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Effects of Frequency on Fatigue Crack Propagation

in

Short-Glass-Fiber-Reinforced Nylon 66

by

Reinhold W. Lang, John A. Manson and Richard W. Hertzberg

Prepared for Publication

in

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FATIGUE CRACK PROPAGATION IN SHORT-GLASS-FIBER-REINFORCED NYLON 66:

EFFECT OF FREQUENCY

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ABSTRACT

The fatigue crack propagation (FCP) response of nylon 66 and composites containing 16 and 31 vol. % short glass fibers was determined at frequencies of 1, 10, 50, and 100 Hz. The sensitivity to frequency was found to depend on glass content and, in the case of the pure matrix, on the value of the stress intensity factor range, ΔK.

Whereas both glass-reinforced compositions were superior to the pure matrix material, an inversion in relative ranking of the former occurred as the cyclic frequency was changed. Also, the strong frequency sensitivity revealed by nylon 66 at low ΔK values disagrees with results reported previously. To explain these phenomena a concept based on the competition between localized and more generalized heating is proposed. For quantitative comparison, values determined for loss modulus as a function of temperature were correlated with the temperature profile measured at the crack tip by means of an infrared microscope. Localized heating at the crack tip is believed to decrease crack growth rates due to a drop in yield strength and the simultaneous increase in plastic zone size which dissipates more energy and effectively blunts the crack. Generalized heating in the bulk material ahead of the crack tip, on the other hand, has an adverse effect on growth rates as a result of an overall decrease in specimen stiffness.

INTRODUCTION

As short-fiber composites are often used in applications that involve cyclic loads, it is important to understand their fatigue

response. While failure of typical engineering plastics at a stress range, $\Delta\sigma$, close to the yield stress may involve gross yielding due to hysteretic heating, failure by propagation of a flaw is common at lower stresses.[1] In the case of reinforcement with short fibers of, for example, glass or carbon, fatigue life under constant-stress-range testing is often improved,[2] presumably in part due to a reduction in temperatures attained during cycling. Clearly it is of interest to examine the effects of cycling on fatigue crack propagation rates in such systems. Since the hysteretic heating rate per unit volume, $\dot{\varepsilon}$, under given loading conditions and heat transfer characteristics depends on the frequency both explicitly and implicitly through the loss compliance D" (equation 1)[3], the effect of frequency must be carefully considered. Specifically, $\dot{\varepsilon}$ is given by

$$\dot{\varepsilon} = \pi f \Delta\sigma^2 D''(f,T)/4 = \text{heat dissipated/time} \cdot \text{volume} \tag{1}$$

where f is the frequency, $\Delta\sigma$ the stress range, and D"(f,T) is the loss compliance as a function of frequency and temperature.

Indeed, reported effects of frequency in both unreinforced and reinforced polymers are complex, reflecting the fact that more is involved than just gross damage due to hysteretic heating. Thus extensive studies of FCP in engineering plastics[1] reveal that increasing frequency may increase, decrease, or not affect FCP rates. An explanation for such diverse behavior has been proposed in terms of a competition between local crack-tip heating (which can, in effect, blunt the crack) and generalized heating of the bulk material (which will lower its modulus). Of course, increasing frequency also increases the strain rate, thus increasing the modulus and decreasing creep[4]. Variable effects of frequency have also been noted in adhesive joints[5,6] and in continuous-fiber composites[7-9]. Within the latter systems sometimes more damage (reflected in lower moduli) is seen at lower frequencies[7], while sometimes complex effects of frequency have been observed[8,9].

With short-fiber systems, a few studies of FCP have been reported[10-12], but the performance relative to that of the pure matrix was not explicitly described. It was decided therefore to extend earlier studies of FCP in polymers to examine the effects of fiber parameters (e.g., volume fraction and orientation), interfacial characteristics, matrix, and frequency and environment. Following a preliminary paper on FCP in injection-molded nylon 66 composite systems[13], this paper examines the effect of frequency and glass content on FCP in short-fiber reinforced nylon 66 containing 1.7% water. By combining observations of temperature rise with measurements of damping, it will be shown that otherwise anomalous effects of frequency can be explained.

EXPERIMENTAL

Materials and Specimen Preparation

Three nylon 66 resins reinforced with various amounts of short glass fibers were supplied by the LNP Corporation:

B-N66: unreinforced nylon 66
B-16G: B-N66 containing 16 vol % (30 wt %) of short glass fibers
B-31G: B-N66 containing 31 vol % (50 wt %) of short glass fibers

The number-average molecular weight $\overline{M}_n$ of B-N66 was determined by dilute-solution viscometry using formic acid (88%) as a solvent, and found to be 1.9×10^4; details are described separately[14].

Values of percent crystallinity (by weight) were determined by both differential scanning calorimetry (Perkin-Elmer DSC model 1B) and by a density gradient column (DGC): 31(±1)% and 42% by DSC (average for all matrix materials) and DGC (for B-N66), respectively. These values agree well with those measured in this laboratory for other commercial nylon 66 materials (25 to 35, and 30 to 43% by DSC and DGC, respectively); the difference presumably reflects uncertainty in the values of heat of fusion and density for the 100%-crystalline reference. Since the material had been stored for several years at ambient conditions the moisture content was measured gravimetrically by drying specimens to constant weight at 105-110°C under vacuum. All matrix materials were found to contain 1.7 wt % water. The glass fibers used were of the E-glass type having an average diameter of 10 μm and a length of 6.4 mm before processing.

Specimens of the compact tension (CT) geometry[1] were cut in two different directions from injection-molded plaques so that the direction of the applied load was either transverse (T) or longitudinal (L) with respect to the major flow direction. Dimensions and gating of the plaques are shown in Fig. 1.

