Microstructure of Plastic Bonded Explosives and Its Evolution after Thermal and Mechanical Aggressions*

CHEN Pengwan[1], HUANG Fenglei[1] & DING Yansheng[2]

(1 State Key Laboratory of Prevention and Control of Explosion Disasters, Beijing Institute of Technology, P O Box 327, Beijing 100081, China)

(2 Institute of Mechanics, Chinese Academy of Sciences, Beijing 100080, China)

Abstract: Because sensitivity of an explosive is highly controlled by its microstructure, it is very important to examine and characterize it. Both scanning electronic microscopy and polarized light microscopy were chosen to examine the microstructure of selected plastic bonded explosives. The initial microstructure of hot pressed plastic bonded explosives and its evolution under thermal and mechanical aggressions were studied. To better reveal the microstructure, polishing and etching techniques were developed to prepare the samples of microscopic examination. The thermal aggressions included low temperature freezing and frozen combustion. Mechanical aggressions included quasi-static Brazilian test, uniaxial compression, ultrasonic wave insult and long-pulse low-velocity impact. The characteristic damage modes under different solicitations were analyzed.

Keywords: polymer bonded explosives; microstructure; damage; thermal aggression; mechanical aggression

1 Introduction

Microstructure of energetic materials is of crucial importance in the initiation process as well as in the reactive response to various solicitations, i.e. in vulnerability evaluation. Numerous studies have shown that the presence of defects, such as pores or cracks, can greatly increase the sensitivity of the energetic materials because they are potential hot spots where chemical reactions are likely to happen and also because they increase the specific area available for combustion. It is therefore very important to characterize, as precisely and as quantitatively as possible, the microstructure of energetic materials.

Different microscopy tools have been successfully used to characterize the microstrcutre of explosives. Scanning electronic microscopy (SEM) is one of the most widely used techniques in this regard[1]. Sharma, et al[2] used atomic force microscopy to examine the surfaces of explosive crystals and for the investigation of hot spot reaction sites. Borne[3] used optical microscopy with matching refractive index to analyze the defects in the bulk of RDX crystals. In addition, environmental scanning electronic microscopy (ESEM)[4] and polarized light microscopy (PLM)[5-7] have also been successfully used to examine the microstructure of explosives.

The present paper has investigated the microstructure of hot pressed plastic bonded explosives (PBXs) and its evolution after different thermal and mechanical aggressions by using scanning electronic microscopy and polarized light microscopy.

2 Experiments

Hot pressed HMX-based PBXN-5 was used in the experiments. PBXN-5 contained HMX 94.5%-95.0% and fluorin rubber 5.0%-5.5% by weight. Molding powder was pressed to various levels of porosity. The pressing temperature was 100 ℃ and the time duration of pressing was 1.5 h. To generate different microstructure, different pressure was used, ranging from 25 MPa to 400 MPa. The initial microstructure of pressed PBXN-5 prepared under different pressure was first examined.

To examine the evolution of microstructure under different solicitations, pressed PBXN-5 samples were subjected different thermal and mechanical aggressions. The thermal aggressions included low temperature freezing and frozen combustion. Mechanical aggression included quasi-static Brazilian test, uniaxial compression,

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ultrasonic wave test and long-pulse low-velocity impact. To evaluate the influences of a thermal load induced by low temperature, low temperature freezing tests were conducted by immersing samples into liquefied nitrogen for 5 minutes. In frozen combustion test, samples were ignited by a flame, and after a few seconds, were dropped in a water bucket where it was instantly cooled and extinguished. Brazilian test, or diametrical compression, in which a disc of material is loaded diametrically in compression, was used to generate tensile stress insults. Uniaxial compression was also conducted to evaluate the influences of compressive stresses. Different strain rates were used in both Brazilian test and compression. Ultrasonic waves are basically a mechanical aggression which is very different from shocks or impacts. In ultrasonic test, samples were submitted to ultrasonic waves in a bath with distilled water for 5 minutes. In addition, a long-pulse low-velocity gas gun with a gas buffer was also developed and used to apply dynamic compression, through which the time duration of dynamic compression can be extended to several milliseconds[8]. The explosive samples were confined in steel tubes during impact. The sample sizes were ϕ10mm×12mm for compression and ϕ20mm×10mm for Brazilian test and low velocity impact respectively.

Low toughness and safety concerns bring additional difficulties in preparing PBX samples for microscopic examination. In microscopic examination, samples were first ground using standard fine silicon carbide papers (800 grid) to obtain a flat surface. Final polishing was carried out in an automatic polishing machine using 1 μm alpha alumina powder, at a load of 50 g, while being lubricated with distilled water. To avoid bringing additional unexpected damage to the samples, especially the damaged or fractured samples, during polishing, samples were first potted in commercial low-viscosity epoxide mounts with traditional amine hardening agent and then cured. To better reveal the details of the microstructure, iso-methyl butyl ketone was selected to etch the surface.

