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Mechanical behaviour of poly (methyl methacrylate)

Part 2 *The temperature and frequency effects on the fatigue crack propagation behaviour*

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The effect of temperature and cyclic frequency on fatigue crack propagation (FCP) rates were investigated for poly methylmethacrylate (PMMA). Test temperature ranged from −30 to 100° C which includes the β transition and approaches T_g. FCP experiments were conducted at frequencies of 1, 10, 50 and 100 Hz. In general, the FCP rates increased with increasing temperature and decreasing cyclic frequency. The crack growth rate is near a maximum at 80° C and 10 Hz. When the experimental conditions approach the glass transition region, e.g. studies at 100° C, the failure mechanism changes and the material becomes more fatigue resistant while simultaneously softening. The frequency dependence of da/dN provided by the Michel–Manson (M–M) model is shown to be valid up to about 80° C, in absence of extensive plastic deformation. FCP results exhibit an interaction between the thermal and mechanical driving forces which is not consistent with the M–M model.

Plots of FCP rate were made against the reciprocal of temperature. The apparent activation energies calculated from these plots varied from 13 to 44 kJ mol^{-1}, and show that the activation energy increases as ΔK increases and as cyclic frequency decreases.

1. Introduction

Since polymers are being used increasingly for loading-bearing applications, understanding the fatigue process in these materials is essential if they are to be used in a safe and reliable fashion. Linear elastic fracture mechanics (LEFM) has been used to describe fatigue crack propagation (FCP) behaviour in polymers. Typically, FCP in these viscoelastic materials is very sensitive to such variables as cyclic frequency and temperature. In general, the FCP rate for polymers decreased with increasing cyclic frequency, although exceptions have been reported [1–4]. This phenomenon has been attributed to crack tip blunting, resulting from local hysteretic heating, and to the inhibition of molecular motion due to a shortened deformation response time [5–7].

By contrast, the effect of temperature on FCP rate of polymers has been studied less extensively than cyclic frequency. Recently, Michel and Manson proposed a model relating FCP rate to both mechanical and thermal driving forces as well as to cyclic frequency [8, 9]. It was shown that this model can describe the FCP behaviour of polystyrene (PS) and poly(vinyl chloride) (PVC) reasonably well at room temperature [9]. An objective evaluation of the Michel–Manson model requires an independent determination of the material properties used in this model, i.e. ΔK_{th}, K_C, and σ, and especially, consideration for how these properties depend upon temperature and loading rate. Therefore, in the present work, PMMA was selected as a model material to examine the Michel–Manson (M–M) equation across the transition temperatures, T_β and T_g, and through a wide range of cyclic frequencies.

The various processes involved in the fracture of solids usually impart special characteristics to the fracture surface. By studying such surfaces, one can gain valuable information, complementing FCP kinetics and providing additional insight into the failure mechanism. The fractography of poly(methyl methacrylate) (PMMA) in tensile and fracture toughness tests, i.e. monotonic loading conditions, has been examined in detail [10–13] including the influence of different loading rates [14] and temperature [15], and different molecular weight [16]. By contrast, the PMMA fracture surfaces of fatigued samples have received less attention and will be characterized as part of this study.

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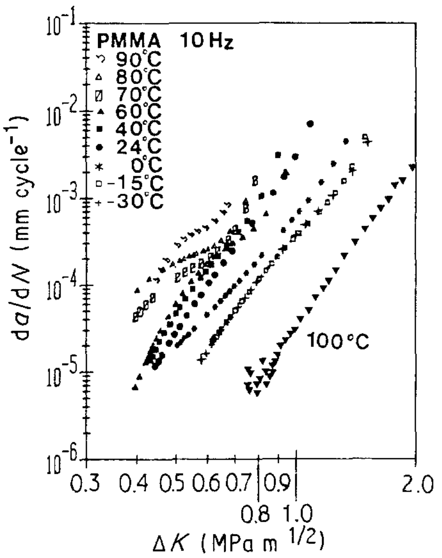

Figure 1 The effect of temperature on the fatigue crack propagation behaviour of PMMA at a cyclic frequency of 10 Hz.

