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Burning Characteristics of the HMX/CL-20/AP/Polyvinyltetrazole Binder/Al Solid Propellants Loaded with Nanometals

Alexey V. Sergienko,[a] Elena M. Popenko,[a] Konstantin V. Slyusarsky,[a] Kirill B. Larionov,[a] Ella L. Dzidziguri,[b] Elena S. Kondratyeva,[b] and Alexander A. Gromov*[a, b]

Abstract: The paper investigated the effect of metal nanopowders additive on the combustion properties of HMX/CL-20/AP/polyvinyltetrazole binder/Al propellants. Using thermal analysis, the authors described the effect of aluminum, boron, zinc, nickel, copper, and molybdenum and identified the combustion in a pressure range from 4 to 10 MPa with a pressure step of 1 MPa. No significant correlation between the oxidation properties of the n-Me powders and the combustion properties of propellants was discovered. An addition of nanopowders caused an increase in the propellant burning rate by approximately 30% for n-Al, n-B, n-Ni, and n-Mo independent from the pressure values. An addition of n-Cu resulted in a burning rate increase by a factor of 4.9 due to coppers' probable catalytic activity during interaction with nitroesters and cyclic nitramines in a solid phase. n-Zn additive increased the propellant burning rate by factors 2.3 and 3.6 at 4 and 10 MPa, respectively, due to catalytic activity of zinc in a gaseous phase.

Keywords: aluminized solid propellant · nanometal · non-isothermal oxidation · combustion · burning rate

1 Introduction

Modern solid propellants are based on active polyvinyltetrazole binders and powerful explosives that contain cyclic nitramines RDX, HMX, and CL-20 [1–3]. A control of their ballistics is a typical issue for such propellants [4–5] as well as an increase of their burning rate [6–8].

Cyclic nitramines are chemically resistant to the influence of conventional catalysts used in solid propellants: metal oxides [9], metal stearates [10], lead salicylates [11], ferrocene, etc. [9, 12]. Nevertheless, complex components [13–15] or metal nanopowders [16–18] show good catalytic activity for the conversion of cyclic nitramines during combustion. Nano-sized metal powders (or metal nanopowders, *n-Me*) have a stronger effect on the propellant's burning rate in comparison with other catalysts [19]. Metal complexes with energy-saturated ligands are known as accelerators of thermal decomposition and combustion of propellants based on RDX, HMX, and CL-20 [6, 20–22]. However, when aluminum is added to such propellant formulations, the catalytic efficiency of the metal complexes decreases [22]. One of the possible ways to control the burning rate of aluminized propellants with active binders and cyclic nitramines is activation of burning of micron-sized aluminum powders (*μ-Al*) with the nano- aluminum (*n-Al*) [23, 24] and other nanometals addition [8, 9].

In this work, we studied the ignition and combustion characteristics of the HMX/CL-20/AP/polyvinyltetrazole binder/Al solid propellant with a partial substitution of μ-Al to n-Me. The aluminum, boron, zinc, nickel, copper, and molybdenum nanopowders were studied by non-isothermal heating (DTA-TG analysis) and combustion in the propellant. The aim of this work was to estimate the effect of n-Me additive on the propellant's burning law and DTA-TG reactivity parameters of the propellant formulations.

2 Materials and Methods

2.1 Nanometals Characterization

The nanopowders of aluminum, boron, zinc, nickel, copper, and molybdenum were used in the present study. All powders were produced by an electric wire explosion method [25]. The exception was boron nanopowder that was obtained via a boron oxide reduction by calciothermic reduction. Boron is not a metal, but its combustion is close to metals with a volatile oxide [24].

[a] *A. V. Sergienko, E. M. Popenko, K. V. Slyusarsky, K. B. Larionov, A. A. Gromov*
National Research Tomsk Polytechnic University, Tomsk, Russia
[b] *E. L. Dzidziguri, E. S. Kondratyeva, A. A. Gromov*
National University of Science and Technology "MISiS", Moscow, Russia

