

Detonation properties of 1,1-diamino-2,2-dinitroethene (DADNE)

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Abstract

1,1-Diamino-2,2-dinitroethene (DADNE, FOX-7) is an explosive of current interest. In our work, an advanced study of detonation characteristics of this explosive was performed. DADNE was prepared and recrystallized on a laboratory scale. Some sensitivity and detonation properties of DADNE were determined. The detonation performance was established by measurements of the detonation wave velocity, detonation pressure and calorimetric heat of explosion as well as the accelerating ability. The JWL (Jones–Wilkins–Lee) isentrope and the constant- γ isentrope for the detonation products of DADNE were also found.

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1. Introduction

1,1-Diamino-2,2-dinitroethene (DADNE, FOX-7) is commonly expected to be an useful explosive combining comparatively high performance and low sensitivity. The unique characteristics of DADNE result from its molecular structure [1], which is conducive to creation of strong inter- and intramolecular hydrogen bonds stabilizing the molecule. Since 1998 when DADNE was synthesized by Latypov et al. [2], it has been a subject of many experimental investigations [3–11]. DADNE has been found to be far less sensitive to impact and shock than RDX (1,3,5-trinitro-1,3,5-triazinane) whereas its explosive properties are comparable to that of RDX. For these reasons DADNE has appeared to be very useful for systems in which its insensitivity is more important than maximum performance.

In our laboratory we dealt with the optimization of DADNE synthesis and crystallization process, the chemical and phase composition of products crystallized from different solvents and the thermal analysis of DADNE samples obtained under different conditions [6–9]. The main objectives of present work were to determine some explosive properties of DADNE. The detonation velocity, pressure and heat were measured. Results of the so-called cylinder expansion test were the basis for determining the acceleration ability (the Gurney energy) and

the detonation energy. Moreover, the coefficients of the JWL (Jones–Wilkins–Lee) isentrope and constant- γ isentrope for detonation products of DADNE were found. Some of those results were also presented in proceedings [10,11].

2. DADNE crystallization and main properties

DADNE was synthesized at our laboratory, according to the method disclosed by Latypov et al. [2] and then modified by Chyłek et al. [7]. In the first step 2-methylpyrimidine-4,6(3H,5H)-dione (**1**) was prepared by condensation of acetamide hydrochloride with diethyl malonate. The condensation product was separated and next nitrated with concentrated $\text{HNO}_3/\text{H}_2\text{SO}_4$ acids at room temperature giving 2-dinitromethylene-5,5-dinitropyrimidine-4,6(1H,3H)-dione (**2**) which was directly hydrolyzed in water to DADNE and dinitromethane (Fig. 1. Particle size distribution of the obtained material is shown in Fig. 3).

The raw product was stabilized by boiling its suspension in 0.1% aqueous solution of NaHCO_3 for 8 h under reflux condenser. In the next stage of purification, samples were recrystallized from different solvents. This was not only aimed at removing chemical impurities but also at obtaining spherical crystals with regular surfaces and diameters within a range of 10–300 μm .

At the beginning simple cooling crystallization from water was carried out, but the obtained crystals were quite small, with

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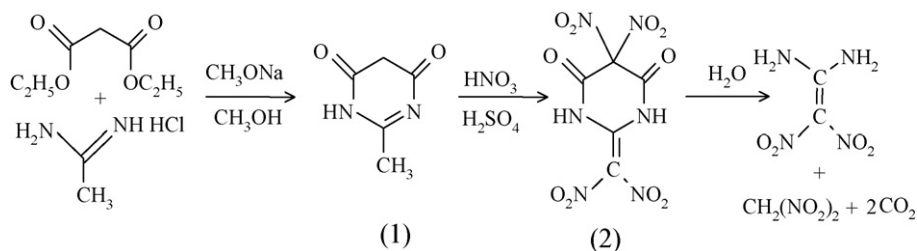


Fig. 1. DADNE synthesis from 2-methylpyrimidine-4,6(3H,5H)-dione [2,7].

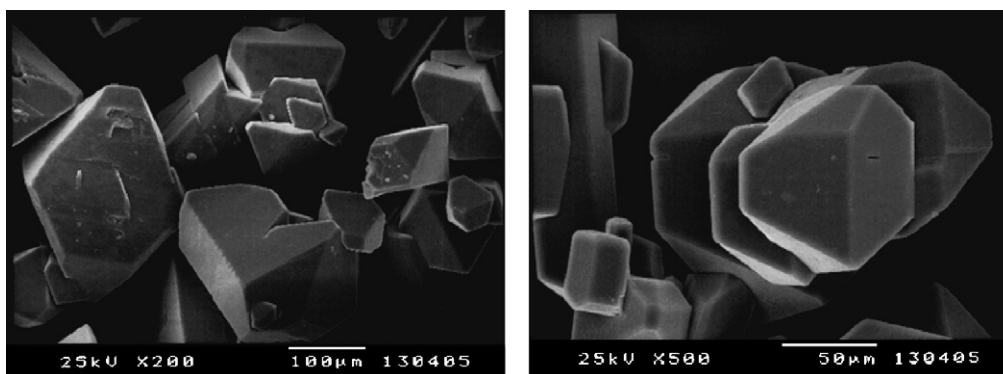


Fig. 2. SEM images of DADNE recrystallized from water/NMP = 75/25 at a cooling rate of 0.3 K/min.

