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High-Rate *J*-Testing of Toughened Polypropylene

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ABSTRACT

In the present work the J-integral approach has been adopted to evaluate the high-rate fracture resistance curve of toughened polypropylenes. High-rate J-testing has been performed according to the multispecimen technique, following different procedures such as ASTM 813-81, ASTM E813-89 and ESIS P1-92. The results of the resistance to crack initiation, J_{IC}, *obtained by means of these different standards, are compared and discussed.*

INTRODUCTION

Since polymeric materials are increasingly used for load-bearing structural applications, a proper characterization of their fracture properties is needed. This is particularly important in the case of high-rate loadings which represent the most severe conditions to which a material can be subjected during its in-service life.

Traditional measurements of impact strength, such as obtained by means of Izod or Charpy tests, provide only an overall evaluation of the fracture resistance and are dependent on specimen geometry and sizes. Better characterization is to be sought in terms of intrinsic material properties which can be achieved by applying the fracture mechanics approach. Linear elastic fracture mechanics (LEFM) testing has been successfully applied to many brittle polymeric materials[1,2] by using substantially the standard procedure recommended with specific reference to metallic materials.[3]

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LEFM testing poses certain restrictions on the specimen dimensions in order to ensure plane strain conditions. Unfortunately, these size requirements are difficult to meet with very ductile materials, such as many toughened plastics, especially because specimen thicknesses larger than those that can normally be manufactured would be required. For elasto-plastic fracture mechanics (EPFM) the plane strain conditions, and therefore also the size requirements, are less restrictive, so that smaller test pieces can be employed.

One of the most widely accepted approaches of EPFM is the J-integral. Several methods for the determination of fracture resistance, J_{IC}, have been developed and standard procedures recommended with specific reference to metallic materials.[4-6] Though similar procedures have also been used by many investigators for polymeric materials, an official standard procedure for polymers is still lacking.[7]

In the present work, the J-integral approach is used to characterize the high-rate fracture behaviour of two samples of toughened polypropylene, following the multispecimen technique according to the ASTM E813-81,[4] ASTM E813-89[5] and ESIS P1-92[6] procedures. Previous works show the results of high-rate J-testing of toughened polymers obtained by using the ASTM E813-81 standard for data handling.[8-10] The aim of the present work is to compare, for the materials examined, the values of the high-rate initiation toughness in Mode I, J_{IC}, obtained according to the different methods mentioned above.

MULTIPLE SPECIMEN J_R CURVE METHOD

The J-integral was originally defined for two-dimensional problems as a path-independent line integral that characterizes the stress and strain field singularities at the crack tip for non-linear elastic or elasto-plastic materials.[11] It can also be expressed in terms of energy as:

$$J = -\frac{1}{B} \cdot \frac{dU}{da}$$

where U is the input energy of the loaded body, that is the area under the load–loadline displacement curve, B is the body thickness and a is the crack length.

To take into account the crack growth, the concept of resistance curve has been developed under some hypotheses,[12] where J is plotted against crack extension as shown schematically in Fig. 1. This concept is not

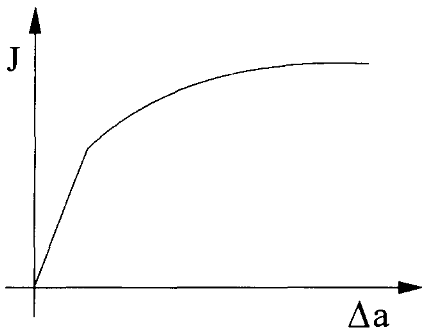

Fig. 1. Schematic J-integral–crack extension, Δa, diagram (J_R curve).

rigorously valid, but it provides a way to evaluate the energy needed to make the crack advance. The first part of the curve, associated with the blunting of the crack tip due to the formation of the plastically deformed zone around the crack tip itself, is frequently represented by a blunting line having the equation $J = 2\Delta a\sigma_y$, σ_y being the yield stress of the material. After the initiation point, J increases, indicating that the resistance to crack propagation is higher than the resistance to crack initiation.

