

CALORIMETRY STUDIES OF EXPLOSION HEAT OF NON-IDEAL EXPLOSIVES

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Heats of explosion of non-ideal RDX-based compositions in four various atmospheres (argon, nitrogen, air and argon/oxygen mixture) were measured. Charges of phlegmatized RDX containing 30% of two types of aluminium powders, coarse aluminium oxide, or fine lithium fluoride particles were fired in a calorimetric bomb of 5.6 dm³ in volume. The influence of inert and reactive additives and the atmosphere filling the bomb on the heat outcome was examined. To estimate the degree of afterburning of the detonation products and reactive particles, thermochemical calculations were also performed for the tested explosive compositions.

Keywords: calorimetry, explosion heat, non-ideal explosives

Introduction

To measure heat of detonation of explosives, certain requirements must be fulfilled. A precise description of a calorimetric method for determining of the detonation heat was given by Ornellas [1–5]. He thoroughly investigated the influence of detonation conditions on the heat released from many explosives by using a calorimetric bomb of 5.28 dm³ in volume [1]. His studies clearly indicate that the heat released from cylindrical heavily confined charges (in gold or ceramic envelopes) fired under vacuum conditions correspond with the detonation heats of explosives. The detonation products from confined charges are those on the Chapman–Jouguet (CJ) isentrope in the freeze out range of 1500–1800 K [1, 4]. The results obtained for unconfined charges correspond with the heats of reactions proceeding under conditions of high temperature and low pressure. Such conditions lead to a decrease in the amount of CO₂ and H₂O, and an increase in the amount of CO and H₂ in detonation products. Finally, the measured heat can be significantly lower than that obtained from cylindrical heavily confined charges. During the first stage of expansion of the detonation products, the gold or ceramic envelope absorbs a significant part of the released energy. Consequently, shock waves reflected at the surface of bomb are weaker and not able to heat detonation products, so that secondary reactions are stopped. Among others, Ornellas measured the detonation heat of explosives with different oxygen balance. Tested TNT and BTF (benzotrifuroxan) are examples of the lowest

oxygen-balanced explosives tested. The oxygen balance to H₂O–CO₂ level is –73.9 and –82.7% for TNT and BTF, respectively. On the other hand, Ornellas measured the detonation heat of EAR (ethylene diamine dinitrate/ammonium nitrite/RDX; oxygen balance –6.2%), PETN (–10.1%) and HMX (–21.6%). The values of 1411 and 1093 cal g^{–1} were obtained for heavily confined BTF and TNT charges, respectively, and 1166, 632 cal g^{–1} for unconfined ones. The heat effects of 1479 and 1334 cal g^{–1} were measured for HMX charges under this same conditions. The difference in heat between confined and unconfined charges of EAR and PETN are similar and essentially within experimental error.

The influence of a metal casing of the explosive charge on the detonation heat measured in a calorimetric bomb was confirmed by Tongchang *et al.* [6] and Makhov [7]. The experiments performed by Tongchang *et al.* showed that the gold and brass shell of the cylindrical charge could be substituted by a thick-walled porcelain shell.

Ornellas also presented the results of detonating PETN and TNT charges in various atmospheres [4]. These results show practically complete reaction of confined charges fired in oxygen atmosphere. But TNT charges detonated in vacuum and in carbon dioxide atmosphere give similar heats released.

The influence of gaseous filling of a chamber on the composition of detonation products was deeply researched by Volk [8–12]. Unconfined charges of explosives were fired in a chamber of 1.5 m³ in volume in vacuum-like conditions and also in argon under pressure in the range from 0.05 to 0.3 MPa. It was concluded,

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that the biggest changes in detonation products composition occurred when the pressure in the chamber was changed from 0 (vacuum) to 0.1 MPa.

Cudzilo *et al.* [13] measured the heat of explosion of TNT, RDX and HMX charges in a bomb of 3.6 dm³ in volume. Charges of explosives without an envelope were fired in argon or air atmosphere under pressure of 1.0 MPa. Inert gas filling the calorimetric bomb was applied to limit the effect of secondary reactions in the detonation products. It was concluded that the after-burning of detonation products in air released additionally 275, 118 and 99% of heat energy (in comparison to results obtained in argon) for TNT, RDX and HMX, respectively. The effect of filling of the calorimetric-bomb cavity with an inert gas on the detonation heat was also reported by Makhov [7].

