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单基微气孔球扁药的静电感度研究

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摘要: 对采用化学发泡工艺制备的具有不同物理特性的单基微气孔球扁药的静电火花感度进行了研究。由于微气孔球扁药堆积密度变化范围较大,对测试仪器的物料装载槽进行了改造,适当调整了放电距离,利用高速摄像的方法对发火概率进行了统计分析。研究结果表明:单基微气孔球扁药的堆积密度和粒径等对静电火花感度有显著的影响,随着堆积密度降低和颗粒直径的减小静电火花感度显著提高。当堆积密度低于 $0.3 \text{ g} \cdot \text{cm}^{-3}$ 时,单基微气孔球扁药的静电火花感度明显高于硝化棉。采用这种改进后的方法对这类低密度的多孔材料的静电感度进行测试基本可行。

关键词: 物理化学; 球扁药; 微气孔; 安全性能; 静电火花感度

中图分类号: TJ55; TQ562; O64

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1 引言

近年来为了获得高燃速的火箭推进剂和发射药,在火药成型过程中加入发泡剂,通过物理或化学发泡的方法可以使药体形成多气孔结构。围绕多气孔发射药和推进剂的成型工艺、内部孔结构、燃烧特征和机理等,国内外有关学者都进行了一些研究^[1-6]。微气孔球扁药是在传统的多孔药基础上发展的一类新型含能材料,其特点是内部结构更均匀,气孔更细小,可以达到微米级甚至纳米级尺度。有关这种单基微气孔球扁药已经开展了燃烧特征方面的研究^[7],但是有关微孔结构球扁药安全性能的研究未见报道。由于具有新的内部结构,为了确保在应用过程的安全,有必要对单基微气孔球扁药结构与安全特性的关系进行研究。为此本文对该类材料的静电火花感度进行了研究。国内外有关火炸药静电感度测试方法还没有统一的标准^[8],国内通用的静电火花感度的测试都是针对粉状材料开展的,粒度要求在 60 目以下。对于单基微气孔球扁药,如果将颗粒进行破碎,其内部结构和整体的物理性能就会发生较的变化。若不改变这类微气孔球扁药的整体结构研究其静电感度,需要对原有的测试方法进

行一些调整。根据这类微气孔材料堆积密度低的特点,本研究主要是在装药量和针距等方面进行了调整。

2 试样与测试方法

2.1 单基微气孔球扁药样品特性

单基微气孔球扁药是采用溶剂法工艺制备得到的,主要制备过程包括物料的分散溶解,乳化发泡,成型及溶剂驱除等几个步骤,所用原料为 D 级硝化棉。将制备得到的单基微气孔球扁药样品进行筛分和干燥后,进行性能测试。

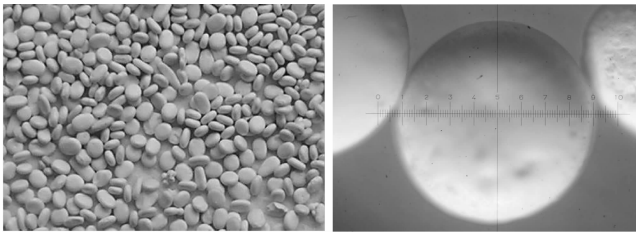
单基微气孔球扁药的外型大致有两种,从显微镜放大图看一种是椭圆形,另一种是圆形,其厚度与药粒的平均直径之比大多为 $1:1.5 \sim 1:3.0$ 。药粒的表面部分是光滑的,还有一些药粒表面也存在孔隙。将药粒仔细从中心切开,观察切面的状态,外观类似于木质结构,在显微镜下才能观察到孔隙。图 1 所示为典型微气孔球扁药的外观,其中(a)为样品的整体外观,(b)为典型药粒的显微观察图。图 2 所示的是典型微气孔球扁药的内部孔状结构,其中(a)和(b)所对应的样品堆积密度分别为 $0.3 \text{ g} \cdot \text{cm}^{-3}$ 和 $0.2 \text{ g} \cdot \text{cm}^{-3}$ 。采用电镜照片得到的切片图,统计分析微孔的孔径及分布情况,由于部分孔隙非常细小,并且相互贯通,很难准确地表征其真实的情况。由于 $10 \mu\text{m}$ 以上的孔隙数量较少,绝大多数孔隙在 $10 \mu\text{m}$ 以下,甚至直径小于 $5 \mu\text{m}$,因此无论堆积密度高低,平均孔径大多在 $10 \mu\text{m}$ 以下。

