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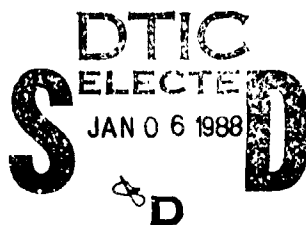
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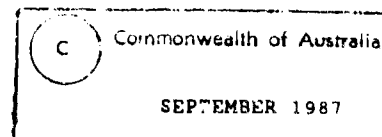MELBOURNE, VICTORIA

REPORT

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MECHANISM OF AND PARTICLE SIZE EFFECTS ON
SHOCK SENSITIVITY OF HETEROGENEOUS PRESSED EXPLOSIVES:
PRELIMINARY ASSESSMENT OF BINDERLESS RDX IN FUZE TRAINS

Robert J. Spear and Victor Nanut

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ABSTRACT

Shock initiation of pressed heterogeneous explosives has been reviewed. The key processes of ignition and buildup and their relative importance under sustained and short duration shocks are described. Particle size effects on shock sensitivity are shown to depend on density and shock duration. $M_{50\%}$ values from gap tests often increase as particle size decreases. Below a critical particle size $M_{50\%}$ then decreases, as predicted from theoretical modelling. This trend was observed here for RDX. Assessment of fine RDX for fuze trains indicated its potential suitability.

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CONTENTS

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1. INTRODUCTION

The initiation and buildup to detonation of solid heterogeneous
pressed explosives is a key process for proper functioning of explosive
trains. Despite the importance of knowledge of this process to ordnance
design, it was not till the early 1960s that the first detailed studies were
published [1-4]. Investigation of these processes, both experimentally and
via theoretical modelling, has expanded considerably over the ensuing twenty-
five years. Two extensive surveys, covering the state of knowledge based on
published and unpublished data up till about 1980, have been published
[5,6]. Lee and Tarver [5] have addressed the subject from a theoretician's
point of view, developing computational models to predict shock induced
initiation of detonation. Price [6] represents the experimentalist, giving
detailed descriptions and comparisons of test methods and the effect of sample
variables on the results obtained.

The purpose of the study described in this report was two-fold. The
first aim was to present as detailed a physical picture as current knowledge
permits of the processes occurring during shock initiation and subsequent
buildup to detonation in heterogeneous pressed explosives. The principal
consideration was to assess and describe the effect of sample parameters on
these processes, particularly the key parameter of explosive particle (grain)
size. The second aim was to commence a study of the suitability of the use of
very fine binderless RDX in fuze trains. Results are presented here for RDX
of particle size <5 μm up to 300 μm subjected to sustained shock.

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2. INITIATION BY SHOCK

It is generally agreed that initiation of heterogeneous solid explosives occurs at inhomogeneities in the pressed solid, where interaction with the incident shock wave produces small localised regions of high temperature ("hot spots"). Energy released by growth of chemical reaction from the hot spots can subsequently become self-sustaining, reinforcing the incident shock wave and leading to detonation. A number of possible mechanisms for formation of the hot spots have been proposed and largely fall into two types.

(i) Shock induced collapse of voids within the filling. Mechanisms for energy generation include impact of the void front onto the void rear and associated microjetting [1,5,7,8], and viscoplastic work done on explosive at the void peripheries [5,7,9,10,11]. The stagnation (of microjets) theory proposed by Seely [12] and elaborated by Stresau [13] is closely related to these mechanisms.

(ii) Impact [13] and friction [5,7,13] between grains, and viscous friction within deforming grains [7]. The related process of shear banding has also been proposed [10,14].

Howe et al [15] have concluded on the basis of theoretical models that the relationship between particle size and threshold pressure for initiation can only be explained if both void collapse and frictional processes are operating. It is generally agreed that shock interaction with voids is the primary mechanism, with friction playing a secondary role. Note that Lee et al [16] have concluded that pore (void) collapse may not be the dominant mechanism for producing reaction sites during shock initiation of TATB at densities below 1.65 Mg/m^3. Experimental evidence has been variously interpreted to mean that shock sensitivity depends principally on the number [1], volume [17] and surface area [15] of voids, but the relative importance of each contribution is not known. Theoretical modelling of shock initiation in heterogeneous explosives has largely centred on the void collapse mechanism [7,8,18] but more recently other mechanisms have been treated [10]. Adiabatic compression of interstitial gases, which is the most important mechanism for initiation under impact [19], is not normally operative under shock conditions [2,20].

The molecular processes occurring during shock initiation are not known. Walker et al in a series of papers [21,22] have proposed that the shock initially causes cleavage of the (explosive) molecules into ions and free radicals. If these reactive species are formed in sufficiently high local concentration they could grow to produce a self-sustaining exothermic decomposition. Isotope labelling studies [23] have indicated that the same bonds (C_α-H) are broken in the rate determining step for both thermal decomposition and shock initiation of TNT. An extension of this concept is that the incident shock does not need to be degraded to thermal energy, i.e. hot spots, but initiation can occur directly by bond shear [22]. A similar "tribochemical" mechanism was proposed some years previously [19], but both these mechanisms are currently considered to be unimportant.

3. BUILDUP TO DETONATION

Initiation (ignition) is followed either by buildup to self-sustaining exothermic reaction and ultimately detonation, or by failure to propagate due to energy losses. Chemical energy release in the buildup and subsequent detonation occurs by grain burning as elaborated by Eyring [24]. However there is considerable disagreement on whether the rate of grain burning or physical processes such as permeability, thermal conduction, convection and diffusivity are the key parameters controlling this process.

Most of the evidence for grain burning being the dominant parameter in buildup following shock initiation comes from study of particle size effects [6,10,13,15,17]. In essence, the higher shock sensitivity of explosive compacts pressed from small particle size materials to very short duration pulses has been attributed to their higher rates of grain burning. Although the burning rate of materials such as HMX is faster for small particle fractions [25], particle size effects on both deflagration-to-detonation transition (DDT) [26] and strand burning [27] of low density high explosives are not consistent with this picture: larger particle size materials show more rapid buildup. One explanation of this apparent contradiction is that there is an abrupt increase in the pressure exponent at higher pressures, and there is some evidence to support this [28]. Large increase in burn rates in closed bomb tests due to crystal breakup at higher pressures has been observed [25], and could also contribute to this disparity. Lee and Tarver [5] concluded that the growth of reaction from the ignition sites apparently proceeds at rates that exceed the linear burn rate-pressure dependence of laminar deflagration in explosives.

