JOURNAL OF MATERIALS SCIENCE 22 (1987) 2503–2508

J testing of toughened nylons

D. D. HUANG
Polymer Products Department, E. I. DuPont de Nemours & Company, Inc., Wilmington, Delaware 19898, USA

J. G. WILLIAMS
Department of Mechanical Engineering, Imperial College of Science and Technology, London SW7 2BX, UK

The J-integral method for measuring fracture toughnesses of tough materials has been applied to three toughened nylons. The J tests were conducted at room temperature at speeds as high as 26 mm sec^{-1}. Plane strain conditions were met in two of the three cases. Some comments about the experimental aspects of applying the J method to polymers are given.

1. Introduction

Several investigators have demonstrated that polymers can be characterized by linear elastic fracture mechanics (LEFM) [1–4]. In general, the scheme designed for metals (ASTM E399) is adequate to measure fracture toughnesses of polymers if the conditions for specimen geometry are met. These conditions were empirically formulated to ensure that the test specimens are in plane strain where fracture toughness is at a minimum. To obtain plane strain conditions, ASTM requires that the specimen thickness be greater than 2.5 $(K_{cl}/\sigma_y)^2$. This effectively guarantees that the thickness will be at least an order of magnitude greater in size than the plastic zone where small scale yielding occurs. The depth of the test specimen is required to be twice the thickness to prevent plastic collapse of the ligament.

LEFM testing of toughened polymers is especially difficult because their yield stresses are low. Thus, very thick specimens (as large as 25 mm) are required to obtain plane strain conditions. Changes in test conditions, such as high loading rates, low temperatures, or both, can limit the ductility of the specimen. These approaches will reduce the minimum required thickness, but this reduction may still be insufficient.

One approach that has been recommended for tough metals is the J-integral approach, originally proposed by Rice [5]. Its application is not limited to small scale yielding – it can be applied to large-scale plasticity cases [6]. In the general elastic–plastic case, J can be considered as the potential energy difference between two loaded identical bodies having slightly different sized cracks. It can also be considered a measure of the characteristic crack tip elastic–plastic field. When plastic deformation does not occur, J takes on additional interpretations. For nonlinear elastic bodies, J represents the energy available for crack extension. For linear elastic bodies, J is equal to G, the strain energy release rate (or the crack driving force).

Begley and Landes [7] demonstrated that the J-integral approach can provide a plane strain ductile fracture toughness, J_{cl}. This is a material parameter and represents the energy required to initiate a crack. It does not describe the propagation process. They demonstrated that J_{cl} values obtained in two different steels agreed very well with G_{cl} values obtained on specimens with much greater thicknesses.

To obtain valid fracture toughness values, J specimens are not required to be in elastic plane strain, but still must be in plastic plane strain. Some constraint of the crack tip region is required to achieve this, but not to the degree in LEFM specimens. Consequently, the minimum thickness requirement is significantly smaller for J testing (as much as 0.2 to 0.3 of the size of the LEFM specimens). The J-integral method has been applied to polyethylene [8], polypropylene [9], and a variety of toughened blends [9]. These studies were conducted at slow rates (0.033 mm sec^{-1}) and low temperatures (down to $-80°$ C) to decrease the required minimum thickness. In the present study, we have achieved the same goal by testing at high rates (up to 26 mm sec^{-1}).

2. The multiple specimen J-integral method

The J-integral method that is under investigation is a multiple specimen technique, similar to ASTM E813. It was originally proposed by Landes and Begley [10]. The first specimen is completely fractured to determine the ultimate displacement. Subsequent specimens are loaded to different subcritical displacements to obtain different levels of crack growth. From the area under the loading curve of each test, a value of J is calculated. Crack growth is marked and measured on the fracture surface. Resistance (J–Δa) curves are then constructed. J_{cl} is found graphically by finding the intersection of the blunting line ($J = 2\sigma_y \Delta a$) and the resistance curve.

In the current investigation, ASTM E813 recommendations have not been strictly followed. The modifications are noted below.

