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Bicyclo-HMX as an energetic additive for composite propellants

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This paper studies the ballistic parameters of composite propellants containing bicyclo-HMX (BCHMX) as an energetic additive. The results were compared with the same composite propellant containing HMX. Based on the results of Stojan vessel measurement, it was found that the burning rates of BCHMX-containing propellants are higher compared to most concentrations of HMX in propellants. Increasing the concentration of HMX in the propellant often leads to a decrease in burning rate, whereas this trend was not observed for higher concentrations of BCHMX. The pressure exponent of all tested propellants is less than 1, while mostly lower values were observed for BCHMX propellant samples. Propellants containing BCHMX are also distinguished by easy ignitability. Using REAL software, it was found that propellants containing BCHMX achieve high specific impulses. When the BCHMX content is over 30 %, the propellant exceeds the specific impulse values of the propellant without added BCHMX and of all tested propellants containing HMX.

Nomenclature

dp = pressure step

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dt = time step

e_0 = burning thickness of propellant grain

E_{50} = impact sensitivity level, 50% probability initiation energy

F_{50} = friction sensitivity level, 50% probability initiation force

I_{sp} = specific impulse

OB = oxygen balance

p = pressure

$p_{\max}$ = maximal pressure

p_z = ignition pressure

T_f = flame temperature

u = burning rate

I.Introduction

Composite propellants are composed of a crystalline oxidizer (most often ammonium perchlorate, AP) incorporated into a hydrocarbon-based polymeric matrix serving as fuel and binder. These high-performance compositions usually surpass widely used double base propellants. To increase the energetic content i.e. specific impulse (I_{sp}) of a composite propellant, powdered aluminum is added to this mixture, which increases the I_{sp} by increasing the flame temperature. Aluminum powder also improves combustion stability and increases density of the propellant resulting in improved volumetric performance [1-6].

The combustion of AP containing composite propellant produces a large amount of hydrogen chloride forming a detectable trail. This unwanted trail can be suppressed by reducing the ammonium perchlorate content in the propellant, which can be partially replaced by energetic materials, particularly nitramines. Hexogen (1,3,5-trinitro-1,3,5-triazinane, RDX) and octogen (1,3,5,7-tetranitro-1,3,5,7-tetrazocane, HMX) are most commonly used for this purpose [1-2].

Bicyclo-HMX (cis-1,3,4,6-tetranitrooctahydroimidazo-[4,5-d]imidazole, BCHMX) is a relatively new energetic nitramine that was first synthesized in the former USSR in the 1980s and its preparation and application were confidential until 2010. Compared to RDX and HMX, BCHMX has a high enthalpy of formation (Table 1) as a result of a rigid molecular structure and deformed valence angles. High heat of formation, together with high crystal density, is also reflected in BCHMX's high detonation parameters which are the reasons why BCHMX has been suggested for various types of plastic bonded explosives [7], melt cast compositions with TNT [8-9] and 2,4-dinitroanisole [10] or as a secondary charge in detonators [11]. A comparison of BCHMX's properties compared to those of HMX and RDX is shown in Table 1.

The combustion characteristics of BCHMX have been investigated by Sinditskii, Egorshev and Berezin [12]: BCHMX burns faster than RDX and HMX and almost as fast as CL-20 (2,4,6,8,10,12-hexanitro-2,4,6,8,10,12-hexaazatetracyclo[5.5.0.0^{3,11}.0^{5,9}]dodecane) in pressure range 0.1-36 MPa. Unlike HMX, BCHMX burns in the sub-atmospheric pressure region. BCHMX also has a higher adiabatic flame temperature (3530 K vs. 3318 K for RDX and 3295 K for HMX at 10 MPa), a property which is closer to that of CL-20 (3636 K at 10 MPa) [12].