The heat of detonation of TNT, RDX and HMX was also examined by Galezowski *et al.* [14] by using a 5.6 dm³ calorimetric bomb filled with nitrogen under pressure from 0.1 to 2 MPa. It was confirmed that nitrogen under high pressure reduces the secondary reactions in detonation products and makes it possible to measure the detonation heat of explosives.

Trzcinski and Paszula [15] investigated the after-burning process of detonation products of non-ideal explosives with high deficit of oxygen in confined conditions. Among others, the heat of explosion was measured in compressed argon, nitrogen and air. There was no meaningful difference between the heat effects measured in argon and nitrogen. After-burning process in air atmosphere increases total heat effect in comparison to results obtained for argon atmosphere of about 270, 240, 180 and 150% for TNT, TNT/Al (85/15), TNT/RDX (50/50) and TNT/RDX/Al (42.5/42.5/15) mixtures.

A lot of work has been accomplished recently with aluminium particle in energetic materials to research the influence of Al particles on the detonation and blast performances. However, there are only a few works devoted to the calorimetry study of high explosives with aluminium. The effect of 15% addition of aluminium powder on the detonation heat of TNT and TNT/RDX mixture was examined by Trzcinski and Paszula [15]. The aluminized explosives produced more energy than the neat explosives detonated in the same gaseous filler. Quite good agreement between the heats of detonation measured in argon with theoretical ones was obtained when 20-30% of aluminium was assumed to be inert in thermochemical calculations.

Makhov [16, 17] studied the calorimetric heats of decomposition of formulations containing C-H-N-O high explosives (TNT, RDX, PETN, BTNENA) and powder aluminium. The effect on the explosion heat of the oxygen balance, aluminium

concentration, high explosive and Al particle size, and charge diameter and density was investigated. Mean particle sizes of Al powders used in these investigations were 0.1, 20, 50 and 200 µm. The explosive charges were confined in a 5-mm thickness solid casing. Prior to the test the bomb was blown out with argon and pumped out. The heat of detonation of the aluminized explosive tested depended unambiguously on their oxygen balance. The heats of explosive decomposition of aluminized HMX, nitroguanidine, and bis(2,2,2-trinitroethyl) nitramine were also measured by Makhov *et al.* [18].

The heat of detonation of some aluminized explosives (PBXs, TNT and Composition B) was evaluated by Brousseau and Anderson [19]. The explosives were confined in copper tubes and fired under vacuum conditions in a calorimetric bomb. Nanometric aluminium (100–200 nm) and three types of aluminium powder with average particle sizes of 2, 12 and 21 µm were used in the study. Addition of aluminium powder significantly increased the heat of detonation of the explosive tested.

Research on the effect of aluminium contents on the calorimetric heat of detonation of RDX-based compositions was carried out by Trzcinski *et al.* [20]. Mixtures containing 15, 30 45 and 60% of two types of aluminium (a mean size 50 mm powder and flaked Al) were tested. Mixture of phlegmatized RDX with aluminium produced more heat of detonation in the calorimetric bomb than the explosive itself. The maximum heat corresponded to an aluminium content of 30%.

The aim of this research was to measure the heat released during explosion of RDX-based compositions containing 30% of a non-explosive component – inert or active – in a calorimetric bomb, under high pressure of four atmospheres: nitrogen, argon, air, and an argon/oxygen mixture. After burning of Al particles and the postdetonation gases were investigated in air and O₂/Ar (20/80) atmospheres. Two types of aluminium powders with different particle sizes (5 and 90 µm) were applied. As commonly known, active components, especially aluminium powders are widely used in explosives to enhance their blast effect and energetic characteristics. For comparison, Al₂O₃ and LiF were mixed with phlegmatized RDX and the heat of explosion of such compositions was investigated.