1. Crack growth (Δa) was measured at the centre of the fracture surface. Since the crack fronts were

0022–2461/87 $03.00 + .12

thumbnail shaped, this corresponded to the maximum crack growth. This is the original proposal by Landes and Begley [10]. E813 recommends making nine equally spaced measurements and averaging them in a prescribed manner. This recommendation was made because the average value would more accurately map the crack front when significant bowing had occurred. It had also been shown that an average value correlated well to other crack extension measurement techniques such as unloading compliance and electrical potential. The single-point technique that was used here is more convenient and leads to a more conservative J_{cl} value (about $5\,kJ\,m^{-2}$ less than the averaged crack growth method for the materials tested here).

2. The span to depth (S/W) ratio was 3.5. E813 recommends a ratio of 4. The span to depth ratio is important in the calculation of J. J can be expressed as [11]

$$J = J_e + J_p$$
$$= (\eta_e U_e + \eta_p U_p)/Bb$$

where J_e and J_p are the elastic and plastic contributions to J, η_e and η_p are the elastic and plastic work factors, U_e and U_p are the elastic and plastic components of the total energy, U_T, B is the thickness, and b is the ligament. For bend specimens, the η_p coefficient is independent of S/W ratio. However, η_e has an S/W dependence. When the S/W ratio is 4 and the specimen is deeply notched (greater than 0.4), η_e and η_p are both equal to 2. Therefore, for J calculations, the total energy does not have to be partitioned into its elastic and plastic portions. For this geometry,

$$J = 2U_T/Bb$$

When the S/W ratio is 3.5, η_e equals 2.2 and η_p equals 2. This leads to a maximum error of 10% when the load–deflection curve is completely elastic. The error decreases as the ratio of elastic to plastic energy decreases.

3. The resistance curve was fitted using data points where crack growth was between two offset lines drawn parallel to the blunting line. The minimum offset was 0.6% of the ligament and the maximum offset was 6% of the ligament. E813 recommends using parallel lines that are offset by 0.15 mm and 1.5 mm. Shih [12] has shown that the value of J is accurately predicted by these estimation procedures if the crack extension is less than 6% of the remaining ligament. This is a source of confusion in the ASTM standard. E813 recommends that the test specimens need to be a minimum size ($B, b > 25\,[J_{cl}/\sigma_y]$). It also recommends fixed offsets for the maximum and minimum crack growths. Since the ligaments can be of any size, the fixed offsets will not always guarantee that the crack growths will be less than 6% of the ligament. However, the 6% criterion should not be considered definitive. Recent work [13] on polymers showed that linearity in the R-curve can exist at crack growths much greater than 6%.

4. The loading rates varied from 0.26 to 26 mm sec^{-1}. This led to loading times as small as 0.05 sec at the fastest speed. E813 recommends that the loading time be greater than 6 sec. ASTM made this recommendation to avoid the confusion of measuring a dynamic fracture toughness. While this is a valid concern, the data that will be presented show no definitive evidence of dynamic effects in this range of loading times.

5. The specimens were notched with cutters that were lapped to a radius between 5 and 12 μm. E813 recommends a fatigue crack. In the current study, J_{cl} is independent of notch root radius in the 5 to 12 μm range. Earlier toughness studies of polymers have shown that notching with a sharp cutter can be acceptable [9, 13].

6. The yield stress was the ultimate stress measured in tension at the appropriate rate. This has been shown by Ward (e.g. [14]) to be the intrinsic yield point for polymers. E813 recommends using the 0.2% offset yield stress or an average between the ultimate stress and the 0.2% offset yield stress.

3. Experimental details

Toughness testing was conducted on rubber-toughened crystalline nylon 6/6, rubber-toughened amorphous nylon, and a medium-toughened crystalline nylon 6/6. These resins are commercially available and are listed in Table I. The resins were injection moulded into 100 mm × 254 mm × 12 mm plaques. The specimens were cut from the plaques, notched to one-half of the depth, and tested in three-point bend. The specimens were essentially tested dry as moulded. The dimensions and notch directions are shown in Fig. 1.

