J Mater Sci (2007) 42:5272–5280
DOI 10.1007/s10853-006-0387-y

Microstructure, deformation and failure of polymer bonded explosives

Pengwan Chen · Fenglei Huang · Yansheng Ding

Received: 18 October 2005 / Accepted: 1 May 2006 / Published online: 31 January 2007

Abstract Polymer bonded explosives (PBXs) are highly particle filled composite materials comprised of explosive crystals and a polymeric binder (ca. 5–10% by weight). The microstructure and mechanical properties of two pressed PBXs with different binder systems were studied in this paper. The initial microstructure of the pressed PBXs and its evolution under different mechanical aggressions were studied, including quasi-static tension and compression, ultrasonic wave stressing and long-pulse low-velocity impact. Real-time microscopic observation of the PBXs under tension was conducted by using a scanning electron microscope equipped with a loading stage. The mechanical properties under tensile creep, quasi-static tension and compression were studied. The Brazilian test, or diametrical compression, was used to study the tensile properties. The influences of pressing pressures and temperatures, and strain rates on the mechanical properties of PBXs were analyzed. The mesoscale damage modes in initial pressed samples and the samples insulted by different mechanical aggressions, and the corresponding failure mechanisms of the PBXs under different loading conditions were analyzed.

P. Chen (✉) · F. Huang
National Key Laboratory of Explosion Science and Technology, Beijing Institute of Technology, Beijing 100081, China
e-mail: pwchen@bit.edu.cn

Y. Ding
Institute of Mechanics, Chinese Academy of Sciences, Beijing 100080, China

Introduction

The study of the microstructure and mechanical properties is of crucial importance for the design, safety evaluation, and life prediction of energetic materials. Energetic materials may be subjected to different external stimuli, e.g. compression, tension and impact, resulting in the change of microstructure and even mechanical failure. Damage caused by external stimuli influences not only the mechanical properties, but also the sensitivity, combustion and even detonation behavior of energetic materials. Studies have shown that the presence of defects, such as pores or cracks, can greatly increase the sensitivity of 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.

Polymer bonded explosives are highly particle filled composite materials comprised of 90–95% by weight of powerful secondary explosive crystals held together by a polymeric binder (5–10% by weight). They are used in both civil and military applications when very high performance is required. The study of the mechanical properties and failure mechanisms of PBXs has drawn tremendous attention in recent years [1–4]. Low strengths and safety concerns bring additional difficulties in preparing samples and conducting mechanical tests of PBXs. Optical and electronic microscopic examinations are effective methods to study the microstructure and its evolution and to reveal the mesomechanical deformation and failure mechanisms of explosive materials. Different microscopic methods including scanning electron microscopy (SEM), environmental scanning electronic microscopy (ESEM)

[5, 6] and polarized light microscopy (PLM) [7] have been successfully used in this regard. The Brazilian test, or diametrical compression, in which a disc of material is loaded diametrically in compression, has been used to generate tensile stress and estimate the tensile failure stress of a material without the inconvenience of uniaxial dumbbell testing [8]. Application of this method to explosives has been undertaken by some researchers [3, 5, 6, 9–11]. The researchers at Cavendish Laboratory, Cambridge, UK have done extensive work in this field by introducing some sophisticated techniques [12–15]. They have used some optical techniques such as moire or speckle to quantify the strains in the binder and the explosive. In addition, they have obtained dynamic data by loading their Brazilian test geometry in a Hopkinson bar. At present, the correlation between the microstructure and the mechanical properties of PBXs, and the mesomechanical mechanisms of damage, deformation and failure of PBXs are not fully understood. The experimental results presented in this paper may provide some further insight into these issues.

Experimental

Two PBXs investigated in the experiment were based on the explosive *β-HMX* (Cyclotetramethylene-tetranitramine). The two PBXs designated as PBX-A and PBX-B contain HMX 94.5–95.0% (type B) and a fluoelastomer binder 5.0–5.5% by weight. The binder used in PBX-A was F_{2641}, a copoly(vinylidene fluoride-chlorotrifluor ethylene) and the binder in PBX-B was F_{2311}, a copoly(vinylidene fluoride-hexafluoropropylene). Molding powder was produced by a conventional slurry process, in which the explosive crystals agglomerated as they were coated with the binder dissolved in an organic solvent. The molding powder was compressed in a mold to produce a consolidated piece with a desired geometry under different temperatures and pressures. For hot pressing, the temperature was 100 °C and the time duration of pressing was 1.5 h. Different pressing pressures were used, ranging from 50 MPa to 200 MPa.

