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14. ABSTRACT

This report outlines the mechanical, optical and microscopic techniques available in the Physics and Chemistry of Solids (PCS) Fracture and Shock Physics group, Cavendish Laboratory, Cambridge, for studying the impact and high strain rate properties of materials. Results are presented for high strain rate stress-strain response of PBX, with effect of particle size and temperature. Results are presented on studies of ultrafine and conventional PETN and RDX.

15. SUBJECT TERMS
EOARD, Energetic Materials, Composites, deformation, PBX, dynamic properties, Hopkinson bar

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13. ABSTRACT (Maximum 200 Words)

During the year, two interim reports were issued. The first (dated June 2002) gave a comprehensive overview of the techniques available in our laboratory for the study of the dynamic mechanical properties and detonation of energetic materials. It also included some preliminary data on the effect of particle size on the high strain rate response in compression of certain ammonium perchlorate (AP) filled PBXs at various temperatures. The second report (dated October 2002) included data from a more thorough study of the particle size effect in these materials. This report analyses the dependence of flow stress on grain size.

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Deformation, PBXs, Dynamic Properties, Hopkinson Bar.			16. PRICE CODE

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1. SUMMARY OF FIRST YEAR'S WORK

During this past calendar year, two interim reports were issued outlining work on this contract. The first (dated June 2002) gave a comprehensive overview of the techniques available in our laboratory for the study of the dynamic mechanical properties and detonation of energetic materials. It also included some preliminary data on the effect of particle size on the high strain rate response in compression of certain ammonium perchlorate (AP) filled PBXs at various temperatures. The second report (dated October 2002) included data from a more thorough study of the particle size effect in these materials. In particular we noted that the effect of particle size on high strain rate flow stress was clearer at a low temperature (-60 °C) as compared to room temperature (see figure 1).

In that second report, we found that the data on flow stress as a function of particle size from the materials that were available to us were best fitted by a power-law of −0.125 (figure 1). However, as can be seen this line of best fit does not go through the data points and their error bars very well (regression coefficient = 0.956). Reexamination of the data showed that the data for the three smallest particle size materials fitted a straight line plot to a much higher level of accuracy (regression coefficient = 0.992) if the flow stress were plotted against the square root of the particle size (figure 2). The data point for the largest particle size material does not lie on this line. However, it should be noticed that the gradient of the line is such that it would predict a negative flow stress for material with this particle size. This is clearly not possible. So presumably the correlation of flow stress with the square root of particle size breaks down at some critical value of particle size. If we had access to more PBX materials (say 5 or 6) with a finer gradation of particle sizes, this point could be checked out more accurately.

2. FUTURE WORK

(a) Cast samples will be obtained to give more consistent results.
(b) PBXs with a large number of different particle sizes will be obtained to check the relation of flow stress with particle size more thoroughly.
(c) Poisson ratios will be measured to check whether the material is incompressible.
(d) High speed photography combined with digital speckle photography will be used to examine the specimens during deformation in the SHPB to check for uniformity of deformation.
(e) ESEM micrographs will be taken before and after deformation to examine damage mechanisms.
(f) The effect of ageing (both natural and accelerated) on high rate of strain stress-strain properties will be examined.

(g) The effect of different energetic particle loadings on the strength of PBXs will be examined.
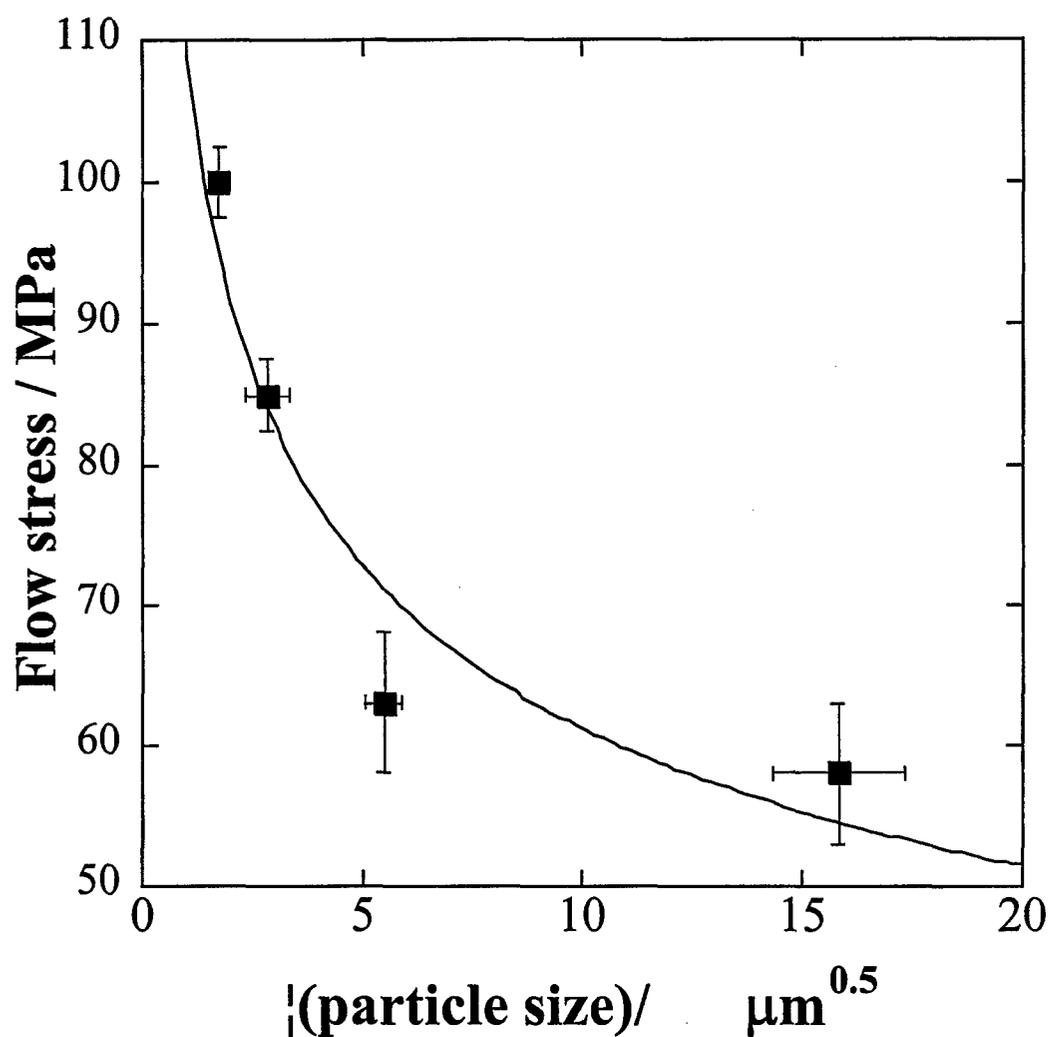

Figure 1. Plot of flow stress versus square root of particle size for all the AP/HTPB compositions studied at strain rate of ca. 4000 s^{-1} and a temperature of -60 °C. The data are best fitted by a power law of exponent -0.25.
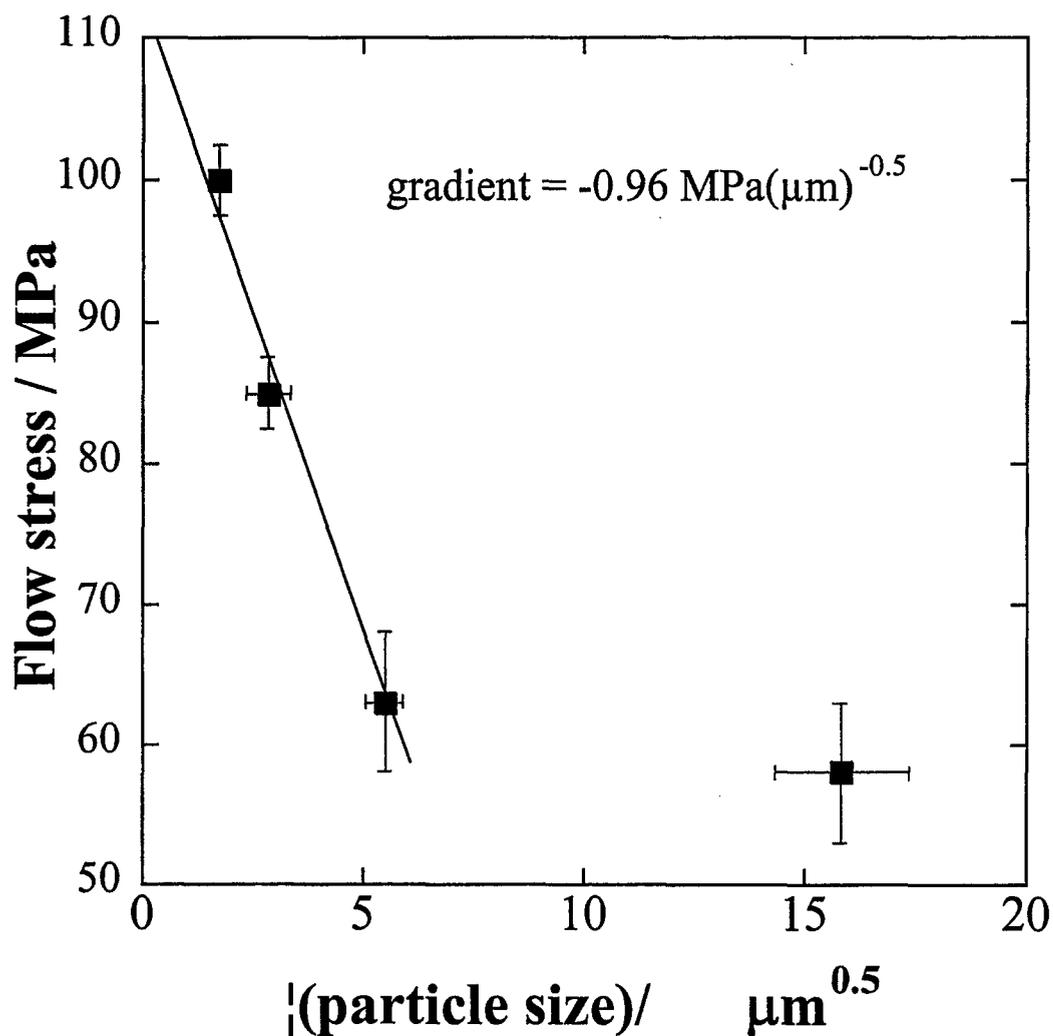
Figure 2. Replot of flow stress versus square root of particle size for all the AP/HTPB compositions studied at strain rate of ca. 4000 s^{-1} and a temperature of –60 °C. The data for the three smallest particle size materials are best fitted by a straight line of gradient –0.96.

The Contractor, hereby declares that, to the best of its knowledge and belief, the technical data delivered herewith under Contract No. F61775-01-WE056 is complete, accurate, and complies with all requirements of the contract.

DATE: 12 . 12 . 02

Professor J E Field
Principal Investigator

I certify that there were no subject inventions to declare as defined in FAR 52.227-18 during the performance of this contract.

DATE: 12 . 12 . 02

Professor J E Field
Principal Investigator

REPORT DOCUMENTATION PAGE

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13. ABSTRACT (Maximum 200 Words)

In our first Interim Report, we outlined the mechanical, optical and microscopical techniques available in the Physics and Chemistry of Solids (PCS) Fracture and Shock Physics Group, Cavendish Laboratory, Cambridge for studying the impact and high strain rate properties of inert and energetic materials. Preliminary results were presented on the high strain rate ($3,500$ s^{-1}) stress-strain response of a PBX consisting of an HTPB binder and AP particles of a range of four different sizes. We have now made a more thorough study of the effect of particle size and temperature on the flow properties. The results confirm that lowering the temperature magnifies the effect of particle size. The report also describes the visit of Professor R W Armstrong and discusses ways of further strengthening the work with EGLIN.

14. SUBJECT TERMS Deformation, PBXs, Dynamic Properties, Hopkinson Bar.			15. NUMBER OF PAGES 12
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1. More research on the Effect of Particle Size on High Strain Rate Properties of an AP/HTPB at Various Temperatures

In earlier research we found that lowering the temperature made the effect of particle size on the flow stress of an AP/HTPB PBX more prominent (Balzer 2001). We therefore performed a more thorough study of the high strain rate properties of this material at both room temperature (22 °C) and at –60 °C. The AP/HTPB PBX used consisted of 66% ammonium perchlorate and 33% HTPB by mass. This was available in four different grain sizes: M1=3μm, M2=8μm, M3=30μm and M4=200-300μm. The room temperature experiments were performed using our low impedance magnesium alloy Hopkinson bar (see our first report on this contract; Walley et al. 2002). The low temperature study was carried out using our Inconel bar system (the mechanical impedance of Inconel is only a weak function of temperature, so the temperature gradient does not distort the elastic wave pulse travelling through it (Kandasamy and Brar 1994, 1995). Cooling was performed by surrounding the ends of the bars with a chamber into which helium gas was passed that had been cooled using liquid nitrogen. The temperature was monitored using chromel-alumel thermocouples.

The PBXs were available in blocks of typical size 10cm by 5cm by 2cm. In order to prepare the samples, strips of PBX approximately 2mm thick, were cut from the block using razor blades mounted 2mm apart. The specimens were taken from these strips using a vertical punch of 6mm diameter. The larger grain size materials were easier to prepare than the smaller grain ones: the cutting instrument moved through the samples much more readily, and the quality of the ends, and in particular the sides, was higher.

The results of this study are presented in figures 1-11. At least three experiments were performed for each grain size at each temperature. It can be seen from figures 1-5 that at room temperature all the compositions strain-hardened, the stresses rising from *ca.* 2 to *ca.* 10 MPa. The strain rates of each curve are tabulated in each figure where original data are presented, and as averages where the average stress-strain curves are given. The fall in stress at the end of each curve represents the unloading of the specimen rather than its failure or fracture. It can be seen from figure 5 that the material containing the largest AP particle size supported the lowest stresses. The dotted lines either side of the solid line in each case represents the standard deviation of the data.

The stress-strain curves at –60°C are different in shape: all the materials flow at constant stress varying between *ca.* 50 MPa for the largest particle size material and *ca.* 100 MPa for the smallest particle size material. Figures

10 and 11 confirm that lowering the temperature magnifies the effect of particle size: the curves are clearly separated by particle size.

References

Balzer, J.E. (2001) "Low-level impact loading of explosives", PhD thesis, Univ. of Cambridge

Kandasamy, R. and Brar, N.S. (1994) "Flow stress and material model study at high strain rate and low temperature" in "High Pressure Science and Technology 1993", ed. S.C. Schmidt, J.W. Shaner, G.A. Samara and M. Ross, publ. New York, American Institute of Physics: pp. 1031-1034.

Kandasamy, R. and Brar, N.S. (1995) "Low temperature and strain sensitivity of steel and aluminum" *Exper. Mech.* **35** 119-123

Walley, S.M., Siviour, C.R., Balzer, J.E., Radford, D.D., Gifford, M.J., Marrah, R., Proud, W.G. and Field, J.E. (2002) "Dynamic deformation properties of energetic composite materials", PCS, Cavendish Laboratory, University of Cambridge, report no. SP1107

M1 Room Temperature
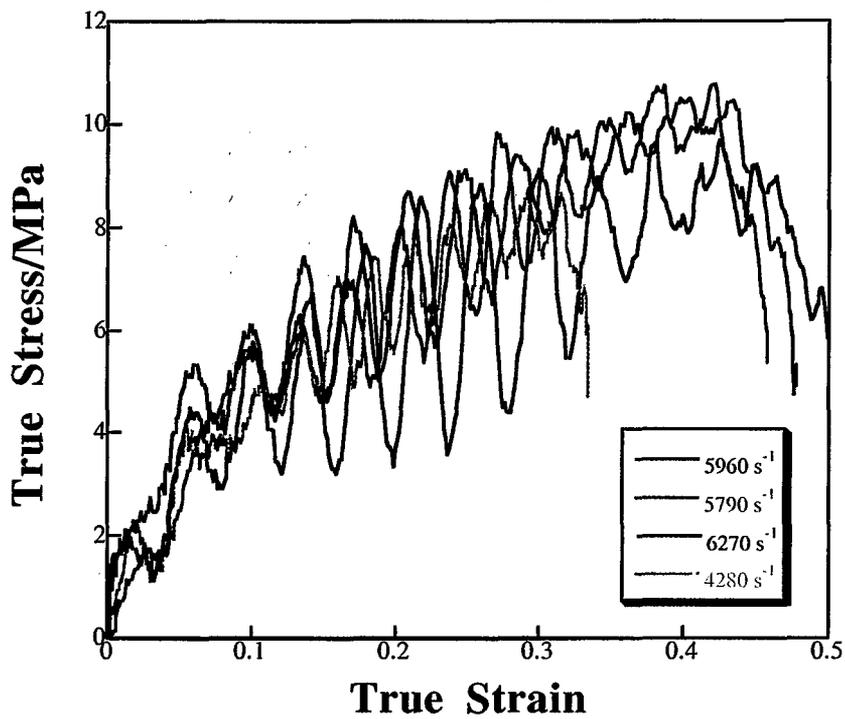
Figure 1. SHPB stress-strain curves for 3 μm AP/HTPB at 22 °C.

M2 Room Temperature
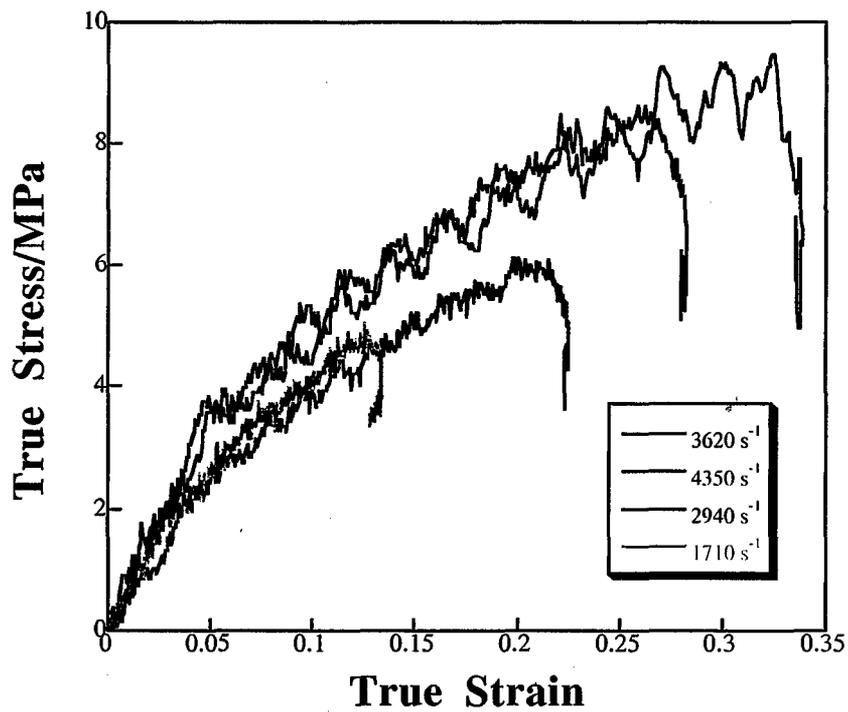
Figure 2. SHPB stress-strain curves for 8 μm AP/HTPB at 22 °C.

