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Measurements of transient high temperatures during the deformation of polymers

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A novel and simple technique for the measurement of the magnitude and spatial distribution of transient high temperatures is described. The technique is applied to the measurement of the temperatures produced during the deformation under impact of a range of polymers. It is found that very high temperatures (up to 700° C) can be obtained in materials which undergo catastrophic failure, and that the temperatures obtained are related to the material's thermal and mechanical properties.

1. Introduction

The deformation of materials at high rates of strain leads to the conversion of mechanical energy into heat. Depending on the mode of deformation this heating may be localized at crack tips or on shear planes or it may produce a more uniform bulk temperature rise. The temperature rises produced during these processes can be very high and for materials such as polymers or explosives can lead to thermal decomposition, or, in the case of metals, phase transformations. Because of the transient nature of this heating, measurement of the temperatures involved is difficult. In this paper we describe a simple technique based on the use of a heat sensitive film which gives the spatial distribution of heating. If used in conjuction with a technique such as high-speed photography, which provides temporal information about the deformation process, it is shown that reasonably accurate measurements of temperature can also be obtained. We have applied the technique to the deformation during impact of a wide range of polymers and the results are described in Section 3.

2. Experimental details

The technique used in this work was first suggested by Coffey and Jacobs [1]. It is based on the use of an acetate sheet coated with a sensitive layer which darkens on exposure to heat. The sheets are commercially produced for the production of transparencies for overhead projectors (3M Type 570). The sheets do not darken when exposed to light or infrared radiation out to 10 μm [1]. Film darkening does not occur when the sheets are torn or impacted with peak pressures up to at least 1 GPa, and the darkening produced by heating is stable at room temperatures for periods of at least a year. However if the film is subjected to high loading stresses at high strain rates ($\dot{\varepsilon} > 10^4$) catastrophic failure of this film backing may occur which will induce darkening of the sensitive layer. Details of the composition of the sensitive layer may be found in [2]. For very short duration heat pulses ($< 10^{-4}$ sec) the film colour obtained is yellowish-brown rather than black and the degree of darkening increases in intensity as the contact time is increased, until the film is fully blackened. This yellow-brown colouration is believed to be due to the reaction in the sensitive layer being unable to go to completion in the time available.

Film darkening is a function of both temperature and time so, in order to use the film to estimate the temperature achieved during deformation, one must know the time over which the deformation occurred and then refer to a set of calibration curves (darkening as a function of time and temperature) for the film. The film was calibrated by cutting out small circular discs (4 mm diameter) and attaching them sensitive side outwards to cylindrical steel rollers (5 mm diameter) using double-sided sellotape. The rollers were dropped down an accurately vertical tube onto a variable temperature hot plate whose surface temperature was measured using a chromel–alumel thermocouple. A simple catcher arm was used to prevent repeated impacts of the roller on the hot surface (Fig. 1). The contact time of the heat sensitive layer on the hot plate was varied by using rollers of different lengths and by incorporating polymeric layers as sandwiches between the film and steel roller. The contact time for the impact of a cylinder depends on the double transit time of the stress wave in the cylinder. With a "soft" polymeric layer added, the contact time is increased.

Contact times were measured by dropping the rollers onto a 1 mm thick piezoelectic crystal mounted on the hot-plate surface and measuring the voltage pulses produced. A range of contact times from 30 to 600 μsec was obtained and plots of the optical density of the film measured relative to a standard reference on a Joyce Mk III B microdensitometer are illustrated in Fig. 2. The film colouration was quite grainy (grains ~ 0.2 mm) and this gave rise to random variations in optical density about the mean value for any given temperature and time of contact. The extent of this graininess is illustrated by the error bars on the calibration curves (Fig. 2)

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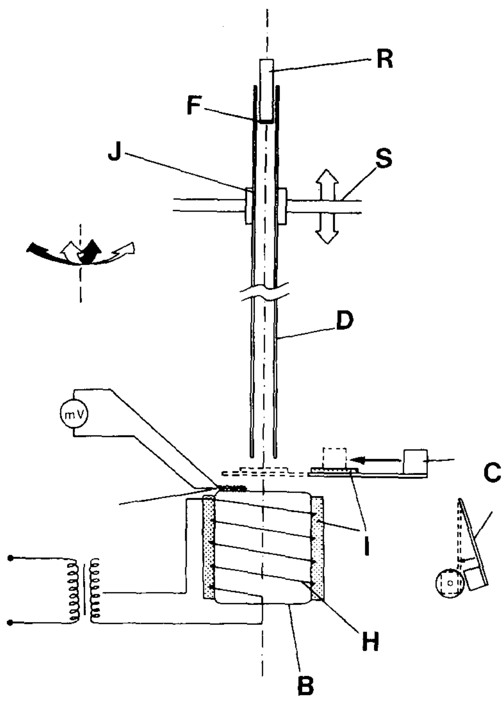

Figure 1 Apparatus for calibration of heat sensitive film. F disc of film, R cylindrical roller, S sliding support, J joint to adjust drop-tube perpendicular to heated surface, D drop-tube, C catcher arm to prevent bounces causing repeated impacts, I thermal insulation, H heater element, B steel block.