According to the multiple specimen method to determine the J_R curve, originally proposed by Landes and Bagley,[13] identical notched specimens are loaded to different levels of the load-point displacement to obtain different extents of crack growth, and successively fully unloaded. Afterwards, the specimens are broken for direct measurements on the fracture surfaces of the crack extension, Δa, which occurred during loading. For each specimen the J value is evaluated by measuring the area U under the load–loadline displacement curve and using the equation

$$J = \frac{\eta U}{B(W-a)}$$

where η is an appropriate factor for the specimen geometry adopted, and B and $W-a$ are the specimen thickness and ligament, respectively. Then the J_R curve is obtained by plotting the J values as a function of the corresponding Δa values.

Different data handling procedures and validity requirements are prescribed by different standards, in order to ensure that the value obtained of J_{IC} characterizes intrinsically the fracture resistance of the material.

The main differences among the three different standards considered in this work for J_{IC} determination are briefly reviewed in the Appendix.

EXPERIMENTAL

Two samples of polypropylene (PP) modified with ethylene–propylene rubber (EPR), at 16 and 26 vol.% rubber content, respectively, were kindly supplied by Himont Italy SpA (Ferrara(I)) in the form of injection-moulded bars of $60 \times 12{\cdot}7 \times 3{\cdot}5$ mm dimensions. SE(B) specimens were obtained, introducing by a fly cutter an edge-notch with a depth to width ratio $a/W = 0{\cdot}5$ and a notch tip radius of about 15 μm.

High-rate J-testing was performed at room temperature and at an impact speed of 1·8 m/s by an instrumented pendulum from Ceast SpA (Turin (I)), which enables the force versus time curve to be recorded during the test.

The multispecimen technique to determine the J_R curve requires the practical ability to stop the tests at different extents of displacement. With this aim a rigid steel plate was fixed to the frame of the instrument, close to the anvils of the specimen, able to be moved back and forth very precisely by means of a screw. So, by suitable adjustment of the position of this plate, it was possible to stop the hammer of the pendulum at different displacements of the specimens. With respect to other experimental procedures,[8,14,15] this device offers the advantage of avoiding direct strokes of the tup against the stopper, which could damage the transducers placed inside the tup itself.

For each J-test, the crack advancement which occurred during loading has been measured, after successive cryogenic fracture at high speed, on the fracture surface of the specimen by means of an optical microscope.

The values of the high-rate yield stress for the materials examined, necessary to draw the blunting line on the plot of the J_R curve (see Appendix), were obtained by extrapolating the yield stress data measured at low rates between 10^{-2} and 10^3 mm/min in tensile tests performed by an Instron machine.

RESULTS AND DISCUSSION

A typical load versus time diagram obtained during an impact test performed to total break on a SE(B) specimen of the PP sample with 26 vol.% EPR added is shown in Fig. 2. The broad maximum observed, due to the ductile behaviour of the material, seems to indicate the non-applicability of the LEFM approach. This has been confirmed by calculating the ratio between the maximum load P_{max} and the load P_Q,[16] P_{max}/P_Q, where P_Q corresponds here to the intersection between the load–loadline displacement curve and the secant line through the origin with a