A basically different picture has been proposed by Mader [8,29]; energy released close to the shock front by thermal explosions of the hot spots can result in shock acceleration. The strengthened shock then produces increasing numbers of hot spots till the shock ultimately builds to a detonation wave.

In summary, the effect of a number of key parameters which could define the key processes in buildup, and which could be used to predict buildup/propagation success, has not been defined with any certainty.

4. THE RELATIVE IMPORTANCE OF INITIATION AND BUILDUP

Experimental evidence is consistent with the ignition process and the buildup to detonation process being separate [17]. Their relative importance in the overall formation of self-sustaining detonation has two aspects. In terms of energy release, it is now accepted that energy contribution from the hot spots formed at initiation is very small relative to the energy released by grain-burning during buildup. ' In other words, initiation occurs by formation of hot spots at the shock front, then these hot

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spots grow and burn in the reaction zone to generate the energy necessary to build to and sustain detonation.

Whether initiation or buildup will be the key process leading to detonation is critically dependent on the nature of the incident shock. Walker and Wasley [30] proposed, on the basis of experiments using short duration/high pressure shocks delivered to nearly voidless explosives by flyer plates, that there existed a "critical energy" criterion for initiation. This criterion states that there is a critical energy per unit area which must be delivered to an explosive to obtain detonation. The relationship

$$\frac{P^2 t}{\rho_o U} \; = \; \text{constant} \quad \text{or} \quad Put \; = \; \text{constant (since } P = \rho_o u U)$$

can be derived [30] where

        P is the shock pressure in the explosive
        t is the shock duration
        ρ_o is the initial density of the explosive
        U is the shock velocity in the explosive
        u is the particle velocity in the explosive

Since $\rho_o U$ changes only very slowly with increasing P, the relationship reduces to $P^2 t \; \cong \;$ constant, the form in which it is normally used. de Longueville et al [31] have derived a related "critical time" concept for shock initiation.

The critical energy criterion is now considered to be a useful engineering development guide applicable to voidless explosives of the conventional CHNO composition. It is not a general relation; although the shock sensitivity of some explosives obeys $P^2 t$ = constant over a considerable range of t [30,31,33], most explosives do so only at very short pulse durations, typically < 1 μs [31-34], while the behaviour of others does not correlate at all [31,33]. The shock initiation of heterogeneous explosives subjected to long (or sustained) shocks in excess of 1 μs duration depends only on the incident shock pressure. Most standard shock sensitivity tests such as gap tests employ sustained shocks. In addition, lower density leads to increased sensitivity to sustained shock but decreased sensitivity to short duration shock.

Howe et al [15] proposed, on the basis of the different observed behaviour under short duration and sustained shock, that $P^2 t$ = constant correlated with buildup, while pressure dependent initiation correlated with the initiation stage.

Tarver et al [35] have recently published a revision of their earlier [5] phenomenological model for shock initiation of heterogeneous explosives. This revision [35] specifically deals with short pulse duration shocks, and the inability of the earlier model to predict accurately behaviour under these conditions. They have now proposed a three step process for initiation by short shock pulses: initiation by formation of hot spots, then slow growth of reaction from the isolated hot spots somewhat analogous to a deflagration in DDT, and finally rapid completion of the reaction by

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coalescence of hot spots. At longer pulse lengths, where initiation becomes
the determining process, it is not necessary to split the buildup into two
processes although it may still occur in this manner. Johnson, Tang and
Forest [36] have also recently published a numerical model of shock initiation
of heterogeneous explosives and have surveyed previously published models,
particularly those relating to polymer bonded explosives (PBXs).

The physical picture which emerges from these studies shows a
gradation of behaviour depending on the incident shock. Very high
pressure/short duration shocks result in up to 20-30% of the explosive being
ignited as hot spots [35]. The success or failure to grow to detonation is
determined solely in the buildup stage, and the calculations of Tarver et al
[35] strongly suggest that this buildup occurs as discrete slow and fast
steps. Presumably the critical stage is the slow step roughly corresponding
to deflagration in DDT. Low pressure sustained shocks result in only a few
tenths of a percent of the explosive being ignited as hot spots [36], and it
is this ignition stage which is crucial to success or failure to grow to
detonation. Further support for this proposition can be found in von Holle
and Tarver's measurement of hot-spot temperatures in shocked explosives using
time resolved infrared radiometry [37]; only relatively small amounts of
explosive ignited close to the shock front for sustained shocks.

Additional insight into the reasons these hot spots grow or fail
will be provided in the following section.

5. THE RELATIONSHIP BETWEEN EXPLOSIVE PARTICLE
SIZE AND SHOCK SENSITIVITY

One of the key parameters controlling the macroscopic behaviour of
shocked pressed explosives is particle size. The short but succint statement
by Stresau and Kennedy [13] best summarises available knowledge: "fine-
particle powders are often harder to ignite than coarse powders, but reactions
in fine powders grow to detonation more rapidly once ignited." Much of the
insight into the microscopic processes which are occurring during initiation
and buildup is derived from studies of particle size effects, as should be
apparent from the preceding sections.

The relationship between shock sensitivity and explosive particle
size has been reported for quite a number of pure pressed materials:
TNT [1,15,17], RDX [31,38,39], HMX [13,20], PETN [12,38-41], tetryl
[12,38,42], HNS [43,44], TATB [16,32,45] and HNAB [34]. A number of other
studies dealing with particle size effects of some of these explosives in
formulations have been published. In general they follow the behaviour of
the pure materials, and no attempt has been made to cite them here. It should
be noted that most of these studies are restricted to comparison of "coarse"
with "fine", ie. only two or sometimes three size fractions are compared.

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Analysis of the data in the references cited above plus others not cited reveals two key points.

(1)   Particle size effects on shock sensitivity can only be discussed in conjunction with two other parameters: density and shock duration.

(2)   The term "shock sensitivity" is used fairly loosely in the literature and the two major uses are not necessarily consistent. Researchers from the atomic weapons laboratories usually equate sensitivity with run distance to detonation, as typified by Pop plots [46]; shorter run distance at the same incident shock pressure is equated with higher sensitivity. In contrast the other criterion is threshold pressure for initiation, or the more readily determined shock pressure for 50% initiation probability; lower threshold (or 50% initiation) pressure means higher sensitivity*. In the remainder of this paper we will be largely using this definition.