In order to use the calibration to obtain a temperature in an impact experiment, it was necessary to measure the time during which the events which caused the darkening took place. These measurements were made by using an Atomic Weapons Research Establishment designed high speed camera at a framing rate of 7 μsec per frame to photograph the processes occurring during impact. The experimental set up is fully described by Heavens and Field [3]. Basically the system allows the impact process to be viewed perpendicular to the plane of the impact by using toughened glass anvil material in a drop-weight machine (see Fig. 3). All the high speed photographic sequences illustrated in this paper have been obtained with this system. Experiments were carried out by placing a small square of the film surmounted by the sample on the glass anvil with the sensitive side in contact with the sample. Impacts were photographed using the high speed camera system, and the temporal information from the photographic sequences was used in conjunction with the calibration curves to obtain estimates of the temperatures reached during sample deformation.

3. Results

Experiments have been carried out on a range of polymers. The materials investigated were polymethylmethracalate (PMMA), polycarbonate (PC), polystyrene (PS), polytetrafluorethylene (PTFE), nylon 6, high density polyethylene (HDPE), polypropylene (PP) and polyvinylchloride (PVC). All the samples investigated were cylindrical and were approximately 5 mm in diameter and 1 mm high. Samples were produced either by slicing a cylindrical rod of the material or by punching discs from flat cast sheets.

Broadly speaking the materials investigated fell into two categories, those which failed by shear banding or cracking (PMMA, PC, PS, PTFE) and those in which mainly bulk plastic deformation was observed (PP, HDPE, nylon, PVC). Fig. 4 is a typical high speed photographic sequence for the latter type of behaviour. It shows a 5 mm diameter disc of PP (central dark region) which expands radially during the course of the impact. No cracking or shearing is observed. Fig. 5 illustrates the pattern left on the heat sensitive film after such an impact. Neglecting the no-flow zone at the centre the darkening is concentrated towards the central area. An indirect estimate of the temperature reached during the flow process can be made by using the high speed photographs to work out the amount of time during which plastic flow had been occurring at the boundary of the darkened region. For PP this time turns out to be 300 μsec. Since darkening is just occurring after 300 μsec the temperature is likely to be ~ 230° C (given by the lower point on the 330 μsec calibration curve). Similar calculations for the other bulk deforming polymers yield temperatures of ~ 400° C for nylon 6 and an upper limit of 200° C for HDPE. Fig. 6 shows a typical trace left on the film for a sample of nylon. It is clear from just a casual visual inspection of these traces that the temperature reached

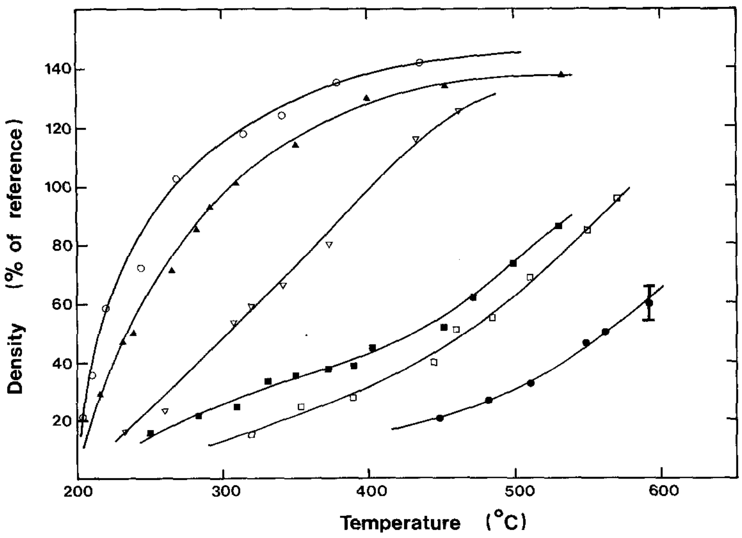

Figure 2 Calibration curves for film. Density is measured relative to a standard neutral density filter. Times represent the contact time of the film with the heated plate. Error bar shown is typical of all points. Contact times (μsec): (●) 30; (□) 50; (■) 140; (▽) 330; (▲) 440; (○) 600.

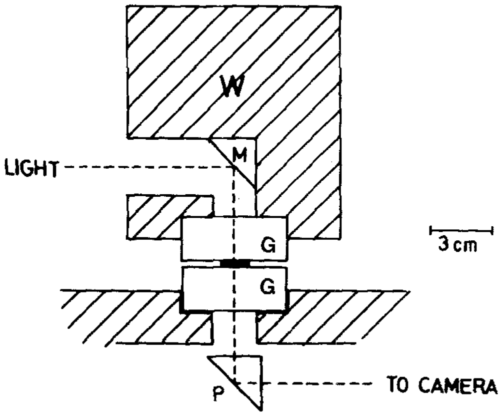

Figure 3 Experimental arrangement during impact: W, drop weight; G, glass blocks; M, mirror.

in the nylon is very much greater than that in PP which is in turn greater than that of HDPE, since *no* colouration is obtained with HDPE samples.

