

## COMBUSTION OF AMMONIUM DINITRAMIDE/POLYCAPROLACTONE PROPELLANTS

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This paper is devoted to the investigation of characteristics and mechanisms of combustion of a composite solid rocket pseudo-propellant of stoichiometric composition based on ammonium dinitramide (ADN) and polycaprolactone (PCL). Experimental data on the dependence of burning rate on pressure in the pressure range of 4–8 MPa for the ADN/PCL propellant and the influence of molecular weight of PCL on this dependence are presented. The temperature of the final combustion products and the temperature profile in the combustion wave of the propellant have been measured by a microthermocouple method at 4 MPa. Also, the influence of a CuO catalyst on pressure dependence of burning rate and temperature profile has been investigated. The flame structure of the propellant ADN/PCL at 0.1 MPa has been studied. Data obtained elucidating combustion mechanisms and place of action of catalyst are discussed.

### Introduction

Ammonium dinitramide (ADN) is a powerful chlorine-free oxidizer, which can replace ammonium perchlorate (AP) in solid rocket propellants. Since the combustion products of ADN-based propellants are not toxic, these propellants are environmentally friendly, and investigation of them is of great interest. Important physicochemical properties and combustion characteristics of ADN and ADN-based propellants were published for the first time in Ref. [1]. ADN-based propellants have a higher specific impulse than AP-based propellants [1,2]. The study of combustion mechanisms of pure ADN was the subject of several investigations [3–6]. It was found that the burning rate of ADN is controlled by reactions in the condensed phase and that the ADN flame has a multizone structure. However, there are only a few papers devoted to the study of the combustion characteristics and the combustion mechanisms of ADN-based propellants and sandwiches with different types of binders, such as hydroxyl-terminated polybutadiene (HTPB) [7,8], glycidyl azide polymer (GAP) [8,9], paraffin [10,11], polycaprolactone (PCL) [12], and polybutadiene polymer [13,14].

One of the objectives of the study of propellant combustion mechanisms is the development of a combustion model that can predict combustion characteristics of solid propellants. The development of a combustion model of composite solid propellant requires information on propellant flame structure. The flame structure of ADN-based sandwiches has been investigated in Ref. [15]. Results showed either no or insignificant effects of diffusion

flames on the processes controlling the propellant burning rate at 0.1–1.4 MPa. However, the burning rates of sandwiches based on HTPB increased nearly 1.5-fold with the pressure increase from 1.5 to 7 MPa [14]. This suggests a possible influence of the HADN/Binder diffusion flames on the burning rate of sandwiches. It has been shown in Ref. [7] that the reactions in the condensed phase control ADN/HTPB propellant combustion.

It is not known at present which factor determines the burning rate of ADN-based propellants. Combustion instability of pure ADN takes place in the pressure range of 2–10 MPa [3]. However, even small amounts of organic additives (~1%) result in a reduction of both burning rates and their scatter in this pressure range [4,16]. In contrast, the addition of Cu<sub>2</sub>O (2%) leads to an increase in the burning rate at 0.1–2 MPa, followed by a decrease above 4 MPa [4]. The burning rate and pressure exponent for ADN-based propellants depend on the type of binder and the oxidizer/binder ratio [11–14]. The chemical mechanism of combustion of ADN-based propellants is not well understood.

The objective of this research is the comprehensive experimental investigation of the combustion mechanisms of ADN/PCL propellants. The following propellant combustion characteristics were studied: pressure dependence of burning rate at 4–8 MPa; temperature profiles in the propellant flame at 4 MPa; the effect of CuO catalyst and the molecular weight of the PCL on the burning rate, pressure exponent  $\nu$ , and temperature profile; and flame structure of ADN/PCL propellant at 0.1 MPa.

