

文章编号: 1006-9941 (2017)05-0384-07

Preparation and Characterization of Metalized Explosive Containing B and Al Powder

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Abstract: Octagon (HMX) based metalized explosive containing boron (B)/aluminum (Al) compound powder, oxidizer ammonium perchlorate (AP) and binder hydroxyl-terminated polybutadiene (HTPB) was designed and prepared. The appearance morphology of B powder, Al powder and B-Al compound powder were observed by SEM. The effects of HMX and AP on the thermal oxidation characteristics of B and Al powder were investigated by TG-DSC. A deeper understanding of reaction kinetic mechanism of the B and Al powder was made. The sensitivities (impact sensitivity, friction sensitivity, electrostatic spark sensitivity, cap initiation sensitivity) and ignition and propagation properties for metalized containing B/Al were measured, which made a fully acknowledge of the safety under different external energy stimuli and ability of detonation propagation. The results show that there are many small particles of B powder on the surface of spherical Al powder in the B-Al compound powder. From room temperature (25 °C) to 1000 °C under N₂ atmosphere, although the pressures have an effect on the thermal decomposition peak temperature of HMX and AP, however partial oxidation of Al powder and B powder only occurs, whereas the combustion does not occur. The impact sensitivity, friction sensitivity and electrostatic spark sensitivity of the metalized explosives containing B and Al powder are 40%–80%, 100% and 3.83–6.40 kV respectively, and all the metalized explosives designed can be initiated by 8[#] industrial detonator, which indicates that the mechanical sensitivity of the metalized explosive containing B and Al powder without binder is high. The mechanical sensitivity is obviously lowered by adding desensitized HMX and AP, and it can be further lowered by adding polyurethane binder. By increasing the 20% content of polyurethane binder HTPB, the impact sensitivity and friction sensitivity of the HMX based metalized explosive containing B powder can be reduced less than 10% and 30%, respectively, revealing that the safety requirements for preparation and processing technology of mixed explosive can be satisfied. Additionally, the metalized explosive of ϕ 50 mm can be initiated by 8[#] industrial detonator and can propagate stable detonation, showing a strong after-effect power ability.

Key words: metalized explosive; preparation; safety; initiation and detonation propagation properties; power ability

CLC number: TJ55; O389

Document code: A

DOI: 10.11943/j.issn.1006-9941.2017.05.006

1 Introduction

By adding active metal powder, the power ability and explosion power of energetic materials can be improved quickly and effectively^[1-2]. Al and B are used in propellants and explosives due to their high oxidation enthalpies, high combustion temperatures, and low molecular weight products.

Commonly, Al is widely recognized as one of the most ideal and suitable active metals with advantages of high oxidation heat, low price and wide source. Specially, the density and combustion heat of Al are 2.7 g · cm⁻³ and 31 kJ · g⁻¹ in the view of thermodynamics respectively, while the density and combustion heat of B are 2.34 g · cm⁻³ and 59 kJ · g⁻¹

respectively^[3]. Considering that the combustion heat of B is almost two times greater than that of Al, many scientists pay close attention to B. However, the melting point and boiling point of B are higher than that of Al, and the liquid B₂O₃ is formed in the early stage of combustion, the oxidation reaction of solid-liquid interfaces is obstructed, which result in that the ignition and combustion characteristics of B powder are poor, so that it is difficult to burn and release the high combustion heat completely. The ignition and combustion characteristics of the amorphous B powder were improved by using technologies of the oxidant AP coating B powder surface and the B-Al composite powder. Under the loading of detonation products of high-energy explosive, the higher combustion heat of the B-Al composite powder was released which have practical value.

