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High-Speed Photographic Study of the Impact Response of Ammonium Dinitramide and Glycidyl Azide Polymer

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A high-speed photographic study has been performed of the rapid deformation of ammonium dinitramide (ADN) and glycidyl azide polymer (GAP) by drop-weight impact. ADN was found to be more sensitive than ammonium perchlorate. Both materials have been suggested as potentially useful oxidizers in explosive and propellant applications. ADN was also found to be sensitized by both hard high-melting point grits (60-μm borosilicate or Pyrex® glass) and brittle polymers. High-density polyethylene was found to suppress deflagration in this material. Heat-sensitive film was used to confirm that the events seen using high-speed photography were indeed deflagrations. GAP was found to be insensitive when impacted in this apparatus, even in the presence of additives or gas bubbles.

Introduction

COMPOSITE propellants are also called heterogeneous propellants and are essentially composed of a binder and an oxidizer.[1] The binder forms the matrix in which the solid oxidizer is suspended. It thus largely governs the mechanical properties of the propellants.[2] To obtain a high specific impulse I_{sp} from a propellant, a high oxidizer loading is necessary (Ref. 3, p. 85). However, a high loading of solids adversely affects the mechanical properties of the composite (Ref. 3, p. 105).

The most widely used oxidizer is ammonium perchlorate (AP). However, it suffers from a number of problems, both military (its exhaust signature makes tracking rockets or missiles relatively easy[4]) and environmental (the exhaust products contribute to acid rain and ozone layer depletion[5–7]). To overcome these shortcomings, the use of ammonium dinitramide (ADN) in place of AP has recently been suggested[8] (Fig. 1, the particle size of the tested powder was determined by sieving: all the powder was retained by a 500-μm sieve and 10% passed through a 699-μm sieve). ADN exhibits high-temperature stability,[4] high-energy density, and does not produce smoke or halogens.[9–12] It is thus a prime candidate as an oxidizer for composite propellants.

There are a number of binders or hydrocarbon fuels, such as polypropylene glycol (PPG),[13] carboxy terminated polybutadiene (CTPB),[14] lactone terminated polybutadiene (LTPB),[15] hydroxy terminated polybutadiene (HTPB),[16] and poly-(butadiene-acrylic acid-acrylonitrile terpolymer) (PBAN)[14] among others, and the choice depends upon availability, cost, ease of processing, and I_{sp} required.[17] HTPB is regarded as the best binder for composite propellants (ca. 91% filling has been reported[15]). Some energetic binders such as glycidyl azide polymer (GAP) (Fig. 2), HHTPB, poly(NIMMO), and poly(GLYN) have also been reported recently,[18–21] with the aim of enhancing the I_{sp} of rocket propellants without adversely affecting their mechanical properties. The use of GAP for propellants results in greater safety in handling, high burning rates, high I_{sp}, and improved mechanical properties.[22,23]

There have been numerous published reports on GAP, including its synthesis,[24–27] structure, thermal behavior (including decomposition),[28–30] physical, chemical, and explosive properties.[31,32] GAP-based propellants have also been evaluated.[23,28,33–39] Similar information has also been published on ADN.[4,10–13]

A literature survey on ADN and GAP indicated that a high-speed photographic study of the initiation of explosion by impact of these explosives had not been reported in the open literature, though presumably laboratories producing them have performed routine sensitivity tests. Therefore, the aim of this investigation was to obtain data on impact initiation and sensitization mechanisms of ADN and GAP by Pyrex (a borosilicate glass) grits and brittle polymers using a drop-weight machine (mass 5.5 kg), combined with high-speed photography (Fig. 3).

A study of the effects of additives is important because they control the sensitivity of explosive mixtures. In some cases, it may be advantageous to increase sensitivity, e.g., in some commercial explosives. However, in other cases it is important to decrease sensitivity from the hazard point of view.

