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HMX 晶体颗粒球形度的定量表征

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摘要: 采用数字光学显微镜成像技术、图像处理技术以及统计技术对 HMX 晶体颗粒形貌进行了定量分析, 对 280 ~ 400 μm , 400 ~ 450 μm 粒径范围数千个球形化处理前后的 HMX 颗粒球形度进行了统计计算, 其颗粒平均球形因子分别为 0.785 和 0.816 及 0.790 和 0.810, 建立了 HMX 晶体颗粒球形度的定量表征方法, 定量表征了 HMX 颗粒的球形化效果。

关键词: 分析化学; HMX; 颗粒形貌; 球形度

中图分类号: O65; TJ55

文献标识码: A

1 引言

炸药的晶体尺寸、晶体表面形貌对于炸药的感度、力学性能、加工性能(流变性能)以及填充密度等都有很大影响。以色列 RAFAEL^[1]研究了基于球形 HMX 高聚物粘接炸药的流变学、感度和机械力学性质,发现使用球形 HMX 填充密度显著增加,流速增加一个量级,力学性能也显著增强。因此对于 HMX 的晶体颗粒形态的表征就显得极为重要。目前对 HMX 晶体颗粒的表面形貌研究多是采用显微照像技术定性分析,而对 HMX 晶体颗粒的表面形貌定量研究(如颗粒球形度和表面粗糙度)较少。我们采用数字光学显微镜成像技术和图像处理技术,对球形化 HMX 颗粒的球形因子进行了统计,初步定量表征了 HMX 颗粒的球形度,为研究 HMX 颗粒的球形化程度与其流变学、感度和机械力学性质的定量关系,奠定了基础。

2 原理

形状因子(shape factor)是对颗粒形状进行定量表征的参数,其定义方法有九种^[2],其中圆度(circularity)^[3]是基于二维图像分析的形状因子定义方法,其计算公式为:

$$\Phi_{\text{circularity}} = \frac{4\pi \cdot A}{P_{\text{rough}}^2} \quad (1)$$

式中, $\Phi_{\text{circularity}}$ 为颗粒圆度, A 为颗粒的投影面积, P_{rough}

为全部投影周长。

本文对于 HMX 颗粒球形因子的定义采用圆度计算公式,通过计算颗粒的球形因子定量表征其球形度。

3 实验

实验采用的 HMX 原材料是由银光化学工业公司提供的粗品,为了改善 HMX 颗粒的表面形貌,对其进行了球形化处理,然后采用水筛法将球形化处理前后的 HMX 颗粒进行颗粒度分级,制备出不同粒径范围的 HMX 颗粒。

采用数字光学显微镜对上述处理前后的 HMX 颗粒进行照像,得到颗粒的二维光学显微图像,然后采用图像处理软件结合 MATLAB 软件对光学显微图像进行分析,计算其球形因子,统计其结果。

4 结果与讨论

4.1 HMX 颗粒形貌分析

图 1 是采用数字光学显微镜得到的 HMX 原始颗粒及球形化处理后的不同粒径的 HMX 颗粒的图像,从图中可以看出 HMX 原始颗粒是由多个不同结晶取向的微晶粒镶嵌构成的孪晶或聚晶,颗粒表面棱角较多。经球形化处理, HMX 颗粒表面棱角减少,表面明显光滑,颗粒的透光性较好,形状接近于球形。

4.2 颗粒球形因子计算

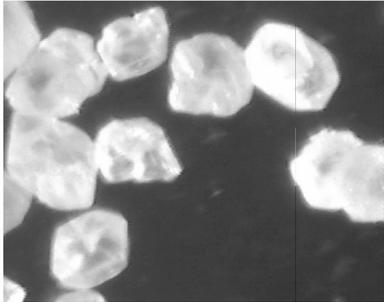
首先采用数字光学显微镜得到 HMX 颗粒的形貌图像(见图 2 a, 图 3 a),然后通过图像处理,去除边界颗粒,划分优选出待分析的颗粒的二维投影图像(见图 2 b, 图 3 b),提取颗粒的投影轮廓,计算每个颗粒的投影面积和投影轮廓的像素,代入公式(1)即得到

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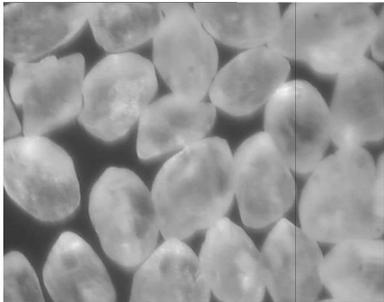
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单个 HMX 颗粒的球形因子(见表 1),而后对所有颗粒的球形因子进行统计分析,定量表征 HMX 颗粒形状分布情况。



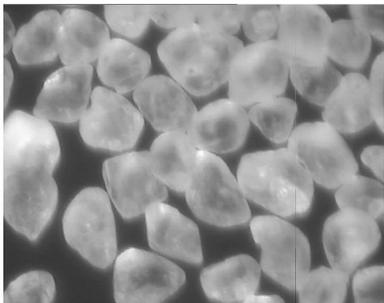
(a) HMX 原始样品(50 倍)

