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The use of a velocity gauge in impact testing of polymers

B. STALDER, H. H. KAUSCH
Laboratoire de Polymères, Ecole Polytechnique Fédérale de Lausanne, 32, chemin de Bellerive, CH-1007 Lausanne, Switzerland

A velocity gauge has been devised for fracture studies on polymers which seems to describe well the state of strain existing under the gauge. In view of the growing interest in impact properties, a detailed examination is made here on the characteristics of this gauge at high loading rates and high crack velocities. A range of polymers were tested which show that the state of elastic strain and the length of the plastic zone are recorded before the crack velocity is measured. An interesting feature resides in the fact that the small dimensions of the gauge permit considering these results as being dynamic parameters, directly linked to the crack tip behaviour. Some further possibilities are pointed out in the field of toughness measurements for high-impact resistant polymers.

1. Introduction

Growing interest is given to impact behaviour of polymers because of the increasing use of plastics in objects of everyday use and technical parts. Many of these are likely to fracture under impact conditions: protection glasses of car lights, cores of skis, vacuum cleaner housings, typewriter daisies, crash helmets and so forth. An empirical way of ensuring that a car bumper, for example, meets the specifications would be to test it under real conditions. The problem, however, is that it must first be manufactured on an arbitrary basis and then tested under all possible crash configurations. At least two different characteristics are involved: the mechanical design of the bumper and the intrinsic properties of the polymer.

To analyse the intrinsic properties at the high load rates which are of concern, one often uses the well-known Charpy test. A swinging pendulum of a determined mass drops from a fixed height and knocks against a three-point bending specimen. The energy lost by the hammer during loading and rupture is indicated on a built-in dial. It is principally the sum of the contributions of fracture initiation, crack propagation and kinetic energy. This test was soon felt to be more appropriate if a continuous load–deflection curve could be obtained by means of an added instrumentation and if fracture mechanics parameters could be drawn from it [1]. Load mesurements may be carried out by placing strain gauges on the tup of the hammer or, in a more sophisticated way, by attaching an accelerometer to the falling weight [2] or making use of laser-Doppler velocimetry [3]. The load–time or energy–time curves are unfortunately difficult to relate to the energy or to the stresses involved at the crack tip, because of the complex stress-wave behaviour [4].

The difficulties encountered with dynamic loading conditions could be diverted with a local probe. A thin layer of conductive material deposited around the crack tip had been devised [5] as a crack velocity gauge. An independent study of its potentialities has shown that its response also depends on the strain field around the crack tip [6, 7]. In the following sections we will examine in detail its possible uses in Charpy-impact tests.

2. Main characteristics of the gauge

The design and the calibration of the gauge were already described elsewhere [6]. However, some of the related problems become more crucial in

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dynamic experiments. A reliable interpretation of the resulting electrical conductance–time curve needs the precise knowledge of the specific characteristics of the gauges. These were critically checked for their compatibility with high rates and high velocities.

2.1. Composition

Commercial graphite sprays, although not intended for this special use, provide a well-fitted mixture of particles, suspension liquid and polymeric binder. In some cases though, the suspension liquid is either too aggressive for the polymer being tested – it could react as a solvent or a crazing agent – or it does not wet the polymer surface because of the inadequate interfacial tension. One should then produce a suitable composition which is to be sprayed with a refillable aerosol can, or is deposited by means of a screen-printing procedure. Attention should be paid to keep the concentration of the polymeric binder to a minimum, just sufficient to ensure some mechanical stability for handling the specimens. A layer which is too compact would not rupture at the same local deformation as the specimen. Actually, there is a minimum opening displacement between the crack surfaces, below which the contact is not annihilated as a bridge is built over the crack. In our experiments, this particular problem has not yet been encountered, even for materials with a crack-tip opening displacement of only some 0.5 μm. This aspect should, however, be kept in mind when brittle and stiff polymers or composites are to be tested.