The behaviour of PVC was similar to that of the materials described above. Most of the heating took place during the bulk plastic stage of deformation and the trace left on the film was similar to that for nylon. Although a ring type fracture of the material did occur at a strain of ~0.4 producing a "tyre" around the main bulk of the sample this did not leave any trace on the heat sensitive film. The temperature reached during bulk deformation was estimated to be 450 ± 100° C and was high enough to produce severe distortion of the sample and heating of the film backing on several occasions.

PC, PMMA, PS and PTFE all deformed first by bulk plastic flow and then with the production of "parallel" and "perpendicular" cracks followed by general random cracking. The word "cracks" is used here because when a specimen is examined after impact it separates along these visible lines. However, both fracture and shear banding can be found with PC, PMMA and PS and with some of the line features shear failure almost certainly occurred initially. The sequence of cracking is illustrated in the drawings of Fig. 7. The deformation could be halted at any of the stages illustrated by judicious variation of the height from which the weight was dropped on the sample. Use of the heat sensitive film in conjunction with variations in the drop height of the weight showed that the amount of film darkening was the same for each set of

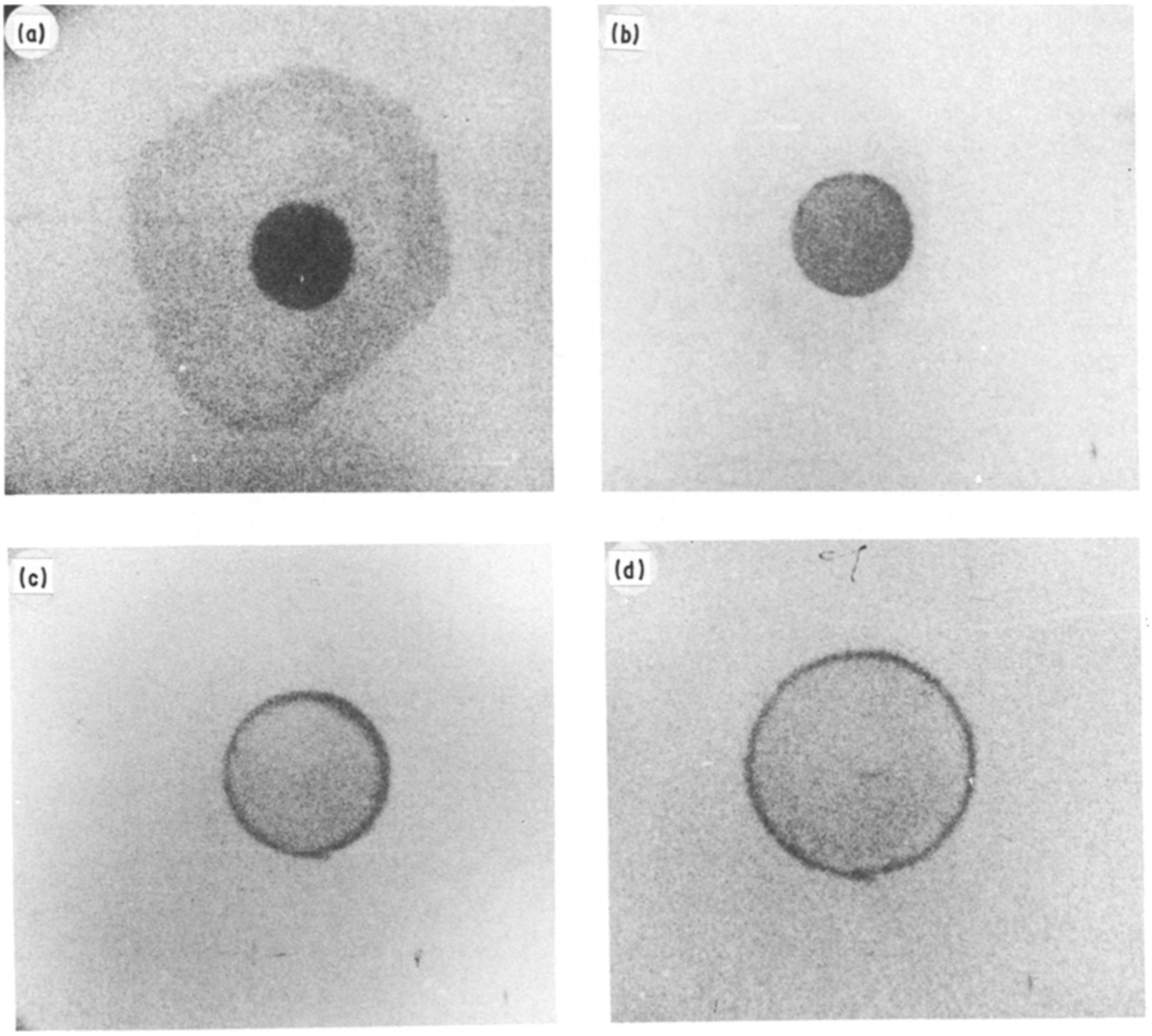

Figure 4 High speed photographic sequence of impact on a 5 mm diameter, 1 mm high PP disc. Times after frame (a); (b), 168 μsec; (c) 268 μsec; (d) 335 μsec; (e) 378 μsec; (f) 402 μsec. The diffuse shading around the disc in frame (a) is due to overwriting on the film.

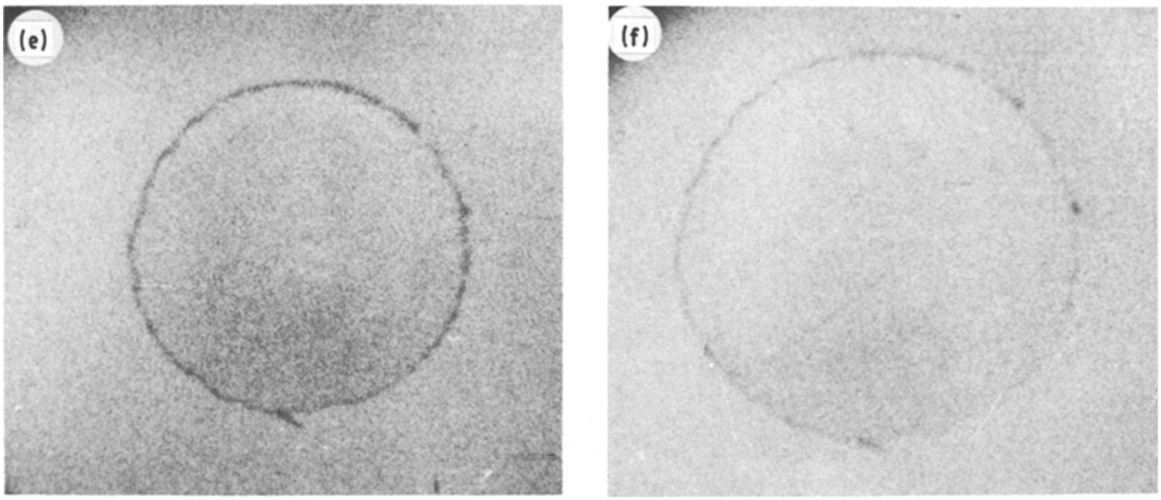

Figure 4 Continued.

cracks and that the darkening took place at the intersection of cracks with the surface.