B powder has been widely used in various kinds of propellants^[4-8]. In recent years, there are some literatures published about metalized explosive containing B and Al powder. Underwater energy of DNTF based B-contained and Al-contained explosive was studied theoretically and experimentally, which indicated that the energy of B-contained explosive was better than that of Al-contained explosive^[9-10]. Besides, better energy characteristics could be obtained if B and Al were used

Received Date: 2016-10-30; **Revised Date:** 2016-11-27

Project Supported: Supported by National Natural Science Foundation of China (Grant No. 11572359, 11502249) and Science and Technology Fund of CAEP (2015B0101012)

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together. Pei et al.^[11] studied the explosion performance of thermobaric fuel containing B, and the shock overpressure of the fuel containing B powder had no apparent advantage compared to the samples containing Al powder in the same measure point. While compared to the fuel containing Al powder, the fireball temperature of the fuel containing B sustained a long time. Recently, the thermal behaviors of various metalized RDX-based explosives with B powder were investigated by a thermogravimetric analyzer (TGA) and a high pressure differential scanning calorimeter (PDSC) technology, indicating that the RDX decomposition and its products could active B powder, and then made its oxidation or nitrogenation easy at high temperature^[12]. Xu et al.^[13-14] studied the combustion heat of the Al/B powder and its application in metallized explosives in underwater explosions. As the content of B powders was increased, the heat of combustion of the metal mixtures increased, and the combustion efficiency of B decreased.

As noted above, the addition of B and Al powders to explosives is a good way to enhance their blast effect, to improve the temperature of the explosion field and to prolong the duration of the higher temperature. However, the application of B in explosive is still at the exploratory stage at home and abroad. Thus, careful measurements are still needed to address specifically the mechanical sensitivity of metalized explosive containing B and Al powder to ensure the safety of preparation and characterization. In this work, a new HMX based metalized explosive containing B and Al powder, oxidizer AP, polyurethane binder was designed and prepared. The thermal properties, mechanical sensitivity, electrostatic spark sensitivity, cap initiation sensitivity, and ignition and propagation properties of the new metalized explosive were measured, which lay the foundation for the subsequent development of detonation reaction zone structure and power ability.

2 Experimental

2.1 Preparation

2.1.1 Raw Materials

HMX; the content was 99.9%, and the particle size D_{50} are 6.7 μm , analytical reagent, and the particle sizes were $\phi 20-50 \mu\text{m}$, Dalian potassium chlorate plant production. Al powder; the content was 99%, analytical reagent, and the particle sizes were $\phi 1-5 \mu\text{m}$, Liaoning Angang Industrial Fine Aluminum Powder Co. Ltd. production. B powder; the content was 99.9%, analytical reagent, and the particle sizes were $\phi 1-5 \mu\text{m}$, Hebei Baoding Pengda New Material Technology Co. Ltd. production. HTPB; molecular mass average was 2000, hydroxyl value was 0.76 mmol/g, Luoyang Liming

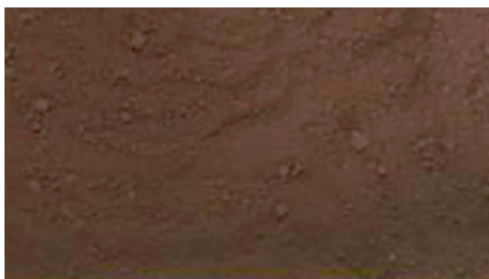
Chemical Academy production.

2.1.2 Powder Mechanical Mixing Method

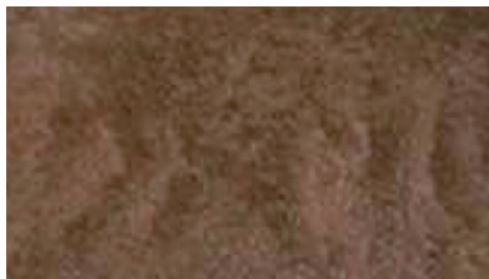
According to the designed formulation, nine kinds of pulverous metalized explosives listed in Table 1 and Fig. 1 consisted of HMX and AP or desensitized HMX and AP, metal powder were prepared. The photographs of three kinds of pulverous metalized explosives in Fig. 1 were taken with a Canon camera to observe



a. JS-1 (containing Al powder)



b. JS-3 (containing B powder)



c. JS-2-1 (containing B and Al powder)

