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Temperature and strain-rate dependence of fracture toughness of phenolphthalein polyether ketone‡

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A strong strain-rate and temperature dependence was observed for the fracture toughness of phenolphthalein polyether ketone (PEK-C). Two separate crack-blunting mechanisms have been proposed to account for the fracture-toughness data. The first mechanism involves thermal blunting due to adiabatic heating at the crack tip for the high temperatures studied. In the high-temperature range, thermal blunting increases the fracture toughness corresponding to an effectively higher test temperature. However, in the low-temperature range, the adiabatic temperature rise is insufficient to cause softening and J_{IC} increases with increasing temperature owing to viscoelastic losses associated with the β-relaxation there. The second mechanism involves plastic blunting due to shear yield/flow processes at the crack tip and this takes place at slow strain testing of the single-edge notched bending (SENB) samples. The temperature and strain-rate dependence of the plastic zone size may also be responsible for the temperature and strain-rate dependence of fracture toughness.

1. Introduction

The application of fracture mechanics parameters, critical strain-energy release rate, G_{IC}, and fracture toughness, J_{IC} or K_{IC}, to describe and characterize crack growth in polymers, is now firmly established [1]. Because of the viscoelastic nature of polymers, subcritical crack growth can take place at G and K levels below G_{IC} and K_{IC} and this depends on both rate and temperature. For a given temperature, T, the rate effect is usually expressed in terms of crack velocity, $\dot{a}$. Theoretical relationships between $G(K)$, $\dot{a}$ and T have been obtained by Marshall *et al.* [2], Atkins *et al.* [3] and Mai and Atkins [4], for several glassy polymers. Various experimental techniques to determine K–$\dot{a}$ relations have also been discussed [5]. In this work our interest was in the variation of K_{IC}, J_{IC} or G_{IC} with T and applied strain rate or stressing rate. Low strain rate can be achieved by conducting quasi-static experiments in single-edge notched bend (SENB) specimens in an Instron machine over a range of crosshead speeds.

Temperature has a significant effect on fracture toughness of many brittle thermoplastics, such as poly(methyl methacrylate) (PMMA) and polystyrene (PS). The fracture toughness of PMMA has been measured over a wide range of temperatures by several investigators [6–8]. In the results of their measurements, it was found that the fracture toughness of PMMA decreased with increasing temperature over a range of −50 °C to the glass transition temperature, T_g. In a previous paper, the temperature dependence of the critical stress intensity factor, K_{IC}, of PMMA was investigated. It was found that K_{IC} decreased with increasing temperature over the range −35 to 60 °C. However, K_{IC} increased near T_g, and reached a maximum at T_g; it then dropped off with increasing temperature. Johnson and Roden [9] measured the critical stress intensity factor (fracture toughness, K_{IC}) over a temperature range from −190 °C to −50 °C and reported that K_{IC}-temperature curves of PMMA exhibited a peak at low temperature (−60 °C) which agreed with the β relaxation at that temperature. On the other hand, the linear elastic fracture mechanics (LEFM) approach is now well established for thermosetting polymers. It was reported that the fracture toughness of epoxy resins did not vary in the low-temperature range, and increased with increasing temperature up to T_g [10–13]. Mizutani [14] suggested that the K_{IC} peak at T_g was related to the large increase in crack-tip plastic zone size at this temperature. In fact, the variation of K_{IC} with T can be accurately predicted from the isothermal–adiabatic transition model put forward by Williams and co-workers for glassy polymers [2, 4].

The effects of strain (loading) rate and temperature on pure and rubber-modified epoxies have been investigated extensively by Kinloch and co-workers [15, 16]. Toughness generally decreases with strain rate at low rates. Toughness also decreases very slightly with temperature initially, but increases as the glass transition temperature is approached (i.e. for $-50\,°C < T < T_g$) [15,16].