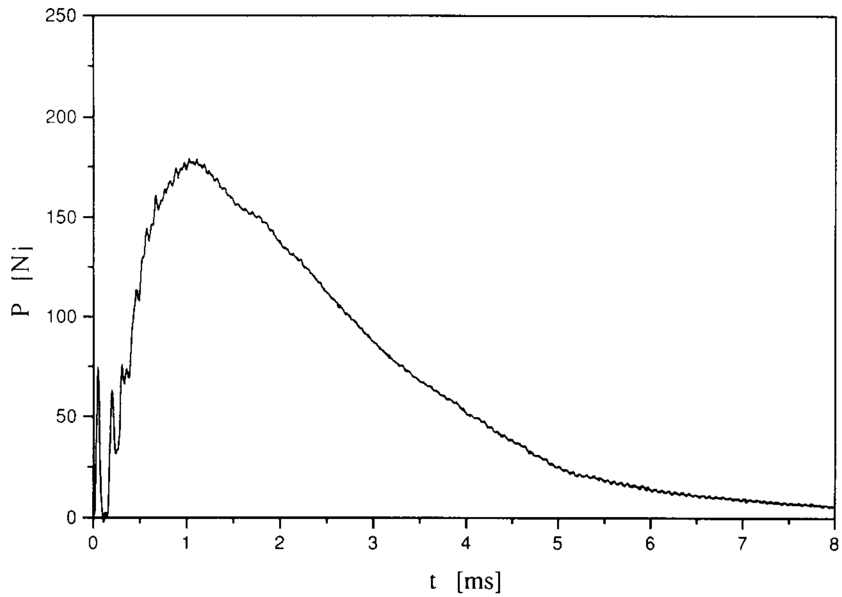

Fig. 2. Load versus time curve in a test at an impact speed of 1·8 m/s for a toughened polypropylene sample at 26 vol.% rubber content.

slope 5% lower than that of the tangent line—with results larger than 1·1. This implies that the test is to be considered invalid for the application of the LEFM approach.[16]

In relation to the application of the J-testing procedures considered, the necessity to stop the tests at different displacements of the specimens gives rise to load versus time diagrams like those shown in Figs 3(a) and (b), which have been obtained for the same material from tests stopped at 2·1 and 4·1 mm of displacement, respectively. The time to the arrest of the striker is clearly detected by the onset of wide-amplitude oscillations due to the vibrations induced in the transducers inside the tup when the striker is stopped.

From a series of tests similar to those reported in Fig. 3, the J values have been determined at different extents of crack growth for both the toughened PP samples examined. These values are reported in Figs 4–6 as a function of the crack advancement Δa. In these three figures the J_R curve for each material has been drawn by interpolating the experimental points following the three different procedures of ASTM E813-81,[4] ASTM E813-89[5] and ESIS P1-92,[6] respectively. It emerges that for the sample with higher rubber content the J_R curve is steeper and has higher values than the J_R curve relative to the other sample, independently from the method adopted. Further, all the different procedures applied seem to give J_R curves that fit the experimental data quite well, at least in the range