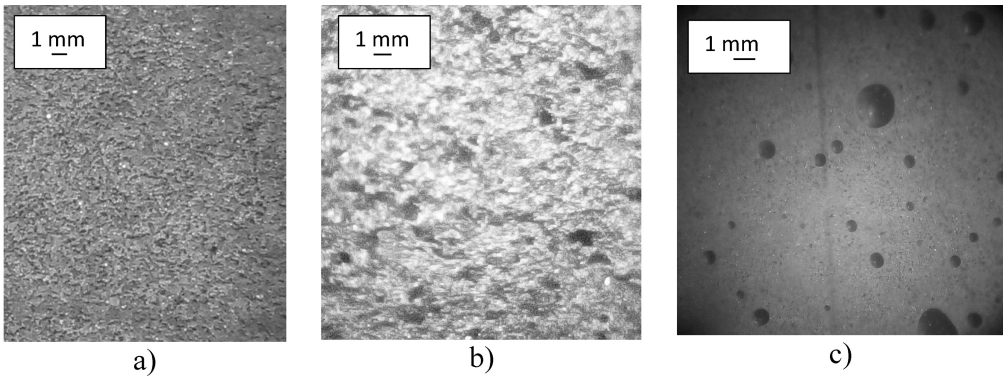

Figure 1. Optical images of the surface of the propellant samples with n-Al (a), n-Ni (b), and n-Cu (c) additives.

2.2 Reactivity Parameters of n-Me and μ-Al

The reactivity parameters [26] for n-Me and μ-Al for comparison were studied by a DTA-TG analysis using a Netzsch STA 449 F1 Jupiter (Netzsch Mastermix Ltd., Germany) analyzer in air with a heating rate 10 °C/min. The temperature of the intensive oxidation onset (T_{on}), the maximal oxidation temperature (T_{max}), and the final degree of metal oxidation (α) were determined using the approach from [23, 26].

2.3 Propellant Formulations

The efficiency of n-Me additives was estimated using the propellant formulation with the following composition: HMX, CL-20, ammonium perchlorate (AP), 19 wt.% of an active binder, 20 wt.% of μ-Al powder, and 0.3 wt.% of carbon black with an average particle size of 150 nm. HMX of two grades (with a particle size of 100–250 μm and 50–100 μm) was used with a mass ratio 25:75. CL-20 powder had the particle diameter of 100 μm. The particle size of the AP powder was varied from 160 to 315 μm. Polyvinyltetrazole rubber was plasticized by nitroester and used as an active binder. Al components were synthesized and tested on purity in Russia.

Metal nanopowders were loaded into a basic propellant formulation by a partial replacement of 6 wt.% of μ-Al. In result, the propellant contained:

- 20 % wt. of μ-Al (basic formulation);
- 14 % wt. of μ-Al + 6 % wt. of n-Me (experimental formulation).

The surface of the tested propellants with n-Me additives (Figure 1a,b) had the same grey color as the basic formulation. The only exception was the propellant with n-Cu: the color of the propellant became greenish with greenish drops of the plasticizer on the surface after mixing (Figure 1c). It indicates a high catalytic activity of n-Cu in that formulation even under room temperature.

2.4 Propellant Burning Test

The burning tests were executed for the propellant samples (cylinders with 15 mm in height and 10 mm in diameter). The burning rate of the propellants was determined in the Vielle bomb (Figure 2). The sample's burning time was measured by a high-speed video camera Panasonic, Japan. The two values of the nitrogen pressure were selected for a precise analysis of 4 MPa (stable burning onset) and 10 MPa (end of the range). The propellant samples were placed vertically and glued to the plastic plate. The number of samples studied at each nitrogen pressure was varied from 10 to 12.

The experimental results were approximated by the Arrhenius law (eq. 1).

$$u = AP^{v} \quad (1)$$

where *A* and *v* are the experimental coefficients; P is the pressure, MPa.

The relative error of the burning rate measurement was ±1.1 % and for the pressure coefficient, the relative error was ±2.5 %.

The effect of the n-Me additive on the burning rate of the propellant was evaluated by the coefficient K (eq. 2) [5] calculated for pressures 4 and 10 MPa:

$$K = u/u_0 \quad (2)$$

here u/u_0 is the burning rate of the propellant with/without n-Me additive at certain pressure, mm/s.

The theoretical burning temperature of the products T_b and standard specific impulse I_s for the studied compositions were calculated as well.

