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Effect of ADN/GUDN Dual Oxidizers on the Combustion Features of Nitrate Ester Plasticized Polyether Solid Propellants

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Abstract: Several laboratory scale research on nitrate ester plasticized polyether (NEPE) solid propellants with and without ammonium dinitramide (ADN) and *N*-guanylurea-dinitramide (GUDN), featured with the same nominal composition, have been prepared and evaluated. The combustion properties (strand burn rate and flame photos) and thermogravimetry (TG) analysis of propellants with ADN and GUDN have been determined. These parameters are compared to those of reference blank compositions. It turns out that the ADN/GUDN dual oxidizer may greatly affect the combustion behavior of NEPE propellants. The addition of ADN particles to the propellant formulations can increase the burn rate and pressure exponent (n), when a fracture of AP or GUDN was replaced by ADN, the n increases from 0.52 to 0.67 and from 0.58 to 0.67, respectively. By replacing AP flakes with the same fraction of GUDN, the burning rate decrease by 18.97% at 7.0 MPa and n increased by 12.04% (1-15 MPa), in comparison to the reference propellant. The involved NEPE propellants with and without ADN/GUDN dual oxidizers at various pressure ranges show multi-flame structure, and the brightness of flame increases with the pressure increases. Addition of ADN can decrease the thermal decomposition temperature. Moreover, when part of AP was replaced by GUDN, the thermal decomposition procedure is close to that of the reference sample.

Key words: nitrate ester plasticized polyether(NEPE) solid propellant; ammonium dinitramide(ADN); combustion property; thermal decomposition

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1 Introduction

High energy solid propellant formulations play an essential role in propulsion for space exploration[1]. The specific impulse or density specific impulse of propellants can be increased by the inclusion of certain high-energy materials (HEMS), such as 2, 4, 6, 8, 10, 12-hexanitro-2, 4, 6, 8, 10, 12-hexanitrohexaazaisowurtzitane (CL-20, HNIW), ammonium dinitramide (ADN) and others[2-3]. Compared with nitramines, such as cyclotrimethylenetrinitramine (hexogen, RDX) and cyclotetramethylenetetranitramine (octogen, HMX), the main oxidizer ammonium perchlorate (AP), used in composite solid propellant has some toxic issues, especially when considering its good solubility. Further problems are generated by AP-based solid propellants during their combustion. Thus, the development of environmental friendly propellants with low signature characteristics and high energetic properties also has been of great interests to researchers[4-6]. Ammonium dinitramide (ADN) and *N*-guanylurea-dinitramide (GUDN), two novel kinds of high energetic materials with high density, extremely more environmentally friendly and good potential application has been widely studied and used in solid propellants and high explosives[7-10]. It was experimentally found that an advantage of ADN over AP is the clean gas

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production and higher heat of formation[11-15]. Though the development of ADN based solid propellants has faced a number of challenges, the research has continued motivated by the high potential.

Throughout the years, many technical problems have been solved, such as synthesis, stability, compatibility and so on. There are some reports on the synthesis, spectroscopy and thermal behavior of ADN and GUDN[16-21]. Nitrate ester plasticized polyether(NEPE) solid propellant, combining the advantages of double-based propellant and composite propellant, is one of the most important high energetic propellants. The development of the smokeless NEPE propellant to meet the tactical missile engine requirements becomes one of the key research directions. Development of energetic materials with improved combustion properties is underway to meet superior performance requirements, and they appear to be the future candidates to compete with the currently used high energy materials with good performance. The GUDN ADN, HMX and RDX are typical energy compounds of this kind[22-26]. However, no data are available regarding the combustion properties of NEPE solid propellant containing ADN/GUDN dual oxidizers based on these materials herein. Therefore, in our study, the coated ADN and GUDN particles have been added to the formulations and six different propellant compositions were designed and fabricated. The objective of this paper is to compare the effects of ADN and GUDN on the basic properties of NEPE solid propellants to those of the corresponding propellants. This paper places an emphasis on the investigation of the combustion performance of NEPE solid propellants, which could be used for solid rocket motor applications.

