

AD-A124 142

THE APPLICATION OF INFRARED MICROSCOPY IN THE STUDY OF
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M T HAHN ET AL. DEC 82 TR-16 N00014-77-C-0633

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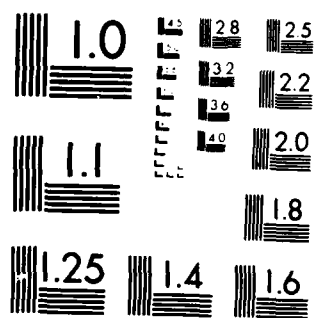
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REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER Technical Report No. 16	2. GOVT ACCESSION NO. AD-A124142	3. RECIPIENT'S CATALOG NUMBER 111
4. TITLE (and Subtitle) The Application of Infrared Microscopy to the Study of Polymer Fatigue		5. TYPE OF REPORT & PERIOD COVERED Technical Report
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) M. T. Hahn, R. W. Hertzberg, R. W. Lang, J. A. Manson, J. C. Michel, A. Ramirez, C. M. Rimnac, and S. M. Webler		8. CONTRACT OR GRANT NUMBER(s) N00014-77-C-0633
9. PERFORMING ORGANIZATION NAME AND ADDRESS Materials Research Center Lehigh University Bethlehem, PA 18015		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS NR356-670
11. CONTROLLING OFFICE NAME AND ADDRESS Office of Naval Research 800 North Quincy Street Arlington, VA 22217		12. REPORT DATE December 1982
		13. NUMBER OF PAGES
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report) Unclassified
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report) This document has been approved for public release; its distribution is unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report) DTIC SELECTE FEB 7 1983 A		
18. SUPPLEMENTARY NOTES Prepared for "Deformation, Yield and Fracture of Polymers", Plastics & Rubber Institute (London).		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Infrared microscopy Fatigue Polymers Fatigue mechanisms Polymer blends Fracture		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A model describing the frequency sensitivity of fatigue crack propagation (FCP) of polymers in terms of localized versus generalized specimen heating is examined. The magnitude of heating at the crack tip and across the unbroken ligament of the specimen is measured with an infrared microscope. The results confirm the earlier hypothesis regarding the observed frequency dependence of FCP behavior.		

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DEFORMATION, YIELD AND FRACTURE OF POLYMERS

THE APPLICATION OF INFRARED MICROSCOPY TO THE STUDY OF POLYMER FATIGUE

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A. Ramirez, C. M. Rimnac and S. M. Webler*

A model describing the frequency sensitivity of fatigue crack propagation (FCP) of polymers in terms of localized versus generalized specimen heating is examined. The magnitude of heating at the crack tip and across the unbroken ligament of the specimen is measured with an infrared microscope. The results confirm the earlier hypothesis regarding the observed frequency dependence of FCP behavior.

INTRODUCTION

The viscoelastic nature of polymeric materials leads to hysteretic heating during cyclic loading. For cyclic loading at constant stress range, $\Delta\sigma$, the rate of heat production per unit volume, $\dot{e}$, is given by (1):

$$\dot{e} = \pi f D'' \frac{(\Delta\sigma)^2}{4} \quad (1)$$

where f is the cyclic frequency and D'' the loss compliance of the material. In unnotched specimens a relatively large portion of the material experiences a high stress so bulk heating may occur (Eq. 1). This may result in a lowering of the modulus or, in extreme cases, actual melting. It should be noted that a decrease in specimen stiffness will lead to greater cyclic damage under load-controlled conditions and will lower the fatigue life. Since the rate of heating increases with increasing test frequency, it follows that fatigue life would decrease in corresponding fashion. We shall define this condition as one reflecting a negative frequency sensitivity.

In fatigue crack propagation (FCP) tests, heating is often localized near the crack tip where the stresses are highest. Barenblatt et al (2) derived an expression for such local temperature elevations for the case where D'' does not vary with temperature and the heated area is small compared to the size of the unbroken ligament of the specimen. In this case, the temperature rise, ΔT , is given by:

$$\Delta T(r, \theta) = [D'' f (\Delta K)^2 \psi(\theta)] / 2 a r \quad (2)$$

where r and θ are polar coordinates measured from the crack tip, a is a coefficient of heat exchange, and $\psi(\theta)$ is a polynomial function of θ and Poisson's ratio. We have suggested that this localized heating can retard FCP

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rates due to crack-tip blunting which lowers the effective stress intensity range, ΔK_{eff} (3). As frequency increases, beneficial local heating would be expected to increase and result in further attenuation of the crack velocity. Such frequency sensitivity is defined as being positive, in that FCP rates are lowered with increasing test frequencies. For many polymers, positive frequency sensitivity is maximized when the test temperature is close to the temperature of the beta transition where energy dissipation and crack-tip heating are maximized (3). This phenomenon has also been observed in the vicinity of the alpha transition (4). In similar fashion, Clutton and Williams (5) have argued that thermal blunting due to localized crack-tip heating in impact tests can enhance the effective fracture toughness of polymers.