To examine the evolution of microstructure under different mechanical conditions, the pressed PBX samples were subjected to different mechanical tests, including the Brazilian test, uniaxial compression, ultrasonic wave stressing and long-pulse low-velocity impact. Ultrasonic waves are basically a mechanical stimulus which is very different from shocks or impacts. In ultrasonic tests, the samples were submitted to ultrasonic waves in a bath with distilled water for 5 min. The frequency and power of the ultrasound were 30 kHz and 100 W respectively. A long-pulse low-velocity gas gun with a gas buffer was also developed and used to apply dynamic loading, through which the time duration of dynamic compression can be extended to several milliseconds. The samples were confined in a steel tube during impact. A full description of the loading assembly can be found in our previous work [16].

SEM was used to examine the microstructure of the PBX samples. Low toughness and safety concerns bring additional difficulties in preparing the PBX samples for microscopic examination. The method of sample preparation here is similar to that of Palmer et al. [3]. Samples were first ground using standard fine silicon carbide papers (800 grit) 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 during polishing, especially the damaged or fractured samples, the samples were first potted in commercial low-viscosity epoxide mounts with traditional amine hardening agent and then cured. To further examine the microstructure of explosive particles, a thorough etching method was adopted. Pressed or damaged PBX samples were kept in a selected solution, e.g. iso-methyl butyl ketone for enough time to totally remove the binder and leave the explosive particles alone, and then the explosive particles were collected for further examination. This method has proven to be an efficient way to reveal the microstructure of explosive particles [16].

Figure 1 shows the loading geometry of the Brazilian test. To eliminate contact failures, curved, rather than flat anvils, were used in the Brazilian test. The use of curved anvils has proven to be applicable in the study of PBXs [3, 6]. The imposition of a compressive

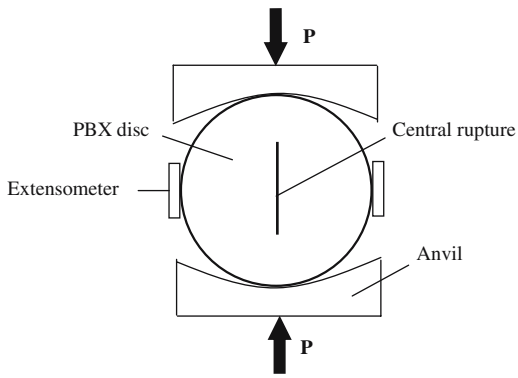

Fig. 1 Brazilian test geometry

load, P, from the two curved anvils on a disc of material causes a tensile failure on the vertical axis of loading. According to simple elasticity theory, if the influences of contact width are neglected, the tensile stress at the center of the disc is a maximum given by [8, 11]

$$\sigma = \frac{2P}{\pi Dt} \quad (1)$$

where D and t are the sample diameter and the thickness respectively. The average horizontal diametrical strain can be measured by two extensometers. Both the creep deformation and the quasi-static tensile deformation under different strain rates were measured. In general, failure occurs along the vertical axis in the Brazilian test, which brings convenience for real time examination of the microstructural evolution. In order to do this, the Brazilian test was also carried out in a SEM equipped with a loading stage. Uniaxial compression under different strain rates was also conducted to evaluate the compressive properties of the PBXs. The sample sizes were ϕ10 mm × 12 mm for compression and ∅20 mm × 10 mm for the Brazilian test and low velocity impact respectively.

Results and discussion

The pressing pressure greatly influences the microstructure of pressed PBXs. At low pressing pressures, a lot of intergranular voids are still present. With the increase of pressure, the intergranular voids decreases, however crystal fractures also occur. When the pressing pressure reaches 100 MPa, extensive crystal fractures can be observed. Please note that the newly formed microcracks in pressed PBXs are randomly distributed without any orientation. Figure 2 shows a micrograph of PBX-A with a porosity of 2.5% pressed under 100 MPa and 100 °C. It is clearly shown that various forms of defects are present in pressed PBX-A, 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. Figure 3 shows the initial microstructure of the HMX particles in the molding powder of PBX-A before pressing, demonstrating absence of obvious microcracks or other defects. Figure 4 shows the microstructure of the recovered HMX particles after pressing at 100 MPa, clearly revealing the presence of extensive crystal fractures and a lot of microcracks. The crystal facture indicated by the black arrow is clearly caused by particle-to-particle contact. It is reasonable to conclude that crystal fractures are mainly associated with particle-to-particle contacts due to an extremely high concentration of explosive crystals in PBXs. Another important phenomenon in pressed PBX is the presence of deformation twinning, an evidence of plastic deformation of explosive crystals during pressing. In the study of the deformation and fracture of *β-HMX*, Palmer and Field [17] observed the existence of elastic twinning and permanent twinning and discussed the influences of deformation twinning on the fracture of *β-HMX*. The presence of deformation twinning demonstrates that explosives crystals can undergo a certain extent of deformation. More experiments are needed to quantify the deformation ability of explosive crystals. The above results also reveal that considerable initial damage is present in hot pressed PBX. Pressing not only consolidates the molding powder, but also induces new damage to explosive crystals.