## Experimental

The ADN used in this study was synthesized at the Zelinsky Institute of Organic Chemistry of the Russian Academy of Science. It contains 2% of ammonium nitrate as an impurity. The melting point (m.p.) of ADN is 365–367 K. Two types of PCL, with different molecular weights of 10,000 (PCL(10,000)), and 1250 (PCL(1250)), have been used. The former polymer at room temperature has a flake form (m.p. 333 K) and the latter one is a waxy solid (m.p. 309–321 K). Ground ADN powder with an average particle size of  $\sim 40 \mu\text{m}$  was used for preparation of propellants. Non-cured ADN/PCL propellant of stoichiometric composition ( $St_b$ ) consisted of 89.08 wt% ADN and 10.92 wt% PCL was prepared by mixing of the ingredients in a dry cell at the temperature slightly exceeding melting point of the polymer. Addition of 2% CuO to  $St_b$  means that in this propellant mass fractions of  $St_b$  and CuO are equal to 98 and 2%, respectively. Strands with diameter of 6 mm and length of 10–12 mm were pressed at pressure of  $\sim 390 \text{ MPa}$ . The density of strands was  $1.58 \text{ g/cm}^3$ . The sides of the strands were protected with thin layer of high-vacuum silicone grease. Measurement of the burning rate at 4–8 MPa was conducted by two methods: by pressure control during the combustion process in a combustion chamber of constant volume in argon and by videotape recording of movement of the burning surface through the combustion chamber windows.

Probe mass spectrometry is the most effective and universal method for investigation of the solid propellant flame structure [17]. It allows the detection of all the stable species present in the flame and also the determination of their concentrations and their spatial distributions. Flame structure has been studied at 0.1 MPa of argon using the mass spectrometric setup [18] with a molecular beam sampling system equipped with a time-of-flight mass spectrometer. The sampling of combustion products was continuous. The frequency of measurements was 25 Hz. Displacement of the burning surface during the one cycle of measurement was  $\sim 0.1 \text{ mm}$  at the rate of movement of burning surface of strand toward the probe of  $2.3 \text{ mm/s}$ . Such a spatial resolution is quite sufficient for investigation of a flame zone with the width of  $\sim 1 \text{ mm}$ . Calibrations [5] were conducted for  $N_2$ ,  $O_2$ ,  $CO$ ,  $CO_2$ ,  $N_2O$ ,  $NH_3$ ,  $NO$ ,  $HNO_3$ , and  $H_2O$ . Calibration for water was carried out by vaporization of drop of water in argon flow. Accuracy of measurement of species mole fractions in the range of 0.1–0.4 ( $H_2O$ ,  $N_2$ ,  $N_2O$ ,  $NO$ , and  $CO_2$ ) was about 10%. For species with mole fractions less than 0.1, the error was 20%–30%. The temperature of the final combustion products was measured by  $\Pi$ -shaped WRe (5%)–WRe (20%) thermocouples with a shoulder length of  $\sim 3 \text{ mm}$ , made of  $100 \mu\text{m}$  di-

ameter wire. The thermocouples were located at a distance of  $\sim 2$ – $5 \text{ mm}$  from the strand surface. The temperature of the final combustion products was corrected for heat loss by radiation [19]. The error in determination of the temperature of final combustion products was equal to  $\pm 25 \text{ K}$ . The accuracy of determination of mole fractions of combustion products as well as temperature of final combustion products of the propellants were obtained by processing of data of several experiments (3–4). Thus, error limits presented relate to reproducibility of the data from experiment to experiment. Temperature profiles in the propellant combustion wave at 4 MPa were measured by ribbon WRe (5%)–WRe (20%) thermocouples (thickness 13–15  $\mu\text{m}$  and width  $\sim 140$ – $150 \mu\text{m}$ ) embedded in the strand. The correctness of the thermocouple's application for measurement of a temperature profile in combustion wave of solid propellants has been analyzed by Zenin [20]. We used his procedure for correction of temperature profile for time response of the thermocouple. It is noteworthy that the known dependence of time response of the thermocouple on its thickness, mass burning rate, and temperature was used. The length of thermocouple's shoulders was enough to neglect the heat loss in the thermocouple's shoulders [20].

## Results

### *Influence of Polycaprolactone Molecular Weight and CuO Additives on the Burning Rate*

Data on the dependence of burning rate ( $r_b$ ) on pressure for baseline propellants with PCL(1250) and PCL(10,000) at 4–8 MPa are shown in Fig. 1. Parameters  $A$  and  $\nu$  of the dependence of burning rate on pressure  $r_b = A \times P^\nu$  ( $r_b$  in mm/s;  $P$  in MPa) are presented in Table 1.

