



Investigation of an Aluminized Binder/AP Composite Propellant Containing FOX-7

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Abstract: This paper presents the results of thermodynamical calculations and investigations of the thermochemical and ballistic properties of aluminized composite solid propellants Binder/AP/Al containing FOX-7. The calculation was conducted by using ICT-Thermodynamic Code. The heat of combustion was determined in a stationary bomb calorimeter IKA C 4000. The breakdown temperatures were taken with the DTA 551 Ex measuring apparatus and the burning rate was measured in a subscale rocket motor utilizing the ESAM v. 3.3.0 system. It was revealed that the introduction of FOX -7 into the propellant composition causes a reduction of the energetic characteristics and the burning rate of the propellant.

Keywords: FOX-7, aluminized composite propellant, burning rate

Introduction

The use of insensitive energetic materials as a component of solid composite propellants arouses more and more interest. It is so because the insensitive materials drastically reduce propellant sensitivity to shock waves. The insensitive behavior is also important for a safe manufacturing process of solid composite propellants. The insensitive energetic material selected as a possible ingredient of solid rocket propellant was 1,1-diamine-2,2-dinitroethylene (FOX-7, DADNE) which is a material well-known and described in literature [1-15].

The paper presents the thermodynamic calculations and investigations of some aluminized Binder/AP composite propellants containing FOX-7 and

a comparison of their properties with those of a propellant not containing FOX-7. The studied compositions are shown in Table 1. The tested propellants were prepared by a classical slurry cast method [16, 17].

Table 1. Composition (% wt.) of the propellant formulations

Components		Propellants		
		P1	P2	P3
AP		79.00	64.00	50.00
Binder	PBAN/Epoxide	11.55	11.55	11.55
	DOA	4.45	4.45	4.45
Al		5.00	5.00	5.00
FOX-7		0.00	15.00	29.00

Thermodynamic Calculations

Thermodynamic calculations were performed to predict the flame temperature, the oxygen balance, the reaction products and the specific impulse for the tested Binder/AP/FOX-7/Al composite propellants. The calculations were performed using ICT-Thermodynamic Code. The code computes chemical equilibrium by solving the non-linear equations derived from the mass action and mass balance expressions [18, 19]. The calculations were performed for isobaric adiabatic combustion at 7.0 MPa, assuming an adiabatic expansion through a nozzle in one-dimensional flow at chemical equilibrium and an expansion ratio of 70:1. The results are presented in Table 2.

Table 2. Calculation results of properties of the propellant formulations

Properties	Propellants		
	P1	P2	P3
ρ , g·cm ⁻³	1.71	1.70	1.70
Specific impulse (I_s), Ns·kg ⁻¹	2432	2396	2343
Volume Specific impulse (I_v), Ns·dm ⁻³	4163	4082	3976
Chamber temperature (T_c), K	3053	2857	2645
Oxygen Balance (OB), %	-19.65	-28.00	-35.79

The composite propellants containing FOX-7 particles offer the advantages of low flame temperature and low molecular mass of combustion products, as well as reduced infrared emissions (the reduction of CO₂ and H₂O in the combustion products in chamber and nozzle) as shown in Figure 1. Moreover,

an increased content of FOX -7 in the propellant decreases the quantity of HCl in the combustion products.