Based on this work, three manuscripts have been prepared. In the first paper of this series [17] several material properties needed as inputs to the M–M model as a function of temperature and loading rate were determined. In this paper, the FCP behaviour and associated fractography are discussed. In the final paper [18], the theoretical aspects of the governing fatigue fracture process will be discussed in terms of the activation energy and activation volume for the fracture.

2. Experimental details

Single-edge-notch (SEN) specimens were prepared from a commercial grade PMMA (Rohm and Haas Plexiglas) with a number average molecular weight of 478 000 g mol^{-1}. These specimens were studied using a servohydraulic closed-loop testing system in manual control. A split oven was used for temperatures above ambient. Experiments at subambient temperature involved passing N_2 gas into a 30 l Dewar flask filled with liquid nitrogen and then passing the chilled gas into the oven through a solenoid valve.

Fatigue experiments were performed under constant load range with minimum to maximum load ratio of 0.1 using a sinusoidal wave form. Crack length was monitored with a travelling microscope with an accuracy of about 0.01 mm. Temperatures ranged from −30 to 100° C, and cyclic frequency was varied from one to 100 Hz (including 1, 10, 50 and 100 Hz).

Fracture surfaces were studied with optical microscopes and an ETEC scanning electron microscope (SEM) at an accelerating voltage of 10 kV. Specimens prepared for use in the SEM were gold-coated prior to viewing.

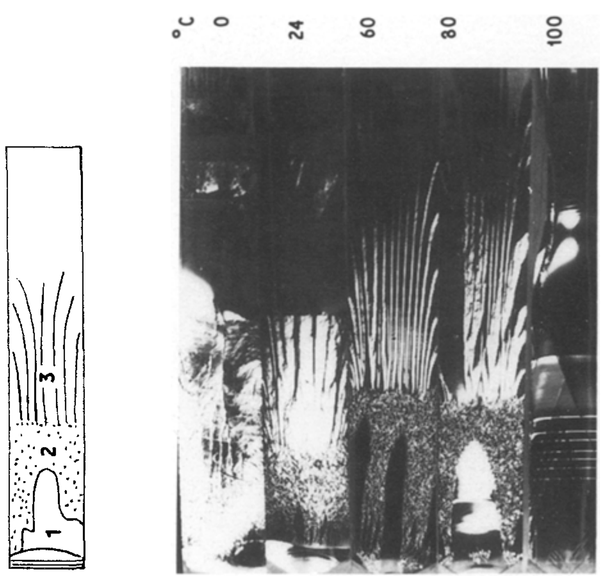

Figure 2 The effect of temperature on the fatigue fracture surfaces of PMMA at 10 Hz. The fracture surfaces are composed of (1) mirror region, (2) mist region, and (3) hackle region.

3. Results

Depending on the experimental conditions, the fatigue specimens failed either in a brittle or a ductile manner. The ductile mode of failure shows not only a high fatigue resistance but also an entirely different fracture surface, suggesting a deformation mechanism different than that for brittle failure.

3.1. Temperature effects

The data show that the FCP rate increases as the temperature increases from −30 to 80° C at all frequencies. However, the results for −30 and −15° C at 10 Hz are virtually the same, Fig. 1.

When the temperature is near the glass transition region, i.e. 100° C, the fatigue resistance suddenly increases (see Fig. 1). At temperature below 100° C and frequencies > 10 Hz, the fracture surfaces have a brittle appearance and are comprised of mirror, mist, and hackle regions. On the other hand, at 100° C there was evidence of ductile behaviour in the form of shear bands and striations at the end of the crack growth region, Fig. 2.

3.2. Frequency effects

Current results for temperatures < 80° C, typically represented by room temperature data, Fig. 3, support earlier work showing that PMMA FCP rates decrease as cyclic frequency increases [10]. However, when the temperature approaches the glass transition region, for example 100° C, crack growth rates increase with increasing frequency, Fig. 4.

3.3. Fractography

The fatigue fracture surfaces are affected by test temperature as shown by the size of the mirror region which increases as temperature increases, Fig. 2. The mirror region is the region nearest the chevron notch which either appears bright, as for the FCP fracture surface at 80° C or dark, as exemplified by fracture surfaces at 60 and 24° C. In the latter case, the mirror regions are elliptical or bullet-shaped dark areas which occur adjacent to areas of mist-type regions. At 0° C, there is almost no mirror region on the fracture surface. For the entire range of temperature and strain rate, there was no evidence of discontinuous growth bands (DGBs) on the fracture surfaces.