$St_b(10,000)$  has a high pressure exponent ( $\nu = 1$ ). Replacement of this binder in the propellant by a polymer with the same structure and chemical composition, but with lower molecular weight (1250) and lower melting point, led to the increase of  $r_b$  (by 1.5 times at 4 MPa and by 1.2 times at 8 MPa) and to the decrease of parameter  $\nu$  to 0.7 (Fig. 1 and Table 1). The burning rate of slightly rich propellant (87% ADN + 13% PCL) was measured for both PCL(1250) and PCL(10,000) at 4 MPa, and it was very close to that of the propellant of stoichiometric composition.

The addition of 2% CuO to propellants  $St_b(1250)$  and  $St_b(10,000)$  increased  $r_b$  at 4 MPa (Fig. 1) and practically did not have an effect on  $r_b$  at 8 MPa. The addition of 2% CuO resulted in the increase of  $r_b$  of  $St_b(1250)$  by 1.23 times and of  $St_b(10,000)$  by 1.38 times at 4 MPa. With the addition of catalyst parameter,  $\nu$  decreased by 1.6 times: to 0.44 for  $St_b(1250)$  and to 0.60 for  $St_b(10,000)$  (Table 1).

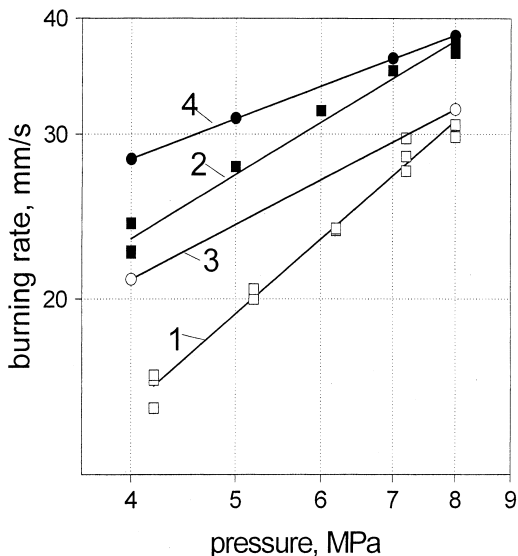


FIG. 1. The dependence of propellant burning rate on pressure: 1 (□), St<sub>b</sub>(10,000); 2 (■), St<sub>b</sub>(1250); 3 (○), 98% St<sub>b</sub>(10,000) + 2% CuO; 4 (●), 98% St<sub>b</sub>(1250) + 2% CuO.

TABLE 1  
Dependence of propellants burning rate on pressure

Propellant	A	$\nu$
100% St <sub>b</sub> (1250)	8.74	0.70
100% St <sub>b</sub> (10,000)	3.79	1.00
98% St <sub>b</sub> (1250) + 2%CuO	15.30	0.44
98% St <sub>b</sub> (10,000) + 2%CuO	9.10	0.60

The main ingredient of the investigated propellants is ADN (89.08%). That is a reason why the effect of the addition of CuO on ADN burning rate has been explored. Combustion of pure ADN over the pressure range of 2–8 MPa is unstable [3]. Our investigation confirmed this fact. However, the addition of 2% CuO to pure ADN resulted in stabilization of ADN combustion. Burning rates of 98% ADN + 2% CuO mixture at 4–8 MPa are compared with those for pure ADN in Fig. 2. It is noticeable that the burning rates of the 98% ADN + 2% CuO mixture are less than the lower boundary of observed scatter of the burning rates of pure ADN. The pressure exponent for the mixture 98% ADN + 2% CuO at 4–8 MPa is negative (similar to additive of Cu<sub>2</sub>O [4]); that is, the burning rate of this mixture decreases with the increase of pressure from 4 to 8 MPa. The different influence of CuO on the burning rate of pure ADN at 4–8 MPa and that of the St<sub>b</sub> propellant may indicate a difference in mechanism of action of CuO in these two cases.

