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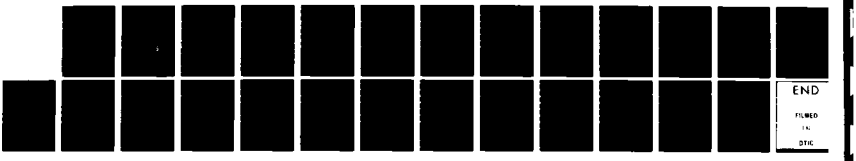

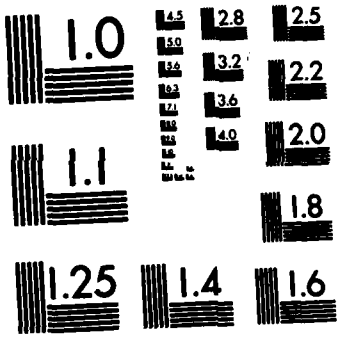

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by

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Abstract

Fatigue crack propagation (FCP) tests were conducted on nylon 66 specimens equilibrated over a range of moisture levels and test frequencies to determine the crack growth rates and the crack-tip temperatures as a function of water content. Frequency-sensitivity was correlated with the amount of crack-tip heating taking place, with crack-tip temperature being found to depend strongly on the estimated loss compliance, D", of the material. The frequency-sensitivity of FCP in nylon was seen also to be affected by mean stress, suggesting that creep processes are often significant in FCP of nylon.

Introduction

When conducting fatigue tests with polymeric solids, one must be ever-mindful of the potential for cyclic-stress-induced heating of the test plaque due to hysteretic energy losses within the material. Under certain conditions, for example, fatigue failures can occur in unnotched specimens because the sample literally melts or is heated to the point where it becomes excessively compliant relative to the displacement capacity of the fatigue test apparatus. Testing procedures for such unnotched specimens and associated failure criteria (including melting) have been established, and are described by ASTM Standard D-671 (1); the relevant literature is reviewed elsewhere (2). As pointed out by Ferry (3), the rate of hysteretic energy dissipation per unit volume, $\dot{\epsilon}$, in a constant stress range experiment varies directly with the test frequency, ω (rad/s), the square of the stress amplitude, σ_o, and the loss compliance of the material, D", as described by a relationship of the form

$$\dot{e} = \omega D'' \sigma_o^2/2 \quad (1)$$

Converting frequency to Hz, and stress amplitude to stress range $\Delta\sigma$ ($\sigma_o = \Delta\sigma/2$), we have

$$\dot{e} = \pi f D'' \Delta\sigma^2/4 \quad (2)$$

Note that the loss compliance is, in turn, a function of the test frequency, f, and temperature T, and structural or compositional factors. For example, D" in nylon 66 (N66) is strongly influenced by the presence of imbibed water. For convenience, D" may also be expressed in terms of dynamic moduli as follows:

$$D'' = \frac{E''}{(E'^2 + E''^2)} \quad (3)$$

where E" and E' are the loss and storage tensile moduli, respectively. (In the case of a constant-strain-range experiment, the loss modulus, E", is the appropriate dissipation term in Equations 1 and 2.)

When fatigue specimens are pre-notched, such hysteretic heating is typically localized near the crack tip plastic zone where the stresses are greatest. The authors recognize that the loss compliance, D", depends on the magnitude of the applied stress and the stress state in addition to the previously mentioned variables. For this reason, it may not be possible to predict the actual crack tip temperature from Eq. 2. Nevertheless, this relationship does show the influence of the major variables associated with hysteretic heating and has been often applied successfully in the analysis of large-strain engineering problems (3b). For example, Attermo and Ostberg (4) used an infrared camera to record a maximum increase in crack-tip temperature of up to 30°C in fatigue tests at 11 Hz of poly(vinyl chloride) (PVC), poly(methyl methacrylate) (PMMA), and polycarbonate (PC). A much larger crack-tip temperature increase of 100°C was measured by a copper-constantan thermocouple in an impact-modified blend of nylon 66 (equilibrated at room temperature to a water content of 2.0%) when tested at 30 Hz (5). By comparison the temperature rise at the crack tip was less than 35°C when the modified nylon was tested at 10 Hz and in the dry-as-molded condition. These latter results are consistent with Equation 2 to the extent that absorbed water (contributing in this case to an increase in D" at the temperature concerned) and higher test frequencies should lead to greater crack-tip heating under cyclic loading conditions.