When the FCP measurements were conducted in the glass transition region (e.g. at 100° C), special

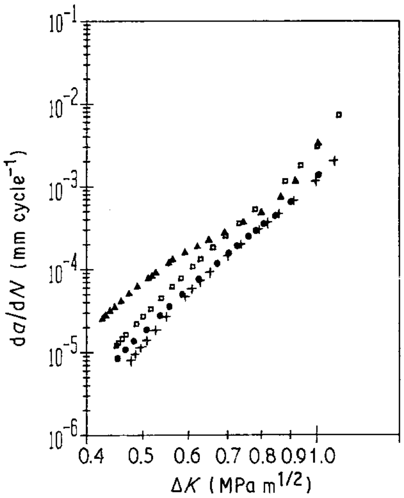

Figure 3 The effect of cyclic frequency on the FCP behaviour of PMMA at room temperature (24°), (+) 100 Hz; (●) 50 Hz; (□) 10 Hz; (▲) 1 Hz.

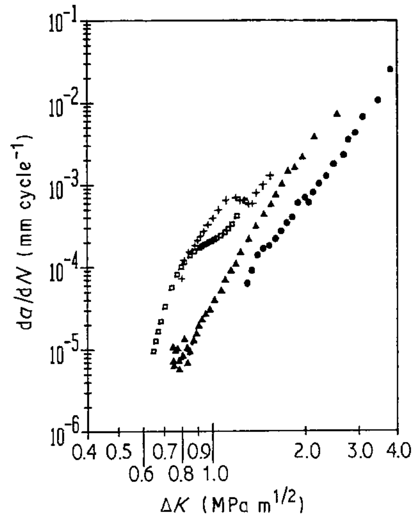

Figure 4 The effect of cyclic frequency on the FCP behaviour of PMMA at 100° C. (+) 100 Hz; (□) 50 Hz; (▲) 10 Hz; (●) 1 Hz.

fracture surface features appeared, including initially "diamond shapes", followed by "triangles", and finally shear lips and fatigue striations, Figs 5 and 6. A series of clear parallel lines were shown on the fracture surfaces of 100° C tests at ΔK greater than about 1.7 MPa m$^{1/2}$ (see Fig. 6). Since these lines are perpendicular to the direction of crack propagation, and their spacing corresponds to the macroscopic growth rate for one loading cycle (Table I), they correspond to fatigue striations.

4. Discussion

4.1 Temperature effects

The effects of temperature on fatigue behaviour have not been widely studied. For PMMA, studies by Radon *et al.* [19] were conducted at 5 Hz for fatigue crack propagation at four different temperatures ranging from −60 to 40° C. The FCP rate decreased with decreasing temperature, similar to the current results. Other studies on PMMA conducted at 1 Hz, show little effect of temperature on FCP rate between −120 and −70° C, but then a rapid rise of a decade or more in FCP rate occurs as the temperature increases from −70° C to room temperature [10]. However, the current results at 10 Hz shows that there is almost no temperature effect below −15° C. The possible factors causing such a difference between the literature and current results might be differences in molecular weight, cyclic frequency, and experimental control mode used by the various researchers.

An actual measurement of the temperature near the crack tip using an infrared microscope at room