Fatigue Crack Propagation Tests

FCP experiments were conducted with an electrohydraulic closed-loop testing machine using standard procedures[1,14]. The applied waveform was sinusoidal with constant load amplitude and a minimum-to-maximum load ratio, R, of 0.1. Environmental conditions were laboratory air at 22-24°C and an average relative humidity of 40%. Tests were conducted at frequencies of 1, 10, 50 and 100 Hz, and the results plotted as log da/dN, the crack growth per cycle, as a function of log ΔK, the stress intensity factor range ($\Delta K = Y\Delta\sigma\sqrt{a}$ where Y is a geometrical variable, $\Delta\sigma$ the applied stress range, and $\underline{a}$ the crack length).
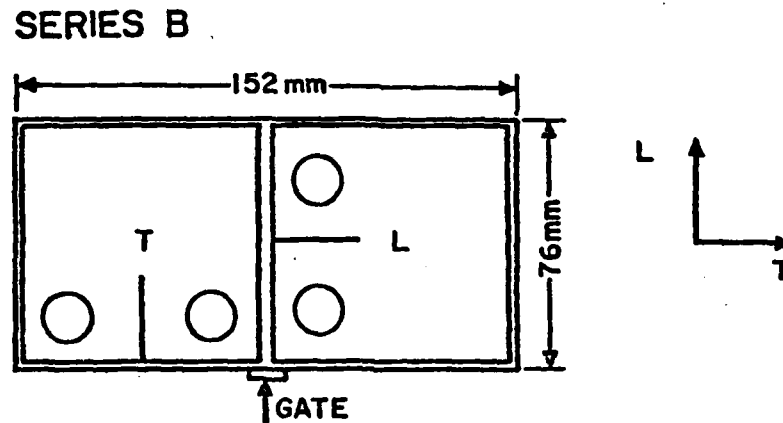
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Fig. 1. Geometry of injection molded plaques and orientation and
position of FCP specimens. T: direction of applied load
transverse to major flow direction; L: direction of applied
load longitudinal with respect to the major flow direction
(nominal plaque thickness = 3.2 mm).

Temperature Rise during FCP

Crack-tip temperatures and temperature profiles ahead of the
crack tip were measured using an infrared radiometric microscope,
model RM-2B, with a 15X objective corresponding to a spot size
0.075 mm (Barnes Engineering Company). Temperatures were recorded
after equilibrium was reached, before the FCP test was interrupted to
record the crack tip position. The estimated error increases from
±0.5°C at around 30°C, to ±(1-2)°C at 65°C, to ±3°C above 65°C.

Dynamic Mechanical Spectroscopy (DMS)

Dynamic mechanical spectra were recorded for each of the mate-
rials tested using an Autovibron apparatus (Model DDV-III-C, IMASS
Corporation, Hingham, MA). Test procedures are described and dis-
cussed by Webler, et al.[15] Specimens for these tests were cut and
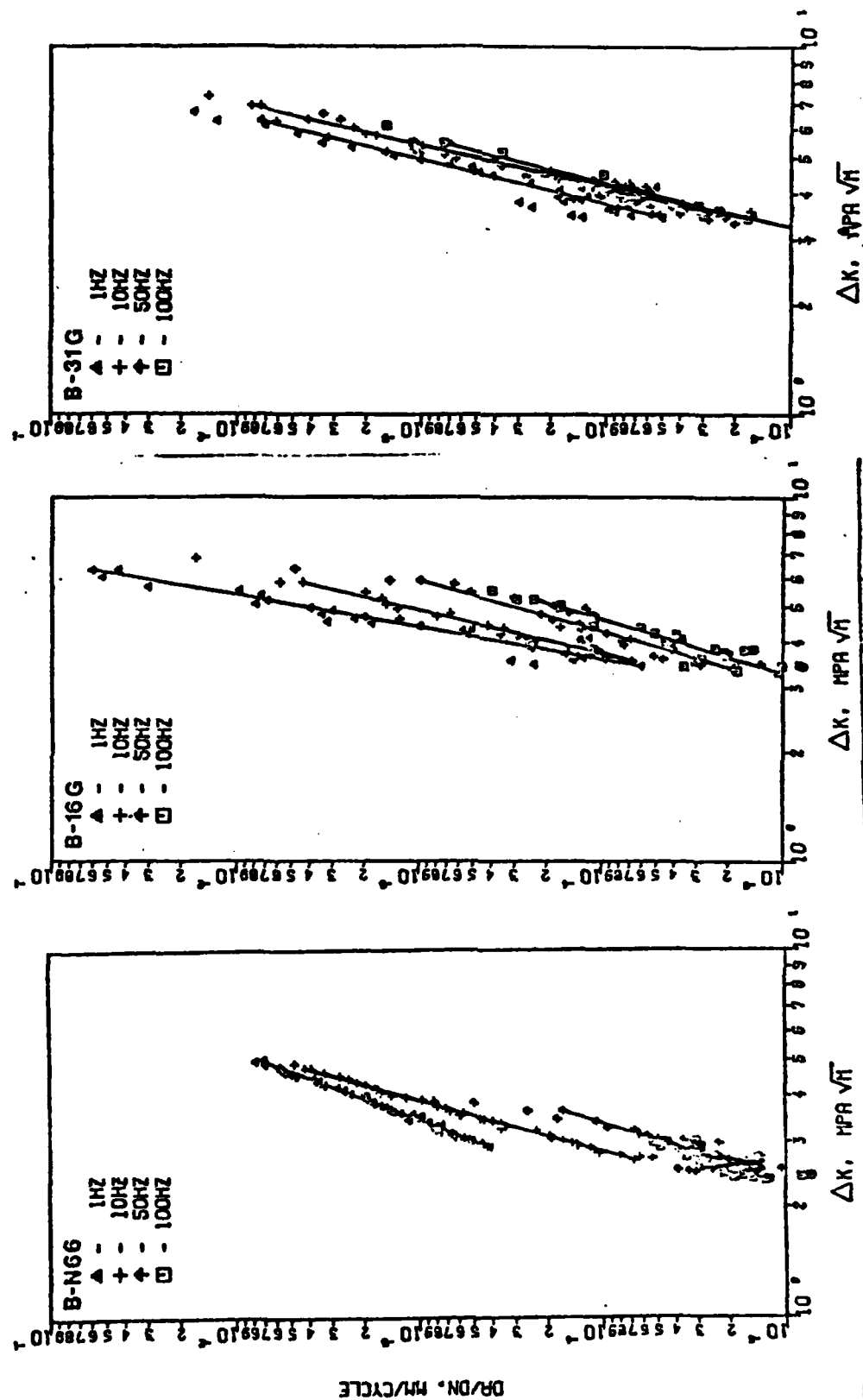loaded in the T-direction according to Fig. 1.

Fig. 2. Effect of frequency on FCP response of nylon 66 (B-N66) and composites containing 16 and 31 vol.% short glass fibers (B-16G and B-31G), respectively.