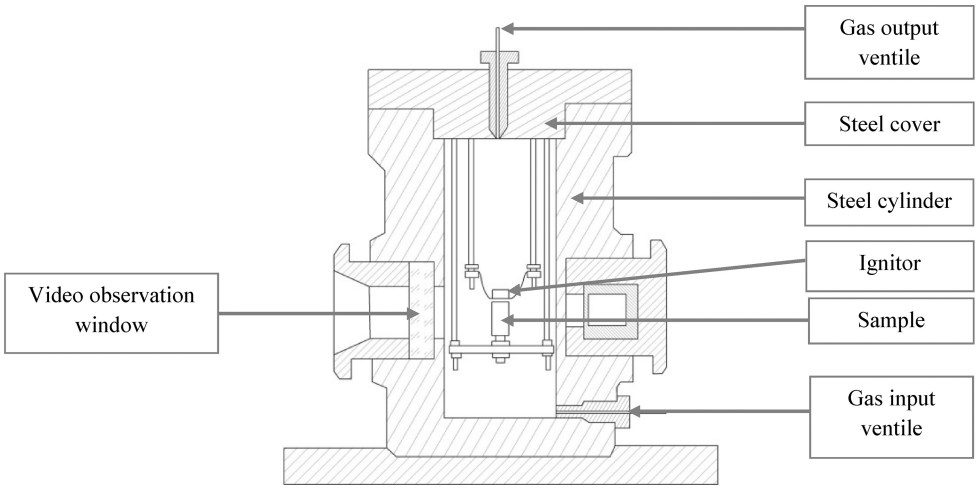

Figure 2. Vielle bomb scheme.

Table 1. Physical characteristics and reactivity parameters of studied metals.

Parameter	Aluminum μ-Al	n-Al	n-B	n-Zn	n-Ni	n-Cu	n-Mo
Active metal content, wt.%	98.5	90.0	94.2	90.3	99.8	92.4	92.0
Specific surface area(BET), m^2/g	0.5	15.5	11.0	5.3	7.5	6.8	5.4
T_{on}, °C	570	510	570	500	340	200	350
T_{max}, °C	610	580	1350	n.a.	410	220	441
α, %	8.5	35.6	9	16.2	80.2	81.6	42.3
Melting temperature of metal, ° C	660	660	2075	419	1453	1083	2617
Boiling temperature of metal, ° C	2518	2518	3865	906	2732	2567	4612
Metal melting enthalpy, kJ/mole	10.8	10.8	23.6	7.3	17.6	13.0	28.1
Theoretical burning enthalpy of metal, kJ/mole	1675	1675	1254	350	240	162	745

3 Results and Discussion

3.1 Physical Characteristics and Reactivity Parameters of Nanometals

Physical characteristics and reactivity parameters of the n-Me by oxidation in air are given in Table 1 as well as the properties of the μ-Al powder for comparison.

Based on the data in Table 1, the content of non-oxidized metals (Al°, B°, Zn° etc.) in the n-Me powders were maximal for n-Ni (99.8 wt.%), while for another n-Me it was rather low (90–94 wt.%). For n-Me, the values of Me° were incomparably high as compared to those of the μ-Me powders. The specific surface area for the studied n-Me was maximal for n-Al (15.5 m^2/g), which corresponds to ~150 nm mean-surface particle diameter.

DTA and TG curves for the non-isothermal oxidation process in air for μ-Al and n-Me are presented in Figure 3. Reactivity parameters for μ-Al and n-Me are given in Table 1. The obtained results are in good agreement with the data presented in periodicals for metal powders [20–21].

According to the DTA-TG data, the temperature of oxidation onset T_{on} was maximal for n-B and μ-Al (570 °C). The minimal values were observed for n-Cu (200 °C) and n-Ni (340 °C). Lower temperature of intensive oxidation onset T_{on} corresponded to a lower maximal oxidation temperature T_{max} and a higher conversion α at the end of heating: minimal values of temperatures T_{max} as well as maximal conversion values α were observed for n-Cu and n-Ni. According to the DTA-TG data n-Cu and n-Ni powders were the most reactive among the studied n-Me (Table 1). These metals are expected to have higher efficiency for propellants. The lowest reactivity was obtained for μ-Al and n-B, while these metals have the highest theoretical burning enthalpy.