3 Results and Disccussion

3.1 Initial Microstructure

Fig.1 shows a micrograph of a molding powder granular in which a lot of HMX grains are gathered together by the binder. A lot of voids are present in the molding powder. The size of HMX grains is in the range of tens to hundreds micrometers. Many surfaces of HMX particles seem clean, revealing that the binder was not uniformly coated on HMX particles during the production of molding powder. The HMX crystals are basically free of cracks. The molding powder was then pressed to various levels of porosity by adjusting the pressing pressure. The pressing pressure greatly influenced the microstructure of pressed PBXN-5. At low pressing pressure, a lot of intergranular voids were still present. With the increase of pressure, the intergranular voids decreased, however crystal fractures also occurred. When the pressing pressure reached 100 MPa, extensive crystal fractures can be observed. Fig.2 shows a micrograph of PBXN-5 with a porosity of 2.5% pressed under 100 MPa. It is clearly shown that various forms of defects are present in pressed PBXN-5, including intergranular voids, debonding of the binder from crystals, and crystal factures. In addition, deformation twinning can also be observed on large crystals in some cases. To further examine the microstructure of HMX crystals, pressed PBXN-5 was keeping in a selected solution for enough time to totally remove the binder and leave the explosive particels alone. Microscopic examination of the recovered HMX crystals showed the presence of extensive fracture of particles and a lot of microcracks[8]. Crystal fractures are mainly associated with particle-to-particle contacts due to an extremely high concentration (over 90 %) of explosive crystals in PBX. Another important phenomenon in pressed PBX was the presence of deformation twinning, an evidence of plastic deformation of explosive crystals during pressing. The above results also reveal that considerable initial damage is present in hot pressed PBX, especially in PBX samples obtained under high pressing pressure. Pressing not only consolidates the molding powder, it also induces new damage to explosive crystals. Further studies are needed to quantitatively characterize the damage generated in pressing and its influences on the properties of PBX.

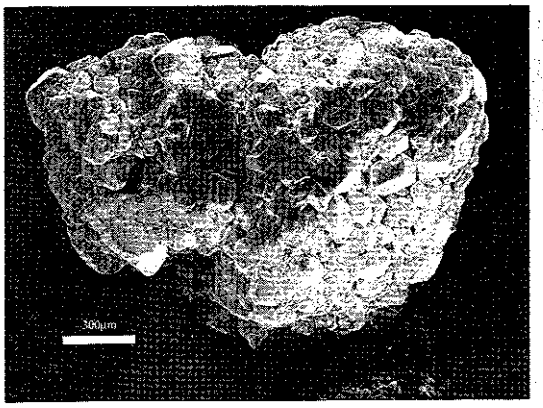

Fig.1 Micrograph of a molding powder granular

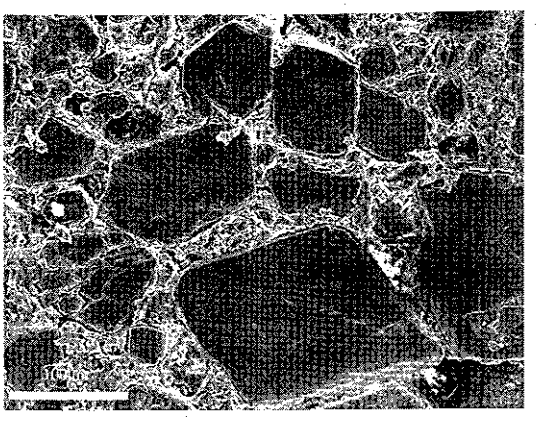

Fig.2 Micrograph of pressed PBXN-5 with 2.5% porosity

3.2 Thermal Aggressions

When PBXN-5 was subjected to freezing in dry ice for 5 minutes, no visible cracks were observed. However when PBXN-5 was subjected to freezing in liquefied nitrogen for 5 minutes, cracks were initiated. Some small cracks were linked together and formed larger ones. The cracks propagated mainly along the crystal boundaries. The results show that the interfacial strength of PBXN-5 is low. Due to the large difference of thermal expansion properties between explosive and binder, PBX has poor resistance to heat shock.