The extent of the bulk deformation and timescale of the cracking were different for the different materials. Fig. 8 illustrates this behaviour for PS. Bulk deformation occurs in frames a and b and then at a strain of 0.003 "parallel" cracks are produced sequentially across the specimen, frames c–g. These cracks are then connected by "perpendicular" cracks, and eventually the sample becomes covered in a network of cracks (frame h). The timespan from first production of the "parallel" cracks to the final multiple cracking is ~250 μsec. Fig. 9 illustrates a similar sequence for PC. Bulk deformation from frames a–e then at a strain of 1.1 cracking occurs. However, in this case the failure of the sample is much more rapid and cracking is complete by frame i, a timespan of 25 μsec. PMMA behaves in a similar manner, cracking occurring when the strain is ~0.04 but the duration of cracking is now ~400 μsec. Table I summarizes the behaviour of these polymers for loading of similar sized samples by impacts with the same weight dropped from approximately the same height (5 kg mass from ~0.5 m).

Of the polymers which cracked during deformation, only very slight darkening was produced when the impact energy was restricted so as to prevent cracking whilst still allowing some plastic deformation to occur. This implies that the temperature achieved during bulk deformation of these materials cannot be much more than 200° C. Fig. 10 illustrates the traces obtained for a crack running through a sample of PMMA. It can be seen that the dark line on the film follows the course of the crack. Measurement of the optical density of the trace together with an estimate of the time of heating yield a temperature of 530 ± 50° C. This is in very good agreement with the value of 500° C quoted by Fuller *et al.* [4] for cracks in PMMA moving at similar velocities but measured by a totally independent technique.

Fig. 11 illustrates the pattern left on the film after the failure of a sample of PC. It is clear that the darkening is irregular and in some areas very intense. Measurement of the optical density of the most intense areas with an estimated heating time of 30 μsec yield peak temperatures ~700° C. For materials which undergo catastrophic failure (PC) the estimated heating time is typically 30 to 50 μsec.