The applied method should provide a layer of graphite which is homogeneous at least at distances larger than the desired absolute precision of crack length measurement. A typical value of resistance is 1 kΩ, measured for a square area of graphite with two opposite electrodes. The fineness of the aerosol is qualitatively given by the minimum mass of graphite which forms a continuous layer, that is to say gives a non-zero conductivity. In the example presented in Fig. 1, a deposit of less than 2 g m^{-2} would just build unconnected spots, the size of which give an idea of the smallest measurable crack growth. The thinner the layer, the better is its ability to follow any deformation and hence crack propagation. These criteria define a useful range centred around 10 g m^{-2} leading to thicknesses of about 5 to 10 μm.

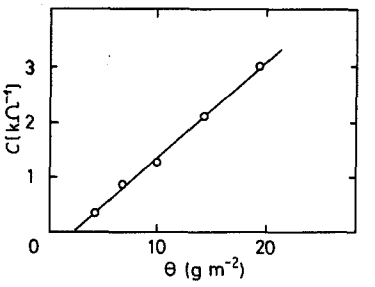

Figure 1 Conductance of the gauges as a function of the mass of graphite (GRAPHITE 33).

2.2. Calibration

it has already been pointed out [6] that for a gauge of width b and length l the simple relationship between conductance and relative crack length

$$R_0/R = 1 - (a/l) + \Delta$$

is not valid for a/l near 0 or 1, because of the establishment of a non-linear distribution of the potential in the vicinity of a crack. The parameter Δ describes the deviation compared to an infinitely "narrow" ($b/l \to 0$) gauge. An experimental investigation gave: $\Delta \simeq 0.20\ b/l$. The extent of the quasi-linear zone (at $\pm$ 1% of a_m) is also a function of Δ. For a square gauge, the useful range is less than 50% of the total length. This implies two major geometric requirements:

1. the gauge must be narrow to increase the measurable length, but not too much, in order to avoid crack propagation out of the graphite area; and

2. the initial crack length a_0 (prior to testing), must be greater than a minimum value $a_{min} \simeq 0.40\ b$ at which the current distribution becomes almost stable with crack propagation.

Provided these requirements are met, together with homogeneity and parallelism of the silver electrodes, the above conductance and crack length is valid.

2.3. Environmental effects

There are some side-effects responsible for a change in resistance, thus limiting the precision of the crack length measured: humidity and temperature. Humidity and eventually other vapours drastically increase the resistance of the gauge. This is probably due to the loss of contact between adjacent graphite particles produced by the adsorbed molecules. Protection against a

high moisture content of the environment can be obtained with a sealed cap. Higher temperatures favour the rearrangement of the particles, which occurs with time, leading to an increase in conductivity. Nevertheless, a stable resistance is reached within less than 1 h. Any variation of these two parameters should be avoided during the test.

2.4. Strain sensitivity

A very important feature of the velocity gauge is the modification of the effective contact area between particles during straining. Fig. 2 shows the relative resistance change as a function of strain in a low stress region, where the supporting material, PMMA, can be considered as elastic (Poisson's ratio, $\nu = 0.38$). Tension specimens with a square gauge, bordered by two silver electrodes perpendicular to the traction axis, were used here showing three noticeable characteristics:

1. the curve is clearly non-linear, although somewhat straight when the strain is released. Its mean slope ranges from 10 to 20. A strong hysteresis is present;
2. the successive deformation cycles differ from the first one, the rate of increase in resistance being lower;
3. the resistance for a given deformation decreases at each cycle. It tends to stabilize, provided the initial maximum strain is not exceeded.

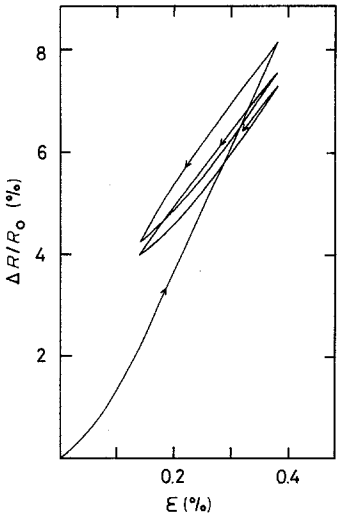

Figure 2 Relative conductance change of a gauge placed on a PMMA tensile specimen as a function of strain.