(a) original HMX particles(×50)



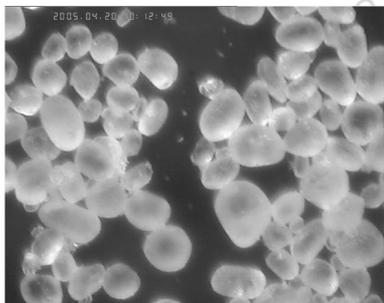
(b) 280 ~ 355 μm 球形化 HMX(75 倍)

(b) 280 - 355 μm spherical HMX particles(×75)



(c) 180 ~ 280 μm 球形化 HMX(100 倍)

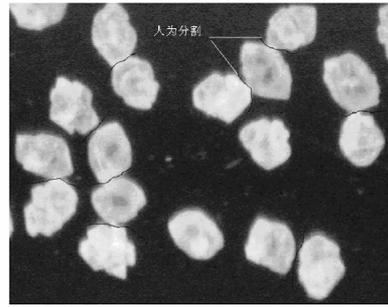
(c) 180 - 280 μm spherical HMX particles(×100)



(d) 125 ~ 180 μm 球形化 HMX(100 倍)

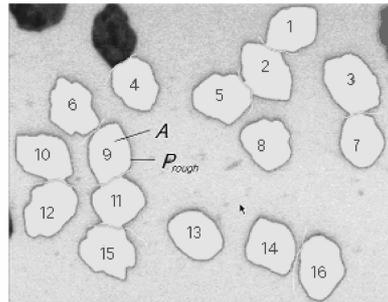
(d) 125 - 180 μm spherical HMX particles(×100)

图 1 不同粒径 HMX 颗粒的光学显微图像
Fig.1 Optical microscopic pictures of HMX particles with different size



(a) 图像处理前的光学显微图像(35 倍)

(a) Optical microscopic picture before image manipulation

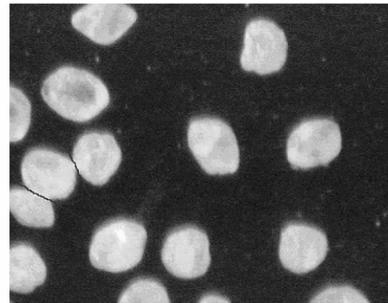


(b) 图像处理后的轮廓提取二维投影图

(b) Planar projection picture after image manipulation

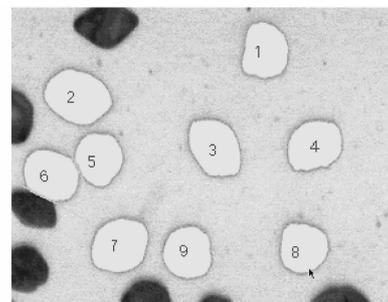
图 2 400 ~ 450 μm 原始 HMX 颗粒的图像处理

Fig.2 Image manipulation of 400 - 450 μm original HMX particles



(a) 图像处理前的光学显微图像(35 倍)

(a) Optical microscopic picture before image manipulation



(b) 图像处理后的轮廓提取二维投影图

(b) planar projection picture after image manipulation

图 3 400 ~ 450 μm 球形化 HMX 颗粒的图像处理

Fig.3 Image manipulation of 400 - 450 μm spherical HMX particles

表1 400~450 μm HMX 颗粒的球形因子举例

Table 1 Example of sphere factors of 400~450 μm HMX particles

particles No.	original	spherical
1	0.822	0.828
2	0.765	0.857
3	0.777	0.815
4	0.824	0.833
5	0.820	0.850
6	0.736	0.836
7	0.842	0.843
8	0.805	0.851
9	0.791	0.843
10	0.705	
11	0.759	
12	0.789	
13	0.862	
14	0.810	
15	0.736	
16	0.813	
average	0.791	0.840
standard deviation	0.042	0.013

HMX 颗粒的投影轮廓对于颗粒球形因子的准确计算至关重要,在进行图像处理过程中,要求原始图像的质量要好,分辨率要高,边缘轮廓清晰,颗粒与颗粒之间不能重叠,对于不能完全分开的颗粒要进行人为分割,如图 2b 中的 1、2、5 颗粒,6、9、11、15 颗粒,在图形处理前就要人为对其进行分割。因此样品制备非常重要,既要保证一个样品板上有足够多的颗粒,又要保证颗粒之间不重叠,如何快速制得合格的样品,尚值得进一步研究。为了保证结果的代表性,需要对样品进行多达百幅图片照相、处理,最终得到近千个颗粒的球形因子数值。如何自动快速获得这些图象,减少人为因素的影响,也是值得探讨的。