Fig. 1 Photographs of three kinds of pulverous metalized explosives

Table 1 Formulation of pulverous metalized explosive

sample	mass ratio/%				note
	HMX+AP	HMX+AP (desensitize)	Al	B	
JS-1	75	—	25	—	
JS-2-1	75	—	12.5	12.5	composite powder 1 [#]
JS-2-2	75	—	12.5	12.5	composite powder 2 [#]
JS-3	75	—	—	25	
JS-4	—	75	25	—	
JS-5-1	—	75	12.5	12.5	composite powder 1 [#]
JS-5-2	—	75	12.5	12.5	composite powder 2 [#]
JS-6	—	75	—	25	
JS-7	—	75	15	10	

Note: The desensitized HMX and desensitized AP were coated with 2% wax.

the appearance of metalized explosives. Two kinds of B and Al compound powder used in this study were compound powder 1[#] and compound powder 2[#] that both had the mass ratio of B and Al as 1 : 1. The difference was that the compound powder 1[#] was made by simple physical mixing method while the compound powder 2[#] was made by mechanical attrition method.

2.1.3 Cast PBX's Method

Through the processes of batching, kneading, vacuum casting and curing, four kinds of metalized explosives consisted of desensitized HMX or RDX, desensitized AP, B and Al compound powder, HTPB and TDI were prepared. The metalized explosive shown in Fig. 2 consisted of HMX and AP (60%), B powder (12%), Al powder (8%), and other additives including the HTPB binder (20%). The components of the three other metalized explosives were shown in Table 2.

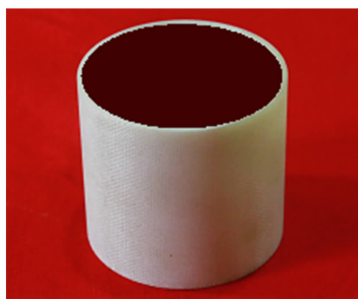


Fig. 2 Photograph of metalized explosive cylinder

Table 2 Formulation of cast metalized explosive

sample	mass ratio / %				
	RDX+AP (desensitize)	HMX+AP (desensitize)	B	Al	others
GR-1	64	–	25	–	11
GR-2	64	–	12.5	12.5	11
GH-1	–	60	8	12	20
GH-2	–	60	4	16	20

2.2 Characterization

2.2.1 SEM Analysis

Scanning electron microscope (SEM) observations were carried out on Apollo300 SEM (Camscan Electron Optics, UK). When the voltage was 30 kV, the resolution was 1.2 nm. When the voltage was 1 kV, the resolution was 2.0 nm. The maximum magnification of the sample was 100 thousands. During the observation of appearance of micron metal powder samples, the magnifications were 0.5–20 thousands.

2.2.2 Thermal Analysis

The DSC-TG experiments were performed by STA 449C DSC-TG and DSC 204 thermal analyzer (NETZSCH Co. German). The sample masses were (2±0.50) mg, heating rate

was 10 K · min⁻¹, the experimental temperatures were 20–1000 °C, the working atmosphere was nitrogen, the flow rate was 20 mL · min⁻¹, and the pressure DSC was operated at 1 MPa.

2.2.3 Measurement of Sensitivity Properties

The mechanical sensitivity of metalized explosive was measured according to specifications of standardization methods GJB 772A–1997 601.1 (Impact sensitivity explosive probability method) and 602.1 (Friction sensitivity explosive probability method)^[15]. The electrostatic spark sensitivity was determined by a model JGY-50 instrument. The sample masses were (20±2) mg, the 50% firing voltage and 50% firing energy were calculated with the up-down method. In reference to the Chemical Physical Hazard Test Guide in the “test of cap initiation sensitivity 5(a)”, the cap initiation sensitivity of sample was measured^[16].

2.2.4 Plate Dent Test

The fiber tube of $\phi 50$ mm×50 mm and 2 mm thick was filled with metalized explosive. In the plate dent test, the booster explosive was TNT cylinder of $\phi 50$ mm×50 mm, the initiating system were PETN tablet and 8[#] industrial detonator, the identification plate was Q₂₃₅ steel cylinder of $\phi 100$ mm×40 mm. The detonation pressure was calculated through the dent depth of identification plate, and power ability of the metalized explosive were relatively evaluated^[17–18].