The crack growth was marked by freezing the test specimens in liquid nitrogen and then breaking them at 260 mm sec^{-1}. The crack front is bowed and the Δa value is measured at the centre of the specimen. An example of the crack growth region in Material B is shown in Fig. 2a. The region next to the initial notch (between lines A and B) is the crack growth region for this specimen. In J testing of polyethylene, it was shown that a stretch zone next to the initial notch formed first, followed by crack growth [8]. This was not observed in the materials in this investigation. This region AB continuously grew with increasing J levels. If it were the stretch zone due to blunting, it would increase with increasing J and then remain constant at J values greater than J_c. The reason for the second texture (BC) before the fast fracture region is unclear. Its texture resembled that of the stretch zone found in the specimens which were loaded to sub-J_c levels. In these specimens, only blunting occurs and only one texture was seen. The second texture was not as prominent in Materials A or C. An example of a fracture surface of Material A is shown in Fig. 2b.

J tests were conducted on the toughened polymers at three different rates, 0.26, 2.6, and 26 mm sec^{-1}.

TABLE I

Material	Description	Trade name
A	Rubber-toughened semicrystalline nylon 6/6	"Zytel" ST801
B	Rubber-toughened amorphous nylon 6/6	"Zytel" ST901
C	Toughened semicrystalline nylon 6/6	"Zytel" 408

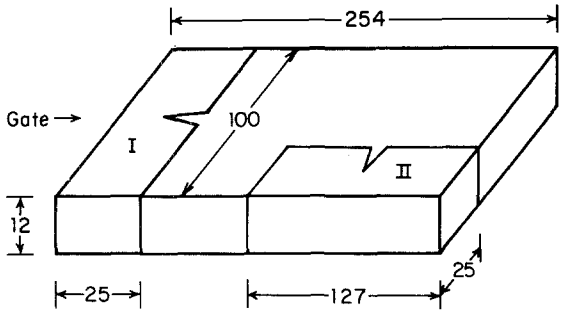

Figure 1 Specimens. All dimensions are in millimetres.

Because their mechanical properties (such as yield stress and modulus) might be rate dependent, additional considerations were necessary. The present study treats rate dependence as an independent variable. Consequently, for a given set of J data, the individual tests, yield stress, and modulus were tested at the same displacement rate. The resulting strain rates for these tests were within a factor of two of one another.

Yield stresses were measured in tension using injection-moulded bars (ASTM D638, Type I). The yield stress as a function of displacement rate is plotted for the three materials in Fig. 3. Elastic moduli were measured in flexure using injection-moulded flex bars (3 mm thick). The moduli of the materials did not vary significantly as a function of rate (all were within 10% of the others). Since the moduli were essentially constant, an average value was calculated for each material. The results are listed in Table II. Poisson's ratios were assumed to be rate independent and equal to 0.41. All tests were conducted in a 23° C and 50% r.h. environment.

A computer-controlled servohydraulic system was used for all mechanical testing. Software was developed to run the machine, acquire data in the form of load–displacement curves, and numerically integrate the curves to calculate J values. Crack growth was measured from the fracture surfaces using a travelling microscope.

4. Results

A sampling of the results is shown in Figs 4 and 5 for Materials A and B, respectively. Each dotted line represents an R-curve that was fitted to the data from a set of specimens from one plaque. The solid line is the blunting line calculated from the appropriate yield stress. The dash-dot lines delineate the crack growth window within which the data must fall to qualify for linear regression.

TABLE II Summary of results

Material	Test rate (mm sec^{-1})	Yield stress (MPa)	J_c (kJ m^{-2})	K_c (MPa m$^{1/2}$)
A	26	50.1	27.5–32.5	8.05–8.75
	2.6	47.5	33.0–41.0	8.80–9.80
	0.26	45.1	39.3–44.5	9.60–10.20
B	26	68.7	13.0–19.0	5.60–6.77
	2.6	62.9	19.0	6.77
	0.26	58.6	18.0	5.69
C	0.26	61.9	18.5	7.41

E_A = 1.96 GPa, E_B = 2.01 GPa, E_c = 2.47 GPa.
Poisson's ratio = 0.41.
All specimens were 12 mm thick.