Experimental

Components characterization and explosive charges preparation

To prepare the tested explosive samples, commercial grade phlegmatized RDX (RDX_{ph}) was used. This mate-

rial contains ca. 94% of pure RDX and 6% of wax (CH_2)_n. Two types of aluminium powder produced by ALPOCO M2 plant, UK, were used in the mixtures with RDX_{ph} . The first one, marked here as Al5, is a fine particle sized aluminium powder with an average particle size of 5 μm . The second one, marked here as Al90, is aluminum powder of particle size in a range from 75 to 90 μm . Both powders were analysed to evaluate the content of metallic aluminium. The analysis consisted of measurement of the volume of hydrogen released as a result of reaction of the samples tested with 40% aqueous solution of sodium hydroxide [21]. The accuracy of the analysis was below 1% of the initial mass of a sample. As may be predicted, the total content of metallic aluminium in the coarse powder (Al90) is high and exceeds 99%. The fine Al powder (Al5) contains ca. 96.5% of active metal, which is natural, since the oxidation of Al is the higher the smaller Al particles are and the higher specific surface of the sample is.

To perform a deeper investigation on behavior of the Al powders after detonation, the aluminium powders were replaced with inert components which have grain size close to applied Al powders. As these components, fine grained lithium fluoride with particle size of ca. 5 μm (LiF No. 01140 Sigma-Aldrich) and aluminium oxide with particle size ca. 75 μm (Merck KGaA, No. 101097) were used. LiF and Al_2O_3 had been chosen not only because they are chemically inert but also they have similar thermochemical properties to aluminium powders. All tested RDX-based compositions contained 70% of RDX_{ph} and 30% of the chosen active (Al5, Al90) or inert (LiF, Al_2O_3) additives.

Some microscopy analyses (SEM and optical microscopy) were made to characterize the components of RDX-based formulations. The shape and size of powders particles were observed at the scanning microscope JEOL 5400 with magnification from 100 to 2000 times. SEM images of used additive particles and phlegmatized RDX are shown in Figs 1–5.

SEM images (Figs 1 and 2) revealed that the Al powders chosen for studying detonation performance of RDX_{ph} /Al mixtures differ significantly from each other in particle size and shape. Particles of Al5 are sphere shaped, whereas particles of Al90 are much less spherical. LiF particles (Fig. 3) are cubic shaped with rounded wedges and they well correspond with Al5 morphology, so that LiF was a good choice to replace Al5 in the mixtures with RDX_{ph} . Sphere-shaped alumina particles (Al_2O_3 , Fig. 4) are built from sintered crystals. The porosity of these particles is high and can influence the density of RDX_{ph} /alumina mixtures. The stability of Al_2O_3 particles during explosion was verified by SEM observation of

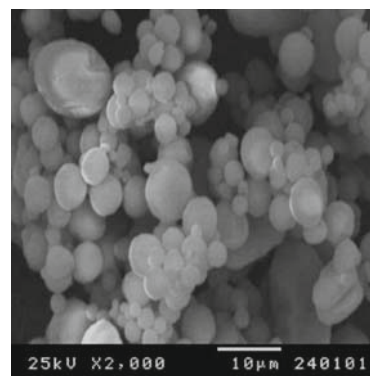


Fig. 1 SEM images of Al5 particles

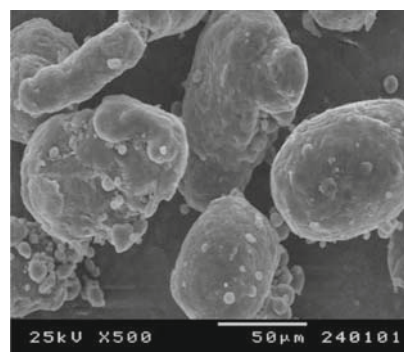


Fig. 2 SEM images of Al90 particles

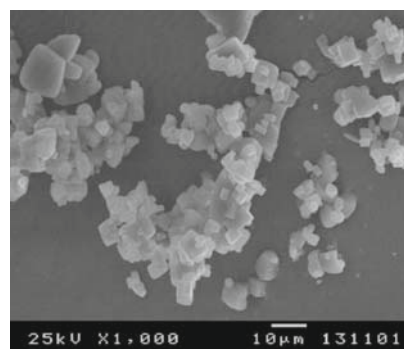


Fig. 3 SEM images of LiF particles

explosion residues. Post-explosion alumina particles had much smaller size than that before explosion. This means that Al_2O_3 particles disintegrated during the explosion process. For the manufacturing of pressed explosive charges, a molding powder was prepared by granulation of RDX_{ph} /additive mixture with chloroform. Firstly, weighed quantity of RDX and chosen powder (LiF, Al_2O_3 , Al5, Al90) were mixed mechanically and after obtaining homogenous composition chloroform was added to wet the mixture. The slurry-like mixture was mixed once again. Then the chloroform was completely evaporated. That procedure leads to a homogenous