M3 Room Temperature
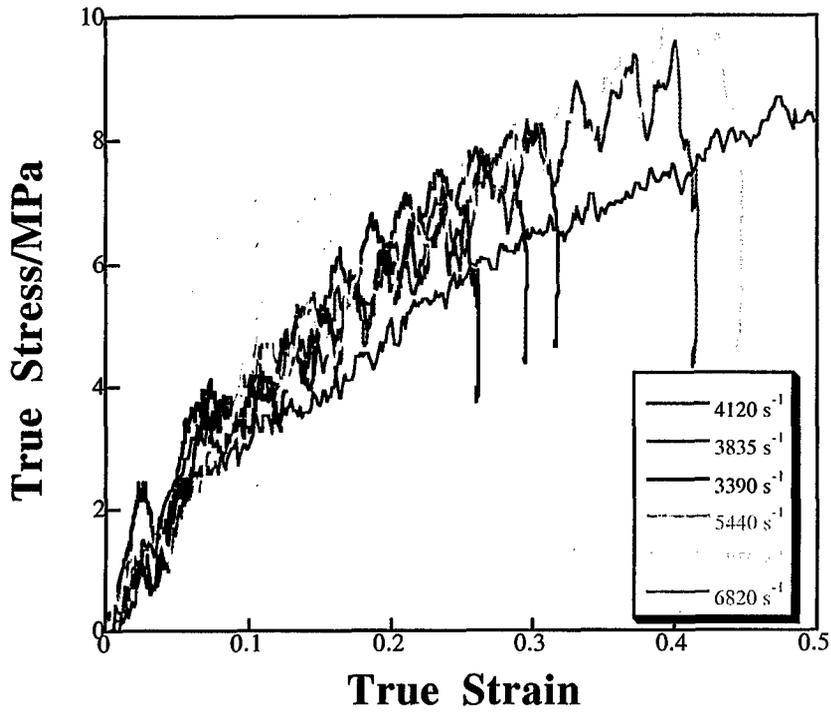
Figure 3. SHPB stress-strain curves for $30\mu m$ AP/HTPB at 22 °C.

M4 Room Temperature
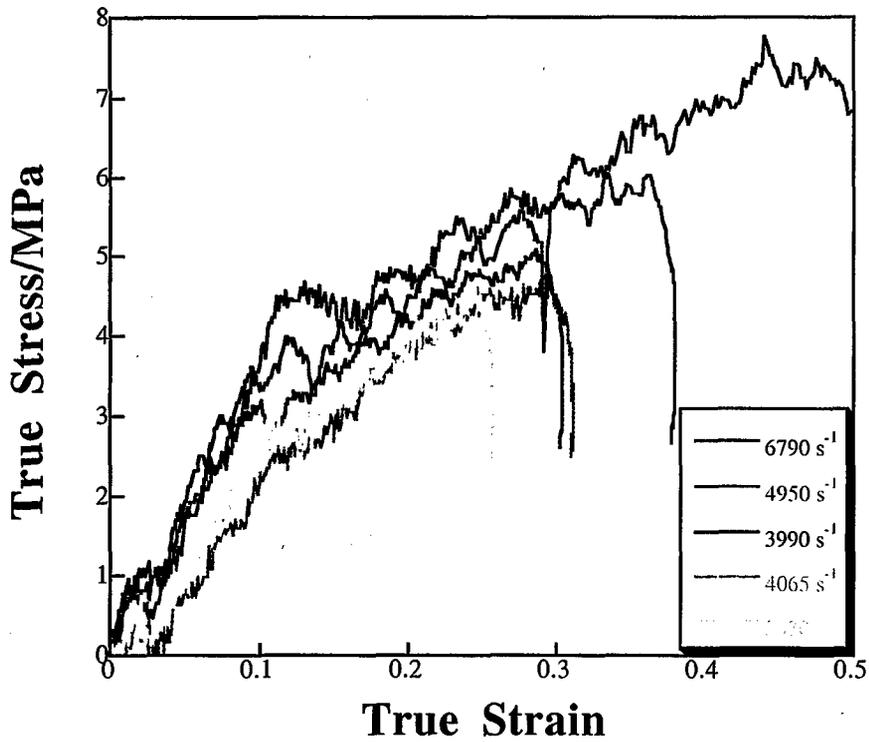
Figure 4. SHPB stress-strain curves for 200-$300\mu m$ AP/HTPB at 22 °C.

Room Temperature
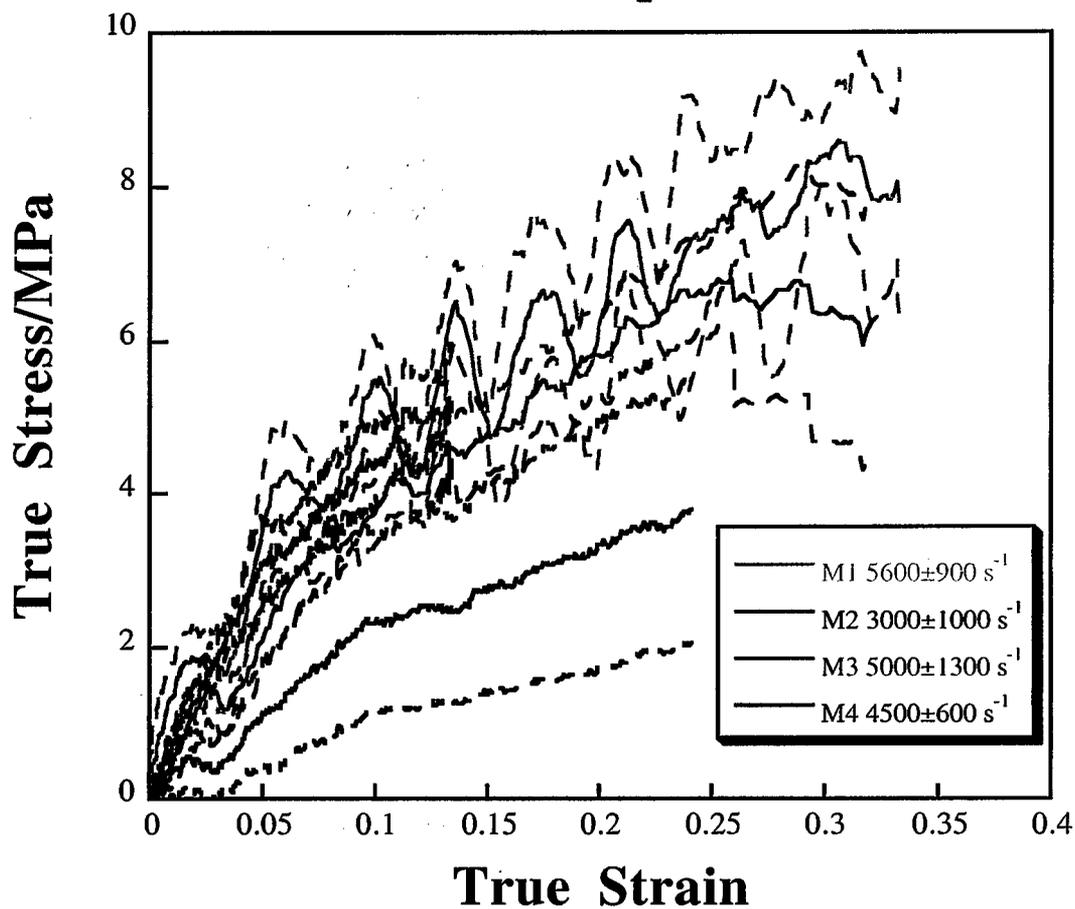
Figure 5. *Average SHPB stress-strain curves for all the AP/HTPB compositions at 22 °C. Dotted lines indicate standard deviation of the data.*

M1 -60°C
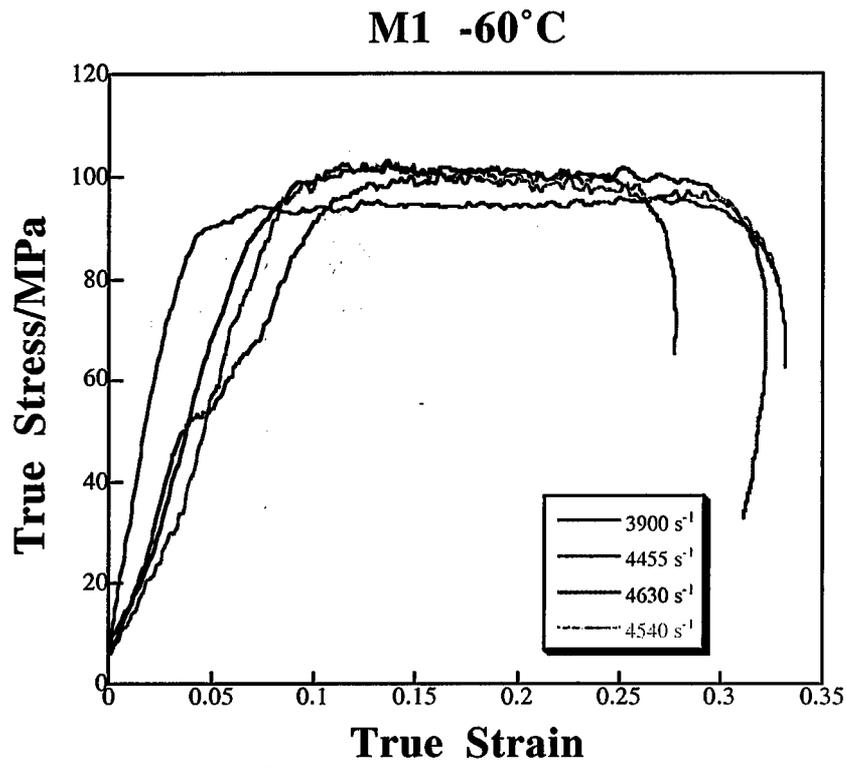
Figure 6. SHPB stress-strain curves for 3 μm AP/HTPB at -60 °C.

M2 -60°C
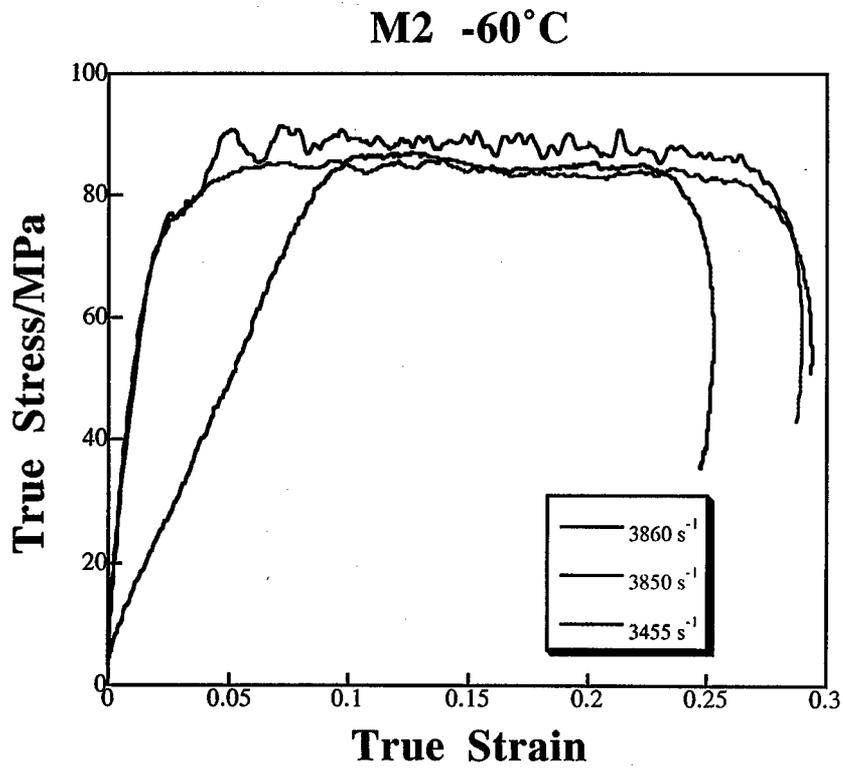
Figure 7. SHPB stress-strain curves for 8 μm AP/HTPB at -60 °C.

M3 -60°C
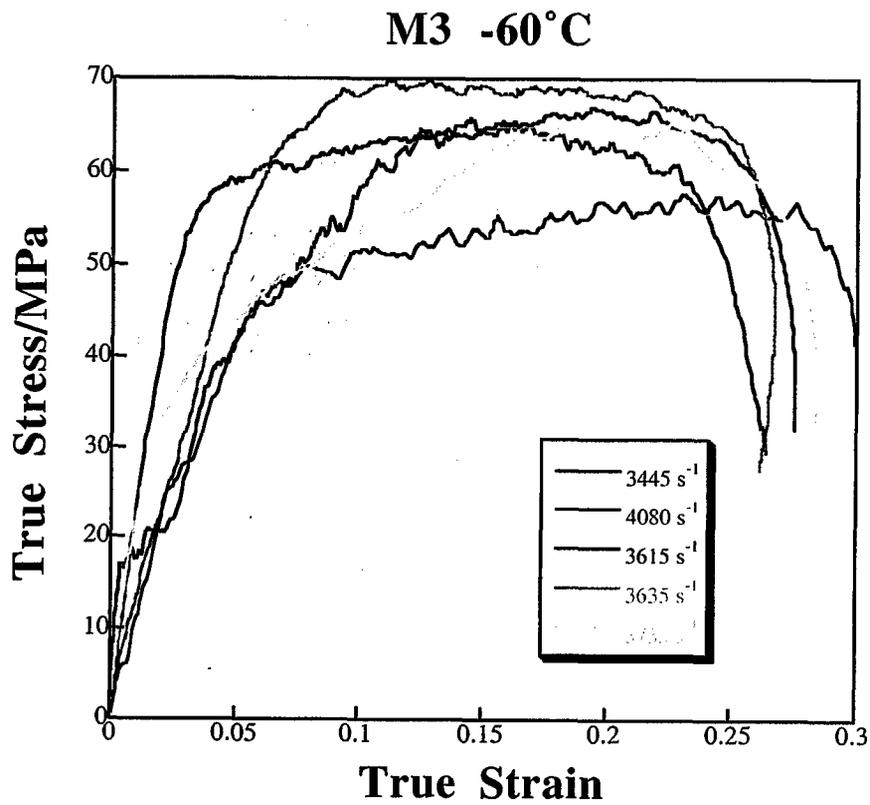
Figure 8. SHPB stress-strain curves for 30μm AP/HTPB at -60 °C.

M4 -60°C
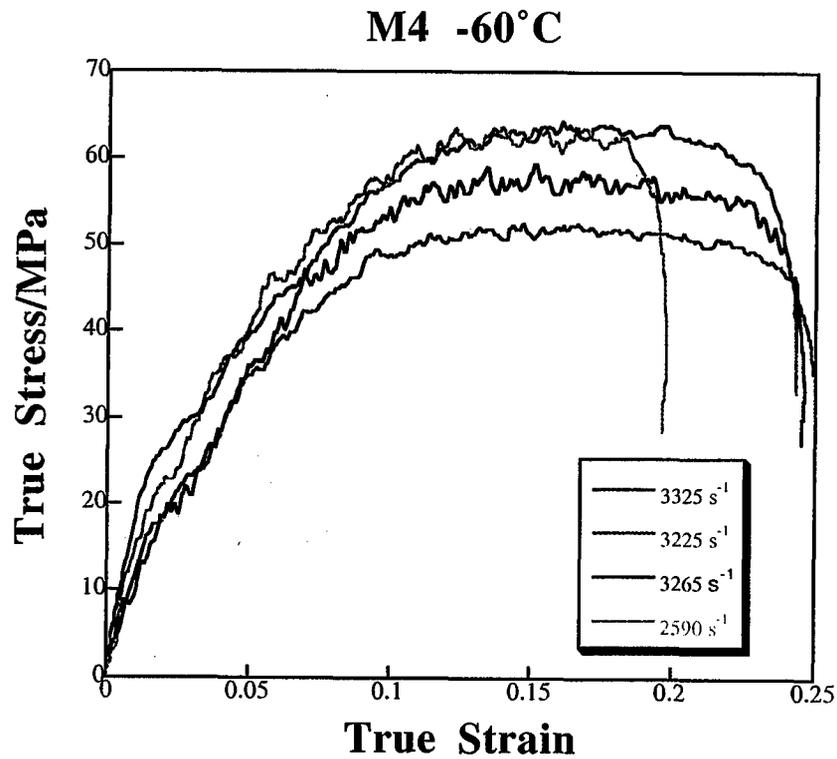
Figure 9. SHPB stress-strain curves for 200-300μm AP/HTPB at -60 °C.

-60 °C
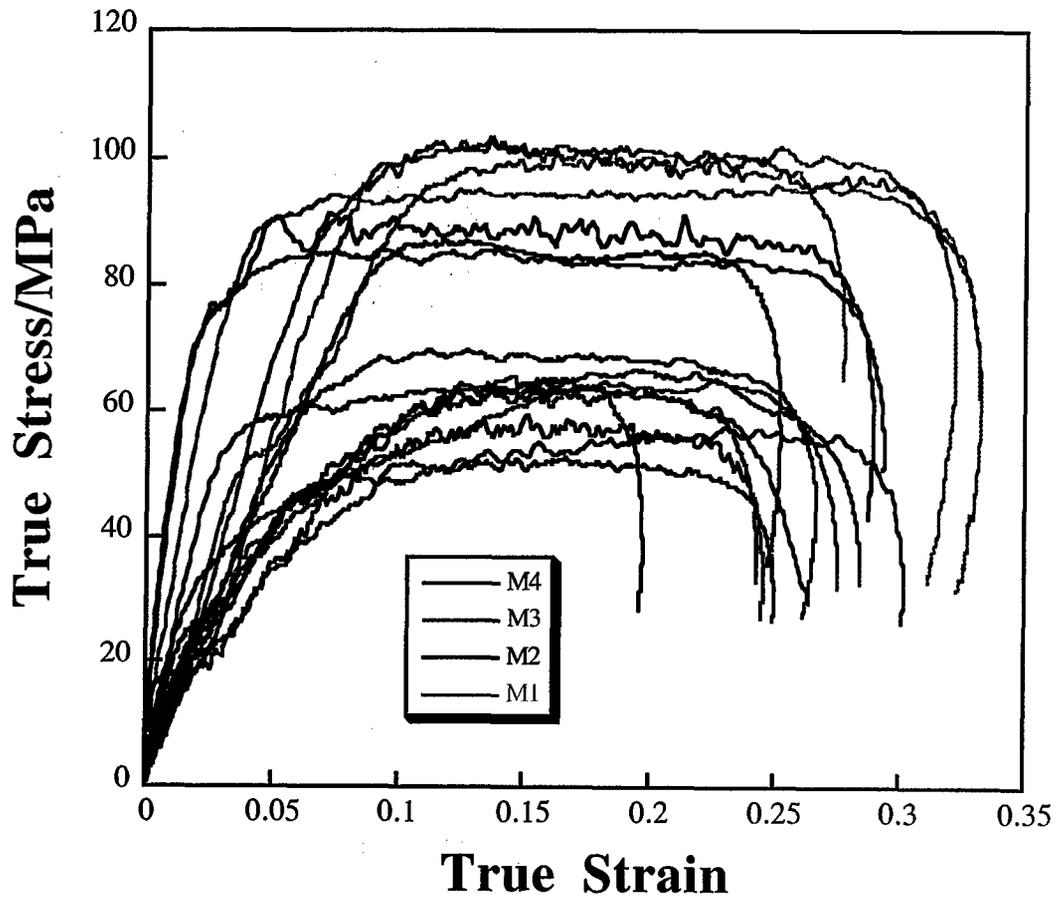
Figure 10. All the stress-strain curves for all four compositions at –60 °C