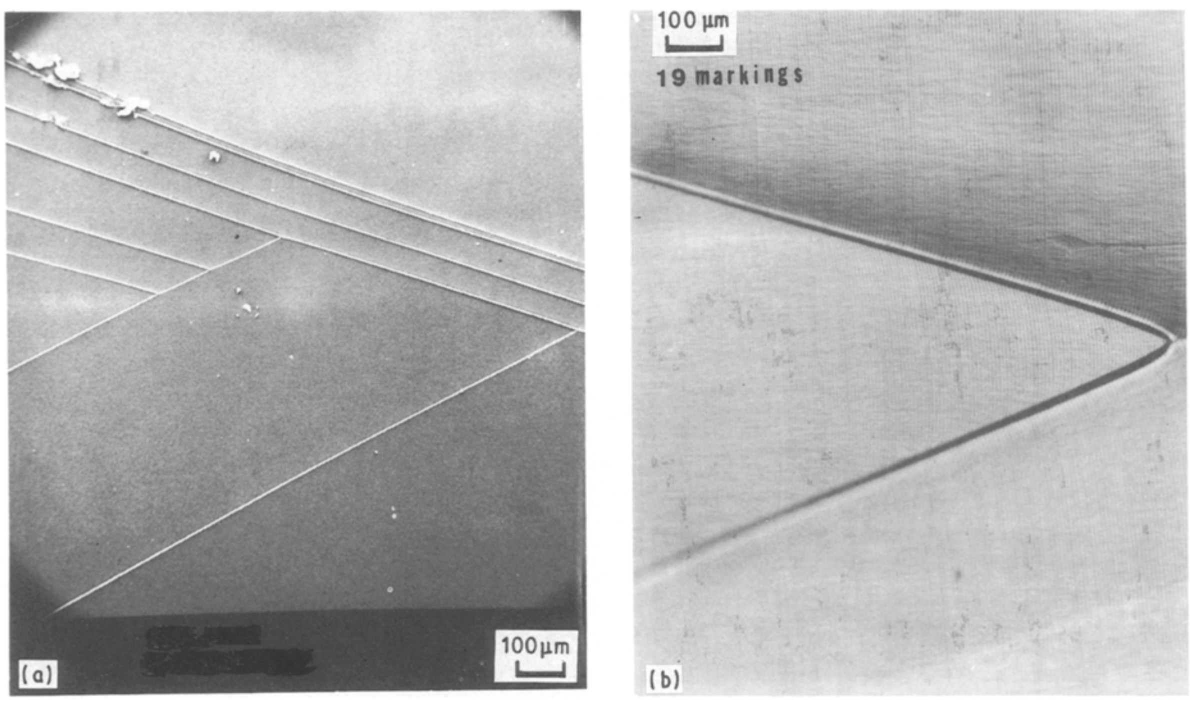

Figure 5 The fatigue fracture surface of PMMA at 10 Hz and 100° C. (a) The "triangle" feature at early part of the fracture surface. (b) Striation markings at later part of the fracture surface.

Figure 6 High magnification images (5000 ×) of a "triangle" on the fracture surface at $K = 2.4\,\mathrm{MPa\,m^{1/2}}$.

temperature showed a negligible change in temperature at the tip for a cyclic frequency of 10 Hz. No measurements of the crack tip temperature rise were made at other test temperatures. At higher temperatures the micromolecular activities of polymers such as chain reptation and disentanglement are increased. If chain slippage is considered to be the main molecular motion contributing to fatigue fracture of PMMA, the fatigue resistance should be low when the temperature is high because chain disentanglement and chain slippage are easier at high temperature. This is believed to be the reason why FCP rates increase as temperature increases.

When the temperature reaches the glass transition region, e.g. 100° C, the fatigue resistance suddenly increased. This result could stem from much more hysteretic heat being generated as compared with that generated at lower temperatures. The hysteretic heating would tend to soften the crack tip material and blunt the crack tip, thus decreasing the stress concentration factor of the crack tip and slowing down the crack growth rate. The hysteretic heating may also contribute to the change in fracture mechanism from brittle to ductile at 100° C.

4.2. Frequency effects

It is known that the hysteretic heat generated during the cyclic loading is proportional to the cyclic frequency,

$$\Delta\dot{T} = \frac{\pi f J''(f, T)\sigma^2}{\rho C_p} \qquad (1)$$

where $\Delta\dot{T}$ is the temperature change per unit time, f represents frequency, J'' represents the loss compliance, σ is the peak stress, ϱ is the density, and C_p is the specific heat [1, 2, 20]. If hysteretic heating occurs throughout the uncracked ligament, which is more likely in the glass transition region than for lower temperatures, then one might expect to see FCP rate increase as frequency increases since hysteretic heating softens the material and leads to an even higher degree of chain mobility. This condition is found at 100° C (see Fig. 4). For temperatures less than 100° C, J'' is smaller, thus, the crack tip heating is not significant. However, high cyclic frequency makes the physical state of the polymer stiffer, and reduces the chance of chain slippage. Therefore, FCP resistance increases as cyclic frequency increases.