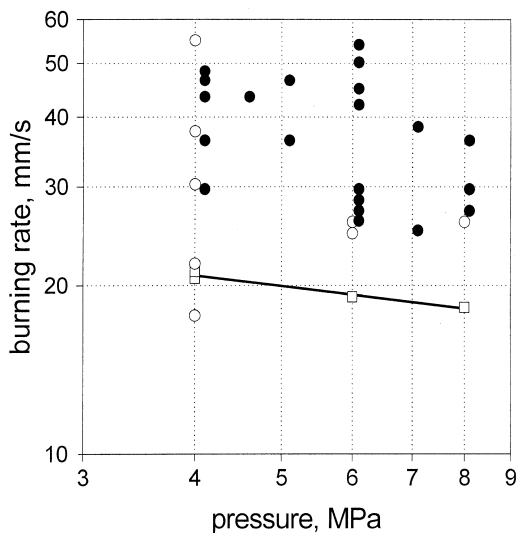


FIG. 2. The dependence of burning rate of mixture ADN + catalyst on pressure: (□) 98% ADN + 2% CuO; (○) ADN (our data); (●) ADN [3].

#### Thermal Structure of the Propellant Combustion Wave

The temperature of the final combustion products of the St<sub>b</sub>(10,000) propellant with radiation correction measured by different thermocouples was equal to  $2870 \pm 25$  K. This temperature is slightly less than the calculated equilibrium temperature (2960 K [21]). The increase of the initial pressure to 8 MPa did not change the temperature of the final combustion products of St<sub>b</sub>(10,000).

The temperature of the propellant burning surface was determined by a method of slope break on the temperature profile [20] in several experiments. In Fig. 3, the part of initial profile (showing slope break) near the burning surface for St<sub>b</sub>(10,000) is presented. We took into account the time response of the thermocouple using the procedure described in Ref. [20] when processing the experimental data. The correction for surface temperature was equal to  $\sim 70$  K. Corrected temperature profiles are shown in Fig. 3. The mean value of burning surface temperature ( $T_s$ ) of St<sub>b</sub>(10,000) was equal to  $630 \pm 10$  K. It is close to estimation of  $T_s$  for pure ADN (640 K) at 4 MPa [6]. The thickness of the thermal layer in the condensed phase (CP) was  $\sim 180$   $\mu\text{m}$ , and the width of the reaction zone in the flame,  $L$ , for St<sub>b</sub>(10,000) was  $\sim 150$   $\mu\text{m}$ . The temperature gradient near the burning surface,  $\varphi$ , was  $\sim 1.9 \times 10^7$  K/m. The addition of 2% CuO to St<sub>b</sub> resulted in the increase of  $T_s$  by 70 K (Fig. 3) and the increase of  $L$  by 50  $\mu\text{m}$  at 4 MPa.

Heat feedback from the flame into the CP by heat conductivity,  $q$ , was calculated by the equation

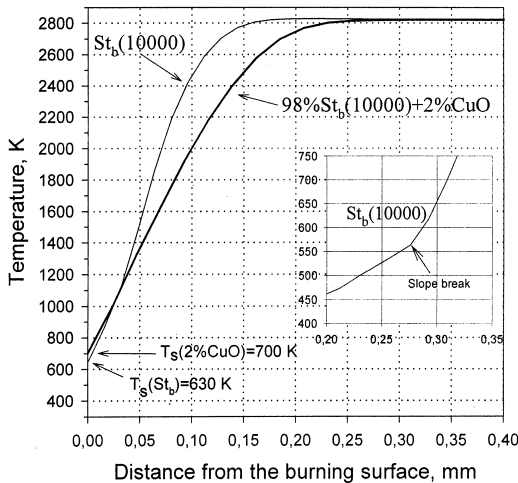


FIG. 3. Corrected temperature profiles in combustion wave of  $St_b(10,000)$  and  $98\%St_b(10,000) + 2\%CuO$  propellants at 4 MPa.