Figure 5 shows a plan view of PBX-A damaged by ultrasonic waves for 5 min. A lot of pull-outs can be observed and some of them are very large and involve

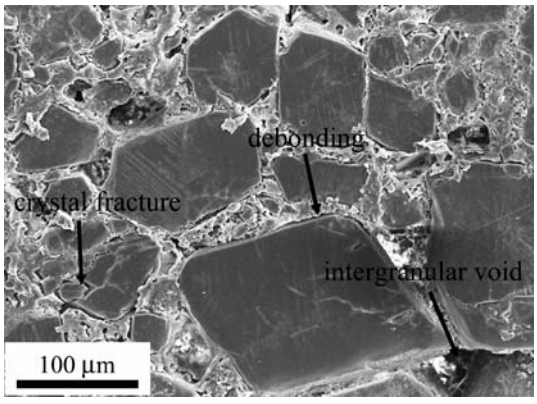

Fig. 2 Microstructure of pressed PBX-A

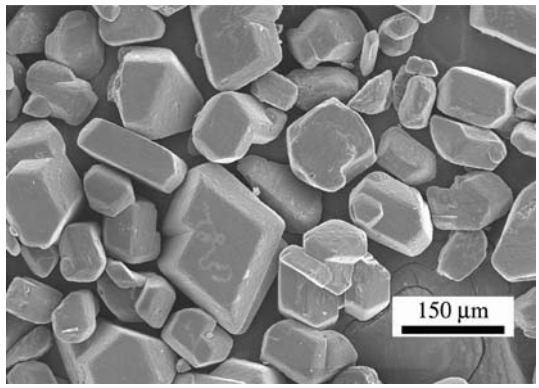

Fig. 3 Initial microstructure of HMX crystals in molding powder of PBX-A

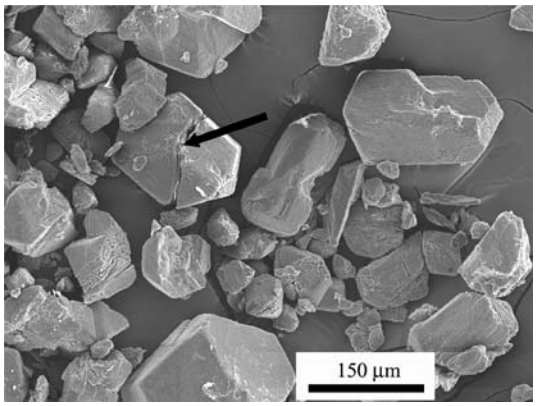

Fig. 4 Microstructure of the HMX crystals in pressed PBX-A

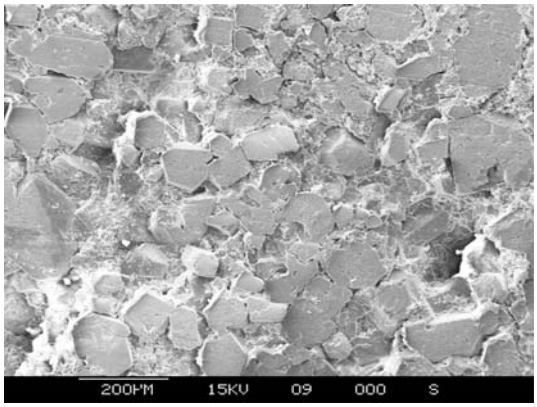

Fig. 5 Microstructure of PBX-A subjected to ultrasound

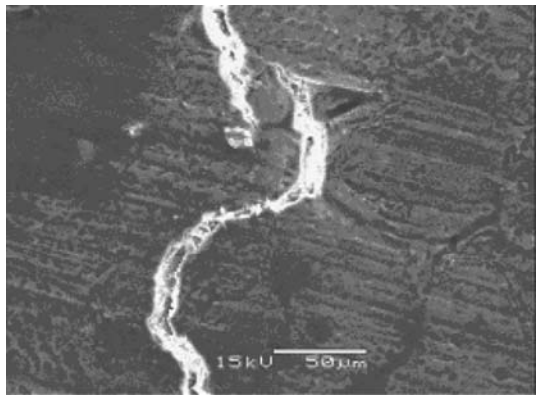

Fig. 6 Cracking along particle boundaries

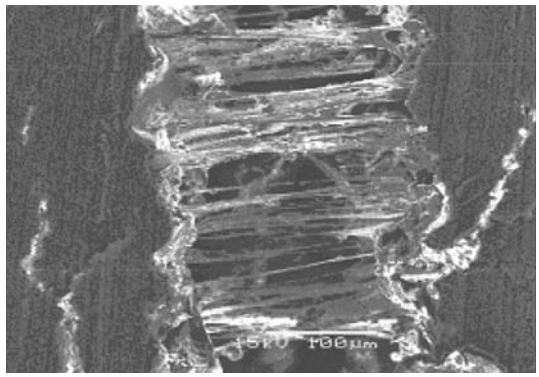

Fig. 7 Binder filaments bridging crack walls

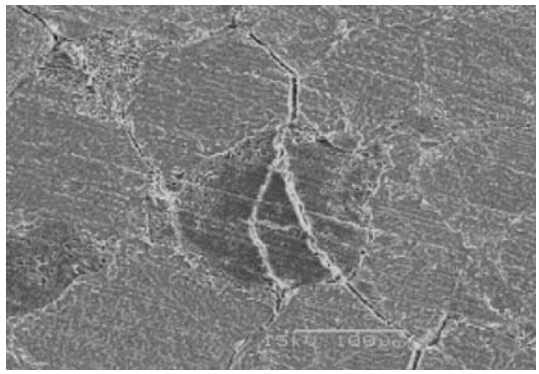

Fig. 8 Crystal fracture on a fracture route

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 the pressed PBXs. Similar results are also observed by Demol et al. [18]. Ultrasonic waves are frequently used in polishing process to clean the samples before microscopic examination, however it seems to be inappropriate for PBX materials.

The failure processes of PBXs observed in the real time microscopic examination of Brazilian tests are similar to those reported by Palmer et al. [3] and Rae et al. [6]. The results show that the failure first starts at several independent sites, usually around the boundaries of the larger HMX filler particles and forms microcracks. These microcracks links up into larger cracks and finally induces the rupture of the samples. The initial damage such as uncoating and debonding generated during the manufacture of molding powder and pressing are usually the origins of failure. Figures 6–8 shows the results of real time microscopic examination of PBX-A in Brazilian tests. Fig. 6 shows that a crack is extending along the boundaries of larger particles. Figure 7 shows some extended binder filaments