-60 °C
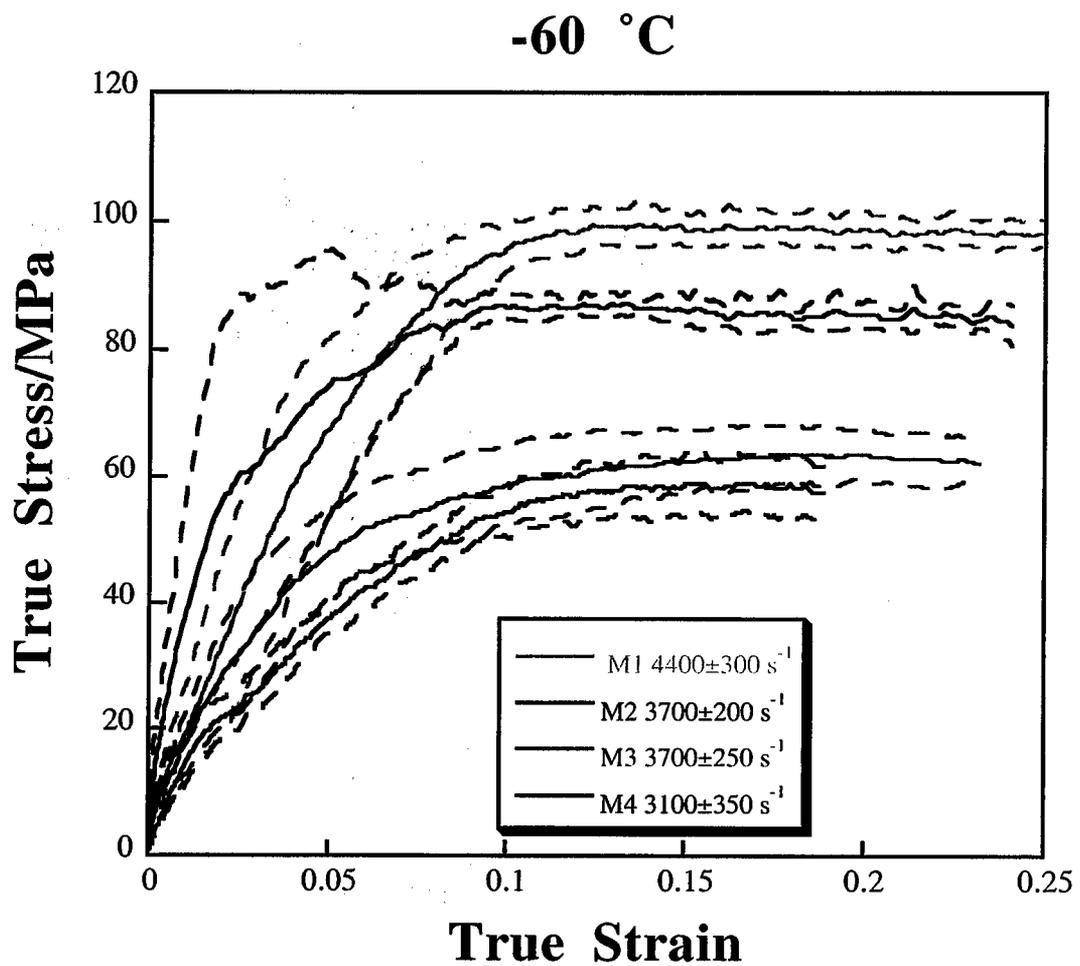
Figure 11. Average SHPB stress-strain curves for all the AP/HTPB compositions at -60 °C. Dotted lines indicate standard deviation of the data.
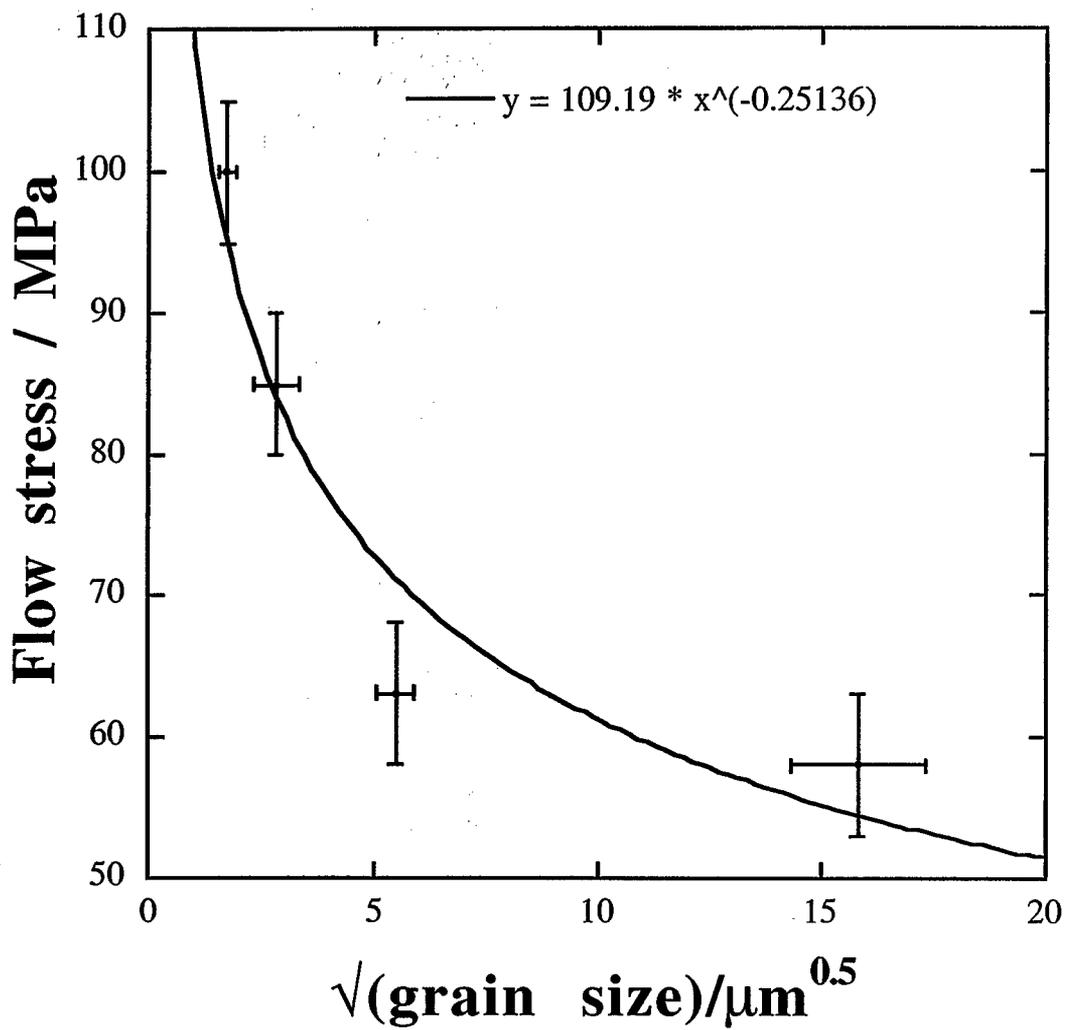
Figure 12. Plot of flow stress versus square root of particle size for all the AP/HTPB compositions studied.

2. REPORT ON PROFESSOR ARMSTRONG VISIT TO CAMBRIDGE

Professor Armstrong visited Cambridge between 26th May and 1st June 2002. Part of the visit was concerned with looking at the facilities for high-strain rate research in the laboratory, and discussing the research covered in the first report. He also gave an interesting colloquium; *Dislocation Properties: Pile-up/crack analogy; grain volume/boundary-controlled dynamics; and, energetic crystal interpretations.*

Suggestions for further work were discussed. All agreed that closer contact between Cambridge and Eglin would be beneficial, especially with regard to manufacture of samples for testing. Research will be performed to study the effects on the mechanical properties of propellant materials of the following parameters;

- Grain size
- Loading density
- Temperature, including one temperature below the glass transition
- Strain rate, including tests up to 70,000 s^{-1} in the miniaturised direct impact Hopkinson bar.

Professor Armstrong asked Cambridge to send him a list of required samples, which is given in Table 1. Regarding the sample sizes, some small tolerance is allowed in the sample length and diameter. However, the surface finish of the samples must be good, and the ends should to be parallel to about 20μm. This we have found precludes cutting samples from larger blocks as the cut surfaces are not sufficiently flat and parallel. It is thought that careful casting into good quality moulds may produce the best results.

Finally, the possibility for a visit by Clive Siviour to Eglin was discussed. This visit would be to oversee the construction, commissioning and use of a Split Hopkinson Pressure Bar, identical to the one in Cambridge. Funding for this new item would have to be additional to the present contract.

Table 1: Sample requirements for suggested propellant tests.

Quantity	Grain size	Sample size	Energetic loading density (by volume)
10		6 mm dia. 2 mm thick	0%
30	1 μm	6 mm dia. 2 mm thick	66%
10	1 μm	1.5 mm dia. 0.75 mm thick	66%
50	10 μm	6 mm dia. 2 mm thick	66%
10	10 μm	1.5 mm dia. 0.75 mm thick	66%
30	100 μm	6 mm dia. 2 mm thick	66%
10	100 μm	1.5 mm dia. 0.75 mm thick	66%
10	1 μm	6 mm dia. 2 mm thick	33%
10	10 μm	6 mm dia. 2 mm thick	33%
10	100 μm	6 mm dia. 2 mm thick	33%
10	1 μm *	6 mm dia. 2 mm thick	95%
10	10 μm *	6 mm dia. 2 mm thick	95%
10	100 μm *	6 mm dia. 2 mm thick	95%

* These samples may need to be of a bimodal distribution.

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Dynamic Deformation Properties of Energetic Composite Materials

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S M Walley, C R Siviour, J E Balzer, D D Radford,
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The report outlines the mechanical, optical and microscopical techniques available in the Physics and Chemistry of Solids (PCS) Fracture and Shock Physics Group, Cavendish Laboratory, Cambridge for studying the impact and high strain rate properties of inert and energetic materials. Preliminary results are presented on the high strain rate (3,500 s^{-1}) stress-strain response of a PBX consisting of an HTPB binder and AP particles of a range of four different sizes. The material with the largest particle size had a significantly lower stress-strain curve than material with smaller particles. These results were obtained using a low impedance split Hopkinson pressure bar (SHPB). An optical extensometer has been used to check the accuracy of the strain measurement in the SHPB for soft polymeric materials. A comparison was made of the results obtained using a miniaturised direct impact Hopkinson bar (DIHB) and a conventional SHPB. Excellent agreement was found at strain rates in excess of 30,000 s^{-1}. The DIHB will be particularly useful in performing tests on nanometric energetic materials. Another comparison was made between the stress-strain curves obtained with an instrumented dropweight and our SHPB. Recently published results on DDT studies of ultrafine and conventional PETN and RDX are summarised.

14. SUBJECT TERMS

Deformation, PBXs, Dynamic Properties, Hopkinson Bar, Drop-Weight Techniques

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1. INTRODUCTION

The Physics and Chemistry of Solids (PCS) Fracture and Shock Physics Group at the Cavendish Laboratory, Cambridge has built up and continues to develop a wide range of techniques available for the study of energetic and inert materials at impact rates of strain. Table 1 lists the mechanical equipment available for measuring the strength properties of materials. Table 2 gives the wide range of high-speed cameras the group possesses. We have found over many years that the technique of high-speed photography gives invaluable information about the initiation and ignition mechanisms of energetic materials, particularly when applied to small-scale, well-characterized specimens. A drop-weight machine modified to allow photography of the deforming specimen has played a key role in this work. Much of the mechanistic information about hot spot mechanisms could not have been obtained from large explosive charges nor predicted in advance of performing the experiments.

In recent years, we have also built up an expertise in optical and microscopical techniques for the measurement of deformation in both quasistatic and dynamic tests (see Table 3). This is providing detailed and accurate information about the response of both energetic (Goldrein *et al.* 1995*a,b*; 2002) and inert (Synnergren and Goldrein 1999; Synnergren *et al.* 1999; Goldrein *et al.* 2000; Grantham *et al.* 2000) materials to various stimuli.

Hopkinson bars play a major role in our group for obtaining the mechanical properties of a wide range of materials at high rates of strain ($10^{+3} - 10^{+5}$ s^{-1}). The first Hopkinson bar to be built in our group was designed for obtaining stress-strain curves of very hard metals at strain rates in the range $10^{+4} - 10^{+5}$s^{-1} (Gorham 1980, 1991; Gorham *et al.* 1984, 1992), the limit at which it is feasible to perform experiments under conditions of 1D stress.

Since then, we have extended our capabilities by constructing compressive Hopkinson bars with a wide range of impedances, from magnesium at the lowest to tungsten at the highest (see Table 4). This allows us to obtain compressive stress-strain curves of materials with yield stresses of a few MPa (soft rubber binders, for example) to a few GPa (armour steels, for example). We have chosen to use a low impedance metal rather than a polymer for the bar material because the mathematics of elastic wave propagation is simpler for an elastic as compared to a viscoelastic bar (Bacon and Brun 2000; Gray III and Blumenthal 2000; Bacon *et al.* 2001; Bussac *et al.* 2002).

Data for constitutive modelling of materials should be obtained over a range of temperatures. We have therefore developed the capability of performing Hopkinson bar tests over a wide range of temperature (-196 to +600 °C). For high-conductivity metals, we use an induction heater. This has the advantage that it can be computer-controlled so that the heating cycle can be as short as a few seconds. This is important in order to minimise changes in the microstructure before the test is carried out. For low conductivity materials such as polymers and PBXs, we enclose the bar ends and specimen within a chamber and pass in helium gas heated

1

TABLE 2
HIGH-SPEED CAMERAS AVAILABLE IN THE PCS GROUP

Make	Framing speed
Kodak Fast video	up to 2×10^3 s^{-1}
Hadland hyspeed (16 mm film)	up to 10^4 s^{-1}
AWRE C4 rotating mirror (140 frames)	up to 2×10^5 s^{-1}
Beckman & Whitley 189 (rotating mirror, 25 frames)	up to 4×10^6 s^{-1}
Hadland Imacon 792 (8 full or up to 24 half frames)	up to 10^7 s^{-1} or streak
IMCO Ultranac (8 full or up to 24 half frames) (fully programmable)	up to 10^7 s^{-1} or streak
IMCO Ultra 8	up to 10^8 s^{-1}

TABLE 3
OPTICAL, MICROSCOPY AND X-RAY TECHNIQUES AVAILABLE IN PCS GROUP

Moiré Interferometry
Speckle Interferometry
Digital Speckle Photography (DSP)
Digital Speckle Radiography
Flash X-ray Image Analysis
Double Exposure Speckle Photography (DESP)
Digital Image Cross-Correlation
Automated Fine-Grid Technique
X-ray Microtomography
Environmental & Conventional Scanning Electron Microscopy (ESEM)
Atomic Force Microscopy (AFM)

TABLE 4
MECHANICAL PROPERTIES OF HOPKINSON BARS AT THE CAVENDISH LABORATORY

Material	Density/kg m^{-3}	Wave Speed/m s^{-1}	Impedance/kgm^{-2} s^{-1}
Magnesium AZM	1798	4920	8.85×10^6
Dural	2711	5040	13.7×10^6
Ti6Al4V	4418	4840	21.4×10^6
Stainless steel	7835	4842	37.9×10^6
Maraging steel	8080	4830	39.1×10^6
Inconel 718	8269	4980	41.3×10^6
Tungsten	16900	4406	75.3×10^6

Results

A number of samples of each grain size were studied. In general, the results from four samples would be consistent, but occasionally there would be an 'outlier'.

Figure 1 shows the stress-strain curves for the four different grain sizes obtained at room temperature. Comparisons of a few representative samples from each grain size are shown in figure 2. Figure 3 shows a comparison of these results with those obtained in previous work at room temperature (Balzer 2001). The agreement can be seen to be good.

Discussion

The graphs presented in figure 1 show there is no rate dependence of the samples over the strain rate range examined: *ca.* 1000-7000 s^{-1}.

It can be seen from the curves presented in figure 2 that there is no obvious difference in the stress-strain curves obtained for material with grain sizes ranging between 3μm and 30μm. However material with a grain size of 200-300μm **does** exhibit a lower stress-strain curve. This suggests that there might be a grain size effect between 3 and 30μm, but the spread in the data obtained has hidden it. Further studies with more accurately prepared samples, for example cast to size, might allow data to be obtained with a much lower variation. Cast specimens might also allow strain rate effects to be distinguished.

A further problem is the oscillation of the stress-strain curve, especially for the smaller grain size samples. This is quite a common phenomenon when testing very soft materials, such as rubbers e.g. Gray III *et al.* (1997, 1998, 2000), Gray III and Blumenthal (2000). These oscillations can be removed by Fourier analysis (see figure 4). The initial parts of the stress-strain curves show ramping, which is almost certainly the effect of the sample stress 'stepping up' as the stress wave passes backwards and forwards through it (Briscoe and Nosker 1984). The oscillations on the curve have the same wavelength as this ramping, and are therefore probably due to the movement of the wave within the sample, although a mechanism for this has not yet been determined. Measurement of the wavespeed in the sample will determine whether the time period of the oscillations is comparable to the travel time in the sample. Use of high-speed photography would possibly also allow the cause of these oscillations to be determined. There are also plans to use speckle photography to measure the deformation fields over the whole sample surface (Huntley and Field 1986; Goldrein *et al.* 1995a; Goldrein 1996). However this would require very accurately prepared cylinders for precise calculation of the strains from the speckle patterns obtained.

