



Concerning the Shock Sensitivities of Certain Plastic Bonded Explosives Based on Attractive Cyclic Nitramines

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Abstract: Plastic bonded explosives (PBXs) based on ϵ -2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazaisowurtzitane (ϵ -HNIW), 1,3,5-trinitro-1,3,5-triazinane (RDX), β -1,3,5,7-tetranitro-1,3,5,7-tetrazocane (β -HMX) and *cis*-1,3,4,6-tetranitro-octahydroimidazo-[4,5-d]imidazole (BCHMX) were prepared using a hydroxyl-terminated polybutadiene as a binder (HTPB) and cured with hexamethylene di-isocyanate (HMDI). Recently published data for analogous PBXs with a polyisobutylene binder (*i.e.* a C4 matrix) are also included. All of these PBXs were tested using the Small Scale Water Gap Test according to STANAG 4490, the results of which are directly proportional to the impact and friction sensitivities of the PBXs studied. Reciprocal ratios between the shock sensitivity of these PBXs and their performance were found using the volume heat of explosion as a variable; a semi-logarithmic analogue, using a representative of the detonation pressure (product ρD^2) as a variable, has shown an opposite trend for three of these PBXs. A semi-logarithmic relationship between the shock sensitivity of the PBXs and the impact sensitivity of their pure nitramine fillers has confirmed the higher shock reactivity of the C4 PBXs filled with both technical ϵ -HNIW and RS- ϵ -HNIW, while for the HTPB PBXs filled with technical grade ϵ -HNIW, the shock sensitivity found was as expected. The shape and size of the particles should not be a reason for the increased resistance to shock of HMX-C4.

Keywords: nitramines, gap test, PBX, sensitivity, performance

1 Introduction

It is well-known that each energetic material (EM) has its own critical initiation pressure, P^* , and initiation density, ρ^* [1, 2]. When the initiation pressure is lower than P^* , detonation of the EM will start and develop with the commonly named “hot spots” mechanism (or it might be extinguished). On the other hand, at a greater pressure and density the detonation proceeds through a homogeneous mechanism which depends on the chemical composition of the given EM [1, 2]. These facts are some of the reasons for the results from shock sensitivity measurements being frequently non-reproducible [3]. For measuring the shock sensitivity of plastic bonded nitramine explosives (PBXs) with softened polyisobutylene as the binder (C4 matrix), a small water gap test [4], standardized by STANAG 4490 [5], was used, however we discovered that the arrangements for the test require a very small adjustment [4]. This adjustment might lead to dissimilar measurement results compared with the original arrangement. Such sensitivity to minor, insignificant changes in the arrangement, is another problem for the specification of shock sensitivity. Nevertheless, in the present paper the same approach was used to measure the sensitivity of nitramine based PBXs with hydroxyl-terminated polybutadiene (HTPB) as the binder. The results obtained, together with those of Ref. [4], are verified on the basis of their comparison with the performance of both of these groups of plastic explosives and with their impact sensitivities. The choice of the experimental samples of PBXs is the same as in Ref. [4], and aims mostly at an evaluation of the possible applications of a relatively new explosive, *cis*-1,3,4,6-tetranitro-octahydroimidazo-[4,5-d]imidazole (BCHMX), in comparison with other most attractive nitramines.

2 Experimental

2.1 Nitramines used

The following nitramines were used for preparing the PBXs: ϵ -2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazaisowurtzitane (ϵ -HNIW or ϵ -CL-20), of both technical (common) quality and reduced sensitivity (RS- ϵ -HNIW [6, 7]), as described in Ref. [4], *cis*-1,3,4,6-tetranitro-octahydroimidazo-[4,5-d]imidazole (BCHMX) prepared according to its patented process [8, 9], 1,3,5-trinitro-1,3,5-triazinane (RDX) and β -1,3,5,7-tetranitro-1,3,5,7-tetrazocane (β -HMX). The last two were obtained from Eurenco, Paris. Granulometric data for the nitramines used is presented in Table 1.

