

J-Integral Studies of Crack Initiation and Crack Tip-Blunting of Phenolphthalein Polyether Ketone

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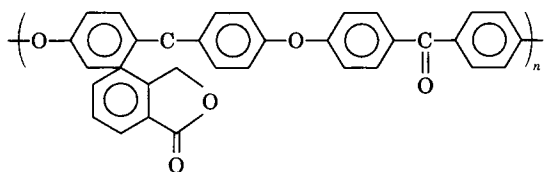
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SYNOPSIS

The J-integral is applied to characterize the fracture initiation of phenolphthalein polyether ketone (PEK-C) for which the concepts of linear elastic fracture mechanics (LEFM) are inapplicable at high temperatures for reasonably-sized specimens due to extensive plasticity. The multiple-specimen resistance curve technique recommended by the ASTM is the basic method employed. The values of J_{IC} increase with increasing temperature. The crack tip-blunting of PEK-C is observed. The parameters, such as crack opening displacement (COD), δ_0 , and the stretching increment Δa_{bl} are introduced to describe the blunting phenomenon. The δ_0 increases with increasing temperature, as does Δa_{bl} . This indicates that the blunting occurs easily as the temperature increases; i.e., as the material's yield stress, σ_y , steadily falls. The relationships between δ_0 , Δa_{bl} , and σ_y are also discussed. © 1994 John Wiley & Sons, Inc.

INTRODUCTION

Phenolphthalein polyether ketone (PEK-C)



is an amorphous material with a high T_g and can be used as an engineering thermoplastic and matrix of composites; understanding its fracture toughness as a function of temperature and crack tip-blunting is becoming more important.

The concepts of linear elastic fracture mechanics (LEFM) were applied successfully to measure the plane strain fracture toughness of brittle polymers. However, for the toughest polymers, valid measurements were restricted to low temperatures. At higher temperatures, extensive plasticity occurred at the crack tip, invalidating the use of LEFM. It was suggested that, under such conditions, other methods

which take into account the plasticity at the crack tip, such as the J-integral, may have to be used.

The J-integral was proposed by Rice¹ as a two-dimensional energy line integral that can be used as an analytical tool to characterize the crack tip stress and strain fields under both elastic and plastic stress-strain conditions. For elastic material, linear or nonlinear, it is path-independent; it characterizes the crack tip stress and strain fields; and it is a measurement of the elastic energy release rate. For elastic-plastic conditions, when analyzed with the deformation theory of plasticity, it is path-independent and it also characterizes the stress-strain field at the crack tip. However, it loses its meaning as an energy release rate, but can be considered as an energy input parameter. Because the deformation theory does not allow for any unloading, the strict application of J is limited to monotonic loading with no unloading and nonpropagating cracks. Hence, it may be used as an elastic-plastic fracture criterion for crack initiation, $J = J_{IC}$, first demonstrated by Begley and Landes^{2,3} for steel and supported by extensive work since then. For viscoelastic materials, such as polymers, the time-dependence introduces an additional complication. However, if the time-dependence is treated as an independent variable and the analysis is at a constant strain rate, mate-

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rials under elastic-plastic conditions may also be analyzed by the J-integral, but subject similarly to the restrictions mentioned above. Hence, in the present study, tests will be performed under monotonically increasing loads at a constraint strain rate to determine J at crack initiation. Some previous work has supported the use of J_{IC} as a fracture criterion for crack initiation.⁴⁻⁷

The J-integral can be determined from its energy rate interpretation¹:

$$J = \frac{1}{B} \frac{dU}{da} \quad (1)$$

where U = potential energy of the loaded body, B = thickness, and a = crack length. Equation (1) can be used to determine J experimentally. Although it represents an exact method, the experimental procedure is rather tedious. Rice et al.⁸ introduced an approximate expression for calculating J in deeply notched, bend-type specimens:

$$J = 2U/B (W-a) \quad (2)$$

where U is measured from the area under the load vs. load point deflection curve and W = specimen width.

Equation (2) is the basis for the multiple-specimen resistance (R) curve method for determining J_{IC} , first developed by Landes and Begley,⁹ and is now the ASTM recommended procedure.¹⁰ This will be the basic method adopted in the present study. It will be briefly outlined in the next section.

