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Publisher *Taylor & Francis*

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## Journal of Energetic Materials

Publication details, including instructions for authors and subscription information:

<http://www.informaworld.com/smpp/title~content=t713770432>

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**To cite this Article** Waschl, J. A.(1998) 'An investigation of PETN sensitivity to small scale flyer plate impact', Journal of Energetic Materials, 16: 4, 279 – 288

**To link to this Article:** DOI: 10.1080/07370659808230235

**URL:** <http://dx.doi.org/10.1080/07370659808230235>

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# AN INVESTIGATION OF PETN SENSITIVITY TO SMALL SCALE FLYER PLATE IMPACT

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## ABSTRACT

The sensitivity of pentaerythritol tetranitrate (PETN) to small scale flyer plate impact has been assessed over the specific surface area (SSA) range 0.1 to 2.5 m<sup>2</sup>/g and for pressing densities ranging from 85% to 97% theoretical maximum density. The sensitivity was found to depend on SSA/mean particle size and density. The highest sensitivity was found at high SSA/low mean particle size and low density. Below a SSA of 0.75 m<sup>2</sup>/g or above a mean particle size of 30 μm, spot size effects appeared to have determined the ease with which PETN was detonated.

## INTRODUCTION

Gap test examinations of pentaerythritol tetranitrate (PETN) have indicated that the sensitivity of PETN decreased as the specific surface area (SSA) increased<sup>1</sup> or the particle size increased<sup>2</sup>. The tests were conducted over a SSA range of 0.2 - 1.8 m<sup>2</sup>/g and a particle size range of ~ 44 - 125 μm. The result was confirmed over the density range 0.75 - 1.6 g/cm<sup>3</sup>.

Under short shock conditions, a different response has been found for hexanitrostilbene (HNS)<sup>3</sup>, another heterogeneous explosive. In the HNS study the sensitivity initially increased, then remained relatively steady and then decreased over the SSA range 1 to 40 m<sup>2</sup>/g. Apparently the ignition and buildup mechanism is dependent on the shock duration and the explosive morphology<sup>4,5,6</sup>.

Using 1.57 mm diameter Kapton flyer plates, Schwarz<sup>4</sup> showed that increasing the pressing density of the PETN acceptor pellet caused the impact sensitivity to decrease. Two other granular explosives, HNS<sup>3</sup> and TATB<sup>7</sup> have shown a similar trend.

Journal of Energetic Materials Vol. 16, 279-288 (1998)  
Published in 1998 by Dowden, Brodman & Devine, Inc.

The purpose of this study was to further investigate the sensitivity of PETN to a short duration shock derived from a small scale flyer plate impact. Of particular interest was the effect of SSA, density, and flyer plate size on the sensitivity.

In the next section the approaches employed to assess the sensitivity of the PETN are detailed. The results of these experiments are provided in section 3 and discussed in section 4. In section 5 the conclusions of this study are presented.

### EXPERIMENTAL

Samples available for this study are listed in Table I. The PETN samples were subjected to a number of tests including scanning electron microscopy (SEM - Cambridge Instruments Model S250 MK11); SSA analysis (ASAP 2000 Surface Area Analyser or Micrometrics Flowsorb II 2300); and particle size measurements ( Malvern 2600/3600 Particle Sizer).

The residual solvent content of several sample specimens was determined by infrared spectroscopy. Solvents considered were acetone and ethanol. Two sample specimens were reanalysed at an interval of several months in order to determine whether any of the residual solvent would evaporate from the samples during that time. In addition CO<sub>2</sub> content was also measured. Differential scanning calorimetry (DSC) was conducted on several specimens to determine the purity of the samples.

The PETN samples were pressed into cylindrical pellets of about 50 mg each. The pellets were approximately 4 mm in diameter and between 2.0 and 2.5 mm long. The standard pellet pressing density was 90% of theoretical maximum density (TMD). A number of samples were also pressed to densities between 85% and 97% TMD. Sample sensitivity was examined via a previously described exploding foil flyer plate generator<sup>3</sup>. Bridge sizes employed were 125, 250 and 375  $\mu\text{m}$  wide.