EXPERIMENTAL RESULTS

Effect of Frequency and Fiber Content on FCP

The effect of test frequency on FCP behavior of B-N66, B-16G and B-31G in the frequency range of 1 to 100 Hz is shown in Fig. 2. Although there is some scatter in the data for the glass-reinforced compositions and for the pure B-N66 at 50 and 100 Hz, consistent with a discontinuous crack growth mechanism, the behavior can be described reasonably well by the Paris relationship[16] [$da/dN = A \Delta K^n$, with material variables A and n given in Table 1]; this relationship has been found to hold in several other short-fiber composite systems[5,6,10-13], as well as in some continuous-fiber systems[17,18]. While crack growth rates in all three systems were found to show a positive frequency dependence (i.e., the higher the frequency the lower the growth rate per cycle), the degree of frequency sensitivity varied with glass content. Thus Fig. 3, in which da/dN for different ΔK levels is plotted versus test frequency on a double logarithmic scale, reveals

Table 1. Parameters[a] A and n of the Paris Relationship for B-N66, B-16G and B-31G

Frequency (Hz)	B-N66 A^b	n	B-16G A^b	n	B-31G A^b	n
1	1.6×10^{-6}	5.2	4.8×10^{-11}	11.4	7.1×10^{-9}	7.4
10	5.9×10^{-8}	7.1	1.5×10^{-9}	8.5	5.4×10^{-10}	8.6
50	1.0×10^{-8}	7.6	3.3×10^{-9}	7.1	2.1×10^{-10}	9.1
100	1.3×10^{-7}	5.3	3.0×10^{-9}	6.8	9.9×10^{-10}	8.0

[a]Determined by regression analysis

[b]Units = [$(mm/cycle) \times (MPa\sqrt{m})^{-n}$].

that nylon 66 containing 16% (vol.) glass fibers is the most frequency-sensitive composition.

The high frequency sensitivity of the composition with an intermediate glass content has an important effect on the relative ranking of these materials with respect to FCP. Whereas both glass-reinforced systems are superior to the pure matrix material over the whole frequency range, an inversion in relative ranking of B-16G and B-31G takes place at around 50 Hz (see Fig. 3). At low cyclic frequencies of 1 and 10 Hz, respectively, crack growth rates at any given ΔK value are higher for B=16G than for B-31G, whereas at 50 Hz no significant difference in FCP behavior could be found. Further
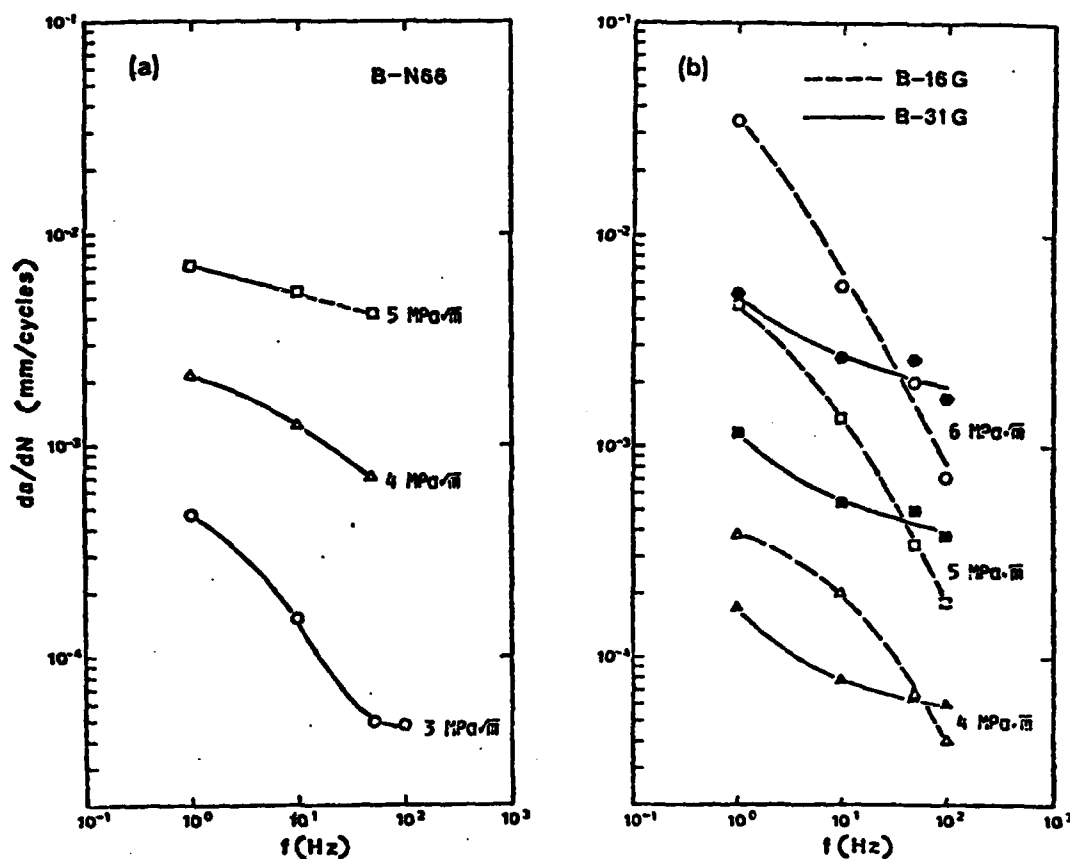
Fig. 3. Effect of frequency on FCP rates for nylon 66 and composites. Numbers at right of each curve give ΔK at which the rate is measured.

increase in frequency to 100 Hz leads to the above-mentioned inversion, with B-16G being somewhat more resistant to FCP than B-31G.

Effect of Orientation on FCP

The results presented in Figs. 2 and 3 were obtained from specimens cut out in the T-configuration according to Fig. 1. To investigate the effect of fiber orientation on FCP, specimens were also tested in the L-configuration at 10 and 100 Hz; however, no significant differences with respect to orientation could be found. A model explaining this somewhat unexpected behavior is discussed in an earlier paper[13]. Thus the growing crack perceives a quasi-isotropic fiber array, even though the specimens can be considered in effect as laminates of plies having different fiber orientations.

Reproducibility of FCP Test

Reproducibility experiments conducted at 10 and 100 Hz for the T-configuration showed excellent agreement. Maximum differences in

growth rates were less than 20% for B-16G and B-31G, respectively.
In the case of B-N66 the results obtained from two different tests
were essentially identical.

Hysteretic Heat Generation

During the course of the fatigue tests significant temperature
rises at the crack tip at frequencies higher than 10 Hz were observed
and first measured by means of a thermocouple. To record the crack
tip temperature more accurately an infrared radiometric microscope
was used, a technique applied also by Attermo and Östberg[19] for
poly(vinyl chloride), poly(methyl methacrylate) and polycarbonate.