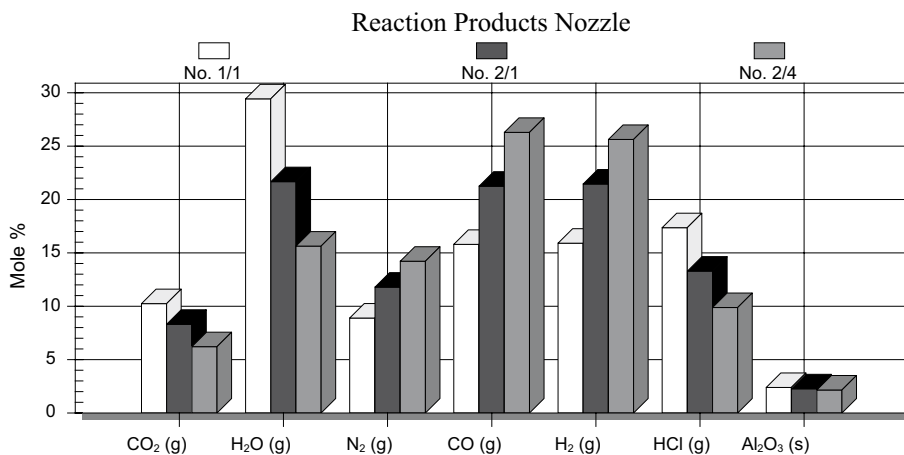


Figure 1. Graphical representation of the calculated reaction products of the tested propellants (No. 1/1 – P1, No. 2/1 – P2, No. 2/4 – P3).

Experimental

Granulometric analysis

Grain size distribution of the Al, AP and FOX-7 crystals was determined with the IPS-U (Infrared Particle Size) analyzer, version 8.12. The working principle of IPS analyzer consists in the measurement of the variations of the IR stream which is dispersed by the particles moving within the measurement zone.

Trimodal AP was used as oxidizer (small sized and large sized). The content of the small sized (fine-grained) AP in the propellant was 15-23.7%. Particle diameters of the fine-grained AP were below 50 μm . Particle diameters of the large sized (two fraction) AP were below 160 and 480 μm (Figure 2). Particle diameters of FOX-7 were below 560 μm . Aluminum powder (Al) particle diameters were below 60 μm (Figure 2).

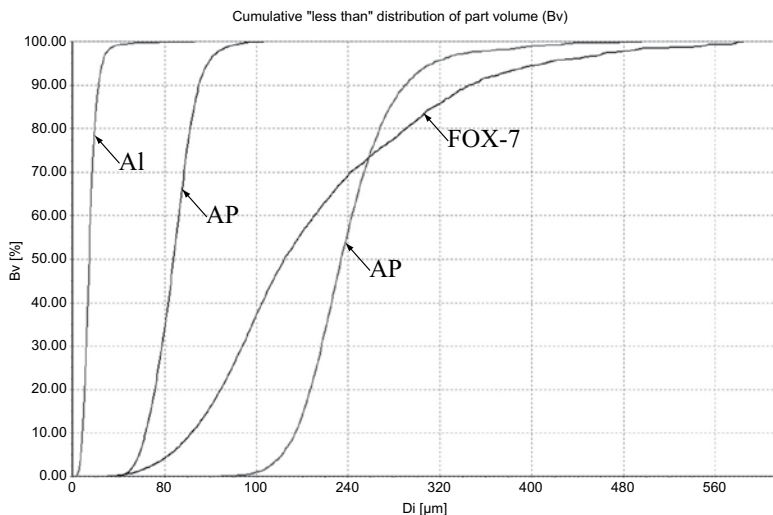


Figure 2. Cumulative distribution of particle volume: Al, AP (two fractions) and FOX-7.

The shape and size of the FOX-7 and AP particles observed under a microscope coupled with a still camera is shown in Figure 3.

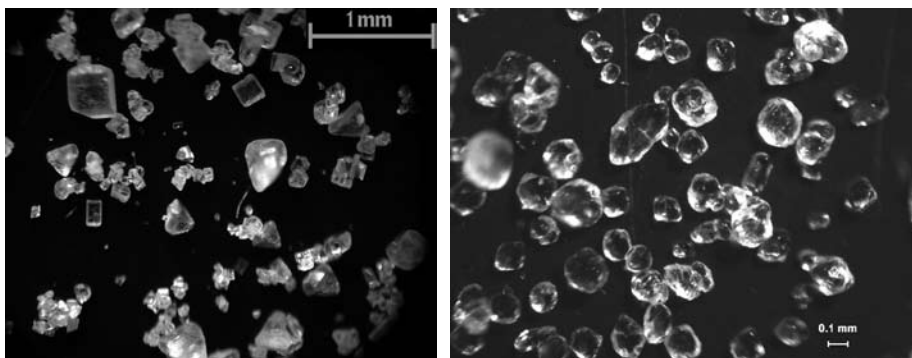


Figure 3. Microscopic pictures of the FOX-7 (left picture) and AP (right picture) particles.