Fig. 4 SEM images of Al_2O_3 particles



Fig. 5 SEM images of RDX_{ph} particles

mixture in which additive particles are bound to RDX particles.

Pressed charges containing Al additives were investigated using SEM and optical microscopy. It was determined that Al particles are much larger before pressing than after this process. Al particles can be partially crushed during charge preparation, yet their observable size was probably lower than the real size because of their merging in the RDX matrix.

The porosity of pressed charges, α , was also calculated. It was defined as:

$$\alpha = \rho_s / \rho_c - 1$$

where s refers to the solid or matrix material, and c to the porous conditions. The densities of solids [kg m^{-3}] are taken: $\rho_{\text{Al}}=2710$, $\rho_{\text{LiF}}=2640$, $\rho_{\text{Al}_2\text{O}_3}=4000$, $\rho_{\text{RDX}}=1800$, $\rho_{\text{wax}}=890$. The porosity of pressed mixtures is shown in Table 1.

The porosity of $\text{RDX}_{\text{ph}}/\text{Al}_2\text{O}_3$ is much higher in comparison to the rest of the mixtures. This feature can influence the detonation characteristics of that composition.

Calorimetric measurements of the heat of explosion

Figure 6 shows a scheme of the calorimetric system used for measurements of the heat of explosion. The spherical bomb, made of stainless steel, has a volume of 5.6 dm^3 . The bomb is placed in a polished steel calorimeter vessel containing distilled water.

Table 1 Density and porosity of tested mixtures

Explosive mixture	Density/ kg m^{-3}	Porosity/ %
$\text{RDX}_{\text{ph}}/\text{LiF}$	1750	8.6
$\text{RDX}_{\text{ph}}/\text{Al}_2\text{O}_3$	1640	25.0
$\text{RDX}_{\text{ph}}/\text{Al5}$	1860	2.7
$\text{RDX}_{\text{ph}}/\text{Al90}$	1850	3.3

The calorimeter is surrounded by constant-temperature jacket. The thermal equivalent of the instrument was determined by burning about 10 g of benzoic acid in oxygen at a pressure of 2.5 MPa. The maximum deviation in determination of the thermal equivalent was $\pm 1\%$.

Weighed samples of explosives were pressed at 10 MPa in a mould with an inner diameter of 30 or 23.5 mm to give ca. 20 or 15 g pellets. The pellets were hung in the center of the bomb. Each charge had a fuse cavity and was initiated with a standard military fuse. To estimate the heat of explosion of the fuse, TNT and RDX_{ph} charges of different mass were detonated in the bomb filled with argon or air. Additionally, the results published in [7, 8] were taken into consideration. Finally, the heat of the fuse was found to be 11724 J in argon or nitrogen and 82850 J in air or the mixture of argon/oxygen 80/20 vol%.

Technical gases were used: argon, nitrogen, air and argon/oxygen mixture at a pressure of 2.0 MPa as an atmosphere in the bomb. At least two measurements were performed for each explosive. To cal-

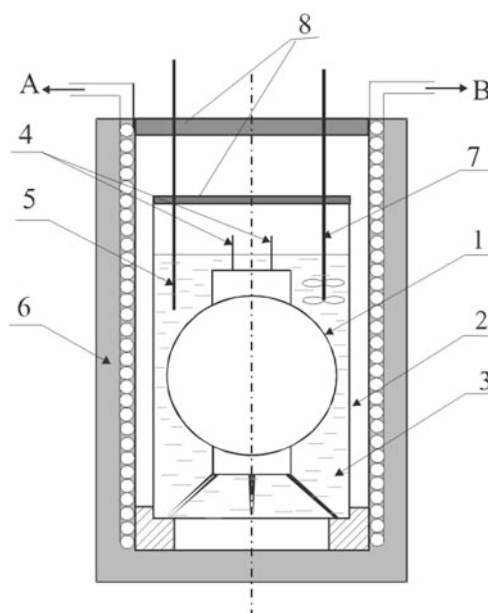


Fig. 6 A scheme of the water calorimetric set: 1 – bomb case, 2 – calorimeter bucket, 3 – water, 4 – ground and insulated voltage terminal, 5 – thermometer, 6 – constant-temperature jacket, 7 – stirrer, 8 – covers

