The effect of several factors on energy characteristicsand burning of pyrotechnic compositions based on silic and trilead tetraoxide in some pyrotechnic devices

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ABSTRACT

Pyrotechnic ignition is one of the important parts of the delay timing devices found in fuses and rockets to provide operational reliability according to technical requirements. In this study, the burning rate and some energy characteristics of the pyrotechnic ignition were described. The research results showed that pyrotechnic compositions containing 85.0% Pb_3O_4, 15.0% Si, and 1.0% NC (over 100%) had a high and stable burning rate (109.0 mm/s) and good ignition ability.

Keywords: Pyrotechnic ignition; Pyrotechnic compositions; Burning rate; Energy characteristics.

1. INTRODUCTION

For pyrotechnics that are not reliably ignited from the primers, specialized mixtures are required to ignite from the primary igniters. These mixtures are called ignition devices and are manufactured in granular form, then compressed directly onto the main composition, sometimes also called intermediate booster compositions [1, 3].

Booster compositions are mixtures that are easily ignited from priming compositions and ignite the main compositions with a certain amount of hot solid slag. Through practical studies, it show that booster compositions are often used in IGLA anti-aircraft missiles with gasless delay trains, self-destruct rings and electric differential detonators [4, 7].

In this study, the authors present the results of our investigation into the influence of raw material particle size, and composition ratio of booster compositions based on silicon and lead tetroxide on the burning rate, heat of combustion, gas-generating volume, and ignition temperature.

2. MATERIALS AND METHODS

2.1. Materials

Crystalline silicon (purity ≥ 99.0%) and reddish-brown crystalline lead tetroxide (purity ≥ 98.5%) with particle sizes of 0.06 mm to 0.15 mm. They were purchased from the Chinese Company Xilong. Third-grade nitrocellulose was purchased from the Z Company (Vietnam).

2.2. Methods

2.2.1. Preparation of pyrotechnic samples

The mixture of fuel and oxidizer (lead tetroxide) is pre-mixed and then evenly mixed through sieves with hole sizes of 0.9 mm, 0.05 mm, and 0.01 mm. The mixture is placed into a cup, and then the binder solution (third-grade nitrocellulose dissolved in acetone) is poured in and stirred until the solvent evaporates, forming a homogeneous plastic mass. The amount of solution was used in order to receive 0.5 wt.% nitrocellulose in samples (over 100%). The mass is passed through sieves with holes size of 1.0 mm and then dried naturally for 24 hours. The pyrotechnic material, with particle sizes of 0.4 to 0.9 mm, is separated using suitable sieves. After drying, the sample is stored in a sealed plastic bag for characterization.

2.2.2. Experimental techniques

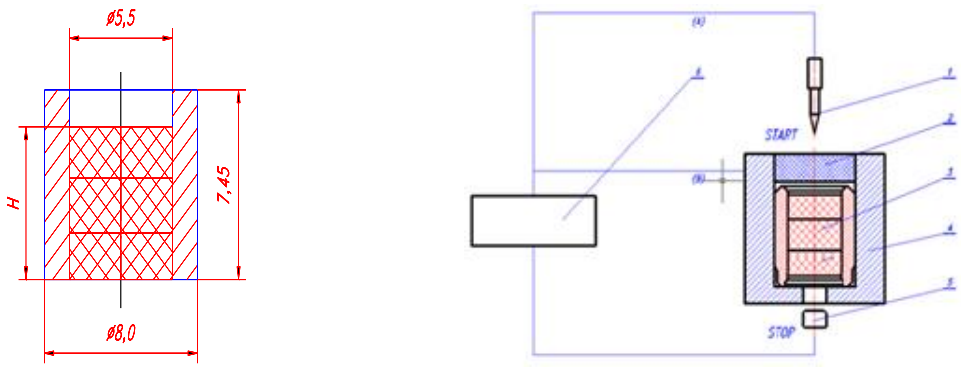
The heat of the combustion was determined by using a Parr-6200 calorimeter with 2 grams of sample weight. Thereafter, the pressure in the calorimeter bomb was measured on a Lutron-9017 manometer, and the volume of gaseous products was calculated as follows:

$$V = \frac{273}{T_c} \cdot \frac{\Delta P \cdot V_b}{1000 \cdot m}$$

Where: V - The volume of gaseous products, ml/gr; ΔP - The difference between values of pressure in the combustion chamber before and after measurement, mbar; T_c - The temperature of the combustion chamber, which equals the room ambient temperature, Kelvin; V_b - The volume of the combustion bomb equals 334 ml.