Table 1. Summary of the particle size distribution* of the nitramines used

Nitramine	for PBXs with the C4 and Formex binders [4]			for PBXs with the HTPB binder [10]		
	Median particle size [mm]	Diameter cumulative [mm]		Median particle size [mm]	Diameter cumulative [mm]	
		10%	90%		10%	90%
ϵ -CL20	13.2	7.3	22.1	24.1	11.6	49.8
RS-CL20	7.8	2.6	16.5	-	-	-
BCHMX	14.7	8.0	25.9	18.3	9.8	38.4
RDX (grade 2)	143.8	26.2	282.3	-	-	-
RDX (grade 5)	12.6	7.2	20.1	-	-	-
RDX (Eurengo)	-	-	-	64.2	17.6	126.7
β -HMX	144.3	67.6	426.3	41.9	15.4	88.5

Note: * data taken from Refs. [4, 10]

2.2 Preparation of the plastic bonded explosives (PBXs)

PBXs bonded with the C4 matrix [4]: the binder was prepared with 25 wt.% of polyisobutylene (PIB), 59 wt.% of dioctyl sebacate (DOS) and 16 wt.% of the oil HM46. The plastic explosives themselves were prepared [4] by mixing 91 wt.% of the particular nitramine with 9 wt.% of the binder in a computer controlled Brabender Plastograph at 70 °C under vacuum for 70 min. The samples of the final plastic explosives were then extruded using a 40 mm single screw extrusion machine to obtain long charges of the plastic explosives with a diameter of 21 mm. The prepared mixtures were designated as RDX-C4, HMX-C4, BCHMX-C4, techn- ϵ -HNIW-C4 and RS- ϵ -HNIW-C4.

PBXs bonded with HTPB were prepared in a 4 L vertical sigma mixer. All liquid ingredients, HTPB (R-45 from ARCO Co.) as a prepolymer with an hydroxyl content of 0.84 meq·g⁻¹, tris[1-(2-methylaziridiny)]phosphine oxide (MAPO) and dioctyl adipate (DOA), except for the curing agent, were added and mixed for 20 min at 40 °C followed by vacuum mixing for a further 20 min to expel any entrapped air. Then, the nitramine was added in three portions during 30 min. Finally, the curing agent, hexamethylene di-isocyanate (HMDI), was added at 55 °C and the ingredients were mixed for a further 30 min. The mixture was then cast and cured at 60 ± 2 °C for seven days under vacuum. The cured HTPB binder system was prepared with an NCO/OH ratio of 1.3. MAPO was 0.5 wt.%, while the DOA was 20 wt.% of the total binder system. The PBXs thus prepared contained 82 wt.% explosive and 18 wt.% binder system and were designated as RDX-HTPB, HMX-HTPB, BCHMX-HTPB and techn- ϵ -HNIW-HTPB.

2.3 Detonation velocity measurements

The detonation velocities were measured using an “Explomet-Fo-2000 Multichannel” instrument (Continitro AG, Geneva, Switzerland). The samples for testing were prepared in the form of a sheet, 20 mm thick, 40 mm wide and 300 mm long. Three optical sensors (probes) were placed in each charge, with the first sensor being placed at a distance of 100 mm from the surface containing the detonator. Each of the other two probes was placed at a distance of 80 mm from the previous one. Charges were set off using a No. 8 electrical detonator. The mean values of three measurements for each sample (max. $\pm 114 \text{ m}\cdot\text{s}^{-1}$) are reported in Table 2.

2.4 Calculation of the detonation parameters

The theoretical heat of detonation, Q_{det} , detonation velocity, D , and detonation pressure, P , for all of the PBXs prepared were calculated by means of the EXPLO5 code [11, 12] in which the BKWN set of parameters was applied as follows: $\alpha=0.5$, $\beta=0.176$, $\kappa=14.71$, $\Theta=6620$ [13]. Thus, the Q_{det} values obtained (see Table 2) represent the heat released in a constant volume explosion for the compositions of the products, taken from the Chapman-Jouguet (CJ) state.