If the loss compliance of even a precracked polymer sample is very high, however, the unbroken ligament of the sample can heat significantly in an FCP test. As with unnotched samples, this may decrease specimen stiffness, and result in greater damage per cycle. Since this detrimental heating increases with increasing frequency, FCP rates will also increase with increasing test frequency. Such negative frequency sensitivity with respect to FCP response has been observed in impact-modified nylon 66 (6). While it is clear that knowledge of specimen temperatures is important in the study of fatigue, previous studies of FCP have lacked such detailed temperature information. Attermo and Östberg (7) used a scanning infrared camera to observe temperatures near the crack tips in fatigue of polymers but did not report on the associated FCP rates.

The objective of this paper is to correlate specimen temperature with fatigue crack propagation rates. To this end, temperatures were recorded at the crack tip and across the unbroken ligament of the specimen using an infrared microscope. This instrument has many advantages over other methods including good spatial resolution, fast response time, precise measurement, easy data acquisition, and non-interference with specimen heating. The effects of several material and test variables on specimen temperature rise are described, as well as the effects of such temperature increases on FCP.

EXPERIMENTAL

Materials

The materials examined included: impact-modified nylon 66 (Zytel ST801), poly(vinyl chloride) modified with 6% methyl methacrylate-butadiene-styrene copolymer (PVC-6% MBS), and acrylonitrile-butadiene-styrene graft copolymer (ABS). Impact-modified dry nylon samples ($M_n=17,000$) (8) were prepared from 8.3 mm thick injection molded plaques. The PVC-6% MBS samples were prepared from material supplied with a weight-average molecular weight, M_w , of 1.69×10^5 . ABS specimens were prepared from 4.8 mm thick extruded sheet.

Loss moduli for the above materials were determined by dynamic mechanical spectroscopy using an automated Rheovibron, model DDV-IIIC. The test procedure is reported elsewhere (9).

FCP Testing

ABS, PVC-6% MBS, and impact-modified nylon were machined into compact-type (CT) specimens with a height-to-width ratio, H/W , of 0.6. For the impact-modified nylons and the PVC-6% MBS, $W=61.0$ mm; the ABS had $W=63.5$ mm. FCP tests were run at constant load range with $R=0.1$ on a closed-loop servohydraulic testing machine. A sinusoidal waveform was used and test frequencies ranged from 1 to 100 Hz. Crack lengths were measured using a traveling micro-

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scope. It was necessary to interrupt the test to make such measurements, since these interruptions allowed the specimen to cool, hold time periods were kept as brief as possible. The measured crack growth rates, da/dN , were plotted as a function of the stress intensity factor range, ΔK .

Temperature Measurements

Temperature measurements were made using an RM-2B infrared radiometric microscope manufactured by Barnes Engineering Co. and equipped with a 15X lens with a spot size of 75 μm . The lens collects infrared radiation with wavelengths between 2 and 20 μm and sends this radiation to a germanium detector. The resulting signal is amplified and the analog voltage is displayed on a dial which allows direct reading of temperatures from 15 up to 165°C. The microscope was mounted on a Gaertner XYZ positioner which allows traversing in three perpendicular directions. A linear variable differential transducer (LVDT), mounted adjacent to the positioner, measured the movement of the microscope in the direction of crack growth. The output voltages of the LVDT and the IR microscope were connected to an X-Y recorder; temperature-distance profiles were then generated semi-automatically by traversing the microscope in the direction parallel to the crack plane. Temperature profiles and maximum temperatures were periodically monitored throughout a test. All temperature measurements were made while the specimens were being cycled, and therefore represent steady-state measurement for each ΔK , test frequency, and material condition.