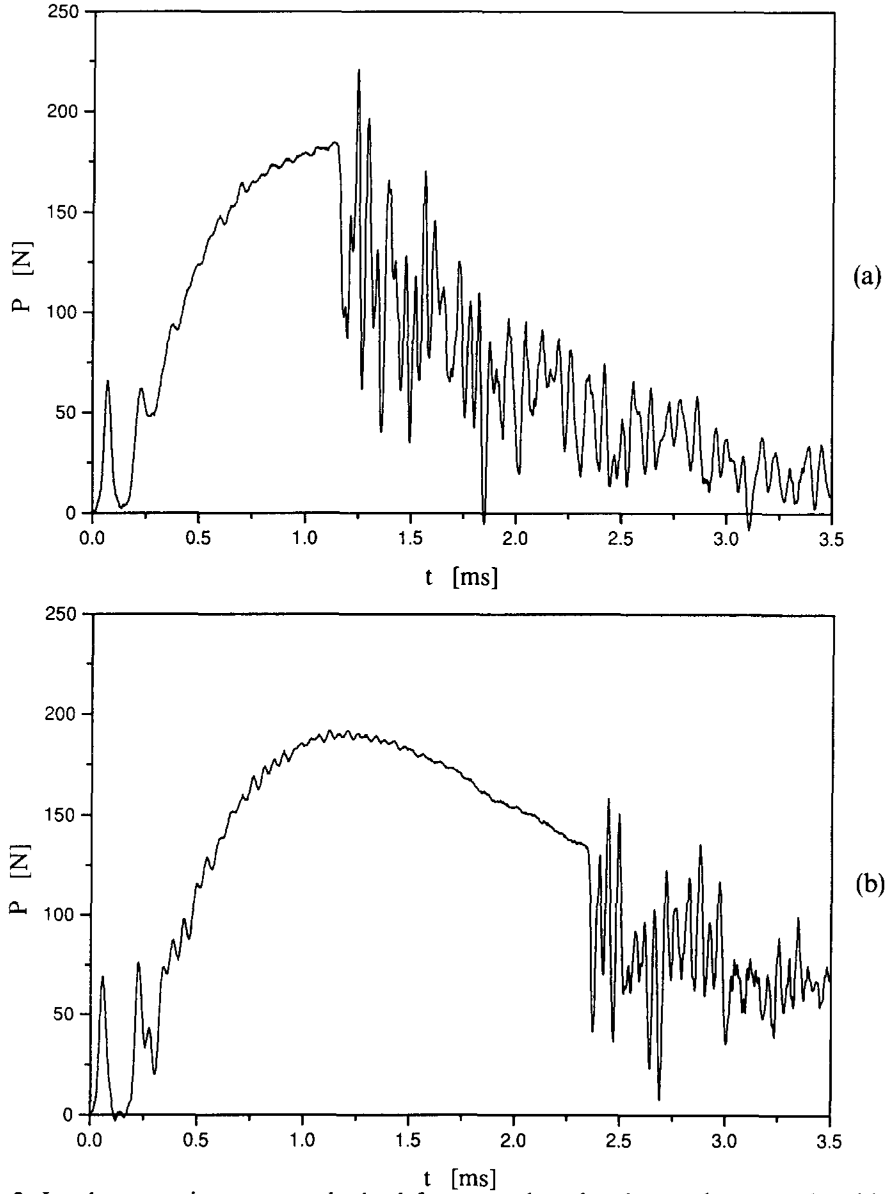

Fig. 3. Load versus time curves obtained for a toughened polypropylene sample with 26 vol.% rubber content, during impact tests stopped at (a) 2·1 mm; (b) 4·1 mm of displacement.

of Δa recommended. In spite of this, the J_{IC} values evaluated according to the different methods can differ significantly, as shown in Table 1.

In the case of the application of the procedure ESIS P1-92, the initiation fracture resistance, J_{IC}, has been determined both at the intersection of the

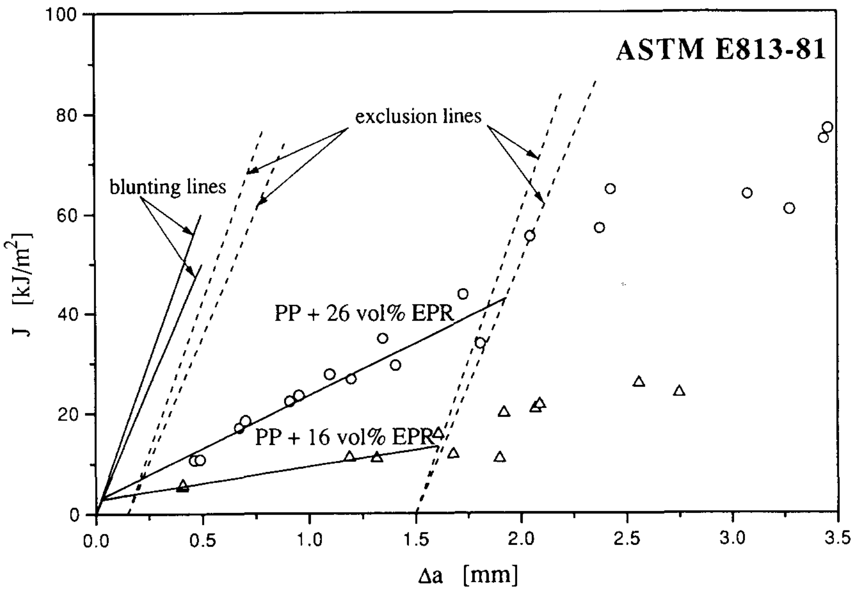

Fig. 4. *J*-Resistance curves obtained according to ASTM E813-81 standard for two toughened PP samples at different rubber contents.