$q = -\lambda(T) \times \phi/m$ , where  $\lambda$  is the coefficient of heat conductivity in the gas phase ( $4.2 \times 10^{-2} \text{ W/m} \times \text{K}$ ) and  $m$  is the mass burning rate. Heat release in the CP reaction layer,  $Q$ , was calculated by formula  $Q = C \times (T_s - T_o) - q + q_m$  where  $C$  is the coefficient of CP specific heat ( $1.26 \times 10^3 \text{ J/kg} \times \text{K}$ ),  $q_m$  is the heat of melting ( $1.34 \times 10^5 \text{ J/kg}$ ),  $T_o$  is initial temperature (293 K). The values of  $\lambda$ ,  $C$ , and  $q_m$  were taken the same as in the case of pure ADN [6]. Heat release rate in the flame was calculated by the approximate formula  $\Phi = C_p \times \phi \times m$ , where  $C_p$  is the coefficient of gas-phase specific heat ( $1.39 \times 10^3 \text{ J/kg} \times \text{K}$  [6]). The addition of 2% CuO to  $St_b$  did not change  $\Phi$ , but decreased  $q$  and increased  $Q$  by a value of  $9.2 \times 10^4 \text{ J/kg}$  (Table 2). As a result, the burning rate increased. The obtained data indicate that reactions in CP control the

TABLE 2  
Results of processing of temperature profiles in the combustion wave of the ADN/PCL propellant without and with 2% CuO and ADN

Propellant	$St_b$	98% $St_b$ + 2% CuO	Pure ADN [6]
$P$ , MPa	4	4	2
$\phi$ , K/m	$1.9 \times 10^7$	$1.3 \times 10^7$	$10^7$
$m$ , kg/m <sup>2</sup> s	25.3	36.3	46.4
$q$ , J/kg	$3.2 \times 10^4$	$1.6 \times 10^4$	$1.3 \times 10^4$
$Q$ , J/kg	$5.4 \times 10^5$	$6.3 \times 10^5$	$5.5 \times 10^5$
$\Phi$ , J/m <sup>3</sup> s	$6.7 \times 10^{11}$	$6.6 \times 10^{11}$	—

burning rate of the ADN-based propellant. The accuracy of data presented in Table 2 is equal to  $\pm 10\%$  for  $\phi$ ,  $\pm 15\%$  for  $q$ ,  $\pm 5\%$  for  $Q$ ,  $\pm 5\%$  for  $m$ , and  $\pm 15\%$  for  $\Phi$ .

#### Flame Structure of Propellant $St_b(1250)$ at 0.1 MPa

Experiments showed that the  $St_b(10,000)$  propellant at 0.1 MPa burned without a visible flame. Brown residue (apparently undecomposed polycaprolactone) remained on the strand holder after the experiments. The temperature of the combustion products was  $\sim 670 \text{ K}$ , which is close to that of pure ADN at 0.1 MPa [5]. So, one can suppose that at 0.1 MPa, only ADN burns, whereas PCL(10,000) only melts and partially decomposes. Replacement of PCL(10,000) by PCL(1250) resulted in the appearance of the visible flame. However, the flame did not cover the whole burning surface of the strand. Separate flame jets moving over the strand burning surface during the combustion were observed. So, the combustion of  $St_b(1250)$  at 0.1 MPa has a torch character with formation of separate seats of burning on the burning surface. During the combustion of the ADN/HTPB (97/3) at 0.1–0.6 MPa, separate seats of burning were also observed [7].

The measurement of temperature and the determination of combustion product composition were conducted in different experiments. The probe (or thermocouple) during the combustion was located either in the luminous zone (torch) or in the dark zone (between torches or far from them). Analysis of the videotape recording allowed the determination of the flame zone, where the probe or thermocouple was located at the moment of measurement.

Results of two experiments on measurement of the temperature profile in the  $St_b(1250)$  flame at 0.1 MPa, which confirm the conclusion regarding

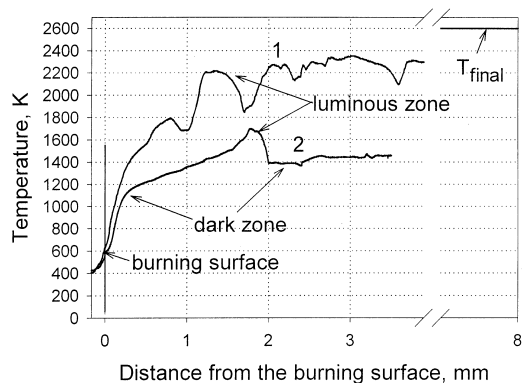


FIG. 4. Temperature profiles in flame of propellant ADN/PCL(1250) at 0.1 MPa

TABLE 3  
Concentrations (in mole fractions) of species and temperature in the flame of propellant  $St_b(1250)$  at 0.1 MPa and of ADN at 0.6 MPa