Overall, the data behave very well. The general shape of the R-curves is similar to those obtained for other materials [7–9]. At low J values, the R-curve follows the blunting line defined by $J = 2\sigma_y \Delta a$. At higher values, the data fall off the blunting line. This part of the curve is essentially linear within the defined window. Although the J_c values vary from plaque to plaque, the individual sets of data point do not show a great deal of scatter. The J_c values for all the materials (using specimen I) are listed in Table II.

4.1. Comparison to earlier results

Preliminary results have been published earlier [15]. The results were similar to those here except for the J_{cl} values of Material B. It was found that, on further study, the J_{cl} values were in the range of 13 to 19 kJ m^{-2} rather than 27 kJ m^{-2} as reported earlier. The results of the early tests were self-consistent – the shapes of the R-curves were similar, there was no more scatter in data (Fig. 5), and the J_{cl} values were constant regardless of the test speed (provided the specimens were thick enough to be in plane strain). As there was no obvious operator error, the source of the scatter in J_{cl} values could be due to differences in materials, processing, or the J technique itself.

Various characterization techniques did not reveal substantial differences between the two sets of Material B specimens. Their thermal characteristics were essentially the same. There was no evidence of crystallinity in either sample (determined by X-ray diffraction and DSC). The toughener contents and morphologies were similar.

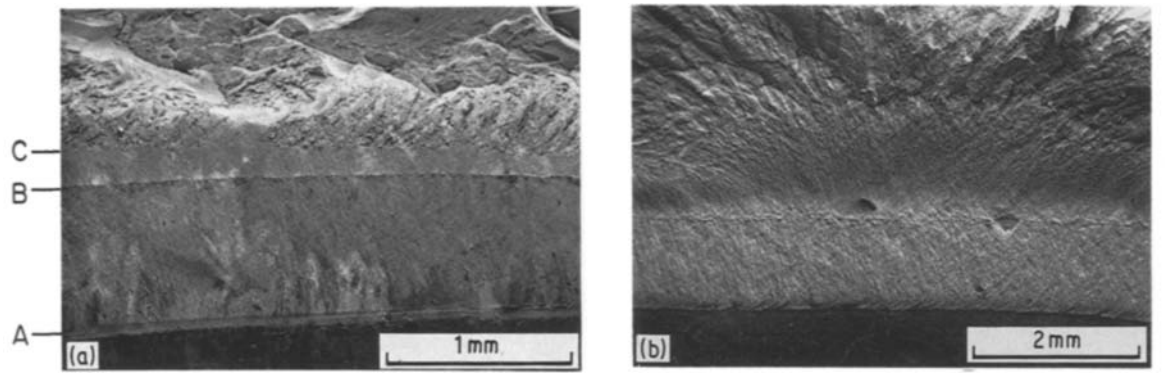

Figure 2 (a) Material B, J_{cl} = 15 kJ m^{-2}. (b) Material A, J_{cl} = 30 kJ m^{-2}.

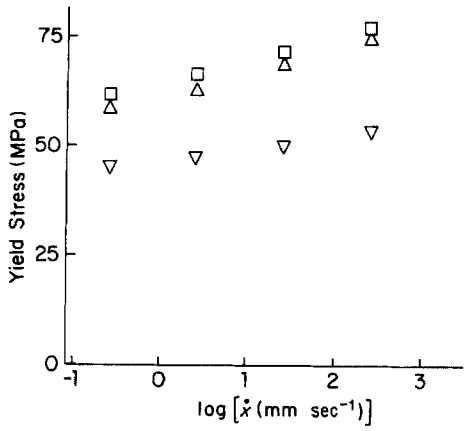

Figure 3 Yield stress plotted against displacement rate for (□) toughened nylon 6/6, (△) rubber-toughened amorphous nylon, (▽) rubber-toughened nylon 6/6.

Because the materials in the specimens seemed to be indistinguishable, additional J tests were conducted on specimens with varying surface treatments to determine whether J_{cl} values could be affected by processing conditions. Tough surface layers can be the result of residual stress or moisture pick-up in the amorphous nylon matrix. Tests were conducted with specimens that were annealed overnight at 120° C and then either quenched in an ice–water bath or slowly cooled in an oven. The J_{cl} values for both sets were between 15 and 18 kJ m^{-2}. Additional sets of specimens were also annealed, then soaked in water for up to 37 days. The surface moisture content rose from 0.6% to 1.3%, but the J_{cl} values were still between 14 and 19 kJ m^{-2}.