Preparation of propellant samples

Propellant samples were prepared using a slurry cast technique. All solid ingredients were stored in water-jacketed ovens at 348 K to minimize the absorption of moisture from the atmosphere. Prior to incorporation into the

mixture, the PBAN pre-polymer was degassed overnight at 348 K in a vacuum drying oven.

The propellant was prepared in a vertical, 1.5-liter planetary action mixer at 348 K, under reduced pressure. After the mixing cycle, the propellant was cast under vacuum into a Teflon coated mould. The propellant slurry was then cured at 348 K for two days in a water-jacketed oven.

The cured propellant was removed from the mould and machined in the shape of cylinders of the following dimensions: height [h] = 50 mm, external diameter [D] = 37 mm, internal diameter [d] = 14, 16, 19 and 23 mm. The front surfaces of the cylinders were coated with a thin layer (about 1 mm) of Poxilina® (Figure 4).

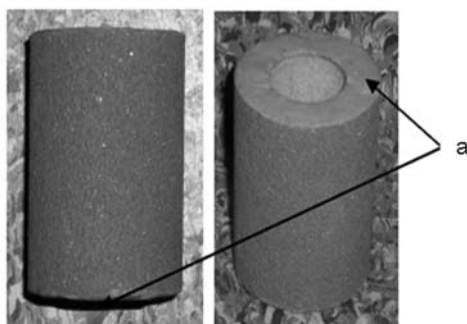


Figure 4. Photographs of example of propellant grains (a – inhibiting surfaces).

Thermal analysis

A differential thermal analysis was performed using a DTA 551 Ex apparatus manufactured by OZM Research. 90 mg of sample was heated at a rate of 15 K/min in the presence of static air. Analyses were performed in open test tubes. A thermocouple protected by a glass sheath was inserted directly into the sample. Data were evaluated using the MEAVY 2.0.0.4 software of DTA 551 Ex analyzer. The onset (T_{onset}) and the maximum (T_m) of the first exothermal effect were evaluated on thermograms. The onset of decomposition peak (T_{onset}) and the maximum peak (T_m) of research propellants are presented in Table 3.

Heat of combustion

The heat of combustion (Q) represents the caloric equivalent of the total combustion energy of a substance. Its value depends only on the composition of the material and not on any other properties of propellant, such as, for example,

loading density. Combustion experiments were conducted in a stationary bomb calorimeter with automatic temperature measurement under reduced pressure ensuring minimum oxygen atmosphere (3.3 kPa pressure). The adiabatic bomb IKA C 4000 calorimeter system was used to determine the heat of combustion. The calorimeter was calibrated by burning standard propellant to determine its effective energy equivalent. The results of the heat of combustion study of propellants are presented in Table 3.

Table 3. Results of propellant investigations

Properties	Propellant		
	P1	P2	P3
T_{onset} , K	515	528	522
T_m , K	528	530	524
Q , $\text{kJ}\cdot\text{kg}^{-1}$	5835	5130	4796

Burning Rate Measurements in Subscale Rocket Motors

Burning rates were determined using subscale rocket motor (SRM) – Figure 5. The distribution of the sample grains of composite solid propellants inside the SRM chamber is shown in Figure 6.

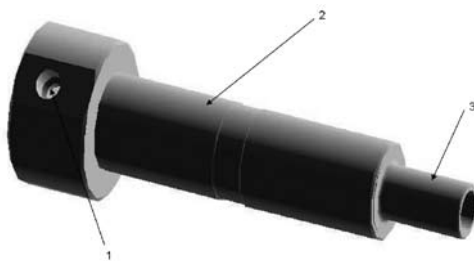


Figure 5. Subscale rocket motor: 1 – pressure tap, 2 – chamber, 3 – nozzle.