The Multiple-Specimen R-Curve J_{IC} Test Method

The procedure is shown schematically in Figure 1. Briefly, it consists of loading several identical specimens to different values of load point deflection and then unloading. After unloading, the specimens are broken open so that the amount of crack extension, Δa , can be measured. For PEK-C, it is enough to break open the specimens by impact at room temperature. The change of fracture mode marks the crack advance sufficiently well. The values of J at the point of unloading are determined from eq. (2) for each specimen and plotted as a function of Δa . This gives a crack growth resistance curve from which J_{IC} is determined at the intersection of the blunting line and the straight line fitted to the actual crack extension points. The blunting line accounts for the formation of a crack opening stretch zone prior to actual material separation. This is illustrated in Figure 2. The stretch zone is approximately

given by $\Delta a = \delta_0/2$, assuming a blunted crack with a semicircular profile. Hence, the blunting line is given by:

$$J = \frac{\sigma_y \delta_0}{y} = 2\Delta a \sigma_y \quad (3)$$

where σ_y = uniaxial yield stress and δ_0 = crack opening displacement.

Crack Tip-Blunting

It is well-known that ductile fracture, starting from a preexisting crack, may be preceded by the following four phases: (i) blunting of the crack followed by the (ii) initiation of crack growth, which then evolves into (iii) stable crack propagation and ends with (iv) unstable and rapid crack propagation. The qualitative and quantitative evolution of the previously mentioned phases are dependent on the properties of the materials and the geometry of the specimens, as well as on the geometry of the crack. Whatever macroscopic parameter is used to characterize these four phases, its relation to the crack growth increment Δa will be of the form shown in Figure 2. One can see that the four phases of ductile fracture are reflected in the four distinct regions of the curve. Some of these are macroscopic manifestation of microscopic changes in the material close to the tip.

The first two phases play an important role in fracture mechanics because they define some fracture parameters. In linear elastic fracture mechanics (LEFM), Irwin's stress intensity factor, K , provides the necessary fracture parameter, the critical value of which in plane-strain opening mode is the fracture toughness K_{IC} . The K_{IC} approach is, however, applicable in the small-scale yielding region only (SSY). So, outside this region of LEFM some other parameters must be introduced, as the J-integral² and the crack opening displacement (COD), δ_0 ¹¹, which attempt to interpret the plastic fracture of the materials.

Since the critical value of the COD has been proposed as a constant for materials by Well,¹¹ a lot of experimental, fractographical, and mechanical methods have been developed and improved; but later it was proved that the direct and accurate measurement of the COD is very difficult if not impossible.¹² However, in the first stages of fracture procedure, where the velocity of the propagating crack is not very high, the method of reflected caustics¹³ yields excellent results for the COD of the lateral faces of the specimen measured exactly on the moving tip of the crack. Moreover, the accurate mea-