Hard high-melting point grits sensitize energetic materials by creating hot spots through frictional mechanisms.[40–42] Brittle amorphous polymers can sensitize energetic materials because their failure can be quite violent, liberating a great deal of heat in shear bands and at crack tips.[43,44] Heat-sensitive film can be used to obtain spatial information on hot spot generation.[45–47] More ductile polymers also reach a temperature high enough to expose the film (about 200°C), but not usually high enough locally to initiate energetic materials, especially if the polymer is semicrystalline as the latent heat of melting absorbs the energy released.[44]

Experimental Procedures

Drop-weight machines have been widely used since at least the beginning of the 20th century[48] to rank the impact sensitivity of both solid (e.g., Refs. 49–59) and liquid (e.g., 42, 60–62) energetic materials. In the standard configuration, the material is placed between hardened steel anvils and deformed by the impact of a weight falling on the upper anvil.[48,51,56,63,64] The major problem with such machines is that different rankings in explosive sensitivity are obtained by different laboratories.[65,66] Coffey and DeVost[65] argue that the major reason for this is that the kinetic energy of the falling weight is partitioned differently on impact in the various machines between plastic

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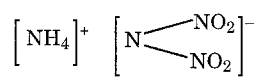

$[NH_4]^+[N(NO_2)_2]^-$

Fig. 1 Chemical formula of ADN.

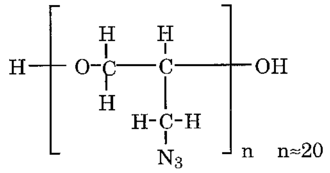

Fig. 2 Chemical formula of GAP.

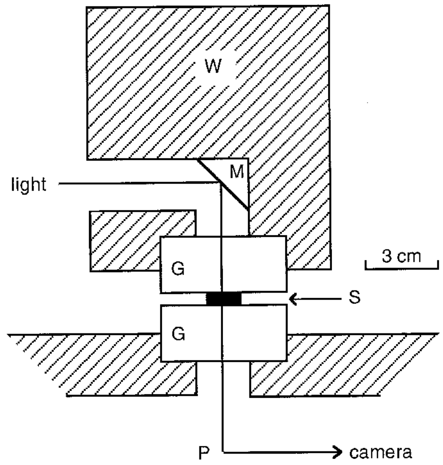

Fig. 3 Schematic diagram of high-speed photography apparatus.[72] *W*, weight; *M*, mirror; *G*, glass anvils; *S*, specimen; *P*, prism. Field of view is 22 mm in diameter.

deformation of the sample and stored elastic energy in the rest of the apparatus (though Kondrikov[59] is less pessimistic about comparisons between various machines used to rank explosive sensitivity). A review of the various machines used to test the hazard sensitivity of energetic materials has recently been published by Duffy and Mellor.[67]

For this reason, it was suggested by Coffey and co-workers[68–70] that reproducibility could be achieved by raising the impact speed so that the energetic sample initiates before significant elastic energy is stored in the rest of the apparatus.[65,71] We have considered implementing this suggestion, but concluded it was impractical for our glass-anvil drop-weight machine, the purpose of which is to enable hot-spot initiation mechanisms to be visualized (e.g., Refs. 44 and 72–76), rather than to rank explosives for their sensitivity. We have, however, recently updated our drop-weight machine to improve planarity of impact and to allow novel loading cycles to be performed.[77–79] Drops can be performed up to a height of 1.3 m.

