



RESEARCH ARTICLE

Performance of TKX-50 in thermobaric explosives

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Abstract

In this study, the behavior of the high-nitrogen compound TKX-50 in model thermobaric formulations was investigated. The addition of 10% Al to TKX-50 reduces the heat of detonation by approximately 90 J/g. Despite this, Al reacts with the detonation products of TKX-50 in an exothermic manner, and the energy contribution was calculated to be approx. 375 J/g. In addition, the overpressure in the explosion chamber filled with argon after detonation of aluminized TKX-50 charges containing 27% Al is approx. 20% higher than in the case of neat TKX-50. Also the maximum temperature of the TKX-50/Al explosion products in the argon filled chamber is higher by 370 K than that of measured after detonating TKX only. What is more aluminum oxynitride with a low nitrogen content has been identified in the solid detonation products of aluminized TKX-50, but only for detonations in argon. Of course, charges made of TKX-50/Al mixture generate significantly higher overpressure and radiant temperature values in a confined space when they are detonated in an air atmosphere. It all means that burning aluminum in nitrogen provides little energy, and even if the concentration of nitrogen in the post-detonation products is much higher than that of oxygen, aluminum oxides are preferentially formed.

KEYWORDS

confined explosion parameters, detonation heat, thermobaric explosives, TKX-50

1 | INTRODUCTION

Current state-of-the-art thermobaric explosives (TBXs) contain high explosives (usually RDX or HMX), metal powders (mainly Al and/or Mg), an additional oxidizer (NH_4ClO_4 or NH_4NO_3) and a binder (usually plasticized HTPB) [1]. During the explosion of TBX charges, a significant part of the metallic additive burns using oxygen from the air. This process occurs in the expanding detonation products which are released. The energy and gaseous products released in these reactions cause a slower

drop in the pressure and temperature in the vicinity of the epicenter of the explosion to occur, thus increasing the blast performance. The advantage of thermobaric ammunition is primarily due to the fact that the warhead carries mainly high-energy combustible components, and the oxygen needed for their combustion is taken from the atmosphere surrounding the explosion site.

Despite the simplicity of this idea, creating an effective TBX is not a trivial task. It is necessary to find a sufficiently reactive (prone to oxidation) metal, which as a

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powder will ignite even before oxygen from the air appears in the detonation products. In order for this to occur, the metal powder must participate in exothermic reactions with primary decomposition products of an explosive compound, i.e. primarily with water and carbon oxides, as well as possibly also nitrogen. All these gases are very inactive oxidizers, and in addition, at this stage of the explosion, the metal is still in the condensed phase and its contact with gaseous oxidizers is very limited (also due to the oxide coatings on the surface of the metal particles). In order to increase the concentration of oxygen and nitrogen oxides in the post-detonation anaerobic stage of combustion reactions, modern TBXs must contain up to 30% of additional oxidizers. However, this means a reduction in the quantity of the explosive component is necessary, and a lowering the detonation parameters is consequently observed.

A possible strategy to eliminate this drawback of oxidizer-containing TBXs could be the replacement of conventional high explosives (RDX, HMX) with high-nitrogen explosives. This promising approach aims to reduce the amount of oxidizer which is necessary, and to partly convert the metallic fuel into aluminum nitride (AlN). If NH_4ClO_4 or NH_4NO_3 do not have to be added to the TBX formulation, then the same mass of formulation is able to contain a higher mass percent of a N-rich high explosive than the oxidizer-containing TBX.

In this work, the energy contribution of the Al micron particles to the heat of detonation and the confined explosion parameters of dihydroxylammonium-5,5'-bis-tetrazole-1,1'-diolate (TKX-50) was investigated. Calorimetric measurements of the heat of detonation of neat TKX-50 and its mixtures with Al powder (10%) were performed under identical conditions. In addition, the overpressure and radiant temperature histories in the explosion chamber filled with air or argon were measured after detonation of 50 g charges prepared from TKX-50 passivated with 3% of paraffin, as well as from mixtures composed of 70% TKX-50, 27% Al powder and 3% paraffin. Solid detonation products recovered from the bomb calorimeter and the explosion chamber after detonations performed in air and argon were also analyzed.

