

Improvement in energy release properties of boron-based propellant by oxidant coating



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ABSTRACT

The energy release properties of a propellant can be improved by coating boron (B) particles with oxidants. In the study, B was coated with four different oxidants, namely, NH_4ClO_4 , KNO_3 , LiClO_4 , and cyclotetramethylenetetranitramine (HMX), and the corresponding propellant samples were prepared. First, the structural and morphological analyses of the pretreated B were carried out. Then, the thermal analysis and laser ignition experiments of the propellant samples were carried out. Coating with NH_4ClO_4 showed a better performance than mechanical mixing with the same component. Coating with KNO_3 efficiently improved the ignition characteristics of the samples. Coating with LiClO_4 was the most beneficial in reducing the degree of difficulty of B oxidation. Coating with HMX was the most beneficial in the heat release of the samples. The KNO_3 -coated sample had a very high combustion intensity in the beginning, but then it rapidly became weak. Large amounts of sparks were ejected during the combustion of the LiClO_4 -coated sample. The HMX-coated sample had the longest self-sustaining combustion time (4332 ms) and the highest average combustion temperature (1163.92 °C).

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1. Introduction

Amorphous boron (B) is an ideal fuel for solid-ducted rockets [1] because of its high gravimetric and volumetric calorific values. After the addition of 40% B (in mass), the theoretical specific impulse of an NH_4ClO_4 /hydroxyl-terminated polybutadiene (HTPB)-based propellant can reach 12 kN s kg^{-1} , 5–6 times higher than that of a conventional propellant [2]. However, in a real application, it is difficult to release all the chemical energy from B. This is because B has a high ignition temperature [3], and most of the B particle surface is covered with an oxide film (mainly B_2O_3) that inhibits the contact and reaction between B and environmental oxygen (O) [4]. Therefore, the energy release properties of B and B-based propellants have been extensively studied both theoretically and experimentally [5–9].

The coating of B particles efficiently improves the performance of B-based propellants. The coating of B with different materials reduces the ignition temperature, increases the combustion temperature, removes the surface oxide film, catalyzes the oxidation, and improves the consistency of the propellant system [10–13]. King et al. [10] used LiF to coat B in as early as 1984. Because

LiF was incombustible, its mass content in the propellant was set to approximately 2–4%. The ignition delay time of B was shortened significantly because LiF removed the surface oxide film. Ming et al. [14] studied the effect of glycidyl azide polymer (GAP) coating on the performance of B-based propellant. GAP helped to remove the surface oxide film by generating H_2O during the combustion. Yeh et al. [11,15] coated B with Mg, and the experimental results showed that Mg was reduced in the first stage (the stage where surface-oxide thickness decreases) combustion time of B when its mass fraction was 0.75%.

The combustion of B consumes significant amounts of O. However, the combustion environment in the gas generator of solid-ducted rockets is rich in fuel [16], and the combustion of B suffers from the lack of environmental O. Therefore, an oxidant is an essential component of B-based propellants. Liu et al. [17] studied the combustion characteristics of B-based propellants with different oxidants under N_2 atmosphere. Although hexanitrohexaazaisowurtzitane (HNIW, CL-20) and cyclotetramethylenetetranitramine (HMX) hindered the generation of B_mC_n , they promoted the generation of BN. However, KClO_4 prevented the generation of BN. A separate study by Shuji et al. [18,19] indicated that KNO_3 promoted the ignition of B-based propellants, both in air and vacuum. Zhang et al. [20] reported that even though the ignition characteristics of LiClO_4 were worse than those of NH_4ClO_4 , its O content was higher. Therefore, LiClO_4 was better suited to be

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Table 1
Formulas of B-based propellants.

NO.	B ^{NH₄ClO₄} ₁₀ /%	B ^{KNO₃} ₁₀ /%	B ^{LiClO₄} ₁₀ /%	B ^{HMX} ₁₀ /%	B/%	NH ₄ ClO ₄ /%	HTPB/%
1 (AP)	38.5	–	–	–	–	31.5	30
2 (KN)	–	38.5	–	–	–	31.5	30
3 (LP)	–	–	38.5	–	–	31.5	30
4 (HMX)	–	–	–	38.5	–	31.5	30
5 (Base)	–	–	–	–	35.0	35.0	30

an oxidant in solid propellants with a high O demand. Moreover, Xi et al. reported that Li₂O (an intermediate reaction product of LiClO₄) catalyzed the combustion of B by further reacting with B₂O₃.