irregular shapes and very porous surfaces. In order to unify shapes and smooth the surface of crystals, suspensions containing 50 g of the product crystallized from water and ca. 50 ml of a solvent were vigorously stirred for a long time (also at an elevated temperature). In the first place pure water and next water/*N*-methyl-2-pyrrolidone (H₂O/NMP) mixtures with different H₂O/NMP ratios were used. NMP was chosen, because it is one of the best solvents of DADNE, so that more DADNE could be dissolved. It was expected that mechanical interactions of crystals between themselves and the container walls, combined with dissolution of the smallest crystals and sharp edges of bigger crystals, would give product more homogenous with regard both to the crystal shape and dimension. However, it must be stated that the attempts to modify the dimension and shape of crystals by agitating them in water or water/NMP suspensions were not successful.

In the next step of investigation, water/NMP mixtures were used to recrystallize DADNE. Several experiments were

performed to assess the influence of cooling rate on the characteristics of the crystals. In each case, the suspensions of growing crystals were agitated while cooling [8,9]. Exemplary SEM images of crystals, obtained at a cooling rate of 0.3 K/min, are presented in Fig. 2. The particle diameters were estimated assuming their spherical shape.

From size distribution measurements, it follows that the fraction of particles with diameters below 50 μm is as high as ca. 60%, but mass fraction in the tested sample looks quite different—particles with diameters within a range of 250–500 μm clearly dominate.

In order to obtain a fine-grain product, DADNE was dissolved in NMP while boiling. The solution was poured into water at an ambient temperature and the suspension was vigorously stirred. After filtering the product was slowly dried in an air atmosphere. The fine-grain product of quite regular shapes was obtained. SEM images of the crystals are presented in Fig. 4.

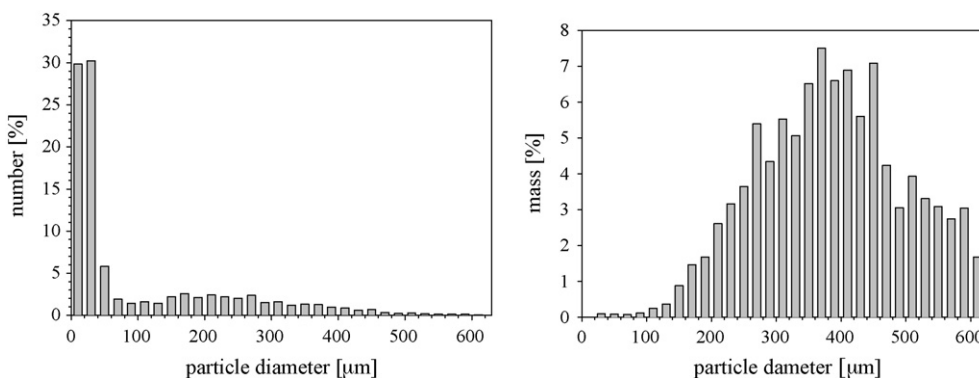


Fig. 3. Particle size distribution of DADNE recrystallized from water/NMP 75/25 mixture.

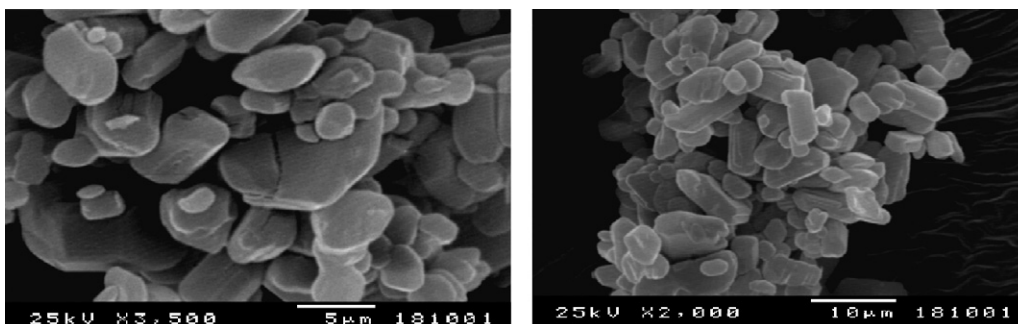


Fig. 4. SEM images of DADNE crystals obtained by pouring the DADNE solution in NMP into water at an ambient temperature.