With materials such as PS, PTFE and PMMA, the *total* deformation process could take several hundred microseconds. However, the high speed photographs

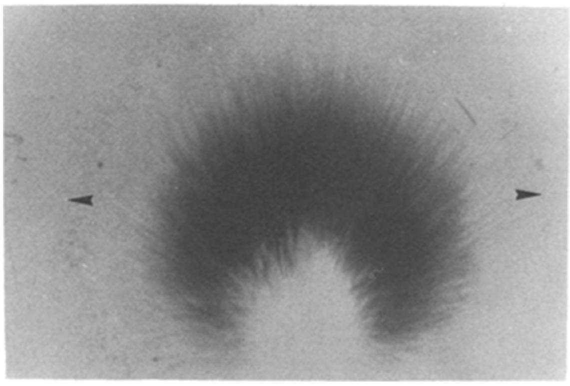

Figure 5 Pattern left on film after impact on a PP disc. Arrows indicate final diameter of the disc. Original in colour.

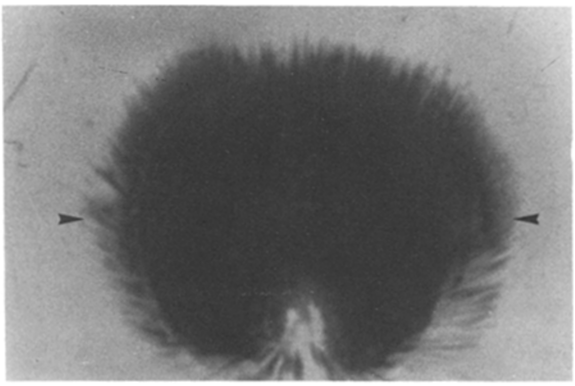

Figure 6 Pattern left on film after impact on a Nylon 6 disc. Arrows indicate final diameter of the disc. Original in colour.

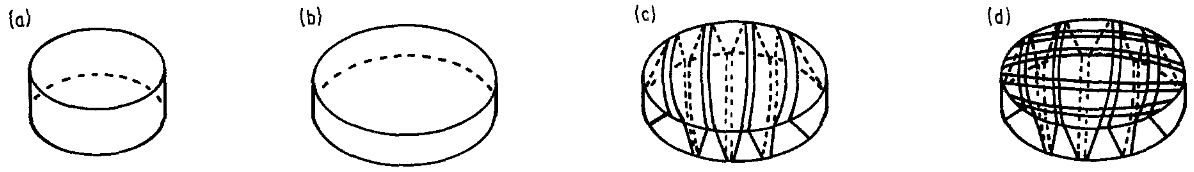

Figure 7 Sketches of shear and cracking sequences observed during impact on PC, PS and PMMA. The sequence was similar for each of the materials although the duration and time between stages differed. (a) represents initial undeformed disc, (b) disc at completion of plastic flow stage, (c) failure by "parallel shear-cracks", (d) continued failure by "perpendicular shear-cracks".

showed that stages of this deformation could take place very rapidly with a set of cracks taking 20 to 30 μsec to form. Fig. 7, frames b–d, is an example of a set of parallel cracks in PS which took ~ 20 μsec to form.

These times are reasonable estimates of the duration of the high temperature regions since measurements by Fuller *et al.* [4] on PMMA and PS, using an infrared detector to observe the temperature rise after the passage of a crack, yield values of ~ 10 μsec for the rise time of the temperature pulse in PMMA and PS for cracks travelling at similar velocities to those we have observed. Taking 1 μm as being a typical thickness of the plastic zone ahead of the crack tip (2 × typical craze thickness) and assuming the temperature drops by diffusion of heat into the bulk material we find that the time taken for the temperature to drop to < 200 from 500° C would be 20 μsec using $T = T_0 \operatorname{erf} [R/(4kt)^{1/2}]$, [5] where T is the temperature after time t, T_0 is the initial temperature rise in a hot region of thickness R and k is the thermal diffusivity which for a polymer is typically $10^{-7}\,\mathrm{m^2\,sec^{-1}}$. The total heating time of the film would therefore be typically 30 μsec. The actual peak temperatures achieved in the polymer may be even higher than the values quoted in Table I, since our estimates of duration of film heating includes the time taken to reach the peak as well as to drop to below values which will cause no further film darkening.

4. Discussion

Our experimental results indicate that in polymers which are subject to cracking very high temperatures are reached in the crack zone. These temperatures are higher than could be achieved by conversion of the energy of the falling weight directly into heat throughout the sample. Assuming no energy losses and complete conversion of mechanical energy into heat, a temperature rise of 600° C would be expected in our samples taking a value of $2\,\mathrm{kJ\,kg^{-1}\,°C^{-1}}$ as a typical specific heat. Differential scanning calorimetry (DSC) experiments over the range 50 to 350° C yielded values of specific heat which varied in an approximately linear value from 1.3 at 50° C to $2.4\,\mathrm{kJ\,kg^{-1}\,°C^{-1}}$ at 350° C for PC and 1.9 at 50° C to $2.1\,\mathrm{kJ\,kg^{-1}\,°C^{-1}}$ at its melting point (130° C) for PP. The specific heat of PMMA has been found to be relatively constant at $2.9\,\mathrm{kJ\,kg^{-1}\,°C^{-1}}$ until decomposition occurred at ~ 360° C.