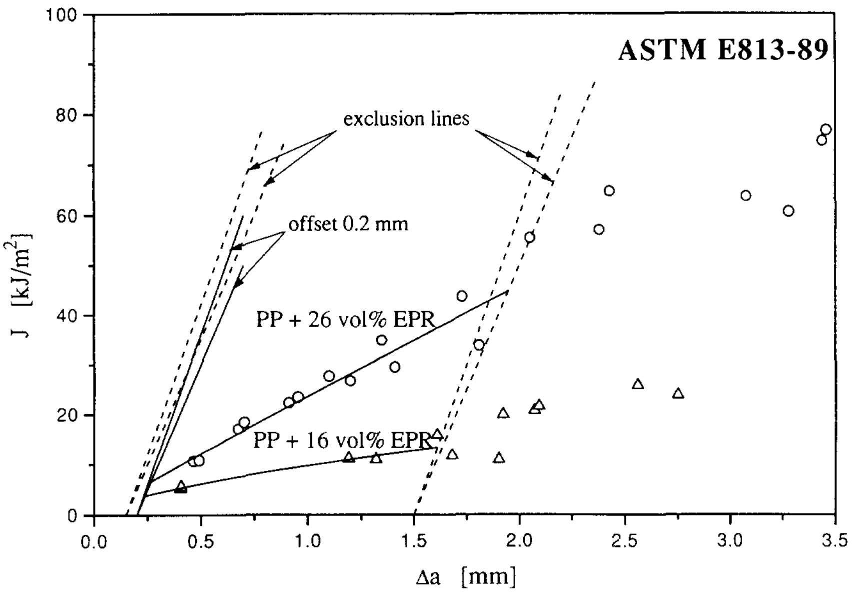

Fig. 5. *J*-Resistance curves obtained according to ASTM E813-89 standard for two toughened PP samples at different rubber contents.

J_R curve with the offset line parallel to the blunting line at 0·2 mm of crack growth, $J_{0\cdot2/BL}$, and directly at 0·2 mm of crack advancement, $J_{0\cdot2}$. Both values are reported in Table 1.

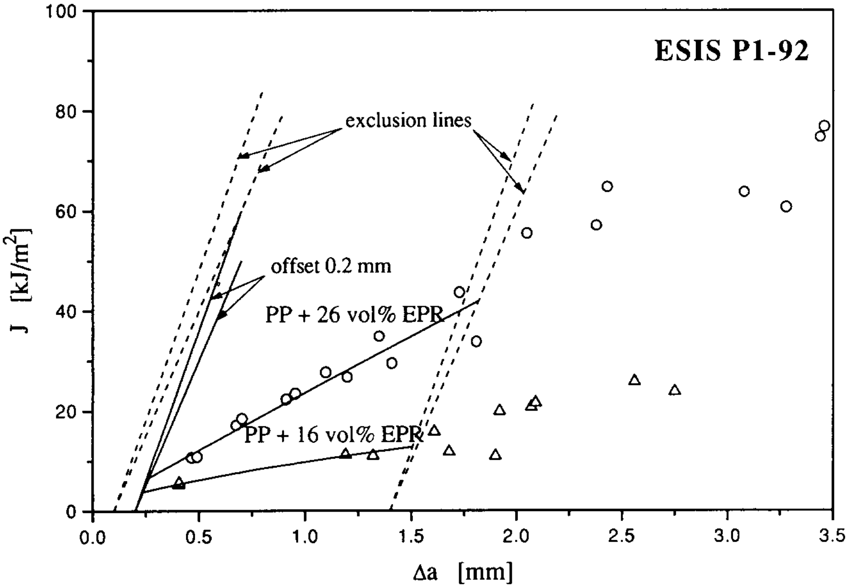

Fig. 6. *J*-Resistance curves obtained according to ESIS P1-92 procedure for two toughened PP samples at different rubber contents.

From the J_R curves obtained by the different methods the values of dJ/da, evaluated at $J = J_{IC}$, have also been determined in order to characterize the resistance of the material to crack propagation, though this parameter is not an intrinsic material property, depending, as it does, on geometry.[9] However, having considered specimens with the same geometry and sizes, we can claim that the values of dJ/da, evaluated at $J = J_{IC}$ for the different J_R curves, are comparable. The values of this parameter obtained from the different J_R curves of Figs 4–6 are also shown in Table 1.

Different considerations must be given to each of the two materials tested. For the sample at low rubber content all the J_{IC} values and the values of dJ/da at $J = J_{IC}$, evaluated according to the different protocols, are found to be in good agreement except those obtained by applying the ASTM E813-81 method. In fact, such a method provides values of both J_{IC} and dJ/da at $J = J_{IC}$ that are underestimated with respect to those obtained following the other procedures.