Higher crack-tip temperatures were recorded in these materials during preliminary experiments with an infrared microscope. This is to be expected since the latter device has much better spatial resolution than a thermocouple. Moreover, the infrared microscope is a non-contacting device and thus does not interfere with the heat flow in a sample, while thermocouples can act as heat

The influence of test frequency on fatigue crack propagation rates has received separate and considerable attention. Of particular note, the frequency sensitivity for a given polymer was found to depend strongly on the frequency of movement of main chain segments responsible for generating the principal secondary transition peak (usually the β peak) at a given test temperature (2,6,7). In this regard, the greatest sensitivity of FCP to frequency occurred when the test frequency was close to the frequency of the β-process at the temperature concerned. It was argued that, when such conditions are met, localized crack-tip heating would occur to lower the yield strength, thus increasing the plastic zone size and acting to blunt the crack tip (8). Since this reduces the severity of the stress intensity at the crack tip, it would be expected that the rate of fatigue crack propagation would decrease. The observed lack of frequency sensitivity in dry-as-molded nylon 66 at room temperature was said to be due to the large difference between the test frequency and the β frequency. However, there have been other reports of frequency sensitivity in nylon (9 - 11) and further work has been done by the present authors in an attempt to reconcile this apparent discrepancy. This paper reports on further measurements of crack-tip temperatures and crack growth rates in nylon 66 as a function of cyclic frequency and the content of imbibed water.

Experimental Procedure

For this study, compact-tension specimens of nylon 66 (M_n = 17,000) (12) were used; details of the FCP tests and the procedures for moisture equilibration are described elsewhere (12).

Measurements of crack-tip temperatures were made using copper-constantan thermocouples (TC) and an Omega digital thermometer. A 1.6-mm-diameter hole was drilled at mid-thickness from the top of the test specimen to the crack plane and a TC inserted as shown in Figure 1. To compare center and surface temperatures, a second TC was taped to the surface of the test specimen at the same eventual crack length location as the buried thermocouple. Measurements of surface temperatures were found to be from 3 to 10°C lower than values recorded at the mid-thickness position (depending on the magnitude of the peak temperature), with the mid-thickness location measurements being used for the comparisons described below. The specimen temperature was measured and recorded as a function of the distance between the TC location and the advancing crack tip. In all cases, the maximum temperature was observed when

the crack tip reached the TC. For comparative purposes, the TC was usually placed at the same relative location corresponding to a stress intensity factor range, ΔK, of approximately 2.5 MPa$\sqrt{m}$. It should be noted that the existence of the TC hole in the test plaque resulted in a relaxation of the stress field in the region of the hole, and caused a reduction in the fatigue crack growth rate (see Figure 2). For this reason, samples prepared with thermocouple holes were not used to provide information on crack growth rates over the full range of ΔK.

Experimental Results and Discussion

Experiments were conducted to determine the extent of crack-tip heating in N66 as a function of test frequency and content of absorbed moisture. In all instances, the temperature at the thermocouple location was plotted as a function of the distance between the crack tip and TC location. Examples of such data are shown in Figure 3, which shows the strong dependence of crack-tip temperature on frequency. Note that the specimen temperature at the TC location increases to a maximum value corresponding to the point where the crack tip and TC locations coincide. Beyond this point, the temperature at the TC location begins to decrease but at a more gradual rate than that associated with the approach of the crack. Most likely, this reflects the fact that the crack tip plastic zone dimension is larger beyond the TC location so that hysteretic heating and associated temperature increases should be greater.

Maximum TC temperatures for the various experimental test conditions are summarized in Table 1. Several interesting observations can be made. First, dry N66 exhibited little heating at either 10 Hz or 50 Hz, while the crack-tip temperature in N66 specimens containing $\geq$2.6% water was at least 37°C at 10 Hz and 51°C at 50 Hz (for $\Delta K \sim$ 2.5 MPa$\sqrt{m}$). Second, for moisture levels above 0%, an increase in the test frequency resulted in higher crack-tip temperatures. For example, at 1 Hz the crack-tip temperature in saturated N66 (8.5% H_2O) was only 28°C, while at 50 Hz the temperature rose to 52°C (ΔK = 2.5 MPa$\sqrt{m}$). Third, the data for saturated N66 at 10 and 50 Hz show that the crack-tip temperature elevation was greater at high ΔK levels. Note that the size of the plastic zone at the crack-tip should increase with increasing ΔK. Since the volume of this plastic zone is the primary heat source in the test sample, a greater hysteretic energy loss should take place as ΔK increases and thus lead to an increase in the crack-tip temperature.