However, the assumption of complete conversion of the mechanical energy of the falling weight into heat in the sample is rather unrealistic. Typically 20 to 30% of the impact energy will be used in elastic energy in the system which causes the weight to recoil. Smaller portions of the impact energy probably totalling another 10% will be used in plastic distortion of the machine components and emitted as sound waves. Thus one would expect at most 2/3 of the impact energy to be deposited in the sample, yielding a maximum expected temperature rise of 400° C which is in agreement with our results (since temperature rise of approximately 450° C is in fact the maximum that is observed in those materials which deform in our equipment solely by bulk plastic deformation).

If one assumes that the temperature produced at the crack tip is due to the release of elastic stored energy one would expect those materials in which the failure was most catastrophic, i.e. the energy release rate was highest, to produce the highet temperatures. Inspection of Table I will confirm that this is indeed the case. However the release rate will not be the only factor in determining potential for production of high temperatures. Consideration must also be given to the magnitude of the stored energy. This will be determined by the stress at which failure occurs and the failure strain less the residual strain ($\varepsilon_f - \varepsilon_r$). A measure

TABLE I

	A^* natural strain	Time of cracking	Max temperature of plastic deformation	Max observed crack temperature	Latent heat† (kJ kg)
HDPE	0.9	–	$\ll 200$	–	120
PP	> 2.5	–	230	–	95
NYLON 6	1.0	–	400	–	130
PVC	0.7	–	450	–	60
PS	0.01	250	< 200	550	–
PMMA	0.04	400	< 200	530	
PTFE	0.25	150	< 200	600	37
PC	1.1	20	< 200	700	7.2

A^* represents the strain at which cracking occurs or the observed maximum plastic strain.
†obtained on a Mettler DSC-30.