2 | EXPERIMENTAL

2.1 | Materials

Dihydroxylammonium-5,5'-bis-tetrazole-1,1'-diolate (TKX-50) was synthesized according to our previously published procedure [2–4]. Al powder was purchased from Alfa-Aesar (Cat. No 11067), and was used as obtained. The purity was higher than 99.5%, with particle size less than $43\ \mu\text{m}$ (325 mesh).

When measuring the heat of detonation, it is necessary to use TKX-50 of high purity. Therefore, for calorimetric measurements, charges containing no binder were prepared, despite the fact that they are characterized by low mechanical strength [5]. The powdery samples were pressed (at 100 MPa) into approx. 5 g cylinder pellets of 25 mm diameter. The detonated charges were composed of 5 pellets, so that the mass of the charge was approx. 25 g.

Samples which were used for testing in the explosion chamber contained 3% paraffin. Paraffin (delivered by ROTH, Art. No CN49.2) was dissolved in chloroform, and the solution in the designed ratio, was added to a pre-weighed sample of neat TKX-50 or TKX-50/Al mixture. The resulting putty-like substance was kneaded/rubbed to obtain a powdery consistency. The resulting granulates were then dried to constant weight, before being pressed into charges with a diameter of 30 mm. TKX-50 is a relatively insensitive explosive, so a 5 g booster of wax-passivated RDX (RDX/wax 94/6) was pressed into the bottom of the charge. The total weight of each charge detonated in the chamber was approx. 50 g, which included 45 g of the explosive being tested.

All of the charges were weighed and measured in order to enable calculation of the densities. Results are shown in Table 1.

2.2 | Calorimetric measurements

A spherical steel bomb with an internal volume of $5.6\ \text{dm}^3$ was the main component of the water

TABLE 1 Composition and density of the tested charges.

Explosive	Charge diameter [mm]	Average density [g/cm^3]
TKX-50	25.0	1.50 ± 0.03
TKX-50/Al (90/10)	25.0	1.52 ± 0.02
TKX-50/Paraffin (97/3)	30.0	1.48 ± 0.03
TKX-50/Al/Paraffin (70/27/3)	30.0	1.55 ± 0.01

calorimeter used for the measurements. The bomb was placed in a polished stainless-steel calorimeter bucket that held 27000 ± 1 g of water. The bucket was surrounded by a constant-temperature jacket, the temperature of which was maintained at 20.0 ± 0.1 °C. The thermal equivalent of the instrument, determined by burning certified samples of benzoic acid with purified oxygen at a pressure of 2.0 MPa, was found to be 163.7 ± 1.2 kJ/K. The error of $\pm 0.73\%$ indicates the ultimate precision of the instrument.

The 25.0 g TKX-50 or TKX-50/Al charges were detonated in the bomb calorimeter which was filled with argon to a pressure of 2.0 MPa. Standard military detonators were used to initiate detonation. The detonator consisted of primary and secondary explosives enclosed in an aluminum cup with mass of approximately 1.6 g. To estimate the energy released by the detonator, TNT charges of different masses were detonated in the argon-filled bomb. Assuming that the heat of detonation of the fuse does not depend on the mass of the TNT charge (i.e. the degree of reaction that occurs between aluminum and the detonator explosion products with the gaseous products of TNT is independent of the TNT mass), the dependence of the total heat effect on the TNT mass should be linear. This assumption enabled the heat released by the fuse to be determined as being 11.4 ± 0.45 kJ. To calculate the heat of detonation of an explosive, the difference between the measured total heat effect and the heat released by the fuse was divided by the mass of the tested charge. Three measurements were performed for the tested explosives, and the results (J/g) were averaged and rounded to the nearest ten.