Particle size distribution of the fine DADNE is shown in Fig. 5. The fraction of particles with diameters below $5\ \mu\text{m}$ dominates (ca. 75%), but the mass fraction is distributed in a quite different manner—particles with diameters within a range of $5\text{--}11\ \mu\text{m}$ are in the majority.

DADNE recrystallized in water/NMP mixture (Figs. 2 and 3) could be pressed in the form of cylindrical pellets with a diameter to length ratio of one up to the density of $1780\ \text{kg/m}^3$. But the density of product obtained from NMP solution (Figs. 4 and 5) was about $1610\ \text{kg/m}^3$. In this case the pellets swelled up after removing them from mould. So that, the product of particle distribution shown in Fig. 3 was the main object of our investigations.

3. Sensitivity to mechanical stimuli

The friction sensitivity of our DADNE was measured by applying a Julius-Peters machine. No friction sensitivity was observed up to loading of $353\ \text{N}$ for both the DADNE sorts (coarse and fine particles DADNE).

The impact sensitivity of coarse-size DADNE was determined by the use of testing apparatus with $5\ \text{kg}$ hammer. The highest drop height was determined at which no reaction was observed (h_0). Ten trials were conducted at consecutive height that was changed with a step of $1\ \text{cm}$. The value of $23\ \text{cm}$ was found as h_0 which corresponds with the impact energy of $11.3\ \text{J}$. For comparison, the value of $6\ \text{cm}$ ($2.9\ \text{J}$) was determined for crystalline RDX under the same conditions. In Ref. [4], the impact energy of $15.5\ \text{J}$ and $12.4\ \text{J}$ was determined for FOX-7 (DADNE) crystals with sizes of $250\text{--}355\ \mu\text{m}$ and those with

sizes less than $70\ \mu\text{m}$, respectively. A $2\ \text{kg}$ drop-weight apparatus was applied in Ref. [4].

The impact sensitivity can also be expressed as the drop height at which 50% initiations occurred (h_{50}). To determine h_{50} for our DADNE the Bruceton procedure was applied [12]. In this procedure the stimulus level is adjusted from one test to the next test (an up-and-down method) in order to group tests as closely as possible around the height at which 50% of specimens explode. The total number of tests performed was 30. The medium height $h_{50} = 35.2\ \text{cm}$ was obtained. This height corresponds with the impact energy of $17.3\ \text{J}$. For comparison, the value of $9.6\ \text{J}$ was determined for crystalline HMX (1,3,5,7-tetranitro-1,3,5,7-tetrazocane). The up-and-down method was also applied in Refs. [3,5] to measure the 50% probability level for DADNE using a 2-kg drop-weight apparatus. The impact energy of $24.7\ \text{J}$ and $31.2\ \text{J}$ was determined for crystalline DADNE and recrystallized one, respectively.

The shock sensitivity of DADNE was determined by using a gap test. The charge configuration used in the experiments is shown in Fig. 6. Pressed DADNE was placed inside a cooper tube of 25-mm inner diameter and 2.5-mm wall thickness. The length of the charge was $100\ \text{mm}$. A booster made of phlegmatised RDX (50-mm diameter, 50-mm height) served as a shock wave generator. From shot to shot, the length of an epoxy-40 vol.% quartz attenuator ($100\ \text{mm}$ in diameter) was changed with 1-mm step. The highest and the lowest gap values were appointed for which the complete detonation and failure of explosion process were observed. The complete detonation of explosive charge was indicated by a clean hole cut in the steel witness plate.

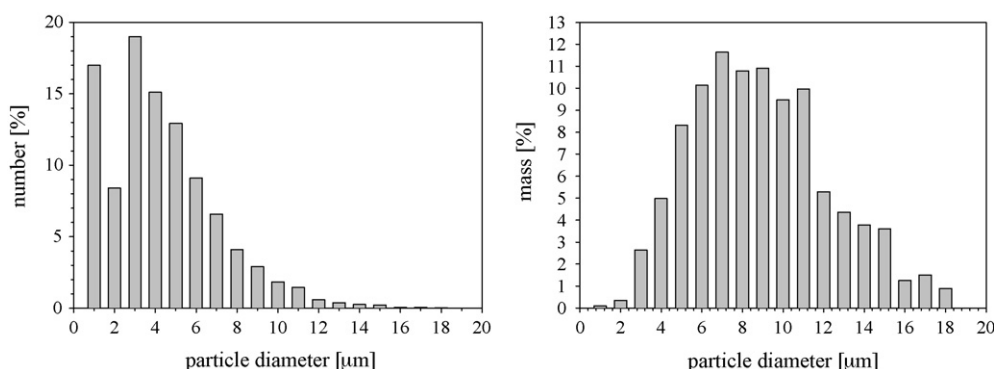


Fig. 5. Particle size distribution of DADNE obtained by pouring the DADNE solution in NMP into water at an ambient temperature.