bridging the crack surfaces, demonstrating that the binder undergoes considerable deformation. Plan views of the fracture routes show that crystal fracture is very rare, but may appear due to the orientation of some larger particles perpendicular to an advancing crack path (see Fig. 8).

PBX-A and PBX-B exhibit different morphology of fracture surfaces due to different mechanical properties resulted from different binder used. The fracture surfaces of PBX-A in creep and quasi-static Brazilian tests are relatively rough, with some particles completely pulled out. Larger particles exhibit clean crystal faces due to the debonding of binder from particles. In contrast, the finer particles appear rough having binder fibrils fractured due to extensive deformation. Figure 9 shows a typical fractograph of PBX-A under quasi-static Brazilian tests, 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. Some microcracks can also be observed on some particles (see black arrows in Fig. 9). Due to the low stress amplitude applied in Brazilian tests, it is reasonable to assume that these microcracks are mainly caused during pressing. The failure of PBX-B occurs at a higher stress level and the fracture surfaces are relatively smooth. Figure 10 shows a typical fractograph of PBX-B under quasi-static Brazilian tests, revealing the presence of more fractured crystals. F_{2311} exhibits better mechanical properties than F_{2641} with a larger tensile strength and elongation at failure. More importantly, when F_{2311} is used for HMX-based PBXs, a better coating quality and interfacial properties can be obtained according to experimental and theoretical studies [19, 20]. The microscopic examination in our experiment clearly shows that F_{2311} can produce better interfacial bonding and macromechanical properties, which can be further supported by the results of mechanical tests discussed below.