Particle size effects on heterogeneous explosives pressed to high relative density, eg. > 98 %TMD typically achieved by isostatic pressing, are clear cut; shock sensitivity increases as particle size decreases. This behaviour is also followed by cast and homogeneous explosives [6,12,48] such as composition B. At these densities approaching crystal density, behaviour under shock is approaching that of homogeneous liquid and cast explosives [48]. One explanation given for the effect of particle size at high density is that these charges have very low permeability and shock sensitivity should therefore be a function of pore surface area which will increase with decrease in particle size [4].

At low (relative) densities of 95 %TMD and less, ie. accessible by normal pressing operations, pronounced dependency on particle size is often observed. The following discussion compares behaviour at identical %TMD.

For sustained shocks such as in gap tests, reaction thresholds as measured by incident shock pressure are nearly always lower for larger particle size materials [13,15,17,38,43] while pressures for 50% detonation probability** are also often lower [12,32,38,40,42]. One explanation is that in relatively low density charges the area of surface exposed to reaction products, which will determine shock sensitivity, will be a function of

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*   An example of such a "confused use" of the term shock sensitivity can be seen in the paper by Taylor [47] where he equates shock sensitivity with shorter run distances in the Introduction yet uses examples derived from lower initiation thresholds in the text.

**   This is the result usually quoted from gap testing and is often referred to as the "50% threshold". It is not a true threshold and differences between the two can be seen in [12,38] for example.

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permeability which will decrease as the particle size decreases [4].
Alternatively the smaller grain size materials will have smaller voids and
hence smaller hot spots, thus energy losses to thermal conduction will be
higher [32].

As pulse duration decreases and shock pressure increases, there is
usually a reversal of behaviour and smaller particle size materials are more
shock sensitive [13,15,17,32,34,43,45]. Initiation under these conditions
was discussed in the previous section: a relatively high proportion of the
shocked explosive is ignited by the incident shock, and Howe et al [15] have
suggested that it is the higher rate of grain burning which enhances the
buildup success of small particle size materials. Another explanation is that
under these higher incident shock pressures the hot spots are hotter, the
reactions are consequently faster, and a state is reached where the number
rather than the size distribution of hot spots is important [32].

An illustration of the relative behaviour discussed in the preceding
two paragraphs is shown in Fig. 1. The curves suggest that threshold shock
pressure is lower for larger particle size materials but the initiation energy
(at short shock duration) is lower for smaller particle size materials. A
similar figure can be seen for PBXN-5 (HMX/Viton A 95:5) in ref. [13] and data
for RDX and TATB are listed in Refs. [16,31 and 45].

The relationship between shock sensitivity and explosive particle
size has been modelled by Taylor [7,47]. Taylor assumed that hot spot
formation occurred by void collapse and calculated hot spot temperatures and
thermal energies for materials at the same density but with decreasing
particle size. It was found that hot spot temperature remained roughly
constant as particle size decreased till a critical pore size was reached;
here the temperature achieved decreased substantially. This decrease in hot
spot temperature was assumed to result in a decrease in ability of the hot
spots to grow (to buildup). de Longueville [31] had earlier suggested that
hot spot temperature determined shock sensitivity.

Taylor then used this calculated data in conjunction with two
published observations,

(1)   explosives under sustained shock display higher chemical
       reactivity as particle size decreases [1,49,50]

(2)   coarser grained explosives exhibit lower reaction
       thresholds during sustained shocks [13,15,31,33],

to suggest that a reversal of shock sensitivity for sustained shock should
occur. That is, as particle size decreases, shock sensitivity increases due
to increased surface area which enhances buildup, but below an (unspecified)
particle size initiation probability decreases due to decrease in hot spot
temperature not compensated for by increased surface area. Taylor cites data
for HNS [44,45,50] as being consistent with these conclusions.

6. RELEVANCE TO THE USE OF BINDERLESS RDX IN FUZE TRAINS

Taylor's calculations are limited to void collapse as the sole mechanism for hot spot formation and Howe [15] has shown previously that this is not normally the case. In addition, the higher chemical reactivity of smaller particle size material, cited as (1) above, is rot necessarily normal behaviour. While it might be tempting to dismiss Taylor's predictions because they are possibly based on incorrect assumptions, if they do predict trends correctly they could have profound importance on future design of fuze trains. For example, reduction of explosive particle size leads to reduction in impact sensitiveness [51,52]. One proposal to use RDX in fuze trains while meeting current fuze/safety guidelines [53,54], ie no material more sensitive than tetryl may be used below the shutter, is to use very fine RDX. However this could be accompanied by reduced shock sensitivity since it is for very small particle size materials that Taylor's predictions seem to be observed. With the exception of HMS and TATB, most of the published data on particle size/shock sensitivity cited earlier refer to "large" particles, eg $75-500 + \mu m$.

We accordingly undertook an experimental study of the relationship between shock sensitivity and explosive particle size for RDX over a range of particle sizes from $< 5 \mu m$ to $300+ \mu m$. We set out to investigate the behaviour of these materials to both short and sustained shocks. The work described here is the first part, using the MRL small scale gap test [55] i.e. sustained shock. The study of the behaviour of these materials when subjected to short duration shock will be commenced following completion of test equipment currently being assembled; preliminary testing in slapper detonators has indicated a pronounced dependency on RDX particle size [56].

7. EXPERIMENTAL

7.1 Preparation of RDX Samples

7.1.1 Grade A Sieve Fractions

RDX Grade A, Class 1, was supplied water wet from Albion Explosives Factory. The material as received was passed successively through a series of sieves under water by gentle brushing. The sieves as used sequentially were 355, 300, 250, 212, 180, 150, 125, 106, 75 and 45 μm. The three samples used in this study, 250-300 μm, 125-150 μm and 75-106 μm consisted of material retained on the smaller pore size sieve and passing through the larger. Material retained on the sieve was rinsed off with distilled water and dried by suction filtration.

7.1.2 Grade E

RDX Grade E was supplied water wet by Weapons Systems Research Laboratory (WSRL), Salisbury. The material as received was dried by spreading thinly on paper and air drying with break up of agglomerates. Approximately two weeks was required to complete drying and reduce agglomeration such that handling properties were adequate. RDX Grade E was prepared by aqueous acetone precipitation of Grade A; full details have previously been published [57].