For the tougher sample, at higher rubber content, the J_{IC} values show some disagreement in relation to the procedure adopted. Specifically, the ASTM E813-81 standard provides a J_{IC} value which is about twice as low as those provided by the other standards considered. This result agrees with that found for toughened nylon at low-rate testing.[17]

TABLE 1

J_{IC} and dJ/da (at $J = J_{IC}$) Values Provided by the Different Procedures Adopted for the Materials Examined

Materials	*ASTM E813-81*		*ASTM E813-89*		*ESIS P1-92*			
	J_{IC} (kJ/m^2)	$\left.\frac{dJ}{da}\right\|_{J=J_{IC}}$ (MJ/m^3)	J_{IC} (kJ/m^2)	$\left.\frac{dJ}{da}\right\|_{J=J_{IC}}$ (MJ/m^3)	$J_{IC}(J_{0\cdot2/BL})$ (kJ/m^2)	$\left.\frac{dJ}{da}\right\|_{J=J_{0\cdot2\,BL}}$ (MJ/m^3)	$J_{IC}(J_{0\cdot2})$ (kJ/m^2)	$\left.\frac{dJ}{da}\right\|_{J=J_{0\cdot2}}$ (MJ/m^3)
PP+16% EPR	2·8	6·6	3·7	10·6	3·6	11·2	3·3	11·8
PP+26% EPR	3·3	21·0	6·6	23·9	6·4	25·6	4·7	26·2

The very good agreement between the J_{IC} value obtained by the ASTM E813-89 procedure and the value $J_{IC} = J_{0\cdot2/BL}$ evaluated according to the ESIS P1-92 recommendation must be mentioned. The same holds for the parameter dJ/da (at $J = J_{IC}$).

Further, it appears that the two methods of evaluating J_{IC} according to ESIS P1-92, that is $J_{0\cdot2/BL}$ and $J_{0\cdot2}$, provide different values. It emerges that for the material at the higher rubber content $J_{0\cdot2}$ is lower than $J_{0\cdot2/BL}$, whereas the values of dJ/da at $J = J_{0\cdot2/BL}$ and at $J = J_{0\cdot2}$ are quite similar for both the materials tested.

The analysis of the results points out the consistence of the ASTM E813-89 and ESIS P1-92 procedures for determining the high-rate fracture resistance of toughened polypropylene.

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REFERENCES

1. Williams, J. G., *Fracture Mechanics of Polymers.* Ellis Horwood, Chichester, 1984.
2. Kinloch, A. J. & Young, R. J., *Fracture Behaviour of Polymers.* Applied Science Publishers, London, 1983.
3. ASTM E399-83a, Standard test method for plane strain fracture toughness of metallic materials. *Annual Book of ASTM Standards,* Philadelphia, PA, **02.02**, 1990, pp. 686–710.
4. ASTM E813-81, Standard test method for J_{IC}, a measure of fracture toughness. *Annual Book of ASTM Standards,* Philadelphia, PA, Part 10, 1981, p. 810.
5. ASTM E813-89, Standard test method for J_{IC}, a measure of fracture toughness. *Annual Book of ASTM Standards,* Philadelphia, PA, **03.01**, 1989, p. 700.
6. ESIS P1-92, *ESIS Recommendations for Determining the Fracture Resistance of Ductile Materials.* ESIS, European Structural Integrity Society, Technical Committee 1, 1992.
7. Development of a standard protocol is in progress by ESIS, European Structural Integrity Society, Technical Committee 4, Polymers and Composites.
8. Savadori, A., Impact testing of plastics: present knowledge. *Polymer Testing,* **5** (1985) 209–41.
9. As quoted in Ricco, T. & Pavan, A., Fracture mechanics of rubber-modified polyamide-6: temperature and rate effect with varying rubber content. *Ang. Makromol. Chem.,* **201** (1992) 23–31.