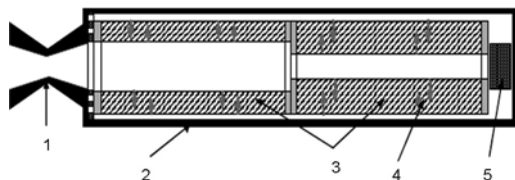


Figure 6. Distribution of the propellant grains under investigation inside the SRM chamber: 1 – nozzle, 2 – chamber, 3 – propellant grains, 4 – direction of burning, 5 – igniter.

A strain gauge was applied to measure pressure during the combustion of two grain loads of a specific propellant arranged as in Figure 6. The pressure over time $p = f(t)$ was registered and the measurement results were processed using the ESAM v. 3.3.0 system.

Based on the measurement data obtained, the average burning rate and the average pressure were determined, since the rate depends on the pressure. This is consistent with the well-known equation of the power dependence of burning rate on pressure, called St. Robert's, St. Venant's or Vieille's law, $r = ap^n$ (r – propellant burning rate, a – coefficient of pressure, n – pressure exponent).

The average burning rate (r_{aver}) was defined as:

$$r_{aver} = \frac{w_b}{t_b} \quad (1)$$

where:

$w_b = (D-d)/4$ – web thickness

$t_b = t_2 - t_1$ – burning time

t_1, t_2 – the start and end burning times for the given range.

The average pressure for the burning time $[t_b]$ was determined by this formula:

$$p_{aver} = \frac{\int_{t_1}^{t_2} p dt}{t_b} \quad (2)$$

where

p_{aver} – the average combustion pressure determined over t_b .

The registered courses of the changes of pressure over time ($p = f(t)$) as well as the numerical integrations of those courses, the so-called pressure impulses, are shown in Figures 7-9.

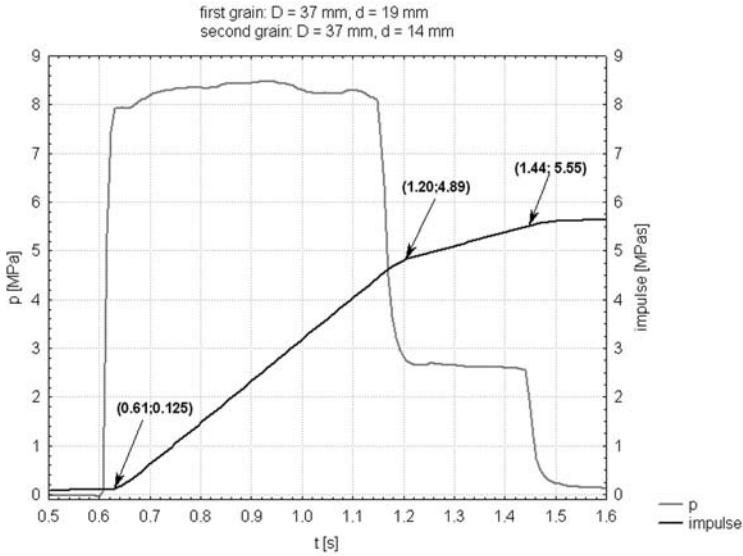


Figure 7. Pressure - time curve from SRM test and integral of this curve for propellant P1.