Knowledge of the transverse and longitudinal sound wavespeeds would allow the Poisson's ratio of these materials to be found. Such a measurement is a check on the assumption that volume is conserved during the deformation. Volume conservation has been assumed on the grounds that the polymer is a minority component of the sample, and this is the only part of the sample which behaves in a non-conservative manner, and that only during the 'elastic' part of the deformation (G'Sell and Jonas 1979, 1981). Since rubber generally conserves

5

3μm Stress-Strain Curves

Sample 9: 2404
Sample 4: 3600
Sample 10: 4280
Sample 7: 5700
Sample 6: 5960
Sample 8: 6270

(a)

8μm Stress-Strain Curves

Sample 1: 1700
Sample 2: 2930
Sample 4: 3620
Sample 3: 4380
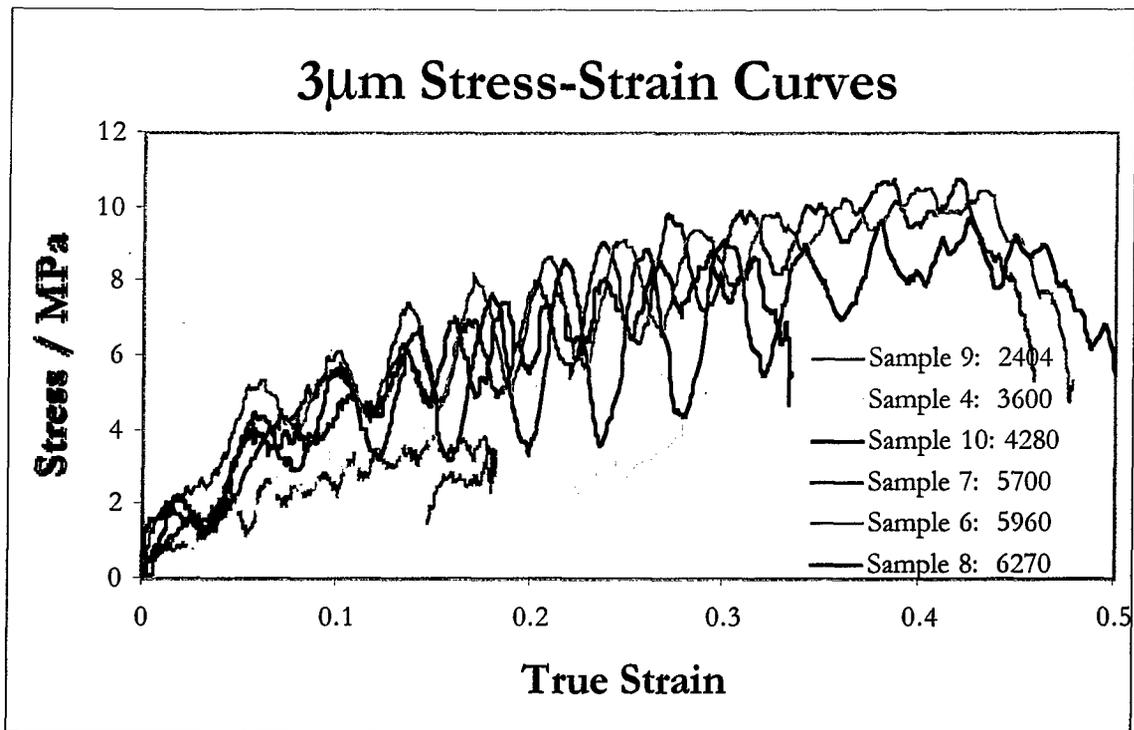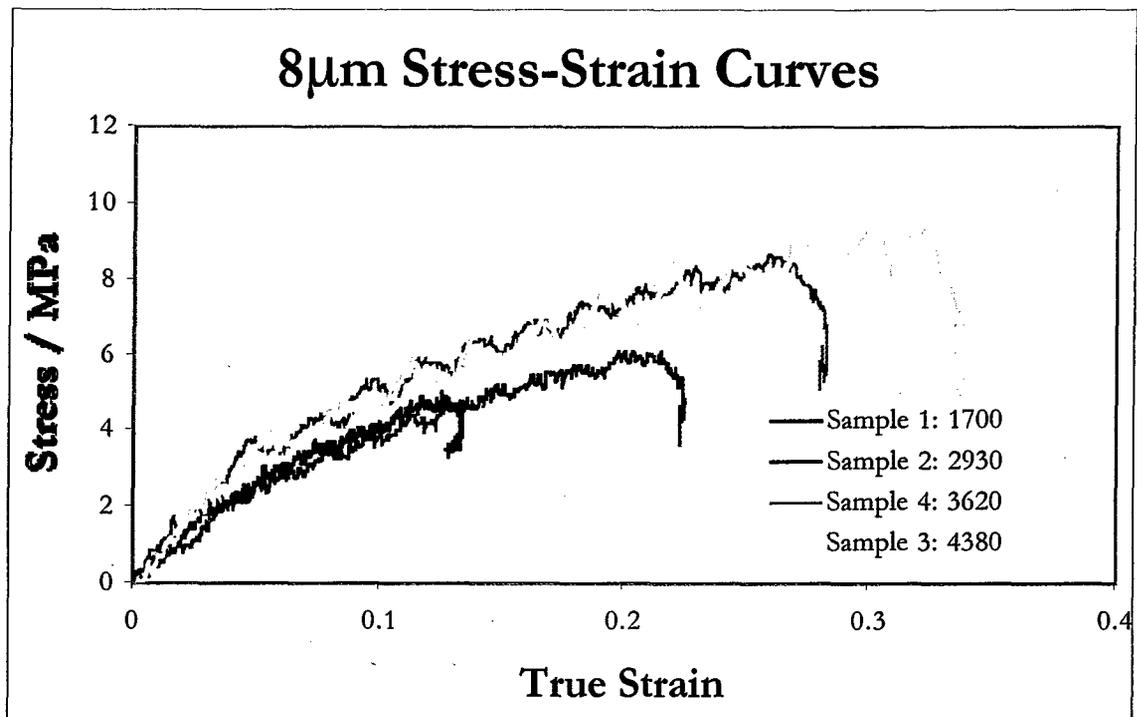
(b)

Figure 1 (caption on next page)

30μm Stress-Strain Curves

— Sample 4: 3360
— Sample 5: 3800
— Sample 6: 4080
Sample 3: 5360
— Sample 2: 5770

(c)

200-300μm Stress-Strain Curves

— Sample 3: 3310
— Sample 2: 3570
Sample 1: 3890
— Sample 5: 3970
— Sample 4: 4030
— Sample 6: 4880
— Sample 7: 6570
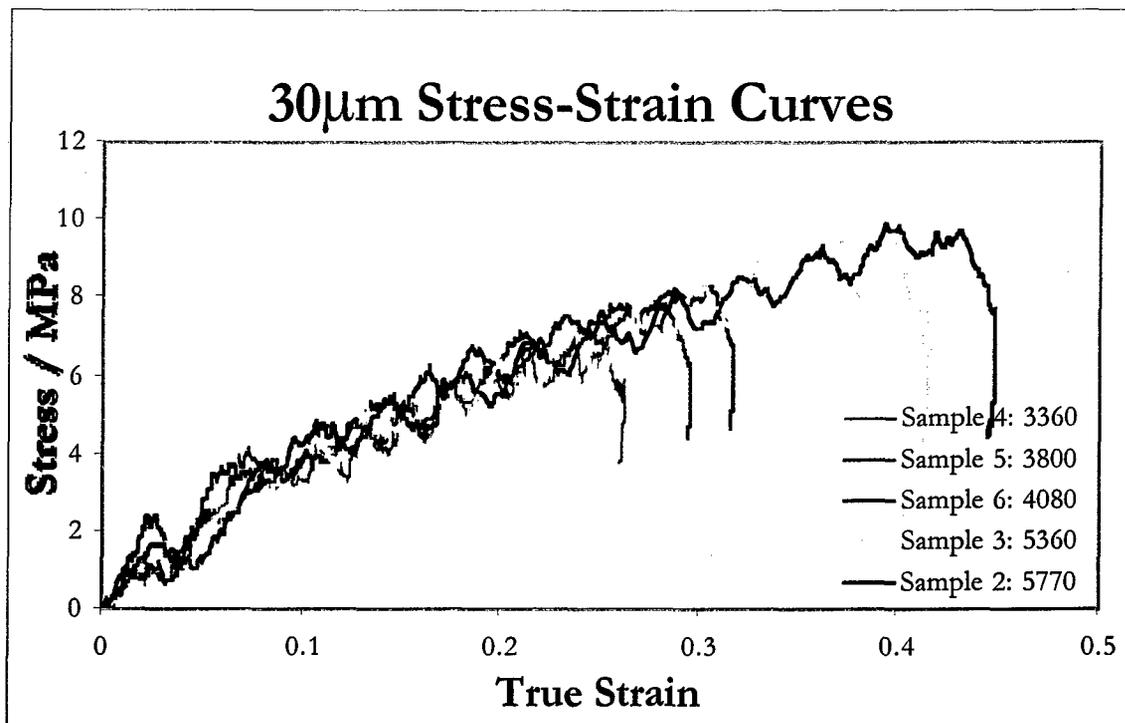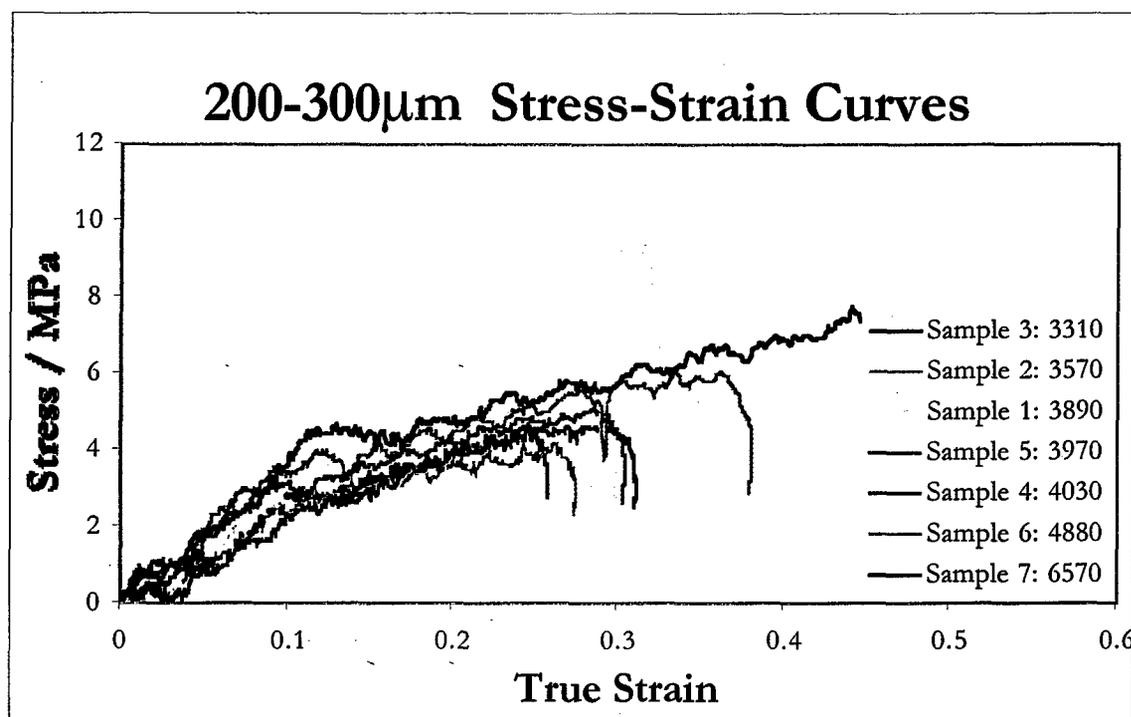
(d)

*Figure 1. Stress-strain curves obtained in a compression SHPB for AP+HTPB PBXs of various AP grain sizes: (a) 3μm, (b) 8μm, (c) 30 μm, (d) 200-300μm*
The figures in the lists on the RHS of the graphs are the strain rates in reciprocal seconds.

All particle sizes
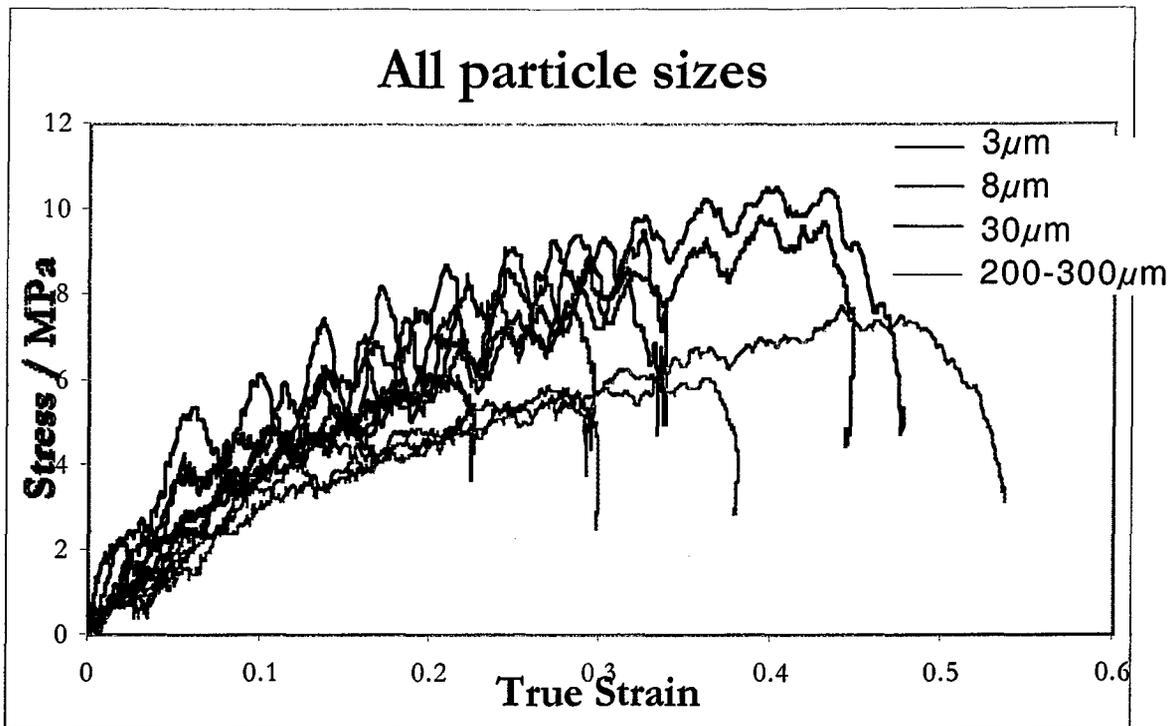
Figure 2. Comparison of SHPB stress-strain data for all grain sizes tested.

Comparison of old and new data
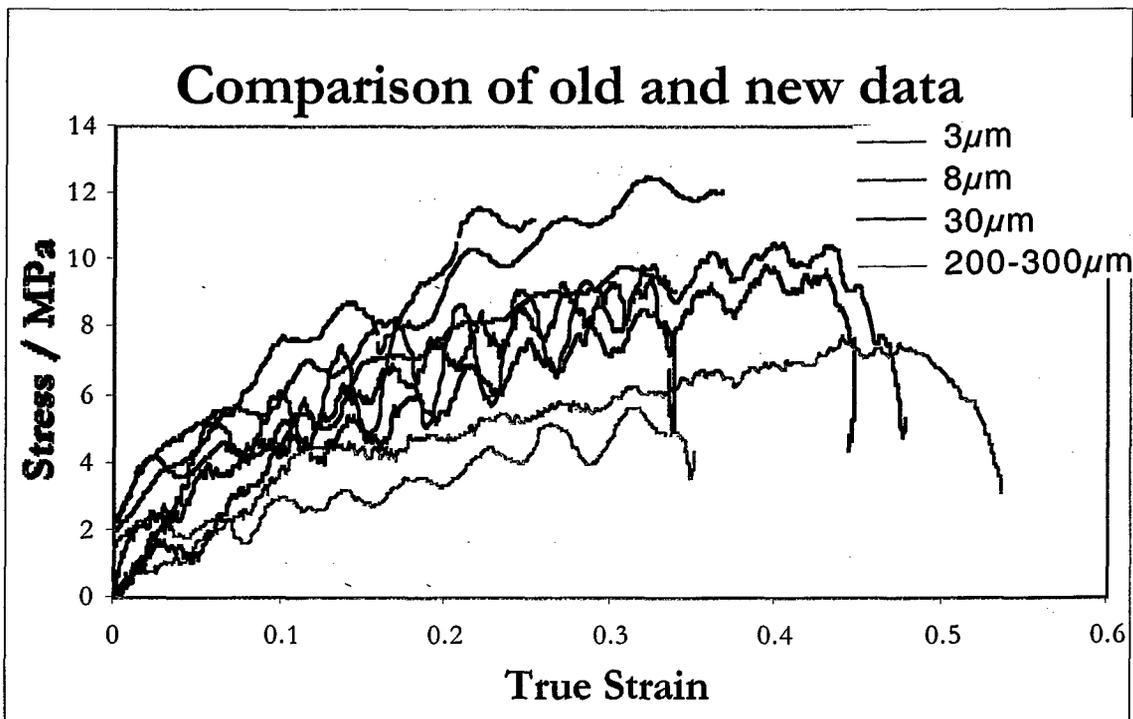
Figure 3. Comparison of old and new stress-strain data at room temperature.

Frequency Domain


(a)

30μm Stress-Strain Curve & FFT
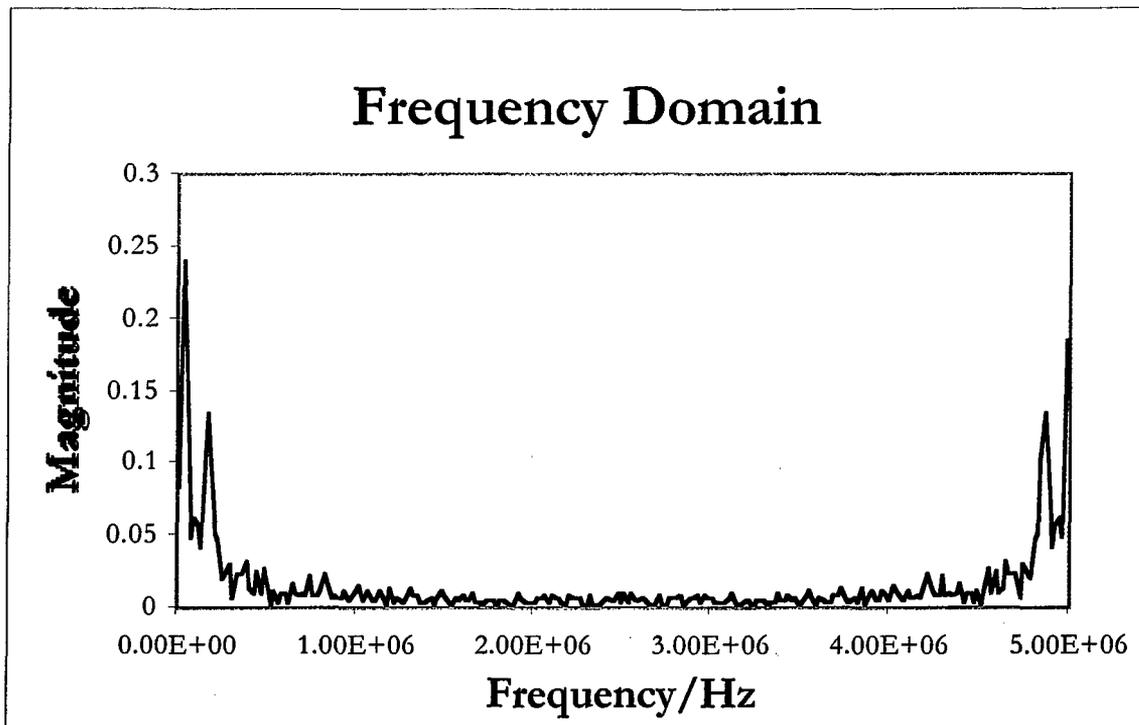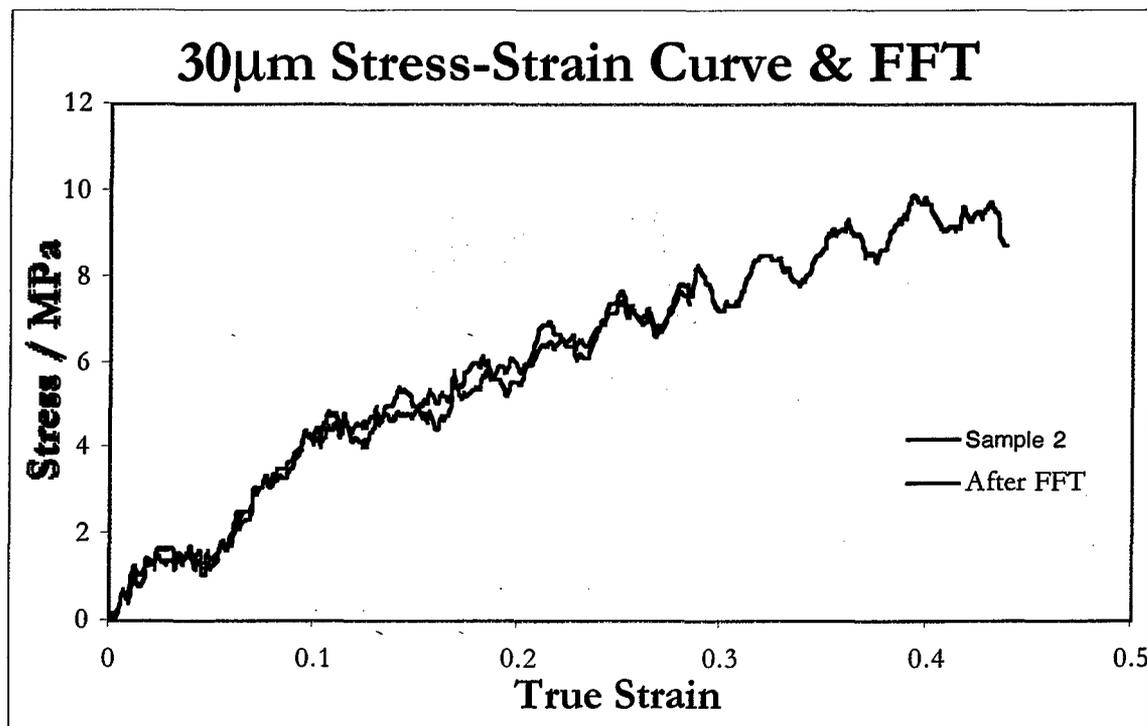
(b)