The morphology of fracture surfaces of PBXs in quasi-static Brazilian tests is also influenced by strain rates. Microscopic examination of PBX samples fractured under higher strain rates shows that more crystal fractures occur with the increase of strain rates. To further investigate the influence of even higher strain rates, a dynamic Brazilian test was conducted. The sample disc was loaded by a drop weight, in which the falling weight was 5.0 kg and the drop height was 0.8 m. Figure 11 shows a typical fractograph of PBX-A under the dynamic Brazilian test, demonstrating the presence of extensive crystal fractures.

Figure 12 shows a typical fractograph of PBX-A in quasi-static compression test. Extensive crystal fractures can be observed, causing the formation of a large number of smaller particles. The corresponding tensile failure stress at the disc center in the Brazilian test and the compressive failure stress in compression test are estimated as 1.2 and 9.8 MPa respectively, revealing that explosive particles with initial microcracks caused by pressing may not fracture under a tensile stress of

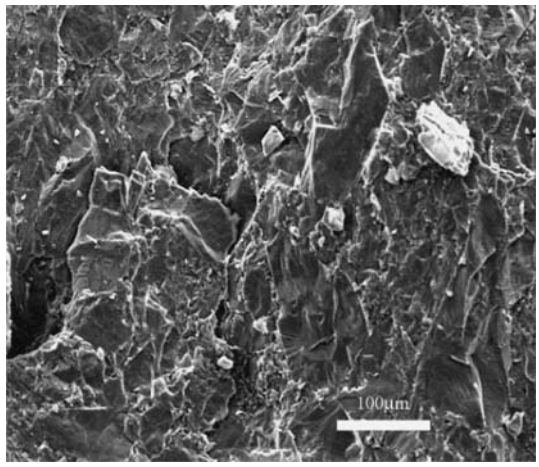

Fig. 10 A typical fractograph of PBX-B under the quasi-static Brazilian test

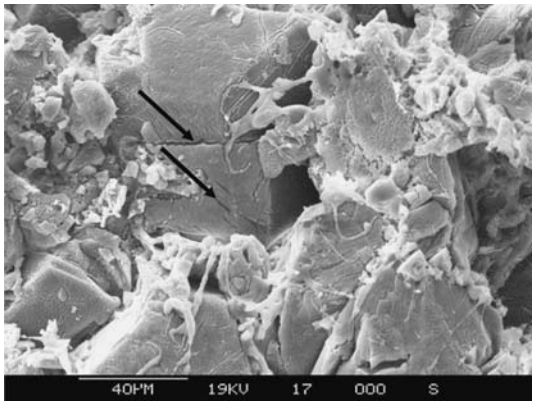

Fig. 9 A typical fractograph of PBX-A under the quasi-static Brazilian test

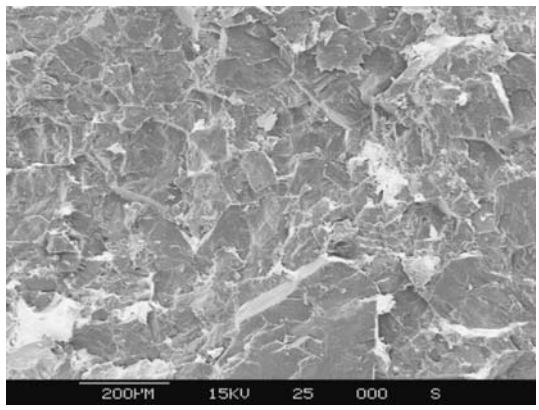

Fig. 11 A typical fractograph of PBX-A under the dynamic Brazilian test

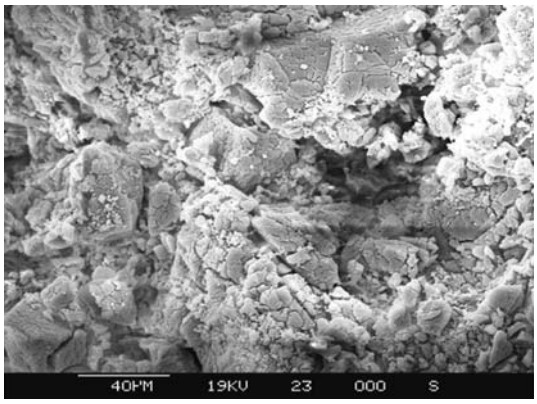

Fig. 12 A typical fractograph of PBX-A under compression

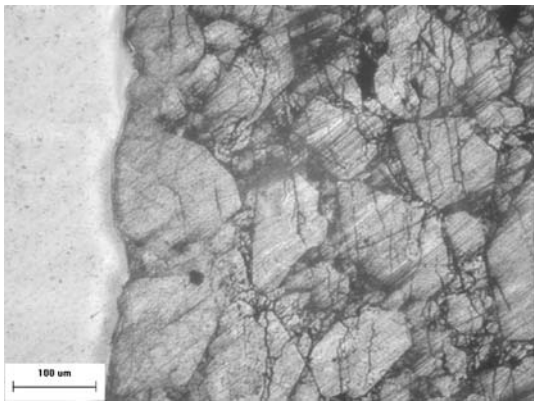

Fig. 14 Microstructure of impact damaged PBX-A (far field)

1.2 MPa, while may fracture under a compressive stress of 9.8 MPa. Determination of the critical failure tensile and compressive stresses of explosive crystals is crucial to understand the mesomechanical phenomena of PBXs. Unfortunately the data of mechanical properties of explosive crystals are very scarce at present due to the difficulties in experiments.