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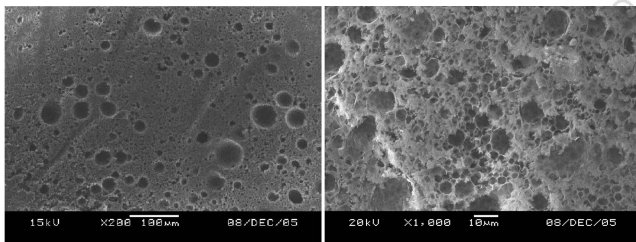
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(a) aspect photo of sample (b) microscope photo of sample

图1 微气孔球扁药的外观

Fig.1 Appearance of the micropores oblate propellant(MPOP)



(a) bulk density $0.3 \text{ g} \cdot \text{cm}^{-3}$ (b) bulk density $0.2 \text{ g} \cdot \text{cm}^{-3}$

图2 微气孔球扁药内部孔隙结构

Fig.2 Inner pore structure of the micropores oblate propellant(MPOP)

2.2 参照样品种准备

为了研究对比不具有孔状结构的单基药的静电感度,本文采用制备单基微气孔球扁药的原料D级硝化棉作为参比样品。

2.3 测试仪器和方法

测试装置: JGY-50型静电火花感度仪, FE-200型数码相机。

方法: 静电火花感度的测量方法参照 GJB/z 736.11-90《火工品试验方法 电火工品静电感度试验》进行。采用数码摄像的方法对发火情况进行记录和分析。在初步试验的基础上选用充电电压: 7 kV; 电容: $0.01 \mu\text{F}$; 针距: 2.5 mm; 装药量: 25.0 mg; 温度: $25 \text{ }^\circ\text{C}$; 湿度: 49%。

判别标准: 试样发生冒烟、燃烧、爆炸等均判为发火。

以上实验均采用爆炸概率法来表征试样的感度, 一组(25发)试验的爆炸概率点估计值按下式计算:

$$P = X/25$$

式中, P 为爆炸概率; X 为25发试验中发生爆炸的次数。

按此方法计算出每一组试验的爆炸概率, 当两组平行试验的爆炸概率无显著性差异时, 以两者的算术平均值作为该试样的感度爆炸概率。

3 结果与讨论

3.1 堆积密度对微孔球扁药静电感度的影响

在上述条件下, 对粒度范围为40~60目的不同堆

积密度的单基球扁药样品静电感度进行了测试, 结果如表1所示。药粒的堆积密度采用 GJB771.101-88 规定的方法测试。药粒的粒度分布采用 GJB771.106-91 规定的方法测试。

表1 不同堆积密度球扁药的静电感度

Table 1 Effects of bulk density on electrostatic sensitivity of MPOP

sample	ρ / $\text{g} \cdot \text{cm}^{-3}$	d /mm	grain size /mesh	fire probability /%
D-class NC	0.19	-	<60	60
MPOP	0.15	0.206	60-100	84
MPOP	0.20	0.206	60-100	68
MPOP	0.30	0.206	60-100	64
MPOP	0.37	0.206	60-100	60
MPOP	0.45	0.206	60-100	56

Note: ρ is bulk density; d is grain diameter.