The multiple specimen test method was also scrutinized. The effect of varying notch root radius was studied first. In the early work, the radius was 12 μm and in the later work, it was 5 μm. The early J tests with blunter notches were repeated. J_c was 30 kJ m^{-2} for Material A and between 17 and 19 kJ m^{-2} for Material B. Thus, the J_c values of both Materials A and B are essentially independent of notch root radius in this narrow range. Some preliminary tests using larger radii (up to 1 mm) were also attempted. The tests were difficult to perform because the crack growth could not be measured. After the specimens were frozen, they did not necessarily break at the end of the crack-growth region. Instead, fracture occurred at unpredictable points around the blunt crack tip. As the plane of the crack growth could not be seen, the amount of crack growth could not be measured. This occurred at radii down to 0.2 mm.

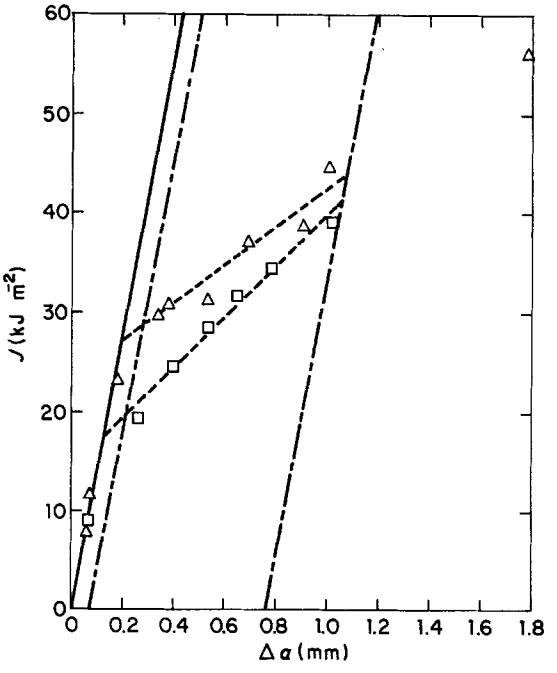

Figure 5 Material B, 26 mm sec^{-1}, thickness = 12 mm. (□) J_{cl} = 17.25 kJ m^{-2}, (△) J_{cl} = 27 kJ m^{-2}.

Another test consideration is the spacing of the data in the crack growth window. Fig. 6 shows an example of two tests with Material A tested at 2.6 mm sec^{-1}. Each set of data covers a different part of the window.

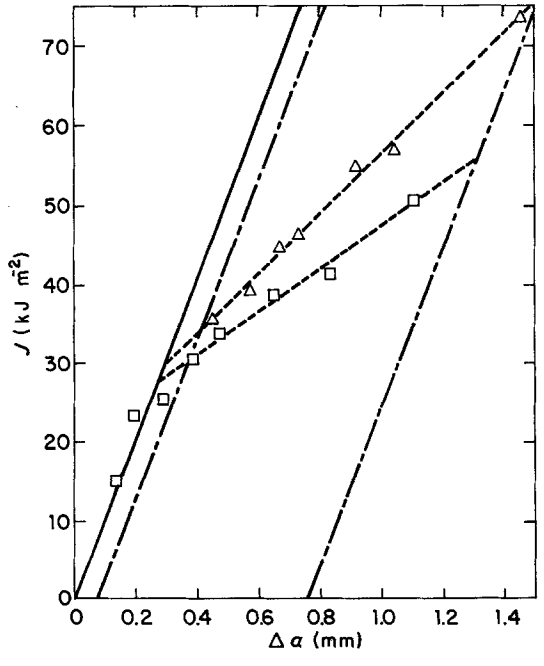

Figure 4 Material A, 26 mm sec^{-1}, thickness = 12 mm. (□) J_c = 27.5 kJ m^{-2}, (△) = 30 kJ m^{-2}.