	$T, K$	$H_2O$	$N_2$	$N_2O$	$NO$	$NH_3$	$HNO_3$	$H_2$	$CO$	$CO_2$	$O_2$
Luminous zone (exp)	~2600	0.39	0.32	0	0.10	0	0	0.03	0.02	0.12	0.02
Thermodynamic calc. [21]	2695	0.40	0.34	0	0.01	0	0	0.03	0.05	0.09	0.03
Dark zone (exp)	~1120	0.32	0.11	0.20	0.20	0.04	0.01	0.01	0.02	0.08	0.01
Pure ADN dark zone at $L_1 \sim 4$ mm (0.6 MPa) [5]	~920	0.31	0.10	0.28	0.23	0.07	0.02	—	—	—	—

torch combustion of this propellant, are presented in Fig. 4. Curve 1 in Fig. 4 corresponds to the case when the thermocouple moved from the torch to the burning surface. Abrupt fallings of temperature on curve 1 are connected with changing of torch location with respect to thermocouple. Curve 2 corresponds to the case when the thermocouple moved to the burning surface from the dark zone. The abrupt increase of temperature at the distance of  $L_1 \sim 2$  mm relates to torch appearance near the burning surface. The videotape recording showed that a dark zone exists near the burning surface. The width of the dark zone varies from  $\sim 1$  mm (near bottom of torch) to 3–4 mm (region between torches). Thermocouple measurements revealed the existence of three zones in the flame (Fig. 4): (1) the narrow dark zone adjacent to the burning surface (width of the zone  $\sim 0.2$ – $0.3$  mm), where the temperature grew from  $\sim 600$  to  $\sim 1150$  K; (2) the dark zone (width of the zone  $\sim 0.5$  to  $\sim 3$  mm), where the temperature slightly increased from 1150 to 1450 K; and (3) the luminous zone (torch), where the temperature increased to 2600 K at the distance of 4–8 mm.

The compositions of the combustion products in the luminous and dark flame zones of propellant  $St_b(1250)$  are presented in Table 3. The temperature of the combustion products in the luminous zone, which is equal to 2600 K, is slightly less than the calculated equilibrium temperature (2695 K [21]); that is, 100% completeness of combustion is not achieved. The presence of NO in the combustion products confirms this conclusion. The element balance in the luminous zone was in satisfactory agreement ( $\pm 5\%$ ) with that in the propellant. The calculated deficiency of carbon in the combustion products determined in the dark zone is equal to  $\sim 50\%$  of the initial amount. This fact indicates that identification of carbon-containing products in the dark zone was incomplete. Besides, we have obtained peaks of the following unidentified masses in mass spectrum of species near the burning surface of the propellant  $St_b(1250)$ : 55, 57, 60, 67, 69, 70, 71, 73, 79, 81, 95, 108, and 115. We suggest that masses from 55 to 115 are responsible for the decomposition products of PCL.

## Discussion

Processing of temperature profiles at 4 MPa showed that the heat feedback from the flame into CP is small in comparison with  $Q$ . Therefore, the reactions in the CP control combustion of the composite propellant as well as in the case of pure ADN.