RESULTS AND DISCUSSION

The earlier hypotheses (3,4,6,10) about the interrelation of viscoelastic damping peaks, hysteretic heating, and crack growth rates were confirmed by correlation of FCP data with the infrared temperature measurements and the dynamic mechanical data. These data are shown in Figure 1a-i for three cases: (1) ABS, which shows negligible frequency sensitivity, (2) PVC-6% MBS, which shows positive frequency sensitivity, and (3) impact-modified nylon 66, which shows negative frequency sensitivity.

In the FCP data for ABS, Figure 1a (10), no significant variation in the growth rates is seen when the frequency varies between 1 and 100 Hz. This is consistent with the measurements of specimen temperature, Figure 1d, which show that negligible heating takes place at the frequencies from 1 to 100 Hz and values of ΔK of up to 1.0 $\text{MPa}\sqrt{\text{m}}$. Positive frequency sensitivity is illustrated in Figure 1b for PVC-6% MBS. A decrease in the crack growth rate occurs as the test frequency increases from 10 to 100 Hz. The specimen temperatures, Figure 1e, show negligible heating at 10 Hz and a ΔK of 1.4 $\text{MPa}\sqrt{\text{m}}$; for the same ΔK value a temperature rise of 9°C is observed at 100 Hz. Note that this temperature rise is localized near the crack tip. We believe that the localized heating at 100 Hz leads to greater crack blunting, thereby accounting for the lower FCP rates. The FCP data for impact-modified nylon 66 are shown in Figure 1c (6). Note the increase in FCP rates with increasing frequency. (The 30 Hz data correspond to a material containing some unknown amount of water but are believed to correctly represent overall data trends.) This negative frequency sensitivity also may be related to the infrared temperature rise in dry samples is significant at 10 Hz and even greater at 30 Hz. The temperature rises observed are certainly sufficient to lower the value of the modulus, resulting in greater cyclic damage as discussed elsewhere (6).

At this point, it is important to consider why more heating takes place in the impact-modified nylon 66 samples as compared with the PVC-6% MBS and ABS samples. Equation 2 shows that ΔT varies directly with the magnitudes of

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D'' , test frequency, and $(\Delta K)^2$. In addition, one must be mindful of the rate of change of D'' with temperature, dD''/dT ; when this parameter is positive and large, specimen heating occurs under autoaccelerating conditions. From Figure 1g,h,i we see that D'' for the modified nylon 66 is the smallest among the three materials and, yet, this material experiences the greatest amount of heating. This condition is surely traced, in part, to the higher ΔK level ($2.9 \text{ MPa}\sqrt{\text{m}}$) associated with the temperature measurements of this material as compared with the ABS ($\Delta K=1.0 \text{ MPa}\sqrt{\text{m}}$) and PVC-6% MBS ($1.4 \text{ MPa}\sqrt{\text{m}}$) polymers. (The ΔK levels chosen for each polymer reflect the relative ranking of the fatigue resistance of these materials.) Also, the D'' -gradient, dD''/dT is much greater in the nylon sample over the temperature ranges experienced by these materials. Even greater temperature elevations would be expected in the nylon sample had it been possible to conduct the fatigue test at 100 Hz (the frequency used in the ABS and PVC-6% MBS materials.) Finally, the greater amount of heating found in the PVC-6% MBS material as compared with ABS is traced to the combined influence of higher ΔK values used in the fatigue test and the magnitude of D'' in the PVC-6% MBS polymer. Thus it is shown that for the cases of positive, negative, or zero frequency sensitivity, the FCP data correlate well with infrared temperature measurements and the viscoelastic damping data.

The preceding results show specimen temperature distributions at one value of ΔK for each material. Additional studies have shown that crack-tip temperatures increase markedly with increasing levels of ΔK and that the rate of increase in temperature with respect to crack length, dT/da , is linked strongly with the K -gradient, dK/da . We have also found that the second-power dependence of ΔK (Eq.2) holds only when D'' for the material in question does not vary over the temperature range encountered. Finally, it has been shown that the degree of heating depends on the specimen configuration. For a given ΔK -level, specimen heating is greater for cases in which the Y -calibration factor of ΔK is low. In this circumstance, the overall cyclic stress level is higher, which contributes to greater hysteretic heating.

CONCLUSIONS

The infrared temperature measurements, together with viscoelastic data, serve to explain positive, negative, and negligible frequency sensitivity in a number of polymers. The observed frequency sensitivity is consistent with previous arguments of the competition between localized and generalized heating. The degree of hysteretic heating was found to depend on the prevailing values of ΔK , test frequency, and loss moduli.