2 Experimental

2.1 Materials and Specimen

Ethylene oxide / tetrahydrofuran co-polyether (PET, M_n=4280 g·mol^{-1}, hydroxyl value: 26.4 mg KOH/g), plasticized with nitroglycerin/1,2,4-butanetriol trinitrate (NG/BTTN=1∶1), was cured with polyisocyanate (N-100), and then micro-sized Al powder (≥99.8%), RDX, GUDN and ADN were used as components of the solid rocket propellant. The following are the percentages, by mass fraction of the ingredients, used in six different propellant formulations: (1)NPA-1: AP+ADN/26.5%+0%; (2) NPA-2: AP+ADN/11.5%+15%; (3) NPA-3: AP+ADN/6.5%+20%; (4) NPA-4: AP+ADN/4%+22.5%; (5)NPA-5: GUDN+ADN/26.5%+0%; (6) NPA-6: GUDN+ADN/11.5%+15%. Bimodal AP (105-147 μm and 1-5 μm) is utilized in the propellant formulation. The percentages of RDX, Al and NEPE propellant binder system is the same.

Six different propellant compositions with different mass fraction of oxidizers were prepared. Propellant formulations were mixed in 500 g batches using a 2 L vertical planetary mixer. All the samples involved in this investigation, which were prepared by a slurry cast technique at the temperature of 35 ℃ and then solidified for 120 h (50 ℃) in a water jacketed oven, were machined to a fixed dimension (shape: length:100-150 mm;width:2-5 cm, height: 2-5 cm). Except where otherwise stated, all propellants were manufactured, processed, and tested at Xi'an Modern Chemistry Research Institute under identical conditions and using identical procedures.

2.2 Equipment and Experimentation

2.2.1 Strand Burning Rate Test

Strand burning rate of the propellants were determined by means of fuse-wire technique[17]. The method involved the combustion of strands (ignited by means of a nichrome wire) with dimensions of 150 mm×5 mm×5 mm in a nitrogen pressurized steel bomb. The burning rates were computed from the time that was recorded for the tests conducted at each pressure for each sample.

2.2.2 Combustion Wave Temperature Test

The Π type double tungsten-rhenium thermocouple was used to test the combustion wave distribution of the solid propellant. The thermocouple (diameter=25 μm) was embedded in the propellant sample (diameter=7 mm, length=120 mm) whose profile was coated by polyvinyl alcohol solvent as a flame-retardant and then exposure to air for drying.

2.2.3 Thermal decomposition analysis

Thermal analysis (TG-DTG experiments) of solid propellants, with various oxidizers, was carried out on a TA instrument (made in USA) at a heating rate of 10 ℃·min^{-1} under N_2 atmosphere (sample mass of about 2.0 mg).

3 Results and Discussion

3.1 Effects of ADN on the Combustion Characterizations of NEPE Solid Propellants

3.1.1 Burning Rate and Pressure Exponent

Propellant burning rates determine the rate of gas generation, which determines the pressure inside the motor and the overall thrust. Burning rates herein are obtained experimentally by burning small propellant strands and measuring the surface regression versus time. Various factors like the particle diameter, oxidizing species, pressure, and temperature affect the burning rate of propellants[11-15]. Table 1 lists the pressure exponent of propellants in different pressure ranges. The strand burning rate change data of NEPE solid propellants containing different mass fraction of oxidizer particles and obtained under different pressures are shown in Fig.1.

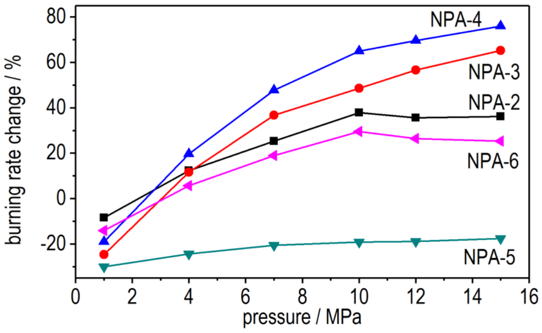

Fig.1 Effect of dual oxidizers on the burn rate of NEPE solid propellant

Table 1 Combustion properties of NEPE solid propellants with different mass fraction of dual oxidizers(initial temperature: T_0=298 K)