Table 2. Detonation parameters of the nitramine explosives studied

Nitramine explosive tested	Loading density [g·cm ⁻³]	Detonation velocity D [m·s ⁻¹]		Detonation pressure P [GPa]	Heat of detonation Q_{det} [kJ·kg ⁻¹]
		experimental	calculated		
techn- ϵ -HNIW	1.98 ^a	9473 ^a	9410 ^a	41.7 ^a	6465 ^a
BCHMX	1.79 ^a	8700 ^a	8840 ^a	33.9 ^a	6447 ^a
RDX	1.76 ^a	8750 ^a	8718 ^a	32.1 ^a	6085 ^a
β -HMX	1.90 ^a	9100 ^a	9225 ^a	38.0 ^a	6075 ^a
techn- ϵ -HNIW-C4	1.77 ^a	8594 ^a	8425 ^a	29.64 ^a	5744 ^a
RS- ϵ -HNIW-C4	1.77	8594	8425	29.64	5744
BCHMX-C4	1.66 ^a	8266 ^a	8056 ^a	25.49 ^a	5602 ^a
RDX-C4	1.64 ^a	8055 ^a	7856 ^a	23.36 ^a	5512 ^a
β -HMX-C4	1.67 ^a	8318 ^a	8130 ^a	25.86 ^a	5564 ^a
techn- ϵ -HNIW-HTPB	1.63 ^b	8167 ^b	7919 ^b	23.73 ^b	5786 ^b
BCHMX-HTPB	1.56 ^b	7746 ^b	7593 ^b	21.23 ^b	5744 ^b
RDX-HTPB	1.52 ^b	7526 ^b	7449 ^b	20.06 ^b	5487 ^b
β -HMX-HTPB	1.57 ^b	7812 ^b	7627 ^b	21.34 ^b	5453 ^b

Note: ^a taken from Ref. [14]; ^b result from this study

2.5 Impact Sensitivity Measurements

A standard impact tester (Julius Peters [15]) with exchangeable anvil was used, the amount of tested substance being 50 mm³. The drop hammers used weighed 2 kg and 5 kg. A probit analysis [16] was performed to determine the probability levels of initiation. The sensitivity obtained was expressed as the drop energy, E_{dr} , versus the initiation percentage. Only the results for a 50% level probability of initiation are used in this article and these are shown in Table 3.

2.6 Friction sensitivity

Friction sensitivities for all of the nitramine explosives studied were determined using a BAM friction test apparatus using the standard test conditions [15]. The sensitivity to friction was determined by spreading a thin layer of about 0.01 g of the dry explosive on the surface of the porcelain plate. Different loads were used to change the normal force between the porcelain piston and the plate. Initiation was observed through sound, smoke appearance, or by the characteristic smell of the decomposition products. Using the probit analysis [16] (35 measurements per sample), only the normal force at which a 50% probability of initiation occurred is reported as the friction sensitivity in Table 3.

2.7 Gap test

The Small Scale Water Gap Test (SCWT) [5, 17] was used to determine the shock sensitivity of the PBX samples prepared with a C4 matrix, as described in Ref. [4]. This method is suitable for materials having a critical diameter of less than 20 mm. A slight change in the SCWT consisted in eliminating the air bubble formed in the water gap during assembly [4]. The 50% point, in mm of water column, and the standard deviation for test sample initiation were calculated using a Bruceton statistical analysis of the Up and Down test sequence, performed with 25 consecutive trials. The results of the SCWT were then presented as the water column height and corresponding pressure for 50% probability of sample activation. The data obtained for the versions of the PBXs studied are presented in Table 3.

3 Results and Discussion

Licht has shown [19] that a high level of performance of EMs is usually accompanied by an increase in their sensitivity and that an insensitive explosive will not exhibit top performance. It has subsequently been shown that this can be considered as a general rule (even if there are some exceptions [3]), but the author has stated that this result has not been proved by theory [3, 19]. Studies

in this area have been centered mainly on impact sensitivity [3, 19-21], and less commonly on friction sensitivity [3, 20], while similar articles on shock sensitivity seem to be almost absent from the literature for the present.