10. Huang, D. D. & Williams, J. G., *J* testing of toughened nylons. *J. Mater. Sci.*, **22** (1987) 2503–8.
11. See for example Turner, C. E., In *Post-Yield Fracture Mechanics*, 2nd edn, ed. D. G. H. Latzko. Elsevier Applied Science Publishers, London, 1984.
12. Hutchinson, J. W. & Paris, P. C., ASTM STP 668. American Society for Testing and Materials, Philadelphia, PA, 1979, pp. 37–64.
13. Landes, J. D. & Begley, J. A., ASTM STP 560. American Society for Testing and Materials, Philadelphia, PA, 1974, pp. 170–86.
14. Server, W. L., ASTM STP 668. American Society for Testing and Materials, Philadelphia, PA, 1979, pp. 493–514.
15. Savadori, A., Bramuzzo, M. & Marega, C., *J*-integral analysis of ductile fracture of PP/EP rubber blends. *Polymer Testing*, **4** (1984) 73–89.
16. ESIS, European Structural Integrity Society, Protocol, *A Linear Elastic Fracture Mechanics Standard for Determining K_C and G_C for Plastics*. ESIS Technical Committee 4, 1990.
17. Hashemi, S. & Williams, J. G., Single and multi-specimen *R*-curve methods for determination of J_{IC} toughened nylons. *J. Mater. Sci.*, **26** (1991) 621–30.

APPENDIX

A brief description of the methods for data handling to determine the value of J_{IC} according to the different standards is reported below.

ASTM E813-81

To establish the validity of the experimental data according to this standard, it is necessary to plot on the J–Δa diagram the blunting line $J = 2\Delta a\sigma_y$, and two exclusion lines parallel to the blunting line with an offset of 0·15 and 1·5 mm, respectively, as shown in Fig. A.1a. All data

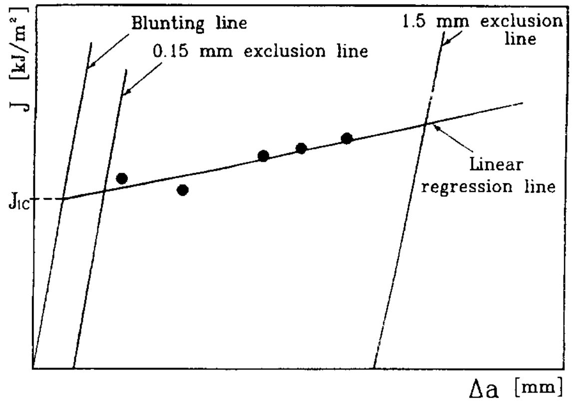

Fig. A.1a. Definition of region of valid data according to ASTM E813-81.

should be placed inside the area enclosed by the two parallel offset lines. Data outside these limits are not qualified for use in R curve development. At least four data points must remain inside this area and at least one remaining data point should be required near the blunting line, otherwise further experimental data are necessary. All data points between the exclusion lines must verify the following requirements:

$$B, (W-a) > 15J/\sigma_y \tag{A.1}$$

The best-fit linear regression line through the qualified J–Δa points represents the J_R curve. The intersection of this linear regression line with the blunting line marks J_Q.

J_Q represents J_{IC}, if the following requirements are satisfied:

thickness	$B > 25J/\sigma_y$	(A.2)
initial ligament	$(W-a) > 25J/\sigma_y$	(A.3)
slope of the regression line	$dJ/da < \sigma_y$	(A.4)

ASTM E813-89

Also in this version of the ASTM E813 standard the J–Δa points used for the determination of the J_R curve must lie between two exclusion lines, each drawn parallel to the blunting line, with an offset of 0·15 and 1·5 mm, respectively. It is also required that at least one point should be placed in zone A and at least one point in zone B, as shown in Fig. A.1b. The

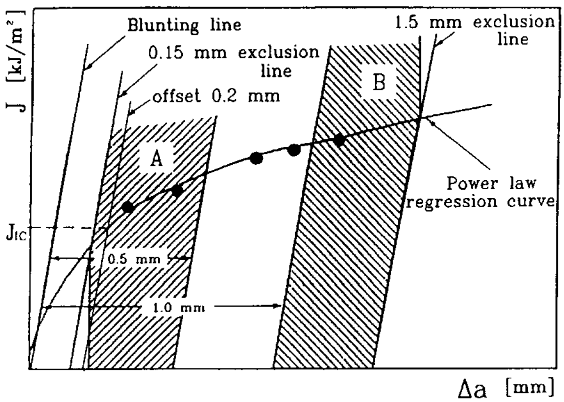

Fig. A.1b. Definition of region of valid data according to ASTM E813-89.

acceptable data are then fitted by a power law regression curve of the form:

$$J = C_1(\Delta a)^{C_2}$$

The intersection of this power law regression curve with a line parallel to the blunting line at an offset of 0·2 mm defines J_Q. Again, this value is assumed as the initiation toughness, J_{IC}, if requirements (A.1)–(A.4), listed above, are satisfied.