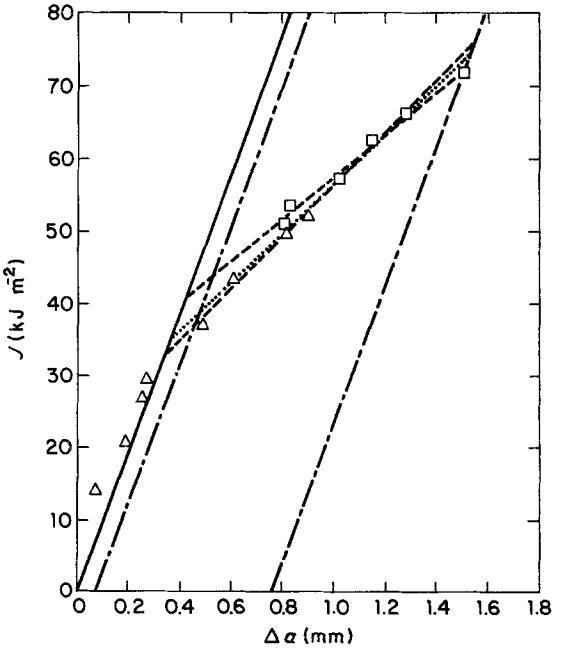

Figure 6 Material A, 2.6 mm sec^{-1}, thickness = 12 mm. (△) J_c = 33 kJ m^{-2}, (□) J_c = 41 kJ m^{-2}, (· · ·) J_c = 35.5 kJ m^{-2}.

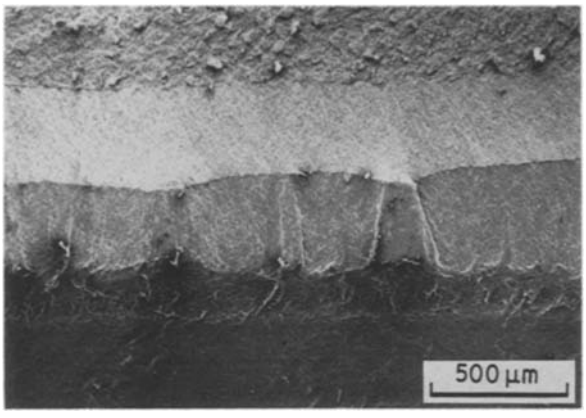

Figure 7 Material B, $J_{cl} = 27\,kJ\,m^{-2}$.

Taken separately, the J_c values vary from 33 to $41\,kJ\,m^{-2}$. Taken together, the data are still acceptable in terms of scatter with J_c equal to $35.5\,kJ\,m^{-2}$. The spacing of the data is more important when the R-curve has a steep slope because minor data scatter can shift the blunting line intercept significantly. This does not appear to be the problem with the data in Fig. 5 for Material B, as the data for both tests are spread throughout the window.

One of the differences in the present technique and the ASTM standard is the use of one measurement of crack growth at the centre of the specimen instead of an average value obtained from nine measurements spread over the thickness. The average measurement presumably accounts for the degree of bowing in the thumbnail shape of the crack front. It would also give a lower Δa value than the single measurement. This yields a higher J_c value. However, this rise (approximately $5\,kJ\,m^{-2}$ for Material B) occurred in all tests and, again, could not account for the range of J_{cl} in the two sets of experiments.

The crack growth regions on the fracture surfaces of the two specimens are different. Fig. 7 shows an example of a crack-growth region of a Material B specimen from an early test ($J_{cl} = 27\,kJ\,m^{-2}$). Compared to the crack-growth region shown in Fig. 2a taken from a later test ($J_{cl} = 15\,kJ\,m^{-2}$), the crack growth in Fig. 7 is rougher, both in surface texture and crack front, as if the crack grew by tearing. This is indicative of all the specimens in the high J_{cl} tests at any of the tested speeds. The rough texture was not a function of notch root radius because the crack growth regions in the specimens in the second set of J tests using $12\,\mu m$ radius cutters were similar to that seen in Fig. 2a.