Many of the above mentioned parameters of BCHMX (especially high heat of formation and density, high burning rate and stable burning at sub-atmospheric pressures) make it very promising for use in propellants. Meanwhile, the scientific literature only reports the use of BCHMX as a component of single base gun powders. Heat of explosion and vivacity of the powder increase with increasing BCHMX content (5-20%) which has a positive impact on bullet velocity [13]. Sensitivity data for composite propellants containing BCHMX have been reported recently [14]; a study of thermal decomposition kinetics for BCHMX/HTPB/MAPO/DOA composition have been published as well [15-16]. Based on thermochemical calculations and the high energy content of BCHMX, we have adopted the hypothesis that it could be a suitable energetic additive for composite propellants that should surpass the currently used RDX or HMX. We focused this study on the burning characteristics of composite propellants containing BCHMX and compared them with composite propellants based on commonly used HMX.

Table 1 Comparison of nitramine properties [17].

Properties	BCHMX	beta-HMX	RDX
Melting point [°C]	268 dec.	275 dec.	204
Theoretical maximum density [g/cm^3]	1.86	1.96	1.82
Enthalpy of formation [kJ/kg]	701^a	297^b	320^b

Oxygen balance [%][c]	-16.3	-21.6	-21.6
Calculated detonation velocity [m/s] / density [g/cm^3] [c]	9050 / 1.86	9169 / 1.91	8855 / 1.82
Max. experimentally determined detonation velocity [m/s] / density [g/cm^3]	8650 / 1.79	9100 / 1.90	8750 / 1.76
Calculated detonation pressure [GPa]	37	39	35
Ignition temperature [°C]	214-224	275	204 dec.
Impact sensitivity [J]	2.98	6.37	5.58
Friction sensitivity [N]	88	95	152
Electric spark sensitivity [mJ]	148.7	236.4	216.4

[a] Value taken from lit. [12]; [b] value taken from lit. [18]; [c] values calculated using the EXPLO5 v6.06.02 thermochemical code [19].

II.Experimental

Two series of propellant samples were prepared. A standard propellant sample, denominated as Control, was used to assess the burning rate of the basic propellant composition without any nitramine. Nitramine-modified propellants were then prepared, containing 10, 15, 20, 25 and 30% of either HMX or BCHMX. The compositions of the composite propellants in the study are summarized in Table 2. The main variable was the AP to nitramine (BCHMX or HMX) ratio. Nitramines were always added to the propellant at the expense of the original amount of AP. The amount of the other components remained constant.

Table 2 Propellant composition by wt. %.

AP	BCHMX or HMX	Al powder	HTPB	DOS	MAPO	IPDI	Ferrocene
59	10	8	16.5	4.18	0.55	0.77	1
54	15						
49	20						
44	25						
39	30						
69[a]	0						

[a] Control sample

A. Materials and method of preparation

The ammonium perchlorate (Bochemie Plc. Bohumín Czech Republic) consisted of a tetra-modal mixture of four particle size fractions: 25% 100-200 μm, 49% 200-300 μm, 11.5% 300-400 μm and 14.5% of finely ground AP (20-50 μm). Hydroxyl-terminated poly-butadiene (HTPB) (KAUČUK, Plc. Kralupy nad Vltavou, Czech Republic) type R45 was used as the binder, tris(1-(2-methyl)aziridinyl) phosphine oxide (MAPO) (Hangzhou Yuhao Chemical

Technology Co., Ltd. China) as a bonding agent, bis(2-ethylhexyl)-sebacate (DOS) (Alfa Aesar) as a plasticizer and isophorone diisocyanate (IPDI) (Aldrich) as curing agent. Powdered aluminum with an average particle size of 10 µm (Albo Schlenk Ltd.) was used. Ferrocene – (bis(η^5-cyclopentadienyl)iron) – (Sigma-Aldrich) was used as a burn rate modifier. Propellant samples in amounts of 150 g were prepared in a custom-made 400 cm^3 double-walled glass reactor heated to 60 °C. All solid components were gradually added to the HTPB in small increments and, after each addition, the mixture was stirred under vacuum. In the case of the nitramine-modified propellants, nitramines were added to HTPB in the early stage to ensure sufficient coating of sensitive nitramines by HTPB and thus increasing safety during subsequent mixing. After mixing of all components, the slurry was additionally mixed under vacuum and the resulting propellant was cast into 3D printed PET molds. Propellant samples were cured at a temperature of 60°C for two weeks.