TABLE 1

wt. % water	Frequency 1 Hz	10 Hz	50 Hz
0	-	T_{max} = 28°C	T_{max} = 26°C
	-	ΔK = 2.5 MPa$\sqrt{m}$	ΔK = 3.1 MPa$\sqrt{m}$
2.6	-	T_{max} = 43°C	T_{max} = 51°C
	-	ΔK = 2.5 MPa$\sqrt{m}$	ΔK = 2.5 MPa$\sqrt{m}$
4.5	-	T_{max} = 39°C	T_{max} = 60°C
	-	ΔK = 2.5 MPa$\sqrt{m}$	ΔK = 2.4 MPa$\sqrt{m}$
8.5	T_{max} = 28°C	T_{max} = 37°C	T_{max} = 52°C
	ΔK = 2.2 MPa$\sqrt{m}$	ΔK = 2.5 MPa$\sqrt{m}$	ΔK = 2.5 MPa$\sqrt{m}$
"	-	T_{max} = 30°C	T_{max} = 47°C
"	-	ΔK = 1.5 MPa$\sqrt{m}$	ΔK = 1.5 MPa$\sqrt{m}$

The effect of moisture level on crack-tip temperature is a direct result of the influence of moisture on the mechanical damping characteristics of N66. As shown in Table 1, an increase in water content from zero to 2.6% resulted in a significant (15°C) increase in crack-tip temperature, while a further increase in water content resulted in crack-tip temperatures that generally differed little from the values for 2.6% water. A reasonable explanation for this observation is possible if trends in published dynamic mechanical spectra of these materials are examined. Figure 4 shows the tensile compliance, D", as a function of temperature for nylon 66 of various moisture levels. [Although these values were calculated from dynamic modulus data obtained using a resonance technique (13), the trends as a function of water content should be generally similar to those expected in the present case.] At room temperature, D" is lowest in the nylon with 0% moisture, and increases with water content over the range shown. Further, as the moisture level increases, there is also an increase in the magnitude of the maximum in D" and a decrease in the temperature at which this maximum occurs. Both the value of D" at room temperature and the location of the peak relative to room

temperature are important. For example, if the initial value of D" is low, little heating will be observed; this appears to be the case for the dry nylon. If the value of D" is higher, more heating will occur, with the amount depending on the position of the peak relative to room temperature. If the damping peak is above room temperature, D" will increase as the specimen heats; specimen heating will then involve an auto-accelerating process. On the other hand, if the peak in D" is below room temperature, D" will decrease with increasing temperature and the crack-tip heating will tend to be self-limiting. Figure 4 suggests that the change from the auto-accelerating to self-limiting crack-tip temperature condition occurs at a moisture level between 3.3 and 6.4%. Since the crack-tip strains experienced by a fatigue sample are larger than those experienced in the determination of D", one would expect the D" peaks to shift to lower temperatures. Hence the moisture content associated with the crack-tip heating transition would be expected to occur at somewhat lower water levels. This is consistent with the crack-tip temperatures observed at 10 Hz. At 50 Hz, the crack-tip temperature is a maximum at a water content of 4.5%, as shown in Figure 5. This would be expected, since the D" peaks will shift to higher temperatures at the higher frequency. Since for the stress conditions experienced by the material at the crack tip, there are no dynamic loss data available at present, the relative tendency for hysteretic heat generation of the materials concerned will be discussed using D" data from conventional small-strain experiments.

The frequency dependence of the increases in crack-tip temperature suggests that the respective FCP rates of moisture-bearing N66 should likewise be a function of test frequency. Unfortunately, published results on this point are not in full agreement. For example, Culver et al. (9-11) reported that FCP rates in N66 were frequency-sensitive over the range from 0.1 to 20 Hz. On the other hand, no clear frequency sensitivity of FCP rates was noted by Hertzberg et al. (15) within the range from 1 to 100 Hz. An attempt was then made to rationalize these conflicting findings in terms of possible differences in material and test variables. Table 2 summarizes the results of this analysis of the data previously published (8-11,15,16) and of the data generated during the present study.