Because the scatter in Material B J_{cl} values could not be explained by material differences or data analysis, any other reason would be speculation. The surface texture roughness may be indicative of a "poor quality" notch which induces a crack growth mode that requires more energy. It is conceivable that the notching flycutters could have been damaged during cutting, causing all of the early sets of specimens to be poorly notched. As the techniques for sharpening, notching, and inspection became better, the later tests were more controlled, giving more valid J_{cl} values. The present procedure has given very consistent results when appropriate care has been taken to get the best possible notch.

5. Discussion

According to the ASTM size criterion, a plane strain value of fracture toughness may be obtained if the specimen thickness, B, meets the requirement $B > 25\,(J_{cl}/\sigma_y)$. This requirement is an empirical one. It was formulated on the basis of experimental evidence in J testing of metals. For the cases that have been studied, only Material A does not meet this requirement. Because of the decrease in yield stress with decreasing test rate, slower rates place the specimen further away from plane strain conditions.

The J_c data for Material A are self-consistent since the J_c values at lower speeds are progressively higher. These specimens are too thin (according to the ASTM recommendation) at any of the tested speeds. However, the value of $30\,kJ\,m^{-2}$ may be a plane strain value since the specimen thickness is only 3 mm too thin. In contrast, the 12 mm thickness appears to be adequate for plane strain conditions in Material B. This thickness is greater than the ASTM recommendation at all speeds. The J_{cl} values are roughly constant, approximately $16\,kJ\,m^{-2}$. Material C was only tested at one speed, $0.26\,mm\,sec^{-1}$, because the crack growth was not stable enough at higher speeds. The value of $18.5\,kJ\,m^{-2}$ should be a plane strain value. More data covering a range of thicknesses and specimen geometries are needed to confirm these values.

To test isotropy of the injection-moulded plaques, one set of specimens was prepared in the specimen II configuration, i.e. the specimens were notched normal to the flow direction instead of parallel to it. The results indicate that there is no difference in toughness in these directions. The J_c values were 31.5, 15.5, and $15.8\,kJ\,m^{-2}$ for Materials A, B, and C, respectively. For Materials A and B, the fracture toughnesses and R-curves of the two directions were similar. However, for Material C there was an unexpected amount of scatter (Fig. 8) using specimen II. Experimentally, the test was difficult because fracture occurred unpredictably. Although the J_c values are similar, the R-curves are clearly different indicating that propagation properties may be different in the two directions.

The ductile fracture toughness obtained by the J method can be related to K_c, the critical stress intensity factor, which is a measure of strength. The equation for specimens under plane strain is $K_{cl}^2 = (J_{cl}E)/(1 - \nu^2)$. By calculating K_{cl} from J_{cl} and using the ASTM requirement for K testing ($B > 2.5\,(K_{cl}/\sigma_y)^2$), a comparison of the recommended minimum thicknesses for each type of toughness test can be made. As shown in Table III, the J-integral method

TABLE III Comparison of minimum specimen thicknesses for J and K testing

Material	Minimum thickness for J (mm)	Minimum thickness for K (mm)
A	15.0	70.3
B	5.8	20.3
C	7.5	35.8

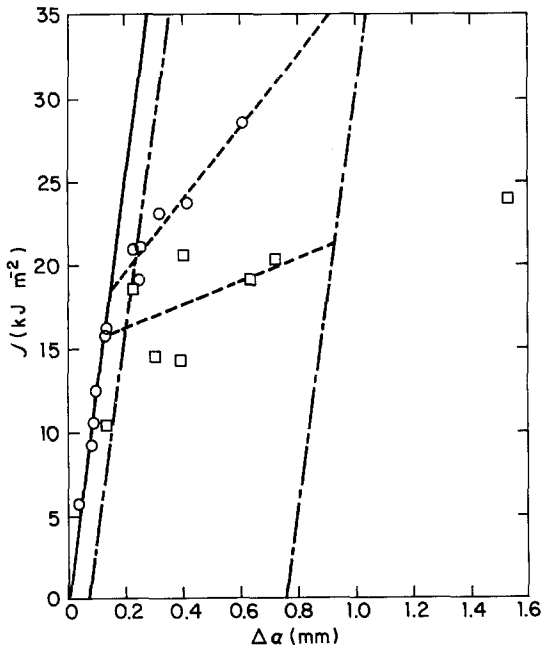

Figure 8 Material C, 0.26 mm sec^{-1}, thickness = 12 mm. (○) I, J_{cl} = 18.5 kJ m^{-2}, (□) II, J_{cl} = 15.8 kJ m^{-2}.

can decrease the minimum thickness requirement by a factor of 3 to 5.