B. Setup for Stojan Vessel measurements

The dependence of the burning rate on pressure was measured using an SV2 (Stojan Vessel) closed vessel (OZM Research). ABSW software (OZM Research) was used to evaluate the measurement results, this evaluation is based on Eq. (1), where e_0 is unit burning thickness of solid propellant grain, p_{max} is maximal pressure in closed bomb, p_z is ignition pressure, dp and dt represent the pressure change as a function of time [20].

$$u(p) = [e_0 / (p_{max} - p_z)] \, . \, (dp / dt) \qquad (1)$$

The uncertainty of the pressure measurement is within 0.5% as given by the manufacturer. The weight and dimensions of used samples were in the range of 55-65 g and 50 mm diameter by 19-22 mm height; exact values were assigned to samples for each measuring run. The thickness measurement deviation is no more than 0.1 mm. The sample was left in the 3D printed PET casting mold which was wrapped in three layers of textile tape as insulation. The uneven surface of the cured propellant sample was cut away to achieve a flat surface. The propellant sample was ignited using 13 ±0.3 g of black powder attached to an iron grid sample holder. A detailed picture of the sample insulation and setup of iron grid holder with the propellant sample is shown in Figure 1. Minimum of two shots were performed for the measurement of each composition. The standard deviation was determined by interleaving the data obtained from two measurements using a power function. The standard deviations of the regression curves from the measured data were in the 0,17-0,86 mm/s range. The pressure exponent was also determined from the regression function used. The burning rate and pressure exponent were evaluated over a pressure range of 3-14 MPa.

Fig. 1 Detailed sample of propellant (left) sample on iron grid holder with attached bag of black powder (right).

C. REAL software calculations

Prediction of specific impulse, flame temperature and calculation of oxygen balance was conducted using software REAL [21] – a thermodynamics code which is used for computer simulation of chemical equilibrium in complex chemically-reacting systems. Enthalpies of formation and concentrations of each component were used as input data. Standard pressures 7 MPa (chamber pressure) to 0.1 MPa (expansion out of nozzle) were used for calculation. For all I_{sp} calculations, the Virial equation of state was used.

D. Sensitivity to impact and friction

Sensitivity to impact was determined using BAM Fall Hammer BFH-12 (OZM Research), friction tests were performed on BAM Friction Apparatus FSA-12 (OZM Research). FEST (Fast and Efficient Sensitivity Testing) method was used to determine the 50% initiation probability levels E_{50} and F_{50}. More detail in [14].

III. Results and discussion

A. Theoretical calculations using REAL software

The specific impulse (I_{sp}) depends on a number of parameters, including the chemical composition of the propellant. In general, if ammonium perchlorate is replaced by nitramine in the composite propellant, the specific impulse decreases [1]. Replacing AP in composite propellants may be done to reduce the amount of hydrogen chloride in the combustion products. At first, using REAL thermochemical code, we calculated I_{sp} (for the considered chamber pressure 7 MPa) for a typical high performance composition AP/HTPB/Al 68/14/18 and 68/12/20 [1, 22] Then the dependence of the specific impulse on the content of BCHMX and HMX in propellant was determined (Figure 2). As the nitramine content in the propellant increases, the specific impulse increases slightly at first and significantly decreases after reaching a maximum value of I_{sp}. In case of BCHMX propellants with 18% aluminum content, the I_{sp} is increased to maximum of 2605 Ns/kg (at 10% BCHMX content) from original 2598 Ns/kg (propellant without nitramines). The same trend was observed for HMX when maximum I_{sp} = 2603 Ns/kg was calculated for propellant with 10% of HMX.

