Letter

A study on mechanical comminution of obsolete HTPB propellants from the rocket motor

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Abstract

With the replacement of weapons as well as the factor of aging effect, it is urgent to deal with obsolete HTPB propellants effectively which is taken out of the retired rocket motor, and transforming it into civil explosives is regarded as a hopeful direction now. Though obsolete HTPB propellant can meet the basic requirements on the energy characteristics as a component of explosives, its particle size must be decreased and distribute uniformly on the millimeter level at least. Therefore, nothing but comminution can ensure symmetrical mixture with other components so as to achieve the purpose of stable detonation. There are many commonly used mechanical comminution methods which can make granularity of the propellants from the original diameter ($10\sim100$ mm) to the final ($0.5\sim5$ mm). But most of them would lead to great loss of AP and Al as the main energetic component inevitably, and the extent of loss has certain relationship with them. Therefore, according to the special characteristics of HTPB, measured by diameter ratio and loss rate of AP and Al before and after the comminution, three kinds of comminution methods (machines) including cage wet grinder, dry cyclone cutter and remote control cutter, are selected comparatively to study which will be fit for HTPB under the premise of security. The result shows that dry cyclone cutter is the most ideal comminution method which can make material diameter size reach about1mm or so, while the loss rates of two active ingredients are 2.6 % and 4.2 % respectively. The following initiation test, in which three kinds of civil explosives prepared by different sizes of samples are detonated by8[#] detonators, can also verify this conclusion above.

Keywords : military chemistry and pyrotechnics, obsolete HTPB propellants, comminution method, initiation tests

1. Introduction

In recent years, the military powers have carried out in -depth study on the preparation of civil explosives and other energetic materials with waste solid propellants¹⁾. For example, it shows that the re-use process of obsolete HTPB propellants can be divided into three steps as following: First, the missile or rocket power system composed by propellants and motor was separated by the high -pressure water jet, and then the HTPB propellant is secondly crushed into the smaller particle through appropriate comminution method after the separation. The third step is to accomplish the preparation of a new novel civil explosive by adding some energy components, and the process used to include mixing, compression and initiation tests, etc. In the three steps, how to choose a safe and effective comminution method iscrucial and indispensable. Now some mechanical comminution such as crushing, grinding, folding broken, splitting pieces and impact crushing, etc, can be used to overcome the internal cohesion of HTPB in theory. Due to the fact that HTPB propellant simultaneously owns the character of high flexibility and sensitivity, some researchers confirm that mechanical sensitivity of HTPB is mainly determined by the AP and the catalyst such as ferrocene³⁾, and if the heat or impact of conventional crushing effecting on them is too violent, hot spots or active sites can easily be produced by shear and distortion, which would be a risk to burn, even to explode possibly. So the key to the successful comminution is not only to offer sufficient energy to tear the internal combination, but also to avoid burning and exploding. This paper is designed to explore the comminution method to a certain extent according to the characteristics and certain re–use vector of HTPB.

methods				
technical parameter	wet cage grinder	drycyclone cutter	remote control cutter	
chargein grain size / mm	\leq 50	≤ 30	≤100	
diameter ratio	30-40	18	≤ 10	
rotate speed / rpm	3000	≤ 25000	_	
power / kw	25	12	30	

 Table 1
 technical parameter of laboratory comminution methods

2. Experiments Part

2.1 Test materials and comminution methods

The cubic HTPB bloom with diameter of $20 \text{ mm} \times 20 \text{ mm} \times 20 \text{ mm} \times 20 \text{ mm}$ (named sample0) will be used to simulate the fragments separated from the retired rocket motor by the high-pressure water jet. The contents of active ingredient AP and Al in it are tested as 63.1% and 14.2% respectively with the "Chinese Standard QJ 914.2-1985: analysis method of composite solid propellant component" before comminution, which should be re-measured before the preparation of civil explosive.