High-speed photography is performed using a continuous access AWRE C4 rotating mirror camera (minimum interframe time 5 μs). In the research reported here, an interframe time of 8 μs was typical. It should be noted that if the anvils survive the impact (which they usually do), the film may be exposed a second time (overwriting) as the light output from the flash tube decays away. This is not usually a problem, indeed, it can be an advantage as it allows direct comparison of initial and final states.[76]

Heat-sensitive film (3M type 370) was used without photography to confirm that what was seen on the high-speed sequences was indeed deflagration. The technique was first suggested by Coffey and Jacobs[45] as a means of recording the spatial distribution of energetic events in an explosive layer or a deforming crystal. However, in conjunction with high-speed photography it also allows hot spot temperatures to be estimated. This is because for very short-duration heat pulses (less than 100 μs) the heat-sensitive chemical layer on the acetate film turns yellowish brown, and the degree of darkening increases with the contact time until the film is fully blackened. Thus, the darkening is a function of both temperature and time. Calibration curves allowing temperature measurements to be made for rapid events may be found in Swallowe et al.[46]

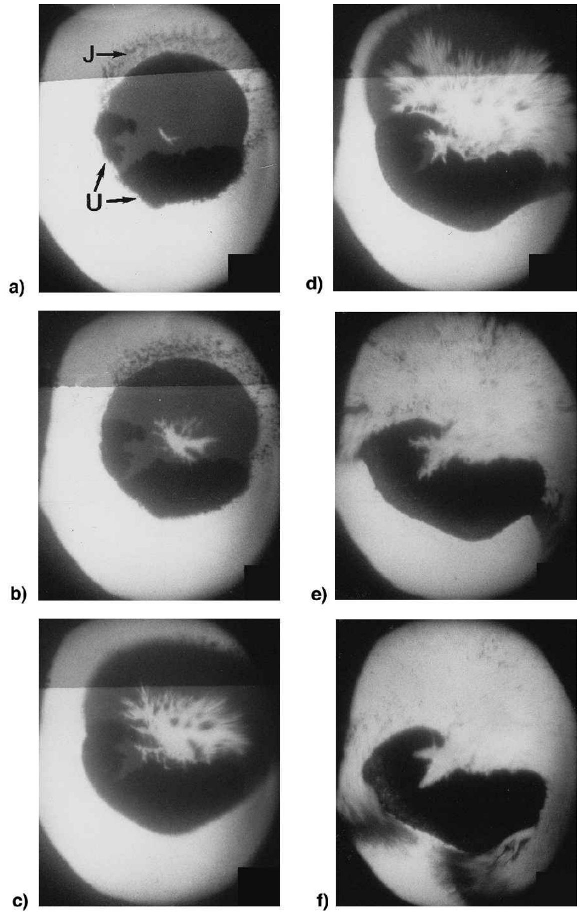

Fig. 4 Selected frames from a high-speed photographic sequence of the deflagration of ADN powder (drop height 1.3 m). Time from the instant of impact: a) 96, b) 112, c) 120, d) 128, e) 144, and f) 168 μs. The straight line and contrast differences in frames a–d are because of a developing error. The light gray area in the upper part of the specimen (as seen in frames a–c) is caused by overwriting.

However, it should be noted that the use of heat-sensitive film is an invasive technique, as the heat-sensitive compound is supplied on a compliant polymer backing. Thus, the mechanical conditions at the specimen/anvil interface are somewhat different to those when the film is not present. There may therefore be differences in explosive sensitivity between drops performed with and without heat-sensitive film. The damage to the film can be related to the violence of the event.

A related study has been performed on the impact response of novel energetic materials.[80]

Results

ADN

ADN deflagrated when impacted from a drop-height of 1.30 m, but failed to ignite when impacted from lower drop heights (1.0 and 0.65 m). Heat-sensitive film in contact with ADN powder was not discolored at these lower drop heights.

Figure 4 shows selected frames from a high-speed photographic sequence of drop-weight impact on an approximately 5-mg layer of ADN powder. As the apparatus is backlit (Fig. 3), the sample appears in silhouette; it originally had a diameter of approximately 9 mm. The early frames (not shown) showed compression and compaction of the layer followed by radial flow at a velocity of approximately 30 m s^{-1}. Jets of very small particles (labeled J in Fig. 4a) travel ahead of the main body of the sample. The jets form in the angle between the specimen and the anvils, and are a common feature (e.g., Refs. 72 and 73). They move preferentially in one direction because of a very slight asymmetry in the impact.