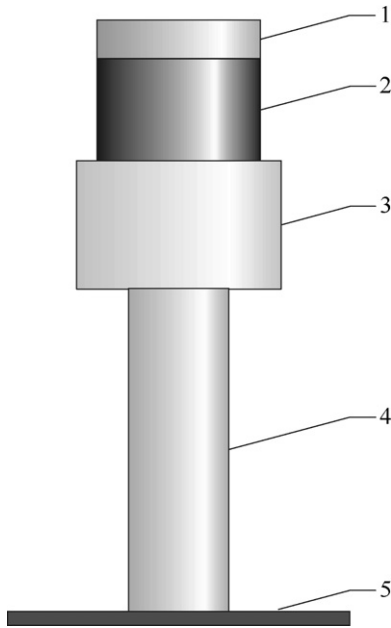


Fig. 6. Gap test configuration: 1—detonator and holder, 2—booster, 3—epoxy gap, 4—DADNE charge in cooper tube, 5—witness steel plate.

The shock sensitivity of DADNE as the usual gap results in the form “detonation–no detonation” is presented in Fig. 7. For comparison, the results obtained for TNT (pressed charges at a density of 1.63 g/cm^3) are also shown. The sensitivity of the fine-grain DADNE is lower than that of TNT, but the coarse-grain DADNE is much more sensitive than TNT.

4. Detonation characteristics

4.1. Detonation velocity and calorimetric heat of detonation

In all tests described hereafter, DADNE was pressed at a density of 1780 kg/m^3 in the form of cylindrical pellets with a diameter to length ratio of one. The detonation velocity of crystalline DADNE was determined by the method of short-circuit

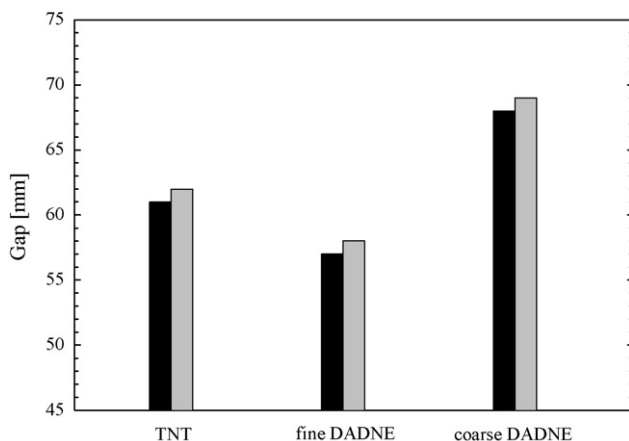


Fig. 7. Thickness of epoxy gap for complete and incomplete detonation in TNT and DADNE.

sensors. In a charge of 20 mm diameter, there were three distances at which time intervals were measured. The detonation velocity was obtained as a ratio between the distance travelled and the corresponding time interval. Measured detonation velocity was $8325 \pm 80 \text{ m/s}$. A theoretical detonation velocity was also estimated by using the thermochemical code CHEETAH [13] with two sets of the BKW parameters (BKWC and BKWS). The thermodynamical data for DADNE were taken from Ref. [3]. Calculated detonation velocity was 8453 m/s and 8499 m/s for BKWC and BKWS sets, respectively.

The detonation heat of DADNE was measured in the calorimetric system described in Ref. [14]. The calorimetric bomb of a volume of 5.6 dm^3 was filled with argon at a pressure of 2 MPa. Detonation of charges of 20-g weight was initiated by an electrical fuse. The average heat of detonation from three tests was $4860 \pm 60 \text{ J/g}$. For comparison, the total detonation energy calculated by CHEETAH code is 4774 J/g for BKWC set or 4884 J/g for BKWS set.

4.2. Detonation pressure

To determine the detonation pressure of DADNE, a variant of the aquarium test was applied [15]. In this method, profiles of an oblique shock wave propagating in a cylindrical layer of water during detonation of a cylindrical charge of an explosive tested is recorded with a X-ray set. The experimental profiles are then compared with results of numerical modeling of the expansion process which are in a form of relation between the position of the front of oblique shock wave in water and the exponent of isentrope (γ) of detonation products. The value of γ corresponding to the solution that overlaps the experimental profile is accepted as the exponent sought. The detonation pressure is calculated according to the following equation:

$$p_{CJ} = \frac{\rho_0 D^2}{\gamma + 1} \quad (1)$$

where D , p_{CJ} denote the detonation velocity and pressure, respectively, and ρ_0 is a density of the explosive tested.

The results of the water test are given in Table 1. The exponent of isentrope of detonation products was determined by comparison of measured and calculated positions of the shock wave front in a plane section located at a distance of one charge radius from the front of detonation wave. The detonation pressure was calculated from Eq. (1). Table 1 also contains some theoretical values of the parameters (with an index “t”) calculated with the thermochemical code CHEETAH (BKWC).