In Table IV, J_{cl} and K_{cl} values are listed for the present study. In addition, literature values [9] of J_{cl} and K_{cl} for a variety of toughened polymers tested at 20° C are listed. The minimum J_{cl} and K_{cl} values for Material A are significantly better than the other materials. The toughness values of Materials B and C, while lower, are still comparable to the best of the rest. While extensive J work has not been conducted on toughened polymers, Material A ranks among the best in toughness and strength for polymers at any temperature. (Linear low-density polyethylene has a J_{cl} value of 18 kJ m^{-2} and a K_{cl} of 7.2 MPa m$^{1/2}$ at −100° C [13].)

6. Conclusions

The J-integral technique provides a suitable means to fracture toughness measurement in toughened polymers with achievable specimen sizes. Test rates (up to 26 mm sec^{-1}) were found to be satisfactory for J tests provided that all material constants were measured at the same test rate. When applied to tough systems, ASTM E813 need not be followed scrupulously. Despite the differences listed earlier, the J data appear sensible and self-consistent. However, because of these differences, the measured J_{cl} values for Materials A, B, and C may be conservative.

TABLE IV Comparative toughness for other toughened polymers

Material	J_{cl} (kJ m^{-2})	K_{cl} (MPa m$^{1/2}$)
A	30.0	8.40
B	16.0	6.20
C	18.5	7.41
Polypropylene [9]	15.5	4.97
Rubber-toughened PVC [9]	3.0	3.09
ABS [9]	3.0	2.68

References

1. J. G. WILLIAMS, "Fracture Mechanics of Polymers" (Ellis Horwood-Wiley, Chichester, 1984).
2. S. HASHEMI and J. G. WILLIAMS, *J. Mater. Sci.* **19** (1984) 3746.
3. J. F. MANDELL, A. Y. DARWISH and F. J. McGARRY, *Polym. Eng. Sci.* **22** (13) (1982) 826.
4. J. F. MANDELL, D. R. ROBERTS and F. J. McGARRY, *ibid.* **23** (7) (1983) 404.
5. J. R. RICE, *J. Appl. Mech.* **35** (1968) 379.
6. K. B. BROBERG, *J. Mech. Phys. Solids* **19** (1971) 407.
7. J. A. BEGLEY and J. D. LANDES, "The J-Integral as a Fracture Criterion", in "Fracture Toughness", ASTM STP 514 (American Society for Testing and Materials, Philadelphia, 1972) pp. 1–20.
8. M. K. V. CHAN and J. G. WILLIAMS, *Int. J. Fracture* **19** (1983) 145.
9. S. HASHEMI and J. G. WILLIAMS, *Polym. Eng. Sci.* **26** (11) (1986) 760.
10. J. D. LANDES and J. A. BEGLEY, "Test Results from J-Integral Studies: An Attempt to Establish a J_{cl} Testing Procedure," in "Fracture Analysis", ASTM STP 560 (American Society for Testing and Materials, Philadelphia, 1974) pp. 170–86.
11. J. D. SUMPTER and C. E. TURNER, *Int. J. Fracture* **9** (1973) 320.
12. C. F. SHIH, W. R. ANDREWS, H. G. DE LORENZI, *et al.*, "Crack Initiation and Growth Under Fully Plastic Conditions: A Methodology for Plastic Fracture", Report NP-701-SR (Electric Power Research Institute, Palo Alto, California, February 1978) pp. 6.1–6.63.
13. S. HASHEMI and J. G. WILLIAMS, *Polymer* **27** (1986) 384.
14. I. M. WARD, *J. Mater. Sci.* **6** (1971) 1397.
15. J. G. WILLIAMS and D. D. HUANG, *Polym. Preprints* **26** (2) (1985) 153.

Received 18 August
and accepted 23 September 1986