随着样品堆积密度的提高, 其静电火花感度不断降低, 从84%降到56%, 而纤维状的D级硝化棉静电火花感度为60%。当堆积密度在 $0.3 \text{ g} \cdot \text{cm}^{-3}$ 以下时, 单基微气孔球扁药的静电火花感度明显高于D级硝化棉。

3.2 粒径对微孔球扁药静电感度的影响

将药粒堆积密度为接近的不同粒径范围的球扁药试样的静电感度进行测试, 结果如表2所示。样品颗粒的平均粒径增大, 其静电火花感度大幅度下降, 从84%降低到20%。

表2 不同粒度球扁药的静电感度

Table 2 Effects of grain diameter on electrostatic sensitivity of MPOP

sample	d /mm	ρ / $\text{g} \cdot \text{cm}^{-3}$	grain size /mesh	fire probability /%
D-class NC	0.206	0.190	60-100	60
MPOP	0.592	0.148	20-40	20
MPOP	0.423	0.148	32-40	44
MPOP	0.298	0.148	40-60	70
MPOP	0.227	0.148	60-65	80
MPOP	0.206	0.148	60-100	84

4 结论

本研究所用的针对单基微气孔球扁药的静电火花感度测试方法基本可行。

单基微气孔球扁药的静电火花感度与样品的特性有一定关系, 发火概率随着样品的堆积密度的减小而提高, 随着样品的平均粒径的减小而提高。当堆积密度在 $0.3 \text{ g} \cdot \text{cm}^{-3}$ 以下时, 单基微气孔球扁药的静电火花感度明显高于D级硝化棉。

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Electrostatic Spark Sensitivity of Single Base Oblate Propellant with Micropores

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Abstract: The oblate propellant with micropores (MPOP) was prepared by chemical foamed process, and the effects of the materials and inner-structure on its electrostatic spark sensitivity were studied by adjusting the distance of discharging and the way of loading. Results show that the lower the bulk density of these oblate propellants is, the higher electrostatic spark sensitivity is. And the smaller the grain diameter of the oblate propellants is, the higher its electrostatic spark sensitivity is. The electrostatic spark sensitivity of MPOP with the bulk density not more than $0.3 \text{ g} \cdot \text{cm}^{-3}$ is apparently higher than nitrocellulose. It is feasible to test the electrostatic spark sensitivity of low-density materials with micropores using this improved method.

Key Words: physical chemistry; oblate propellant; micropore; safety behavior; electrostatic spark sensitivity

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Specific Heat Capacity of Carbohydrazide Nitric Acid Energetic Coordination Compounds

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Abstract: The specific heat capacity of four carbohydrazide nitrate energetic coordination compounds $[M(\text{CHZ})_3](\text{NO}_3)_2$, ($M = \text{Mn}, \text{Co}, \text{Zn}, \text{Ni}$) were determined by differential scanning calorimeter (DSC) under a temperature range, and regression equations for the specific heat capacity changing with temperature were obtained by Origin 7.0. The equations of $\text{Ni}(\text{CHZ})_3(\text{NO}_3)_2$ and $\text{Mn}(\text{CHZ})_3(\text{NO}_3)_2$ are functions of sixth degree, second degree and third degree in some range of temperature, equations of the others mostly are functions of fourth degree or fifth degree, where the relative coefficient square is more than 0.987, and standard deviation is smaller than 0.017. Except $\text{Zn}(\text{CHZ})_3(\text{NO}_3)_2$, the specific heat capacity of the compounds changes more, and one or more peaks appear in the curves of the specific heat capacity. TG and FTIR were carried through. At $200 \text{ }^\circ\text{C}$, only $\text{Co}(\text{CHZ})_3(\text{NO}_3)_2$ loses 8.64% of its mass. As FTIR spectra are not same at different temperature, it is possible that the crystal transformation results in the change of the specific heat capacity.

Key words: analytical chemistry; differential scanning calorimeter; specific heat capacity; $\text{Mn}(\text{CHZ})_3(\text{NO}_3)_2$; $\text{Co}(\text{CHZ})_3(\text{NO}_3)_2$; $\text{Ni}(\text{CHZ})_3(\text{NO}_3)_2$; $\text{Zn}(\text{CHZ})_3(\text{NO}_3)_2$