3 Results and Discussions

3.1 Appearance of B and Al Compound Powder

The micro morphology of Al powder, B powder and two kinds of B and Al compound powders were observed by SEM. The SEM images at various scales are shown in Fig. 3.

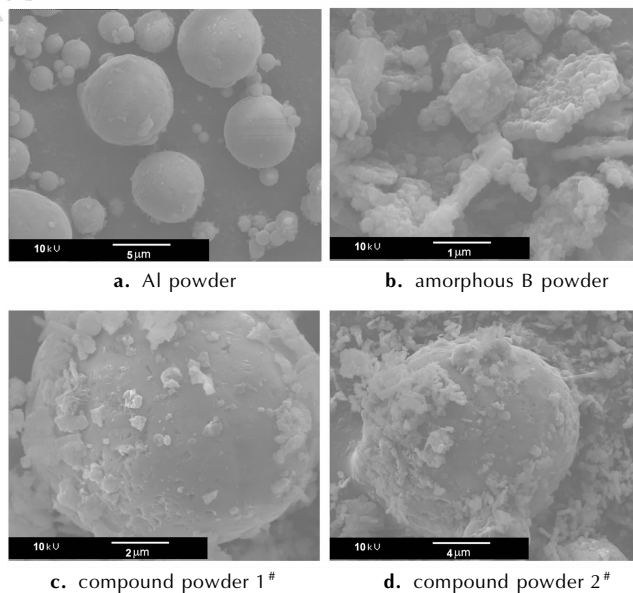


Fig. 3 SEM images of metal powder

The Al powder is 1–5 μm spherical particles, and partial particles have agglomeration, as shown in Fig. 3. The structure of B powder is amorphous flake crystallite shape whose sizes are between 1–5 μm . For the B-Al compound powder, there are many little amorphous flake B powder on the surface of Al powder.

3.2 Thermal Properties

The TG-DSC curves at atmospheric pressure and DSC curves at atmospheric pressure or 1 MPa pressure of powdery AP, HMX, HMX+Al, and HMX+Al+B obtained at a heating rate of $10 \text{ K} \cdot \text{min}^{-1}$ are shown in Fig. 4.

It can be seen from Fig. 4 that the endothermic peak of AP sample at about 245°C is a crystalline transformation peak. The thermal decomposition of large AP particles ($\Phi > 200 \mu\text{m}$) is different from that of small AP particles and there are two

decomposition peaks for large AP particles^[19]. In this paper, the AP sample is small particles ($\Phi = 20\text{--}50 \mu\text{m}$), so there is only one decomposition peak at temperature 410.4°C . The peak is wide and gentle at atmospheric pressure, while the exothermic peak is narrow and sharp and the released heat is also increased at 1 MPa pressure. The decomposition peak of HMX sample at 1 MPa pressure is slightly decreased and becomes very sharp. By adding Al powder in HMX, the crystalline transformation peak, the exothermic decomposition peak of HMX and the endothermic melting peak of Al powder are all slightly decreased. Under the conditions of room temperature to 1000°C and N_2 atmosphere, although the pressure have effects on thermal decomposition peak temperature of HMX and AP, Al powder and amorphous B powder occur to partial oxidation but no combustion.