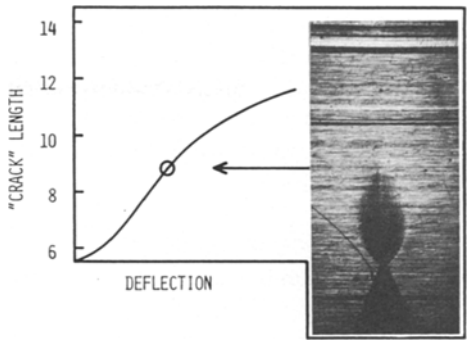

Figure 3 Comparison between the measured "crack" length and the length of the plastic zone for a PVC specimen.

The very high biaxial deformation present in the side surface of a yield zone causes a drastic decrease in local conductance. As an example, a notched beam of PVC, the span and section of which were, respectively, 50 mm and 12.5 mm × 10 mm, was tested in three-point bending at a slow crosshead speed. An increasing yield zone appeared followed by a crack. The response of the gauge is reported in Fig. 3. Besides recording the value of R_0/R, an optical determination of the plastic zone length was performed using the reflections from the plane areas. A very good correlation to within some 4% was found. Over the deformed region, the conductivity is found to be less than a tenth of the original one. This effect is obviously not too far from the one resulting from a crack. It appears therefore that the graphite gauge depicts the length of both the crack and the preceeding yield zone. Very tough polymers provide a smooth variation of resistance with deflection and the first increase in crack length may not be easy to notice, unless use is made of the load–time curve and of the topology of the fractured surface. The latter information, on the other hand, is indispensible to correlate the inner to the surface behaviour recorded by the gauge.

An absolute calibration of the influence of elastic strains has not yet been elaborated mainly because of lack of reproducibility of the strain sensitivity from one gauge to another. Consequently, the variations of the strain field, caused by the stress concentration area of the crack tip during crack propagation, are responsible for an uncertainty in the crack length measurement. It is generally restricted to a few per cent, although greater for tension-type than for bending-type specimen. Even though it is not measured

quantitatively, this strain-induced deviation of conductivity permits one to identify in impact testing the precise moment at which the hammer touches the specimen. Thus, the time to fracture is very easily determined in conjunction with the subsequent crack speed.

2.5. Conductance measurement

A wirewound potentiometer, R_v, is placed in series with the graphite gauge, R_x. A constant voltage U_0 is applied to them. The resulting current is then equal to $U_0/(R_x + R_v)$. Having chosen in our experiments a value of R_v (less than 10 Ω) which is small compared to an R_x of 300 to 1000 Ω. The voltage drop in R_v may be approximated by $U_m \simeq U_0 R_v/R_x$. By inserting the established relationship between conductance and crack length, one obtains:

$$U_m = \frac{U_0 R_v}{R_0}(1 + \Delta - a/l).$$

The relative crack length is a simple function of known or readily determined constants and of the measured potential U_m:

$$\frac{a}{l} = (1 + \Delta) - \frac{R_0}{U_0 R_v} U_m$$

R_0 is the resistance of the unnotched gauge. The voltage U_0 is restricted to a maximum of 5 V so that the gauge behaves like an ohmic resistor. The possible limit seems to be far above that value but unacceptable heating of the gauge, and hence of the substrate, may occur and alter the deformation and rupture processes or even soften the polymer. It has been proven that no measurable heating effects are observed with a voltage $U_0 = 5$ V applied to a 300 Ω gauge over several hours. The heat produced by Joule effect can dissipate without any noticeable temperature increase.

It is of major importance, when measuring fast fracture, to ensure that the electronic components used are able to follow the maximum voltage-drop rate obtained. Capacitive or inductive components may severely limit the frequency response. The gauge system (R_v and R_x) proposed here presents a global settling time of 1 μsec, determined by the potentiometer and not by the graphite gauge which gives better results.

*Hottinger Baldwin Messtechnik, KWS 3020B, Germany.
†René Maurer TMP, Switzerland.
‡Hewlett Packard 9836, USA.