Figure 4. (a) Frequency spectrum obtained by Fast Fourier Transform (FFT) analysis of one of the 30μm AP particle size stress-strain curves. The frequency of the oscillation can be clearly seen to be around 5 MHz. (b) Comparison of stress-strain curves with and without removal of the main oscillatory frequency component.
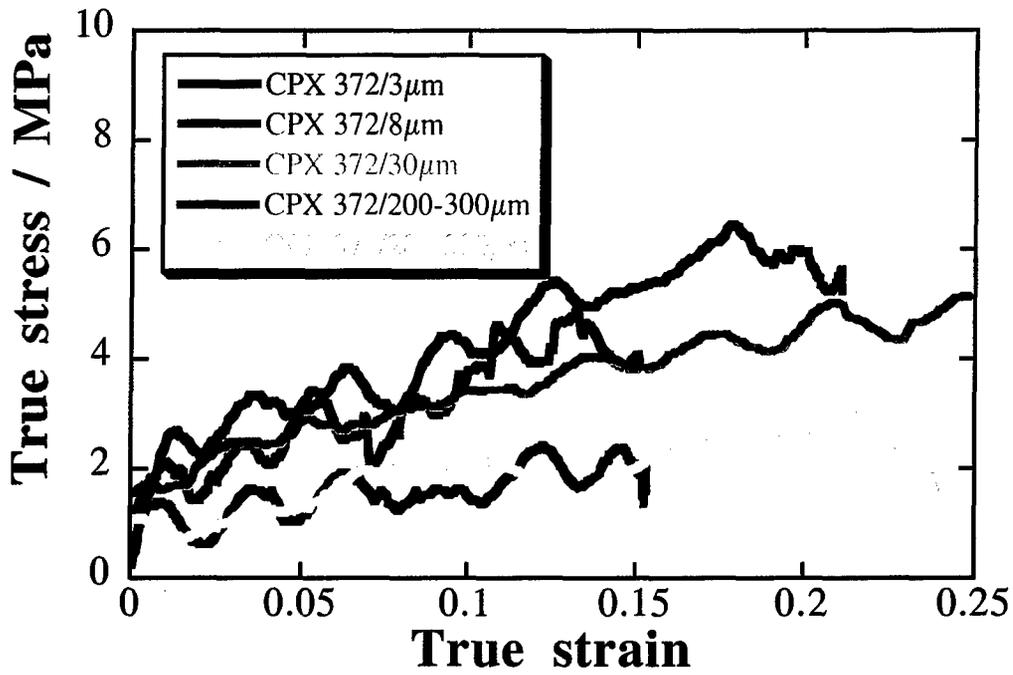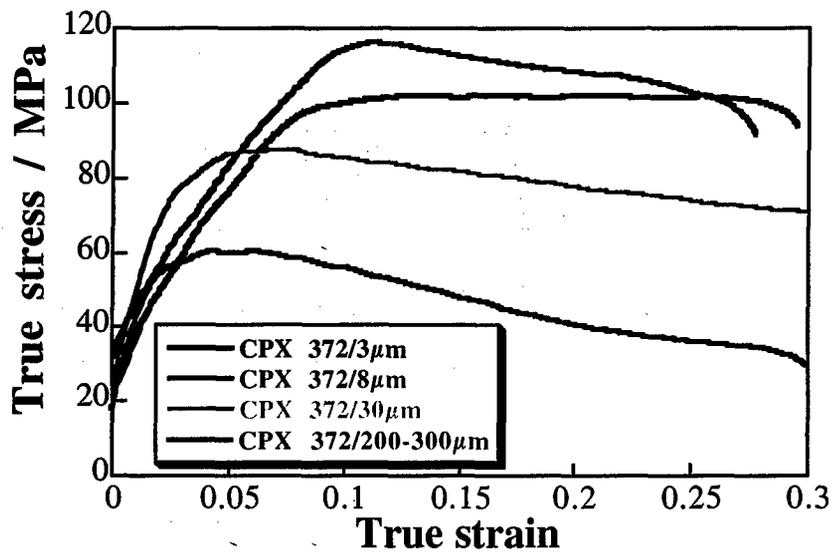
Figure 5. SHPB stress-strain curves obtained for the AP+HTPB PBXs of various AP particle sizes for two different temperatures: (a) +60°C, (b) –60°C.
From Balzer (2001).

3. Recommissioning of Miniaturised Direct Impact Hopkinson Pressure Bar and Comparison with a Conventional Split Hopkinson Pressure Bar System

In order to be able to carry out uniaxial compression tests at strain rates up to the limit of Hopkinson bar techniques (10^5 s^{-1}), Gorham and Field developed in this laboratory a miniaturised direct impact Hopkinson pressure bar system (Gorham *et al.* 1984); Gorham *et al.* 1992). It has also been used to obtain very high strain rate data of soft materials such as polymers and PBXs (Walley *et al.* 1989, 1991, 1992; Walley and Field 1994).

The advantage of not having an input bar is that the strain rate that can be applied is not limited by dispersion of the signal within the pressure bar (Dharan and Hauser 1970). The advantages of miniaturisation are that (i) radial inertia within the specimen is kept small (see below), (ii) the range of frequencies can be transmitted down the output bar is increased, and (iii) the bar can be made from a hard, relatively brittle material such as tungsten carbide. Our system consists of a tungsten carbide (WC) pressure bar 3mm in diameter instrumented with high gain semiconductor strain gauges (BLH Electronics type SPB1-03-12U1) which need no amplification, so there are no complications introduced by bandwidth limitations of amplifiers. Specimens are typically 1mm diameter and 0.5mm thick (pinhead size).

Tungsten carbide was chosen as a bar material because of its strength (which allows very strong metals to be tested) and its low Poisson ratio (0.22, compared to typically 0.33 for most metals), which reduces elastic wave dispersion effects (Bancroft 1941; Davies 1948; Safford 1988, 1992; Mason 1999). It also has a high acoustic impedance which helps to ensure a nearly constant strain rate during an experiment. It has not been used widely in Hopkinson bar applications because of its brittleness. Indeed we have found that WC bars of more conventional length (0.5m long) and diameter (12.7 mm) have a very limited lifetime. Our miniaturised bar has, however, survived many hundred, if not thousands, of impacts. This is presumably due to the lower stresses imparted and the small intrinsic flaw size. Materials with lower Poisson ratio v do exist (e.g. beryllium v = 0.02-0.05, diamond v = 0.07, and plutonium v = 0.19), but none of them are suitable for routine use in a normal laboratory. The only one of these three ever to be used as a bar material is beryllium (Jones 1966; Bateman *et al.* 1996). Safford (1988) suggested that a rod made from beryllium-2%copper (not to be confused with copper-2% beryllium) might be even more suitable, especially if nickel plated to contain the toxic dust and fumes, as its elastic properties are close to that of pure beryllium (Silversmith and Averbach 1970), but as far as we know no-one has acted on this suggestion.

According to the analysis of Pope and Field (1984), there are several quantities that need to be known accurately if a stress-strain curve is to be derived from the signal recorded from the output bar: (i) the initial dimensions of the specimen, (ii) the mechanical impedances Z_i (= $\rho_i c_i$), where ρ_i is the density and c_i the wavespeed

## PC Stress Strain Curves
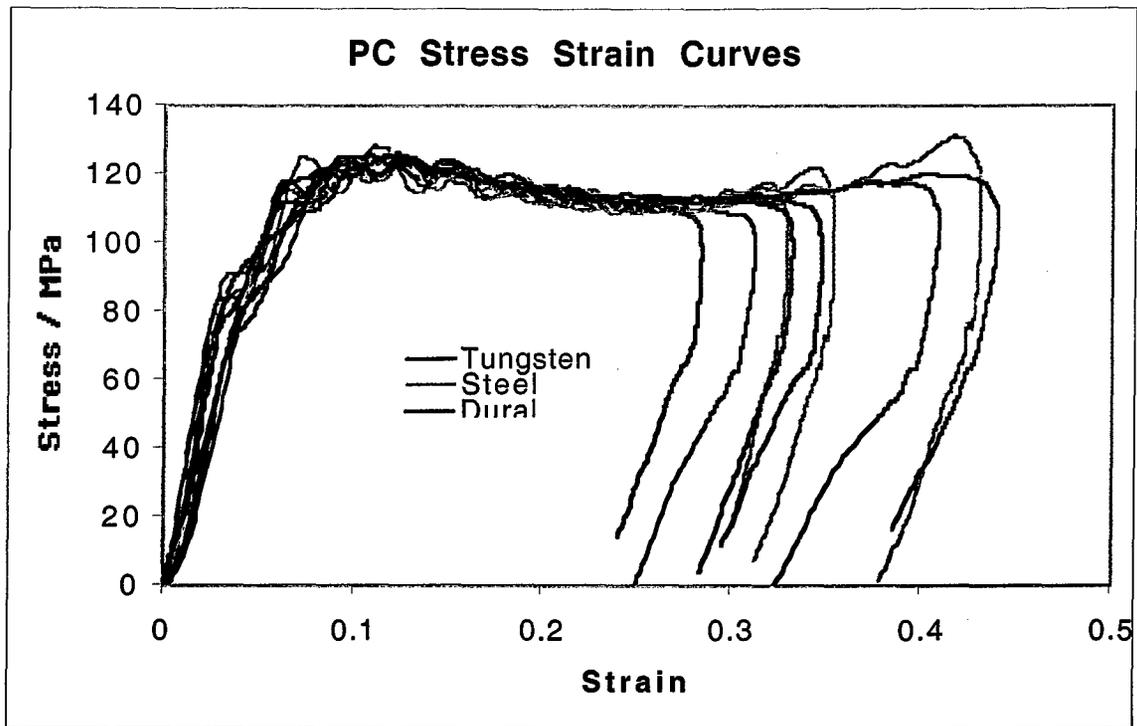
Figure 6. Stress-strain curves obtained for polycarbonate in our compression split Hopkinson pressure bars with a wide range of impedances.
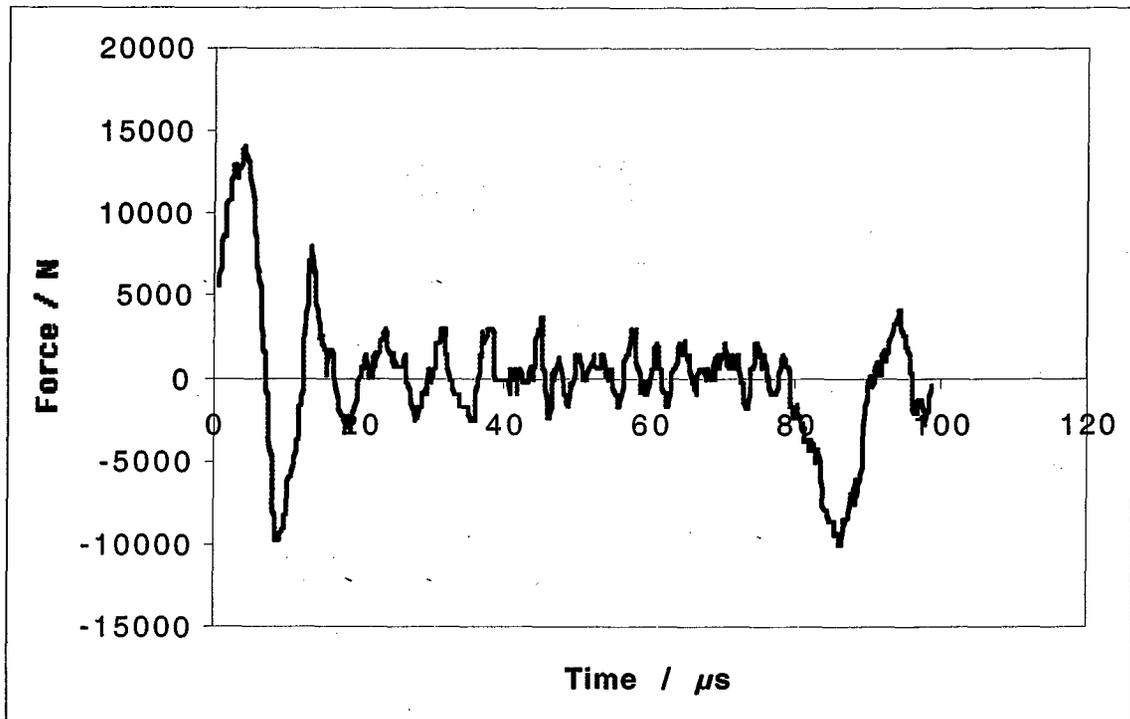

Figure 7. Plot of the difference between the stress-strain curves obtained for a polycarbonate specimen calculated two different ways.

We recently recommissioned the miniaturised direct impact Hopkinson bar for a project to obtain the stress-strain curves of copper specimens at very high rates of strain. Initially we believed we could do this using our low impedance dural SHPB and miniature specimens. But this bar material proved not to be hard enough. Steel bars were then used, with the disadvantage that the transmitted signal is smaller (see figure 8). But the maximum strain rate we could obtain was in the region of 15,000 s^{-1}. The reason for this upper limit is that the impact speed of the striker bar must be kept below that which causes the strain gauges to spall off. It should be emphasised that this is a high strain rate for SHPB testing, but wasn't high enough for the particular application.

The main reason we can obtain such high strain rates with our conventional SHPBs is that we use semiconductor strain gauges which need no amplification. This means we can test very small specimens that only transmit a small signal into the output bar and still have a large signal-to-noise ratio (see figure 8).
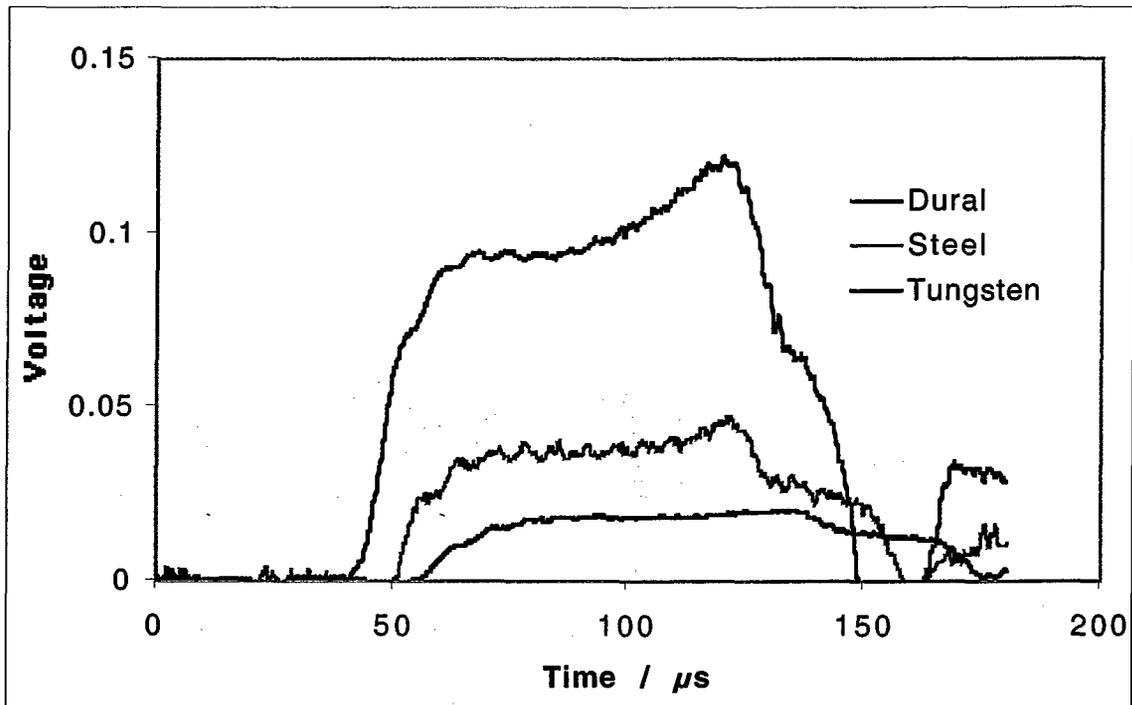

Figure 8. Output bar signals obtained for polycarbonate using three bars of widely varying impedances.

Two other experimental tests were performed to check that the strain in soft specimens was being calculated correctly. The first was to use an optical extensometer. An optical extensometer records the movement of a two black/white boundaries. For the SHPB, these boundaries can be simply created using black and white tape on the input and output bars (see figure 9). It can be seen (figure 10) that the agreement between the strains calculated from the strain gauge outputs using the standard Hopkinson bar equations and those calculated from the displacements measured using the optical extensometer is excellent. The second technique was high-speed photography (see figure 11). The camera used was an IMCO Ultranac. The strains calculated from the high-speed photographic

sequence are compared with those calculated from the strain gauge output are compared in figure 12. The scatter in the data can be seen to be worse than for the optical extensometer, but even so, the agreement is good.
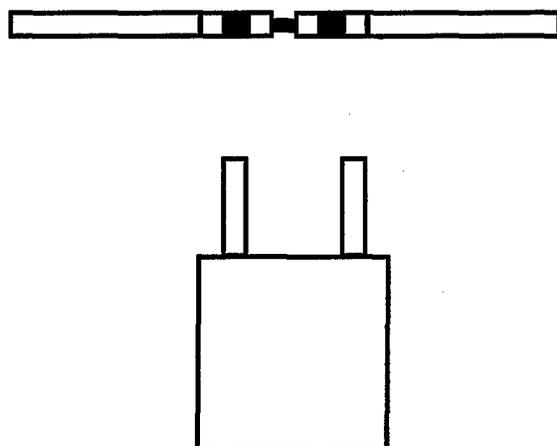
Figure 9. Schematic diagram of optical extensometer system set up to take measurements from a compression SHPB.
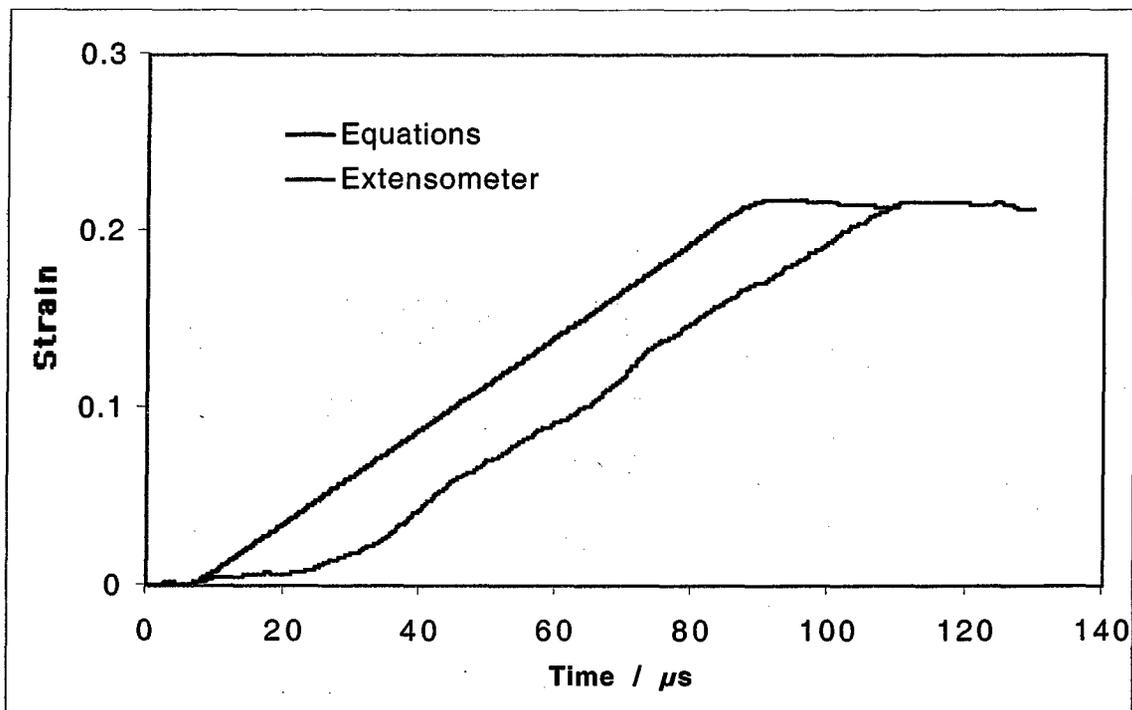
Figure 10. Comparison of strains calculated from SHPB strain gauge outputs and measured using the optical extensometer (traces are time-shifted for clarity).
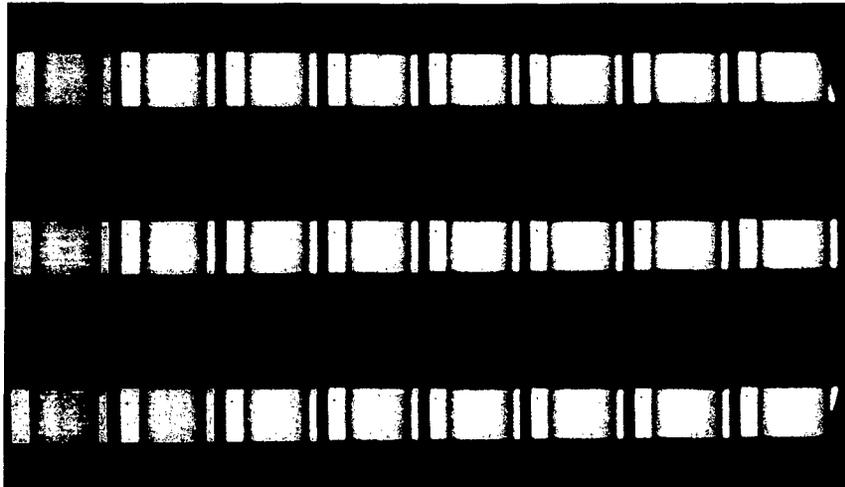
Key to Frame Sequence
1 6 7 12 13 18 19 24
2 5 8 11 14 17 20 23
3 4 9 10 15 16 21 22