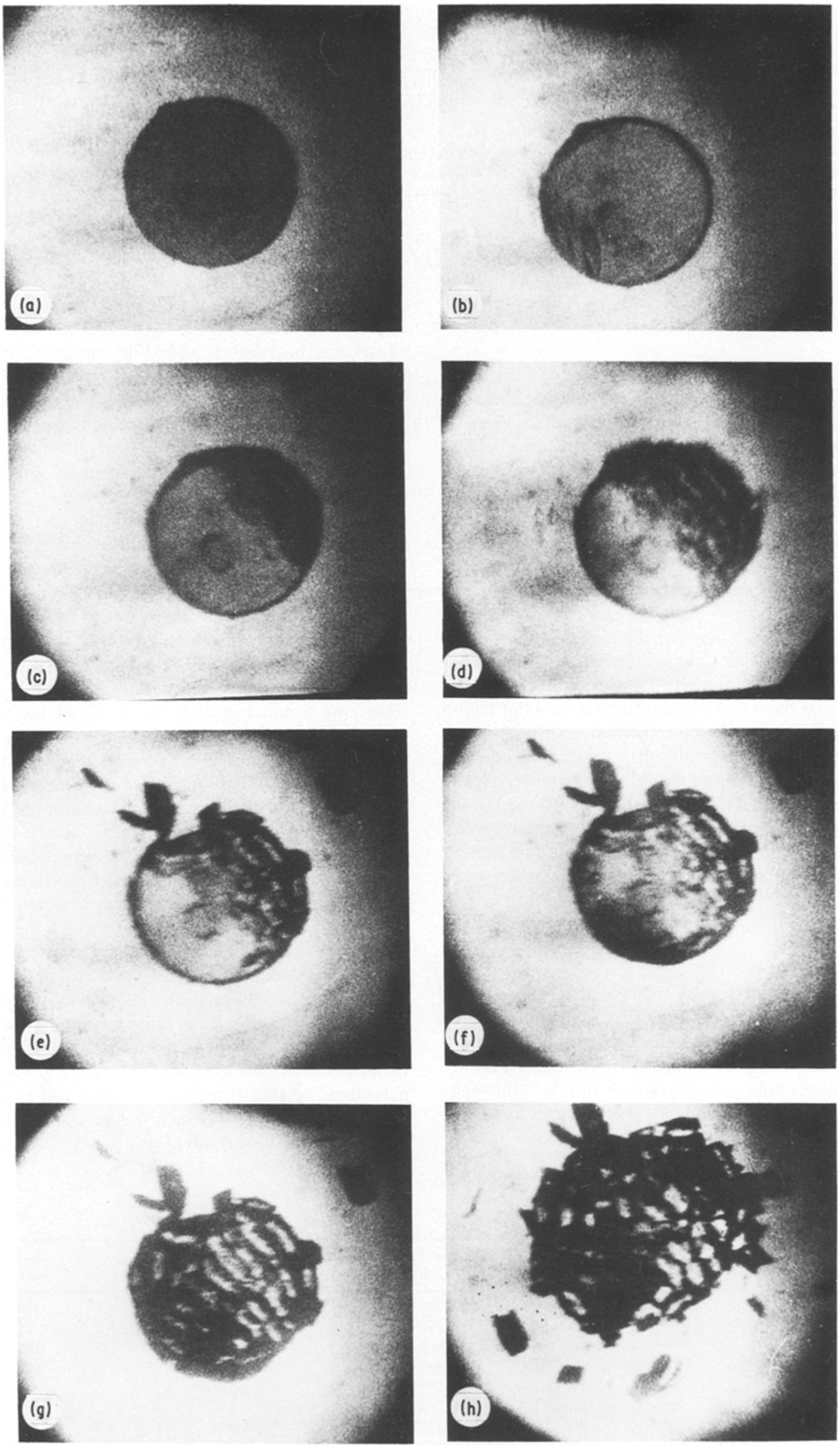

Figure 8 Impact on a 5 mm diameter, 1 mm high disc of PS. Times after frame (a): (b), 35 μsec; (c) 42 μsec; (d) 49 μsec; (e) 189 μsec; (f) 196 μsec; (g) 203 μsec; (h) 259 μsec.

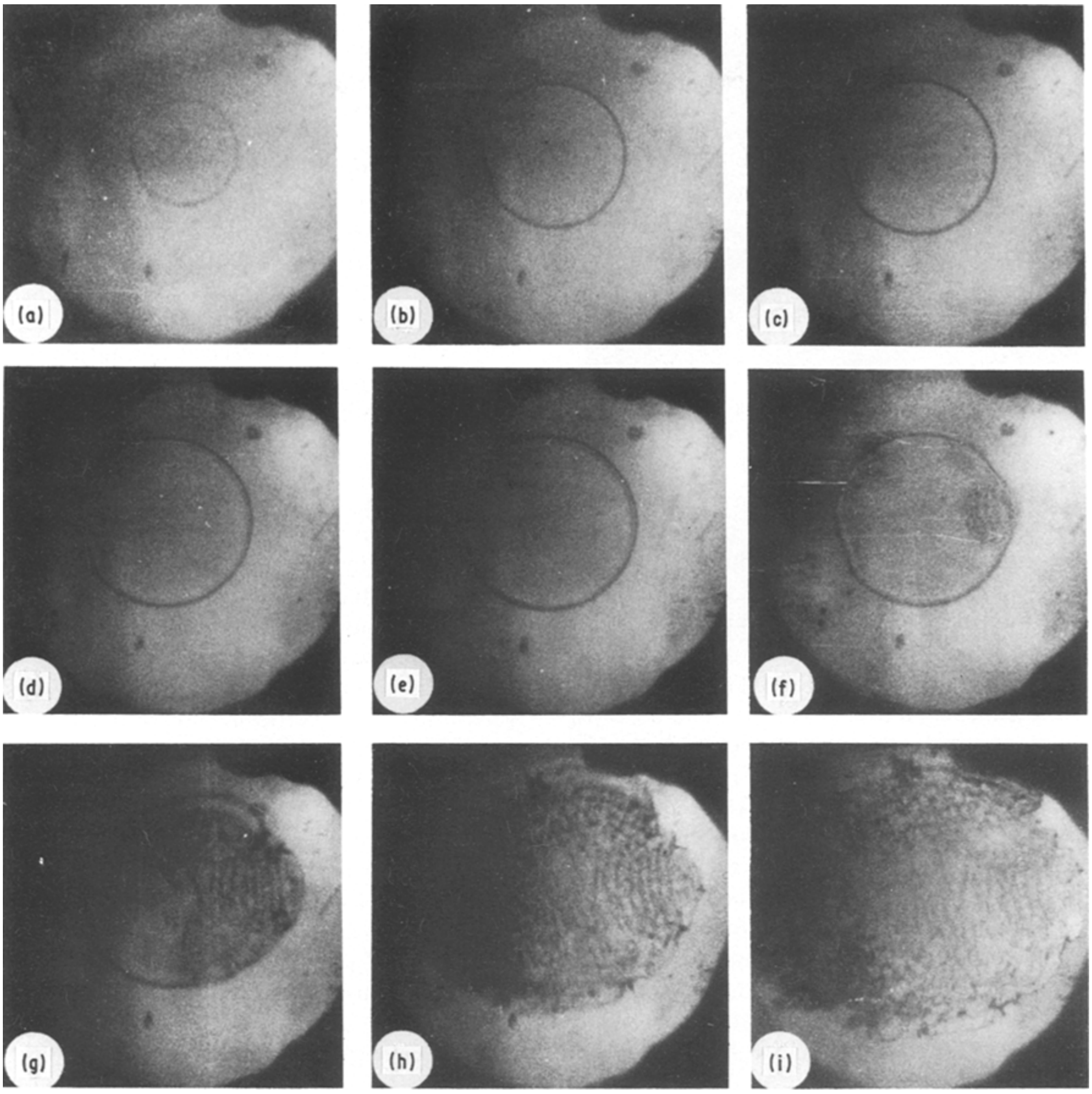

Figure 9 Impact on a 5 mm diameter, 1 mm high disc of PC. Times after frame (a): (b) 161 μsec; (c) 188 μsec; (d) 241 μsec; (e) 268 μsec; (f) 275 μsec; (g) 281 μsec; (h) 288 μsec; (i) 295 μsec.