The bright spot near the center of the sample in Fig. 4a is an ignition event that quickly spreads throughout the sample in the top half of the frames. Again, because of the anvil surfaces not being perfectly parallel, the ignition moves preferentially in one direction rather than radially. This is reasonable as the more rapidly deforming material is likely to create more hot spots and, hence, to promote more rapid propagation in this direction. The products can be seen to expand upward (Figs. 4c and 4d) and then spread around the sample (Figs. 4e and 4f). The lower part of the sample was unconsumed. An indication of the amount of unconsumed explosive can be ascertained by examining the darker areas in Fig. 4a, labeled U, which are similar in shape, but slightly smaller than the dark area in Fig. 4f.

ADN was sensitized by both 60-μm Pyrex powder and the three brittle polymers studied: polycarbonate (PC), polystyrene (PS), and poly(methyl methacrylate) (PMMA). This means that the drop height necessary for deflagration was decreased and the number of initiation sites was increased by the presence of these additives. ADN was desensitized by being placed in contact with a disc of high-density polyethylene (HDPE) (semicrystalline, ductile polymers with a high latent heat of melting tend to suppress deflagration[44]). The polymer discs were placed on top of the powder and gently pressed down so that the top of the disc was at the same level as (or slightly higher than) the top of the powder.

The addition of 60-μm Pyrex glass particles caused a large number of ignition sites throughout the sample (Fig. 5), by frictional rubbing between the particles and the anvils. This process provides the hot spots for ignition of neighboring explosive particles. The first sign of ignition (labeled I) can be seen in Fig. 5b. This can be seen developing further in Fig. 5c, after which many more ignition sites form (Fig. 5d). Figure 5f shows extensive reaction around small areas of unconsumed material. An estimate of the amount of unconsumed material can be made by examining Fig. 5a, in which overwriting enables a direct comparison between the initial and final states of the sample (the light gray areas are the reaction channels). This correlated well with the unconsumed material found adhering to the anvils after impact.

Figure 6 shows selected frames from an experiment in which a PMMA disc was positioned at the center of a sample of ADN powder. The initiation of deflagration was delayed compared to the pure ADN powder because of the polymer disc bearing some of the load, which otherwise would have been borne by the powder. However, when the polymer eventually failed, the number of hot spots was greater than for the pure powder (fracture and shear banding in polymers can cause local hot spots sufficient to ignite energetic powders in physical contact with them[44,46]). Ignition events occur at the boundary between the PMMA and ADN, and these are labeled I in Figs. 6a and 6b. Reaction proceeds vigorously from these sites. Note in