2.3 | Pressure and temperature history measurements

In order to determine the quasi-static pressure after detonation of the tested explosives in a closed volume, the charges were placed in a steel chamber with volume 150 dm^3 (Figure 1), and filled with air or argon under 0.1 MPa pressure. A charge was hung in the center of the chamber. A standard electric detonator and a 5 g RDX/wax booster were used to initiate detonation of the 45 g charges of TKX-50/paraffin and TKX-50/Al/paraffin formulations. The former is hereafter designated as TKX50_{ph}, the latter as TKX50_{ph}/Al. Charges of RDX/wax weighing 50 g were also tested for comparison. Three tests were performed for each explosive in the chamber filled with air or argon. Signals of overpressure from two piezoelectric gauges (PCB Piezotronics, model 102B) located at the chamber wall were recorded by a digital storage scope. To limit the influence of the gas-

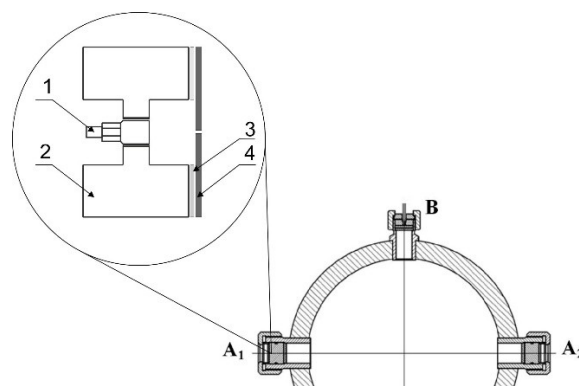


FIGURE 1 Schematic diagram of the 150 dm^3 chamber: A₁, A₂ – pockets with pressure gauges, B – fiber optic sensor, 1 – a PCB gauge, 2 – gauge socket, 3 – Teflon seal, 4 – steel diaphragm with a 3 mm hole.

dynamical processes on the averaged overpressure history, the gauges were placed in special pockets (Figure 1).

To measure a light intensity spectrum, a fiber optic sensor was placed in the wall of the chamber in a such way as to be directed to the charge. Light from the fireball was sent to a spectrometer USB2000+ via an optic fiber cable. Spectra were collected in the visible and infrared range of wavelengths (from 300 nm to 1100 nm). Radiation intensity was recorded using a 2048-element linear silicon CCD array. A single measurement was made by integrating the intensity over 1 ms. After this time, the data was transferred to the computer. The data transfer rate was 2 ms. Thus, the frequency of spectrum measurement was performed at about 330 Hz.

2.4 | Purification and analysis of the solid detonation products

The solid detonation products which were extracted from the bomb calorimeter and explosion chamber after the detonation of three charges of aluminized TKX-50, were sieved through a $250 \mu\text{m}$ sieve and then introduced into 20% hydrochloric acid. The suspension which was obtained was heated at 60 °C for 8 hours under reflux, after which time, the remaining precipitate was filtered off, washed with distilled water and ethanol and dried to constant weight. During leaching of the detonation products recovered from the argon filled chamber with HCl (aq), intense evolution of hydrogen was observed, indicating the presence of a high metallic aluminum content. After purification, the mass of the sample decreased by 43.2%, while the mass reduction of the products of air explosions was almost twice lower (22.0%).

X-ray powder diffraction patterns were recorded using a Rigaku SmartLab 3 kW diffractometer equipped with a Detex250 line detector and working in Bragg-Brentano geometry (2-theta range of 10–90° and a step of 0.02°). A Cu lamp with Ni filter was used, and the voltage and current were 40 kV and 30 mA respectively. The samples under investigation were placed on a Si510 monocrystal. The ICDD standard database type PDF4+2022 was used to identify the phases.

3 | RESULTS AND DISCUSSION

3.1 | Heat of detonation results

Results of the heat of detonation measurements are shown in Table 2.

The heat of detonation of TKX-50/Al = 90/10 mixture is lower than that of neat TKX-50 by 90 J/g. Despite this, Al reacts with the detonation products of TKX-50 in the post-detonation stage in an exothermic manner, because the difference between the heat of detonation of the mixture and the heat of detonation of pure TKX-50 (4185 J/g that is 90% out of 4650 J/g) corresponds to 375 J/g (4560–4185 = 375). Thus, the energy contribution, given in the last column in Table 2, is the energy released due to reactions of Al with TKX-50 detonation products.