Figures 13–14 show the plan-view images of damaged PBX-A impacted at a velocity of 171 m/s with the projectile incident from right. Figure 13 is a plan-view image of the region near the incident surface (near field), while Fig. 14 is an image of the region near the back surface (far field). Though visible macrocracks are not observed in damaged PBX-A samples, a lot of microcracks are present. The figures also show that the density of microcracks in far field is larger than that in near field, implying that impact induced damage is not homogeneously distributed. Please note that the substantial preferred orientation of the microcracks and the coalesced larger cracks in damaged PBX-A is in the vertical direction in contrast to the random distribution in initial pressed PBXs.

Figure 15 shows the morphology of HMX particle after impact damage. It clearly shows that impact induces a lot of cracking, resulting in a large number of microcracks and the formation of smaller particles. To quantitatively evaluate the particle fractures, the HMX powder was collected for particle size distribution analysis by using the above-mentioned thorough etching method. The results show that before impact the average particle size of HMX is 119.3 μm, however after impact at a velocity of 164.4 m/s, the average particle size decreases to 89.4 μm due to extensive particle fractures. At the above impact velocity, no fragments were observed in PBX-A samples. However at the same velocity, cast Composition B (containing TNT 40% and RDX 60% by weight) was severely fragmented [21]. This demonstrates that despite a low concentration of binders, PBXs exhibit better resistance to impact loading than Composition B.

Due to a high concentration of filled particles, the scatter of mechanical test data of pressed PBXs is usually higher than conventional particle-filled composites. Statistical results in our tests show that the

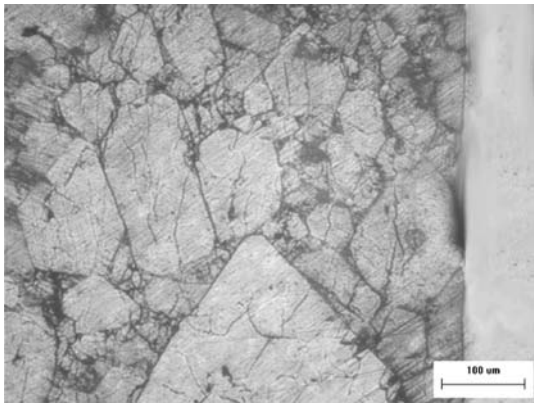

Fig. 13 Microstructure of impact damaged PBX-A (near field)