Several attempts have been made to describe the heat generation
and the associated temperature rise of polymers subjected to cyclic
loading (for a review, see reference 1, ch. 2). While most of the
equations proposed cannot predict absolute temperature rises since
heat losses to the surrounding environment are not accounted for (for
exceptions, see Crawford and Benham[20] and Oberbach[21]) they do show
the influence of the major variables associated with hysteretic
heating. For example, in reducing equation (1) the temperature
rise per unit time (neglecting heat losses), $\Delta\dot{T}$, becomes

$$\Delta\dot{T} = \frac{\pi f D''(f,T,HOH)\Delta\sigma^2}{4c_p\rho} \tag{2}$$

where f is the frequency, D'' the loss compliance under constant
stress conditions, $\Delta\sigma$ the stress range, ρ the density, and c_p the
specific heat. Note that D'' itself is a function of frequency and
temperature, and, in the case of nylon, of water content; c_p may
also vary with temperature and water content.

Effect of Frequency on Hysteretic Heating

In Fig. 4 the crack tip temperature is plotted versus test
frequency at a ΔK of 2.6 and 3.1 MPa$\sqrt{m}$ for the unreinforced and
glass-reinforced compositions, respectively. For the latter mate-
rials, a linear relationship as in eq. 3 fits the data well:

$$\Delta T = C(\Delta K, v_f) \cdot f \tag{3}$$

where ΔT is the temperature rise at the crack tip, f is the frequency
and C, the slope, is a function of the applied ΔK and fiber volume
fraction, v_f. It should be noted that this first-order dependence
of ΔT on frequency is in agreement with equation (2). The devia-
tion from linear behavior observed for B-N6 at higher frequencies
(>40 Hz) is possibly related to the higher heat losses to be expected
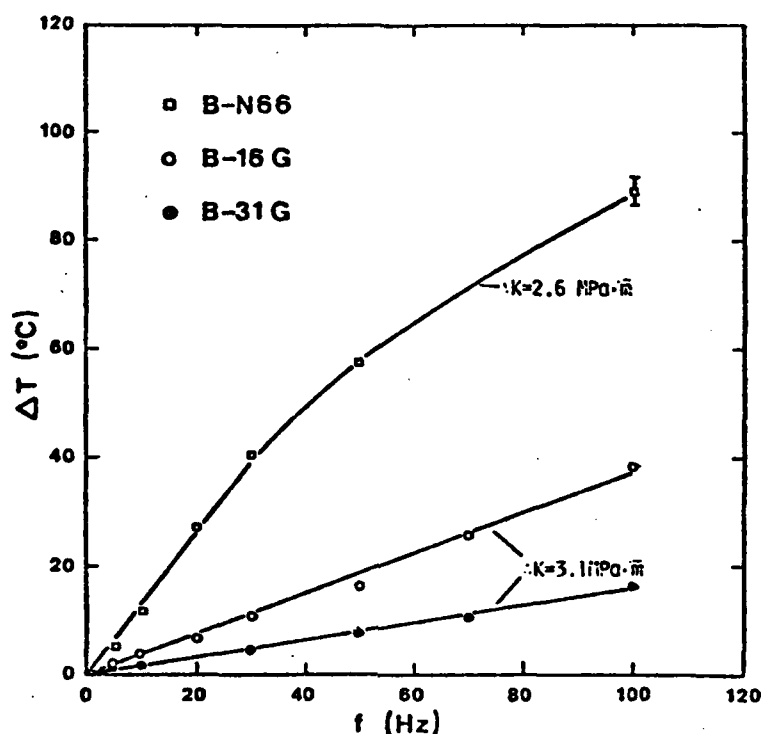at the higher temperatures attained. Furthermore, as will be seen

Fig. 4. Effect of frequency f or crack tip temperature rise ΔT for
B-N66, B-16G and B-31G [ΔT=C(ΔK,v_f)·f].

later, D" reaches a maximum at about 80°C, corresponding to a ΔT of
about 55°C at ≈50 Hz, and decreases again at higher temperatures.
This fact may also have contributed to the observed nonlinearity of
ΔT versus f for B-N66 at higher frequencies.

Effect of ΔK on Hysteretic Heating

The effect of the applied stress intensity factor range ΔK on
the crack-tip temperature rise is shown in Fig. 5 for B-N66 at 10 Hz
and for B-16G and B-31G at 10 and 100 Hz. It was found empirically
that the data can be described to a first approximation by an equa-
tion of the form

$$\Delta T = B(f, v_f) \, \log \frac{\Delta K}{\Delta K_{T,th}} \tag{4}$$

where the constant B is a function of frequency and glass content
and $\Delta K_{T,th}$ is the empirical threshold value for ΔK below which no
detectable temperature rise occurs. [In other cases, a simple
power dependence of ΔT on ΔK may be more appropriate.[22]] It is
interesting that the defined parameter $\Delta K_{T,th}$ has a value in these
systems between 2.2 and 2.4 MPa√m (independent of frequency and
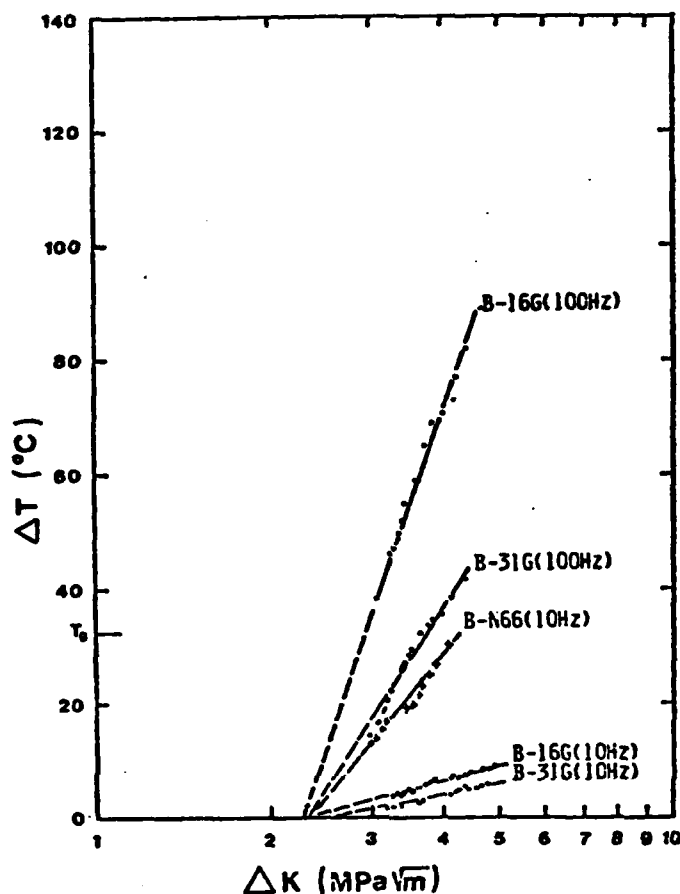
Fig. 5. Effect of stress intensity factor range on crack tip
temperature rise in B-N66 at 10 Hz and in B-16G and B-31G
at 10 and 100 Hz.