Macroscopically the recovered sample subjected to frozen combustion had an undulating, gooey appearance and was brown in color compared with the pristine white of unburned PBXN-5. Cross sectional views showed the presence of three characteristic zones including fusion zone, heat affected zone and base material. The fusion zone was characterized by uneven edges and internal void regions which resembled bubbles. This may be the result of decomposition gases trapped in the melt as it solidified. The heat affected zone was dominated by extensive interfacial debonding. The zone of base material resembled unheated, original materials. Fig.3 shows a plan view of recovered sample. Because part of binder has been destroyed by combustion, HMX crystals are exposed. The plan view is dominated by pits and pocks, especially for HMX crystals. At high resolution, some of the pits and pocks reveal ordered, columnar crystal growth, indicating possible phase transition from $\beta - HMX$ to $\delta - HMX$. Second harmonic generation(SHG) or powder X-ray diffraction (XRD) analysis is needed to clarify the possible phase transition..

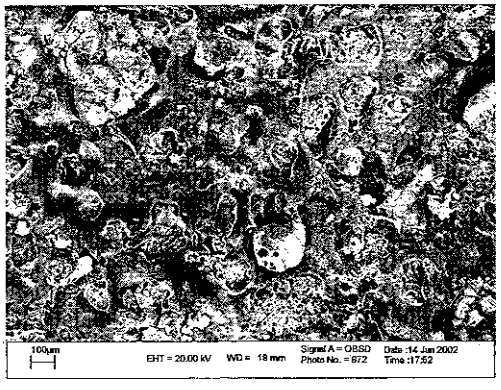

Fig.3 Plan view of PBXN-5 subjected to frozen combustion

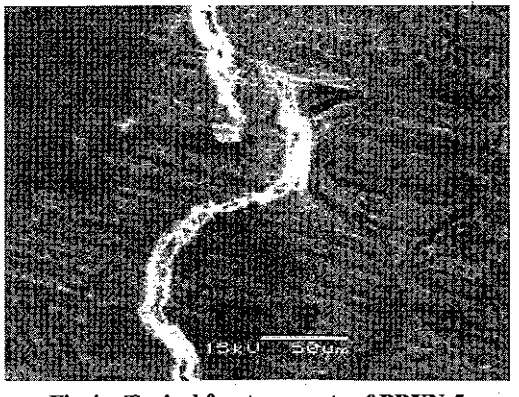

Fig.4 Typical fracture route of PBXN-5 under quasi-static Brazilian test

3.3 Mechanical Aggressions

In quasi-static Brazilian test, the failure first started at several independent sites, usually around the boundaries of the larger HMX filler particles and formed microcracks. These microcracks linked up into larger

cracks and finally induced the rupture of samples. The fracture route predominantly followed the boundaries of HMX particles (see fig.4), while crystal fracture was very rare. The fracture surfaces in Brazilian test were rough, with some partiles completely pulled out. Larger particles exhibited clean crystal faces due to the debonding of binder from particles. In contrast, the finer particles appeared rough having binder fibrils fractured due to extensive deformation. Fig. 5 shows a typical fractograph of PBXN-5 under Brazilian test, in which both clean crystal surfaces and binder filaments can be observed. Examination of fracture surfaces also showed that the failure predominantly followed the boundaries of the explosive fillers due to interfacial debonding, with few fractured crystals.

The fracture surface of PBXN-5 under uniaxial quasi-static compresson was markedly different from that under Brazilian test, as shown in Fig.6. In compression, extensive crystal fracture can be observed, causing the formation of a large number of smaller particles. The tensile failure stress in Brazilian test and the compressive failure stress in compression test were estimated as 0.8 MPa and 8.8 MPa respectively. The explosive particles are separated in tension, so the binder plays a predominant role. While in compression the explosive particles are pressed and contacted with each other due to the extremely high concentration of explosive crystals, so the explosive particles play a more important role. This causes a great difference between the tensile strengths and compressive strengths of PBX.

Fig.7 shows a plan view of PBXN-5 damaged by ultrosonic waves for 5 minutes. A lot of pull-outs can be observed and some of them are very large and involve many grains. The balls of the initial molding powder may have an influence on that process. The grains do not seem to be broken and no intragranular cracks can be detected. Besides, nearly all the contacts between the grains are debonded. The above results show that ultrasonic waves are able to seriously damage pressed PBXN-5. Ultrasonic waves are frequently used in polishing process to clean the samples before microscopic examination, however it seems to be inappropriate for PBX materials.