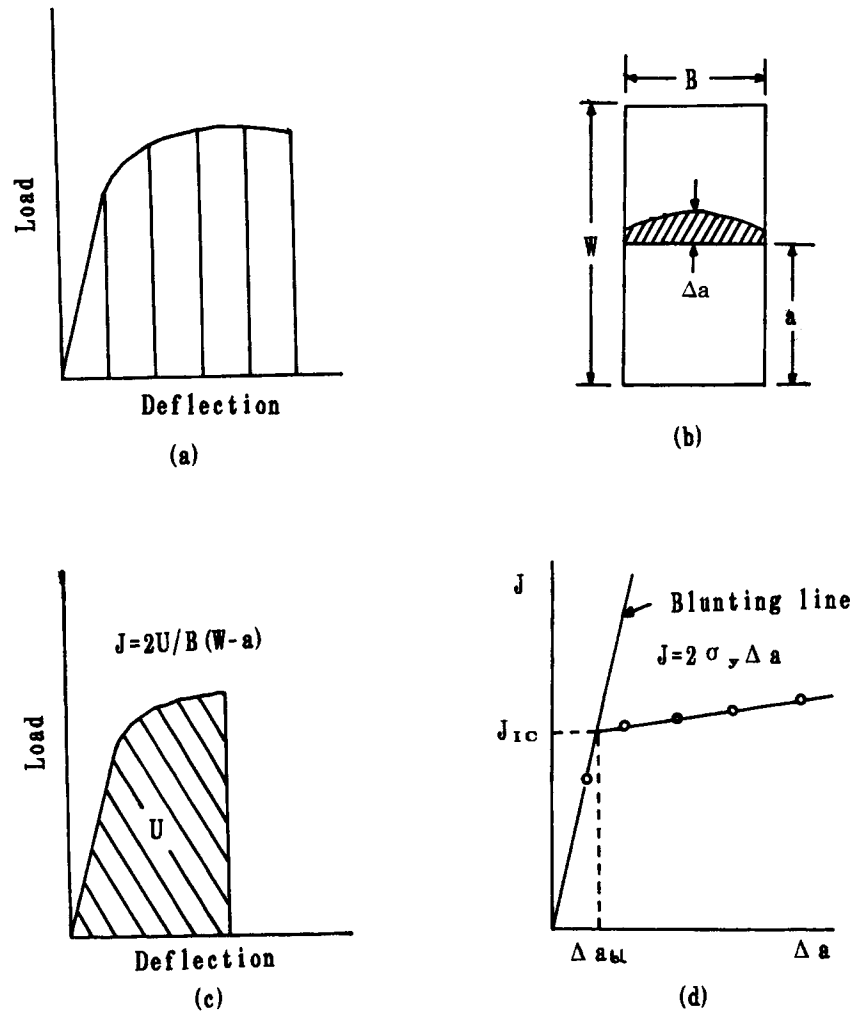


Figure 1 Procedure for J_{IC} measurement: (a) load identical specimens to various deflections; (b) measure crack extension; (c) calculate J for each specimen; (d) plot J vs. Δa and find J_{IC} .

surement and the nature of the evolution of COD is directly related to the qualitative and quantitative evolution of the blunting line. In particular, the existing theories for blunting, such as the theory of Rice-Johnson,¹⁴ Pelloux,¹⁵ and McMeeking,¹⁶ are related to the concept of a blunting line which is constructed indirectly and quite inconsistently.¹⁷

Another interesting quantity for fracture mechanics, which is also difficult to measure, is the crack increment of stretching, Δa , during the first phase of crack growth.¹⁸ While Rice and Johnson studied the case of blunting for plane-strain cracked plates, Saka, Abe, and Tanaka¹⁹ solved numerically a case of mixed-mode deformation of edge-cracked plates under plane-strain conditions. By studying the slip-line fields around cracks of different inclinations, they showed that blunting under mixed

modes of deformation yields cracks whose flanks form shapes resembling "Japanese swords."

Blunting under plane-stress conditions is much more complicated than under plane-strain conditions, since in this case the slip-line field created around the blunted crack affords only six relationships for its solution, while the number of unknowns are seven. To address this difficulty, Theocaris's method²⁰ was based on the form of the deformed cracks in plane stress and the slip-line field created around the crack tip, which was detected experimentally by using this scanning microscope and z-modulation of the microscope. In this way, and for the first time, slip-line fields of cracks under mixed-mode and plane-stress conditions were presented, and interesting differences between the cases of plane strain and plane stress were established.

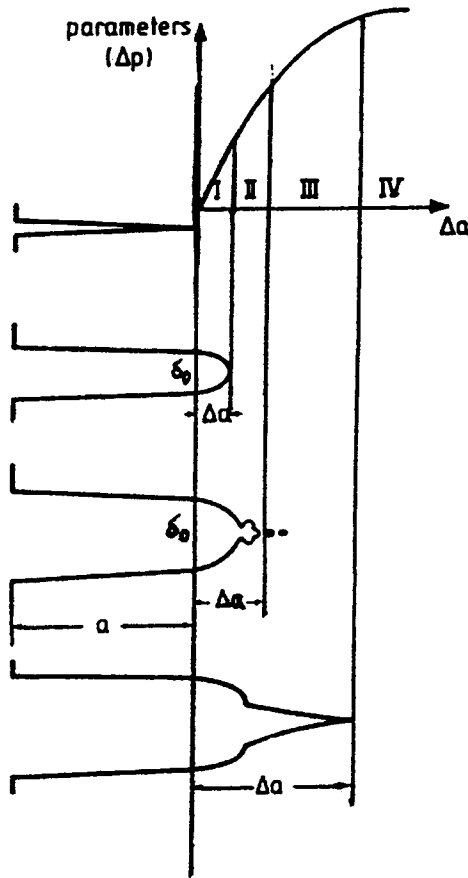


Figure 2 Load-stretching diagram indicating the four characteristic phases of crack evolution: (I) blunting; (II) initial crack growth; (III) stable crack propagation; (IV) unstable crack propagation.