For series of propellants containing 20% aluminum, effect of both nitramines is the same and resulting maximum I_{sp} of 2619 Ns/kg is calculated for propellant with 15% of either BCHMX or HMX. Similar trend of declining values of I_{sp} can be observed as previously stated. During the calculation, increasing BCHMX content in the propellant slightly enhances I_{sp} compared to HMX, but the difference is basically negligible as can be seen in Figure 2.

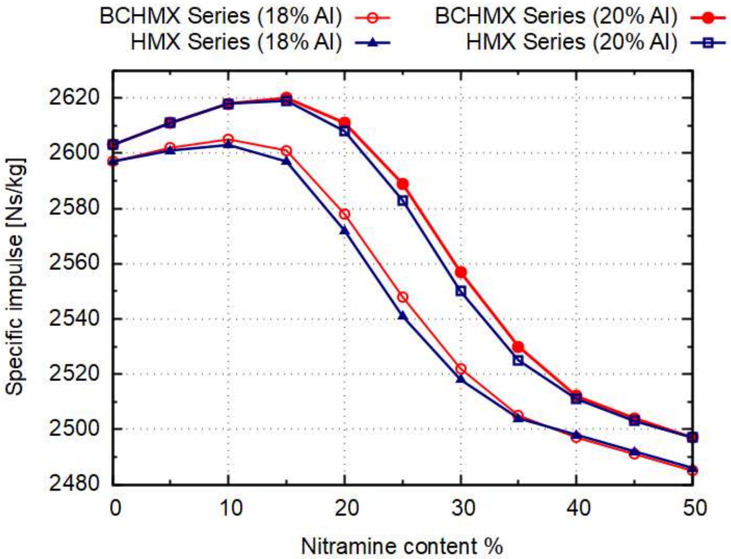

Fig. 2 Calculations of specific impulse for high energy AP/Al/HTPB (containing 18 or 20% Al) propellants with respect to BCHMX increasing content

The specific impulse was also calculated at 7 MPa for compositions described in Table 2; calculations were conducted for compositions containing up to 50% of BCHMX, where ammonium perchlorate is replaced by nitramine.

These compositions contain 8% aluminum, thus lower specific impulse is expected. However, the lower content of aluminum and AP is beneficial in terms of the reduction of primary and secondary smoke [2].

Calculated values of specific impulse as a function of nitramine content in the propellant are shown in Figure 3. The specific impulse for the Control propellant is 2309 Ns/kg. The addition of smaller amounts of BCHMX into the propellant results in an expected slight decrease in the specific impulse. The lowest value was calculated for the mixture with 20% BCHMX, at which point the trend is overturned and further addition of nitramine results in an increase of specific impulse until at 30% BCHMX content the composition has a specific impulse at the level of the composition without nitramine (Control propellant); any further addition of nitramine increases the specific impulse practically linearly. The composition with 50% BCHMX shows the specific impulse 2347 Ns/kg. The specific impulse of the composition with 50% BCHMX compared to the Control is 38 Ns/kg higher, corresponding to an increase of 1.65%. This increase of I_{sp} is significant since, even a 1% increase in I_{sp} can increase the range of long-range ballistic missiles by more than 7% [23].

The behavior of the compositions with HMX is similar, at first, with the increase in HMX content there is a decrease in specific impulse (HMX content up to 20%) and then an increase in I_{sp}. However, this composition does not achieve the same specific impulse as the control composition until HMX content exceeds 45%, and even at 50% HMX the increase in the specific impulse is only negligible.