sample	strand burning rates / mm·s^{-1}						burning rate pressure exponent					
	1 MPa	4 MPa	7 MPa	10 MPa	12 MPa	15 MPa	1-4 MPa	4-7 MPa	7-10 MPa	10-12 MPa	12-15 MPa	1-15 MPa
NPA-1	2.83	5.89	7.59	9.00	10.18	11.76	0.53	0.45	0.48	0.68	0.65	0.52
NPA-2	2.59	6.61	9.51	12.40	13.81	16.01	0.43	0.65	0.74	0.59	0.66	0.67
NPA-3	2.13	6.58	10.38	13.37	15.94	19.43	0.81	0.82	0.71	0.96	0.89	0.81
NPA-4	2.29	7.05	11.21	14.85	17.27	20.69	0.81	0.83	0.79	0.83	0.81	0.81
NPA-5	1.98	4.45	6.03	7.27	8.26	9.69	0.58	0.54	0.53	0.70	0.72	0.58
NPA-6	2.43	6.22	9.03	11.66	12.87	14.73	0.68	0.67	0.72	0.54	0.61	0.67

From Fig.1 and Table 1, it can be seen that the ADN/GUDN dual oxidizers can affect the combustion behavior and change the burning rate significantly when part of AP or GUDN replaced by ADN particles in the formulations. Compared [NPA-1] with [NPA-5], it can be seen that GUDN can decrease the burning rate of propellant, while the pressure exponent increases a little from 0.52 to 0.58 at 1-15 MPa, which may be attribute to the low enthalpy of formation (-365.79 kJ·mol^{-1}), high active energy 277 kJ·mol^{-1} (200-225 ℃) and higher ignition temperature (192 ℃) than that of ADN (160 ℃). When fracture of AP or GUDN was replaced by ADN the pressure exponent increases from 0.52 to 0.67 and from 0.58 to 0.67, respectively. It indicates that the burning rates and pressure exponent of NEPE solid propellants increase with an increase in the pressure and the mass fraction of ADN in the formulation. As shown in Table 1 sample [NPA-1], designated as the reference composition, by replacing AP flakes with different amount of ADN, burning rate approximately increases by 25.30% [NPA-2], 36.76% [NPA-3], and 47.69% [NPA-4] at 7.0 MPa, respectively, corresponding to 15%, 20% and 22.5% ADN replacement of the AP powder, and the same trend to compare samples [NPA-5] with [NPA-6]. By replacing AP flakes with the same fraction of GUDN (sample [NPA-5]), the burning rate decrease by 18.97%(7.0 MPa) and pressure exponent increase by 12.04% (1-15 MPa), respectively. However, when the pressure exponent of NEPE solid propellant with either ADN or GUDN is larger than 0.50, it should be decreased in future work.

From the energetic viewpoint, the heat release and heat feedback to the combustion surface of propellant for ADN particles are higher than that of GUDN ones, and its promoting effect on the propellant combustion process is the main function in the low-pressure range. From the heat transfer viewpoint, the addition of ADN, with high oxygen balance, to the propellant formulation can increase the heat adsorption effectively during the combustion process[22-23].

3.1.2 Flame photos

In order to understand the effects of ADN particles on the combustion flame structure, images of ADN-based NEPE solid propellant flames structures with different mass fraction of oxidizers at 4 MPa and 7 MPa are shown in Fig.2.