3. Fracture behaviour of some polymers

3.1. Experimental procedure

All the experiments were performed using a Frank–Charpy pendulum designed for plastics. Among four available hammers, of 0.5, 1, 2 and 4 J, the heaviest was used. In most cases, this energy was high enough to warrant that crack initiation and, in part, propagation was under a constant hammer velocity of 2.9 m sec^{-1}. The distance between the anvils was 50 mm. The original tup was replaced by a hammer nose instrumented with strain gauges in order to record load. This cell was linked to a d.c.-strain gauge amplifier.* The output signal was recorded by a digital transient memory† and then sent to a computer‡ which stores and processes the data (cf. Fig. 4). A dynamic calibration was performed after each set of experiments, based on the comparison between the area of the load–displacement curve and the total amount of energy lost by the hammer, E_t, read on the built-in scale of the pendulum:

$$E_t = \int_{t_{init}}^{t_{fin}} P(t)\, V(t)\, dt.$$

As some of the initial kinetic energy of the hammer is lost during the impact event, the hammer velocity, $V(t)$, is not a constant and needs to be calculated. Absolute values of load were determined that way, although they were not used in this study.

Specimens were cut from extruded plates or from injection moulded beams. Table I gives a list of the polymers tested. The beams were 12.5 mm deep and 60 mm long. The thickness varies according to Table I.

V-notches were milled in a two-stage operation. At first a depth of 2.5 mm was removed with a 22 teeth, 45° slotting cutter. Subsequently, another 0.5 mm were milled off with a similar cutter having 20 rounded teeth and two contiguous sharp teeth. The notch obtained by this procedure seems to be sharp enough to simulate a natural crack in the case of impact testing. Tests were performed on PMMA and UP at slow crosshead speed to compare the value of K_{IC} for such notches and for natural cracks obtained by forcing a razor blade into the

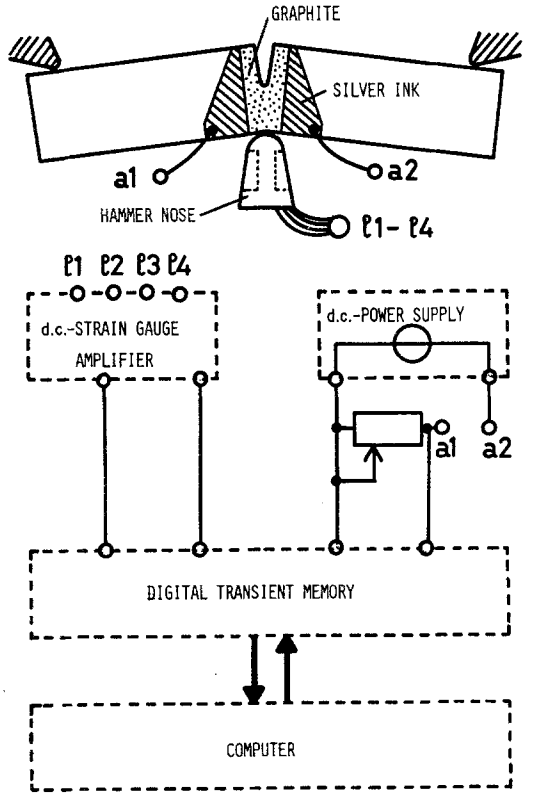

Figure 4 Block diagram of the measuring system.

material. The mean K_{IC} value was the same, although individual values were more dispersed in the case of natural cracks, because the crack front was never completely straight. As a final operation, the lateral area of all beams was sprayed with graphite[§] over a square region 12.5 mm by 12.5 mm. Two silver electrodes[¶] were drawn on the graphite layer, at a distance of approximately 5 mm one from the other. Two thin metallic pins were inserted in the specimen at the location of the electrodes so that they linked the gauge to the leads of the measuring circuit. The potential U_m is recorded on the second channel of the digital oscilloscope. The final evaluation and presentation of both load and crack length data is made on the computer.

The transient memory records a maximum of one point each half microsecond. Assuming that twenty points at least are required to estimate correctly the position of the crack over the whole gauge, the upper limit of measurable crack velocity is 500 m sec^{-1} for a gauge of 5 mm length and 5000 m sec^{-1} for a length of 50 mm. Even for the smallest gauges, this limit is adequate to measure the fracture behaviour of high polymers. The precision on the crack length is $\pm 0.2\%$, determined by the 8-bits configuration of the digital transient memory.