Taking into consideration the above remarks, we introduce in the present article a method of calculation of COD and Δa_{bl} in the case of ductile fracture. As test material, the phenolphthalein polyether ketone (PEK-C) was chosen since it presents a significant amount of plasticity before fracture and shows blunting processes.

Theoretical Background

In the small-scale yielding model introduced by Wells¹¹ and the Dugdale strip yielding based models,²¹ there is an implicit assumption that the COD occurs at both the original crack tip and the elastic-plastic boundary. However, nothing is implied regarding radial displacements ahead of the crack tip.

By considering shear strains in relation to the Prandtl field, Rice and Johnson¹⁴ predicted for plane-strain conditions that the crack tip would be deformed by radial displacement tangentials to the

original crack tip, approximately doubling the respective displacement ahead of the position of the tip.

Pelloux¹⁵ has proposed a simple model for the formation of the stretch zone at the crack tip. Moreover, he found that shear initiation along a plane at 45° to the crack tip, until work-hardening makes further shear on another 45° plane more favorable. Thus, deformation takes place by incremental shear that alternates between the two planes at 45° to the crack direction, leading to an extension of crack equal to half the crack flank opening displacement.

Later on, McMeeking,¹⁶ by using the method of finite elements for large geometric changes, found different shapes for the blunting developed. In general, we can write:

$$\Delta a_{bl} = \lambda \delta_0 \quad (4)$$

where Δa_{bl} is the incremental crack extension and δ_0 is the COD, while the coefficient λ takes the values between 0.55 and 0.65 for the Rice-Johnson model, and the value 0.37 for the McMeeking model. By combining the above three models, we can find a mean coefficient $\bar{\lambda}$ with values of the order 0.5. The values of λ and $\bar{\lambda}$, as well as the above-mentioned models, concerned plane-strain conditions.

EXPERIMENTAL

Materials

The material used was phenolphthalein polyether ketone (PEK-C) supplied by Xu Zhou Engineering Plastic Co., China.

Uniaxial Tensile Test

Test specimens were dumbbell-shaped with dimensions of 3.14 × 3.78 × 25 mm. Uniaxial stress-strain relations were determined as a function of temperature. Several tensile properties (such as yield stress, σ_y , and Young's modulus, E) were determined.

Fracture Mechanics Test

Test specimens were three-point bend bars with dimensions of B = 8 mm; W = 16 mm; and length, L = 80 mm; and single-edge notched to an initial crack length, a, of 8 mm (a/W = 0.5). The initial crack was prepared as follows. A sawcut was made with a band saw to a depth of 6.5 mm, and the final 1.5 mm was done with a sharp single-point fly cutter.

Table I K_{IC} , J_{IC} , σ_y , E , ν , δ_0 , and Δa_{bl} as a Function of Temperature

T (°C)	100.00	120.00	140.00	160.00	190.00
J_{IC} (kJ/m ²)	2.17	2.70	2.88	3.55	3.75
K_{IC} (MPa m ^{1/2})	1.94	2.10	2.16	2.37	1.87
σ_y (MPa)	71.12	59.83	55.75	46.78	31.6
E (GPa)	1.49	1.41	1.39	1.36	0.80
ν	0.373	0.375	0.377	0.378	0.381
Δa_{bl} (μ m)	15.2	22.1	25.9	37.8	76.7
δ_0 (μ m)	35.52	52.28	60.21	88.29	138.33

Tests were performed on an Instron testing machine at a crosshead speed of 5 mm/min. A span-to-width ratio of 4 was used. Tests were performed between 100°C and 190°C.