The detonation velocity and pressure of the tested DADNE are slightly higher than that of RDX phlegmatised by 6% wax: $D = 8270 \text{ m/s}$ and $p_{CJ} = 26.3 \text{ GPa}$ for the density $\rho_0 = 1638 \text{ kg/m}^3$ [15].

Table 1
Experimental and calculated detonation properties of DADNE

Test no.	D (m/s)	γ	p_{CJ}	D_t	γ_t	$p_{CJ,t}$
1	8405	3.31	29.2	8453	3.335	29.34
2	8375	3.39	28.4			

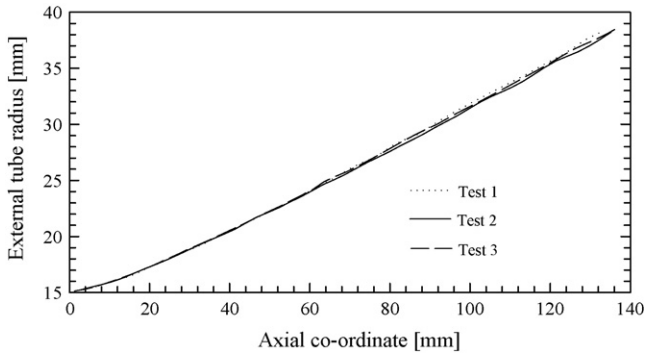


Fig. 8. Radial displacement of the external surface of the tube versus an axial co-ordinate.

4.3. Acceleration abilities

The cylinder test results were the basis for determination of acceleration abilities of detonation products of DADNE. The process of acceleration of copper tube by detonation products was recorded with the impulse X-ray apparatus. The tube was 300 mm long with internal diameter of 25 mm and wall thickness of 2.5 mm. Three tests were performed. Profiles of the tubes are given in Fig. 8.

To determine the radial velocity of the copper tube, the data obtained from the cylinder test were recalculated using the method proposed in Ref. [16]. Fig. 9 shows the tube velocity as a function of the relative volume of detonation products. For comparison, the velocity of copper tube for three volumes of expanding tube, estimated by CHEETAH, are also shown in Fig. 9.

The acceleration ability of explosive can be described by so-called Gurney energy, which is defined as a sum of kinetic energies of driven tube and detonation products related to unit mass of explosive. For cylindrical envelopes, the Gurney energy

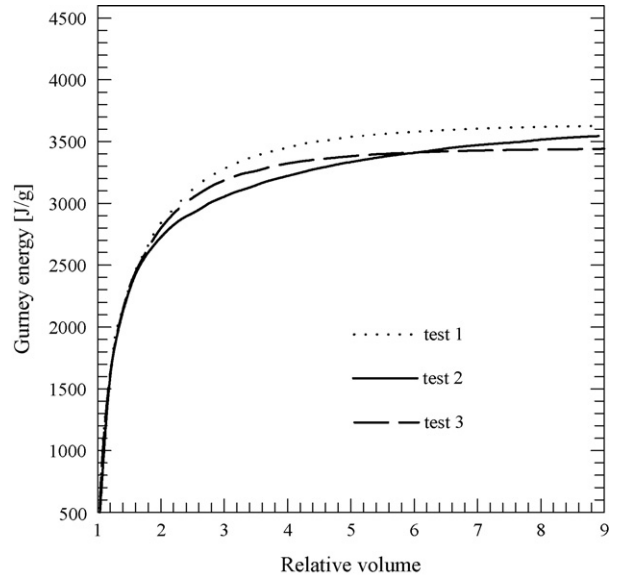


Fig. 10. Dependence of the Gurney energy on the relative volume of detonation products.

is expressed by the following relation [17]

$$E_G = \left(\mu + \frac{1}{2} \right) \frac{u_L^2}{2}, \tag{2}$$

where μ denotes the ratio of tube mass to explosive mass, u_L is the tube velocity. The calculated dependence of Gurney energy on the relative volume of detonation products is shown in Fig. 10.

The characteristics of DADNE obtained from the cylinder test results are summarised in Table 2. The Gurney energy and velocity ($u_G = \sqrt{2E_G}$) were determined for the relative volume of nine. For this volume the driven copper tube is still ductile and is not ruptured yet. For comparison, the data determined for TNT and phlegmatised RDX are also presented in Table 1 [18]. DADNE acceleration abilities are as good as that of phlegmatised RDX.