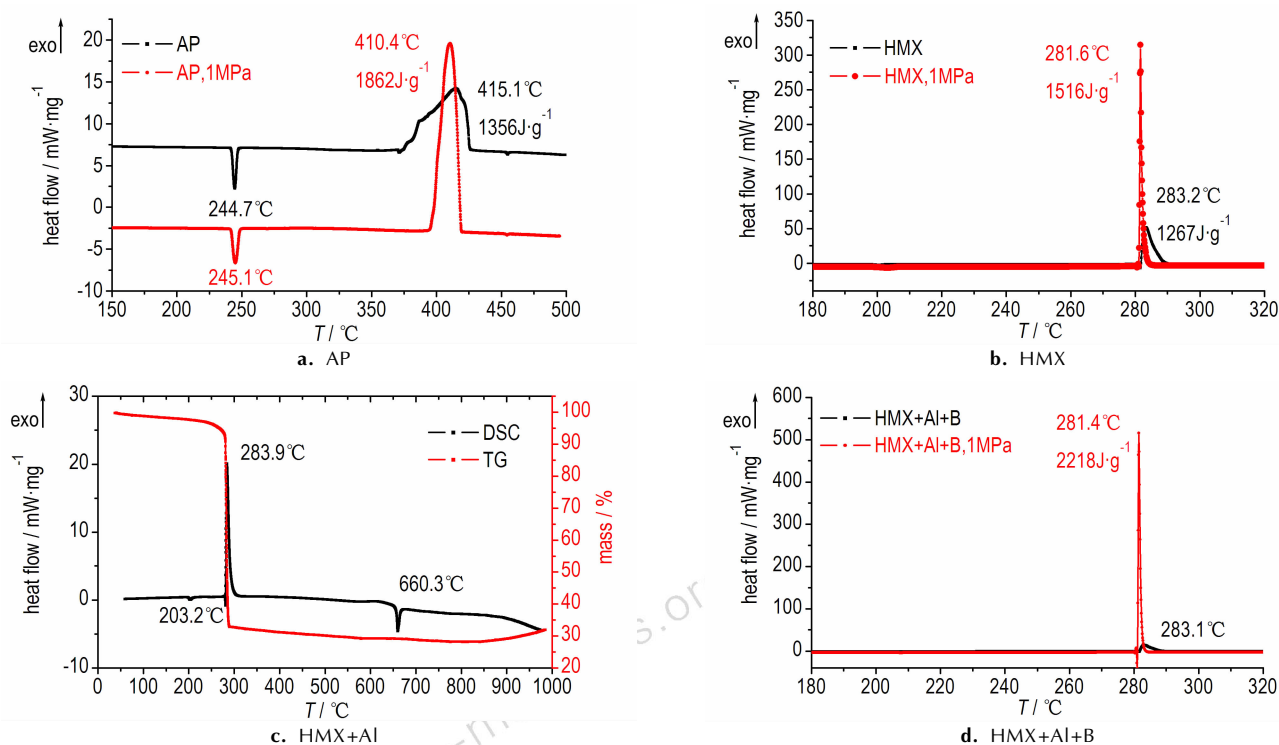


Fig. 4 DSC and TG-DSC curves of AP, HMX, HMX+Al, and HMX+Al+B

3.3 Safety

The safety of the metalized explosive containing B and Al powder was evaluated by sensitivity test, and the safety risk assessment for preparation and processing of kilogram grade sample was also evaluated. The mechanical sensitivity, electrostatic spark sensitivity and cap initiation sensitivity of six kinds of powdery metalize explosive are shown in Table 3.

The impact sensitivity, friction sensitivity and electrostatic spark sensitivity of the metalized explosives containing B and Al powder are 60%–80%, 100% and 3.83–6.40 kV, respectively, and the metalized explosive has cap initiation sensitivi-

ty, as shown in Table 3. The results show that the sensitivity of the metalized explosive containing B and Al powder is higher without binder, therefore the safety in the preparation and characterization test should be paid attention to. When the metalized explosive is impacted by drop hammer, it is often accompanied by a large noise and flash, due to the reaction energy release of burning metalized powder if explosion happens. Specially, for metalized explosive containing B and Al powder, the noise and flash are greater, the T10A steel sleeve is exploded into two parts, and the drop hammer is pushed up by the counterforce. The results indicate that the metalized ex-

plosive has strong after-effect power ability. When the metalized explosive is acted by friction hammer, it is often accompanied by a large noise and flash if explosion happens, which is only observed in the friction sensitivity test of CL-20 and BTF explosive^[20-22].