Through calculation, we can see that when the mass ratio between HTPB and coolant (water) reaches 1:5 for wet crushing method and under the conditions of the speed \leq rpm, working time \leq 1h for dry crushing method, they can both ensure that the impact on the HTPB is less than the critical value⁴. Therefore, under the input is 100 g and work time is 5 min in each runs, three kinds of laboratory comminution methods including wet cage grinder, dry cyclone cutter and remote control cutter are chosen to test for the crushing effect on HTPB under the premise of security, technical parameter of which are shown in Table 1, and the generated samples are named as I, II, III separately.

2.2 Comminution test

(1) Wet cage grinder. Dr Gu Jian-liang in Nan-Jing University carried out a careful study on the comminution of



Fig. 1 Sketch of wet cage grinder 1-steel pole, 2-shaped block, 3-powder grains.



Fig. 2 Sketch of dry cyclone cutter. 1-inlet, 2-powder grains, 3-rotor, 4strain net, 5-cutter, 6-discharge hole

the waste multiple double-base powder and propellants with wet cage grinder^{5,6)}, which attains success. Based on the fact that both HTPB and double-base drug belong to the energetic high polymer with resinoid, wet cage grinder is put forward to carry out with speed reaching 3000 rpm, and then the crushing radio can exceed 30–40. After sample 0 is put into the grinder, during the circle outward movement under the centrifugal force and gravity, the following high-speed rotations of the lap steel bar can break them entirely. The safety of whole process can be ensured by adding on-going cool water as coolants, and its sketch is shown in Fig. 1.

(2) Dry cyclone cutter. As the internal structure of HTPB is similar to that of common rubber, the grinding equipments which suit rubber may be appropriate for HTPB in principle. In order to solve the issues of gathering groups for sample I and great loss of energetic components when contacting with water, the dry cyclone cutter with appropriate speed and working time in common use for rubber is tested for HTPB. The sample 0 is carried from the hopper to the grinding chamber through the screw conveyor, and sheared by the high–speed rotating blade, then transported into the cyclone by negative pressure. The way of heat dissipation is of the air–filled type through the pan mill, which can also ensure test security without coolants in the course. The sketch of dry cyclone cutter is shown in Fig. 2.

(3) Remote control cutter. Due to the high risk of operation, the most secure measure is to minimize the opportu-



Fig. 3 Sketch of remote control cutter.

nities of contacting with them directly for the operators. Sample 0 are transformed into the cutting CNC lathe while the machine is adjusted with its size and position as 3 mm cube condition. Under the design of remote control and the isolation operation, above comminution can proceed within the explosion–proof rooms through industrial television system which can be remotely monitored and controlled⁷, and the general layout of equipment is shown in Fig. 3.

2.3 Initiation Test

Currently, 8[#] detonator is used to evaluate the initiation sensitivity of the explosive grain that is composed by three different HTPB samples to determine which of them can be used as main component of civil explosives. The production of explosive grain is as follows: samples are put into the kraft paper tube with $\Phi45\,\text{mm}$, apparent density of explosive gain filled with samples II is measured $0.95-1.10 \,\mathrm{g/cm^3}$, in order to compare the initiation at the same apparent density, and the other samples are control the density with $1.0 \,\text{g/cm}^3$ or so by pressed gently with a wooden stick, and the height is generally charged three times as large as the diameter for the grain. Then, an $8^{\#}$ detonator is inserted and fixed into the explosive grain or the booster primer circle, connected with the detonation cable tightly after the top of grain is sealed. The depth of pitting coming from initiation on a piece of 45[#] steel separator with $100 \text{ cm} \times 100 \text{ cm} \times 1 \text{ cm}$ is recorded after initiation tests in order to assess the success or failure of initiation. If the 8[#] detonator can not initiate the sample explosive, then adds 10% insensitive RDX processed into cer-



Fig. 4 Experimental set-up of initiation test. 1-8# detonator, 2-insensitive RDX, 3-explosive grain, 4-separator

Table 2	Result of comminution tests and Initiation tests.
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sample	granularity /mm ⁻	loss rate/%		ignition condition Φ45mm	
		AP	Al	a	b
Ι	_	66.1	65.5	failed	failed
II	1	42.0	4.2	success	success
III	3	1.4	1.4	failed	success

 $a:8^{\#}$ detonator

b: insensitive RDX

tain circle whose diameter is same as explosive grain to improve its initiation sensitivity as priming charge until being able to detonate, sketch of experimental set-up shown as Fig. 4.

