at high bending stress (45.0 MPa). (a) Overview of fracture surface. Notice the featureless appearance. (b) Cleavage markings on the same sample as (a), indicative of unstable crack propagation to fracture.



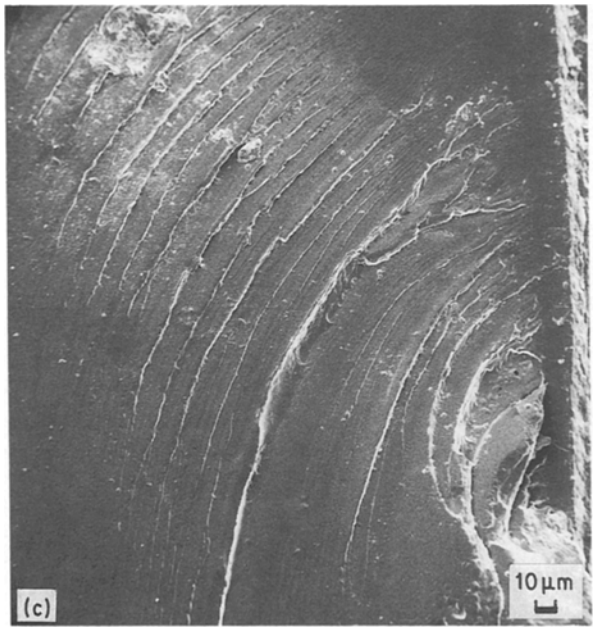


Figure 8 Microfractographic appearance associated with subcritical crack advance in PMMA. (a, b) Bending stress = 22.5 MPa (a) Crack origin. Notice the surface flaw, ratchet lines, and striations. (b) Same area as (a). (c) Bending stress = 27.0 MPa. View of cyclic markings near fracture origin. Morphology of the markings indicates that they are not striations.

Delrin indicates that both materials fail by a combination of stable crack growth and fast fracture, with the relative amount of each process depending on the prevailing stress level.

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References

1. R. J. CRAWFORD and P. P. BENHAM, *Polymer* **16** (1975) 908.
2. R. W. HERTZBERG, "Deformation and Fracture Mechanics of Engineering Materials" (Wiley, New York, 1978) p. 467.
3. I. CONSTABLE, J. G. WILLIAMS and D. J. BURNS, *J. Mech. Engng Sci.* **12** (1970) 20.
4. R. J. CRAWFORD and P. P. BENHAM, *J. Mater. Sci.* **9** (1974) 18.
5. R. W. HERTZBERG and J. A. MANSON, "Fatigue of Engineering Plastics" (Academic, New York, 1980) p. 36.
6. J. D. FERRY, "Viscoelastic Properties of Polymers" (Wiley, New York, 1961).
7. S. B. RATNER and S. I. KOROBOV, *Mekhanika Polimerov* **1** (1965) 63.
8. J. L. O'TOOLE, G. P. KOO and M. N. RIDELL, *Polym. Engng Sci.* **10** (1966) 363.
9. R. J. CRAWFORD and P. P. BENHAM, *J. Mater. Sci.* **9** (1974) 1297.
10. G. P. KOO, M. N. RIDELL and J. L. O'TOOLE, *Polym. Engng Sci.* **7** (1967) 182.
11. M. A. MINER, *J. Appl. Mech.* **11** (1954) A-159.
12. ASTM E466-82, "Conducting Constant Amplitude Axial Fatigue Tests of Metallic Materials", Vol. 3 (American Society for Testing and Materials, Philadelphia, Pennsylvania, 1983) p. 596.
13. M. T. HAHN, R. W. HERTZBERG and J. A. MANSON, *Rev. Sci. Instrum.* **54** (1983) 604.
14. N. G. McCRUM, "Anelastic and Dielectric Effects in Polymeric Solids" (Wiley, London, 1967) p. 540.

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