Table 3 Sensitivities of pulverous metalized explosive

sample	<i>I</i> / %	<i>F</i> / %	<i>E</i>		<i>C</i>
			V_{50} / kV	E_{50} / J	
JS-1	80	100	3.83	0.220	+
JS-2-1	70	100	5.17	0.401	+
JS-3	60	100	6.40	0.614	+
JS-4	70	100	10.40	1.62	+
JS-5-1	60	100	12.60	2.38	+
JS-6	40	100	17.10	4.39	+
HMX ^[18]	80-100	100	2.552	0.0994	+
AP ^[20]	32	28			+

Note: The impact sensitivity, friction sensitivity, electrostatic spark sensitivity and cap initiation sensitivity are expressed by symbols *I*, *F*, *E* and *C*, respectively.

Based on RDX or HMX, adding oxidizer AP, B-Al compound powder and binder HTPB, four kinds of metalized explosive containing B and Al powder were designed and prepared. Before curing, samples of 40 mg metalized explosive into the impact or friction devices, then pressed into tablet and put into the oven to curing. Afterwards, the mechanical sensitivity of metalized explosive was measured, the results are listed in Table 4.

Table 4 Mechanical sensitivities of cast metalized explosive %

sample	<i>I</i>	<i>F</i>
GR-1	88	100
GR-2	60	100
GH-1	0	24
GH-2	8	20

It is shown that the impact sensitivity of the GR-1 formulation of RDX based explosive containing B powder is 88%, the friction sensitivity is 100%, as shown in Table 4, therefore the mechanical sensitivity of this metalized explosive is very high. In the case of other components unchanged, by replacing the B powder into B and Al compound powder, the impact sensitivity is decreased to 60%, and the safety requirements of preparation and processing of mixed explosive still can't be satisfied. Increasing the binder content of HTPB, the impact sensitivity of the GH-1 and GH-2 formulation of HMX based explosive containing B powder is less than 10%, the friction sensitivity is less than 30%, so that the safety requirements for the preparation and processing technology of mixed explosive are satisfied.

3.4 Ignition and propagation properties

The plate dent test of nine kinds of metalized explosives, powdery HMX and AP, TNT and cast metalized explosive cylinder of $\phi 50$ mm were done. Device of plate dent test are shown in Fig. 5, photographs of some identification plate are shown in Fig. 6, and results of plate dent test for metalized explosive are shown in Table 5.