3.2. Determination of the time to fracture

Kalthoff *et al.* [8], in their excellent work, proposed a simplified procedure to determine the dynamic stress intensity factor, K_{Id}, based on the measurement of the time to fracture, t_f. t_f is understood here as the time interval between the first contact and the moment when crack propagation begins. A predetermined impact response curve, satisfying the experimental conditions, is then sufficient to deduce K_{Id}. Although this method was devised primarily for steels, many experiments were performed by Böhme and Kalthoff [9] on a particular epoxide resin showing its potential usefulness in the impact testing of polymers.

Several methods may be used to measure t_f. Kalthoff *et al.* [8] recorded the signals of two strain gauges, one on the hammer tup to identify the moment of first contact, the second at the crack tip of the specimen to reveal the onset of crack propagation. The latter may be replaced by a simple conducting line, cut by the crack.

TABLE I Materials used and thickness of specimens

Symbol	Material		Thickness (mm)
PMMA	Polymethylemethacrylate	Plexiglas 233, Röhm AG	10.1
PC	Polycarbonate	Makrolon, Bayer AG	9.9
PE	High-density polyethylene	BP	11.0
PVC	Polyvinylchloride		10.2
EP	Epoxy	Araldit B, Ciba-Geigy	10.0
UP	Unsaturated polyester	VP 1530	9.4
PA	Polyamide 12	L20G, EMS AG	5.8

[§]GRAPHIT 33, Kontakt Chemie, Rastatt, Germany.
[¶]LIETSILBER 200, Demetron, Hanau, Germany.

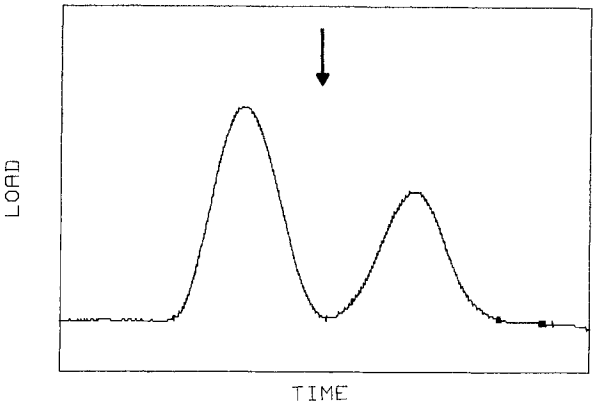

Figure 5 Load–time curve obtained for a PVC specimen. The arrow indicates the onset of crack propagation.

The first contact may also be determined using the electrical switch composed of the metallic hammer and a conductive layer deposited on the rear face of the specimen. A load–time curve (Fig. 5) recorded on a PVC specimen clearly shows the beginning of the impact but does not give the time when the crack propagation actually begins. Even if taking into account the delayed response of the load cell, due to the travel of the stress waves, it is impossible to find a significant point (for example maximum load) on this diagram. This is generally the case when fracture initiation occurs during an oscillating load–time curve. This will be different if a graphite gauge is applied to one or both lateral sides of the specimen since it gives a precise measurement of the required time to fracture and records successively the beginning and the end of the loading event (see Fig. 8). The onset of crack propagation with the specimen of Fig. 5 was easily determined by this means, the event is marked by an arrow on the diagram.

3.3. Crack velocity

The first aim of the graphite gauge is the determination of the crack length as a function of time. A crack velocity may then be deduced for each point on the specimen. As reported in the previous paper [7], a precise measurement is obtained for brittle polymers. In the case of semi-ductile and ductile polymers, the length of the plastic zone (l_p) is roughly added to the crack length. Despite their inaccuracy, the terms "crack length" and "crack velocity" will be referred to for each case in the following considerations and figures.

The record of $a(t)$ for one representative specimen of PA 12 (see Table I) is presented in Fig. 6.