Table 3. Survey of data obtained for shock sensitivity in this paper, and for impact and friction sensitivities from the literature [4, 18, 20]

Nitramine explosive tested	Sensitivity						
	Impact		Friction		Shock		
	[J]	Ref.	[N]	Ref.	water gap [mm]	pressure [GPa]	Ref.
techn- ϵ HNIW	4.1	[18]	69.0	[18]	-	-	-
RS- ϵ -HNIW	10.8	[4]	84.4	a	-	-	-
BCHMX	3.2	[18]	66.1	[18]	-	-	-
RDX	5.6	[18]	148.5	[18]	-	-	-
β -HMX	6.4	[18]	154.4	[18]	-	-	-
techn- ϵ -HNIW-C4	14.2	[20]	148	[20]	19.7 \pm 0.7	1.87	[4]
RS- ϵ -HNIW-C4	17.0	[4]	-	-	16.3 \pm 0.8	2.38	[4]
BCHMX-C4	11.6	[20]	181	[20]	16.6 \pm 1.1	2.33	[4]
RDX-C4	21.1	[20]	214	[20]	14.9 \pm 0.4	2.64	[4]
β -HMX-C4	20.2	[20]	193	[20]	14.1 \pm 1.1	2.81	[4]
techn- ϵ -HNIW-HTPB	10.8	a	214	a	15.1 \pm 0.9	2.61	a
BCHMX-HTPB	9.6	a	322	a	15.3 \pm 0.5	2.56	a
RDX-HTPB	14.6	a	>360	a	13.6 \pm 0.5	2.91	a
β -HMX-HTPB	15.2	a	>360	a	13.6 \pm 0.7	2.92	a

Note: ^a result from this study

Figure 1 presents a semi-logarithmic relationship between the shock sensitivity and performance, expressed as the product of the experimentally determined loading density, ρ , and the square of detonation velocity, D^2 (*i.e.* a representation of detonation pressure) – we are here talking about relationships between experimental data. This divides the group of PBXs into three sub-groups for which the RDX-C4 data represent an intersection point of the corresponding partial relationships; the straight line for BCHMX-HTPB, RDX-C4 and HMC-C4 could be construed as an exception from the general trend of the sensitivity-performance relationship (for a discussion about this relationship see Refs. [3, 19]). On the other hand, performance based on thermochemical calculations (*i.e.* volume heat of explosion) correlates in all of the cases here with shock sensitivity, with the RDX-HTPB data as an intersection point of the corresponding partial relationships (see Figure 2).

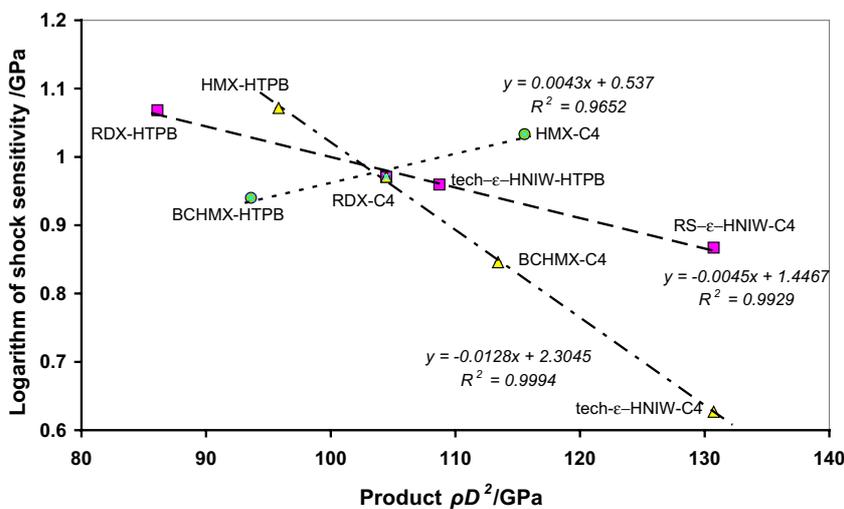


Figure 1. Semi-logarithmic relationship between shock sensitivity and a representation of detonation pressure (*i.e.* product of loading density and the square of experimental detonation velocity)

In Figures 1 and 2, it is interesting that both of the C4 PBXs, derived from the same quality HNIW (*i.e.* techn- ϵ -HNIW) and its RS-analogue (reduced sensitivity-analogue, *i.e.* RS- ϵ -HNIW), correlate well with these relationships, the first one according to Figure 2 with, logically, relationships for the more reactive BCHMX mixtures. It is also evident in Figures 1 and 2, that HTPB, in the quantities used for the preparation of the corresponding PBXs, seems to suppress the “higher sensitivity” of techn- ϵ -HNIW, in comparison with the situation when this quality of HNIW is incorporated in the C4 binder. It should be mentioned that not only are nitramine fillers related in light of their molecular structure, but also both binders have some macromolecular-structural likenesses (even though their rheological properties can be different); these similarities should be one of the reasons for the PBX groupings in the sense of the partial relationships in Figures 1 and 2.