3.2 Ballistic Properties

Ballistic properties of the propellants loaded with n-Me as well as density values are presented in Table 2. For n-Zn, n-Cu, and n-Ni, the K coefficient value was dependent on pressure. K values for n-Zn, n-Cu, and n-Ni changing with pressure was significant. For all the modified formulations except for n-Cu, the *A* values lie in a range of 3.5–4.0. The *v* values were in a range of 0.7–0.8 for all the samples except

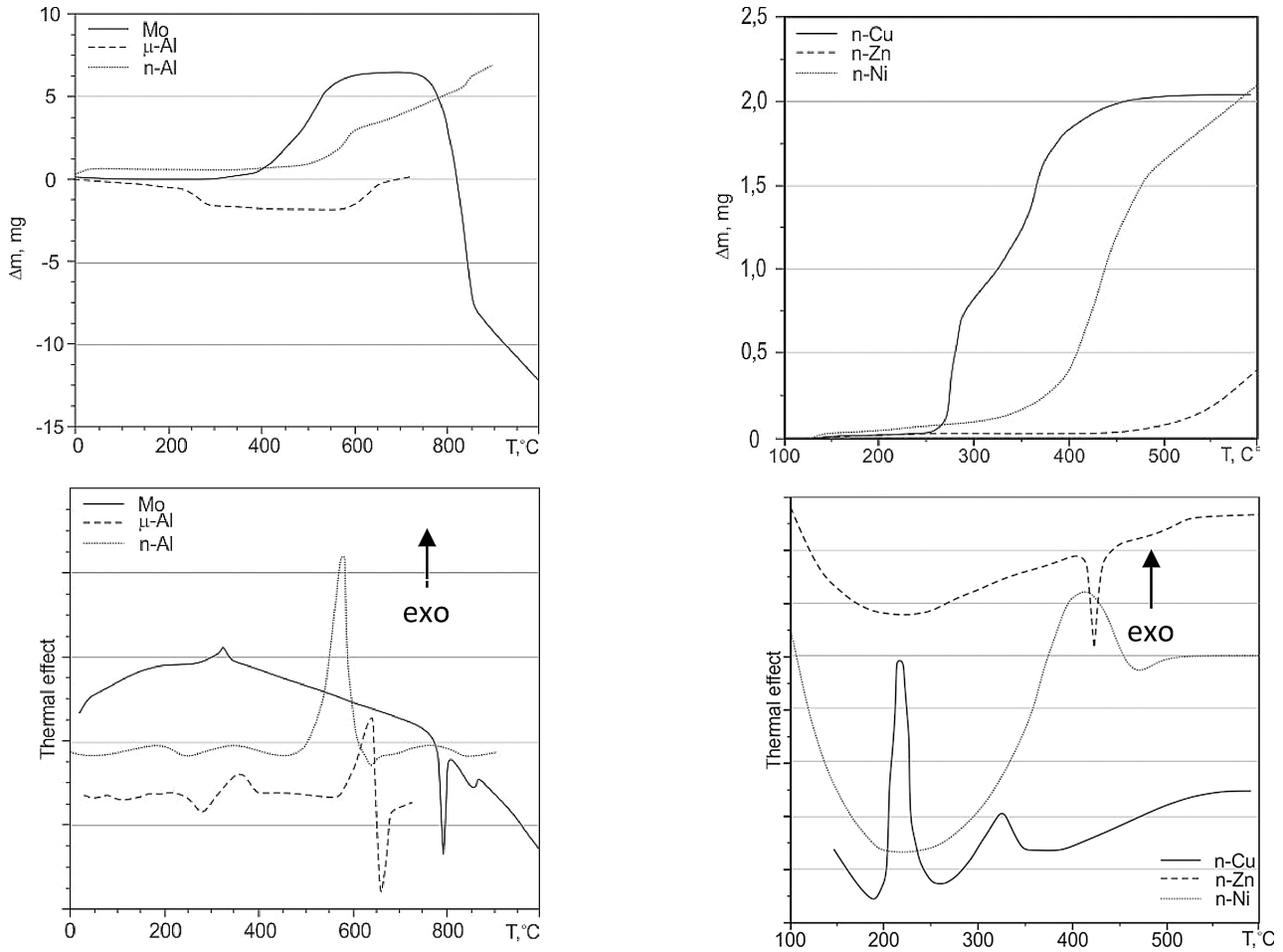

Figure 3. TG and DTA-curves of μ-Al and n-Me oxidation process.

Table 2. Ballistic, energetic properties, density and porosity of basic formulation with μ-Al and n-Me loaded propellants.