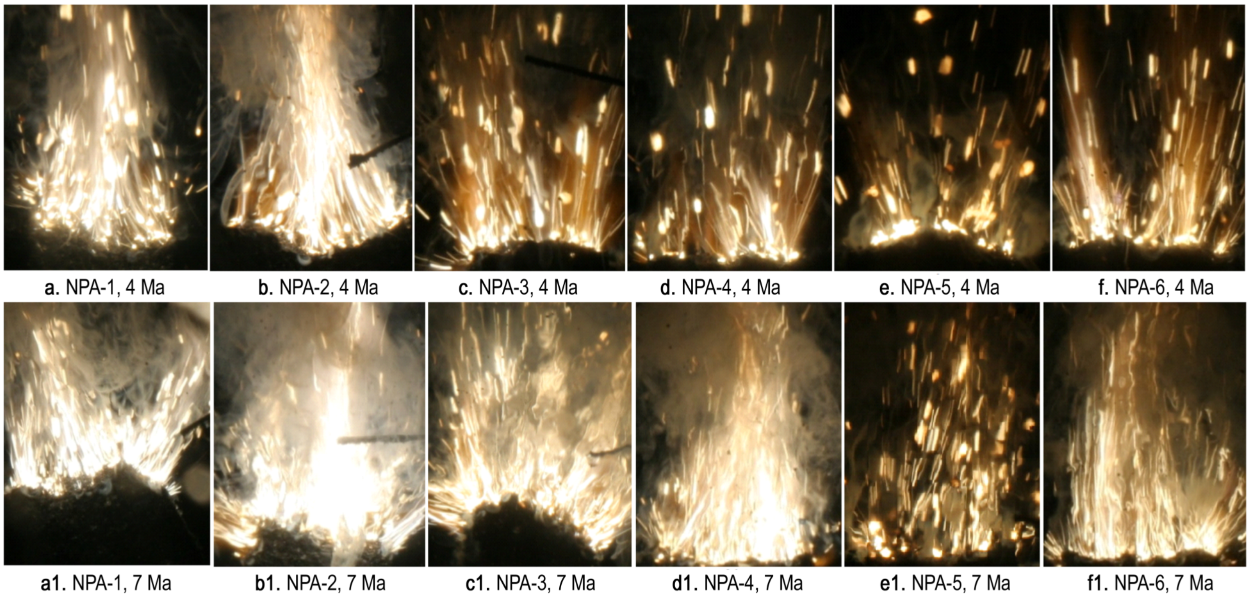

Fig.2 Flame photos of NEPE solid propellants with different mass ratio of dual oxidizers (×500)

From Fig.2, it can be seen that the combustion performance of NEPE solid propellants containing different mass fraction of oxidizers with and without ADN particles present multi - flame structures and the brightness of flame structures is increased in pressure. There are many sparks on the propellant surface during the combustion process, which can be attributed to the addition of the aluminum metal particles to the propellant formulation. It can also be found that the brightness of flame is increased with increase in the mass fraction of ADN particles in the formulations, this phenomenon is accord with the burning rate results mentioned above. Moreover, it was found that the burning rate of ADN is controlled by reactions in the condensed phase and a multi - zone flame structure has been established, which is in agreement with the burning rate results of the propellants. It can also be found that although the metal oxidation process follows a common set of events, aggregation/agglomeration phenomena near the burning surface are noticeably different depending on the enforced operating conditions and details of the solid propellant formulation. The micron-sized aluminum used for the present set of experiments, gives large agglomerate with respect to nano-sized aluminum as it is generally agreed in published literatures[24-26]. It is also pointed out that the agglomeration size varies inversely with the propellants burning rate, consequently, the agglomerate size inverse correspondingly near the burning surface with the oxidizers content and particle size. Furthermore, the accumulating aluminum particles (filigrees or clusters) act as thermal sink of the heat feedback from the gas-phase flame to the propellant surface, contributing to a further change in the burning rate. Amonotonic trend of agglomerated size decrease and the increase of pressure is also observed in this case, which is agreement with that of literature[29-30]. Understanding of these effects opens the path to improved ballistic performance and it should be performed in future work.

3.2 Effects of ADN on the Thermal Decomposition of NEPE Solid Propellants

ADN particles have important effects on the combustion properties of dual-oxidizer propellants. For example, the influence mechanism can be related with the thermal decomposition of propellants. Fig. 3 shows the TG-DTG curves of ADN-based NEPE solid propellants with dual oxidizers.