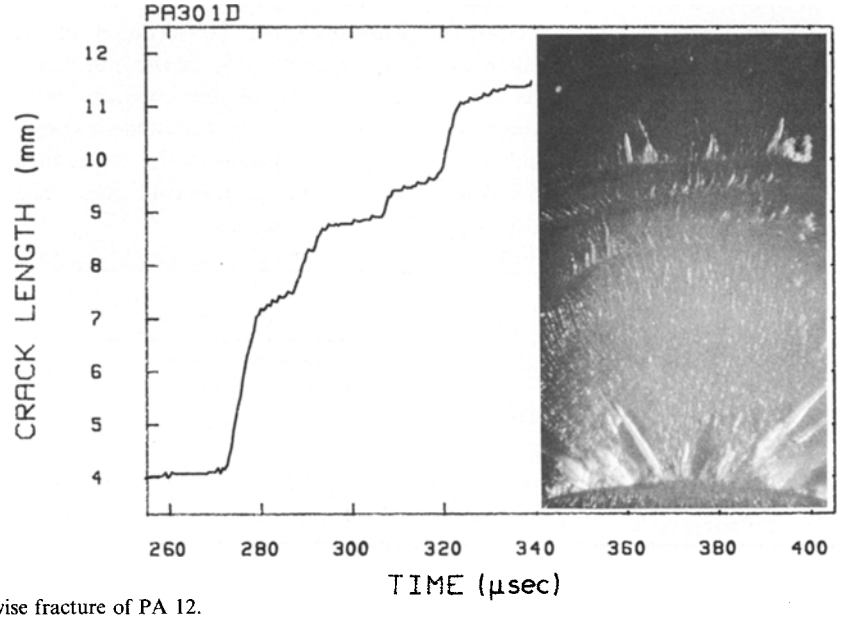

Figure 6 Stepwise fracture of PA 12.

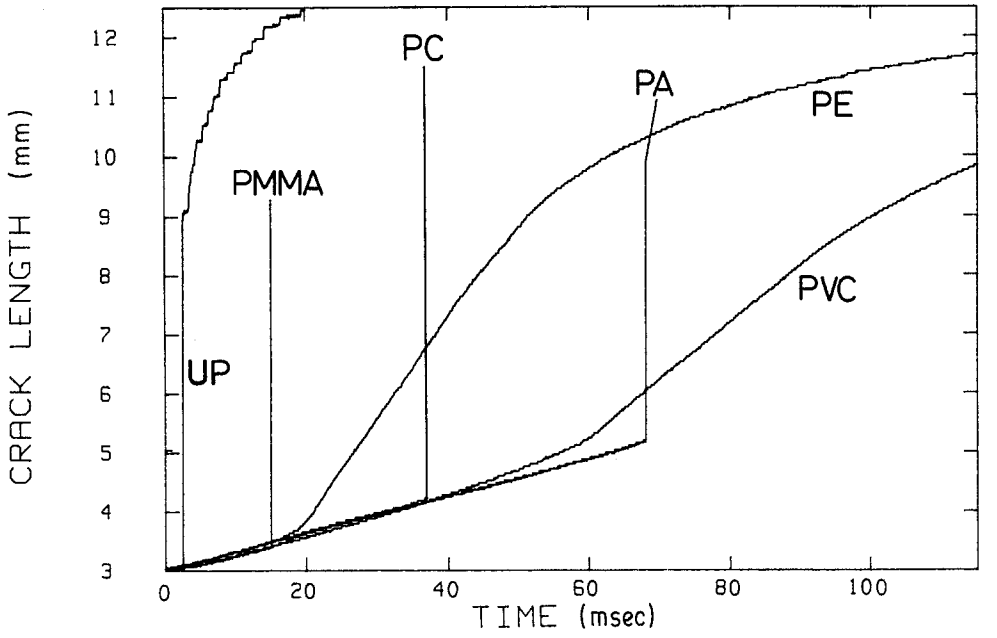

Figure 7 Characteristic curves at slow rate.