**TABLE 1.** Characteristics of the PETN samples.

Sample	IR spectroscopy (%)		CO <sub>2</sub> (%)	DSC melting onset (K)	SSA (m <sup>2</sup> /g)	Mean Particle Size (μm)
	Acetone	Ethanol				
Base					0.14	54
4/90					0.81	58
6/86	0.6	not detected	not detected	414.1	0.84	22
7A/90	not detected	not detected			0.75	30
9/90				414.0	0.83	26
10/90	1	not detected			0.93	18
11/90					1.49	16
12/90	not detected	not detected	not detected		-	46
13/90	1	0.2			0.79	15
18/90					2.46	7
19/90	0.3	0.05			0.84	21
19A/90					0.42	-
21/90	0.5	not detected	0.2		2.18	10
8/91					1.06	22

The voltage across the bridge was monitored during bridge burst. The peak in the voltage trace was taken as the time of bridge burst<sup>3</sup>. For a number of the shots, a piezoelectric time of arrival detector (TOAD) was placed over the top surface of the explosive to detect detonation breakout<sup>3</sup>. The detonation breakout time, the bridge burst time and the flyer plate transit time were used to estimate the total run time of the detonation wave through the explosive. The difference between this estimated total run time and the theoretical run time based on the known velocity of detonation of PETN provides the excess transit time. For prompt initiation, the excess transit time is close to zero. All measurements were recorded on a digital oscilloscope (Tektronix DSA 602).

The Bruceton technique<sup>11</sup> was employed to determine the mean firing energy level ( $E_{50\%}$ ). Several exploratory shots were fired before the Bruceton set commenced in order to determine a starting point near  $E_{50\%}$ . The Bruceton step size was 5 mJ or 10 mJ and the analysis was usually based on a set of twenty shots. The number of shots was limited by the small quantity of explosives available. Additional cursory evaluations of a number of the explosive samples were also undertaken. For these samples, an estimate of  $E_{50\%}$  was determined from less than ten shots and quoted as a range.

## RESULTS

The low solvent and  $\text{CO}_2$  content shown in Table I indicate that the samples are almost pure; consistent with the onset of melting result. The solvent quantities did not change over a period of several months for the two samples reanalysed. The small quantity of  $\text{CO}_2$  that was detected was probably from the dry ice used in the manufacture of the sample.

Figure I is a plot of the relative  $E_{50\%}$  as a function of SSA. A similar trend for decreasing mean particle size was found. The data were normalized to  $E_{50\%}$  for PETN sample 6/86 (SSA 0.84  $\text{m}^2/\text{g}$ , 90% TMD) and for 250  $\mu\text{m}$  wide bridges. The error bars indicate a 95% confidence interval. The arrow indicates that the mean firing energy value exceeded the maximum firing energy employed in the experiments.

A comparison of the sensitivity of PETN as a function of bridge size is shown in Table II. In those cases where about ten shots were conducted an estimated range for  $E_{50\%}$  is shown. A greater than symbol indicates that the mean firing energy value exceeded the maximum firing energy employed in the experiments.

Usually detonation of the PETN pellet was evident from the damage to the stripline and surrounds, however, the recorded excess transit times were occasionally calculated at hundreds of nanoseconds instead of tens of nanoseconds. The event associated with this long excess time, although not a detonation,

was difficult to classify as often no pellet remained and the damage to the stripline and pellet holder was less than for a detonation. These events were termed 'low order' when significant damage was sustained by the stripline. When little or no damage was evident the event was classified as a no fire.