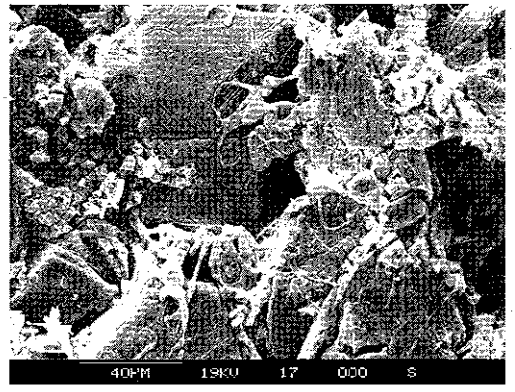

Fig.5 Fractograph of PBXN-5 under Brazilian test

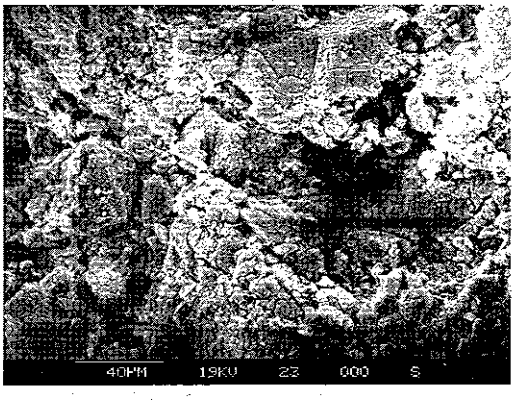

Fig.6 Fractograph of PBXN-5 under compression

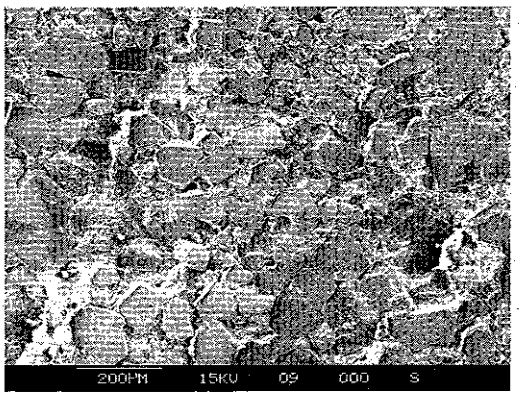

Fig.7 Plan view of PBXN-5 submitted to ultrasonic waves

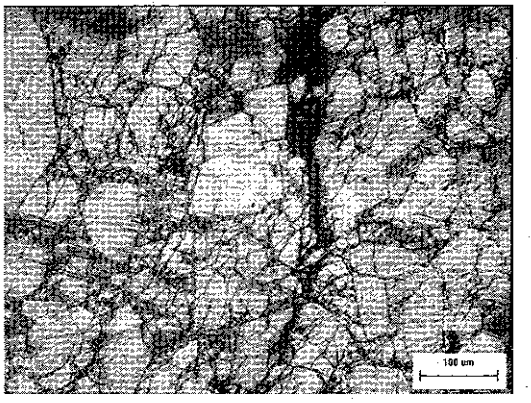

Fig.8 Plan view of PBXN-5 damaged by low-velocity impact

Fig.8 shows a plan view of PBXN-5 damaged by a steel projectile at an impact velocity of 108 m/s. Impact direction is from the right of the figure. The impact induced a large number of microcracks in explosive crystals. A vertical larger crack across explosive crystals can also be observed. Particle size distribution analysis showed that after impact the average size of HMX crystals was 114.5 μm, showing a decrease of 15 μm, compared with 130.8 μm before impact. At this impact velocity, no fragments were observed in PBXN-5 samples. However at the same velocity, Composition B (containing TNT 40 % and RDX 60 % by weight) was severely fragmented. This demonstrates that despite the low concentration of binder, PBXN-5 exhibits better resistance to impact loading than Composition B.

4 Conclusions

Scanning electronic microscopy and polarized light microscopy prove to be efficient tools to characterize the microstructure of plastic bonded explosive. The microstructure of pressed PBX is quite different from that of molding powder. The first step of pressing is elimination of large voids by deformation of the prills. Few crystal fractures occur in this step. As the pressure increases, considerable crystal fractures are induced. The cracking allows elimination of intragranular voids. The characteristic initial damage modes present in pressed PBX include intragranular voids, crystal fractures, interfacial debonding and deformation twinning. The high concentration of explosive crystals results in extensive particle-to-particle contacts, which in return causes extensive fractures during pressing. When PBXN-5 is submitted to low temperature freezing, cracks can be initiated. When PBXN-5 is subjected to frozen combustion, extensive debonding occurs. Different failure modes correspond to different mechanical insults. Interfacial debonding is the predominant mechanism under quasi-static Brazilian test, and crystal fracture is very rare. Extensive crystal fractures are present in compression. Low velocity impact also induces extensive crystal fractures. Despite the low concentration of binder, it still noticeably improves the mechanical properties of PBXs. Ultrasonic waves may induce severe damage to PBX, causing extensive debonding and even pull-outs.

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