The initial detonation products of TKX-50 contain mainly nitrogen, carbon oxides, water and small amounts of nitrogen oxides and free oxygen. All of these gases can react with Al in exothermic reactions in the post-detonation stage. The heats of combustion of aluminum in O₂, CO₂, CO, H₂O and N₂ are: 31.00 kJ/g, 14.07 kJ/g (CO₂ reduced to CO), 11.59 kJ/g, 8.02 kJ/g (H₂O_(g)) and 6.41 kJ/g, respectively [6]. So that thermodynamically, reactions between Al and oxygen or carbon oxides are preferred, while kinetically, reactions with nitrogen are preferred, because its concentration in the detonation products is the highest. From the values for the heats of combustion of Al given above it follows that 375 J of energy is released by burning 12.8 mg of Al in oxygen, or 58.5 mg of Al in nitrogen. In a 1 g sample of TKX-50/Al (90/10) there is 100 mg of Al present, which means that under the conditions used for the calorimetric measurements, the percentage of Al which reacts with the decomposition products of TKX-50 ranges from

12.8% to 55.8%. The amount of oxygen present in 0.9 g of TKX-50 is sufficient to oxidize 24.9 mg of Al. Consequently, increasing the Al content in the mixture should result in an increase in the heat of detonation. Unfortunately, TKX-50/Al mixtures which contain more than 10% Al are not able to detonate after initiation with only a standard detonator.

3.2 | Quasi-static pressure results

Six overpressure profiles were recorded for each of the tested compositions both in argon and air atmospheres. Figures 2 and 3 show overpressure histories recorded for the TKX50_{ph} and TKX50_{ph}/Al charges in the chamber when filled with air and argon, respectively. The overpressure records have an oscillating nature caused by shock reverberations at the chamber wall and vibrations of the measuring system. Placing the sensor in the socket caused the average overpressure to increase in the initial phase. To determine the quasi-static pressure (QSP), the measured dependence of overpressure on time was approximated by the function:

$$\Delta p = a(1 - e^{-bt}) + ce^{-dt} \quad (1)$$

where a , b , c and d are constants, and t is time. The first term in Eq. (1) indicates the growth in the average

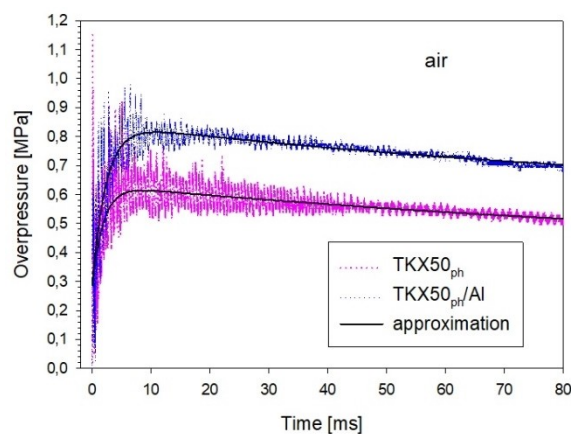


FIGURE 2 Overpressure histories recorded after detonation of the TKX50_{ph} and TKX50_{ph}/Al charges in the chamber filled with air, as well as the approximated overpressures (solid line).

TABLE 2 Average results obtained from detonation calorimetry performed in an argon atmosphere of 2 MPa.

Explosive	Average heat of detonation [J/g]	Standard deviation	Energy contribution of Al [J/g]
TKX-50	4650	50	–
TKX-50/Al (90/10)	4560	35	375

TABLE 3 Measured and calculated values for the maximum overpressure in the chamber.

Parameter	TKX50 _{ph}		TKX50 _{ph} /Al		RDX _{ph}	
	Air	Ar	Air	Ar	Air	Ar
Δp_{max} [MPa]	0.63	0.48	0.81	0.58	0.79	0.64
Δp_{cal} [MPa]	0.97	0.70	1.14	0.92	1.04	0.79
$\Delta p_{max}/\Delta p_{cal}$ [%]	65	69	71	64	76	81
Δp_{inAl} [MPa]	-	-	0.76	0.49	-	-
$\Delta p_{max}/\Delta p_{inAl}$ [%]	-	-	107	118	-	-