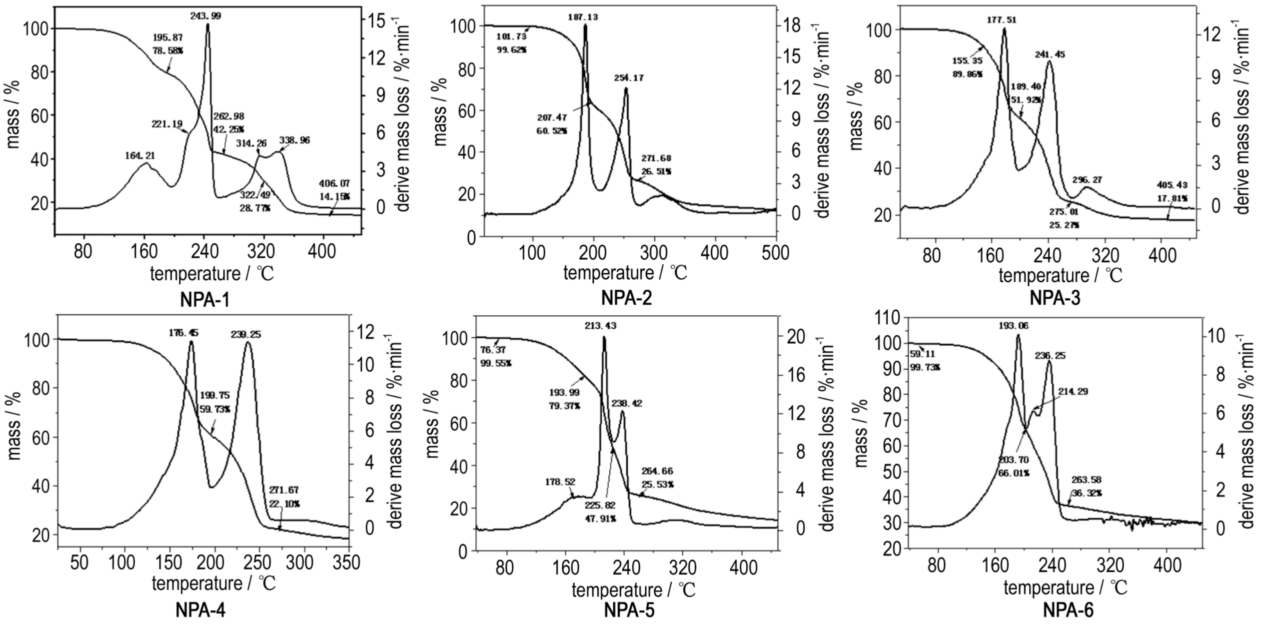

Fig.3 The TG-DTG curves of NEPE solid propellants with different mass fraction of dual oxidizers

From Fig.3 [NPA-1] sample, it was found that the first mass loss stage begins at about 121.1 ℃ and ends at 195.8 ℃, accompanied with about 20.4% mass loss, corresponding to the mass fraction of nitric ester that evaporates and decomposes. The second mass loss stage begins at 196.3 ℃ and ends at 262.9 ℃, accompanied with 35.3% mass loss. This is mainly attributed to the partial decomposition of the RDX, which is in agreement with the mass fraction amounts of RDX in the formulation. The third stage begins at 264.1 ℃ and ends at 359.2 ℃, and is accompanied with a 28.1% mass loss, which is in agreement with the decomposition of part of AP and part of PET binder. In the temperature range of 360.1−450.0 ℃, the mass loss is less than 10.0%, which indicates that there are a few remains at the end of the decomposition. When part of AP were replace by the same mass fraction of ADN in the formulation, showed in Fig.3 [NPA-2]-[NPA-4] samples, there are also three mass changes. The first decomposition peak temperature increased, the second decomposition peak temperature decrease, and the third one become small until disappear with an increase in the mass fraction of ADN particles in the formulation.