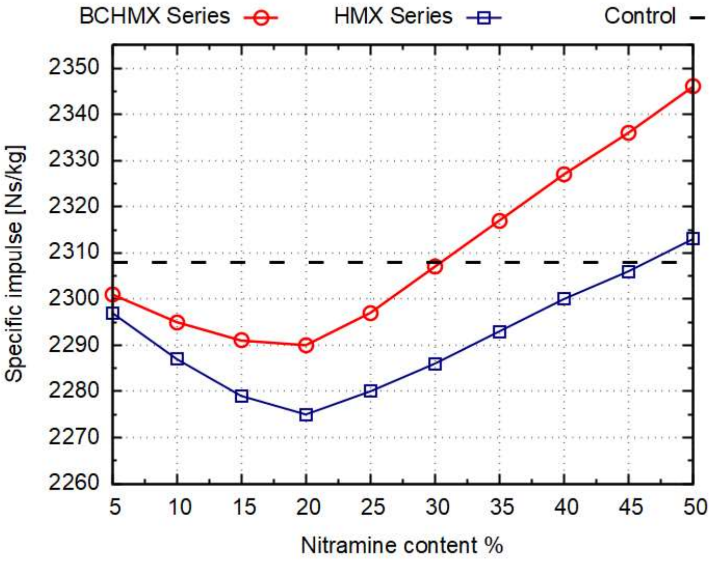

Fig. 3 Calculation of specific impulse for composition given by table 2

Despite lowered content of AP and aluminum in basic composition (control), the specific impulse can be increased when BCHMX is incorporated. Furthermore, calculated flame temperatures for considered nitramines are higher compared to Control propellant as can be seen at Table 3. Addition of BCHMX to these propellants could partially compensate the energy loss due to mentioned lowering of AP and aluminum content.

Table 3 Resulting calculations of oxygen balance and flame temperature

AP %	**BCHMX or HMX %**	**OB % BCHMX**	**OB % HMX**	**T_f BCHMX [K]**	**T_f HMX [K]**
64	5	-55.8	-56.1	2327	2327
59	10	-58.3	-58.8	2326	2321
54	15	-60.8	-61.6	2315	2270
49	20	-63.8	-64.4	2262	2206
44	25	-65.8	-67.2	2200	2149
39	30	-68.4	-69.9	2211	2167
34	35	-70.9	-72.7	2236	2185
29	40	-73.4	-75.5	2261	2204
24	45	-75.9	-78.3	2287	2223
19	50	-78.4	-81.1	2315	2243

*Control propellant: OB: -53,3%; T_f: 2168 K

B. Burning rate measurements

The effect of the concentration of BCHMX and HMX on pressure-burning rate dependency was investigated for the propellant samples prepared for this study. A graphical representation of the pressure-burning rate dependency for samples containing BCHMX and HMX is shown in Figures 4 and 5.

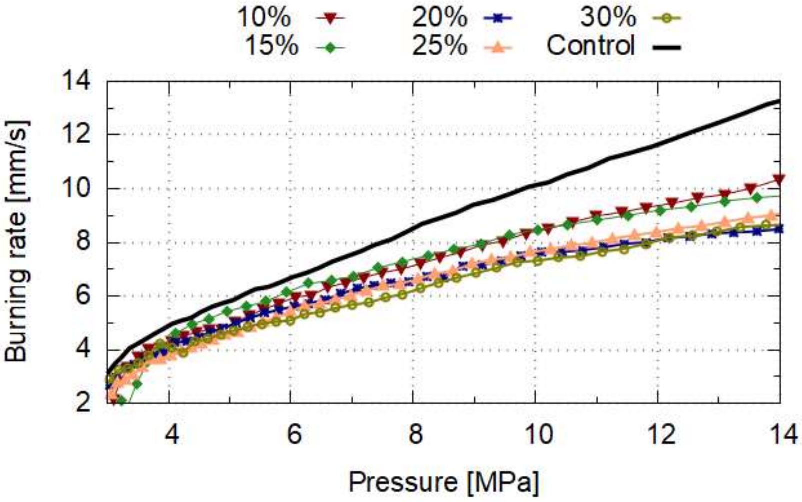

Fig. 4 Burning rate-pressure dependency for BCHMX propellant series.