TABLE 2

Investigator	Water Content	R	Frequency	Frequency Sensitivity	Specimen Type
Hertzberg et al. (15)	0	0.1	1-100	None	CT
This study	8.5	0.1,0.5	0.2,10	Variable	CT,WOL
"	2.35	0.1	1-100	Moderate	WOL
Lang et al. (8)	1.7	0.1	1-100	Strong	CT[a]
Bretz (16)	0.2-4.5	0.1	1-100	Moderate	CT
Arad et al. (9,10)	?	0.67	0.1-5	Strong	CCT
El-Hakeem and Culver (11)	0	0-0.6	0.2-20	Strong	SEN

[a]specimen thickness, 3 mm; thickness in other specimens listed > 6 mm.

It is readily apparent that these data were generated with a widely varying set of test parameters, all of which would be expected to affect the materials' FCP response. For example, large amounts of imbibed water should increase the materials' damping capacity at room temperature (as measured by D") by shifting the glass transition temperature closer to ambient. As a result, higher crack-tip temperatures would be anticipated under cyclic loading conditions. The extent of crack tip heating would depend on the temperature of the D" relative to ambient as discussed above. Proceeding further, localized crack-tip heating should increase resistance to fatigue crack growth through a plastic blunting process. Therefore, so long as the temperature increase remains localized, FCP rates would be expected to decrease with increasing test frequency. It should be noted that water-bearing N66 samples exhibit frequency-sensitive behavior even though the test temperature is far removed from the temperature associated with the β peak (7). However, the α-transition occurs in the vicinity of room temperature in such samples, so that this transition can provide the necessary damping to cause localized increases in crack-tip temperature, resulting in lower FCP rates. This is consistent with the models proposed by the present authors (2,6-8) and by Williams (17). Thus as suggested earlier (2, ch. 3), it is the magnitude of any damping peak and the temperature at which it occurs that determines frequency sensitivity in polymeric materials, not the β-transition per se.

A higher water content in N66 should also enhance creep-dominated crack extension. Therefore, FCP rates would be expected to decrease with increased test frequency since the creep-cracking component would be minimized. The effect of water content on the frequency sensitivity in N66 is summarized in Figures 6 and 7 at two different stress-intensity levels (ΔK = 2 MPa$\sqrt{m}$ and 3 MPa$\sqrt{m}$). Note that, with the exception of the dry N66, FCP rates in N66 are frequency-sensitive. This sensitivity appears to be larger at lower test frequencies and is consistent with the results reported by Culver et al. (10-12).

The effect of mean stress level (described by the ratio, R of the minimum to the maximum load) is also seen to influence the FCP frequency sensitivity. Surely the amount of creep damage should increase with increasing mean stress, thereby reflecting the greater superimposed cyclic damage by exhibiting a stronger frequency sensitivity. Note that a stronger degree of FCP frequency sensitivity is reported at higher R ratios (Table 2).

Finally, the specimen geometry is seen to influence the degree of frequency sensitivity. For example, the specimen thickness affects frequency sensitivity in N66; the thinner the specimen, the greater the tendency towards plane-stress conditions, the lower the rate of FCP, and the greater the frequency sensitivity (see Figures 6 and 7 and reference 8 for discussion). Greater frequency sensitivity is also reported in samples with the center-cracked (CCT) and single-edge notch (SEN) configurations as compared with the compact-type (CT) and wedge-open loading (WOL) specimens. This is readily understood in terms of the stress-intensity factor calibration factors, Y, for these samples. That is,

$$\Delta K_{CCT} = Y_{CCT}\, \Delta\sigma_{CCT}\sqrt{a}$$
$$\Delta K_{SEN} = Y_{SEN}\, \Delta\sigma_{SEN}\sqrt{a}$$
$$\Delta K_{CT} = Y_{CT}\, \Delta\sigma_{CT}\sqrt{a}$$
$$\Delta K_{WOL} = Y_{WOL}\, \Delta\sigma_{WOL}\sqrt{a}$$

If a given crack size were introduced into these four samples and the same stress intensity level applied, the gross stress acting on these samples would depend on the respective Y calibration factor. Hence,

$$Y_{CCT}\, \sigma_{CCT} = Y_{SEN}\, \sigma_{SEN} = Y_{CT}\, \sigma_{CT} = Y_{WOL}\, \sigma_{WOL}$$

Since $Y_{CCT} < Y_{SEN} \ll Y_{CT}$ and Y_{WOL}, it follows that σ_{CCT} and σ_{SEN} would be much larger than σ_{CT} and σ_{WOL}. Therefore, a greater creep component would be expected

to contribute to a greater frequency sensitivity. (This circumstance is considered elsewhere (18)).