Figure 11. High-speed photographic sequence of the deformation of a 4mm long cylindrical PC specimen. Interframe time 5µs.
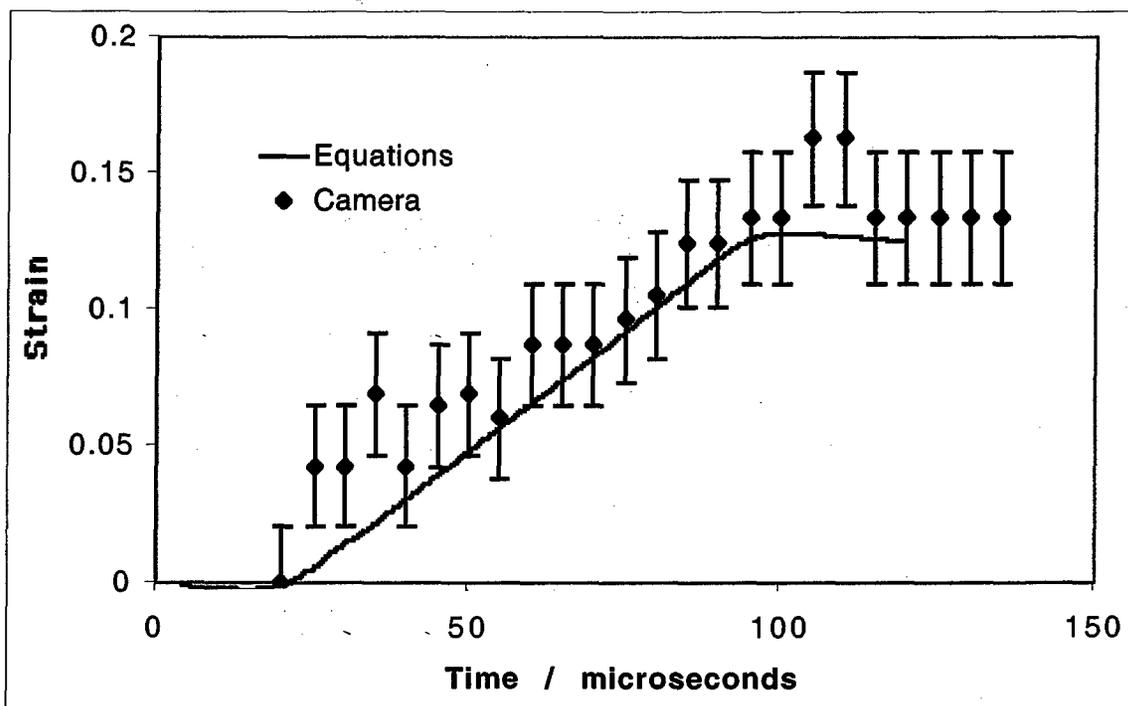
Figure 12. Comparison of strains measured from the sequence shown in figure 11 with that calculated from strain gauge output.

We discovered that the miniaturised tungsten carbide bar required regauging. We decided to use encapsulated semiconductor gauges (BLH Electronics type SPB1-03-12U1) as these are less prone to mechanical damage. They have, however, a longer gauge length than those originally used.

A series of experiments were then performed on 1mm diameter, 0.5mm thick copper specimens. The strain rate was increased in stages by firing the striker bar at increasingly higher velocities until a strain rate of 70,000s^{-1} was reached. Small specimens are required to ensure that inertial stresses do not dominate the measured response (see figure 13). These stresses are given by the following equation (Gorham 1991):

$$\sigma_i = \rho\left[\left(\frac{a^2}{16} + \frac{b^2}{6}\right)\left(\frac{\partial \varepsilon}{\partial t}\right)^2 + \left(\frac{b^2}{6} - \frac{a^2}{8}\right)\frac{\partial^2 \varepsilon}{\partial t^2}\right] \qquad (1)$$

A plot of this for copper specimens of the size used is shown in figure 13.
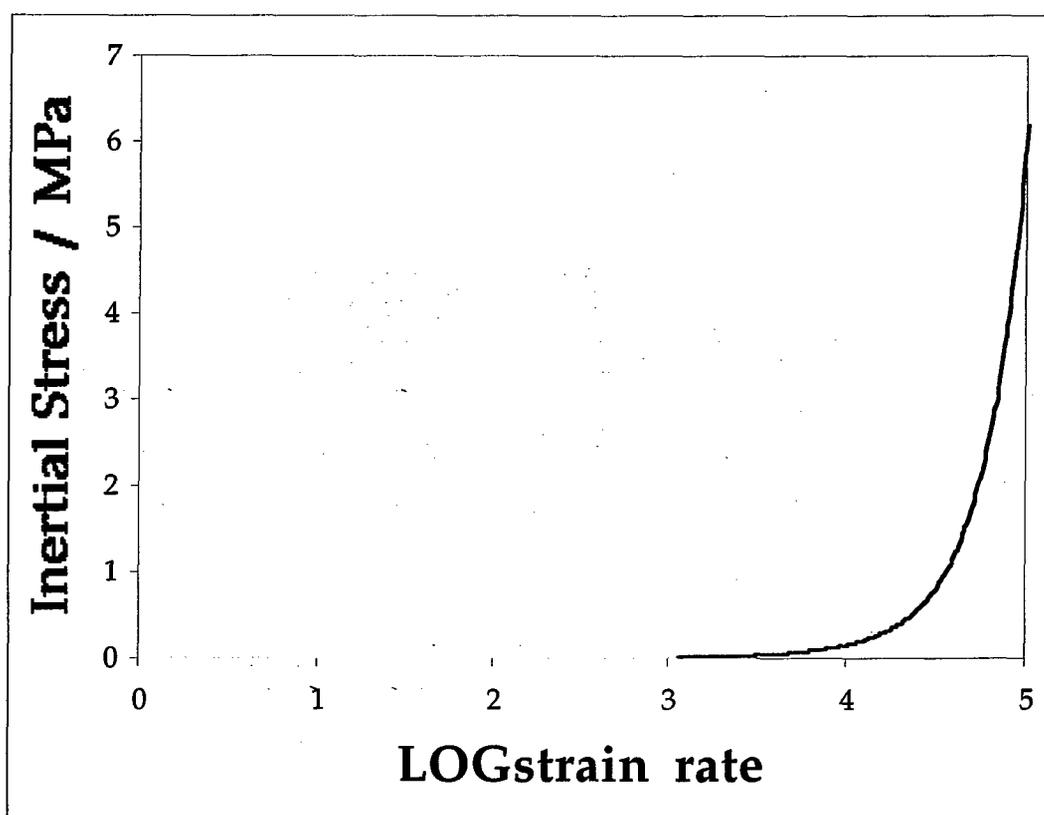

Figure 13. Graph of the inertial stress developed in copper when subjected to compressional deformation at various strain rates. Specimen dimensions: 1mm diameter × 0.5mm thick.

It can be seen that even at strain rates of 100,000 s^{-1} inertial stresses are only around 6 MPa, negligible compared to the flow stress of copper of around 1000 MPa (see figure 14). This demonstrates one of the major benefits of miniaturisation. It should be emphasised that inertial stresses are only active when material is being accelerated. When the specimen has reached steady flow conditions, inertial stresses drop back to zero.

Figure 14 presents the first direct comparison we have made between results obtained in one of our conventional SHPBs and our miniaturised DIHB. The agreement up to a strain of *ca.* 0.45 can be seen to be excellent. This gives us confidence that the DIHB can be used to obtain good data at strain rates approaching 100,000 s^{-1}. This piece of apparatus will play a key role in the project to study the effects of strain rate in various polymers and PBXs with the aim of creating and validating physically-based constitutive relations for these materials.
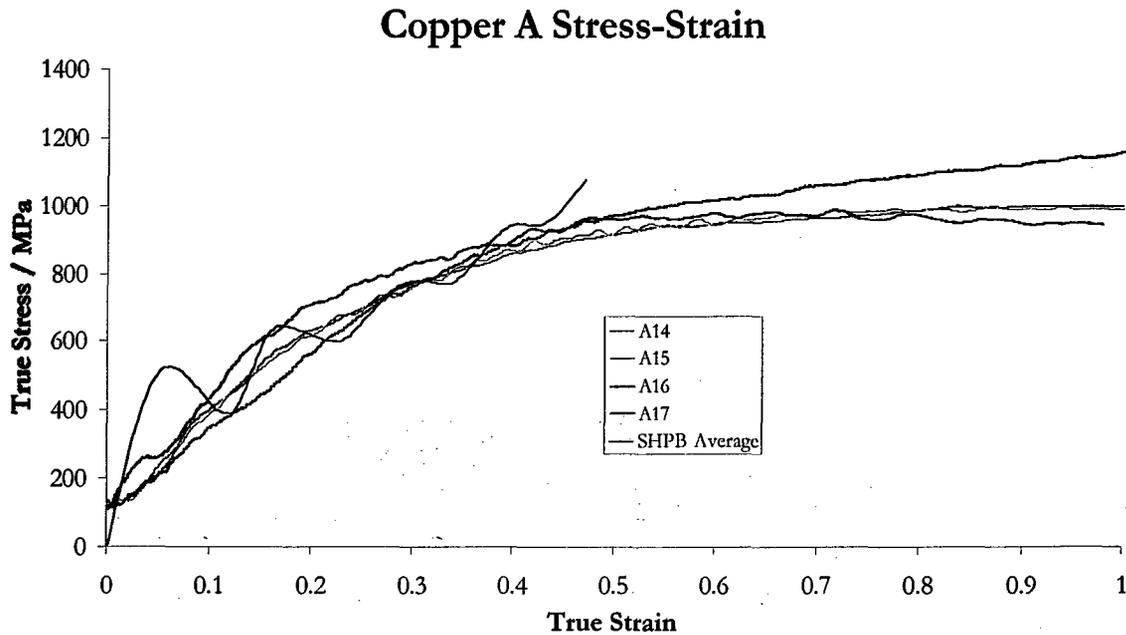

Figure 14. Comparison of stress-strain curve obtained in a conventional SHPB at a strain rate of ca. 15,000 s^{-1} with stress-strain curves obtained in our miniaturised DIHB at strain rates of between 45,000 and 62,000 s^{-1}.

4. Development of Gauged Dropweight Machine

Background

The dropweight machine that we have used extensively for taking high-speed photographic sequences of the rapid deformation of energetic and inert materials (figure 15) has been operated for most of its history without any means of recording force-time data. Two recent attempts were made to instrument it: one with strain gauges attached to a metal ring below the glass anvil (Walley *et al.* 1995) and one with accelerometers attached to the dropweight itself. Accelerometers were found to output a very noisy signal due to reverberations within the dropweight: the oscillations were of similar amplitude to the signal. This has been a frequent observation in dynamic testing rigs of various types (Mooij 1981; Cain 1987; Sahraoui and Lataillade 1990, 1998; Aggag and Takahashi 1996). However, much cleaner signals were obtained using the instrumented ring (Walley *et al.* 1995).
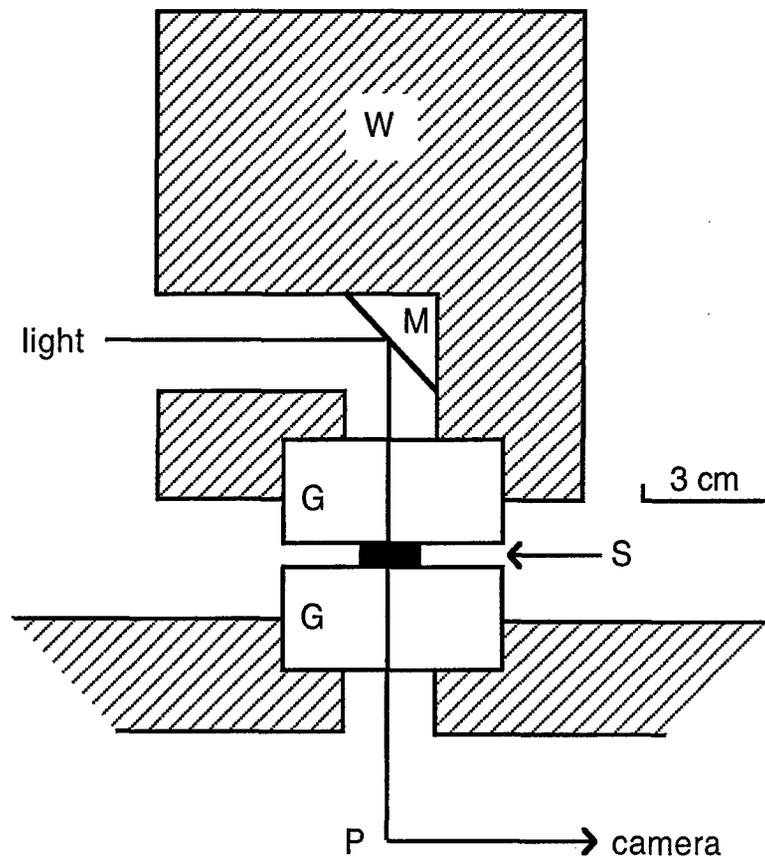
Figure 15. Schematic cross-section diagram of the high-speed photography dropweight apparatus. W weight; M mirror; G glass anvil; P prism; S specimen.

Recently we modified this apparatus so that it can operate as a conventional dropweight machine. The stimulus for this is a project examining the rapid deformation and failure of hard metal cylinders of various aspect ratios. We found that specimens with diameters larger than 4mm could not be deformed in our

It can be seen in figure 16 that the dropweight loadcell consists of three maraging steel cylinders arranged in a stack. The specimen is sandwiched between the upper two. Foil strain gauges are attached to the lowest anvil. This arrangement ensures that the instrumented cylinder is not damaged by the deforming specimen and so can be used many times. It also ensures that the strain field in the gauged anvil is close to uniform (according to the St Venant Principle).

When in use, the stack is enclosed within a steel jacket with sections cut away to allow photography and recording of signals (figures 17 & 18).

Recent work has shown that the arrangement of figure 16 gives the best signal (see figures 20 and 21). Closer examination of the signals near the origin shows that the first oscillation in the black and red traces has structure due to reflections within the block to which the force transducer is bolted (see figure 18). These could be eliminated by using a block of metal that takes the elastic wave energy away and does not allow it to return (our previous dropweight apparatus sat on a blacksmith's anvil, the horns of which presumably have this function: Heavens and Field 1974). The other, more long-lived oscillations have a period commensurate with the elastic wave travel time within the stack of cylindrical anvils. Better impedance matching with the falling-mass may reduce their magnitude.

Again using some soft Pb-Sn solder specimens, a check was made of the reproducibility of the transducer using configuration 1 of figure 19. This can be seen to be excellent, even out to long times.