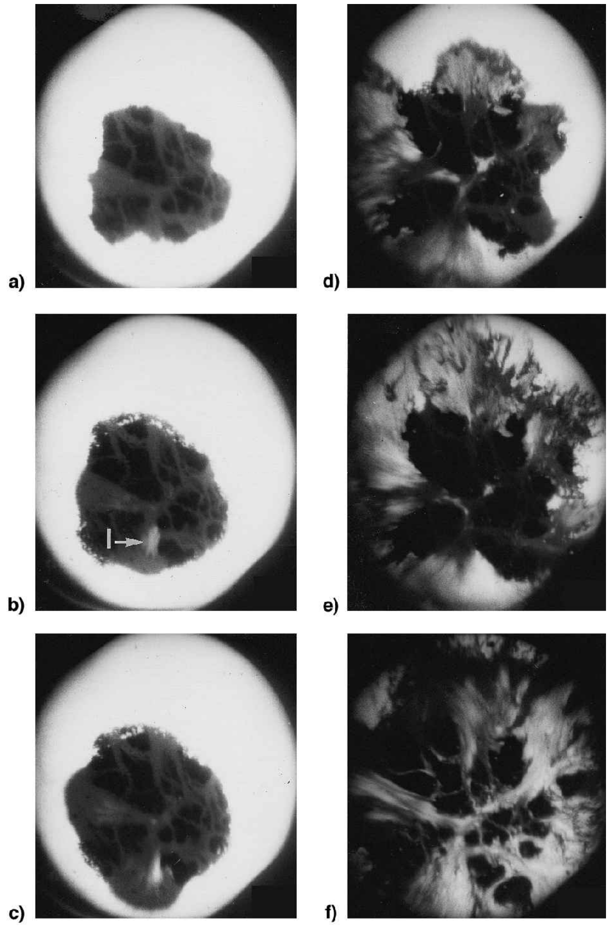

Fig. 5 Selected frames from a high-speed photographic sequence of the deflagration of ADN powder mixed with 60-μm Pyrex powder (drop height 1.3 m). Time from the instant of impact: a) 0, b) 40, c) 48, d) 80, e) 96, and f) 144 μs. Note that there was overwriting in this sequence so that the pattern of gray lines seen in frames a–e correspond to the channels opened up by the burning gas in frame f.

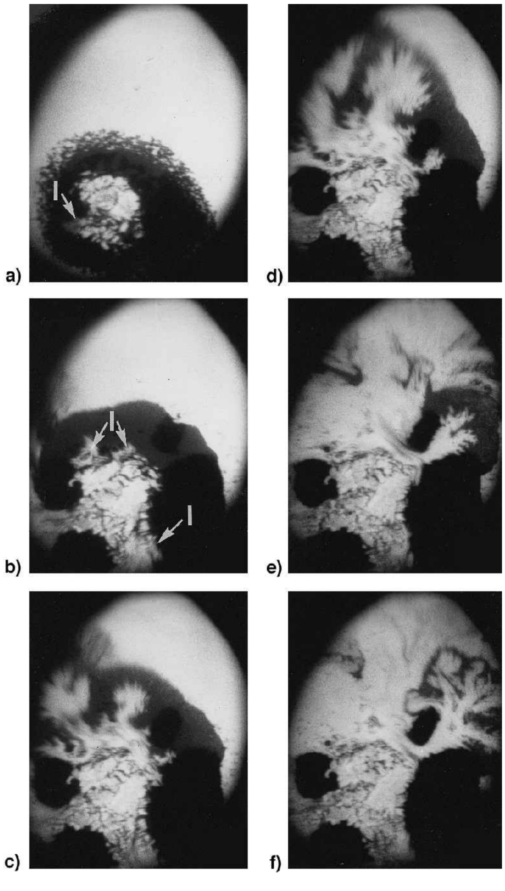

Fig. 6 Selected frames from a high-speed photographic sequence of the deflagration of ADN powder in contact with a disc of PMMA originally 2 mm in diameter. Drop height was 1.3 m. Time from the instant of impact: a) 352, b) 424, c) 440, d) 448, e) 472, and f) 496 μs. Deflagration can be seen to initiate from the periphery of the shattered polymer disc.

particular, the tree-like growth of the burning on the right-hand side in Figs. 6e and 6f.

A comparison was made between the sensitivity of ADN and AP when tested in this apparatus and mixed with the Pyrex powder. ADN was found to be slightly more sensitive than AP, giving a mild deflagration at 0.1 m drop height and a strong deflagration at 0.2 m. AP did not, however, deflagrate under these conditions at 0.2 m.

GAP

GAP was found to be very insensitive when deformed in this apparatus, even in the presence of additives. A number of striking features were, however, observed in the high-speed photographic sequences obtained in impacts from a height of 1.3 m. Figure 7 shows the sequence of events when a single drop of GAP (approximately 9 mg mass) is impacted by our drop-weight. The unimpacted drop has an irregular shape in Fig. 7a. Contact between the upper anvil and the drop occurs between Figs. 7a and 7b, so that the drop in Fig. 7b exhibits a circle of contact. By Fig. 7c the original shape of the drop has almost been obliterated, and it continues to expand as a circular film of fluid at about 45 m s^{-1} up to Fig. 7d. At around 500 μs the weight begins to rebound causing tensile cavitation at the edges of the film in Fig. 7e (see also Ref. 78). As the weight rebounds further, the shape of the original drop reappears in the foam created by this tensile cavitation. This is an observation that is not fully understood, but is probably caused by the polymeric nature of the liquid. It shows the value of high-speed photographic recording because its behavior would not easily be predicted.