As phenolphthalein polyether ketone (PEK-C) is being increasingly used in engineering applications

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there is also a need to understand rate and temperature effects on the fracture toughness. In this paper, we report the strain-rate and temperature dependence of fracture toughness of PEK-C. Strain-rate effects were studied using the quasi-static SENB specimens. Temperature effects on PEK-C were investigated in the temperature range 100–190 °C. The results are discussed in relation to the micromechanism of the deformation process at the crack tip.

2. Experimental procedure

Fracture mechanics characterization was performed in three-point bending with a span-to-width ratio of 4, using an Instron 1121 dynamometer, at different temperatures, at a constant crosshead speed of 5 mm min^{-1}, and at room temperature at different crosshead speeds. Specimens were single-edge-notched 80 × 16 × 8 mm bars. Sharp notches were introduced by scalpel-sliding a razor blade having an on-edge tip radius of 13 μm.

For material that exhibited brittle behaviour, the critical stress intensity factor, K, at fracture initiation was determined according to ASTM Standard E399-83 for metals [17]. Tests were carried out with specimens of varying notch length, a. The values of K were obtained from the slope of the plot $\sigma_c Y^2$ versus a^{-1}, σ_c being the critical stress for fracture initiation, and Y a geometrical factor taken from [17].

For the characterization of fracture resistance in more ductile materials, the J-resistance curve, J_R, according to the multispecimen technique covered by ASTM Standard E813-81 [18] was used. Each of several specimens were loaded to a different deflection, and J was calculated from the input energy, U, measured at the final deflection according to the expression

$$J = 2U/(W - a) \tag{1}$$

in which B, W, and a are the specimen thickness, width, and initial notch length, respectively. The specimens tested were then fractured completely at a higher speed in order to change the fracture regime and, therefore, the fracture surface morphology. The initial crack length and the amount of crack extension could thus be measured from the surface of the completely broken samples with a microscope.

The J values obtained are then plotted against the Δa measured, giving the J_R curve. Because, with ductile materials, some crack-tip blunting may occur prior to the real crack propagation, to determine the onset of crack extension according to [18], the J versus Δa curve is extrapolated to intersect the blunting line, which is assumed to be expressed by

$$J = 2\Delta a \sigma_y \tag{2}$$

in which σ_y is the tensile yield stress, while the intersection gives the fracture resistance, J_{IC}, at fracture initiation, the slope $dJ/d\Delta a$ of the linear region of the J_R curve represents the resistance to crack propagation.

In all the fracture tests performed, the size requirements set by Standards E399-83 and 813-81 for the validity of both K_{IC} and J_{IC}, respectively, were always met.

3. Results

The results of fracture mechanics tests are shown in Figs 1 and 2, respectively. In the temperature range from room temperature to 70 °C, the material exhibited brittle fracture behaviour; it gave valid LEFM data. So we used K_{IC} to describe its fracture behaviour. With increasing temperature, the material became more ductile, LEFM was invalid. Another method, the J-integral, was used. In this temperature range (from 70–190 °C), J_{IC} was converted to K_{IC} by the relation

$$K_{IC}^2 = EJ_{IC}/(1 - \nu^2) = EG_{IC}/(1 - \nu^2) \tag{3}$$

where E is the elastic modulus and ν is Poisson's ratio.

From room temperature to 70 °C, K_{IC} was also converted to J_{IC} by Equation 3. At room temperature, at different strain rates, K_{IC} was obtained, and was converted to G_{IC} by Equation 3.

4. Discussion

It is common experience that the resistance to cracking in many engineering materials depends on the rate of deformation, and the test temperature in which cracking takes place. Traditionally, the effects of rate and temperature have been interpreted in terms of simple stress–strain curves, tensile strain-to-fracture, stress/time-to-failure curves, and the like. Nowadays, however, such effects are given in terms of fracture toughness parameters and they can be studied either individually or in combination.