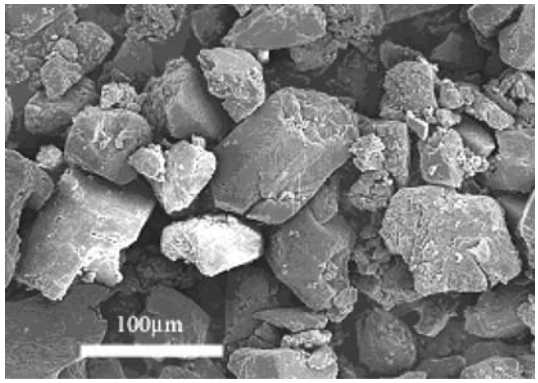

Fig. 15 Morphology of HMX particle after impact

standard deviation of mechanical data of pressed PBXs was 5–10%. Typically a total of six replicates per formulation were used in the mechanical test. Figure 16 shows some tensile stress-strain curves of the two PBXs obtained by Brazilian tests under a strain rate of 4.2×10^{-4} s^{-1}, in which curve c represents the result of PBX-A pressed under 200 MPa and 100 °C, while curves a, b, d and e represent the results of PBX-B pressed under different pressures (ranging from 50 MPa to 200 MPa) and different temperatures (100 and 20 °C). The stresses are calculated using formula (1). The results show that the tensile mechanical properties are greatly influenced by pressing pressures. The tensile strengths of PBX-B increase from 1.5 ± 0.1 MPa to 2.2 ± 0.2 MPa with an increase of the pressing pressure from 50 MPa to 200 MPa. The pressing temperatures have a greater influence on the mechanical properties of PBXs than the pressures. The tensile strength of cold pressed PBX-B (20 °C) is about 1.0 MPa, while the strength of PBX-B pressed at an elevated temperature 100 °C is increased to about 2.2 MPa. PBX-B exhibits a larger tensile strength and failure strain than PBX-A, implying that the mechanical properties of PBXs are largely influenced by the binder despite its low concentration.

Both the tensile and compressive properties of PBXs are influenced by strain rates. At a strain rate of 3.3×10^{-5} s^{-1}, the tensile failure stress of PBX-A is about 0.9 MPa, compared with about 1.4 MPa at a strain rate of 4.2×10^{-4} s^{-1}. Figure 17 shows the compressive stress strain curves of PBX-A under different strain rates, in which the maximum stresses in the curves correspond to uniaxial compressive strengths.

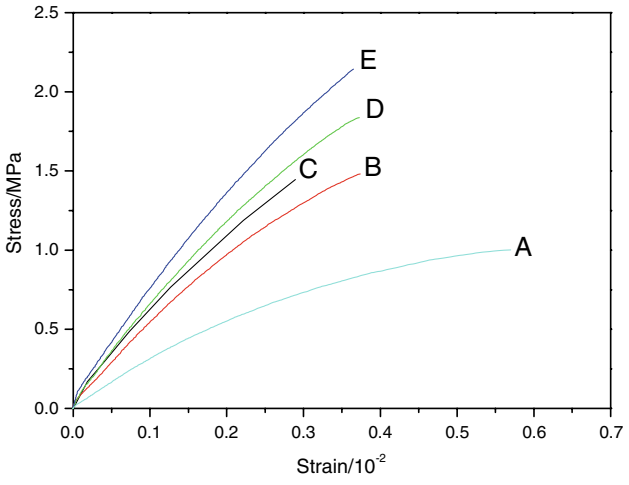

Fig. 16 Tensile stress strain curves of PBXs. (**A**) PBX-B(200 MPa, 20 °C); (**B**) PBX-B(50 MPa,100 °C); (**C**) PBX-A(200 MPa,100 °C); (**D**) PBX-B(100 MPa,100 °C); (**E**) PBX-B(200 MPa,100 °C)

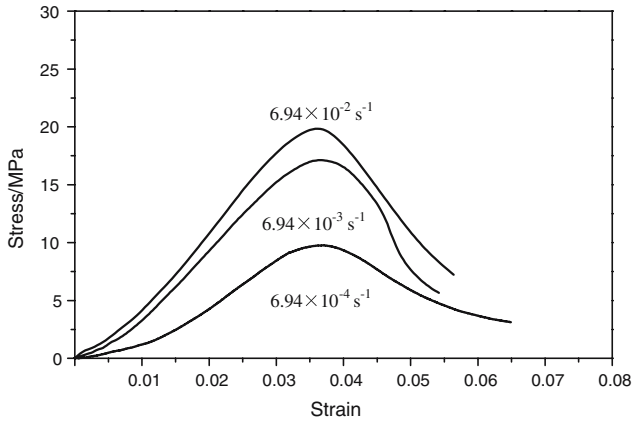

Fig. 17 Compressive stress strain curves of PBX-A under different strain rates

At a strain rate of 6.9×10^{-4} s^{-1}, the compressive strength is 9.8 ± 0.6 MPa; while at a strain rate of 6.9×10^{-2} s^{-1}, the compressive stress increases to 19.8 ± 0.9 MPa. The figure also shows that the strains corresponding to the maximum stresses do not change noticeably. Wiegand [22] reported similar results in the study of the compressive properties of PBXs and other explosives and proposed a failure criterion of constant critical strains for explosives and propellants.