7.1.3 Ball Milled

Ball milled RDX, nominal size 4 , was supplied water wet by WSRL. The material was dried as described above for Grade E. Ball milling was carried out using RDX Grade A under inert solvent.

7.2 Characterisation of RDX Samples

7.2.1 Particle Size Measurements

Particle sizes were determined using a Malvern Particle Size Analyzer Model 2600/3600. The samples as slurries were dispersed using energy from an ultrasonic bath for 1 minute, then transferred to an optical cell and placed in the laser beam path. The finer particle size materials, Grade E and ball milled RDX, were dispersed in HPLC grade chloroform. The coarser materials were dispersed in distilled water with a small amount of Decon 90 surfactant.

Samples were prepared by dividing about 1 g of the powder into small fractions (10 to 20 mg) by using a rotary sample divider [58] constructed at MRL. Sample splitting of powder slurried in liquid was preferred to splitting the dry powder due to the poor flow of dry RDX powders especially for small particle size fractions.

The principle of the Malvern is based upon Fraunhoffer diffraction of a laser beam by small particles. The data thus generated is automatically collected and subsequent data reduction is performed using computer programmes supplied with the instrument. The results are presented as total mass per sampling band (of particle size range), not total number of particles per sampling band. The particle size distributions are obtained by fitting the light energy distribution to several particle size distribution models. The model which was found to be most applicable was a "model-independent" 16 parameter fit over 15 size bands.

7.2.2 Scanning Electron Microscopy

A Cambridge Instruments Model S250 MkII scanning electron microscope with a tungsten electron gun was used. The instrument was operated at

15-21 kV in the secondary electron mode. The samples were prepared by mounting crystals of the formulation with PVA adhesive onto a stub coated with PVA adhesive. The sample was then sputter coated with a conducting film of gold. Micrographs were generally obtained for tilt angles of 30 degrees.

7.2.3 Shock Sensitivity: MRL Small Scale Gap Test [55]

The MRL small scale gap test (SSGT) has been described previously in detail [55]. The system consists of a donor of a PETN filled exploding bridgewire (EBW) detonator, an acceptor of two 12.7 mm diameter x 12.7 mm height pressed cylinders of the explosive under study, with the gap being of laminated brass shim. Detonation of the acceptor is confirmed by a sharply defined dent in a 25 mm square x 12.7 mm thick mild steel witness block. A typical run consists of 25-30 firings conducted using the Bruceton staircase method [59], the result being expressed as the gap in mm at which detonation probability is 50%. Donor EBWs were UK Mk 3 supplied by AWRE Aldermaston.

RDX acceptor pellets were pressed to the required density on an Instron Universal Testing Machine operated as a press. Complete experimental details of this procedure are given in [60]. It was found that the pellets, particularly from the finer powders, possessed poor mechanical strength resulting in cracking or breaking upon ejection from the mould or subsequent handling. Two strategies were used to overcome this problem.

(i)   The RDX Grade A powders (2.50 g) were pressed in two sequential stages each of 1 min duration. The pellet was removed from the mould at the completion of the second 1 min period. In the case of the 125-150 μm and 75-106 μm sieve cuts a thin film of oleic acid was applied to the mould prior to addition of the powder; this facilitated ejection of the pellet. Pressing loads to achieve the required 90 %TMD were:

250-300 μm sieve cut; 1260 kg (76.6 MPa)
125-150 μm sieve cut; 1520 kg (92.4 MPa)
 75-106 μm sieve cut; 1700 kg (103.3 MPa)

(ii) The Grade E and ball milled RDX were first tamped into the mould then a cavity was formed in the mildly consolidated powder with a 3 mm diam. plunger. Acetone (0.02 mL) was injected into the cavity and the drift was placed lightly on top in the mould. After leaving the assembly in this condition for about 5 min, it was pressed for a single 3 min period. This treatment was necessary to form pellets strong enough for ejection from the mould; it also lowered the force necessary to press the pellets and facilitated their ejection. Pressing loads to achieve the desired 90 %TMD were:

Grade E: 1000 kg (60.8 MPa)
Ball milled: 350 kg (21.3 MPa)

Densities were determined by accurate weighing and dimensional measurements.

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7.2.4 Rotter Impact Sensitivity: Figure of Insensitiveness (F of I)

Impact sensitiveness was determined on a Rotter Apparatus [61] using a 5 kg weight. Results from 50 caps tested by the Bruceton procedure were used to determine the F of I values relative to RDX Grade G - 80. The values quoted are derived from the height for 50% initiation probability and are rounded to the nearest 5 units. Average gas volumes for positive results are also quoted.

8. RESULTS AND DISCUSSION

8.1 Characterisation of RDX Samples

Median particle sizes and 16-84% probability ranges for the five RDX samples are listed in Table 1 and the particle size distributions are depicted as bar charts in Figs 2 and 3. All samples exhibit quite narrow particle size ranges and, with the exception of the 75-106 μm and 125-150 μm samples (Fig. 2) there is minimal overlap with other samples. All the sieve cuts have a tendency to contain significant amounts of particles smaller than their lower sieve limit; this results mainly from fracture of smaller crystals from the bridged agglomerates during treatment in the ultrasonic bath (see Section 7.2.1).

Scanning electron micrographs (SEM) of four of the five samples at magnification X200 are shown in Figs 4a-d. The recrystallised Grade A samples (Figs 4a and b, and the 250-300 μm sieve cut not shown) have the typical rounded irregular appearance. Small crystals growing off the faces, referred to above, can clearly be seen. The Grade E and ball-milled materials exhibit a different structure which becomes more apparent at higher magnification (X1000 and X2000) in Figs 5a-c. The Grade E material consists of a range of shapes from elongated spheros to rounded rods and other unusual forms. Such a proportion of particles with large length to diameter ratio makes accurate determination of particle size very difficult. However the crystals are clearly much larger than the rounded ball-milled material and much smaller than the 75-106 μm Grade A sieve fraction.

8.2 Impact Sensitiveness

Rotter F of I was determined on only two of the RDX powders; the largest (250 μm) and smallest (3.9 μm). Results are listed in Table 1.

Of the two, the 3.9 μm ball-milled RDX exhibits lower sensitiveness, ie higher F of I. This F of I is similar to tetryl [61], and the clear trend would suggest that RDX of even smaller particle size should exhibit significantly lower impact sensitiveness than tetryl and thus meet the criterion for fuze safety guidelines [53,54]. US experience is that impact sensitivity can be reduced substantially if the RDX is sufficiently fine [62].