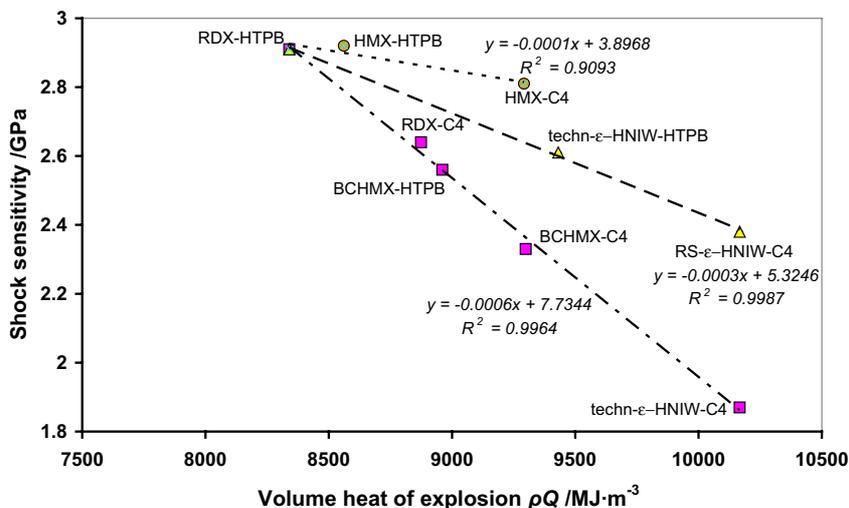


Figure 2. Relationship between shock sensitivity and volume detonation heat (*i.e.* product of loading density and the calculated heat of detonation in $\text{kJ}\cdot\text{kg}^{-1}$)

The relationships in Figure 2 were mainly expected and are in concordance with Licht's conclusions [3, 19]. Moreover, these results confirm the physical meaning and reproducibility of the results from shock sensitivity measurements using the Small Scale Water Gap Test (SCWT) [5, 17] modified according to Ref. [4]. The above-mentioned exception in Figure 1 needs, further, more detailed study, but it is clear that the volume heat of explosion is more representative of the chemistry of detonation than is the detonation pressure.

Figure 3 shows the relationship between the shock and impact sensitivities of the PBXs studied, as described in Ref. [4]. Here, data for the HMX-C4 explosive, together with those of the techn- ϵ -HNIW-C4 explosive, are outside the data groups in this figure. Furthermore, the sensitivities for the RS- ϵ -HNIW-C4 PBX do not have the most convincing correlation here; it was reported in Ref. [4] that, during the blending of RS- ϵ -HNIW into the C4 matrix, the ϵ -polymorph was irreversibly changed into the γ -analogue, which means that the resulting PBX should be marked as γ -HNIW-C4. Theoretically, if the polymorph change had not taken place, data for the HMX-C4, RS- ϵ -HNIW-C4 and techn- ϵ -HNIW-C4 explosives should lie on the same straight line.