4.1. Crack-tip blunting

The concept of crack-tip blunting was originally proposed by Kinloch and Williams [19] to explain unstable "stick–slip" fractures observed in epoxies under quasi-static conditions. Blunting occurs as a direct result of plastic shear flow at the crack tip. Evidence in support of this mechanism is the "stretched zone" formed at the crack initiation in PEK-C tested with SENB specimens at ambient temperature. A "stretched zone" can only be formed if there is plastic flow and blunting of the sharp crack tip.

4.2. Thermal blunting

Thermal blunting [20] and not plastic blunting is the major reason for the high toughness values. This assumes that heat is generated in a linear zone of crazed materials, and that the crack-tip temperature rise, ΔT, is

$$\Delta T = G_{IC}/(\pi \rho c k t)^{1/2} \tag{4}$$

where ρ is the density, c is the specific heat, k is the thermal conductivity, and t is the loading time.

4.3. Temperature effect

Fig. 1 shows the variation of fracture toughness, K_{IC} and J_{IC} of PEK-C with temperature. It was found that in the temperature range from 12–70 °C, K_{IC}

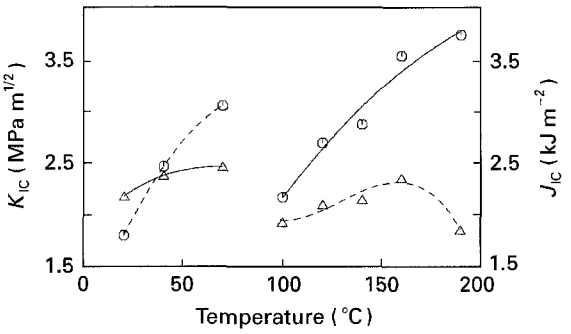

Figure 1 (○) J_{IC} and (△) K_{IC} of PEK-C as a function of temperature.

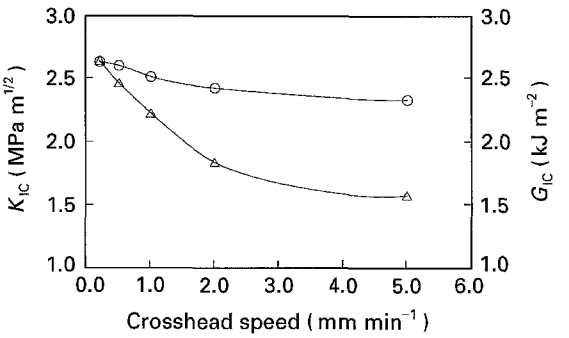

Figure 2 (○) K_{IC} and (△) G_{IC} of PEK-C as a function of crosshead speed.

increased slightly with increasing temperature and J_{IC} increased rapidly. Above 70 °C, K_{IC} increased near T_g, peaked at T_g and then dropped off with increasing temperature. J_{IC} increased with increasing temperature. In the high-temperature range, the trend is primarily due to the increasing amount of crack-tip blunting as the yield stress is decreased when the temperature increases. We have already indicated that adiabatic heating would induce crack-tip blunting by softening a zone of material prior to unstable crack propagation. The whole process is to increase the fracture toughness as would be measured from an effectively higher test temperature (which is equal to the sum of the ambient temperature and the adiabatic temperature rise, ΔT).

At low temperature, the adiabatic temperature rise is not sufficient to cause thermal blunting. Consequently, fracture toughness should decrease with decreasing temperature providing no other relaxation processes exist. However, Fig. 1 shows that the reverse is true, because there is a loss peak at about 70–100 °C for PEK-C. We suggest that the viscoelastic loss of this β-transition is solely responsible for the increasing fracture toughness observed in the temperature range from 12–70 °C. Heat is usually evolved with the β-transition which, in principle, helps in softening and blunting the crack tip. However, at these very low temperatures, the temperature rise is not high enough to cause thermal blunting to occur. Even so, the viscoelastic loss processes due to the β-transition would still contribute to the total fracture toughness.