Conclusions

1. The amount of crack-tip heating which occurs during FCP tests of nylon is clearly related to the initial value of the loss compliance, D", and to the location of the peak in D" relative to the test temperature. Heating will occur if D" is high at the test temperature. If D" increases with the temperature, as ambient temperature is exceeded, specimen heating will auto-accelerate, but heating will be self-limiting if D" decreases with temperature.

2. Frequency sensitivity in moisture-bearing nylon at room temperature is partly caused by the peak in the loss modulus resulting from the α-transition, which, like the β-transition, causes localized crack-tip heating and acts to blunt the crack-tip and to slow FCP.

3. Increasing the mean stress, by increasing R ratio or increasing the gross stress by changing the specimen configuration, increases the crack growth rates and the frequency-sensitivity of nylon. This suggests that creep can affect the crack growth rates in nylon.

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Figure 1: Compact tension specimen with thermocouple location.

Figure 2: FCP behavior in specimen drilled to accommodate TC.

Figure 3: Center and surface temperature vs. distance from crack tip to TC for FCP tests of nylon 66 (4.5% H_2O) at 10 Hz and at 50 Hz.

Figure 4: Loss compliance, D", of nylon 66 as a function of temperature for moisture levels of 0, 0.9, 3.3 and 6.4%. Data converted from Woodward et al. (13).

Figure 5: Crack tip temperature vs. moisture level for nylon 66 tested at $\Delta K = 2.5$ and f = 10 Hz and 50 Hz.

Figure 6: Crack growth rate vs. test frequency of nylon 66 with various moisture levels. $\Delta K = 2.0$ MPa$\sqrt{m}$ and R = 0.1.

Figure 7: Crack growth rate of nylon 66 vs. frequency at $\Delta K = 3.0$ MPa$\sqrt{m}$ and R = 0.1.

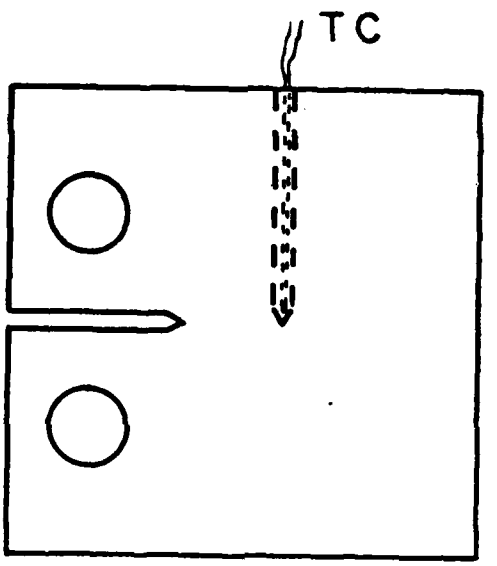
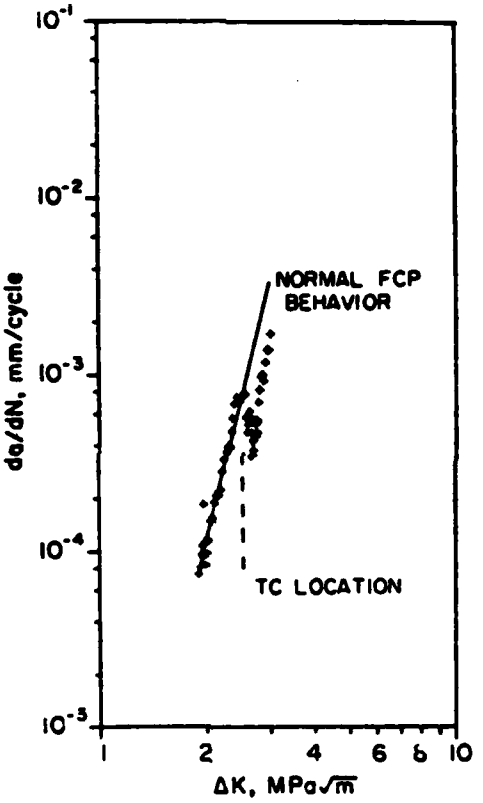