To ensure the anvil is only struck once, we have developed a 'one-hit' catcher device (see figure 22). This is operated only on the rebound so that it does not interfere with the mechanics of the impact.
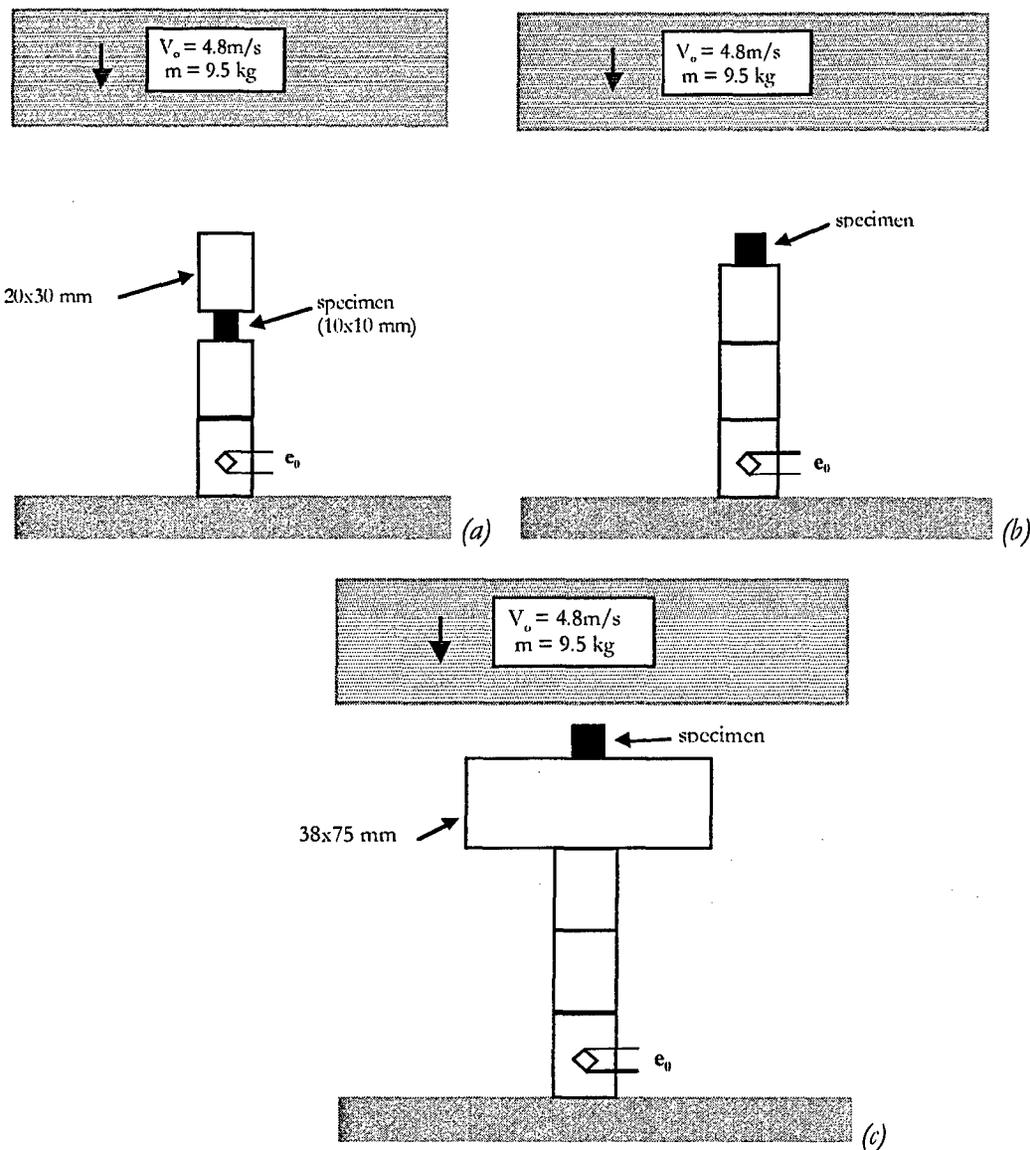
*Figure 19. Three different configurations used to examine best location for specimen.
(a) Configuration 1; (b) configuration 2; (c) configuration 3.
Note that the falling mass was larger in these experiments
compared to those reported later in this document.*
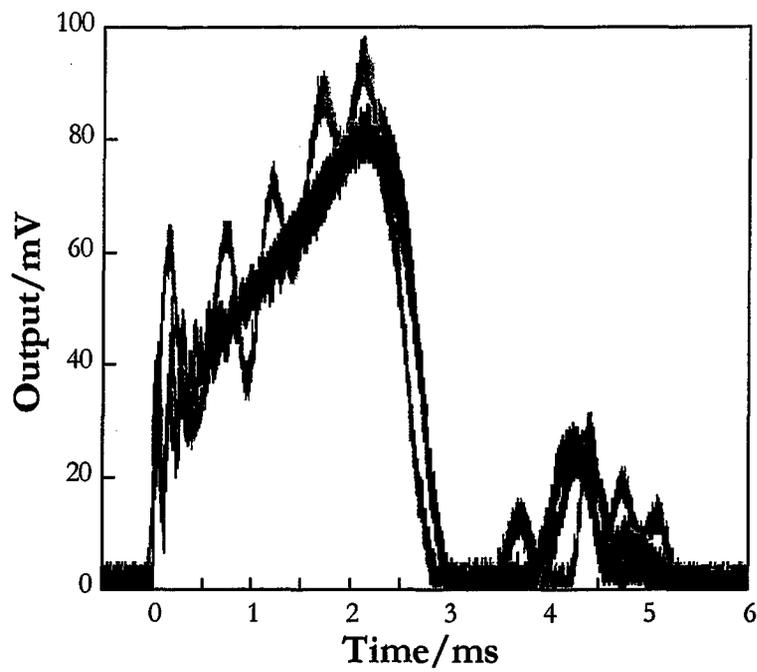
(a)
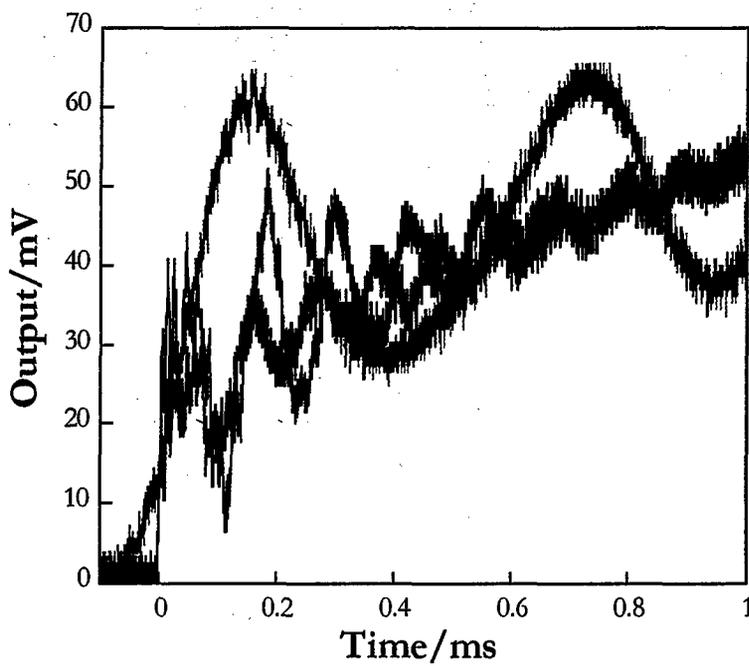
(b)

Figure 20. Transducer output for the three configurations shown in figure 19.
Black line: configuration 1; Red line: configuration 2; Green line: configuration 3.
A. whole signals; (b) enlarged view of signals close to origin.

25
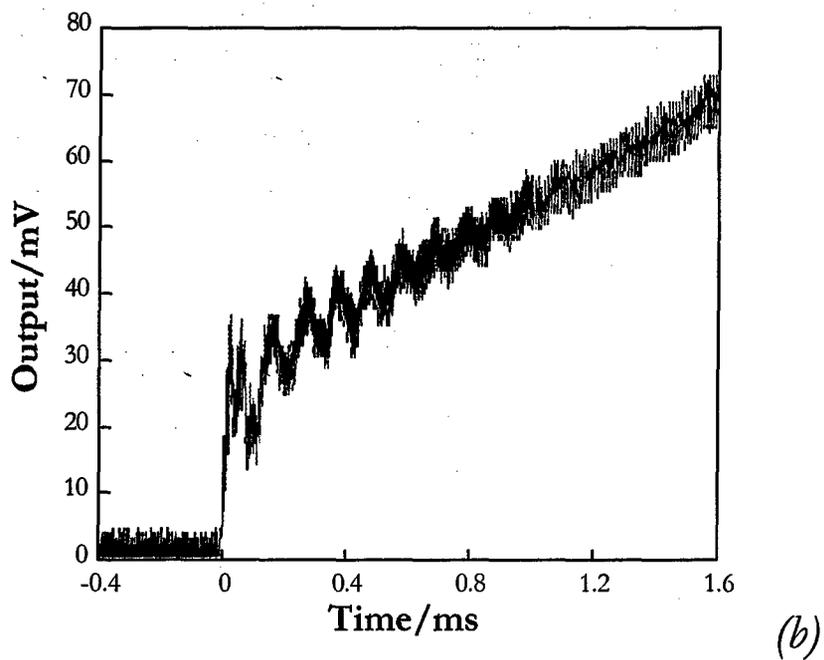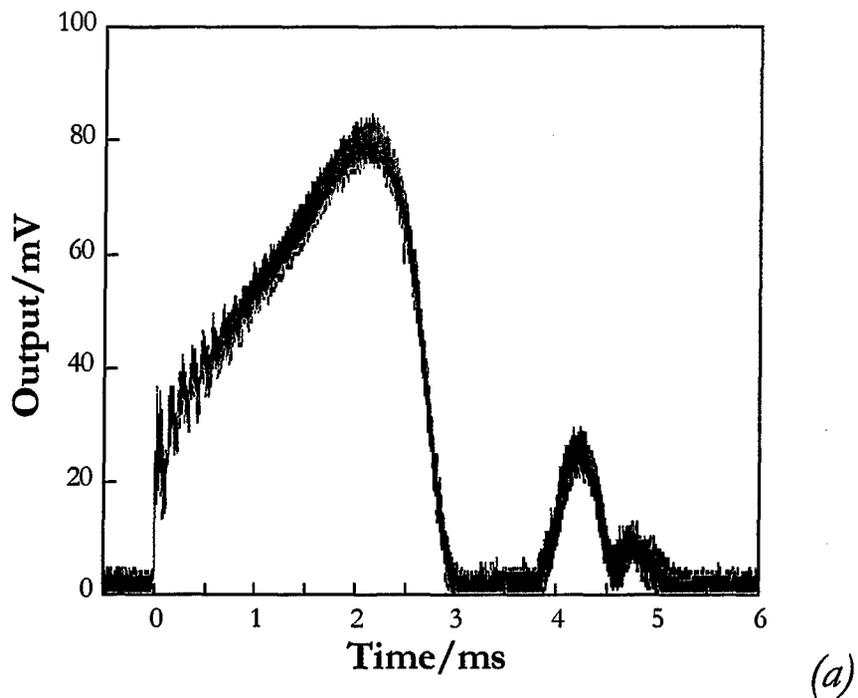
Figure 21. Check of reproducibility of output of dropweight force transducer.
(a) Whole traces; (b) enlarged view close to origin.

Figure 22. Photograph of the 'one-hit' catcher device holding the dropweight after rebound.

where F is the gauge factor of the gauges used and ε is the strain in the gauges (Anon. 1976). This particular configuration is linear.

We decided, however, to calibrate the instrumented anvil. This was done in two different ways: (i) the anvil was loaded statically in an Instron mechanical testing machine; (ii) the anvil was loaded dynamically in the rig by dropping the weight onto the transducer with no specimen present. The static calibration curve is presented in figure 24. The anvil was loaded and unloaded three times to check reproducibility.
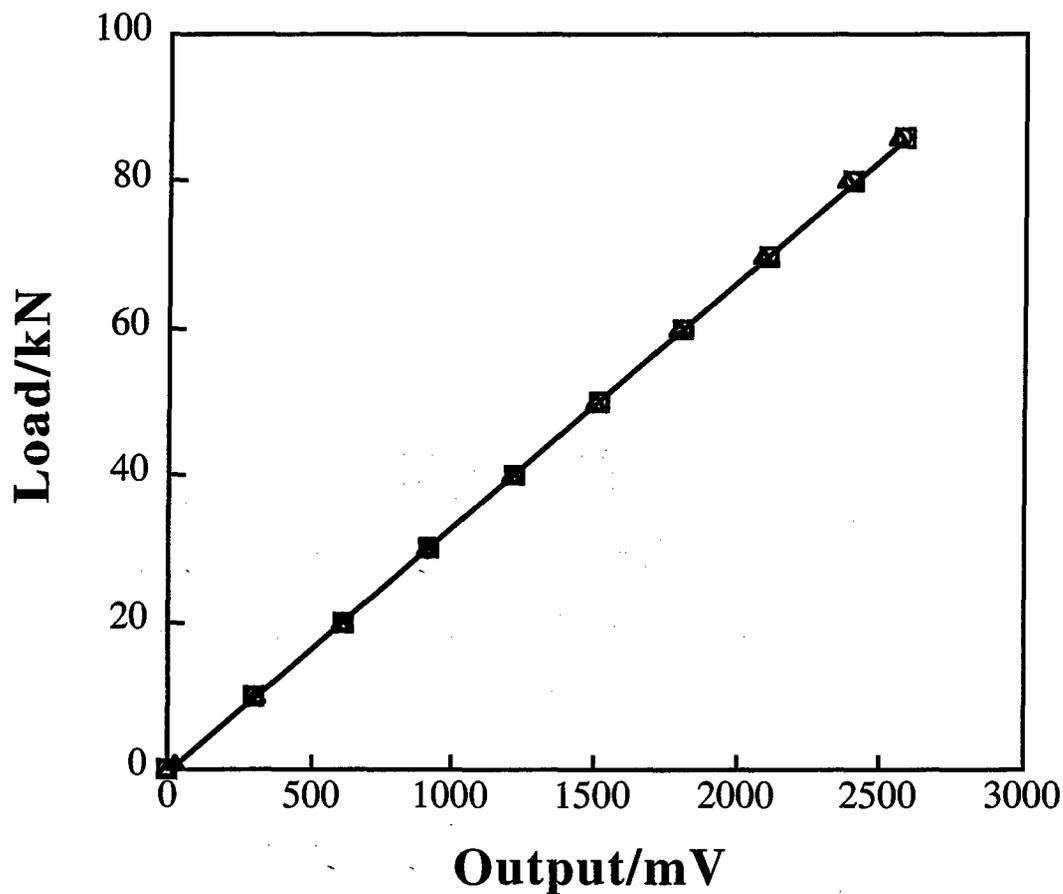
Figure 24. Static calibration curve of the instrumented anvil using the amplifier set to 100x amplification. The calibration factor is 33,380 N/V.

In order to perform a dynamic calibration, the mass of the dropweight needs to be known accurately, and also the impact and rebound speed. This is because the impulse imparted to the transducer is given by $m\Delta v$ Ns. The dynamic calibration factor k is given by:

$$k = \frac{m\Delta v}{\int V \mathrm{d}t} \qquad . \qquad (3)$$

The impact and rebound speeds were measured by attaching a grating with a pitch of 2mm to the side of the dropweight. This passed between a diode laser and an optical fibre connected to a photodiode as the weight falls and rebounds. The measurement is taken as close as possible to the impact point. A typical signal recorded from this set-up is shown in figure 25. The corresponding voltage signal from the Wheatstone bridge is shown in figure 26.
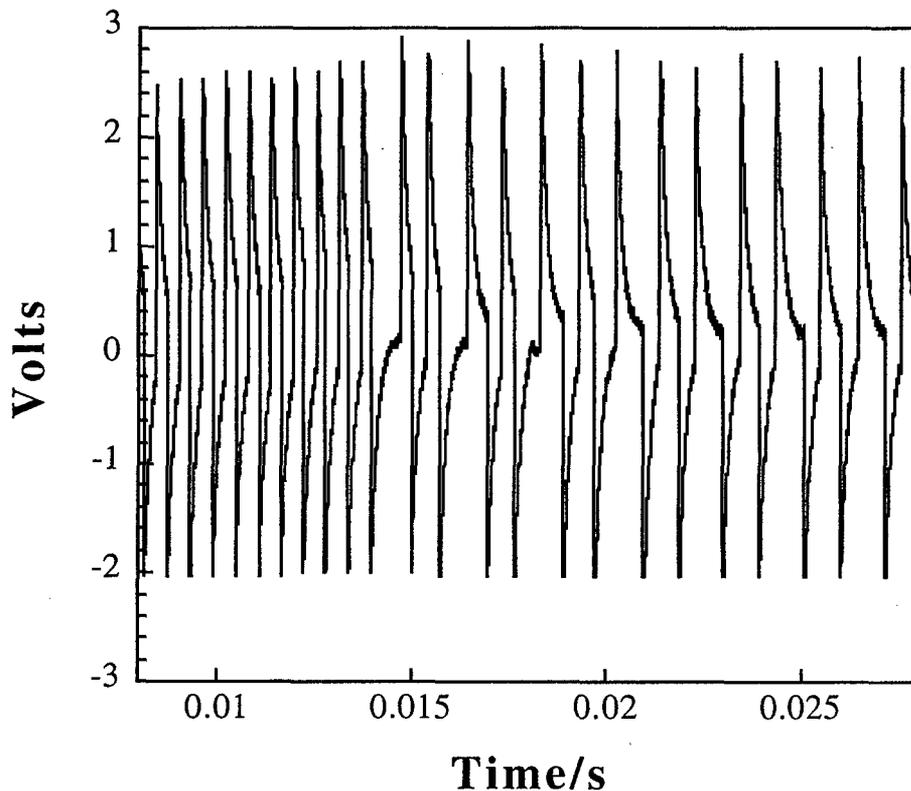
Figure 24. Output from the photodiode as the weight impacts and rebounds from the force transducer.
Calculated impact velocity=3.419 m/s. Calculated rebound velocity=1.964 m/s.

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from subsequent wave reflections: Bussac *et al.* 2002). The dropweight, by contrast, is excited below its resonant frequency. One consequence of this is that the loading time is long, governed by the time taken by elastic wave activity within the impacting mass to produce a net reversal of the momenta of its component parts. The mass, therefore, is subject to a very complex pattern of wave activity within it during impact. This is the reason that transducers should not be attached to it in order to measure the force on the specimen.

A check was then made that the dropweight gave the same stress-strain curve as our compression split Hopkinson pressure bar (figure 27): the agreement can be seen to be good despite the larger oscillations on the stress-strain curve obtained using the dropweight. Note that the dropweight curves go out to larger strains. The data is shown terminating at strains less than the specimens were actually taken to in the dropweight for two reasons: (i) it allows a clearer comparison with the Hopkinson bar data, and (ii) the true stress-true strain curves become less accurate at larger strains due to barrelling of the specimens.
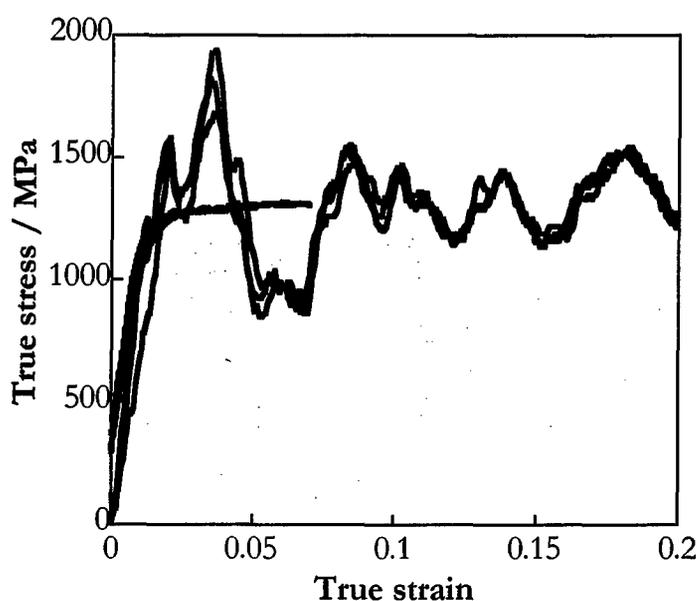
Figure 27. Comparison of stress-strain curves obtained using a compression split Hopkinson pressure bar (red lines) with that obtained using the dropweight (black lines) for Ti6Al4V specimens 4mm diameter, 8mm long.

If it is desired in future to combine high-speed photography with force-time measurement, the stack of steel cylinders would be replaced by the glass anvils (shown schematically in figure 15) and an instrumented annulus placed below the lower glass anvil. We already have experience of this constructing such a system (Walley *et al.* 1995).