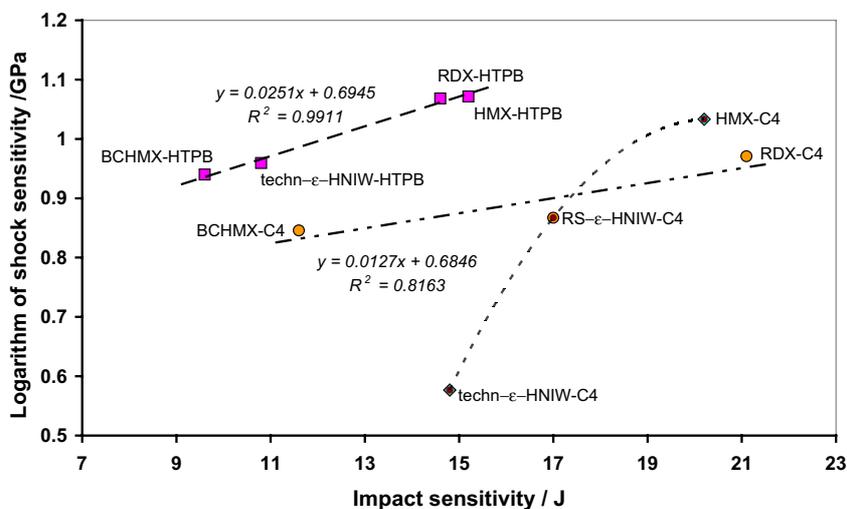


Figure 3. Semi-logarithmic relationships between shock and impact sensitivities of the PBXs studied

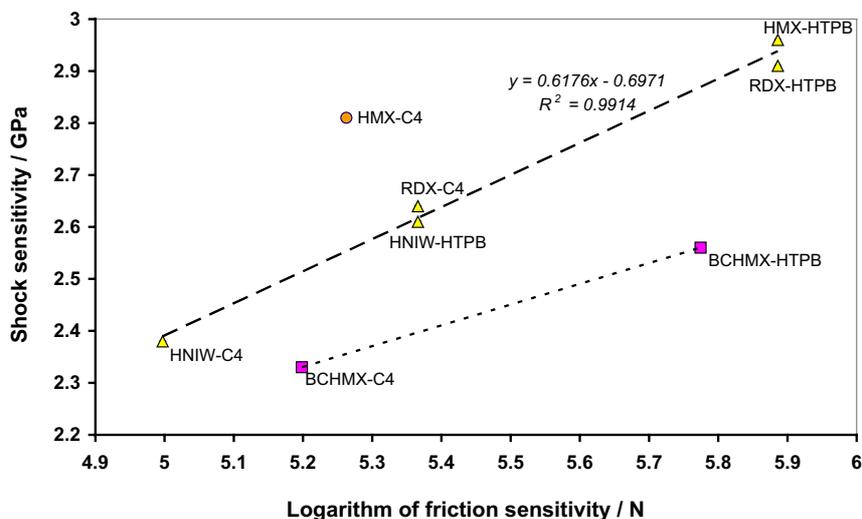


Figure 4. Approximate relationship between shock and friction sensitivities of the PBXs studied

An approximate comparison between the shock and friction sensitivities of the PBXs studied is represented by the semi-logarithmic relationship in Figure 4. Data for the PBXs based on BCHMX are outside the relationship of the other

PBXs. This might be due to the molecular structure of BCHMX (a crowded molecule and therefore with increased initiation reactivity [22]).

In Ref. [4] there is a comparison of the shock sensitivity of the C4 PBXs with the impact sensitivity of their pure nitramine fillers. Extending this comparison with data from the HTPB PBXs from the current paper gives a semi-logarithmic relationship, as shown in Figure 5. In contrast to Figures 3 and 4, data for HMX-C4 correlates in Figure 5 as expected. This fact leads to the presumption that the size and shape of the β -HMX crystals used are not necessarily a reason for the HMX-C4 data positions on the charts in Figures 1-4. The size and shape of the nitramine crystals (for their SEM pictures see Ref. [4]), which were used in Ref. [4] and are used in this study for the PBX preparations, are mutually quite different, but on the logical relationships derived in this study, this fact does not have any influence.