In principle, da/dN approaches infinity as the maximum stress intensity factor, K_{max}, approaches the fracture toughness, K_c, of the material. Thus, K_c can be estimated from fatigue measurements by using K_{max} for the last point of the FCP curve. It is found that the estimated fracture toughness and the measured fracture toughness from three-point bending experiments [17] bear a linear relationship of

$$K_c\,(\text{measured}) = 0.5225 + 1.053K_c\,(\text{fatigue estimate}) \qquad (2)$$

where K_c is the fracture toughness in the unit of $\mathrm{MPa\,m^{1/2}}$, Fig. 7. However, it appears that the K_c value associated with three-point bending toughness tests is always greater than the fatigue value. Since it is very difficult to catch the very final point just before the onset of fast fracture in fatigue experiments, especially those conducted at high frequency, fatigue K_c values should be less accurate than the three-point bending K_c results and typically lower in value.

4.3. Fractography

As one might expect, changes in fatigue conditions will influence fracture surface morphology. Fig. 2 shows the influence of temperature on fatigue fracture surfaces of PMMA at a cyclic frequency of 10 Hz. The fracture surfaces at or above room temperature (except 100° C) show that the mirror region (either light or dark bullet shaped areas) is larger at higher temperature. The mirror region represents the

TABLE I Striation spacings for FCP specimen at 100° C and 10 Hz

Fatigue crack propagation rate, da/dN (mm/cycle)	Stress intensity factor range, ΔK ($\mathrm{MPa\,m^{1/2}}$)	Crack length (mm)	Striation spacing (mm)	Striation spacing da/dN
0.00108	1.74	20.4	0.00125	1.16
0.00237	1.99	22.8	0.00227	0.96
–	–	23.5	0.00250	–
–	–	26.2	0.00610	–
–	–	27.5	0.01470	–
–	–	29.1	0.01724	–

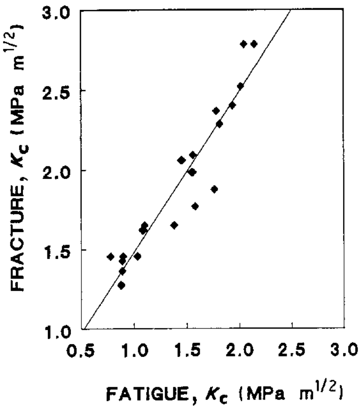

Figure 7 The relationship between the fracture toughness obtained from three-point bending experiments and estimates from the last point of FCP curves.

formation of a single craze wherein polymer chains are coordinated together into discrete fibrils and stretched in the same direction. By contrast, the mist region reflects craze bundling, i.e. multiple parallel crazes. At high temeprature, since fatigue resistance is low and the mirror region is large, it is suggested that single craze deformation dissipates less input energy than multiple craze deformation.

For 100° C, at four different cyclic frequencies (1, 10, 50 and 100 Hz), the fracture surface shows a dramatic change compared with those at lower temperatures, (see Figs 2 and 5). Now, a series of parallel triangles appear in the early portion of the fracture surface and very clear striation markings, as well as shear lips, in the central and final portions of the fracture surface. This fracture surface is clearly different from that for specimens tested at temperatures below 100° C.

Striations, corresponding to the successive positions of the advancing crack front as a result of individual load excursions, not only identify a cyclic loading condition, but also provide quantitative information regarding the kinetics of the fatigue cracking process [1, 21, 22]. A useful empirical relationship [23] between the striation spacing and ΔK for metals, is given by

$$\text{striation spacing} = 6\,(\Delta K/E)^2 \tag{3}$$

where E is the Young's modulus. For polymers, the dependence of striation spacing on ΔK varies from the third to twentieth power depending on the polymer [1]. The current PMMA results show a fifth power relationship between striation spacing and ΔK. Since the striation represents the position of the crack front after each loading cycle, the relationship between striation spacing and ΔK can be used to measure the FCP rate at any given location on the fracture surface.