More than four specimens ($a/W = 0.5$) were tested at each temperature. Specimens were loaded to various deflections corresponding to different amounts of crack extension and then unloaded. After unloading, the specimens were broken open by impact so that the fracture surface could be examined. The subsequent fracture by impact was brittle in nature and could be clearly distinguished from the ductile nature of crack extension. The crack front at unloading after crack extension was generally curved, showing the typical thumbnail fracture. Maximum extension generally occurred about mid-thickness. It appears that the crack first initiates at the mid-thickness where the degree of constraint and stress intensity are greatest, and then extends outwards towards the edges of the specimen. Observation of the fracture surface next to the initial notched crack showed what appeared to be a stretch zone. This was taken to be due to crack blunting prior to actual material separation.

Measurements of the initial crack length, a , and the crack extension, Δa , were made on the fracture surface using a traveling microscope.

RESULTS AND DISCUSSION

Yield Stress and Young's Modulus

The temperature-dependence of yield stress and Young's modulus was shown in Table I. Yield stress and Young's modulus decreased with increasing temperature.

J_{IC} Value

The value of J for each specimen was calculated using eq. (2). Figure 3 shows the results at various test temperatures plotted on a single graph as J vs.

Δa . Also shown on the same figure are the blunting lines based on eq. (3) and the least-square fitted lines to the crack extension points. J_{IC} is determined at the intersection of these two lines. The results of J_{IC} are listed in Table I. Also listed in the same table are the equivalent values of K_{IC} , given by the relation:

$$K_{IC} = (EJ_{IC}/(1 - \nu^2))^{1/2} \quad (5)$$

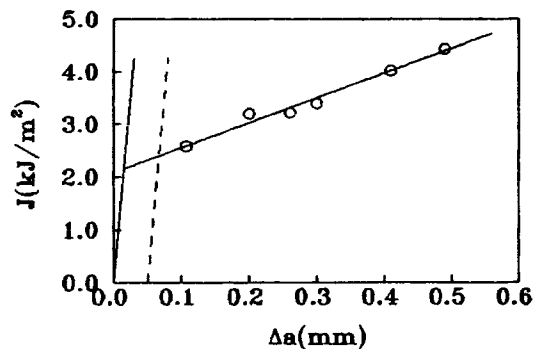
where E = modulus of elasticity, ν is the Poisson's ratio.

The values of J_{IC} and equivalent K_{IC} are also plotted as a function of temperature in Figure 4. From Figure 4 it can be seen that the value of J_{IC} decreases with temperature, although the value of K_{IC} increases first with increasing temperature and then decreases. This difference is due to the greater increase of the material stiffness with decrease in temperature. Evidently, as the temperature is lowered, the materials maintain a higher load-carrying capacity due to the greater increase in stiffness; however, its energy-absorbing capacity decreases as the fracture becomes more unstable. Figure 3 also shows that decreasing the temperature decreases the resistance to crack propagation, as is evident by the leveling of the R-curve with decreasing temperature. The fracture mode becomes more unstable with low temperature.

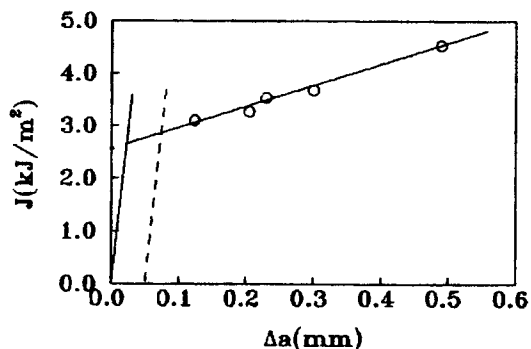
Crack Opening Displacement

The various models such as those of Irwin²² and Dugdale²³ have been proposed to describe the extent and shape of localized plastic deformation zone at a crack tip. From these models, one may define a parameter known as the crack opening displacement (COD), δ_0 , and the value of δ_0 for the onset of crack growth is given by:

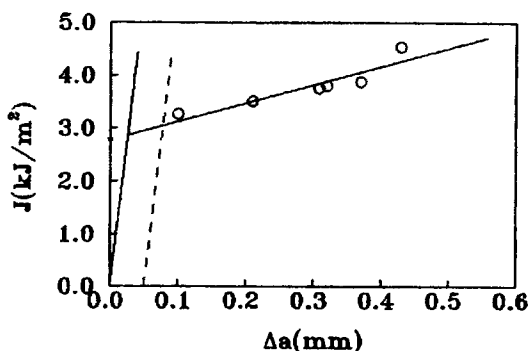
$$\delta_0 = K_{IC}^2/E\sigma_y \quad (6)$$



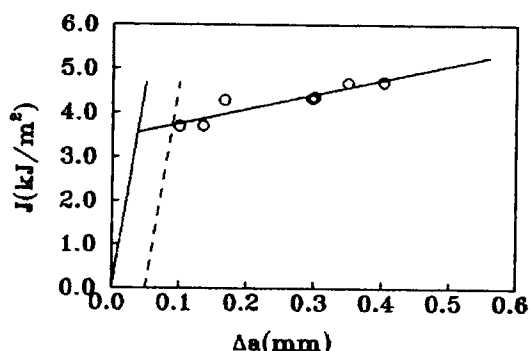
(a) Temperature 100°C.



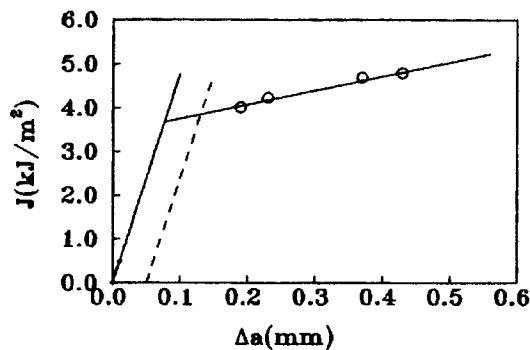
(b) Temperature 120°C.



(c) Temperature 140°C.



(d) Temperature 160°C.



(e) Temperature 190°C.

Figure 3 J vs. crack extension for each test temperature.

where K_{IC} is the stress intensity factor at the onset of crack growth, σ_y is the tensile yield stress, and E is the Young's modulus.

The value of δ_0 obviously reflects the degree of the crack tip-blunting, and the values were therefore calculated for PEK-C. The relevant value of K_{IC} was taken from the results presented above (Table I),

and the values of σ_y and E were determined from the uniaxial tensile test shown in Table I. Values of crack opening displacement, δ_0 , at the onset of crack growth are shown as a function of test temperature in Figure 5. The values of δ_0 increased with increasing temperature. This suggested that the extent of localized plastic deformation at the crack tip, and

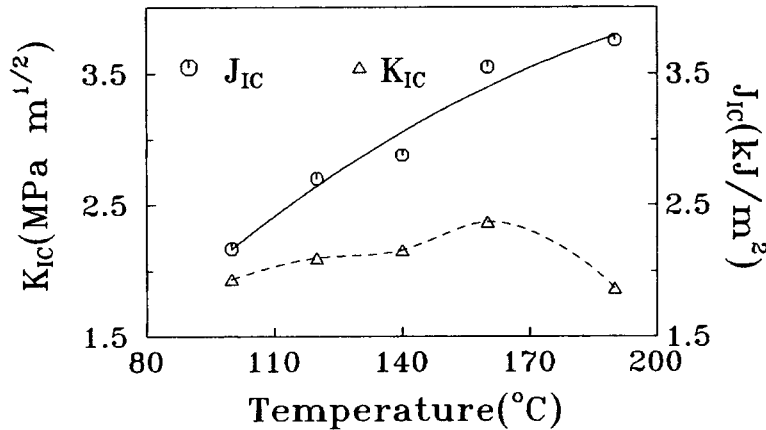


Figure 4 J_{IC} and K_{IC} vs. temperature.

the associated crack tip-blunting, increased steadily as the temperature increased, i.e., as the material's yield stress steadily fell. Figure 6 showed variation of yield stress, σ_y , vs. the crack opening displacement, COD, δ_0 , at various temperatures. Values of δ_0 increased with decreasing yield stress. This indicated that when the yield stress was small, the crack tip was easily blunted.