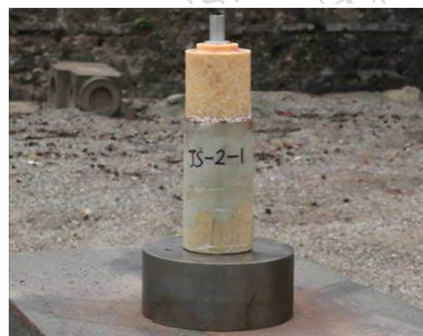


Fig. 5 Device of plate dent test

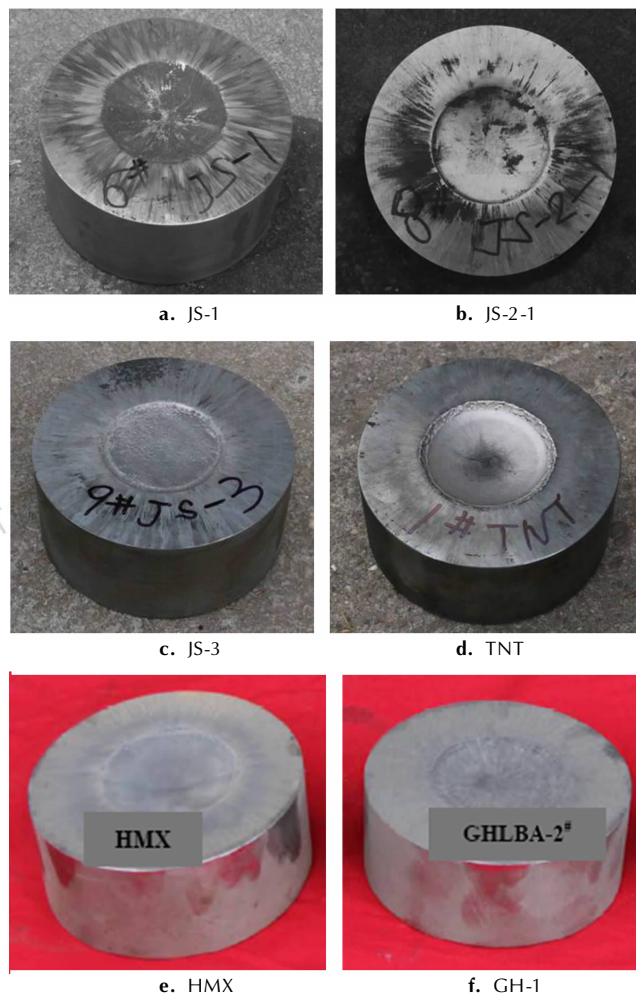


Fig. 6 Photographs of some identification plate

It is shown that the identification plate has an obvious dent, which indicates that the design and preparation of metal-

ized explosives have better ignition and propagation properties, as shown in Fig. 6 and Table 5. By comparison, it is found that the greater the density of explosive, the deeper of dent is. When the quality of metal powder Al : B is 1 : 1, the dent is not the deepest. The detonation pressure of TNT, HMX and AP at different densities is calculated by using VLW code, and the linear relationship between the dent depth and the detonation pressure is shown in Fig. 7. The linear equation is $p=10.0714+1.2591 h$ (1) where, h is the dent depth of identification plate, mm; p is the detonation pressure, GPa.

Table 5 Results of plate dent test for metalized explosive

sample	$\rho/g \cdot \text{cm}^{-3}$	h/mm	p/GPa
JS-1	1.334	4.655	15.93
JS-2-1	1.202	3.363	14.31
JS-2-2	1.151	2.925	13.75
JS-3	0.927	1.747	12.27
JS-4	1.202	4.299	15.48
JS-5-1	1.059	3.451	14.42
JS-5-2	1.008	2.774	13.56
JS-6	0.957	1.776	12.31
JS-7	1.171	2.093	12.71
GH-1	1.508	4.560	15.81
TNT	1.620	7.829	19.87
HMX	1.283	5.423	17.00
HMX	1.147	3.039	13.88
AP	1.080	1.305	11.69

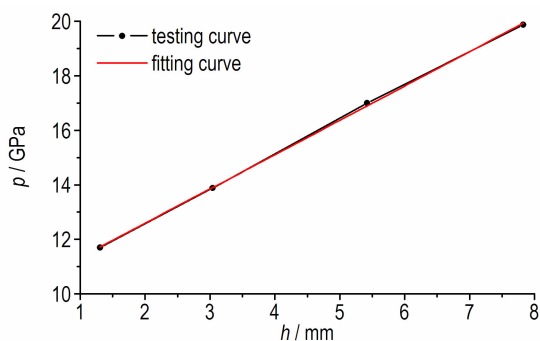


Fig. 7 Curves of relationship between dent depth and detonation pressure

In addition, the cap initiation sensitivity and detonation ability test of metalized explosive were carried out. About 700 g JS-2-1 metalized explosive was put into the fiber tube of 80 mm diameter. The charge density is $0.9415 \text{ g} \cdot \text{cm}^{-3}$. The identification plate above steel rim is Q_{235} steel plate of 1 mm thick while the identification plate below steel rim is Q_{235} steel plate of 8 mm thick, as shown in Fig. 8a. The test results show that metalized explosive has cap initiation sensitivity, and the Q_{235} steel plate of 1 mm thick is exploded into pieces, and the Q_{235} steel plate of 8 mm thick is exploded with a large hole of more than 80 mm diameter, as shown in Fig. 8b, indicating

that the metalized explosive has great after-effect power ability.