glass content) — a range of ΔK corresponding to the passage of most
of the fatigue life. If indeed $\Delta K_{T,th}$ should turn out to be a
true material property, its existence would imply that the concepts
of linear elastic fracture mechanics could be valid for cases in
which most of the fatigue life is passed at values of $\Delta K < \Delta K_{T,th}$.
An analogous transition point separating thermally- from non-
thermally-controlled fatigue failure in unnotched samples has been
characterized by Crawford and Benham (see ref. 1, ch. 2) and defined
as the change-over stress. However, one must distinguish between
thermal failure where actual melting occurs (as in unnotched fatigue
tests) and thermally influenced FCP ($\Delta K > \Delta K_{T,th}$) where mechanical
failure still prevails (as in precracked samples). Clearly more
evidence is needed in order to assess the theoretical significance
of this presently empirical parameter.

Temperature Profiles

Temperature profiles ahead of the crack tip were recorded at various ΔK levels. Fig. 6 shows typical curves for the temperature rise as a function of the distance from the back edge of the sample. The location of the crack tip is indicated by arrows. As expected, ΔT decreases exponentially as the distance from the crack tip increases.

Dynamic Mechanical Spectroscopy (DMS)

Since DMS is a useful technique to obtain elastic and visco-elastic parameters of polymeric materials subjected to cyclic loading, the storage modulus E' and the loss modulus E'' were determined over a temperature range from $-120°C$ to $+180°C$ at a frequency of 110 Hz (Fig. 7). With respect to the interpretation of the FCP results it is interesting to note that the glass transition temperature T_g, here taken as the temperature at which E'' reaches a maximum, is in all cases in the range 50 to 60°C.

To evaluate the relative ranking of the investigated materials as to their ability for heat generation in terms of eq. (2), the temperature dependence of D'' is required (see Fig. 8). Equation (4) was applied:

$$D'' = \frac{E''}{E'^2 + E''^2} = \frac{1/E''}{1+(\tan^2 \delta)^{-1}} \tag{4}$$

The following observations may be made with respect to hysteretic heating during a fatigue test. While dry nylon 66 heats up only slightly[23], extensive heating was observed when 1.7% water was present. However, on adding glass fibers the tendency for heat generation decreases as the fiber content increases. (See Figs. 4 to 6.) The following facts can explain the latter behavior:

(1) the higher the fiber content, the lower the amount of material with high damping.

(2) the higher the fiber content, the greater the thermal conductivity and consequent heat loss.

(3) the higher the fiber content, the lower the average stresses in the matrix at a given applied load (in an isotropic or quasi-isotropic system).
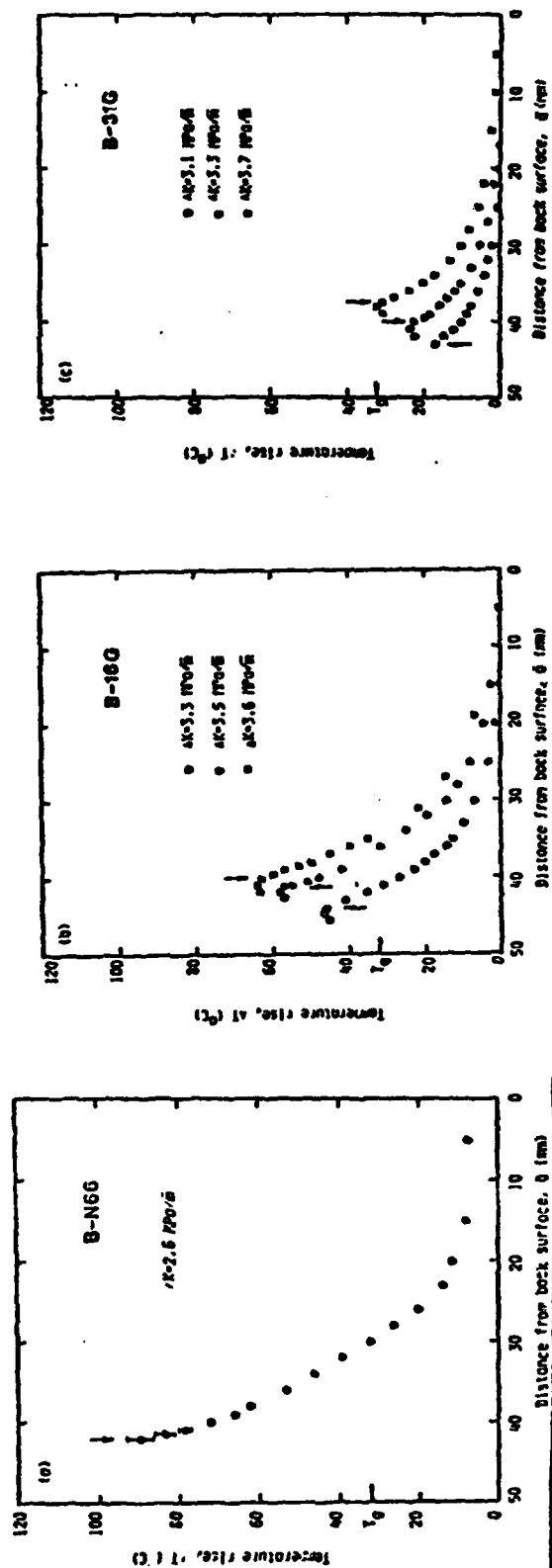
Fig. 6. Temperature rise ahead of the crack tip in B-N66, B-16G, and B-31G as a function of the distance from the back (unnotched) edge of the sample. Arrows indicate location of the crack tip. Frequency: 100 Hz.

(4) the lower the fiber content, the higher D''_{max} and the higher the tendency for autoaccelerated heatup (see Fig. 8).