culate the explosion heat of the explosive mixtures, the difference between the measured total heat effects and the heat released by the fuse was divided by the mass of the tested charge. The obtained results are presented in Tables 2 and 3.

The detonation heat of neat RDX_{ph} in argon atmosphere was measured in works [14] and [20]. The values of 5508 and 5344 J g⁻¹ were obtained, respectively. The heat of detonation in air evaluated in this work was 11840 J g⁻¹. So, the detonation energies released by the explosive component in the RDX-based mixtures tested were about 3740–3860 and 8290 J g⁻¹ for inert gaseous

filler and oxygen enriched atmosphere, respectively. These values are in a quite good agreement with the heats measured for the compositions containing inert additions (Tables 2 and 3).

As it was expected, mixtures of RDX_{ph} with Al produce significantly more energy of explosion in the calorimetric bomb than the mixtures with inert additives. Undoubtedly, the additional heat effects in air or oxygen/argon atmosphere are produced by chemical reactions between aluminium and oxygen present in the bomb. It was noticed that mixtures with Al5 powder generate a bit more energy than that of containing Al90.

Table 2 Calorimetric heats measured at 2 MPa for the mixtures with inert additives

RDX _{ph} /LiF			RDX _{ph} /Al ₂ O ₃		
Gaseous filler	Charge mass/g	$Q/J\ g^{-1}$	Gaseous filler	Charge mass/g	$Q/J\ g^{-1}$
Nitrogen	20.42	3910	Nitrogen	20.11	3941
	20.16	3737			
	14.95	3866			
	14.35	3937		20.02	3756
		$Q_{aver}/J\ g^{-1}=3863$			$Q_{aver}/J\ g^{-1}=3848$
Argon	20.75	3843	Argon	20.01	3904
	21.26	3912		20.09	3734
		$Q_{aver}/J\ g^{-1}=3877$			$Q_{aver}/J\ g^{-1}=3819$
Air	20.09	7685	Air	19.97	7369
	20.18	7827		20.09	7211
		$Q_{aver}/J\ g^{-1}=7747$			$Q_{aver}/J\ g^{-1}=7290$
O ₂ /Ar	20.01	7916	O ₂ /Ar	19.99	7764
20/80	20.07	7790	20/80	20.00	7748
		$Q_{aver}/J\ g^{-1}=7953$			$Q_{aver}/J\ g^{-1}=7756$

Table 3 Calorimetric heats measured at 2 MPa for the mixtures with reactive additives

RDX _{ph} /Al90			RDX _{ph} /Al5		
Gaseous filler	Charge mass/g	$Q/J\ g^{-1}$	Gaseous filler	Charge mass/g	$Q/J\ g^{-1}$
Nitrogen	20.04	7036	Nitrogen	19.95	7267
	20.06	7064		20.00	7337
		$Q_{aver}/J\ g^{-1}=7050$			$Q_{aver}/J\ g^{-1}=7302$
Argon	20.25	6996	Argon	19.99	7244
	20.05	7139		20.04	7216
		$Q_{aver}/J\ g^{-1}=7067$			$Q_{aver}/J\ g^{-1}=7230$
Air	15.12	16624	Air	15.07	17323
	15.06	16644		14.98	16719
				14.98	16854
		$Q_{aver}/J\ g^{-1}=16634$			$Q_{aver}/J\ g^{-1}=16965$
O ₂ /Ar	15.05	16850	O ₂ /Ar	14.98	17073
20/80	15.13	16961	20/80	14.97	16976
		$Q_{aver}/J\ g^{-1}=16905$			$Q_{aver}/J\ g^{-1}=17025$

This might be caused by differences in the relevant additives' specific surface, and a higher degree of fine powdered Al consumption in reactions.