The ignition temperature was determined using the AET-700 Automatic Explosion Temperature Tester according to STANAG 4491. The sensitivity to thermal pulses of the pyrotechnic is also assessed by the success rate of igniting the compressed composition in a copper tube using a spark from a slow-burning fuse (a total of 10 sample tubes were tested).

The burning rate was obtained as the ratio between the distance traveled by the combustion front and the corresponding time interval, which was determined using the burning time measuring device MS02-99 with a measuring range of 1.0 µs to 9999.9 s. A certain amount of the pyrotechnic composition was pressed into a copper tube, with the height of the sample being measured. The sample was ignited by a cap MG-8 (see figure 1).



a) *(b)*

Figure 1. *Setup of the experiment for determination of burning rate:*
a) A tube of pressed pyrotechnic composition;
b) Schematic diagram: 1- Fire needle; 2 - MG-8 cap; 3 – Tube of pyrotechnic composition;
4 - Case; 5 - Light sensor; 6 - Time measuring device.

2.2.3. Computational techniques

The authors used Real software to calculate several energetic characteristics [5].

3. RESULTS AND DISCUSSION

3.1. Influence of oxidizer-fuel content on several energetic characteristics

The authors determined the combustion heat and specific gas volume of samples in different oxidizer-fuel contents (table 1).

Results in table 1 show that the oxygen balance of the mixture approaches zero as the Pb_3O_4 is closed at about 97%. The combustion heat reaches the highest value of 363.5 cal/gr) at the silicon content of 10% (the reaction occurs almost completely). The heat of combustion decreases with rising silicon content because the reaction occurs more completely and the amount of solid products increases. Meanwhile, the specific volume is almost unchanged and is about 18 to 19 ml/gr. Gas products are mainly produced due to the decomposition of nitrocellulose. The main products are mostly solids (Pb, SiO_2, Si), which meet the requirements for primer composition. Their content increases with rising the silicon content and reaches the maximum at silicon content of 25%. Thus, the pyrotechnic composition based on Si and Pb_3O_4 is a type that produces little gas, ensuring use in a closed system. Compared with theoretical calculations (4.6 ml/gr), the volume of gas produced in the experiment is much larger. This is because the calculation conditions are a vacuum environment, while in reality, the measuring device cannot suck all the air out of the measuring bomb.

Table 1. *Influence of oxidizer-fuel content on energetic characteristics of pyrotechnics based on silicon and lead tetroxide.*

No.	Contents,%		K_b, %	Q_v, cal/gr		V_0 ,ml/gr		Solid products, moles/kgr		
	Si	Pb_3O_4		*Theor.*	*Expe.*	*Theor.*	*Expe.*	Pb	SiO_2	Si
1	10	90	-3.8	380.3	363.5	4.6	18,2	3.99	2.66	1.05
2	15	85	-9.9	370.1	361.3	4.6	18,2	3.77	2.52	2.97
3	20	80	-16.0	358.2	352.6	4.6	19,0	3.55	2.37	4.88
4	25	75	-22.2	345.9	336.5	4.6	19,2	3.33	2.22	6.80

3.2. Influence of oxidizer-fuel content on thermal sensitivity

Sensitivity to thermal pulses is an important characteristic used to assess the reliability of ignition from primary igniters and the safety of use [1, 6]. In this study, the sensitivity of the pyrotechnic composition to thermal pulses was determined by the ignition temperature and its ability to ignite when exposed to a spark from a slow-burning fuse (table 2).

The results presented in table 2 indicate that the pyrotechnic composition has a relatively low ignition temperature (below 500 °C) compared to similar booster compositions such as Mg/BaO_2 with a ratio of 12/88 (570 °C) and $B/BaCrO_4$ with a ratio of 5/95 (700 °C) [6]. The higher the silicon content, the higher the ignition temperature. When tested for sensitivity to the spark of a slow-burning fuse, a sample with 25% silicon content had only 9 out of 10 tubes successfully ignited. Therefore, the silicon content in this pyrotechnic composition should not exceed 20%.

Table 2. *Influence of oxidizer-fuel content on sensitivities to heat.*

No.	Contents,%		T_{ig}, °C	*Spark sensitivity*
	Si	Pb_3O_4		
1	10	90	482	10/10
2	15	85	485	10/10
3	20	80	487	10/10
4	25	75	494	9/10

3.3. Study of the influence of some factors on the burning rate

3.3.1. Influence of oxidizer-fuel content

The ratio of fuel to oxidizer is the most important factor affecting the burning rate. The sample, with a specified mass, is divided into three portions, and each is sequentially compressed into copper tubes at a pressure of 2000 kg/cm². The height of the column of the pyrotechnic in the tube is approximately 3.5 mm. The experimental results are displayed on table 3.