ACKNOWLEDGEMENTS

We thank the Polymers Program of the National Science Foundation (Grant No. DMR 77-10063) and the Chemistry Division of the Office of Naval Research for partial support of this effort. Fellowships for two of the authors were provided by the International Business Machines Foundation (CMR and MTH). E. I. duPont de Nemours and Co., Westlake Plastics, and B. F. Goodrich provided materials for this study.

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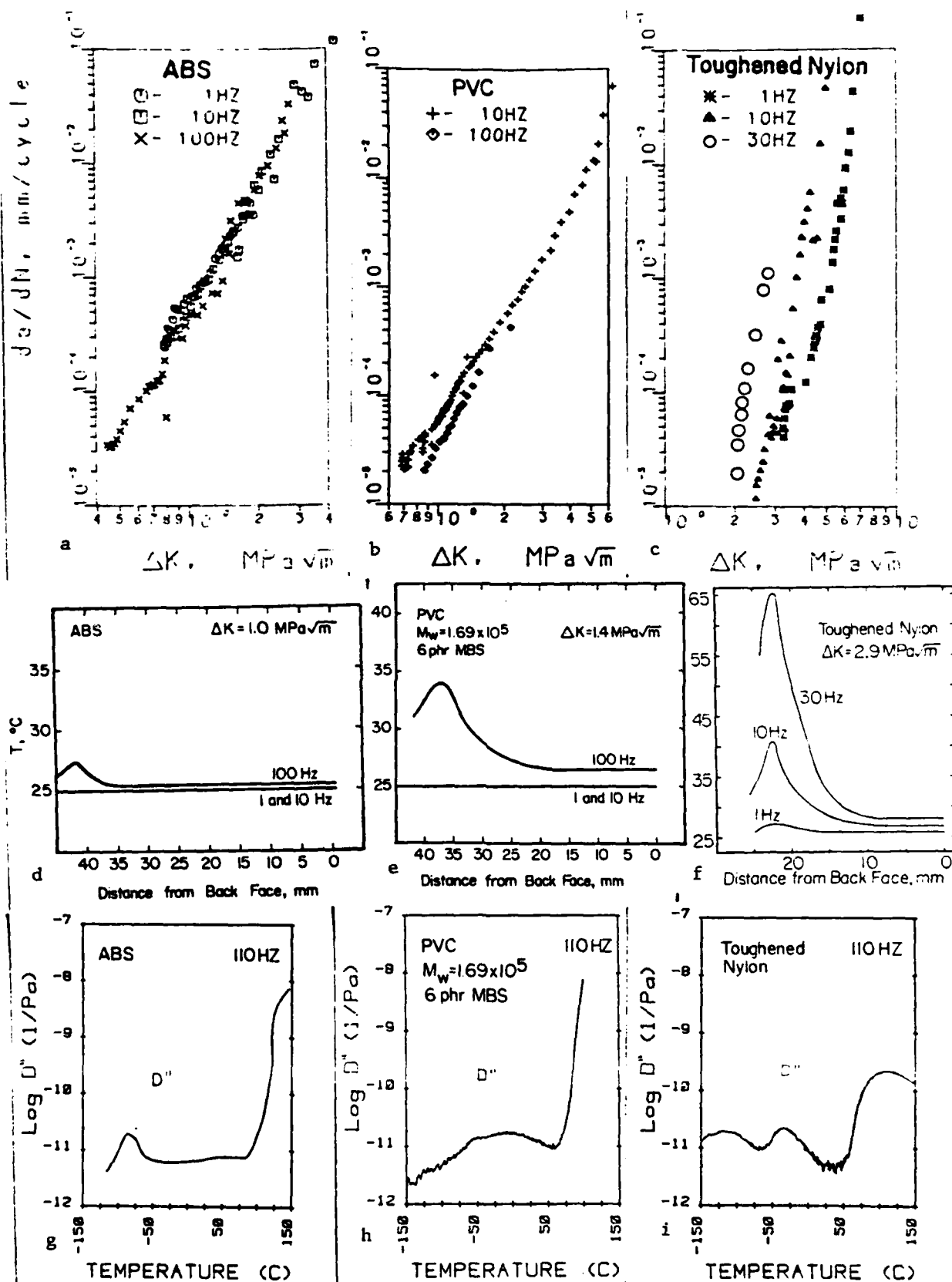


Fig. 1a,b,c--Fatigue crack growth data for ABS, rubber-modified PVC and toughened nylon, respectively; d,e,f--temperature profiles for the above; g,h,i--loss compliance vs. temperature for the above.

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