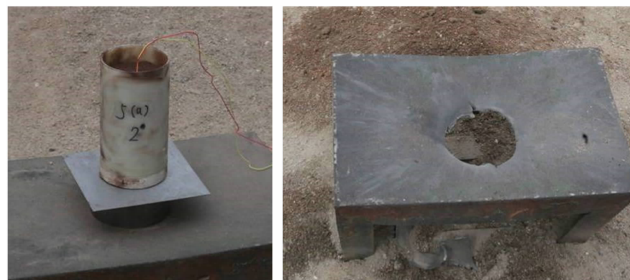


Fig. 8 Cap sensitivity test of metalized explosive

4 Conclusion

(1) For investigation of the thermal properties, mechanical sensitivity, electrostatic spark sensitivity, cap initiation sensitivity, ignition and propagation properties of metalized explosives, the HMX based metalized explosives containing B and Al are designed and prepared by powder mechanical mixing method and cast PBX's method.

(2) From room temperature ($25 \text{ }^\circ\text{C}$) to $1000 \text{ }^\circ\text{C}$ and under N_2 atmosphere, pressures have effect on thermal decomposition peak temperature of HMX and AP, while Al powder and amorphous B powder occur to partial oxidation but no combustion.

(3) The mechanical sensitivities of the powdery metalized explosives are so high that the safety requirements for preparation and processing technology of mixing explosive cannot be satisfied although it can be obviously lowered by adding desensitized HMX and AP. While for the cast metalized explosives including mass compositions 35% of metal powder, 20% of AP, 60% of HMX and AP, 20% of HTPB and other additives satisfy the safety requirements.

(4) The nine kinds of metalized explosives of $\phi 50\text{mm}$ containing B and Al powder prepared all have cap initiation sensitivity and can propagate stable detonation, showing strong after-effect power ability. According to the experimental results, the detonation parameters and after-effect ability of metalized explosive can be improved by increasing the casting density.

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硼铝金属化炸药的制备及性能表征

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摘要: 以奥克托今 (HMX) 为基, 加入氧化剂高氯酸铵 (AP)、硼铝复合粉和粘结剂端羟基聚丁二烯 (HTPB), 设计和制备硼铝金属化炸药。用扫描电子显微镜 (SEM) 观测了硼粉、铝粉及硼铝复合粉的外观形貌; 用热重-差示扫描量热 (TG-DSC) 分析了奥克托今 (HMX) 和高氯酸铵 (AP) 对硼铝粉热氧化特性的影响, 对硼铝粉的反应动力学机理进行了深入了解; 为掌握金属化炸药对各种外界能量刺激的安全性以及传播爆轰波的能力, 测试了硼铝金属化炸药的撞击感度、摩擦感度、电火花感度、雷管起爆感度和起爆特性。结果表明, 硼铝复合粉中, 球形 Al 粉的表面有许多小颗粒的硼粉; 在室温 ~1000 °C 范围和 N₂ 气氛下, 虽然压力对 HMX 和 AP 的热分解峰温有影响, 但是, Al 粉和 B 粉仅发生部分氧化, 不能燃烧; 硼铝金属化炸药的撞击感度为 60% ~ 80%, 摩擦感度均为 100%, 电火花感度为 3.83 ~ 6.40 kV, 可以用 8# 工业雷管直接起爆, 表明无粘结剂的硼铝金属化炸药感度较高, 使用钝化 HMX 和 AP 后其感度明显降低, 添加聚氨酯粘结剂后其感度进一步下降, 当聚氨酯粘结剂含量为 20% 时, HMX 基硼铝金属化炸药的撞击感度小于 10%, 摩擦感度小于 30%, 显示能满足混合炸药制备及加工工艺的安全要求; 此外, 直径 ϕ 50 mm 的硼铝金属化炸药可以用 8# 工业雷管直接起爆, 能稳定传播爆轰波, 表现出较强的后效做功能力。

关键词: 金属化炸药; 制备; 安全性; 起爆传爆特性; 做功能力

中图分类号: TJ55; O389

文献标志码: A

DOI: 10.11943/j.issn.1006-9941.2017.05.006