3. Results and Analysis

In order to choose the optimum comminution method, the initiation test is done to measure whether HTPB can be prepared for the civil explosives, and diameter ratio and the loss rate of component are measured as the standard of crushing effect, shown in Table 2.

3.1 Comminution Test

The shapes of sample I, II, III are separately shown in Figs. 5, 6, 7, and it can be seen from Table 2 that although wet cage grinder can effectively crush double-base propellants, but it is not suitable for HTPB propellants. First of all, the matrix structure of HTPB propellant is so different from that of double-base propellants that its polymeric ally bound binder is not easily squeezed as the elastic body. Although situation can be slightly improved by extending the grinding time, the materials after comminution will turn into a cohesive group, like cotton wool when contacting with water, which result in that it can not be



Fig. 5 Sample I assumes to be cotton wool form when meeting the water.



Fig.6 Sample II is about1mm which can be separated from AP at the bottom.



Fig.7 Sample III exhibits the cubeabout $3 \text{ mm} \times 3 \text{ mm} \times 3 \text{ mm}$.

separated from the machine, and the problem could not be explained quite simply and conclusively. Secondly, AP and Al exposed to the cooling water on the fractured surface are easily scoured down and dissolved in it. With the reduction of diameter and the increase of surface area, AP and Al will become easier to be out of the adhesives. The loss rate is determinate as high as 66.1% and 65.5%, which makes the conditions of reuse no longer available. Therefore, wet cage grinder can not be used to crush HTPB. Figure 6 shows that the diameter of sample II is the smallest about1mm after being crushed by dry cyclone cutter, but the distribution for the diameter size is not very uniform because of the constraints of the safe working hours. The content of Al is almost constant before-and-after comminution, while AP decreases 42%, but most of the lost AP can be separated from sample through sifting for recycling, and the final loss rate is only 2.6%. The remote control cutter is regarded as the most secure and reliable one, but limited by the state of the industrial art, whose sample has exhibited cube with limit value of particle size 3mm uniformity and has little loss of components. However, its shortcoming that includes both longer operating time and more cost should not be ignored.

3.2 Initiation Test

From the subsequent initiation tests we can see, sample I fails to detonate because the internal energetic compo-



Fig. 8 Initiation (separator) tests.

nents have been devastated in the comminution process, and the separate adhesive can not be detonated, which also confirms the previous inference. The diameter of sample II is the smallest, and the losing components can be ignored, so a stable detonation takes place at a very high rate. But due to the uneven distribution of particles in the charge time, there is still possibility of putting out the explosion. The result of sample III is semi-explosion or no explosion, and then the latter detonation situation has been improved by adding booster primer. It is considered that the diameter of sample III is so large that detonation wave can not normally transmitted. If they are prepared for civil explosives, it is necessary to improve the state of the art to continue further comminution, which limits the re-use adhibition immensely.

4. Conclusions

(1) Although wet cage grinder can make sure a safe comminution process, the problem that particle agglomerated when wet has not been resolved, so this method does not adapt to HTPB.

(2) Remote Control cutter is regarded the most safe and reliable method, but limited by the process conditions, sample III is too large in diameter to be set off by $8^{\#}$ detonator, so it still needs to be further crushed to prepare for civil explosives.

(3) With the appropriate speed and working time, dry cyclone cutter can meet the safety requirements of comminution. The loss rate and the particle size can make it suit to prepare common civil explosives to a higher degree.

In view that working time and the maximum speed are both restricted strictly to prevent the underlying accident for dry cyclone cutter, a new novel comminution method named liquid nitrogen freezing would be used soon by replacing it in the next phase. Through freezing the material to be glassy state below -30° C, the method can not only reduces the risk greatly, but also makes HTPB so crisp as to crush easily⁸⁾, which is regarded as the developmental direction of comminution industrialization.

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