From table 3, it indicates that as the fuel content increases, the burning rate rises due to the increased combustion heat. According to the requirements, the primer composition must have a high burning rate. However, to ensure stable combustion, the mechanical strength must be sufficiently high. Mechanical strength is reflected by the compression ratio. According to references [1, 2], the compression ratio should be between 0.7 and 0.9. Thus, a sample with 10% silicon content (compression ratio of 0.68) does not meet the requirement. Among the two samples with silicon contents of 15% and 20%, the first sample, with higher combustion heat and burning rate, is preferred for further research.

Table 3. *Influence of oxidizer-fuel content on burning rate.*

No.	Contents,%		ρ, g/cm^3	Compression ratio	*u*, mm/s
	Si	Pb_3O_4			
1	10	90	4.70	0.68	112.1
2	15	85	4.63	0.75	109.0
3	20	80	4.10	0.73	93.9

3.3.2. Effect of particle size of silicon

To study the effect of silicon particle size on the burning rate, the authors used silicon with four different sieve sizes of 100#/cm, 70#/cm, 58#/cm, and 38#/cm to prepare samples (with 15% silicon content). The oxidizer Pb_3O_4 had a sieve size of 70#/cm. The burning rates are presented in table 4.

Table 4. *Effect of particle size of silicon on the burning rate.*

No.	Particle size, #/cm	*ρ*, g/cm^3	Compression ratio	*u*, mm/s
1	100	4.49	0.69	115.8
2	70	4.63	0.75	109.1
3	58	4.67	0.76	79.7
4	38	4.75	0.78	56.9

From table 4, it can be seen that as the silicon particle size increases, the burning rate decreases. This is explained by the fact that smaller particle sizes provide a larger surface area for contact between the fuel and oxidizer, leading to faster chemical reactions. Silicon with a sieve size of 100#/cm is difficult to process and generates dust during production. In contrast, silicon with sieve sizes of 58#/cm and 38#/cm results in a lower burning rate. Therefore, only silicon with a sieve size of 70#/cm is suitable for further research.

3.3.3. Effect of compression pressure

To study the effect of compression pressure on the burning rate, the author prepared samples with a Si/Pb_3O_4 ratio of 15/85 (silicon with a particle size of 70#/cm). The samples were compressed at 5 different pressures. After compression, the burning rate was measured and the packing density ratio was calculated. The results are presented in table 5.

Table 5. *The dependence of the burning rate on compression pressure.*

No.	*P*, kG/cm^2	*ρ*, g/cm^3	Compression ratio	*u*, mm/s
1	1400	3.76	0.61	124.1
2	1700	4.07	0.67	116.3
3	2000	4.63	0.75	109.0
4	2300	4.69	0.77	98.2
5	2600	4.71	0.77	96.4

From table 5, it can be seen that an increase in compression pressure leads to a decrease in the burning rate. This can be explained by the fact that increased compression pressure raises the density, making it more difficult for gas products to penetrate the unburned pyrotechnic layers, which slows down the heating and ignition process. With a compression pressure smaller than

2000 kG/cm², the compression ratio is less than 0.7, which does not meet the requirements for stable burning. Conversely, if the compression pressure is too high, the material becomes difficult to ignite (due to a smooth surface) and there is a risk of explosion. Therefore, the optimal compression pressure value is P = 2000 kG/cm².

4. CONCLUSIONS

Theoretical and experimental research results show that the optimal content for the primer composition based on silicon and lead tetroxide is Si/Pb_3O_4 = 15/85, with the silicon particle size of 70#/cm, and compressed at a pressure of 2000 kG/cm². This composition ensures a stable burning rate and energy characteristics, as well as mechanical strength. This pyrotechnic mixture can be effectively produced to meet defense requirements.

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TÓM TẮT

Nghiên cứu ảnh hưởng của một số yếu tố đến đặc trưng năng lượng cháy của thuốc hỏa thuật trên cơ sở silic và chì tetra oxit

Thuốc hỏa thuật mồi cháy được sử dụng trong chi tiết mồi cháy của ngòi đạn, tên lửa. Nghiên cứu này trình bày ảnh hưởng của một số yếu tố như thành phần, cỡ hạt, áp suất nén tới tốc độ cháy, một số đặc trưng năng lượng và độ nhạy với xung nhiệt của loại thuốc hỏa thuật. Kết quả nghiên cứu cho thấy, thuốc hỏa thuật có chứa 85,0% Pb_3O_4, 15,0% Si (lọt sàng 70 #/cm) và 1,0% NC (tính ngoài) được nén với áp suất 2000 kG/cm^2 có tốc độ cháy cao và ổn định là 109,0 mm/s.

Từ khóa: Thuốc mồi cháy; Tốc độ cháy; Đặc trưng năng lượng.