While the data in Fig. 8 are for a frequency of 110 Hz, the relative behavior at other frequencies is expected to be at least qualitatively similar.
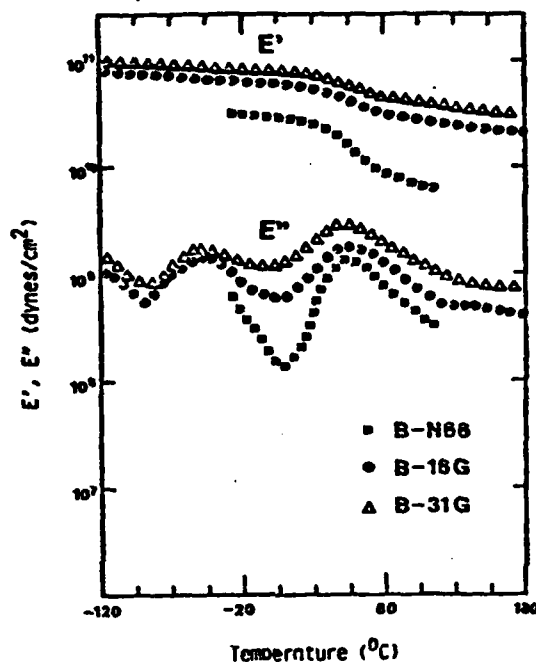
Fig. 7. Dynamic mechanical spectra for B-N66, B-16G, and B-31G.

DISCUSSION

Since cyclic growth rates are frequently related to the plastic zone size at the crack tip[24] or to the crack-opening displacement (COD)[25,26], let us examine the dependence of these parameters on frequency. Irwin[27] derived the size of the plastic zone, r_y, for plane stress conditions from stress field equations by setting the y component of stress, σ_y, equal to the yield strength, σ_{ys}:

$$r_y \simeq \frac{1}{2\pi} \left(\frac{K}{\sigma_{ys}}\right)^2 \quad \text{(plane stress)} \tag{6}$$

For conditions of plane strain r_y is smaller, and given by[28]

$$r_y \simeq \frac{1}{6\pi} \left(\frac{K}{\sigma_{ys}}\right)^2 \quad \text{(plane strain)} \tag{7}$$
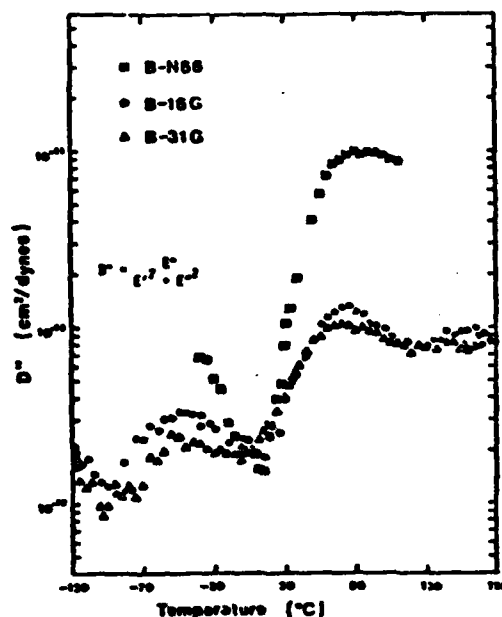
Fig. 8. Dynamic loss compliance D" as a function of temperature for B-N66, B-16G, and B-31G.

The crack-opening displacement, δ, at the crack tip is given by[26,29]

$$\delta \simeq \frac{K^2}{E\sigma_{ys}} \tag{8}$$

At constant ΔK, clearly factors which decrease σ_{ys} will increase r_y, while factors which decrease σ_{ys} or E will increase δ. A temperature rise due to a frequency increase will therefore _increase_ both r_y and δ, while the associated increase in strain rate may be expected to _decrease_ r_y and δ, at least to some extent. An increase in frequency can clearly have contradictory effects.

The next question is the role of δ and r_y in determining FCP rates. In general, it is desirable to keep δ as small as possible to avoid reaching the critical value required for crack extension. The case of r_y seems to be not so straightforward. While it has been suggested that suppressing the plastic zone at the crack tip should increase the resistance to cyclic crack propagation[4], we believe that an _increase_ in plastic zone size may be beneficial because it results in more effective crack tip blunting and dissipates more energy at the crack tip. Consequently, according to equations 6 and 7 ($r_y \propto 1/\sigma_{ys}^2$), any mechanism leading to a decrease in σ_{ys} on a localized scale at the crack tip is expected to lead to an _increase in FCP resistance_. Furthermore, in general the elonga-

tion at yield, ε_{ys}, and at break, ε_b, also increase with decreasing σ_{ys} (e.g. due to a temperature rise[30]), thus allowing for a larger critical COD, δ_c, for crack extension. The localization of the phenomena concerned should be important, for r_y may be expected to depend on the local value of σ_{ys} at the crack tip whereas δ is controlled by the modulus and yield strength profile of the whole unbroken ligament. However, since the temperature rise of the materials tested does not only occur in a localized region at the crack tip but also can be seen to be significant some distance away from it, Fig. 8, two competitive events occur simultaneously. Hence, with high damping polymers and their composites the overall effect of frequency on fatigue crack growth rates will be determined by a balance between the

- temperature rise at the crack tip resulting in an <u>increase</u> in r_y and δ_c (beneficial), and the

- temperature rise of the bulk material ahead of the crack tip resulting in a modulus decrease and a simultaneous <u>increase</u> in crack opening displacement (detrimental).

This concept also is in agreement with the conclusion from experimental findings (see ref. 1, ch. 3) that localized heating at the crack tip leads to attenuated crack growth rates, but generalized heating of the bulk material should be deleterious to FCP resistance.

In the case of the materials studied, it seems likely that the effect of frequency is dominated by consequences of hysteretic heating, for σ_{ys} is only slightly increased by an order-of-magnitude increase in strain-rate but greatly decreased by relatively small increases in temperature[30], and the modulus is nearly independent of frequency in the range studied[31].