The existence of discontinuous growth bands (generally, DGBs are limited to the mirror region) is molecular weight dependent. For PMMA, results from the published literature show that DGBs occur only for specimens of molecular weight less than $2 \times 10^5\,\mathrm{g\,mol^{-1}}$ [24]. Since the PMMA used in this study had $M_n = 4.8 \times 10^5$ ($M_w = 9.9 \times 10^5$), no DGBs were expected and none were found in the mirror region.

4.4. Michel–Manson model

A major objective of the present work is to use PMMA as a model material for evaluating the Michel-Manson (M–M) equation for fatigue crack propagation. This equation includes the effects of temperature, cyclic frequency, and the mechanical driving force [2, 8, 9].

$$\frac{\mathrm{d}a}{\mathrm{d}N} = \tau_0^{-1}\frac{c}{f}\exp\left(\frac{-E_a + \sigma_{ym}V^{\#}}{RT}\right) \times [(\Delta K - \Delta K_{th})^4/\sigma^2(K_c^2 - K_{max}^2)]^m \tag{4}$$

where τ_0, c, m are constants, f is cyclic frequency, E_a is the activation energy of the failure process. $V^{\#}$ is the activation volume, R is the gas constant, ΔK and ΔK_{th} are the applied stress intensity range and threshold range, respectively, σ is the yield strength, $\sigma_{ym} = \sigma(1 + R)/2$, R is the ratio of minimum to maximum cyclic load, K_c is the fracture toughness, and K_{max} is the stress intensity factor at maximum cyclic load.

According to the current results, the frequency dependence of $\mathrm{d}a/\mathrm{d}N$ provided by Equation 4 is shown to be valid up to about 80° C, as long as there is no marked inelastic behaviour, as shown in Fig. 3 At 100° C, FCP rates increase as frequency increases which is in opposition to the model prediction, Fig. 4.

Moreover, the M–M model represents the product of two driving forces, thermal and mechanical. All the parameters in the M–M equation can be independently measured except the constant c and the exponent m. As written, the M–M expression implies that the effects of temperature and cyclic frequency can be separated from the mechanical driving force. If this is so, then the exponent m should be dependent only on the material, and should be constant for different temperatures and frequencies. To simplify the model evaluation, one may consider the energy term as a constant; and, since it is known that FCP rate depends on both temperature and cyclic frequency, Equation 4 may be rewritten as

$$\frac{\mathrm{d}a}{\mathrm{d}N} = \left(\frac{A'T}{f}\right)[(\Delta K - \Delta K_{th})^4/\sigma^2(K_c^2 - K_{max}^2)]^m \tag{5}$$

and,

$$\frac{\mathrm{d}a}{\mathrm{d}N} = \left(A'\frac{T}{f}\right)K^{*m} \tag{6}$$

where A' is a constant including the apparent activation energy. K^* represents the whole mechanical driving force term, in which K_c and σ have been corrected for temperature and frequency, but ΔK_{th} is assumed to be constant. Limited experimental results show ΔK_{th} to be insensitive to frequency at room temperature. Based on the current data, by plotting log ($\mathrm{d}a/\mathrm{d}N$) against K^*, m is found to vary from 0.17 to 1.1 and also depends on temperature. Therefore, strictly speaking, the M–M model as originally proposed is incorrect. One of the possible explanations for the variation in m is that the ΔK_{th} term depends on temperature [25]. At present, there is insufficient ΔK_{th} data for one to assess this possibility. A constant ΔK_{th} was used in calculating K^*. This could be a reason for the dependence of m on temperature.