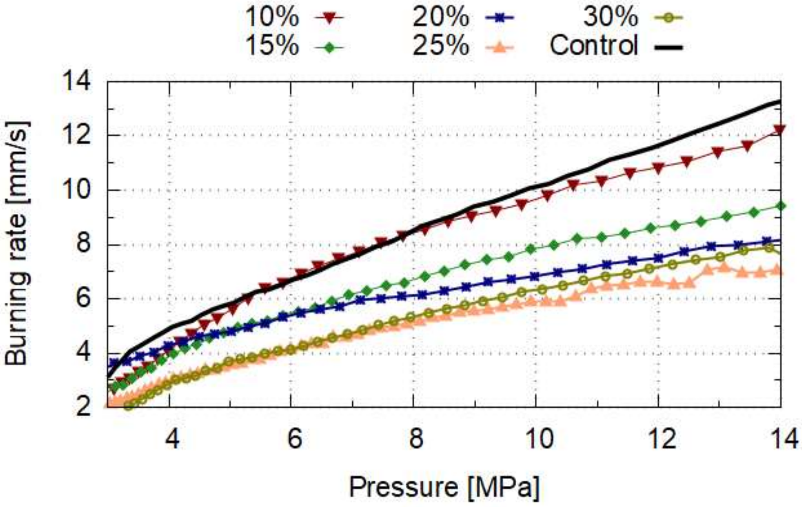

Fig. 5 Burning rate-pressure dependency for HMX propellant series.

The replacement of ammonium perchlorate with nitramine in propellant results in a decrease of burning rate as described in the literature [24, 25]. This effect is to some extent noticeable for all propellants containing either of the studied nitramines, especially for samples with a higher content of nitramine. The burning rates of the propellants containing 10-15% BCHMX (fastest burning samples containing BCHMX) are at the same level as propellants with

15% of HMX. Samples with higher BCHMX content (20, 25 and 30%) show a lower burning rate with no significant effect of the BCHMX content on the burning rate of the propellant. In the case of HMX-containing propellants, the burning rate gradually decreases with increasing nitramine content, an effect that is more noticeable than for the BCHMX propellant samples. The highest burning rate was observed for the propellant containing 10% HMX which is comparable to the Control sample burning rate in the pressure range of 5-9 MPa.

C. Determination of pressure exponents

Pressure exponents in the pressure range of 3-14 MPa were calculated from the experimental data for all the propellant samples used in the study. The pressure exponent is an important parameter in terms of combustion stability – low values of pressure exponent (< 1) are desirable for rocket propellants in terms of burning stability and safety [26]. The pressure exponents for the propellant samples with different nitramine content range between 0.66 and 0.83 for BCHMX and between 0.60 and 0.92 for HMX (compared to 0.83 for the Control sample without nitramine). A graphical comparison of the calculated pressure exponents is shown in Figure 6. The pressure exponents for the propellant containing BCHMX seem to be less dependent on the nitramine content and overall are lower than for the Control propellant.

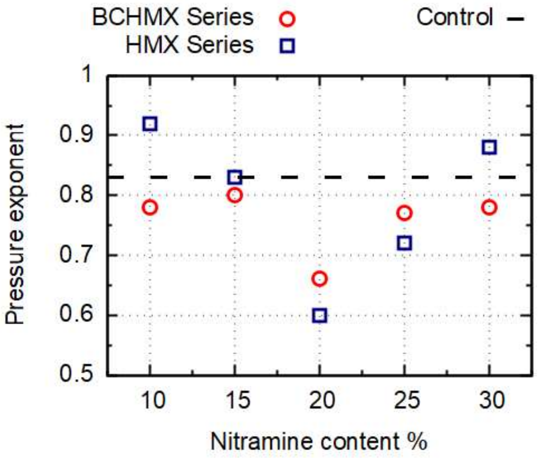

Fig. 6 Pressure exponents of nitramine series.