Explosion heats for each tested mixture measured in argon and nitrogen atmosphere are similar. Thus, argon and nitrogen can be interchangeably used as inert gases. Explosion heats measured in air (nitrogen/oxygen ~80/20) and argon/oxygen 80/20 mixture are also close each another for the tested mixtures. Argon and nitrogen can be used as inert gases interchangeably in presence of oxygen.

Additionally, the heat of explosion under a pressure of 0.1 MPa of argon was also measured for some chosen RDX-based mixtures and for 20 g charge of TNT. The obtained results are presented in Table 4.

The explosion heat of TNT under pressure of 0.1 MPa of argon is relatively low and it strictly corresponds with heat measured for TNT in [14]. In this paper a significant increase in heat released from 3185 to 4439 J g⁻¹ after increasing nitrogen pressure from 0.1 to 2.0 MPa was observed. This means that secondary reactions inside the reshocked detonation products are reduced with increasing the initial pressure. This phenomena is observed especially for neat explosives (without a metal additive) with high negative oxygen balance ([4, 14, 15]). In the case of RDX_{ph}/Al₂O₃ mixture, which has relatively small deficit of oxygen to H₂O–CO₂ level, the decrease in argon pressure from 2 to 0.1 MPa lowers the heat of explosion by about 6%. Similar effect was observed by Ornellas for confined and unconfined charges of explosives with a small negative oxygen balance [4].

Despite the significant deficit of oxygen (–55%) in the RDX_{ph}/Al90 mixture, no noticeable difference is observed between the explosion heat measured at 2 and 0.1 MPa pressure of argon. This effect may be caused by two phenomena occurring during the process of recompression of the detonation products in the bomb filled with an inert gas under low initial pressure. Secondary reactions in the detonation products decrease the heat effect [4]. On the other hand, an increase of heat is caused by the reaction of aluminium particles with the detonation products. Both processes are limited when the initial pressure of argon is 2 MPa.

Table 4 Calorimetric heats measured at 0.1 MPa pressure inside the bomb

Mixture	Charge mass/g	$Q/J\ g^{-1}$	$Q_{aver}/J\ g^{-1}$
RDX _{ph} /Al90	20.35	6944	6984
	20.20	7023	
RDX _{ph} /Al ₂ O ₃	20.24	3577	
TNT	19.92	3266	

Thermochemical calculations

Thermochemical calculations in this work were performed by the use of the CHEETAH code [22]. The BKWS set of parameters [23] for the BKW equation of state and covolume factors for gaseous species were applied. The results of calculations are presented in Table 5.

The heat of explosion measured in 2 MPa inert atmosphere was compared with the total energy of detonation calculated using CHEETAH code. The total energy, E_d , is a sum of mechanical and thermal energies. The mechanical energy represents the amount of energy available to do mechanical work and it is equal to the expansion work of detonation products and determined for the volume of detonation products corresponding to pressure $p=1$ atm. The thermal energy represents the amount of energy locked up as heat of detonation products at $p=1$ atm [22]. For calculations, the composition of detonation products was frozen at the temperature of 1800 K on the isentrope beginning at the CJ point [1, 4].

Comparison of calculated detonation energy with calorimetric heat for the mixtures containing inert additives shows quite good agreement (Fig. 7). This means that there are no secondary reactions inside the bomb filled with inert atmosphere (argon or nitrogen). But a similar comparison performed for the mixtures with aluminium powders indicates that Al particles react partially with the detonation products, because the calorimetric heat measured for these mixtures is greater than the detonation energy calculated for the compositions containing inert additives. On the other hand, it is lower than the detonation energy calculated for complete reactions of the aluminium. Assuming that only aluminium oxide is present in the calculated solid products, we can conclude, that about 50% of aluminium mass is consumed inside the bomb.