The Matrix: B-N66

At first glance, the qualitative trends in FCP rates with frequency (Fig. 3) appear to be consistent with the arguments presented above. Certainly the increase in temperature at the crack tip should favor blunting of the crack tip. However, as shown in Figs. 4, 6, and 8, the matrix material shows a much stronger tendency towards hysteretic heating at and ahead of the crack tip than is the case with the fiber-reinforced specimens. Indeed, with the temperatures noted, one might expect to find a deleterious effect of increasing frequency due to a severe reduction in E and σ_{ys} in the bulk material with a consequent large increase in δ. It should also be noted that the strong sensitivity to frequency seen appears to contradict other findings reported previously[23,31] for nylon 66. [Such conflict is common in the literature[1,4,32,33].] In addition, crack growth rates were found to be lower than those reported by Bretz et al.[14,23], who used the same test procedure and comparable

materials with respect to water content, molecular weight and crys-
tallinity. Clearly some factor must exist that can overcome the
otherwise deleterious effects of high heat generation ahead of the
crack tip.

In fact, there is one difference between this study and those
of Bretz et al.[14,23]: the specimen thickness. Bretz et al. used
specimens having a nominal thickness of 6.2 mm, whereas our speci-
mens were 3.2 mm thick. As pointed out earlier[13], it seems likely
that differences in specimen thickness cause the observed variations
in FCP response due to differences in the stress state. Thus the
thinner the specimen, the greater is the tendency toward plane-
stress rather than plane-strain conditions, the larger is r_y and
the higher the expected resistance to FCP. Similar effects of thick-
ness on FCP have been reported for polycarbonate[34,35]. Indeed, a
preliminary examination of the fracture surface revealed the presence
of shear lips (indicating plane stress conditions) whose width in-
creased with ΔK (at constant frequency) and with frequency (at con-
stant ΔK). This phenomenon (not present in the reinforced samples)
reflects an additional contribution to the dissipation of energy, and
is consistent with the occurrence of a frequency-induced drop in
σ_{ys} ahead of the crack tip, and with the concomitant decrease in FCP
rates observed (for the relationship between shear lip width and
σ_{ys} see ref. 1, ch. 4).

As a result of the change in stress state the question of its
effect on the frequency sensitivity arises. The following reasons
can be put forth to explain why the frequency dependence might change
with specimen thickness. In general, the temperature rise under
cyclic loading conditions will decrease as specimen thickness de-
creases since the heat transfer to the surrounding environment is
enhanced by the increase in the surface-to-volume ratio of the
specimen. Thus, in the case of a precracked FCP specimen, the bulk
material ahead of the crack tip is expected to heat up less as the
specimen thickness decreases. On the other hand, it may well be
that the temperature rise on a local scale at the crack tip increases
as the specimen thickness decreases due to the larger plastic zone
volume associated with plane stress conditions (compare equations
6 and 7). Hence progressively more heat will be generated at the
crack tip at higher frequencies with thinner specimens leading to a
larger drop in σ_{ys} and in turn to an increase in plastic zone size.
As discussed earlier, both effects (larger decrease in σ_{ys} at crack
tip and less decrease in stiffness of the bulk material with increas-
ing frequency in thinner specimens) should be beneficial in terms of
FCP thus explaining the increased frequency sensitivity of thinner
specimens at low values of ΔK.

The effect of ΔK on the <u>relative</u> frequency sensitivity is also
of interest. From Fig. 3a it is evident that the frequency sensi-
tivity of B-N66 varies with the applied ΔK range, being highest at
the lowest ΔK. An explanation for this finding is the favorable
tendency towards the localized temperature increase at smaller crack

lengths (i.e. at low values of ΔK). As pointed out previously (1, ch. 3), the distinction between localized and generalized heating depends on the size of the hysteretically heated damage zone relative to the dimensions of the entire specimen. An arbitrary way to quantify this statement for the case of FCP specimens is to define a quantity R_L as the ratio of the length of the unbroken ligament experiencing a temperature rise above a reference temperature T_{ref}, e.g. $T_g \approx 55°C$, to the length of the ligament that remains below T_{ref}. To gain both the low yield strength at the crack tip and the high modulus or stiffness of the unbroken ligament, R_L should be small.*

It is apparent from the trends shown by the temperature distributions in Fig. 6 that the ratio R_L, which reflects the competition between localized and generalized heating, shifts towards the latter (i.e. increasing R_L) as the crack length and consequently ΔK increase. Hence, the benefits gained with increasing frequency at low ΔK (small R_L) diminish due to the increasing tendency for generalized heating at high ΔK (larger R_L) leading to the decrease in frequency sensitivity with increasing ΔK. Again we see a complex balancing of effects.

The Reinforced Specimens

Generally similar arguments may also be used for the glass-reinforced compositions. However, instead of using the term "plastic zone" it seems more appropriate to use the term "damage zone" with regard to the plastically deformed volume at the crack tip. As with the neat matrix, the positive frequency sensitivity, (see Figs. 2 and 3b) is believed to be caused by crack-tip heating that leads to a decrease in σ_{ys} and an increase in ε_b, which in turn leads to an increase in damage-zone size. Visual observations revealed that the size of the damage zone at the crack tip increased relatively more with increasing frequency in B-16G than in B-31G in which a change is barely detectable. Clearly a larger damage zone is desirable because of both the higher energy necessary to creat it and the increased blunting effect at the crack tip. In fact, the diffuse damage zone in continuous-fiber-reinforced plastics is known to blunt the effect of a flaw; discontinuous crack growth is often observed (see ref. 1, ch. 5).

The larger increase in damage zone size with frequency for B-16G is rationalized as follows. From Fig. 4 it can be seen that the crack-tip temperature rise ΔT varies more with frequency in B-16G than in B-31G. Moreover, although the dependence of ΔT on ΔK is only moderate for the composites at 10 Hz, it becomes more prominent as the frequency is increased (Fig. 5). Most important,

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*This statement is true only if the region experiencing at temperature rise is close to the plastic zone in size. It cannot be applied to the limiting case of $R_L = 0$ in which no heatup occurs at all.

however, whereas the crack tip in B-16G at 100 Hz is above the glass transition temperature T_g ($T_g \sim 55°C \equiv \Delta T \sim 32°C$) over the whole ΔK range this is not the case for B-31G which heats up much less at the same frequency. Hence, the larger decrease in σ_{ys} with frequency for B-16G causes the observed change in damage zone size (recall equations 6 and 7).

Another way to view this is to consider Fig. 6b, which shows that the temperature at $\Delta K = 3.3$ to 3.6 MPa$\sqrt{m}$ drops below T_g at a distance less than 8 mm ahead of the crack tip so that most of the unbroken ligament still retains a high modulus. Hence it is concluded that the higher frequency sensitivity for B-16G and the inversion in the relative ranking of the glass-reinforced materials in terms of their FCP resistance at different frequencies is a result of the higher frequency-induced temperature rise at the crack tip in B-16G and its effect on yield strength and damage zone size.