D. Ignitability of propellant samples

It was found that the addition of BCHMX to the propellants improves the ignitability. Figures 7 and 8 show the pressure versus time record for the combustion of propellants containing BCHMX and HMX. An initial pressure step between 2-3 MPa corresponds to the ignition of 13 g +/- 0.3 g of black powder, while the subsequent gradual pressure evolution corresponds to the propellant combustion. The easy ignition can be recognized as a smooth and gradual increase of the pressure evolution, as can be seen in Figure 7 for BCHMX propellants. In the case of HMX compositions (Figure 8) it is noticeable that the pressure drop corresponds to the time delay between the burn out of the black powder and the ignition of the HMX propellant sample. It indicates a more difficult ignition of propellant. As can be seen at Figure 8 there is a significant pressure drop and large scattering in the p-t curves after black powder burnout. This phenomenon is due to greater heat loss rate into the walls of closed vessel than the rate of gaseous product evolution during HMX propellant combustion. In the case of BCHMX propellants evolution of gaseous products is fast enough to compensate the heat losses and thus facilitate initial ignition.

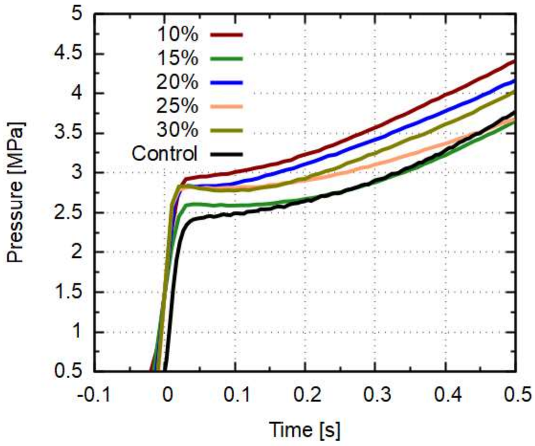

Fig. 7 Pressure development during combustion of BCHMX propellant series in SV2.

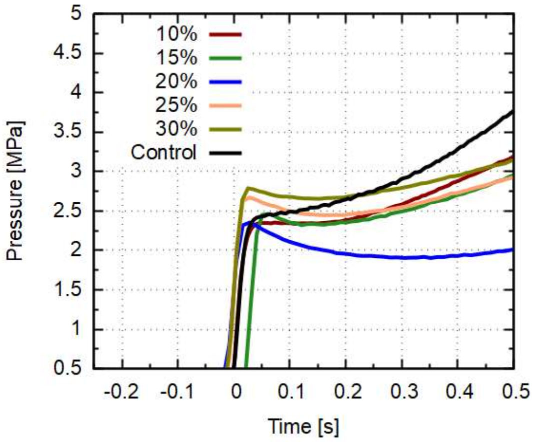

Fig. 8 Pressure development during combustion of HMX propellant series in SV2.

E. Sensitivity to friction and impact

Impact and friction sensitivities of the composite propellants containing nitramines (HMX or BCHMX) were investigated for samples in two forms – as plates and powder. The plate form is a sample that has been cut from the cast propellant and thus represents the sensitivity of the entire propellant mass. The powder sample then represents the sensitivity of filings that may be produced during the mechanical processing of the cast propellant. The powder was prepared by carefully sandpapering of the cut propellant cube. Plates were prepared by cutting a 2x2x5 mm plate from the propellant.

The sensitivity to friction of the investigated composite propellant (Figure 9) did not increase with the addition of nitramine and in the case of the powder sample, in fact, a decrease in sensitivity was noted. No significant differences were observed between the used nitramines. An anomaly was observed in the sensitivity of the powder sample containing 10% BCHMX, which is significantly lower than would be consistent with the overall trend. The reason for this behavior is not entirely clear. The friction sensitivity F_{50} of the propellant without added nitramine was around 80 N (both forms), while the sensitivity of nitramine containing propellants ranged from 70-120 N for plates to 100-180 N for powder, which is for handling purposes comparable to RDX (127 N) [27].