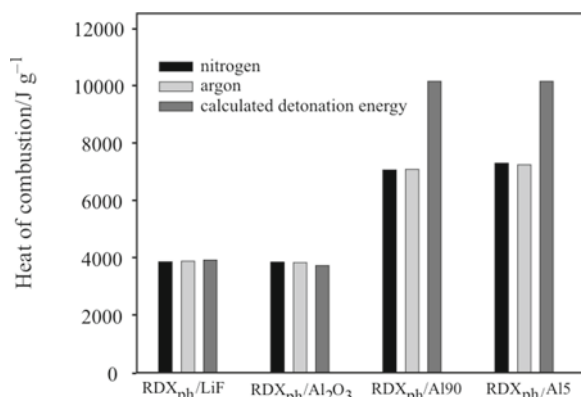
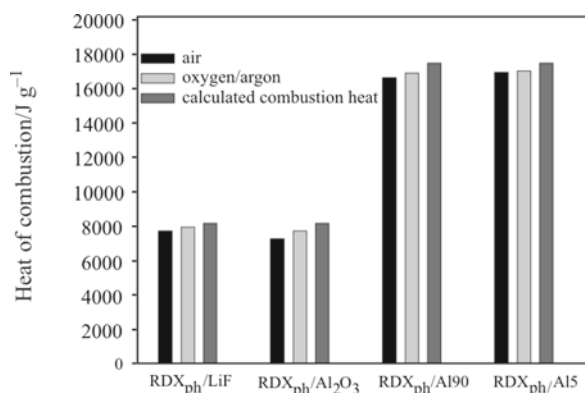


Fig. 7 Comparison of the heat effect of detonation measured in inert atmospheres with the calculated detonation energy for tested compositions

Table 5 Comparison of the calorimetry data at 2 MPa and the results of thermochemical calculations

Explosive mixture	Gaseous filler	$Q_{aver}/$ $J g^{-1}$	$E_d/$ $J g^{-1}$	$Q_c/$ $J g^{-1}$
RDX _{ph} /LiF	nitrogen	3863		
	argon	3877		
	air	7747	3916	8180
	oxygen/ argon 20/80	7953		
RDX _{ph} /Al ₂ O ₃	nitrogen	3848		
	argon	3819		
	air	7290	3723	8180
	oxygen/ argon 20/80	7756		
RDX _{ph} /Al90	nitrogen	7050		
	argon	7067		
	air	16634	10150	17497
	oxygen/ argon 20/80	16905		
RDX _{ph} /Al5	nitrogen	7302		
	argon	7230		
	air	16965	10150	17497
	oxygen/ argon 20/80	17025		


Fig. 8 Comparison of the heat effect of detonation measured in oxygen enriched atmospheres with the calculated combustion heat for tested compositions

The heat of explosion measured in the bomb filled with an atmosphere containing oxygen (air or oxygen/argon 20/80) was compared with the heat calculated for a constant-volume explosion of a charge and gaseous filler (Fig. 8). Because only completely oxidized species are present in the calculated explosion products, we can consider the

heat calculated in this way as a combustion heat, Q_c . From the data shown in Fig. 8 it follows that for all the tested explosives the calorimetric heat measured in an oxygen-rich gaseous filler is slightly lower than the theoretical heat of combustion. This fact indicates that the presence of oxygen in the atmosphere filling a bomb leads to complete interaction between products (including aluminium) and oxygen.

Conclusions

The most important conclusions from the results obtained are:

- Mixtures of RDX_{ph} with aluminium particles produce significantly more energy during explosion in the calorimetric bomb than the mixtures with inert additives regardless the type of atmosphere.
- Mixtures with fine aluminium powder (Al5) produce a bit more energy than that of containing coarse aluminium (Al90).
- Explosion heats measured for all tested mixtures are almost the same in argon and nitrogen atmosphere. Argon and nitrogen can be used as inert gases interchangeably.
- Explosion heats for all tested mixtures are similar in air and argon/oxygen mixture. Argon and nitrogen can be used as inert gases interchangeably in presence of oxygen.
- Calculated detonation energy and calorimetric heat measured in an atmosphere without oxygen are in quite good agreement for the mixtures containing inert additives. But the calorimetric heat measured for the RDX_{ph}-aluminium mixtures in similar conditions is greater than the detonation energy calculated for the compositions containing inert additives and it is much lower than the detonation energy calculated assuming reactive aluminium. About 50% of aluminium mass is burnt inside the bomb filled with argon or nitrogen.
- The presence of oxygen in the compressed gas filling a bomb leads to complete combustion of the detonation products and aluminium particles.

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