采用上述方法,分别对 400~450 μm、280~400 μm 粒径范围的原始 HMX 颗粒和球形化 HMX 颗粒的球形因子进行统计计算,结果列于表 2,400~450 μm HMX 颗粒球形因子的分布情况如图 4 所示。假设分布为正态分布,分别采用 F-检验法和 U-检验法对球形化处理前后球形因子测试数据的精密度和平均值进行显著性检验,结果表明精密度和平均值均具有显著性差异,说明球形化处理后 HMX 颗粒的球形因子增大,球形化 HMX 颗粒比 HMX 原始颗粒的球形因子的精密度更高,离散性更小,图 4 也显示出球形化 HMX 颗粒比 HMX 原始颗粒的球形因子分布明显变窄,说明球形化 HMX 颗粒的外形更加规整均匀。

表2 不同粒径 HMX 颗粒的球形因子

Table 2 Sphere factors of HMX particles with different size

HMX particle size and type	400~450 μm		280~400 μm	
	original	spherical	original	spherical
particle number	924	950	801	1109
average sphere factor	0.790	0.810	0.785	0.816
standard deviation	0.039	0.034	0.042	0.033
F-test	1.405		1.597	
F	$\alpha = 0.05, F_{0.95}(\infty, \infty) = 1$			
U-test	12.158		17.377	
U	$\alpha = 0.05, Z_{\alpha/2} = 1.96$			

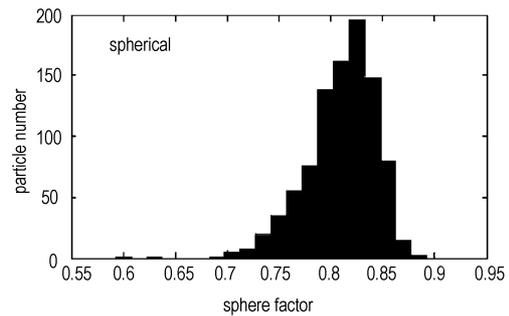
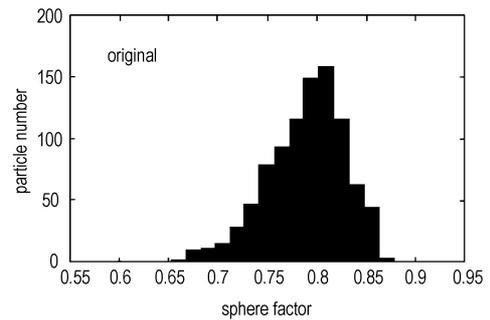


图4 400~450 μm HMX 颗粒的球形因子统计分布图

Fig.4 Sphere factor distribution of 400~450 μm HMX particles

5 结论

通过以上研究,建立了 HMX 颗粒球形度的定量表征方法。测试结果表明,采用光学显微镜成像技术和图像处理技术计算颗粒球形因子的方法可以定量区分不同 HMX 颗粒的球形度,量化 HMX 颗粒球形化处理效果。

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Study on Mechanical Isotropic of PBX

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Abstract: The mechanical properties of two types of specimens were tested, which were sampled from different direction (transverse and longitudinal) in two PBX formed by mould pressing and isostatic pressing respectively. The experimental results show that both of tensile and compressive strength between transverse and longitudinal directions in isostatic pressing PBX are almost the same, in other words, isostatic pressing PBX is mechanically isotropic; and although compressive strength between transverse and longitudinal does not show obvious difference for mould pressing PBX, but tensile strength shows apparent difference, mould pressing PBX is anisotropic.

Key words: materials mechanics; polymer binder explosive (PBX); mechanical property; isostatic pressing; mould pressing

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Quantitative Characterization of HMX Particle Sphericity

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Abstract: The shapes of spherical HMX particles were studied by digital optical microscope and image manipulation technology. The sphericity of HMX particles were identified and calculated, the average sphere factors of HMX particles are 0.785, 0.816, 0.790 and 0.810, respectively. The shapes of HMX particles are quantitatively characterized through statistically calculating the sphericity of thousands of HMX particles with particle size of 280 – 400 μm and 400 – 450 μm . The results show that this method can distinguish the circularity of different HMX particles and quantitatively characterize the HMX spheroidizing effect.

Key words: analytical chemistry; HMX; shape of particle; sphericity

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Safety of Heating TNT in Microwave Oven

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Abstract: The tests for understanding safety and potential application of melting TNT by microwave heating were carried out in the anti-explosion room by the commercial microwave oven with output power of 0 ~ 600 W, which was improved for safety performances. The effect of output power, loading capacity of TNT and container on temperature-time curves were studied. The process for melting TNT were recorded. The surface chemistry and the molecular structure of TNT samples before and after microwave heating were analyzed. The results show that the surface and the structure of TNT do not change after microwave heating when microwave power is 120, 240, 360 W; loading capacity of TNT is 10, 50, 100 g; container is of glass or plastic; heating time is 40 min; the maximum heating velocity is 66 $^{\circ}\text{C} \cdot \text{min}^{-1}$; the maximum heating temperature is 146 $^{\circ}\text{C}$, respectively. Furthermore, combustion or explosion does not occur in the tests.

Key words: physical chemistry; microwave heating; TNT; safety; explosive