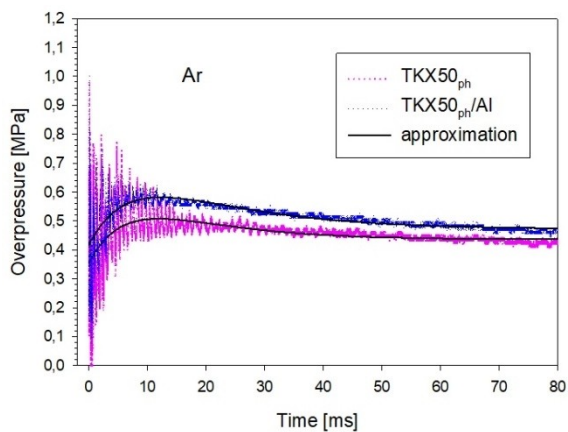


FIGURE 3 Overpressure histories recorded after detonation of the TKX50_{ph} and TKX50_{ph}/Al charges in the chamber filled with argon, as well as the approximated overpressures (solid line).

overpressure which was caused by the shape of the socket and afterburning of the detonation products. The second term describes the small decrease observed in the average overpressure histories during the recording time (80 ms). Approximations of the overpressure histories according to Eq. (1) are shown in Figure 2. Function (1) reaches a maximum value Δp_{max} for a time t_{max} calculated from the equation:

$$t_{max} = \ln\left(\frac{ab}{cd}\right) \frac{1}{b-d} \quad (2)$$

Values of Δp_{max} determined on the basis of the recorded overpressure histories are given in Table 3. The parameter Δp_{max} was defined as the quasi-static pressure for the tested explosives.

To estimate the theoretical overpressure in the chamber, thermochemical calculations were performed using the CHEETAH code, [7] with modified BKWS library of the detonation products [8]. The overpressure Δp_{cal} was calculated for an explosion at constant volume for a charge, and in which air or argon were present in the chamber. Thermochemical equilibrium was assumed in these calculations. The overpressures Δp_{inAl} were also

calculated with assuming chemical inertness of Al. The results of these calculations are presented in Table 3.

As can be seen in Table 3, the values of Δp_{max} are lower than the calculated Δp_{cal} values for all of the charges detonated in air. The ratio $\Delta p_{max}/\Delta p_{cal}$ is 65 % for TKX50_{ph}, and slightly higher (71 %) for the aluminized charge. This means that the mixing of the detonation products with air in the chamber, as well as their thermochemical equilibrium are not perfect. The overpressure and degree of afterburning of detonation products in the air-filled chamber are higher for RDX_{ph} than for TKX50_{ph}. The reason for this appears to be the higher heat of detonation of the former (5340 J/g [9]) compared to the latter (4650 J/g). The overpressure Δp_{max} for TKX50_{ph} is lower than that of TKX50_{ph}/Al, suggesting that Al reacts with the detonation products of TKX-50 and with oxygen from the air. This conclusion is confirmed by comparing the measured Δp_{max} with calculated QSP values assuming Al activity (Δp_{cal}) and Al chemical inertness (Δp_{inAl}). Similar relationships are observed in Table 3 for the explosion parameters in the argon-filled chamber, but the measured and calculated overpressure values are much lower in this case.

3.3 | Fireball spectra analysis and temperature results

Figure 4 shows typical light intensity spectra which were recorded using the spectrometer after detonation inside the explosion chamber. The continuous part of the spectra was used to derive the fireball temperature by the method described in detail in [10]. The wavelength interval considered was between 500 and 850 nm. The spectral radiation intensity was obtained from the raw signal by multiplying the experimental intensity with the sensitivity factor.

As can be seen in Figure 4, in the spectra of the TKX50_{ph}/Al explosion products there are also sharp absorption and emission peaks. The sharp lines at 589.1 nm (absorption) and 819.5 nm (emission) are attributed to sodium cations and atoms [8]. They are

intense, because TKX-50 may contain up to 0.5% of the sodium salt of 5,5'-bistetrazole-1,1'-diol, which is the direct precursor in the synthetic route [1–3]. The peaks observed at 766.5 nm and 769.9 nm indicate emission of radiation by potassium ions. These peaks were present in all of the spectra which were obtained – also in the case of RDX_{ph} .