The impact behaviour of this polymer is quite distinct from the ones presented in the next section on Fig. 8. Its time to fracture is the longest. An extensive deformation, maybe followed by a stable crack, occurs at the notch tip before the crack propagates rapidly at a velocity rising up to 500 m sec^{-1}. The propagation proceeds by steps with an intermediate white zone where slow growth seems to occur at some 10 to 20 m sec^{-1}. De Charentenay *et al.* in their study on PA 11 and PA 12 [10], found the same stepwise propagation which they explained as a bouncing effect leading to a loss of contact between the striker and the specimen. It is probably in part a material property since other polymers with similar elastic constants and the same geometry behave quite differently. An insight into that kind of unstable behaviour can be obtained in studying the whole relationship between stress-intensity factor and crack velocity. The graphite gauge has proved to be more economical and accurate for that purpose than many other available methods [11]. It must be pointed out that the interpretation of $a(t)$ curves may be obscured by the dynamic state of stress [12] or by the inherent geometric instability of the specimen ([13] pp. 244–51).

3.4. Critical "plastic zone" length

The graphite gauge is strain sensitive in its whole area. Nevertheless, the major zone in that respect is located in front of the crack tip, where stresses and strains are very important. Contrary to a crack which effectively separates the gauge, an elastically or plastically strained region produces a gradually variable conductivity ahead of the crack tip. The resulting potential deviation, therefore, does not define a physical point. The apparent increase in crack length is related to the strained or plastic zone but it is not its actual length.

Some specimens of the materials listed in Table I and prepared in the way described for impact tests were loaded in three-point bending under a constant crosshead speed of 1 m min^{-1} with a span of 40 mm. This procedure allowed recording of the crack length curves under quasi-static conditions (Fig. 7). The nominal strain rate ([13] p. 270) was 0.78 sec^{-1} against 90 sec^{-1} in the impact tests.

The growth rates of the "plastic zones" were similar for all materials, about 0.03 m sec^{-1}, but different maximum lengths had to be attained before the crack propagated. This critical length is linked to what happens at the vicinity of the crack and not in the whole specimen. Its quantity should then be valid also under dynamic loading conditions. The result for the impact tests performed are presented in Fig. 8. The variation in mass (variable thickness) and in elastic moduli are responsible for the differences in the deformation curves.

The time scales of Figs. 7 and 8 were set to

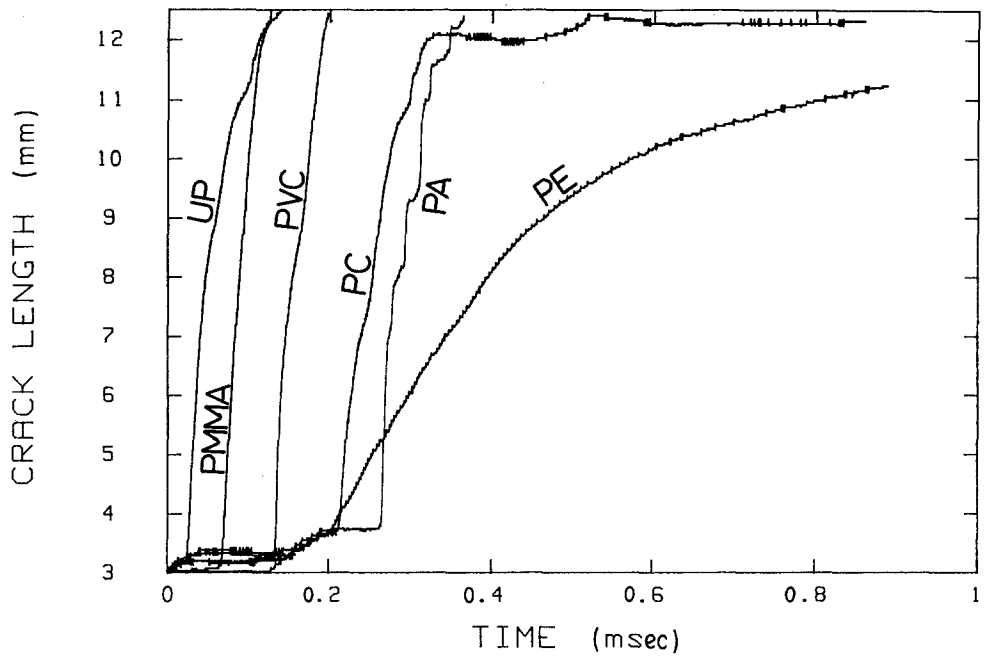

Figure 8 Characteristic curves under impact conditions.

give a ratio of 115.2 like that of the maximum strain rates. Comparing both sets of experiments, it appears (Table II) that the critical zone sizes are smaller under impact conditions, except for UP and PE, for which the values do not change much. The lengths measured for PA are accompanied by a question mark, because they may include an early stable crack growth.