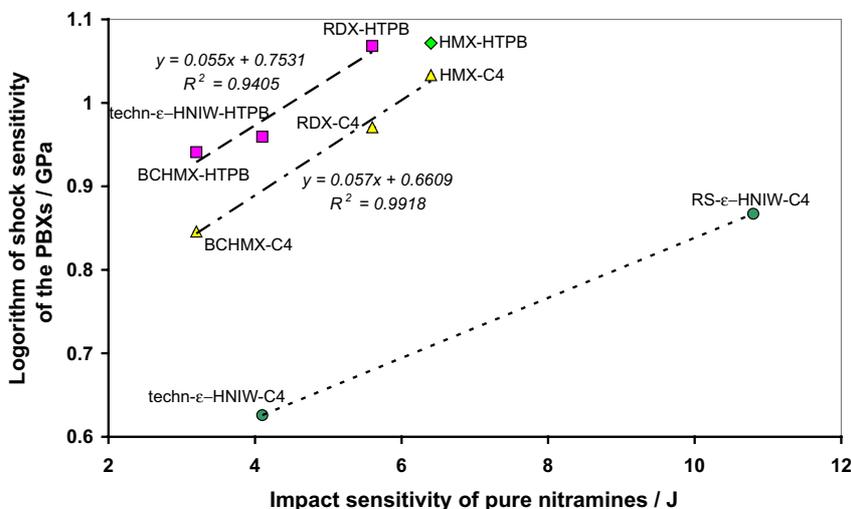


Figure 5. Relationship between shock sensitivity of the PBXs studied and the impact sensitivity of their pure nitramine fillers

The positions of the HNIW PBXs in Figure 5 are interesting. The data for techn- ϵ -HNIW-HTPB correlate as expected (as is the case for their molecular-structural similarity), and HNIW with a C4 matrix is relatively very sensitive to shock. Apart from the lower binder content (in comparison with HTPB PBX), here there might be a significant influence from the micro-cracks in the HNIW crystals in the case of RS- ϵ -HNIW; as has already been mentioned, RS- ϵ -HNIW becomes completely changed into γ -HNIW during kneading with the C4 matrix,

whereas technical ε -HNIW is unchanged in this process [4]. During this polymorphic transition, micro-regions with gradients of mechanical stress are formed because of the dislocation effect; the subsequent process is then controlled by micro-stresses that arise at the front of this $\varepsilon \rightarrow \gamma$ transition [23] and leads to micro-cracks in the HNIW crystals [4]. Traditional problems with the “high” mechanical sensitivity of the ordinary (*technical*) quality of ε -HNIW (*i.e.* impact sensitivity of 2-4.5 J) [4] should reflect the disturbance in the optimal placing of molecules in the crystal lattice [3, 4]. However, the HTPB binder behaves as if it suppresses this deficiency found in technical ε -HNIW, as is seen mainly in Figure 5.

4 Conclusions

The plastic bonded explosives (PBXs) of nitramines with polyisobutylene (C4 matrix) and hydroxyl-terminated polybutadiene (HTPB) binders have provided results from the small scale gap test according to STANAG 4490 (in the sense of Ref. [4]). These results are in a semi-logarithmic relationship with a representative of detonation pressure (*i.e.* the product ρD^2), and are in linear relationships with performance derived from the thermochemistry of detonation (*i.e.* the volume heat of detonation). Both these kinds of relationships are reciprocal to shock sensitivity, with a small exception being observed in the case of the ρD^2 ordinate. From the proportional semi-logarithmic relationship between the shock and impact sensitivities, the C4 PBXs filled with ε -HNIW-C4 and HMX surpass HNIW-C4 due to the higher initiation reactivity. Comparison of the shock sensitivity of these PBXs with the logarithm of their friction sensitivity confirms a greater initiation reactivity for the PBXs filled with BCHMX, while the C4 PBX with HMX has shown the lowest sensitivity. A semi-logarithmic relationship between the shock sensitivity of the PBXs studied and the impact sensitivity of their pure nitramine fillers (*i.e.* RDX, β -HMX, BCHMC, technical ε -HNIW and RS- ε -HNIW) has shown higher shock reactivity for both kinds of ε -HNIW with the C4 binder; in the case of HMX-C4 this relationship suggests that its higher resistance to shock is not related to the size and shape of the β -HMX grains used. This factor did not have an influence even with other nitramines used, even though their granulometric composition was actually quite different. In the case of sensitive technical ε -HNIW, the HTPB matrix seems to act a suppressor of such sensitivity (in comparison with the influence here of the C4 matrix). The higher shock reactivity of RS- ε -HNIW included in the C4 matrix is due to micro-cracks in its crystals (see Ref. [4]), when the

epsilon-modification changes to the gamma-analogue during incorporation of this particular nitramine into the C4 matrix [4]. All of the relationships found during this study confirm the reproducibility and the physical implications of the gap test used, with slight modification, as discussed in Ref. [4].

Acknowledgements

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