Figure 5 shows the radiance obtained for the $\text{TKX50}_{\text{ph}}/\text{Al}$ charge. The peaks resulting from the presence of sodium and potassium impurities were omitted (the emission was approximated by a straight line segment).

The temperature was estimated by a non-linear polychromatic fitting of the spectral radiance variation with the wavelength using Planck's law [8].

The changes in temperature with time for the TKX50_{ph} and $\text{TKX50}_{\text{ph}}/\text{Al}$ charges detonated in the air atmosphere are shown in Figures 6 and 7. Only moderate

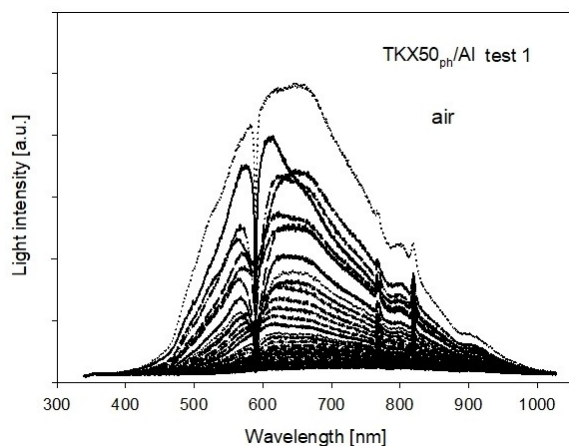


FIGURE 4 Light-intensity spectra for an $\text{TKX50}_{\text{ph}}/\text{Al}$ charge in air.

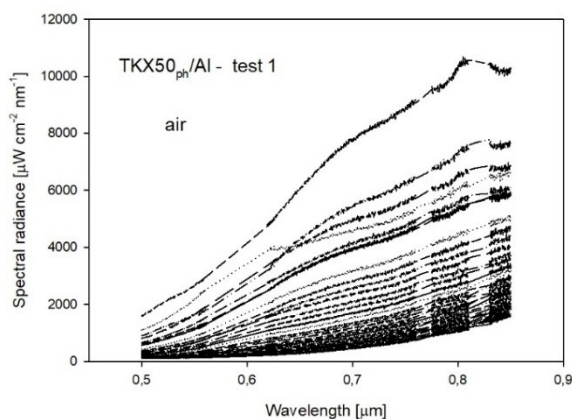


FIGURE 5 Spectral radiance derived from the recorded light intensity of a $\text{TKX50}_{\text{ph}}/\text{Al}$ charge in air.

differences in the temperature/time profiles were observed for the same explosive from measurement to measurement.

Adding aluminum to TKX-50 significantly increases the maximum temperature of the explosion products in the chamber (by 700 K), and almost triples the duration of time at which a temperature exceeding 1600 K is maintained (Figure 7). This is the reason for the increase in Δp_{max} which is observed for aluminized TKX50_{ph} charges detonated in air (Table 3). Plateau visible in the temperature profiles at 2100–2200 K (Figures 7 and 8), is most likely the result of intense combustion of aluminum particles in the air.

A comparison of the temperature variations for the tested charges detonated in the chamber filled with air and argon are presented in Figures 8 and 9, respectively.

As expected, the temperatures measured in the air atmosphere are significantly higher than those recorded in

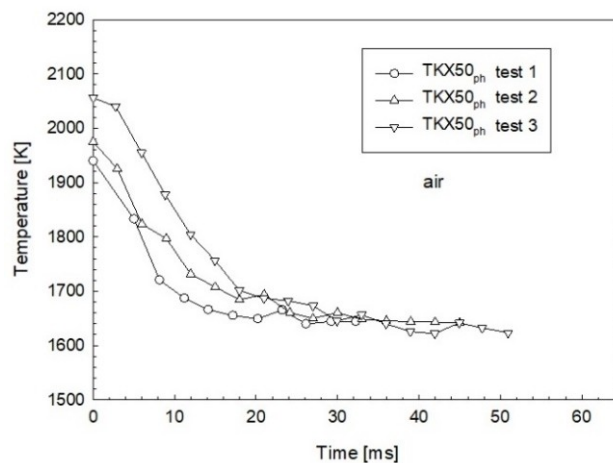


FIGURE 6 Temperature profiles for TKX50_{ph} charges detonated in the chamber filled with air.