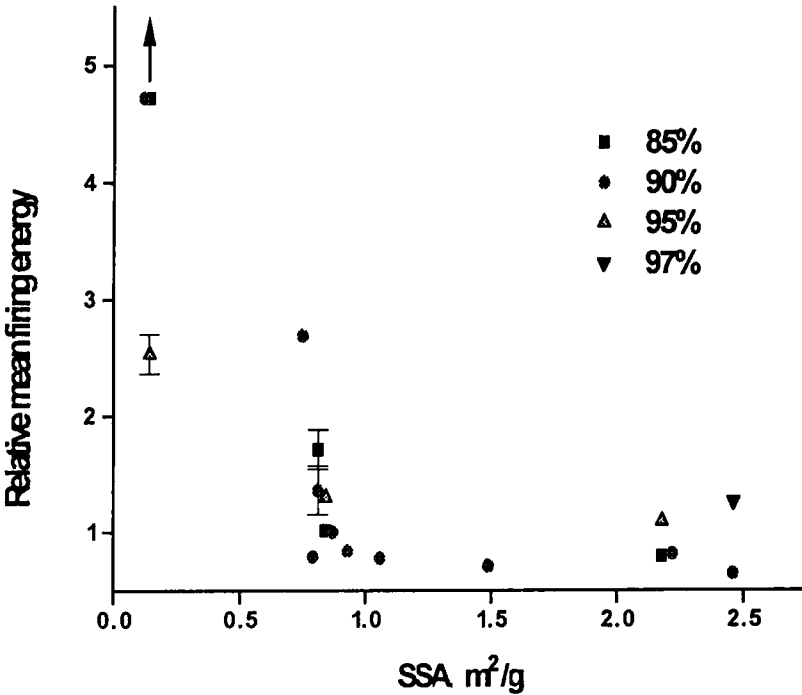


FIGURE I

Normalised mean firing energy levels for PETN as a function of SSA. Pressing densities for PETN were 85%, 90%, 95%, and 97% TMD. The error bars indicate a 95% confidence interval, otherwise the range is covered by the individual markers. The arrow indicates that the mean firing energy value exceeded the maximum firing energy employed in the experiments.

**TABLE 2.** Flyer plate impact sensitivity of various PETN samples pressed to 90% TMD. Flyer plate dimensions were 125, 250, and 375  $\mu\text{m}$  wide. The data were normalized to  $E_{50\%}$  for PETN sample 6/86 (SSA 0.84  $\text{m}^2/\text{g}$ , 90% TMD) and for 250  $\mu\text{m}$  wide bridges.

Relative mean firing energy					
Sample	Base	12/90	6/86	21/90	18/90
SSA ( $\text{m}^2/\text{g}$ )	0.14	-	0.84	2.18	2.46
Mean particle size ( $\mu\text{m}$ )	54	16	22	10	7
Bridge size ( $\mu\text{m}$ )					
125	> 4.72	> 4.72	1.11 $\pm$ 0.07	0.66 $\pm$ 0.08	0.56 $\pm$ 0.05
250	> 4.72	> 4.72	1.00 $\pm$ 0.02	0.81 $\pm$ 0.02	0.64 $\pm$ 0.03
375	3.20 $\pm$ 0.17	> 4.72	1.35 $\pm$ 0.07	1.08 $\pm$ 0.07	-

### DISCUSSION

The failure to detonate the low SSA samples is probably a manifestation of the minimum spot size of the PETN samples being approached. As the minimum spot size is approximately related to the failure diameter it is reasonable to expect that the minimum spot size will increase with increasing particle size in a similar fashion to the failure diameter<sup>9</sup>.

For the same explosive sample, as the bridge size decreases the  $E_{50\%}$  also decreases; reflecting the reduction in energy required to launch the smaller flyer plate. A change in the trend is indicative of encroachment on the minimum spot size. The results of Table II provide evidence that the minimum spot size

for the Base sample is near 375  $\mu\text{m}$ , and is probably greater than 375  $\mu\text{m}$  for 12/90. For all the other samples shown in Table II, the minimum spot size is less than 125  $\mu\text{m}$ , although the slight increase in  $E_{50\%}$  for 125  $\mu\text{m}$  bridges against 6/86 suggests that for 6/86, the minimum spot size is again being approached when using the smallest bridge size.

The relationship found between  $E_{50\%}$  and bridge size for the Base sample provides a clue to the importance of void numbers in determining the sensitivity of the low SSA samples. As the bridge size increases more voids may be involved in the decomposition of the explosive. Therefore, if a larger bridge results in a lower firing energy (and therefore a lower flyer plate velocity) than a smaller bridge, as happens for the Base sample in Table II, then the number of voids that form hot spots is the critical factor in determining the sensitivity of the sample. This also indicates that the minimum spot size for low SSA PETN is dependent on a minimum number of voids.

In higher SSA samples, there are many smaller voids that are more closely packed together. Thus for higher SSA samples there is no shortage of voids and once the material is ignited, the rapid grain burning achievable<sup>10</sup> results in energy being released to the shock wave. The formation of a detonation wave follows.