8.3   Shock Sensitivity

Results for shock sensitivity (SSGT) of compacts of the five RDX samples pressed nominally to 90.0 %TMD (1.622 Mg/m^3) are listed in Table 2. Binderless RDX typically exhibits relatively poor mechanical strength and ejection from pressing moulds and subsequent handling presented a problem. Since the MRL SSGT uses unconfined 12.7 mm diameter acceptor pellets, pressing suitable pellets to 90.0 %TMD proved difficult, particularly for the finest materials (Grade E and ball-milled). In these cases a very small volume (0.02 mL) of acetone was reproducibly added to the powder in the mould prior to pressing. Full details are described in Section 7.2.3. This process resulted in poor reproducibility of pellet densities although pressed under identical conditions; this can readily be seen in the density standard deviations which are substantially larger than those for the Grade A sieve cuts (Table 2). All mean densities were very close to 90.00% TMD.

The gaps for 50% initiation probabilities together with calculated standard deviations are plotted in Fig. 6; 95% probability limits are additionally cited in Table 2. The results for the 250-300 μm Grade A sieve cut and Grade E RDX had high standard deviations. This is often observed for larger particle sized material and results from the relatively large range of shock pressure from the true threshold through to full detonation [38]; in the MRL SSGT the criteria for "go" is a dent in the witness block and shallow or full dents are not differentiated. In the case of Grade E RDX, the principal cause was probably the high standard deviation of the acceptor pellet densities. However, the 3.9 μm ball-milled material also has a high standard deviation on pellet density but a low standard deviation on the $M_{50\%}$ result. This could mean that density dependence on shock sensitivity is decreasing at very small particle size, but further studies would be needed to substantiate this.

The clear trend observed in Fig. 6 is for the shock sensitivity of the pressed materials to increase steadily as median particle size decreases from 250 μm (Grade A 250-300 μm sieve cut) to 21.5 μm (Grade E). There is then a small decrease in shock sensitivity for the 3.9 μm ball-milled RDX, although the large standard deviation on the Grade E result makes this comparison not statistically significant. The large uncertainty in the Grade E result made examination of material of intermediate particle size, eg 12.5 μm, of little relevance. Although study of smaller (than 3.9 μm) RDX would have been very useful, the only possibility of obtaining sufficient material for a gap test was from the milling facilities at WSRL; shipment to MRL under water would have resulted in crystal growth to approximately the same size as the 3.9 μm RDX.

Scott [38] examined three RDX sieve cuts pressed to 1.517 Mg/m^3 (84 %TMD); 74-125 μm RDX was more shock sensitive than 177-250 μm RDX, but both were substantially more shock sensitive than sub-44 μm RDX. Similarly Roth [39] found the shock sensitivity trend for RDX fractions pressed to 1.54 Mg/m^3 (85.3 %TMD) to be 330 μm < 54 μm < 25 μm, but 8 μm RDX was substantially less shock sensitive. de Longueville et al [31] studied coarse (200-400 μm) and fine (40-80 μm) RDX pressed to 1.55 Mg/m^3 (86 %TMD). For sustained shock delivered by flyer plate they found the coarse RDX to have a

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lower shock threshold than the fine RDX. Chick [20] studied shock sensitivity of HMX pressed to 1.14 Mg/m^3 (60.0 %TMD) using a SSGT similar to the MRL SSGT. Coarse (176 μm) HMX had a higher shock threshold than fine (8.8 μm) HMX [20].

Although the trends observed here and in the earlier studies on RDX are similar, we did not observe the substantial decrease in shock sensitivity for our finest material that Scott [38] and Roth [39] detected. Densities in the three studies were similar. There are two possible explanations for these differences.

(i)   The Grade E and ball-milled RDX are clearly different in crystal form from the Grade A RDX sieve fractions (Figs 4 and 5). In particular the ball-milled RDX represents very rough, imperfect material in contrast to the smooth surfaced Grade A RDX. Such crystal imperfections have been shown to substantially increase the shock sensitivity of HMX [63]. Thus the result for the ball-milled RDX could be a compromise between a particle size effect leading to much reduced shock sensitivity while the "rough" crystal form with increased surface area enhances shock sensitivity.

(ii)   The SSGTs used by Scott [38] and Roth [39] used a confined RDX acceptor, in contrast to unconfined acceptor pellets used here. Ignition probability will not depend on confinement, but buildup will [64]. The confined tests are largely tests of ignition probability [64], whereas the unconfined MRL SSGT will introduce an additional factor from buildup due to greater energy losses from side rarefactions. Since buildup becomes increasingly more favourable at smaller particle size/higher surface area, this will compensate for decreased ignition probability at very small particle size, producing a curve like Fig. 6.

It is interesting to note that the trend observed here and by Scott [38] and Roth [39] is also followed for buildup time in shocked low density (0.4 Mg/m^3) RDX [65] and run distance in DDT of HMX [66-68]. These combined results qualitatively support the modelling predictions of Taylor [5,47].

The relationship between particle size and shock sensitivity determined by gap test has been extensively studied for TATB [32]. The clear trend is for decrease in shock sensitivity with decrease in particle size over the range 81-10 μm. The conclusion reached was that shock sensitivity to sustained shock correlated inversely with specific surface area (SSA) [32]. Dinegar [40] had earlier noted only a very small reduction in shock sensitivity for low density PETN as SSA was increased over a very substantial range. The contrast in the behaviour of TATB, a very unreactive energetic material, with very reactive RDX or PETN probably arises because of response to hot spot variation; whereas RDX or PETN will pick up from many hot spots produced by sustained shock, TATB has very specific requirements for hot spot size and intensity [32]. Thus any generalisation of behaviour to sustained shock must take into account the nature of the energetic material.

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9. CONCLUSION

Shock initiation of heterogeneous pressed explosives has been reviewed. Mechanisms for initiation are reasonably well understood but the relative importance of void collapse and frictional processes are not known with certainty. Study of the molecular processes associated with initiation has only just begun. Initiation is followed by buildup which occurs by grain burning. There is considerable disagreement as to whether the rate of grain burning or physical processes such as permeability, thermal conduction, convection etc are the key parameters controlling this process. It is frequently observed that the critical energy criterion P^2t = constant [21,22] is obeyed by many explosives for short pulse duration shocks but not sustained shock. This observation coupled with density effects has been interpreted to mean that buildup is the dominant process determining success/fail for short duration shocks while initiation is the key process under sustained shock. These conclusions are supported by modelling studies.