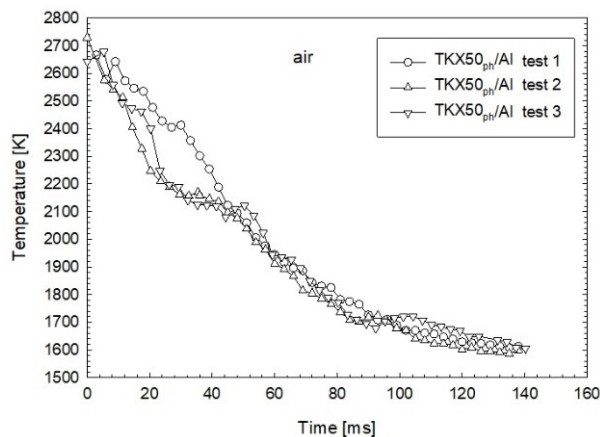


FIGURE 7 Temperature profiles for $\text{TKX50}_{\text{ph}}/\text{Al}$ charges detonated in the chamber filled with air.

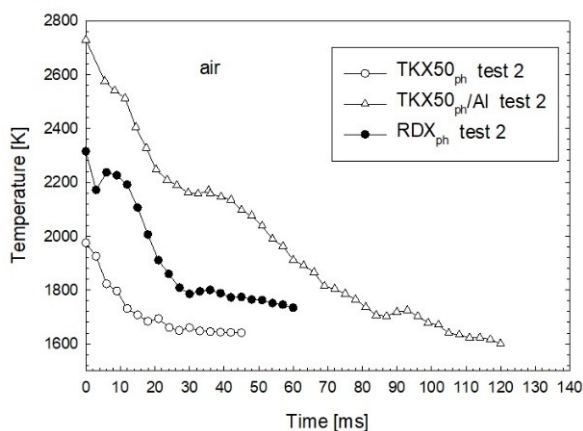


FIGURE 8 Comparison of temperature histories for the charges detonated in the explosion chamber filled with air.

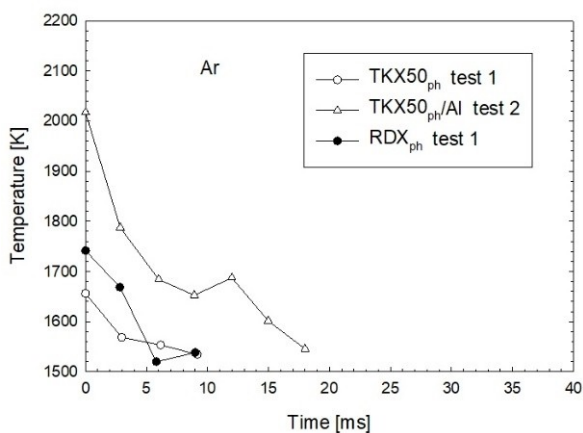


FIGURE 9 Comparison of temperature histories for the charges detonated in the explosion chamber filled with argon.

argon. Also, the duration of time the elevated temperature in the post-explosion cloud is maintained is much longer in the former case. It is noteworthy that the temperatures measured for RDX_{ph} are higher than those obtained for $TKX50_{ph}$, which corresponds well with the results obtained from QSP measurements (Table 3).

3.4 | Phase composition of solid detonation products

XRD patterns of the detonation products recovered from the explosion chamber and calorimetric bomb after three measurements both in air (red, detonations only in explosion chamber) and in argon (black, detonations in the chamber; blue detonations in the calorimetric bomb) are shown in Figure 10. Only in the case of detonations performed in an argon atmosphere, is aluminum oxide