The low firing energy levels for PETN at 90% TMD and over the SSA range 0.75 - 2.5  $\text{m}^2/\text{g}$  (Figure I), indicate that an optimum combination of the void number and volume has been found for the 250  $\mu\text{m}$  bridges. For 250  $\mu\text{m}$  bridges against HNS, the optimum combination occurs from 10 to 20  $\text{m}^2/\text{g}$  after which the sensitivity again begins to decrease<sup>3</sup>. It would be interesting to investigate whether such a reversal was present in PETN. The difficulty at this stage, however, is in the production of higher SSA PETN.

In general the  $E_{50\%}$  points are quoted with errors of less than  $\pm 10\%$ . Only batch 4/90 at both 85% and 90% TMD and Base at 95% TMD have much larger uncertainty. For the 4/90 sample the uncertainty may be a reflection



of the diversity in the microstructure of the sample. Sample 4/90 consists of irregular shapes with rough flaky surfaces that were not exhibited by any of the other samples investigated. Thus the rich morphology of 4/90 may affect the range over which detonation can be achieved. For the Base sample the uncertainty is due to the difficulty in obtaining reliable initiation when operating near the minimum spot size.

Decreasing the density increases both the void volume and the number density of the voids. As the voids grow in size and number density, the separation between the voids must also decrease. Under shock impact, the larger void volume can be expected to generate larger hot spots that may coalesce more quickly than at higher densities. The energy released is therefore coupled to the advancing shock more efficiently at the lower density. Hence, the explosive is expected to detonate at a lower impact pressure<sup>11</sup> or firing energy.

From the PETN data of Figure I, two exceptions to this expected trend are apparent; samples Base and 4/90 - both consisting of large particles. An explanation for the reversal in the expected trend may be found in the pressing of the pellets. It is possible that during the pressing operation that the particles may be fractured<sup>12</sup> producing smaller mean particle size distributions than in the original powder form. Such a change in particle size would result in increased sensitivity as the pressing density increased beyond some maximum. This effect is probably dependent on the original crystal size of the explosive. Note as the SSA increases that the sensitivity of the PETN continues to decrease up to a pressing density of 97% TMD (the limit of the press). Finer material is apparently more difficult to fracture during pressing.

As the purity of the PETN samples are all about equal it is difficult to determine a sensitivity effect due to purity. From the available data in Table I an indication of the effect of solvent content can be made. The acetone content of sample 10/90 is shown to be twice that of sample 21/90. Sample 10/90 has a lower SSA than 21/90. Therefore 10/90 would not be expected to be more sensitive than 21/90. As Figure I

indicates, the sensitivity of 10/90 and 21/90 is essentially the same. Thus it seems reasonable that solvent content of less than 1% is not expected to be an important factor in determining the impact sensitivity of these PETN samples.

### CONCLUSION

The sensitivity of PETN to small scale (250  $\mu\text{m}$  wide) flyer plate impact was found to increase rapidly for an SSA range of 0.1 to 0.75  $\text{m}^2/\text{g}$  when pressed to 90% TMD. This rapid change in sensitivity is due to the increasing spot size of the PETN. Above 0.75  $\text{m}^2/\text{g}$  the sensitivity was found to be relatively constant and independent of SSA to a maximum SSA of about 2.5  $\text{m}^2/\text{g}$ . Similar comments apply when assessing the sensitivity in terms of particle size. The sensitivity of the PETN to 250  $\mu\text{m}$  flyer plates impact appears to become independent of particle size for mean particle sizes smaller than about 25 - 30  $\mu\text{m}$  and is related to an optimum combination of void volume and number. At low SSA, PETN exhibits an increase in sensitivity as the density increases from near 90% TMD. This effect, which is due to crystal fracture, was not observed for high SSA PETN.

Above an SSA of 0.75  $\text{m}^2/\text{g}$ , the PETN samples were initiated promptly. Below that range, the PETN samples exhibited unpredictable behaviour which included long excess times and reduced output. This is consistent with the development of an incomplete reaction.

The purity of the PETN samples produced was high. The results indicated that residual solvent content of up to 1% acetone would not influence the sensitivity of the PETN samples.

### ACKNOWLEDGMENTS

B. Jones conducted all the SSA measurements and pressed and fired most of the PETN, L. Redman produced many of the PETN batches, D. Davidson carried out the IR spectroscopy, I. McDermott and F. Griffo provided the micrographs, and M Fitzgerald provided the DSC results.

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