of the residual strain may be obtained by carrying out experiments to just less than ε_f and observing the strain in the sample after the experiment. Information on the failure stress and strain can be obtained from standard compressional stress–stress curves. Unfortunately this information is not readily available for the materials of interest at the appropriate strain rates ($\dot{\varepsilon} \sim 10^2\,\text{sec}^{-1}$). Work on the production of stress–strain curves at strain rates of 10^2 to 10^3 to high total strains is currently in progress in this laboratory.

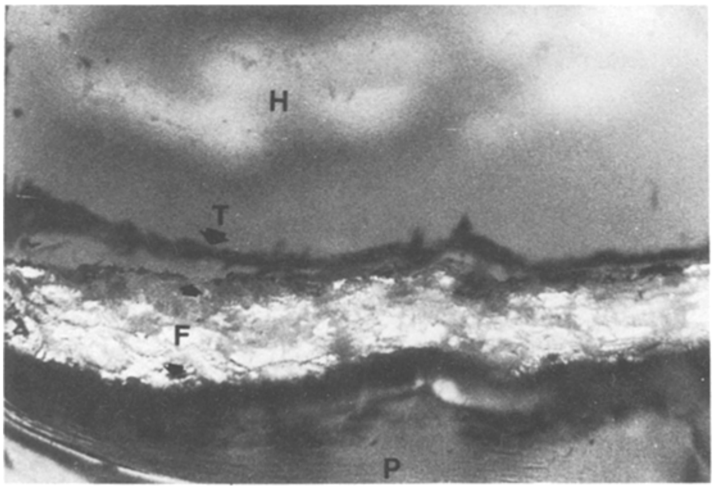

Figure 10 Trace obtained from crack running through PMMA. F is the fracture surface, T the trace left on the film, H the film and P the remaining portion of the PMMA on the film surface. Original in colour.

Figure 11 Pattern left on film after catastrophic failure of PC. The PC is still in contact with the film and is here viewed through the film. Original in colour.

However Table I does reveal that those materials which fracture at higher strains produce higher temperatures.

The prediction of a high yield stress leading to higher temperature during failure is supported by the work of Krishna Mohan and Field [6] who carried out impact experiments on explosive layers and observed that those materials which had the highest yield strengths were most likely to ignite in an impact experiment. However, in order to make a quantitative comparison between the estimates of temperatures produced during deformation and the mechanical properties of the materials it will be necessary to produce a set of stress–strain curves for the polymers at strain rates appropriate to our impact conditions.

Another factor which can limit the potential for high temperature production is the latent heat of any phase transformation that may occur before a high temperature is reached. The most common will be melting or softening of the material and one finds that those materials which have a definite melting range with a high latent heat produce the lower temperatures. Measurements of the latent heats made on the materials using a Mettler DSC 30 are included in Table I.

It is not too surprising that some polymers reach high local temperatures (well above their softening temperatures) during rapid deformation. Polymers in general have relatively low thermal capacities and conductivities and still retain a high viscosity even when the softening temperatures are exceeded. High temperatures are favoured, particularly in those which fail at high stresses such as, for example, PC. In contrast, polymers such as HDPE and PP have much lower yield stresses and noticeably higher latent heats (see Table I). Both of these factors favour bulk, rather than localized, deformation and the attainment of lower temperatures.

It is clear that there are a large number of factors which determine how high a temperature is likely to be produced in a material during impact and that prediction of the likely temperature rises is a difficult task. The method of direct measurement outlined in this paper does however produce a useful experiment window onto these short timescale temperature excursions.

The initial use of this film technique by Coffey and Jacobs [1] was for studies of "hot-spots" produced during the mechanical deformation of explosives. The technique is certainly useful for giving the location and distribution of "hot-spots". It has also helped us confirm earlier research which suggested that high temperatures could be reached during the deformation of some polymers [3] and that the presence of these polymers in explosives could cause sensitization [7, 8].

5. Conclusions

The use of heat sensitive film has been demonstrated to give spatially resolved information on the temperatures produced during the deformation of polymers. It is a simple technique which, when calibrated, gives quantitative results or, uncalibrated, can be used to provide comparative information on a range of materials. It is of particular use with soft materials, such as polymers, which do not cause damage to the backing layer during deformation. The present work has considerably extended the range of the technique from the first proposals of Coffey and Jacobs [1] who only undertook a very limited calibration and did not use cameras to follow the progress of the deformation. The technique should have wide application in the many areas of research in which transient temperatures are produced.

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