Propellants with metal	K		Burning law	T_b, °C	I_s, $N \cdot s \cdot kg^{-1}$	Density, kg/m^3		Porosity, %
	4 MPa	10 MPa				Theoretical	Real	
μ-Al (basic formulation)	1.0	1.0	$u = 3.1\ P^{0.7}$	3655	2573	1787	1748	2.2
n-Al	1.3	1.3	$u = 4.0\ P^{0.7}$	3655	2573	1787	1737	2.8
n-B	1.2	1.2	$u = 3.5\ P^{0.7}$	3502	2496	1779	1747	1.8
n-Zn	2.3	3.6	$u = 3.5\ P^{1.2}$	3447	2467	1832	1804	1.6
n-Ni	1.3	1.5	$u = 3.5\ P^{0.8}$	3415	2454	1838	1803	1.9
n-Cu*	4.9	4.8	$u = 16.2\ P^{0.7}$	3504	2479	1838	1779	3.2
n-Mo	1.3	1.3	$u = 3.9\ P^{0.7}$	3140	2463	1841	1813	1.5

*cured sample, see also Table 3.

for n-Zn. Thus, all n-Me were given a high impact in a gaseous phase of the propellant burning.

Sorted by an increase of the burning rate, metals formed the following reactivity row: n-Cu > n-Zn > n-Al ≈ n-Ni ≈ n-Mo > n-B. The efficiency of the n-Me modified propellants did not significantly corresponded to the n-Me reactivity at non-isothermal oxidation. n-Al, n-Ni and n-Mo with very different reactivity parameters have nearly equal values of the efficiency coefficients K during combustion (Table 2). Low efficiency of n-B by combustion was apparently caused by the formation of a liquid oxide layer on the surface of its particles, which prevented diffusion of the oxidant to the boron in the particle. The highest increase in the burning rate was obtained with n-Cu and n-Zn. The burning rate was increased with the factors 4.9 and 2.3 at pressure 4 MPa and with the factors 4.8 and 3.6 at 10 MPa.

The increased *A* value for the samples with n-Cu allowed us to suppose that copper plays the role of catalysts for

heterogeneous chemical reactions in the studied formulation. Copper is known to accelerate a release of NO_2 from nitroester. Increased v value for samples with n-Zn suggests its catalytic activity to appear at gaseous phase.

Relative difference between the burning temperatures for all the samples was 13%, whereas for specific impulses, it was less than 5%. The highest temperature and specific impulse values were calculated for the basic propellant and the n-Al modified propellant. The lowest temperature was obtained for the n-Mo modified sample, whereas the lowest void and the standard specific impulse were calculated for the n-Ni modified propellants.

The different density and porosity of propellant samples, of course, affects the burning characteristics (Table 2). The minimal real density has the n-Al loaded formulation and the maximal one has the Mo-loaded sample. Generally, the porosity values for all samples were low (1.5–2.2%), however, for n-Al and, especially, for n-Cu the porosity values were relatively high. In the case of n-Al it might be connected with the highest specific surface (15.5 m^2/g) and for n-Cu – with the chemical reaction inside the sample started already at room temperature (see also Figure 1, c).

3.3 Effect of n-Me Additive

In [4–8], the effect of n-Me on the propellants' combustion was explained by a mechanism of the n-Me influence on the burning rate from the position of the thermal theory of Zeldovich-Belyaev. It is based on the model with the leading stage in the gas phase and the diffusional mechanism of combustion of metal particles in the gas-phase reaction. According to the thermal theory, the burning rate of the metallized propellant is determined by the superposition of heat release rates during a homogeneous reaction and heterogeneous diffusion oxidation of metal particles (3).

$$u = \sqrt{a \cdot r_{\text{hom}} + (1-a) \cdot r_{\text{het}}} \approx \sqrt{a \cdot P + (1-a) \cdot P/d^2} \quad (3)$$

where:

- d is the diameter of a metal particle in the flame;
- $r_{\text{hom}}, r_{\text{het}}$ are the heat release rate of homogeneous and heterogeneous reactions;
- a is the share of the homogeneous reaction heat release;
- P is the pressure.

Thus, the total heat release is not the main factor of the process that only defines the rate of heat input into the so-called "reaction area." The more metal burns near the surface of the fuel, the higher the heat release and the burning rate have to be. The amount of metal burned in this zone depends on the following factors: the metal ignition temperature; the width of the "reaction zone," the time of metal particles residence and composition of the oxidizing atmosphere. Very reactive n-Me initiate earlier ignition of large particles of μ-Al, which leads to an increase in the heat flux from the "zone of influence" and increases the burning rate in the macroscale. The incendiary effect of small particles is due to their radiation and a direct heat action when colliding with large not-yet-ignited aluminum particles. In this case, metals with lower ignition temperature are preferable for such purposes (n-Ni, n-Cu and n-Mo, Table 1).