The addition of another additive increased the complexity and consistency of B-based propellants [11]. Hence, the coating of B particles using an oxidant has a special advantage. According to the working principle of a solid-ducted rocket [22], an oxidant coating can promote the primary combustion reaction between B and the oxidant. Further, the incomplete combustion products thus generated can react during the secondary combustion and break the oxide film. In this study, four oxidants including NH₄ClO₄, KNO₃, LiClO₄, and HMX were used to coat B and prepare the propellant samples. Moreover, a mechanically mixed control sample was prepared. The structural and morphological analyses of the pretreated B were carried out using Fourier transform infrared spectroscopy (FTIR), X-ray diffraction (XRD), and scanning electron microscopy (SEM). The thermal oxidation characteristics of these five samples were analyzed using thermogravimetric differential scanning calorimetry (TG-DSC). The kinetic analysis of the samples was performed at a high temperature. Moreover, the combustion characteristics of the samples were studied using an experimental laser ignition system. A thermometer and high-speed camera were used together for the investigation of the combustion process.

2. Materials and methods

2.1. Materials

The chemical formulas of the five propellant samples used in this study are listed in Table 1. The superscript of B represents the oxidant coating material (using the recrystallization method [23]). Subscript 10 indicates that the mass ratio of B and the coating material is 10:1. Sample 5 (Base) was not coated with any oxidant. The recrystallization method comprises four steps: (i) preparation of the saturated solution of a coating material, (ii) addition of a certain proportion of B, (iii) ultrasonic dispersion of the mixture for 15 min, and (iv) evaporation of the turbid liquid to dryness at 60 °C. Then, the pretreated B particles were mixed, and other components were added. The propellant samples were solidified at 70 °C for 5 days prior to the experiments.

The B particles used in this study were obtained from Baoding Zhongpuruituo Technology, China, and had a nominal purity of 99% and an average grain diameter of 2 μm. NH₄ClO₄, KNO₃, and LiClO₄ (purity ≥99.5%) were purchased from Aladdin Industrial, China. The HMX sample was prepared by Northwestern Polytechnical University, China, and had a purity of 99.6%. The HTPB was obtained from Zibo YiLiMo Trade, China. The hydroxyl value and viscosity were 0.7676 mmol/g and 2.92 Pa s (40 °C), respectively.

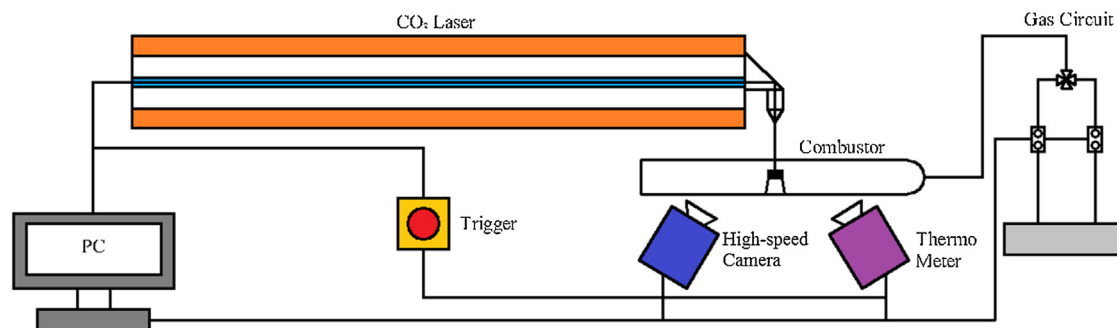
2.2. Devices and methods

The FTIR analyses were carried out using a Nicolet 5700 infrared spectrometer using KBr pellets. A PANalytical X'Pert PRO X-ray diffractometer was used for the XRD analysis. The SEM images were recorded using a Hitachi SU-70 field-emission SEM after gold sputtering for 60 s.

A TA DSCQ1000 atmospheric thermal analysis system was used for the thermal analysis of the samples. Approximately 10 mg of the sample was packed in Al₂O₃ crucible for each TG-DSC experiment. The sample was heated from room temperature to 1000 °C at a rate of 5 °C min⁻¹. The tests were performed in air at a constant gas flow of 120 mL min⁻¹. At least four tests were repeated for each samples to determine the reproducibility.

A laser ignition testing system was developed to study the combustion characteristics of the samples. This system comprised of four parts: a laser ignition module, combustion diagnosis module, gas regulation module, and data acquisition module (Fig. 1). The CO₂ laser heated and ignited the samples in the combustor. A thermometer (Kleiber Infrared GmbH) and a high-speed camera (Redlake GE4900-T12) were used together to detect the combustion of the samples.

In this study, the power of the CO₂ laser was set to 450 W, and the heating time was set to 3 s. Approximately 50 mg of the sample was packed into a quartz crucible for each experiment, and air was used as the reaction gas. The sampling frequency of the high-speed camera was set to 500 Hz, and the measurement period was set to 5 s. The focus of the infrared probe of the thermometer was aimed at the surface of the samples, and the thermometer was turned on simultaneously with the CO₂ laser using a synchronized trigger. The sampling frequency of the thermometer was set to 100 Hz, and the

**Fig. 1.** Schematic of laser ignition testing system.

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