It has ever been reported[27-30] that the first decomposition peak temperature moves back and the second decomposition peak temperature moves ahead, when several mass fraction of AP is replaced by ADN. Thus, the temperature difference $\Delta T=T_2-T_1$ (being T_2 is the second peak temperature and T_1 is the first peak temperature) decreases, which can be concluded that adding ADN to the propellant formulation can decrease the thermal decomposition temperature, especially for the first decomposition stage of NEPE propellant. Moreover, the first decomposition peak temperature move back and the second decomposition peak temperature move forward, when the same mass fraction of AP were replaced by ADN particles from the propellants [NPA-1] to [NPA-4], the reason may be that the temperature difference (ΔT) for the second peak temperature minus the first peak temperature decrease, thus it can be concluded that the decomposition of NEPE solid propellants can be catalyzed greatly by the addition of ADN to the propellant formulation. Also, the decomposition process of the propellant containing ADN particles indicates that the gaseous products formed during decomposition exert higher feedback to the deflagrating propellant surface, which can increase the burning rate. Consequently, it is in agreement with the burning rate results.

The thermal decomposition procedure of [NAP-5] propellant is similar with the [NPA-1] propellant, when AP particles were replace by GUDN particles in the formulation, and its thermolysis process can also be divided into three stages (stages Ⅰ-Ⅲ). The fast stage of mass is considered to be the result of the thermolysis of NG and BTTN plasticizers at the temperature range of 1178.5 ℃. The second mass loss stage accompanied with 32.1% mass loss, which is in agreement with the mass fraction amounts of RDX in the formulation. The third mass loss stage accompanied with 22.4% mass loss, which is in agreement with the mass fraction amounts of GUDN in the formulation. However, the first thermal decompositions stage of [NPA-6] propellant is in the 60.2-203.7 ℃, accompanied with 33.7% mass loss, which corresponding to the mass fraction of nitric ester that evaporates and partial decomposition of ADN particles. The second and the third thermal decomposition stages overlap in part, and the mass loss stage begins at 203.7 ℃ and ends at 263.6 ℃, which corresponding to the mass fraction of GUDN and partial decomposition of ADN. In essence there is no visible difference on the last decomposition procedure for the two types of propellants.

4 Conclusions

(1) The preparation of NEPE solid propellants with ADN/GUDN dual oxidizers is feasible, which can be casted under vacuum and cured safely.

(2) The addition of ADN particles to the propellant formulations can increase the burn rates and pressure exponent. When a parts of AP or GUDN were replaced by ADN, the pressure exponent increases from 0.52 to 0.67 and from 0.58 to 0.67, respectively. The addition of GUDN particle could decrease the burning rate, and increase pressure exponent from 0.52 to 0.58 at 1-15 MPa.

(3) The flame photos of NEPE solid propellant with ADN and GUDN at various pressures present multi - flame structures, and the brightness of flame increases with the increase of pressure. There are many sparks on the propellant surface during the combustion of NEPE solid propellants, which attributes to burning aluminum metals in gas phase.

(4) The addition of ADN to NEPE solid propellant formulation can decrease the thermal decomposition temperature. When AP was partially replaced by GUDN, the thermal decomposition procedure becomes similar with that of the reference one.

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ADN/GUDN双氧化剂对NEPE固体推进剂燃烧性能的影响

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摘　要： 设计并制备了含*N*-脒基脲二硝酰胺盐（GUDN）和二硝酰胺铵（ADN）的硝酸酯增塑聚醚（NEPE）固体推进剂样品，测试了推进剂的燃烧性能（燃速和压强指数）、燃烧火焰结构和燃烧波温度分布，并与不含GUDN和ADN的推进剂性能进行对比。结果表明，GUDN/ADN双氧化剂对NEPE推进剂的燃烧性能有明显的影响，推进剂配方中添加ADN可提高推进剂的燃速和压强指数，含15%、20%和22.5%的ADN替换高氯酸铵（AP）可使推进剂在7.0MPa下的燃速提高25.30%、36.76%和47.69%，GUDN使推进剂在7.0 MPa下的燃速降低18.97%，而压强指数在1~15 MPa提高12.04%，而且在不同压力下含双氧化剂的NEPE推进剂的燃烧火焰结构呈多火焰结构，而且火焰的亮度随着压强的增大而变亮。

关键词： 硝酸酯增塑聚醚（NEPE）固体推进剂；二硝酰胺铵（ADN）；燃烧性能；热分析

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