Metal oxides can make a certain contribution to the acceleration of combustion with a catalytic activity. In this case, highly dispersed γ-alumina is an effective catalyst for combustion of propellants based on AP and inert binders like HTPB [27] as well as the propellants based on ammonium nitrate (AN) [28]. Copper oxide accelerates the double-base gun powders combustion due to its decomposition products (nitrogen oxides). The accelerated combustion of propellants with n-Cu powder was possibly due to the combined effect of the metal and its oxide. Only the propellants with n-Cu showed signs of interaction of copper with the components of the propellant during the manufacturing process: after mixing the formulation acquired a greenish gray color, its volume increased and gaseous products were released. A few hours after mixing, the mass was cured and the samples acquired the necessary strength. Table 3 shows the burning characteristics of the cured and uncured n-Cu contained samples.

Table 3. Combustion properties of cured and uncured propellants with n-Cu.

Propellant sample	K		Burning law
	4 MPa	10 MPa	
Cured n-Cu	4.9	4.8	$u = 16.2\ P^{0.65}$
Uncured n-Cu	3.7	4.1	$u = 9.8\ P^{0.8}$

The cured sample was characterized by an increased burning rate and a smaller value of the pressure coefficient v in comparison with the uncured one. A three-dimensional polymeric net formed during curing kept soot and metal particles in the reaction layer during combustion. By increasing the residence time of metal particles in the burning zone of the propellant, they accumulated on the burning surface and formed a metal porous cake [28]. As a result, the share of the metal oxidized in the condensed phase increases and the catalytic activity of n-Cu also increased. Finally, due to the increased heat generation, the burning rate increased, whereas its dependence on pressure ν decreased.

The propellants with n-Zn were characterized by a high dependence on the burning rate under pressure ($v = 1.2$). Combustion of compositions with n-Zn revealed an anomalously high dispersion of the burning sample. At the end of thecombustion, the entire internal surface of the combustion chamber was covered with a thick and porous layer of finely dispersed condensed combustion products (Figure 4).

Figure 4. Condensed combustion products of the propellant loaded with n-Zn.

This phenomenon occurs due to intense evaporation of the zinc melt at 419 °C and, perhaps, boiling at 904 °C (Table 1). The calculated temperature of the combustion products in a chamber was high (3447 °C, Table 2).

4 Conclusion

The effect of partial replacement of micron-sized aluminum on Al, Zn, Cu, Ni, Mo, and B nanopowders was studied in HMX/CL-20/AP/polyvinyltetrazole binder/Al propellants. The non-isothermal oxidation of n-Me was studied by a DTA-TG analysis. The combustion properties of n-Me loaded propellants were studied in the Vielle bomb. No significant correlation between the reactivity parameters of n-Me oxidation process and the burning rate was found in [29]. The partial replacement of μ-Al on n-Me resulted in the increased burning rate by 30% for n-Al, n-Ni, n-Mo and n-B and by maximum 390% and 260% for n-Cu and n-Zn samples, respectively. At the same time the standard specific impulse did not changed significantly, i.e. by less than 5%.

Analyzing the obtained results, the conclusion could be made that the mechanism of the influence of the studied n-Me on the burning rate is different. n-Al, n-Ni, and n-Mo had the thermal mechanism of combustion. They increase the heat flow from the zone of the gas phase adjacent to the burning surface by initiating an earlier ignition of the micron-sized particles of μ-Al. Ignition effect of small particles was defined by their radiation and collisions with larger particles. It explains the same effect of different n-Me on combustion: the main requirement was lower ignition temperature (in comparison with μ-Al). The accelerating effect depends on frequency of nanoparticle collisions with μ-Al particles.

n-Cu and n-Zn have rather high reactivity with respect to energy-saturated components of the propellant and catalyze their transformation in a burning wave. Copper catalyzes homogeneous reactions while zinc catalyzes heterogeneous reactions in gaseous phase in combustion wave. At the same time, copper powder starts the reaction with the propellant components during its production at room temperature, which indicates its incompatibility with the components of such propellant. That's why using n-Cu is undesirable in propellants with active binders.

The replacement of a small part of micron-sized aluminum could be recommended for the HMX/CL-20/AP/polyvinyltetrazole binder/Al propellants if an increase in the burning rate is required without a substantial change in the formulation and fractional composition.

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