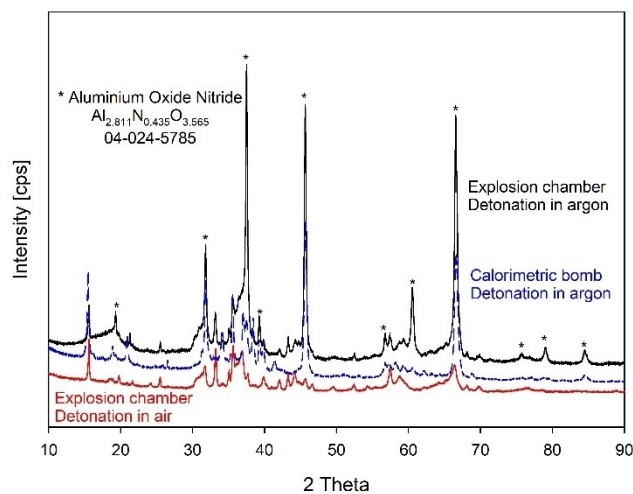


FIGURE 10 XRD patterns of the detonation products of aluminized TKX-50 obtained in an argon atmosphere (black and blue) and in an air atmosphere (red).

nitride (AlON) with formula $Al_{2.811}N_{0.435}O_{3.565}$ the dominating phase. The reflexes for AlON in the detonation products recovered from the explosion chamber are (as expected) much more intense than those observed for the products recovered from the calorimetric bomb. This is explained by the fact that the charges detonated in the chamber contained 27% Al, while the heat of detonation was measured for a TKX-50/Al composition containing only 10% Al. In addition to AlON, diaoyudaoite ($NaAl_{11}O_{17}$, 01-079-2288) was also identified in the detonation products from tests performed in an argon atmosphere.

The main components of the detonation products resulting from the detonations performed in air are corundum (04-004-5434) and aluminum oxide with formula $Al_3O_{3.76}$ (04-016-0538). The phases corresponding to aluminum oxynitrides are essentially not observed in the detonation products of TKX-50/Al mixtures obtained in an air atmosphere. This shows that in the presence of oxygen aluminum does not react with nitrogen, even if the concentration of nitrogen is very high. It can also be assumed that in the aluminum-nitrogen compounds formed in the anaerobic phase, nitrogen is displaced by oxygen when atmospheric oxygen occurs in the expanding detonation products.

4 | CONCLUSIONS

Mixtures of TKX-50 with micron-sized aluminum powder (containing 10% and 27% Al) have been prepared and the influence of Al addition on the heat of detonation as well as on the confined explosion parameters

in argon and air atmospheres has been investigated. The following conclusions were obtained.

- (1) The heat of detonation of TKX-50/Al=90/10 mixture is lower than that of neat TKX-50 by 90 J/g. Despite this, Al exothermically reacts with the detonation products of TKX-50 in the post-detonation stage and the energy contribution equals to 375 J/g.
- (2) The maximum overpressure for TKX-50_{ph} (TKX-50/Paraffin=97/3) measured in an air atmosphere is by approx. 30% lower than that of TKX50_{ph}/Al (TKX-50/Paraffin/Al=70/3/27), which suggests that Al reacts with the detonation products of TKX-50 and with oxygen from the air. Similar relationships are observed for the explosion parameters in the argon-filled chamber, but the measured overpressure values are much lower in this case (by 25–30%).
- (3) Adding aluminum to TKX-50 significantly increases the maximum temperature of the explosion products in the chamber (by 700 K in an air atmosphere), and almost triples the duration of time at which a temperature exceeding 1600 K is maintained. As expected, the temperatures measured in an argon atmosphere are significantly lower than those recorded in air, but still aluminized TKX-50 performs better than neat TKX-50 (by 370 K).
- (4) Only in the case of detonations performed in an argon atmosphere, is aluminum oxide nitride with formula $Al_{2.811}N_{0.435}O_{3.565}$ the dominating phase. The phases corresponding to aluminum oxynitrides are essentially not observed in the detonation products of TKX-50/Al mixtures obtained in an air atmosphere.

In conclusion, burning aluminum in nitrogen provides little energy, and even if the concentration of nitrogen in the post-detonation products is much higher than that of oxygen, it is not able to compete with it.

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CONFLICT OF INTEREST STATEMENT

The authors declare no competing financial interest.

DATA AVAILABILITY STATEMENT

Data may be requested via the authors.

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SUPPORTING INFORMATION

Additional supporting information can be found online in the Supporting Information section at the end of this article.

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