ESIS P1-92

In this procedure the exclusion lines, parallel to the blunting line on the J–Δa diagram, should be drawn with an offset of 0·1 mm and Δa_{max},

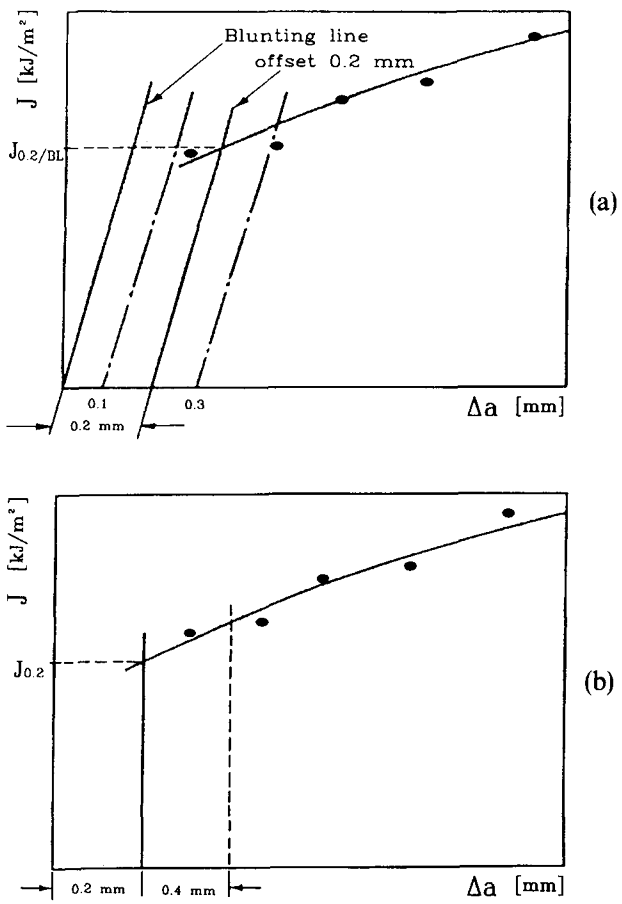

Fig. A.2. Determination of (a) $J_{0\cdot 2/BL}$, (b) $J_{0\cdot 2}$ according to ESIS P1-92 procedure.

respectively, being $\Delta a_{max} = 0{\cdot}1(W-a)$. The area between the two exclusion lines is divided into four zones equally spaced along the Δa-axis. At least one data point should lie in each zone. The best-fit curve through the data points, which lie inside this area, is determined using an equation of the form

$$J = C_1(\Delta a + C_3)^{C_2}$$

This standard allows the determination of two parameters for estimating J close to the onset of crack initiation on the J_R curve: $J_{0{\cdot}2/BL}$ which measures the fracture resistance at 0·2 mm crack growth beyond crack initiation and $J_{0{\cdot}2}$ which measures the fracture resistance at 0·2 mm of total crack growth including crack tip blunting.

(i) The parameter $J_{0{\cdot}2/BL}$ is defined by the intersection of the best-fit curve with a line parallel to the blunting line with an offset of 0·2 mm, as shown in Fig. A.2(a). If

$$J_{0{\cdot}2/BL} < J_{max}$$

being $J_{max} = \min\{(W-a)\sigma_y/25 \text{ and } B\sigma_y/25\}$, $J_{0{\cdot}2/BL}$ is assumed as the J_{IC} value.

(ii) The parameter $J_{0{\cdot}2}$ is defined by the intersection of the best-fit curve with a line corresponding to a constant total crack growth of 0·2 mm, as shown in Fig. A.2(b). If

$$J_{0{\cdot}2} < J_{max}$$

where J_{max} is defined above, $J_{0{\cdot}2}$ is assumed as the J_{IC} value.