Stretching Increment Δa_{bl}

Another parameter that described the degree of crack tip-blunting, the stretching increment Δa_{bl} , was introduced. The values of Δa_{bl} could be determined at the intersection of the J_R-curve and the blunting line. Temperature-dependence of Δa_{bl} was shown in Figure 7. The Δa_{bl} increased with increasing temperature, indicating that the crack tip was easily blunted when temperature increased. Figure 8 showed the variation of the yield stress vs. the

stretching increment Δa_{bl} for various temperatures; values of Δa_{bl} increased as the yield stress decreased. This indicated that when Δa_{bl} is large or yield stress is small, the crack tip-blunting occurs easily.

The Relation Between Δa_{bl} and δ_0

Figure 9 presented the relation between Δa_{bl} and δ_0 . The slope of the linear straight line is 0.59. This suggests that the crack tip-blunting of PEK-C is the Rice-Johnson model. The Δa_{bl} increased with increasing δ_0 . The larger the Δa_{bl} and δ_0 , the easier the crack tip becomes blunt.

Discussion

Although the multiple-specimen R-curve technique requires from four to six specimens to measure a single value of J_{IC}, it is the simplest and most reliable method in the present study. One of the major dif-

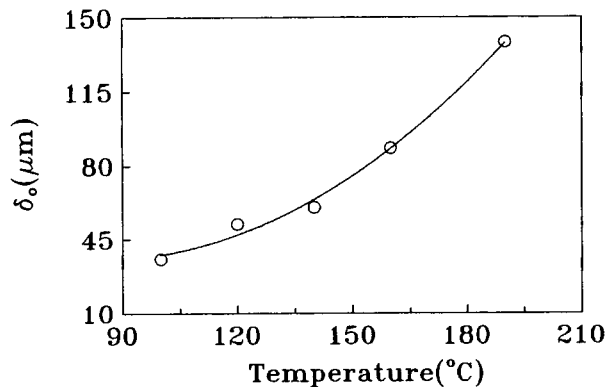


Figure 5 Temperature-dependence of crack opening displacement (δ_0).

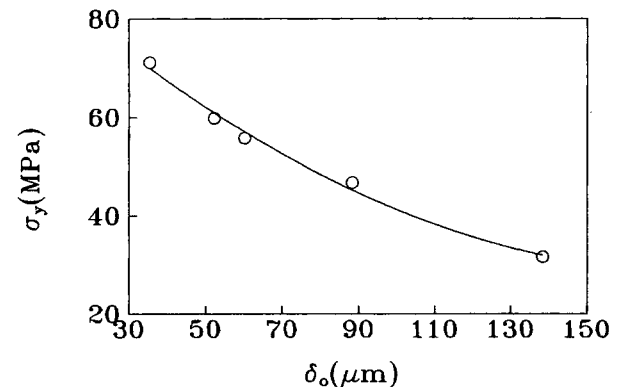


Figure 6 Variation of the yield stress, σ_y , vs. the COD, δ_0 , for various temperatures.

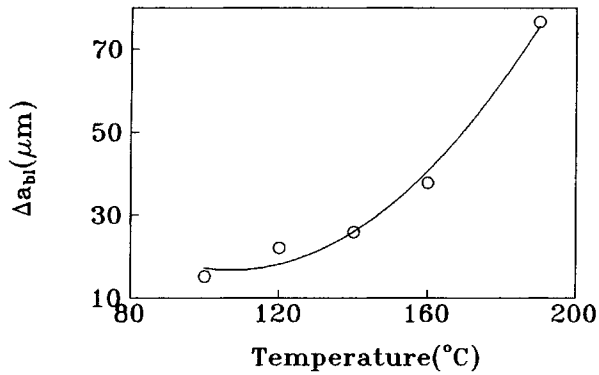


Figure 7 The Δa_{bl} vs. temperature.

difficulties is the determination of the measurement point for J_{IC} , i.e., strictly at the point of fracture initiation. Since the material is opaque and the crack initiation occurs first at the mid-thickness, it is impossible to observe initiation visually with the aid of a traveling microscope and mark the load-deflection record accordingly. The single-specimen elastic compliance technique is considered unsuitable in the present case since the fracture toughness of most polymers is time-dependent and varies with the strain rate. Repeated unloading and reloading, although small, will alter the strain rate and also the measured fracture toughness. Other reliable methods for monitoring crack growth in polymers are unavailable now, although ultrasonic technique may prove the most promising. Hence, the present method enables the extent of crack growth to be measured reliably.