Figure 1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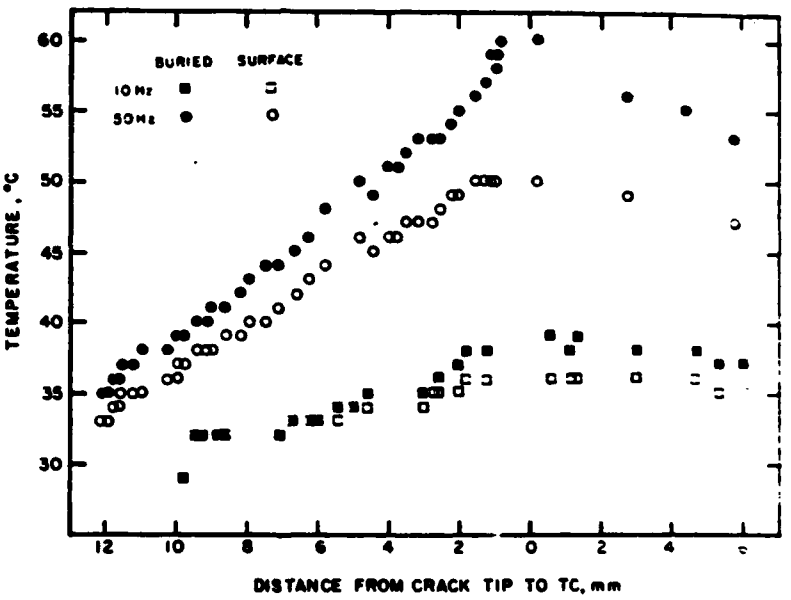
10^{-2}
10^{-3}
10^{-4}
10^{-5}
da/dN, mm/cycle
NORMAL FCP BEHAVIOR
TC LOCATION
1
2
4
6
8
10
ΔK, MPa√m

Figure 2


BURIED
SURFACE
10 Hz
50 Hz
TEMPERATURE, °C
60
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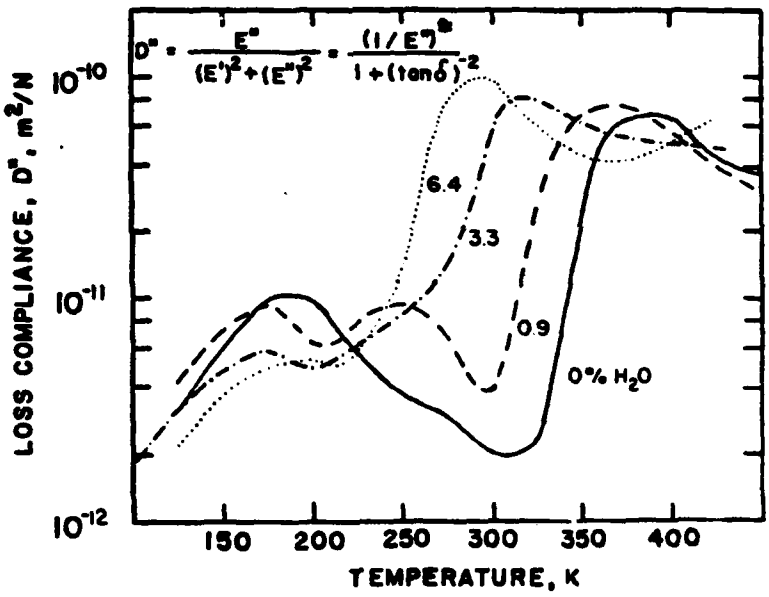
DISTANCE FROM CRACK TIP TO TC, mm

Figure 3


$D^* = \frac{E''}{(E')^2+(E'')^2} = \frac{(1/E')^*}{1+(\tan\delta)^{-2}}$
LOSS COMPLIANCE, D^*, m^2/N
10^{-10}
10^{-11}
10^{-12}
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3.3
0.9
0% H_2O
150
200
250
300
350
400
TEMPERATURE, K