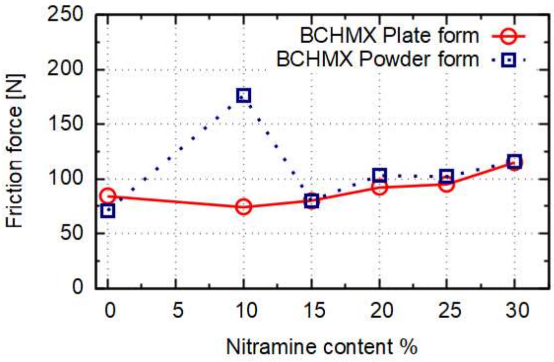

Fig. 9 Friction sensitivity of BCHMX containing composite propellant F_{50} value over BCHMX content.

The sensitivity to impact (Figure 10) of investigated propellants without nitramine E_{50} was found to be 14 J for plate form and 7.2 J for powder form. The sensitization of the propellant by the addition of nitramine is not affected by its content, but changes in relation to the combination of sample form and nitramine. The sensitivity of propellant containing HMX is in range 5.8-8.3 J regardless of the sample form, which makes these samples approximately as sensitive as RDX (6.9 J according to Künzel [27]). In case of BCHMX-modified propellant, the addition of BCHMX increases the impact sensitivity for both forms of the sample. Impact sensitivity for plate propellant containing BCHMX is 6.3-7.2 J (RDX level), while for powder propellant the sensitivity is increased to 2.3-3 J, i.e., approximately to the PETN level (3.9 J) [27].

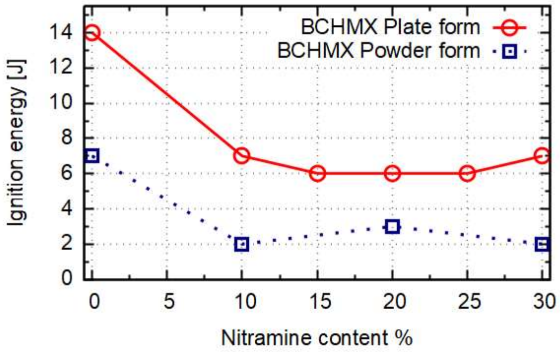

Fig. 10 Impact sensitivity of BCHMX containing composite propellant E_{50} value over BCHMX content.

The results of sensitivity measurement were recently published in conference and they are described in more detail in this work [14].

IV.Conclusion

The aim of the study was to compare the ballistic parameters of composite propellants containing various amounts of BCHMX (cis 1,3,4,6-tetranitrooctahydroimidazo-[4,5-d]imidazole) and HMX (1,3,5,7-tetranitro-1,3,5,7-tetrazocane).

Based on calculations it was found, that for high-performance propellant containing 18-20 % aluminum, the specific impulse is slightly increased by replacing the ammonium perchlorate by either of the considered nitramines at concentrations of 10 and 15% respectively.

Further work was focused on the compositions with 8% aluminum. Specific impulse was calculated for propellants containing 0-50% of nitramines. At first, replacing of ammonium perchlorate by nitramines results in a decrease of a specific impulse, with the minimum value at about 20% of nitramine. With further addition of nitramines, the specific impulse increases. Propellant containing 30% of BCHMX or 45% of HMX exceeds the specific impulse of unmodified propellant.

In terms of burning rates, the propellant containing 10% HMX achieved the highest burning rate of all HMX modified compositions where, in the pressure range of 5-9 MPa, the burning rate was comparable to the Control sample without nitramine. Higher burning rates were observed especially for higher contents of BCHMX in the propellants compared to propellants containing the same amounts of HMX. The pressure exponents for the propellant containing BCHMX are lower than for the propellant without nitramine. The BCHMX-containing propellants exhibited better ignitability compared to the HMX-containing propellants.

The sensitivity to friction does not increase with the addition of nitramines. Impact sensitivity of propellants increases with the addition of nitramines, especially when the propellant is in powder form.

Funding Sources

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