The effect of particle size on shock sensitivity is not straightforward. Although the statement "fine powders are often harder to ignite than coarse powders, but reactions in fine powders grow to detonation more rapidly once ignited" is largely correct, pressing density and shock duration strongly influence particle size effects. Heterogeneous explosives pressed to high relative density (> 98 %TMD) increase in shock sensitivity with decrease in particle size. At lower relative densities (≤ 95 %TMD) gap tests (sustained shock) often show lower reaction thresholds for larger particle materials but the reverse if $M_{50\%}$ values are compared. There is evidence that very small particle size materials show a reversal to decreased $M_{50\%}$ values. Smaller particle size materials invariably are more shock sensitive to short duration shock.

Five RDX powders of narrow particle size range, median 250, 138, 100.0, 21.5 and 3.9 μm, were studied both to further define particle size effects on shock sensitivity and also to make a preliminary assessment of the potential for using very fine particle size RDX in fuze trains. The 3.9 μm ball-milled RDX had F of I 85 and was thus comparable with tetryl. Shock sensitivity to sustained shock for the powders pressed to 90.0 %TMD increased with decreasing particle size 250 < 138 < 100.0 < 21.5 μm, while the 3.9 μm RDX showed a small decrease in sensitivity. These results together with those previously published [38,39] suggest that the prediction of Taylor [5,47] that shock sensitivity to sustained shock may decrease below some minimum particle size could be correct.

Clearly very fine RDX, ie < 3.9 μm , could be less impact sensitive than tetryl, thus meeting current fuze safety guidelines [53,54]. However shock sensitivity could be substantially reduced. We are in the process of extending these studies, both by examining behaviour under short duration shocks delivered by flyer plate, and also by studying very fine RDX.

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10. ACKNOWLEDGEMENTS

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TABLE 1

Particle Size Data and Impact Sensitiveness for
RDX Powders

RDX Sample	Particle Size Data (μm)		Impact Sensitiveness	
	Median	16%-84% Probability Range	Rotter F of I[a]	Evolved Gas (mL)
Grade A				
250-300 μm sieve cut	250	220 - 300	75	14.5
125-150 μm sieve cut	138	95 - 179		
75-106 μm sieve cut	100.0	73.3 - 138.0		
Grade E	21.5	10.8 - 36.0		
Ball-milled	3.9	approx. 1 - 9.4	85	17

[a]   Relative to RDX Grade G = 80.

TABLE 2

Shock Sensitivity (SSGT) of RDX Pellets Pressed
Nominally to 90 %TMD

RDX Sample	Pellet Densities Mg/m^3 (%TMD)		Shock Sensitivity (mm) [a]		
	Mean	Std. Dev.	$M_{50\%}$	$L_{95\%}$	Std. uev.
Grade A				·	
250-300 μm sieve cut	1.622 (90.00)	0.002 (0.11)	3.360	3.622-3.100	0.12
125-150 μm sieve cut	1.622 (90.00)	0.001 (0.06)	3.513	3.553-3.470	0.019
75-106 μm sieve cut	1.620 (89.89)	0.002 (0.11)	3.551	3.609-3.495	0.027
Grade E	1.623 (90.04)	0.014 (0.79)	3.785	4.115-3.455	0.15
Ball-milled	1.624 (90.13)	0.009 (0.51)	3.742	3.795-3.688	0.025