Another check on the pressures developed during impact is to use a pressure-sensitive film (see figures 28 and 29). A photograph of a piece of this film that was

in contact with a polymer disc deformed in the dropweight machine is shown in figure 28. A false colour pressure-distribution map of this impact is shown in figure 29.
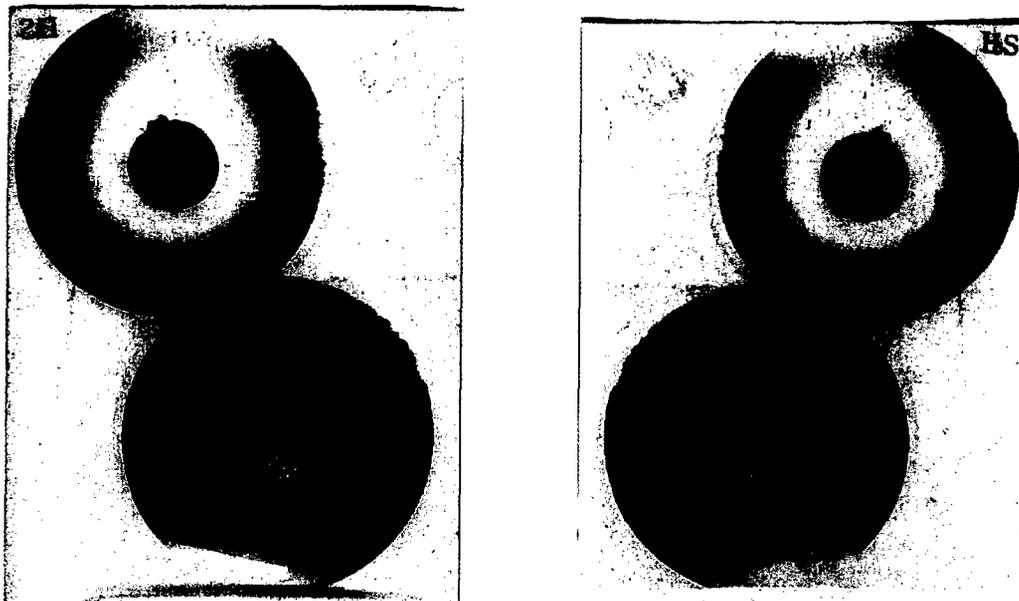
Figure 28. Photographs front and back of pressure-sensitive film after two drops in the dropweight machine using glass anvils. The top drop was onto a 5mm diameter, 0.7mm thick nylon 6 specimen. The bottom drop was glass anvil onto glass anvil. The red colour indicates high pressure.
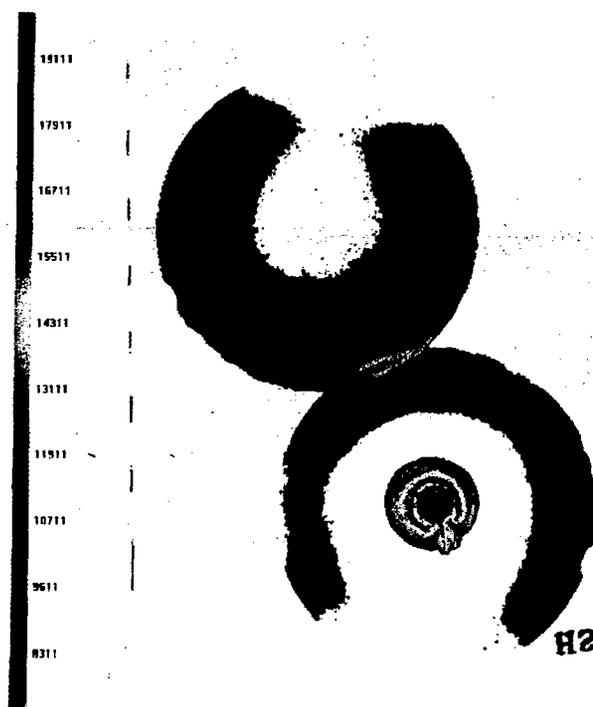
Figure 29. False-colour maps of pressure distribution computed by the manufacturer for the pressure sensitive film shown in figure 28. As the humidity and temperature were not measured in these experiments, the results are qualitative only.

5. DEFLAGRATION-TO-DETONATION (DDT) STUDIES

As part of an on-going programme to investigate the properties of ultrafine energetic materials a number of techniques have been developed to probe their response to different stimuli. A few of the different techniques that have been utilised will be outlined below together with some results that have originated from them. There is not the scope in this summary to outline all of the techniques in detail, some references are provided for further reading.

Materials

The materials that have been used are ultrafine PETN and RDX prepared by a proprietary method by ICI Nobel, Ardeer, U.K. The material is composed of approximately micron sized primary grains that form loose sponge-like secondary agglomerations. The density of the loose powder on delivery is ~15 % of the theoretical maximum density (TMD). The ultrafine HNS that was used was HNS IV as supplied by Bofors, Sweden.

For comparison, conventional grain size materials were also provided by ICI and Bofors. These typically had a grain size of approximately 180 μm.

Photographic studies of confined materials

A steel confinement (figure 30) with an inlaid polycarbonate window has been developed that allows the photographic study of materials as reaction travels along the column. For studying phenomena such as the deflagration-to-detonation transition, the level of confinement available in this set-up is essential for the mechanism to operate.
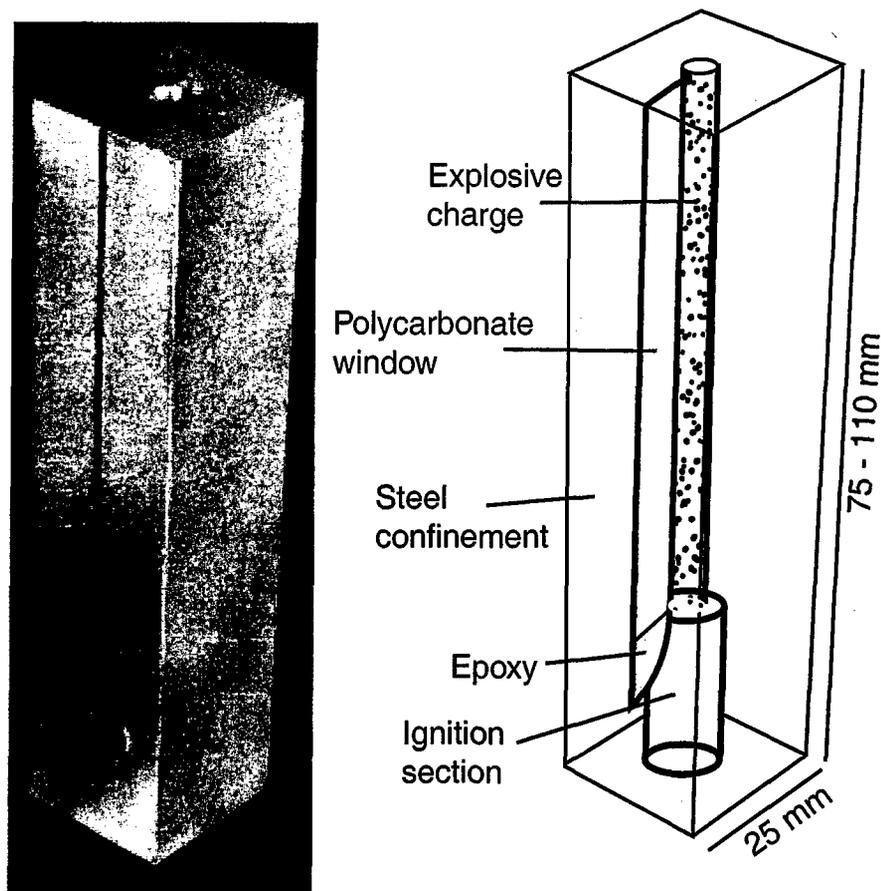
Figure 30. A photograph and a schematic of the windowed steel confinement.

Typically an image converter camera operating in streak mode is used to view the passage of reaction along the column. Because this level of confinement has been found to be necessary when studying the deflagration-to-detonation transition the charge is usually ignited at the base of the column using a specially designed hot-wire pyrotechnic ignition system. This allows direct ignition of the charge of secondary explosive without the venting of gases given off in the early stages of the reaction.
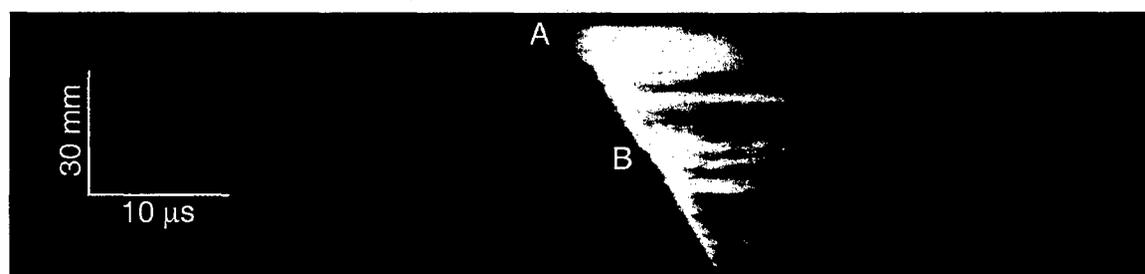
Figure 31. Streak record of a type II deflagration-to-detonation transition in ultrafine PETN. A - Point at which initiation takes place; B - Detonation wave travelling at 5.6 ± 0.3 mm μs⁻¹.

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The triggering of the high-speed camera is carried out using optical fibres positioned along the column. These can also be used to trigger fast responding detonators in order to send a detonation wave through the column prior to the charge undergoing a deflagration-to-detonation transition.
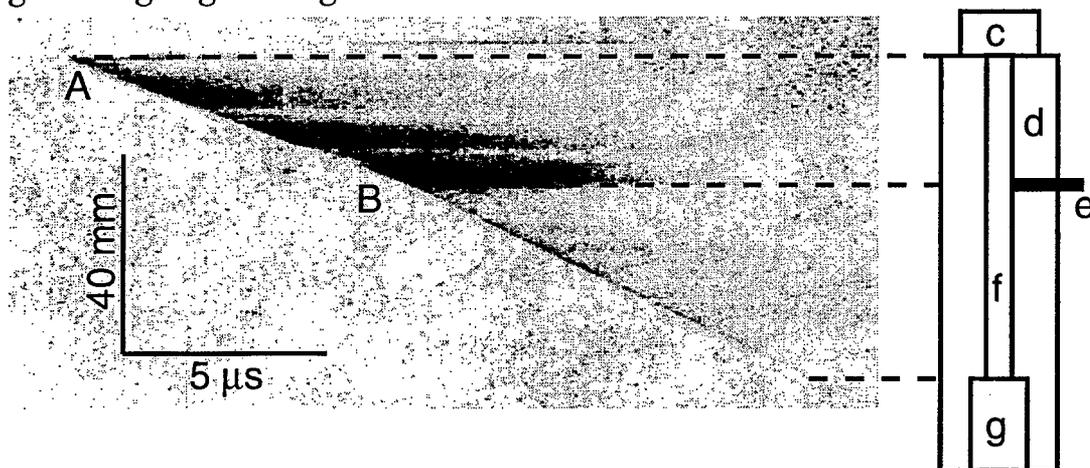
Figure 32. Negative streak record of an ultrafine PETN charge directly initiated after the reaction has reached an optical fibre placed along the column. A schematic of the charge is shown. A – point at which the charge is initiated; B – point reaction had reached when the detonation was initiated; c – EBW detonator; d – steel confinement; e – optical fibre; f – PETN charge; g – ignition section.

Directly initiated experiments – long duration shocks
Where the charges are directly initiated using a conventional detonator, polymethylmethacrylate (PMMA) confinements are used. These are typically 25 mm diameter, 25 mm long cylinders. PMMA offers visual access for high-speed photography. In their most basic guise, these confinements have been used for measuring detonation velocities in pressed charges of PETN.
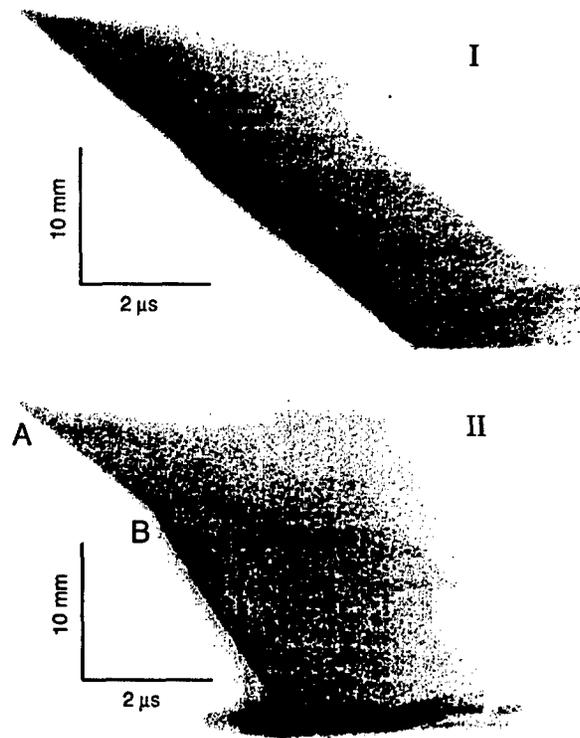
Figure 33. Negative streak records from two directly initiated charges of PETN. Charge I was a solid charge and has a steady detonation velocity of 4.1 ± 0.1 mm μs⁻¹. Charge II had a 1.5 mm diameter axial channel drilled through it. At the point of initiation 'A' the detonation velocity is 3.9 ± 0.1 mm μs⁻¹. At B this accelerates to 8.2 ± 0.1 mm μs⁻¹.

A variation on this experimental set-up has allowed a 'gap-test' to be developed that uses a C8 detonator as the donor charge and a PMMA disc as the 'gap'. The reproducibility of the results is particularly good with go/no-go boundaries being defined to within 0.01 mm providing the quality of the acceptor charge is good.

Directly initiated experiments – short duration shocks
A laser-driven flyer plate system has been developed within the laboratory for imparting short duration high pressure shocks. The laser propels millimetre diameter metre flyers with a thickness of the order a few microns at velocities up to 10 mm s⁻¹. It has been shown using this technique that only ultrafine materials are susceptible to initiation from shocks this thin. The system has been used for go/no-go studies of a range of materials to assess their suitability for use in system employing this type of initiator.
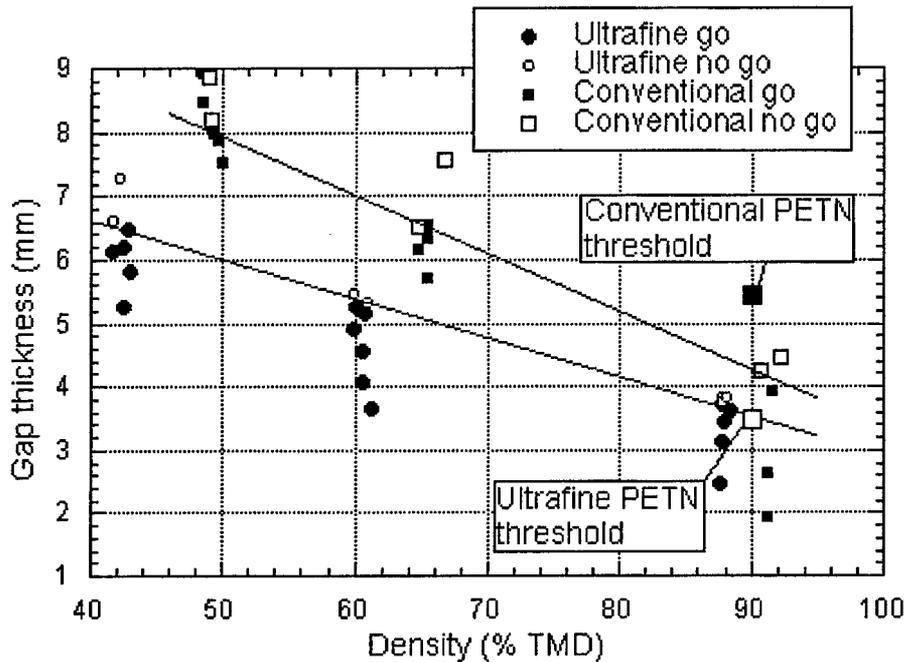
Figure 34. Plot of the results of gap testing comparing ultrafine and conventional RDX. Thresholds for PETN at a given density are also shown.

Thermocouples

Thin type K thermocouples have been used in a number of experiments involving energetic materials. Placed in columns during deflagration-to-detonation transitions they can only give an indication of the temperature during the fast reactions found later in the process. Due to their relatively low thermal mass the response time is sufficiently good to give temperature measurements during the slow build up prior to a type II DDT event. The confinements used for these experiments are cylindrical steel tubes.
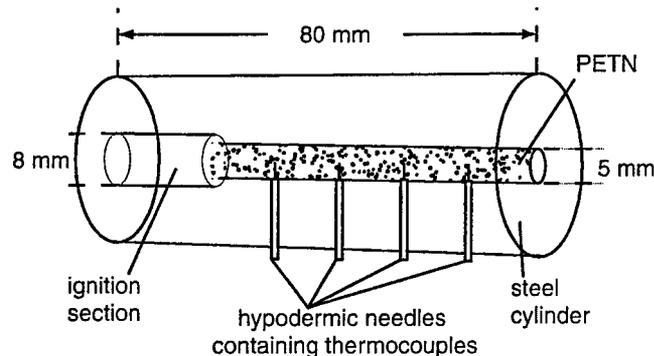
Figure 35. Schematic showing the way in which thermocouples have been inserted into the energetic column for monitoring the build-up to a type II DDT event.
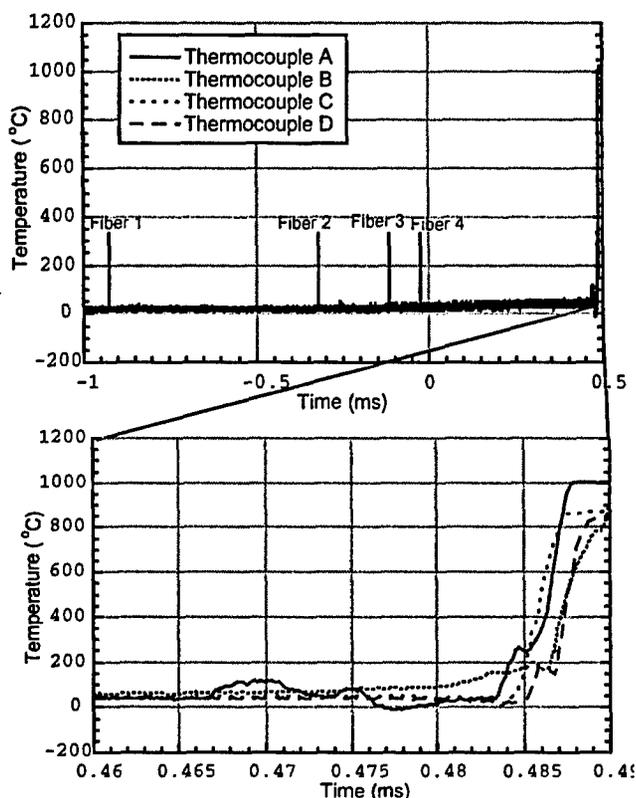
Figure 36. The output from four thermocouples placed in a column of ultrafine PETN during a type II DDT event.

The thermocouples have also been used for monitoring the temperature in an ignition cell that is used for critical hot-spot studies.

Recent publications from our research group in this area
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J.E. Field, M.J. Gifford and W.G. Proud, "Hot-spot mechanisms for explosives", in "Proceedings of NIMIC Workshop on Small Scale Testing of Energetic Materials"; Florida, USA, January 2000, publ. NATO Insensitive Munitions Information Center.

M.J. Gifford, "The role of hot-spots in the ignition and growth of explosion" PhD Thesis, University of Cambridge, 2000.

M.J. Gifford, M.W. Greenaway, W.G. Proud and J.E. Field, "Properties of ultrafine energetic materials", in "Theory and Practice of Energetic Materials", ed. L. Chen and C. Feng, pp. 3-11, publ. China Science and Technology Press, Beijing (2001)

M.J. Gifford, P.E. Luebcke and J.E. Field. "A new mechanism for deflagration-to-detonation in porous granular explosives", *J. Appl. Phys.* **86** (1999) 1749-1753.

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M.J. Gifford, W.G. Proud and J.E. Field, "Observations on type II deflagration-to-detonation transitions", in "Proceedings of the APS Meeting on Shock Compression of Condensed Matter- 2001" (to be published by American Physical Society) (Paper was winner of Tektronix prize)

M.J. Gifford, W.G. Proud, J.E. Field, "Development of a method for quantification of hot-spots" *Thermochim. Acta* **384** (2002) 285-290.

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