Additives of binder to ADN decrease the burning rate in contrast to the influence of additives of binder on the burning rate of AP. Additives of binder probably inhibit reactions of ADN decomposition in the CP. The influence of binder properties (molecular weight, melting point) on the burning rate of the composite propellant indicates that the effectiveness of inhibition of ADN decomposition reactions in CP likely depends on these properties. The rate of pyrolysis of PCL(1250) is significantly higher than that of PCL(10,000). Experiments on combustion of  $St_b$  propellants at 0.1 MPa described above confirm this conclusion. The difference between the rates of pyrolysis of PCL(10,000) and PCL(1250) could be a reason for their different influence on the burning rate of the propellant. Slower pyrolysis of PCL(10,000) leads to more accumulation of it on the burning surface. As a result, reactions of decomposition of ADN become significantly slower in the case of the  $St_b(10,000)$  propellant. The effect of inhibition of ADN decomposition in the condensed phase by minor additives of hydrocarbon fuels was discussed earlier by Strunin et al. [4]. The addition of organic fuel (plasticizer, rubber) in the amount of only 1% resulted in a significant decrease of the burning rate of ADN at 0.1 MPa. The following increase of fuel content up to 5% did not have much effect on the burning rate. Similar effects for ADN/HTPB propellants were observed in Ref. [7]. ADN/HTPB propellants with 3 and 7% HTPB had almost the same burning rate at 0.6 MPa, which is less than the burning rate of pure ADN by 3 times at this pressure. Such a behavior can be explained only by assuming that fuel inhibits ADN decomposition. Thus, it is unlikely that the decrease of the burning rate of ADN with the addition of hydrocarbon fuel can be explained solely by heat loss from evaporation and decomposition of the fuel.

Mass peaks 17, 30, 44, and 46 were detected near



the burning surface of  $St_b(1250)$  at 0.1 MPa at a temperature of  $\sim 600$  K. The ratio between the peaks is not given here, but it corresponds to the ratio of these peaks in the mass spectrum of ADN vapor [5]. The comparison of combustion products composition of  $St_b(1250)$  in the dark flame zone with that of pure ADN at 0.6 MPa at a distance from the burning surface  $L_1 \sim 4$  mm (Table 3) shows that the compositions of nitrogen-containing species and the temperatures are close. A similar conclusion was made in the investigation of ADN/HTPB (97/3) propellant [7] and ADN/GAP (82.5/17.5) sandwiches [9]. One can suppose that in a narrow flame zone adjacent to the burning surface of propellant ( $\sim 0.3$  mm at 0.1 MPa), many of the same reactions as in the dark zone of the pure ADN flame at 0.6 MPa occur. The 200 K difference in temperatures is connected to the oxidizer and binder interactions in CP and (or) the reactions between their decomposition products with the formation of CO and  $CO_2$  in the narrow zone near the burning surface.

Experimental results indicate that the CuO catalyst acts in the CP. Usually, the effect of additives on propellant burning rate has a complicated character. Combustion chemistry of ADN-based propellants is close to that of double-based propellants. In both cases, reactions of nitrogen-containing species in the CP and in the gas phase play an important role. According to Ref. [22], in the case of double-based propellants, the accumulation of catalyst on the burning surface takes place. This leads to the additional heat release in CP on the burning surface, and as a consequence, the burning rate increases.

### Conclusions

The comprehensive study of the combustion of the ADN/PCL propellant showed that the burning rate of this propellant is controlled by reactions of both oxidizer and binder. Reactions of ADN decomposition are responsible for heat release in the condensed phase. On the one hand, PCL inhibits reactions of ADN decomposition in condensed phase. This leads to a decrease of the burning rate. On the other hand, reactions between decomposition products of PCL with those of ADN result in intensification of gas-phase processes in flames. CuO catalyst at 4 MPa increases the rate of reactions and heat release in the condensed phase. The mechanism of influence of CuO catalyst on combustion of ADN-based propellants is probably similar to the mechanism of its influence on double-based propellants.

### Nomenclature

A factor in pressure dependence of burning rate  
 C coefficient of solid specific heat

$C_p$  coefficient of specific heat in gas phase at constant pressure  
 L width of the reaction zone in the flame  
 $L_1$  distance from the burning surface  
 m mass burning rate  
 P pressure  
 q heat feedback from gas into solid  
 $q_m$  heat of melting  
 Q heat release in the reaction layer of the condensed phase  
 $r_b$  burning rate  
 T temperature  
 $T_o$  initial propellant temperature  
 $T_s$  burning surface temperature  
 $\lambda$  coefficient of heat conductivity  
 $\nu$  pressure exponent  
 $\varphi$  temperature gradient in gas close to the burning surface  
 $\Phi$  heat release rate in the gas phase

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