[a] All figures are in mm of brass shim.
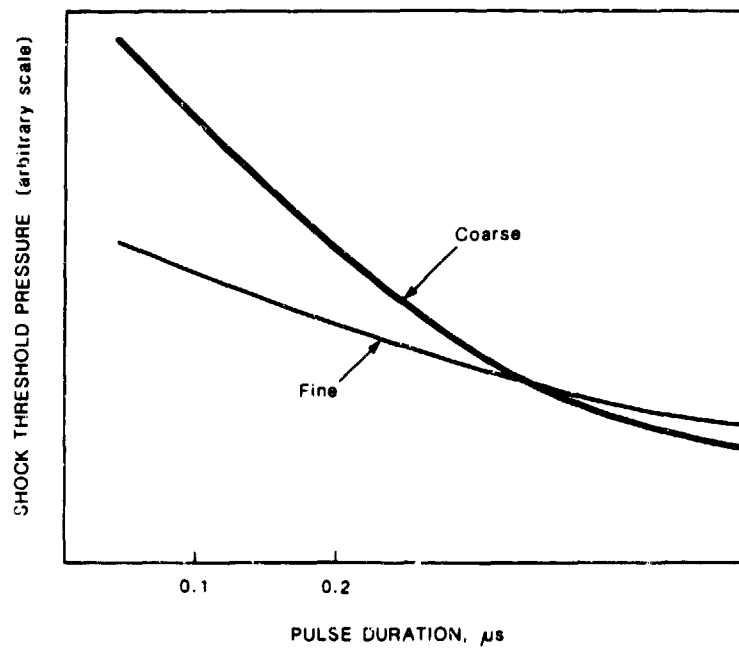
FIGURE 1   The relationship between threshold shock pressure and shock duration for coarse and fine particle size explosive pressed to the same density.
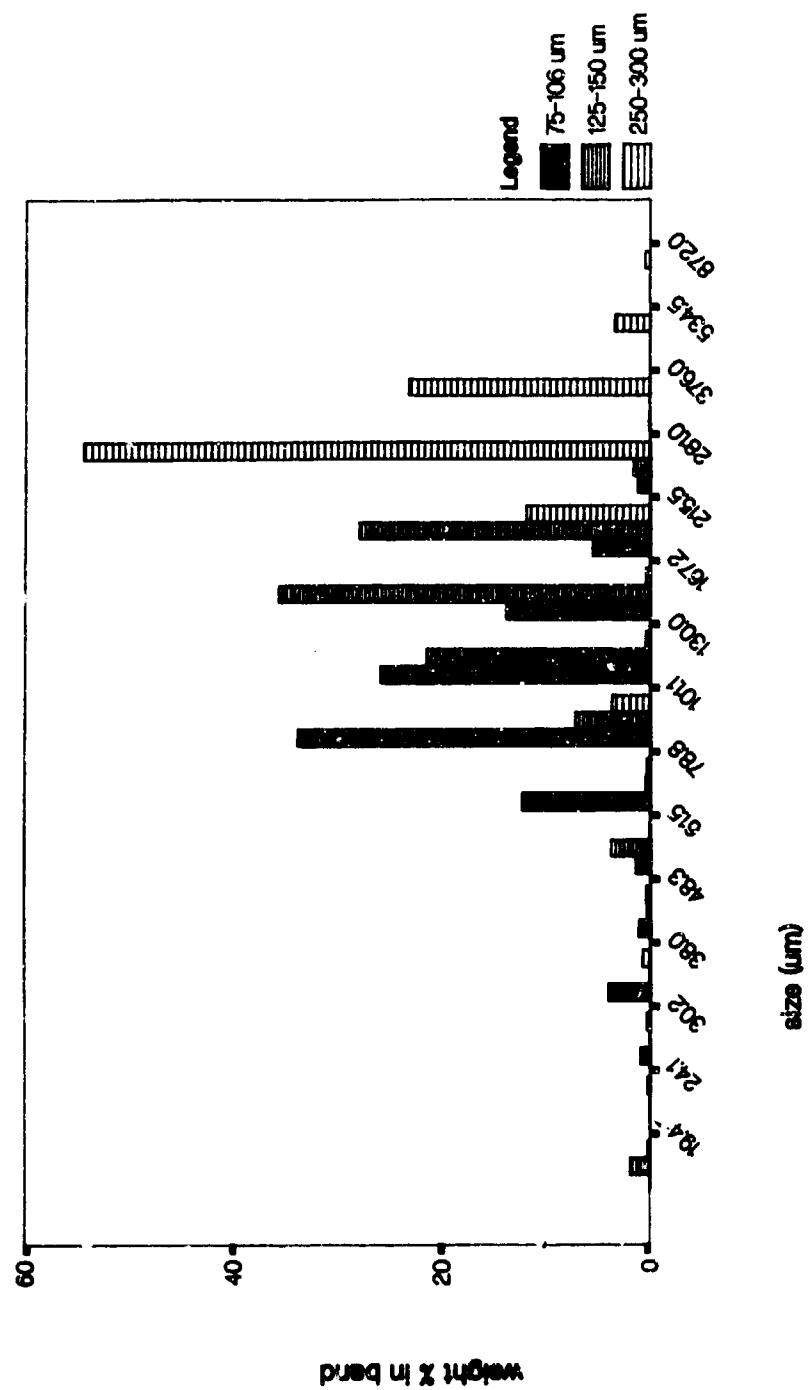
FIGURE 2   Bar chart showing particle size distribution of three sieve cuts of RDX Grade A over 16 sample bands.
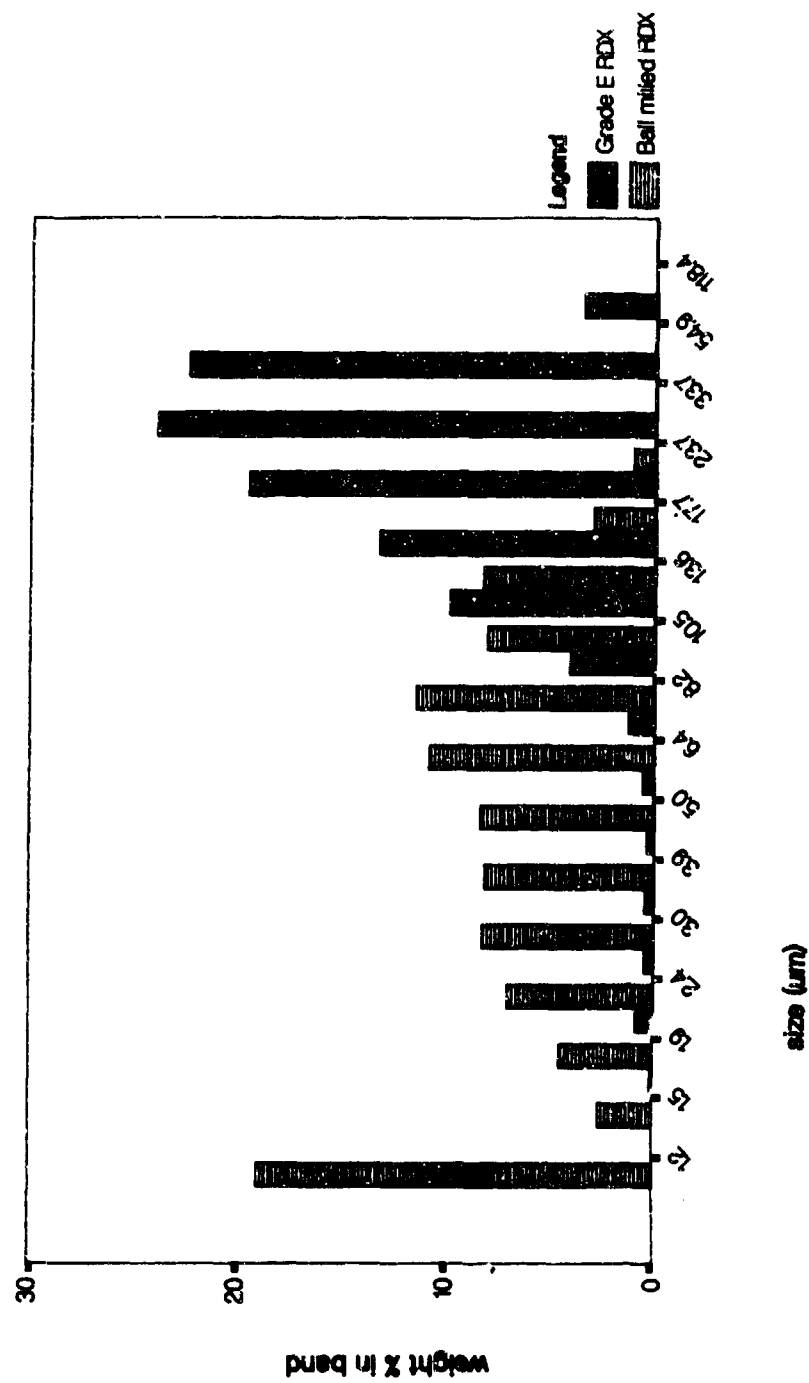
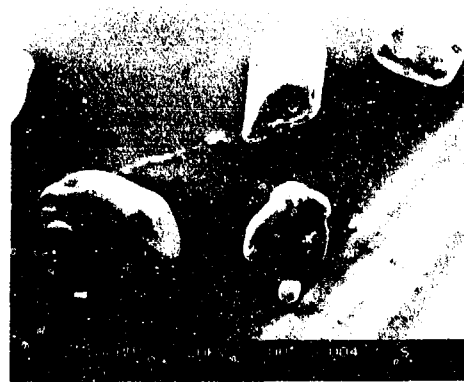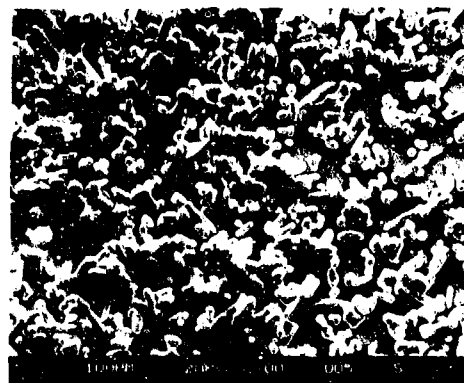FIGURE 3   Bar chart showing particle size distribution of two samples of RDX over 16 sample bands.
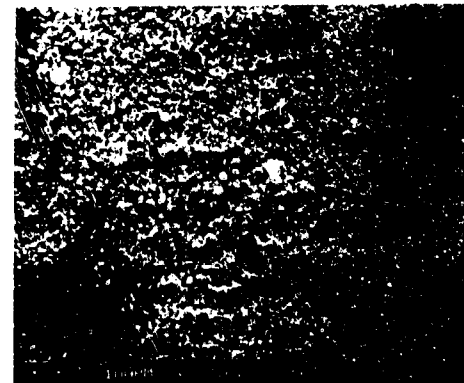
a   b
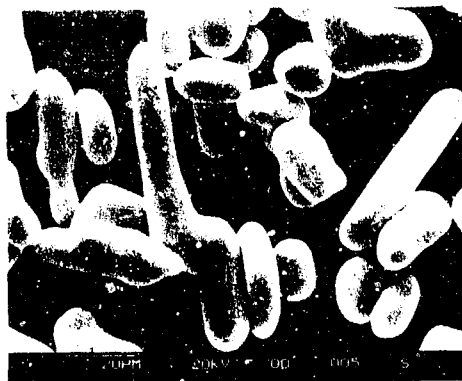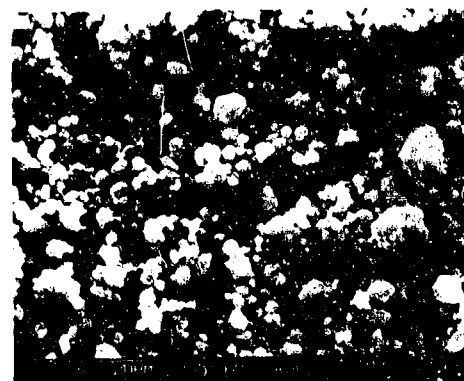
c   d