4.4. Strain-rate effect

Strain rate has a significant effect on the fracture toughness of many polymers and it has been investigated extensively by Yamini and Young [10], Kinloch *et al.* [15, 16], Williams and co-workers [21, 22] and Low and Mai [23] at different testing conditions. The degree of strain-rate effects on the fracture toughness, K_{IC} and G_{IC}, of PEK-C may be discerned from the plot given in Fig. 2; there is a general trend for K_{IC} and G_{IC} to decrease with strain rate at a constant temperature, the effect being more significant at high temperatures than low. In the low strain-rate SENB domain, the negative rate dependence of K_{IC} and G_{IC} is entirely the result of the plastic blunting mechanism. Plastic blunting obviously decreases with increasing strain rate because of the increasing yield strength. Adiabatic heating does not play any part in the crack-tip blunting here.

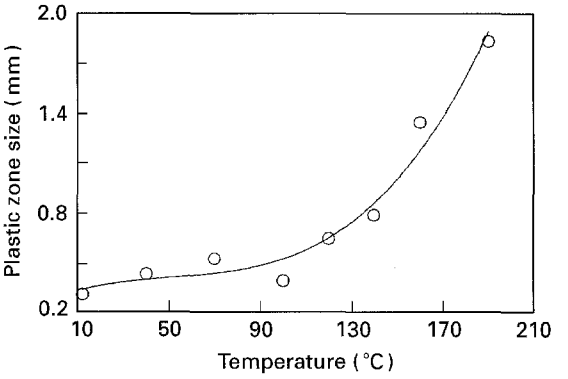

Figure 3 Plastic zone size versus temperature.

In general, the fracture toughness of polymers depends upon strain rate and temperature and there is a general equivalence between these two factors. The fracture behaviour observed at low temperatures and high strain rates can be reproduced at high temperatures with slow strain rates. This behaviour is known as the time–temperature superposition [24].

4.5. Plastic zone size

It is known that plastic deformation occurs at the crack tip during crack growth in polymers. The length of the plastic deformation zone at the crack tip, R, is related to the fracture toughness, K_{IC}, by

$$R = \pi/8(K_{IC}/\sigma_y)^2 \quad (5)$$

where σ_y is the yield strength. The R-temperature curve is shown in Fig. 3. Below 70 °C, the values of R were almost constant and above 70 °C increased vigorously. R decreases with increasing strain rate, as shown in Fig. 4. R increases with increasing temperature and so does the fracture toughness; R decreases with increasing strain rate, and fracture toughness also decreases with increasing strain rate. So the dependence of R on temperature and strain rate can also be responsible for the dependence of fracture toughness on temperature and strain rate.

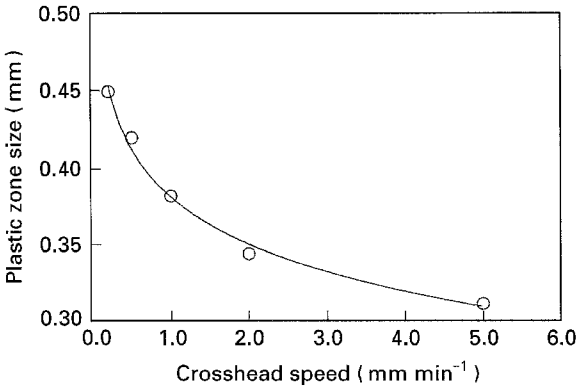

Figure 4 Plastic zone size versus crosshead speed at room temperature.

5. Conclusion

The failure mechanisms of phenolphthalein polyether ketone (PEK-C) were investigated over a wide range of temperatures and strain rates. A substantial variation in fracture toughness with rate and temperature was observed. In order to explain these rate- and temperature-dependent results, two separate crack-blunting mechanisms were proposed: thermal blunting due to crack-tip adiabatic heating and plastic blunting associated with shear yield/flow processes. Thermal blunting was found to occur at high temperatures. For low temperatures, the fracture toughness is dependent on viscoelastic loss processes and not thermal blunting. Plastic blunting was predominant at very low strain rates. The temperature and strain-rate dependence of plastic zone size may also be responsible for the temperature and strain-rate dependence of fracture toughness.

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