4.4. Detonation energy

The results of cylinder test can also be used to estimate the detonation energy. In Ref. [16] it was shown that there was a correlation between the velocity of driven tube at given volume of the detonation products and the detonation energy of an

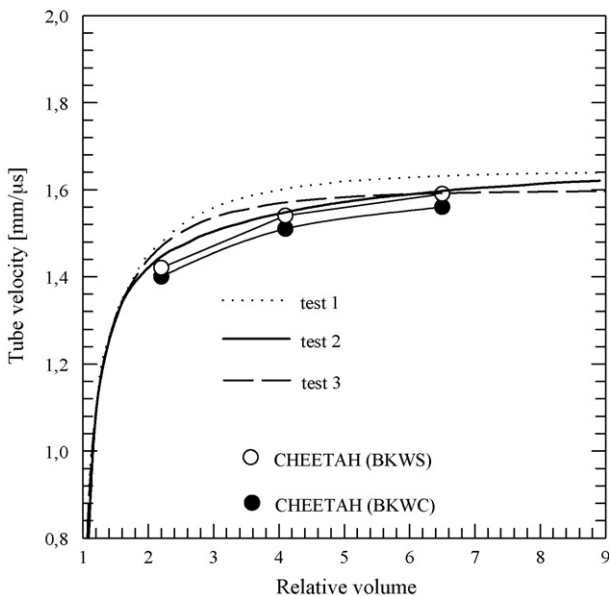


Fig. 9. Dependence of the tube velocity on the relative volume of detonation products.

Table 2
Characteristics of explosives determined from the cylinder test results

Explosive	Density (kg/m ³)	Detonation velocity (m/s)	Gurney energy (J/g)	Gurney velocity (m/s)
DADNE	1780	8325 ± 50	3540 ± 100	2660 ± 40
TNT	1590	6910	2795	2364
RDX/(CH ₂) _n	1650	8390	3734	2733
94/6				

explosive. The relation can be written down as follows

$$\frac{e_0}{e_0^s} = \frac{\mu + 1/2}{\mu^s + 1/2} \left(\frac{u_L}{u_L^s} \right)^2 \quad (3)$$

where e_0 and e_0^s are the detonation energy of a given explosive and a standard explosive, respectively u_L and u_L^s denote the tube velocity determined at given volume of detonation products of the explosives, μ and μ^s denote the ratio of tube mass to explosive mass.

In order to estimate the velocities u_L and u_L^s , the velocity of copper tube was determined from the results of cylinder test and the dependency of velocity square on reciprocal volume of detonation products was constructed. After that, the dependence was extrapolated to the relative volume of 10 and the velocities corresponding to that volume were found in Fig. 11.

Using phlegmatised RDX as a standard explosive for which the detonation energy ($e_0^s = 5344$ kJ/kg) was taken from Ref. [19], the detonation energy DADNE was calculated from Eq. (3). The values of 5120 kJ/kg (test 1), 4980 kJ/kg (test 2) and 4830 kJ/kg (test 3) were obtained. The average detonation energy of ca. 4980 kJ/kg is slightly higher than the calorimetric heat of detonation.

4.5. Effective exponent of isentrope

So-called effective exponent of isentrope is often used to calculate the detonation parameters of explosives or some characteristics of the detonation products. The value of the effective exponent is not determined from the parameters in the CJ point but on the basis of the real isentrope of the detonation products. The effective exponent of isentrope can be estimated from results of the cylinder test. In Ref. [20] the effective exponent is determined by comparison of the experimental profile of the copper tube with that obtained from numerical modeling of the expansion process. The detonation products, driving the tube,

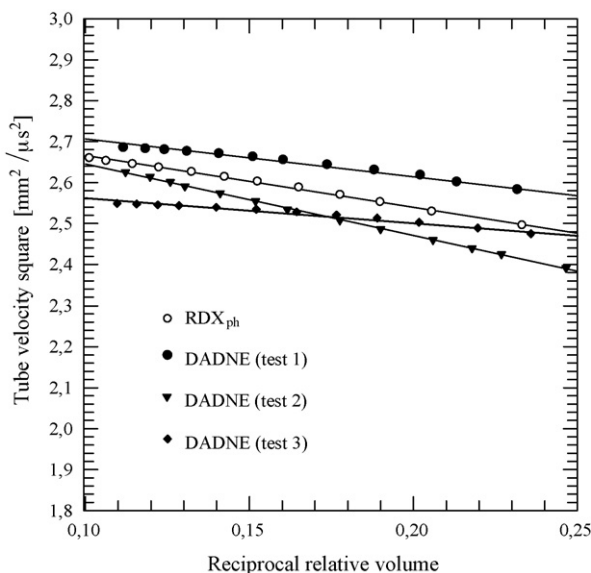


Fig. 11. Dependence of the square of velocity of the copper tube on reciprocal volume of the detonation products.

are described by the constant- γ equation of state. The algorithm used to determine the effective isentropic exponent is as follows.