One final aspect worth consideration in the case of composites experiencing a temperature change is that of thermally induced internal stresses. In cooling a glass reinforced polymer from processing temperature to room temperature, tensile stresses develop in the matrix in a tangential direction around the fibers. These can be caused simply by the mismatch in the thermal coefficients of expansion or by shrinkage of the matrix due to morphological changes, e.g. crystallization. An increase in temperature as observed in our FCP experiments should either decrease these tensile stresses, or if stress relaxation occurs over a long period of storage time (or due to annealing) should result in compressive stresses in the matrix. Accordingly, a temperature increase is expected to be beneficial from the standpoint of internal stresses.

CONCLUSIONS

Several observations and conclusions may be summarized:

1. By testing the FCP behavior of nylon 66, and nylon 66 containing 16 and 31 vol % of short glass fiber, respectively, it was found that the Paris law holds reasonably well.

2. While crack growth rates for all materials decreased with increasing frequency in the range of 1 to 100 Hz, the degree of frequency sensitivity was discovered to depend on the glass content and in the case of unreinforced nylon 66 also on ΔK. Whereas both glass-reinforced compositions were superior to the pure matrix material an inversion in relative ranking of the former occurred as the cyclic frequency was changed. Also, the strong frequency sensitivity revealed by nylon 66 at low ΔK values disagrees with results reported previously.

3. To explain these phenomena a concept based on the competition between localized and more generalized heating has been used successfully. Localized heating at the crack tip is believed to decrease crack growth rates due to a drop in yield strength and the simultaneous increase in strain capability. This in turn increases the plastic zone size which dissipates more energy and effectively blunts the crack. Generalized heating in the bulk material ahead of the crack tip, on the other hand, has an adverse effect on growth rates as a result of the increase in crack opening displacement which is a consequence of the decrease in modulus.

4. The combination of measurements of loss compliance as a function of temperature with direct measurements of temperature profiles at the crack tip provides a powerful approach to explaining effects of frequency on FCP in nylon 66 and short-glass-fiber reinforced nylon 66, and presumably in other polymeric systems as well.

ACKNOWLEDGMENTS

REFERENCES

1. R. W. Hertzberg and J. A. Manson. "Fatigue in Engineering Plastics," Academic Press, New York, 1980.
2. J. Theberge, B. Arkles, and R. Robinson, Ind. Eng. Chem., Prod. Res. Dev. 15:100 (1976).
3. J. D. Ferry, "Viscoelastic Properties of Polymers," 2nd ed., Wiley, New York, 1970.
4. H. A. El-Hakeem and L. E. Culver, Int. J. Fatigue, 1:133 (1979).
5. S. Mostovoy and E. J. Ripling, Polym. Sci. Technol. 9B, 513 (1975).
6. J. A. Marceau, Y. Moji, and J. C. McMillan, paper presented at the SAMPE Symp., 21st April 6-8 (1976).
7. K. L. Reifsnider, W. W. Stinchcomb, and T. K. O'Brien, ASTM STP 636, p. 171 (1977).
8. W. W. Stinchcomb, K. L. Reifsnider, L. A. Marcus, and R. S. Williams, ASTM STP 596, p. 115 (1975).
9. C. T. Sun and W. S. Chan, ASTM STP 674, 418 (1979).
10. A. T. DiBenedetto and G. Salee, Polym. Eng. Sci. 19:512 (1979).
11. P. A. Thornton, J. Composite Mater. 6:147 (1972).
12. M. J. Owen and P. T. Bishop, J. Phys. D: Appl. Phys. 7:2036 (1974).

13. R. W. Lang, J. A. Manson and R. W. Hertzberg, Org. Coatings and Plast. Chem. 45:778 (1981).

14. P. E. Bretz, R. W. Hertzberg, and J. A. Manson, in press, J. Mater. Sci., 1981.

15. S. M. Webler, J. A. Manson, and R. Lang, Polymer Preprints, 22:1, p. 257, March, 1981.

16. P. C. Paris and F. Erdogan, J. Bas. Eng. Trans. ASME Ser. D85: 4, 528 (1963).

17. M. D. Campbell and B. W. Cherry, in "Fracture Mechanics and Technology," (G. C. Sih and C. L. Chow, eds.), Vol. 1, p. 297. Sijthoff and Noordhof, Alphen aan den Rijn, The Netherlands, 1977.

18. D. J. Wilkins, this publication.

19. R. Attermo and G. Östberg, Int. J. Fract. Mech. 7:122 (1971).

20. R. J. Crawford and P. P. Benham, J. Mater. Sci., 9:18 (1976).

21. K. Oberbach, Kunstoffe 63:35 (1973).

22. C. M. Rimnac, Ph.D. research, Lehigh University, 1981.

23. P. E. Bretz, R. W. Hertzberg, J. A. Manson, M. T. Hahn and R. W. Lang, "Effect of Test Frequency and H_2O Content on Localized Crack-tip Heating in Nylon 66," to be published.

24. A. J. McEvily, R. C. Boetner and T. L. Johnson, in "Proceedings of 10th Sagamore Army Materials Research Conference," Syracuse University, New York (1963), p. 95.

25. L. R. Hall and R. C. Shah, Eng. Fract. Mech. 3:169 (1971).

26. J. G. Williams, J. Mater. Sci., 12:2525 (1977).

27. G. R. Irwin, in Handbuch der Physik, Vol. VI, S. Flugge, ed., Springer, Berlin (1958), p. 551.

28. F. A. McClintock and G. R. Irwin, ASTM STP 381:84 (1965).

29. G. R. Irwin, Eng. Fract. Mech. 1:231 (1968).

30. M. I. Kohan, "Nylon Plastics," Wiley, New York, 1973.

31. R. W. Hertzberg, M. D. Skibo, J. A. Manson, and J. K. Donald, J. Mater. Sci. 14:1754 (1979).

32. S. Arad, J. C. Radon, and L. E. Culver, J. Appl. Polym. Sci. 17:1967 (1973).

33. S. Arad, J. C. Radon, and L. E. Culver, Eng. Fract. Mech. 4:511 (1972).

34. J. A. Manson and R. W. Hertzberg, Crit. Rev. Macromol. Sci. 1:433 (1973).

35. G. Pitman and I. M. Ward, J. Mater. Sci., 15:635 (1981).

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