Figure 4

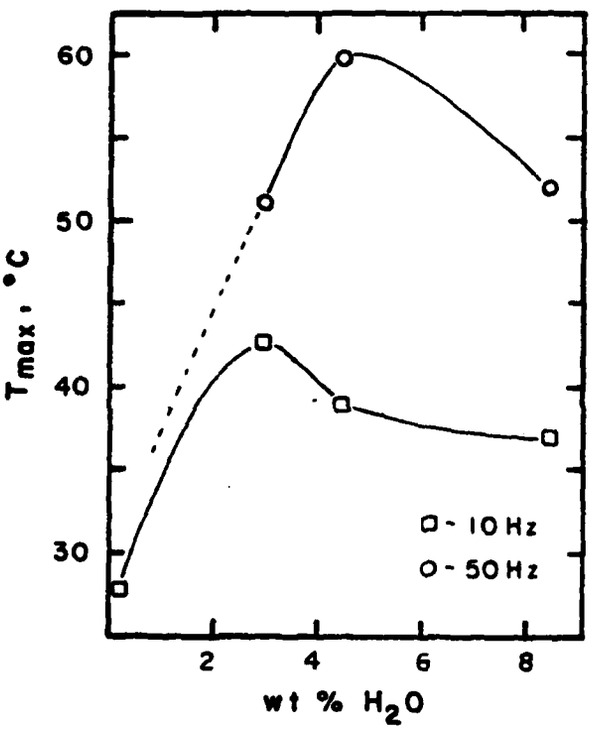
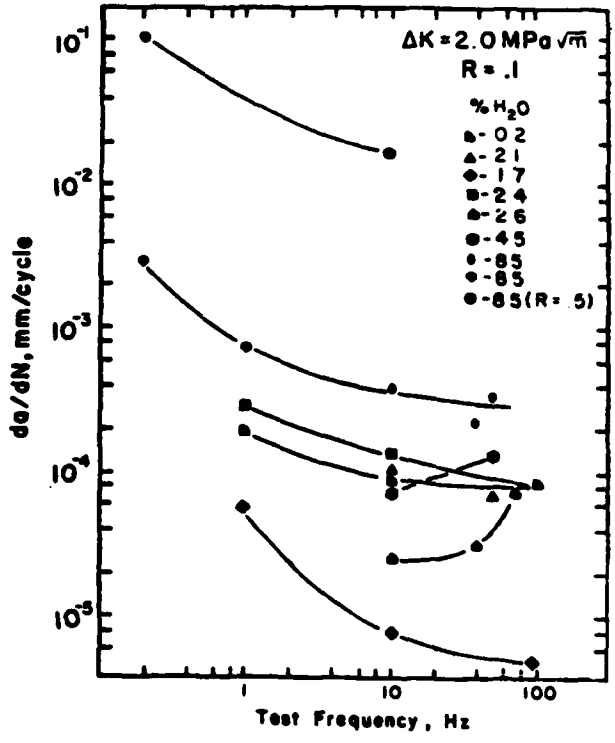


Figure 5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R = .1
% H2O
▲- 0.2
▲- 2.1
◆- 1.7
■- 2.4
▲- 2.6
●- 4.5
●- 8.5
●- 8.5
●- 8.5 (R = .5)
da/dN, mm/cycle
10^-1
10^-2
10^-3
10^-4
10^-5
1
10
100
Test Frequency, Hz

Figure 6

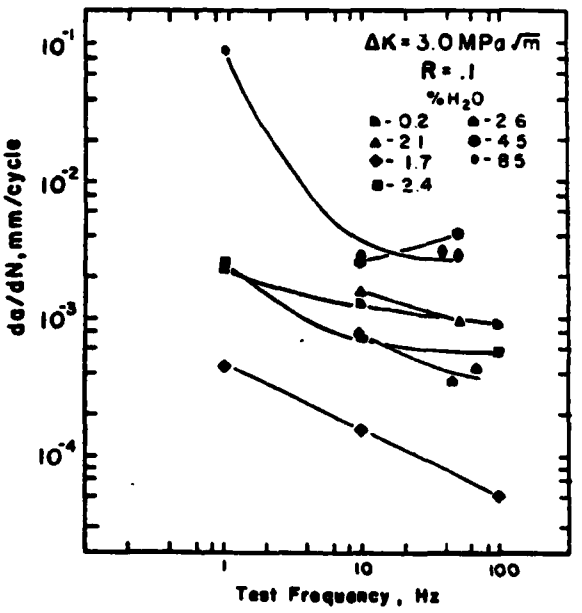
ΔK = 3.0 MPa √m
R = .1
%H2O
■ - 0.2
▲ - 2.1
◆ - 1.7
● - 2.4
▲ - 2.6
● - 4.5
• - 8.5
10^-1
10^-2
10^-3
10^-4
da/dN, mm/cycle
1
10
100
Test Frequency, Hz

Figure 7

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