The problem of driving the cylindrical liner is solved numerically for n values of the exponent γ_i ($i = 1, n$). For each γ_i a discrete dependence of the outer tube radius on the axial coordinate is derived. This dependence is interpolated by spline functions and the values of $r_{ej}(\gamma_i)$ at chosen points x_j ($j = 1, m$) are calculated. The effective exponent γ_{ef} is determined by minimising the function

$$f(\gamma) = \sum_{j=1}^m [r_{ej} - r_{ej}(\gamma)]^2, \quad (4)$$

where r_{ej} is the experimental dependence obtained from the cylinder test.

This method was applied in the present work to estimate the effective exponent of isentrope for DADNE detonation products. Exemplary experimental and calculated profiles of the copper tube driven by the detonation products of DADNE are presented in Fig. 12.

A satisfying conformity of experimental and theoretical profiles were achieved when the values of γ_{ef} were 3.19, 3.26 and 3.22 for tests no. 1, 2 and 3, respectively. So, the average value γ_{ef} for the DADNE detonation products is 3.22.

4.6. JWL isentrope

Jones, Wilkins and Lee proposed the equation of the isentrope for the detonation products of explosives in the following form

$$p = Ae^{-R_1 V} + Be^{-R_2 V} + CV^{(-1-\omega)} \quad (5)$$

where A , B , C , R_1 , R_2 and ω are constants for given explosive, $V = v/v_0$, $v = 1/\rho_0$. The basic method of determination of these coefficients uses the results of the cylinder test. Besides it, some connections between coefficients following from the conservation laws written for the CJ point are used in this method [21]. As a result, parameters A , B , and C are expressed as functions of R_1 , R_2 , ω and ρ_0 , D , p_{CJ} and the detonation energy E_0 . The initial density ρ_0 as well values of detonation velocity D and pressure p_{CJ} are established for DADNE experimentally in this work. The detonation energy E_0 was determined in our previous

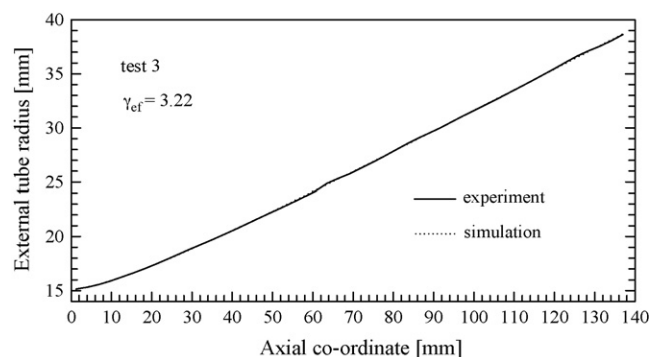


Fig. 12. Experimental and calculated profiles of a copper tube driven by detonation products of DADNE.

Table 3
JWL isentrope of DADNE

Explosive characteristics	JWL isentrope constants
$\rho_0 = 1780 \text{ kg/m}^3$	$A = 1414.339 \text{ GPa}$
$D = 8325 \text{ m/s}$	$B = 21.6637 \text{ GPa}$
$\gamma_{\text{CJ}} = 3.35$	$C = 1.23412 \text{ GPa}$
$p_{\text{CJ}} = 28.4 \text{ GPa}$	$R_1 = 5.54$
$E_0 = 8.9 \text{ GPa}$	$R_2 = 1.51$
	$\omega = 0.32$

work [9]. Thus, only the constants R_1 , R_2 and ω remain to be determined.

They are calculated by the method in which the experimental dependence of radial displacement of the outer tube wall on the axial co-ordinate is compared with that obtained from a numerical simulation [21,22]. The set of JWL constants is chosen for which the experimental and simulated displacements are sufficiently close to each other. The R_1 , R_2 and ω are obtained from comparison of the experimental and calculated radial position of the tube wall at chosen m values of the axial co-ordinate x_j . So, the values of these parameters are determined by minimising the function

$$f(R_1, R_2, \omega) = \sum_{j=1}^m [r_{ej} - r_{ej}(R_1, R_2, \omega)]^2 \quad (6)$$

where r_{ej} and $r_{ej}(R_1, R_2, \omega)$ are the experimental and calculated positions of external surface of the tube, respectively.

Using the model and the results of the cylinder test no. 3, the constants of the JWL isentrope of DADNE detonation products were calculated (Table 3).

Fig. 13 displays how different JWL and constant- γ isentropes for DADNE investigated are. For comparison, the JWL isentrope calculated by using the CHEETAH (BKWC) code is also shown.