Mechanical tests demonstrate that the tensile strengths of PBXs are much lower than the compressive strengths, about one tenth of the compressive strengths. The microstructure of PBXs may account for this result. In general, the modulus of the binder is several orders of magnitude lower than that of the crystalline explosive, and the strength of the crystalline explosive is much higher than that of the binder. The explosive particles are separated in tension, so the

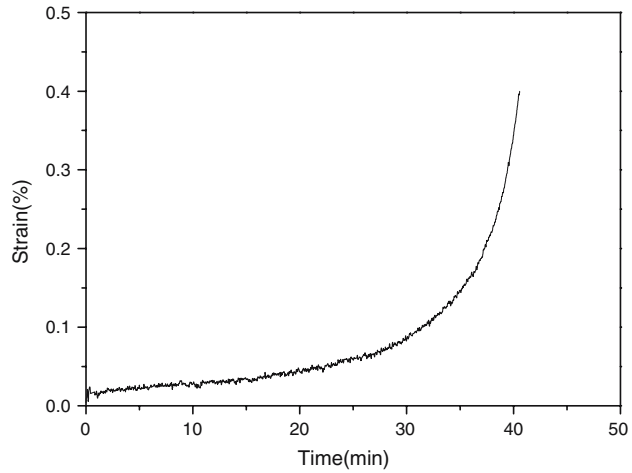

Fig. 18 A tensile creep curve of PBX-A

binder plays a predominant role during deformation. While in compression the explosive particles are pressed and contacted with each other due to the extremely high volume faction of explosive crystals, so the explosive particles play a more important role during deformation.

Figure 18 shows a tensile creep curve of PBX-A obtained under a constant stress of 0.71 MPa at the disc center, revealing similarity to a typical creep curve of a pure high polymer. An instantaneous elastic–plastic response, the second stage named as steady state creep stage and the third stage named as accelerating creep stage can be observed, while the first stage is not obvious. According to the damage mechanics, the third stage is the result of interaction of the steady-state creep and the damage creep, and it is mainly the damage creep that contributes to the ultimate failure. The time dependent creep behavior of PBX is mainly due to the contribution of the binder, demonstrating that PBXs still exhibit viscoelastic properties despite a low concentration of binders.

The failure strains in quasi-static tension, tensile creep and quasi-static compression are relatively small, revealing the nature of brittle material of PBX. The extremely high concentration (over 90%) of brittle explosive particles may account for this.

In the present study, the tensile strain obtained from Brazilian tests reflects an average tensile strain along horizontal diameter. To obtain the whole field deformation of the PBX samples and further investigate the mesomechanical mechanisms of deformation and failure of PBXs, we have applied high resolution moiré interferometry to study the deformation of PBXs. In addition, application of high resolution AFM scanning moire method to the study of deformation and failure PBXs is currently underway. The results will be discussed elsewhere.

Conclusions

The characteristic initial damage modes present in pressed PBXs 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. Ultrasonic waves may induce severe damage to PBXs, causing extensive debonding and even pull-outs. Impact may induce more extensive particle fractures, resulted in the change of particle morphology and particle size distribution. Impact-induced damage is not homogenously distributed, with the far field more severely damaged.

PBXs are brittle materials with low strengths and low fracture strains. The tensile strengths of PBXs are much lower than the compressive strengths. The mechanical properties of PBXs are largely influenced by the binder. Despite a low concentration of binder, PBXs exhibit rate dependent and time dependent mechanical behavior. The Brazilian test has proved to be a useful and efficient method in studying the deformation and failure of PBXs.

Different failure modes correspond to different loading conditions. Interfacial debonding is the predominant failure mode in quasi-static tension and tensile creep under the Brazilian test. In the Brazilian test, the initial failure tends to start around the edges of larger filler particles and often occurs at several independent sites simultaneously. The increase of strain rates causes more explosive crystals to fracture. In the dynamic Brazilian test, the predominant failure mode is crystal fracture. Extensive crystal fractures are also present in compression. Low velocity impact also induces extensive crystal fractures.

Acknowledgements The authors of this paper acknowledge the financial support from the National Natural Science Foundation of China under contract number 10002022 and Joint foundation of Chinese National Natural Science Committee and Chinese Academy of Engineer Physics under contract number 10076021.

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