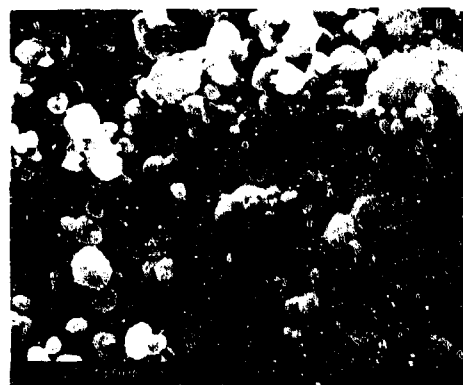FIGURE 4   Scanning electron micrographs of four of the RDX samples studied, magnification X200.

(a)   sieve cut nominally 125–150 μm, Grade A
(b)   sieve cut nominally 75–106 μm, Grade A
(c)   precipitated, Grade E
(d)   ball-milled.

a

b

c

FIGURE 5   Scanning electron micrographs of the two smaller particle size
RDX samples, depicted in the previous figure, but at higher
magnification.

      (a)   Grade E, magnification X1000
      (b)   Ball milled, magnification X1000
      (c)   Ball milled, magnification X2000.
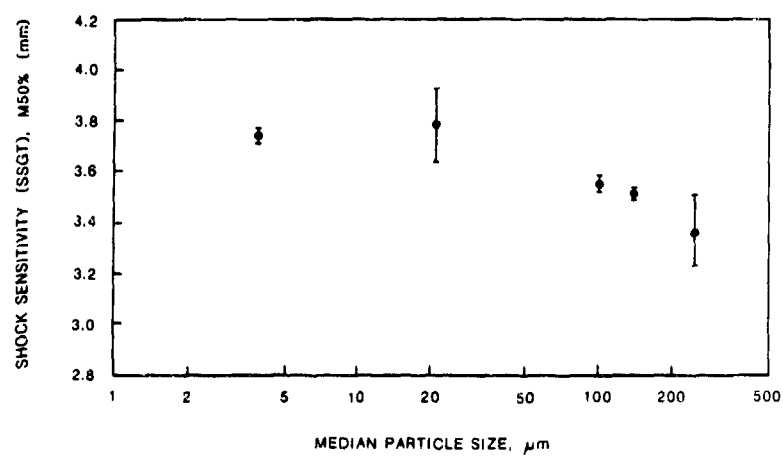
FIGURE 6   A plot of shock sensitivity (SSGT, $M_{50\%}$ in mm) against median
            particle size (logarithmic scale) for five RDX samples pressed
            nominally to 90.0 %TMD. Bars represent calculated standard
            deviations.

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Mechanism of and Particle Size Effects on Shock Sensitivity of
Heterogeneous Pressed Explosives: Preliminary Assessment
of Binderless RDX in Fuze Trains

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ABSTRACT

        Shock initiation of pressed heterogeneous explosives has been
reviewed. The key processes of ignition and buildup and their relative
importance under sustained and short duration shocks are described. Particle
size effects on shock sensitivity are shown to depend on density and shock
duration. $M_{50\%}$ values from gap tests often increase as particle size
decreases. Below a critical particle size $M_{50\%}$ then decreases, as predicted
from theoretical modelling. This trend was observed here for RDX. Assessment
of fine RDX for fuze trains indicated its potential suitability.