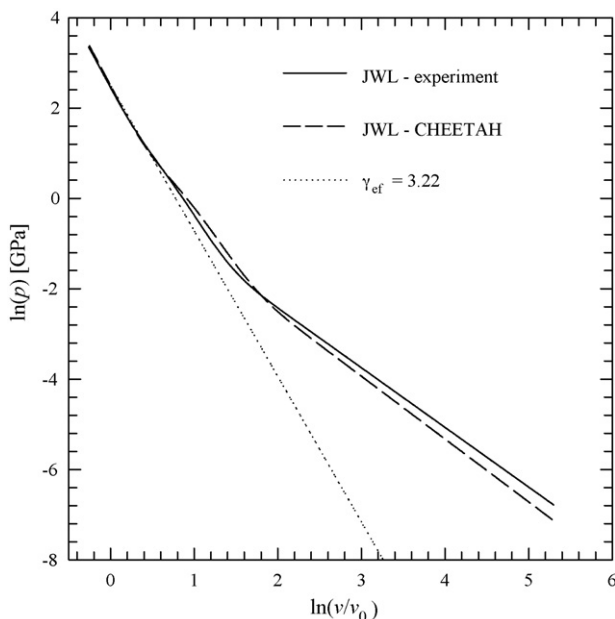


Fig. 13. The constant- γ and JWL isentropes for detonation products of DADNE.

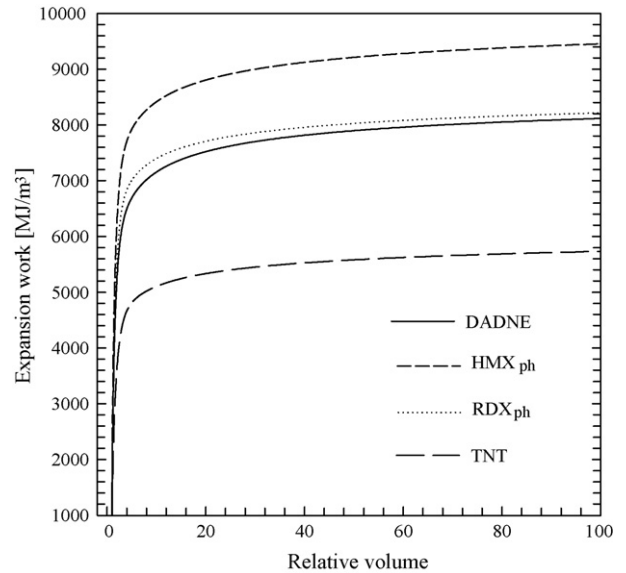


Fig. 14. Expansion work as a function of the relative volume of detonation products.

After determining the JWL isentrope, the expansion work of detonation products can be calculated from the following equation:

$$w(v) = -e_c + \int_{v_{\text{CJ}}}^v p_i dv \quad (7)$$

where $e_c = (p_{\text{CJ}} - p_0)(v_0 - v_{\text{CJ}})/2$ denotes the energy of explosive compressed at the front of detonation wave, p_i is the pressure on the isentrope.

Dependence of the expansion work on the relative volume of detonation products of DADNE is presented in Fig. 14. For comparison, similar curves for TNT, phlegmatized RDX and phlegmatized HMX taken from Ref. [23] are also shown. DADNE expansion work is comparable to that of phlegmatized RDX.

5. Summary

1. The cooling crystallization of DADNE from water, NMP and water/NMP was performed. Crystals of definitely better quality were obtained when the binary system ($\text{H}_2\text{O}/\text{NMP}$) was used for recrystallization. In this case coarse crystals are regular in shape, with smooth walls and sharp edges. An attempt to spheroidize the crystals, by stirring suspensions of the crystals in various solvents, was not successful.
2. In order to obtain a fine-grain product, DADNE was dissolved in NMP and poured into water at an ambient temperature. The fine-grain product of quite regular shapes was obtained.
3. The sensitivity of DADNE to impact was measured by using a 5 kg apparatus and friction sensitivity was determined by a Peters machine. No friction sensitivity was observed. DADNE is considerably less sensitive to impact than crystalline RDX and HMX.
4. The shock sensitivity of DADNE was determined by using a gap test. The sensitivity of the fine-grain DADNE is lower

than that of TNT, but the coarse-grain DADNE is much more sensitive than TNT.

5. The detonation velocity of DADNE pressed at 1780 kg/m³ is 8325 m/s for charges of a 25 mm diameter. The calorimetric heat of detonation determined in a 5.6 dm³ steel bomb filled with compressed argon is 4860 J/g. The pressure of detonation of DADNE is 28.4 GPa. Tested DADNE has the same detonation performance as phlegmatised RDX.
6. The Gurney energy determined for the relative volume of detonation products of nine is 3540 J/g. The acceleration ability of DADNE is comparable to that of phlegmatised RDX.
7. The detonation energy of DADNE estimated from the results of a cylinder test is 4980 J/g and it is slightly higher than the calorimetric heat of detonation.
8. The effective exponent of DADNE isentrope is 3.22. The set of constants of JWL EOS is as follows: $A = 1414.339$ GPa, $B = 21.6637$ GPa, $C = 1.23412$ GPa, $R_1 = 5.54$, $R_2 = 1.51$, $\omega = 0.32$, $E_0 = 8.9$ GPa.

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