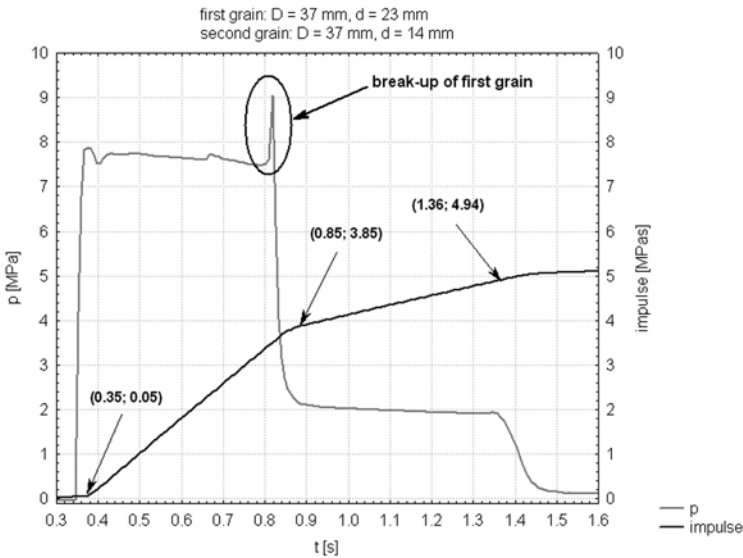


Figure 8. Pressure - time curve from SRM test and integral of this curve for propellant P2.

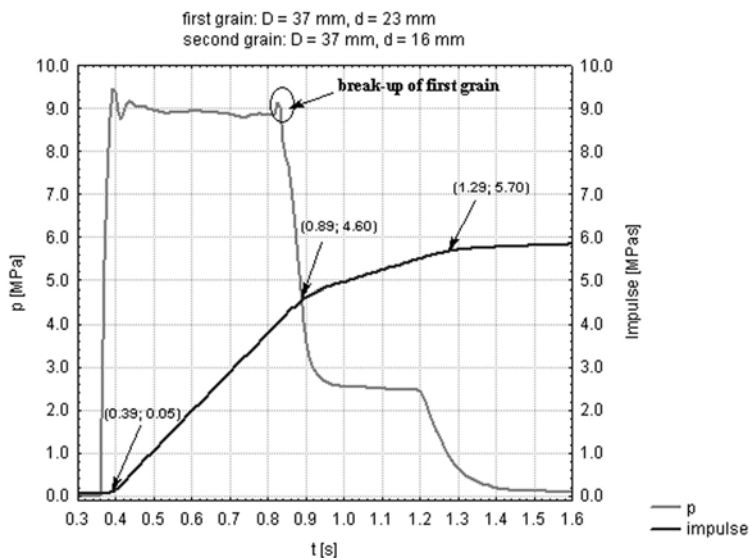


Figure 9. Pressure - time curve from SRM test and integral of this curve for propellant P3.

The values (w_b , t_b , p_{aver} , r_{aver}) calculated on the basis of the registered courses of pressure vs. time during the burning of the studied samples in SRM (Figures 7-9) are presented in Table 4.

Table 4. Results of investigations

Properties		Propellant		
		P1	P2	P3
First range	w_b , mm	4.50	3.50	3.50
	t_b , s	0.59	0.50	0.53
	p_{aver} , MPa	8.08	7.60	9.10
	r_{aver} , mm s ⁻¹	7.63	7.00	7.00
Second range	w_b^* , mm	1.25	2.25	1.75
	t_b , s	0.24	0.51	0.40
	p_{aver} , MPa	2.75	2.14	2.75
	r_{aver} , mm s ⁻¹	5.20	4.41	4.38

$$* w_b = (w_b)_{second\ grain} - (w_b)_{first\ grain}$$

The t_b and p_{aver} parameters were calculated for the points marked with arrows – Figures 7-9.

Conclusions

The most important conclusions from this paper are as follows:

1. It is concluded from the thermodynamic calculations that the introduction of FOX-7 instead of AP causes an obvious decrease of the propellant burning temperature.
2. An increase of FOX-7 content in propellant worsens the oxygen balance and the specific impulse of propellant.
3. Introduction of FOX-7 instead of AP causes a decrease of the energetic properties (Q) of propellant by about 17.8% (propellant P1 and P3).
4. The burning rate of the propellant decreases with the increasing weight percentage of FOX-7 in propellant.

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