4. Conclusions and further possibilities

The performance of the graphite gauge is tightly linked to the type of polymer being tested. Although the original idea was to measure the crack velocity in highly crosslinked and consequently brittle polymers, the results presented in the previous section show that the usefulness of this method is not restricted to that application. The resistance change observed before the crack propagates is the result of the state of strain present under the gauge. With the limitation that the dimensions of the gauge are small as compared to those of the specimen, a dynamic stress intensity factor could then be deduced. Such a measurement has not yet been pursued because of the intrinsic noise of the gauge which alters the signal. This approach could be an interesting alternative to the now common load instrumentation which eventually measures the overall response at the tup. An initial load peak is always present which denotes the acceleration of the specimen. This and the subsequent bouncing make it very difficult to interpret the tup load in terms of state of stress or strain near the crack tip. Some researchers attempted to overcome this difficulty by placing a plastic material between the hammer and the specimen: aluminium for tests with steel [14], plasticine in the case of polymers ([10], [13] p. 242). Bouncing is almost suppressed and the inertial peak is spread out. This procedure, however, necessitates two comments:

1. it still is not clear whether the quasi-static stress intensity factor calculated from the tup load is equivalent to the dynamic stress intensity factor describing the magnitude of the stresses at the crack tip;

2. the slow initial loading rate might disturb

TABLE II Critical zone length, l_p, for both loading conditions

l_p (mm)	PMMA	PC	PE	PVC	UP	PA
$\dot{e} = 0.78\,\text{sec}^{-1}$	0.4	1.2	0.6	2.2	0.1	2.2 (?)
$\dot{e} = 90\,\text{sec}^{-1}$	0.1	0.8	0.7	0.3	0.15	0.8 (?)

the original crack tip geometry allowing blunting of the crack tip in the case of viscoelastic materials.

The formation of a plastic zone, even limited, tends to absorb the vibrations due to dynamic loading. Among the examples shown in Fig. 8, high-density polyethylene behaves as a semi-ductile material (small-scale yielding). As soon as a crack propagates, load and crack length against time curves become quasi-static. Assuming the length of the plastic zone to be constant, the energy release rate may be deduced for the slope of the energy input against crack growth curve and the elastic energy stored in the beam at each moment. The calculated value of G_{IC} ($5.5\,kJ\,m^{-2}$) and the corresponding crack velocity ($30\,m\,sec^{-1}$) are derived from one single impact experiment.

High impact resistant polymers, such as some rubber-modified polyamides, generally show an extensive plasticity and fail by ductile tearing. In order to test specimens of reasonable size, it is necessary to use as a criterion the J-integral proposed by Rice. A load–deflection curve and the related crack length are needed in this procedure. Kobayashi [15] solved the problem by incorporating a depth-variable stock-block on an instrumented Charpy impact test machine. The length of the arrested crack is evaluated *a posteriori*. Successive points on the J-curve are then measured with one specimen each. A continuous measurement of the crack length could simplify this tedious work. It needs the actual crack growth to be followed. The gauge described in Section 2 cannot dissociate crack growth and plastic zone growth, because of its strain sensitivity.

According to measurements performed in our laboratory, the strain sensitivity of a polymer–graphite composite decreases when the volume content of graphite is lowered. Research is under way to obtain a high-resistivity layer almost insensitive to strain, based on an elastomeric matrix which may bear the high plastic deformation present at the crack tip without fracturing. That development might permit the determination of the J-curve in impact on one single specimen.

It must be pointed out, however, that the global behaviour of the through-thickness crack must be inferred from what occurs at the surface for each case presented here. Although troublesome, this drawback cannot be avoided.

Acknowledgement

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