The actual fracture process itself is very complex. Upon loading, the sharp crack tip begins to stretch and blunt before material separation actually takes place. The point of first crack advance, at which J_{IC} is properly defined, is difficult to ascertain even by

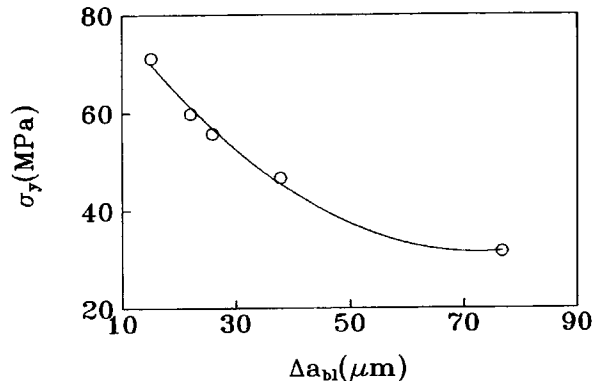


Figure 8 Variation of the yield stress, σ_y , vs. the stretching, Δa_{bl} , for various temperatures.

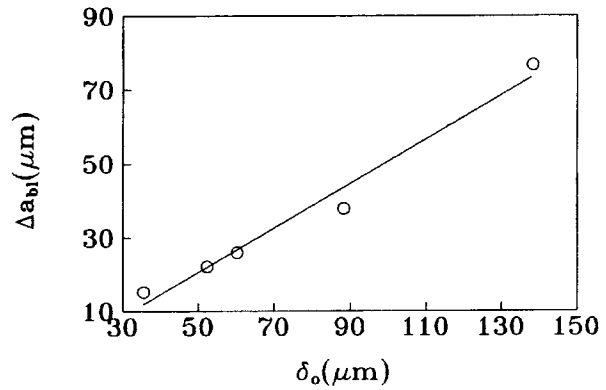


Figure 9 Variation of the COD, δ_0 , vs. the stretching, Δa_{bl} , for various temperatures.

direct observation of the fracture surface subsequent to fracture. The transition from the stretch zone to the material separation region is not clearly defined. Hence, measuring the crack extension only and extrapolating to zero crack extension to obtain J_{IC} value is a difficult task. Although the blunting line eq. (3) may not describe the blunting process exactly, it seems to be adequate to describe the stretch zone at initiation. The interpolated value of crack extension at initiation, Δa_{bl} (obtained from the intersection of the blunting line and the crack extension line) increased with increasing temperature (Fig. 7).

It should be noted that the calculated J values using eq. (2) are not true J values after crack initiation, and are used only for extrapolation purposes. The exact determination of J for an advancing crack is not resolved at the moment. In the present case, for extrapolation, it is sufficient to fit a straight line to the crack extension points. Clark et al.²⁴ have noted that the measurement point for determining J_{IC} should be limited to short crack extensions, since they deviate at large crack extension. This effect is shown by the data at some temperatures, and two straight lines are fitted in these cases.

In spite of the difficulties mentioned above, the usefulness of the present method is demonstrated when the J_{IC} values at various temperatures are converted into equivalent K_{IC} values and plotted as a function of test temperature. As shown in Figure 4, the portion of the curve derived from the J -integral studies in this study completes the plane-strain fracture toughness vs. temperature curve.

CONCLUSIONS

The J -integral method is shown to be useful to determine the plane-strain fracture toughness of phe-

nolphthalein polyether ketone in the region where conventional LEFM testing methods are unsuitable. The values of J_{IC} increase with increasing temperature. The crack tip-blunting of PEK-C is also observed. The parameters, such as crack opening displacement (COD), δ_0 , and the stretching increment Δa_{bl} are introduced to describe the blunting phenomenon. The δ_0 and Δa_{bl} increase with increasing temperature. This suggested that the crack tip-blunting increases steadily as the temperature increases, i.e., as the material's yield stress steadily falls. The δ_0 and Δa_{bl} increase as the yield stress decreases. A linear relationship between δ_0 and Δa_{bl} is observed.

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