The ultimate objective of any study of FCP in polymers is to contribute to the development of a general model to quantitatively describe fatigue crack propagation. The Michel–Manson model represents a step in this direction. However, refinements to the M–M model are necessary because it predicts the wrong frequency dependence for temperatures near T_g and it implies that the thermal and mechanical driving forces are clearly separable, a view contradicted by the present findings. In the course of trying to develop a general model for FCP, a number of regression analyses were tried wherein the data was divided into two groups, results satisfying either plane strain or plane stress conditions. Interestingly, both groups of data could be fit reasonably well (about 92% of the variability accounted for by regression) by a simple model of the form

$$\ln\left(\frac{da}{dN}\right) = A + B\ln(f) + \frac{C}{T} + D\ln\left(\frac{\Delta K}{K_c}\right) \tag{7}$$

where A, B, C, D are the regression coefficients. The only difference between both data sets was that in the case of plane stress conditions, namely near T_g, an additional term involving ln (plastic zone size) had to be included to achieve the same degree of fit for the regression analysis. Given the reversal in the effect of cyclic frequency on FCP in PMMA as temperature increases, one might construct a general model as the sum of two components each representing a different temperature regime. Such a model could have the form

$$\text{growth rate} = \left[1 - \left(\frac{T}{T_g}\right)\right]^2$$

$$\text{(low temperature regime)}$$

$$+ \left(\frac{T}{T_g}\right)^2 \text{(high temperature regime)} \tag{8}$$

4.5. Activation energy

In order to evaluate the thermal activation energy, FCP rates (da/dN) were plotted against the inverse of temperature as shown in Fig. 8 for $\Delta K = 0.6\,\mathrm{MPa\,m^{1/2}}$. Although da/dN is not a time dependent rate, it can be used as a rate variable (da/dt) by multiplying da/dN by the cyclic frequency (dN/dt) to give (da/dt). However, since cyclic frequency is constant, such a change will not affect the slopes of the straight line in Fig. 8 which are correlated to the apparent activation energy. These results show that the apparent activation energy is not constant, but rather decreases as frequency increases. A similar trend was also found $0.5\,\mathrm{MPa\,m^{1/2}}$ where apparent activation energy values were 33, 23 and $17\,\mathrm{kJ\,mol^{-1}}$ for frequencies of 1, 10 and 100 Hz, respectively. The dependence of the activation energy on frequency and ΔK is similar to what Phillips *et al.* [4] observed in PVC.

The measured activation energies are below values reported for the β process of PMMA (71 to $126\,\mathrm{kJ\,mol^{-1}}$) [17, 26]. The fact that the apparent activation energy for FCP is below that for the β process may be explained with the aid of the Zhurkov and Bueche model [27] wherein the apparent activation energy represents the difference between the activation energy for a process such as the β transition, and the product of stress and activation volume.

5. Conclusion

In order to evaluate the Michel–Manson model, the fatigue crack propagation rates of PMMA were studied for different temperatures and cyclic frequencies. According to these results, the conclusions are:

1. For plane strain conditions, the FCP rate increases with increasing temperature and decreasing cyclic frequency.
2. The crack growth rate shows a maximum near 80° C and 10 Hz.
3. Near the glass transition region, the failure mode is ductile and the material becomes more fatigue resistant while simultaneously softening. A triangular fracture surface feature indicating shear deformation is found under these conditions.
4. The frequency dependence of da/dN provided by the Michel–Manson model is shown to be valid up to about 80° C, in absence of extensive plastic deformation. FCP results exhibit an interaction between the thermal and mechanical driving forces which is not consistent with the M–M model.
5. The apparent activation energies of FCP processes increase with increasing ΔK and decreasing cyclic frequency.
6. The apparent activation energies for FCP are lower than the reported activation energy for the β transition.

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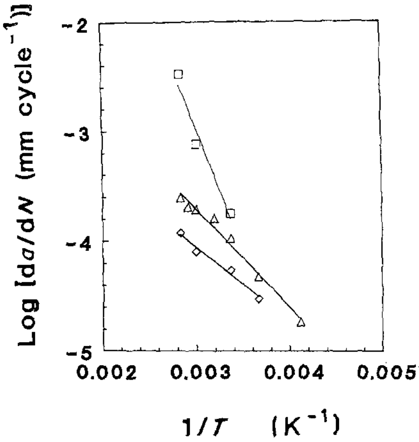

Figure 8 Arrhenius plot of fatigue crack propagation rates. It shows that the apparent activation energies are 44, 17 and $13\,\mathrm{kJ\,mol^{-1}}$ for frequencies of (□) 1, (△) 10 and (◇) 100 Hz, respectively. PMMA. $\Delta K = 0.6\,\mathrm{MPa\,m^{1/2}}$.

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