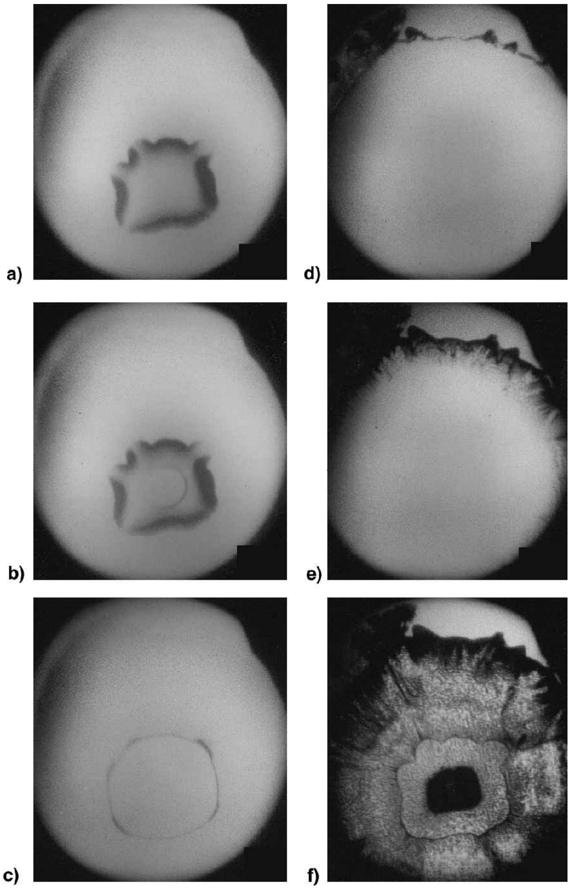

Fig. 7 Selected frames from a high-speed photographic sequence of the rapid deformation of a drop of GAP impacted from a drop height of 1.3 m. Time from the instant of impact: a) 0, b) 8, c) 56, d) 480, e) 520, and f) 560 μs. On the rebound of the weight, the original shape of the drop can be seen (compare frames a and f) among the foam created by cavitation in tension.

Figure 8 shows that this memory effect can be destroyed if Pyrex glass powder is present: the original shape of the drop only reappears in that part of the liquid film not containing glass powder (Fig. 8f). Otherwise the sequence of events up until rebound is very similar to that seen in Fig. 7.

Figure 9 shows the effect of placing a small disc of polycarbonate (PC) in the middle of a drop of GAP. The sequence starts just before the PC disc starts to fail, which it does some time between Figs. 9a and 9b. Fracture and shear banding is complete in approximately 10 μs. The direction of failure is from top left to bottom right in the frames. This causes what appears to be a spall plane in the liquid just inside the boundary of the film, bottom right in Fig. 9c, labeled S. The boundary there then becomes unstable and a jet forms moving at approximately 60 m s^{-1}. The boundary directly opposite (top left) also becomes unstable, taking on a ragged appearance.

Previous work has shown that collision between drops is a possible ignition mechanism in energetic liquids.[81] Therefore, the experiment was tried where two drops of GAP were constrained to expand between impacting glass anvils and collide (Fig. 10). The impact speed was measured from the film as being about 45 m s^{-1}. This sequence shows that the two drops simply coalesced smoothly (Figs. 10e and 10f), with apparently no boundary forming between them (Figs. 10c and 10d).

Although the impact speed is admittedly low compared to the liquid-drop or jet impact case (analyzed in Refs. 82–84), low-molecular-weight liquids such as nitroglycerine and a water-based liquid gun propellant (63% hydroxylammonium

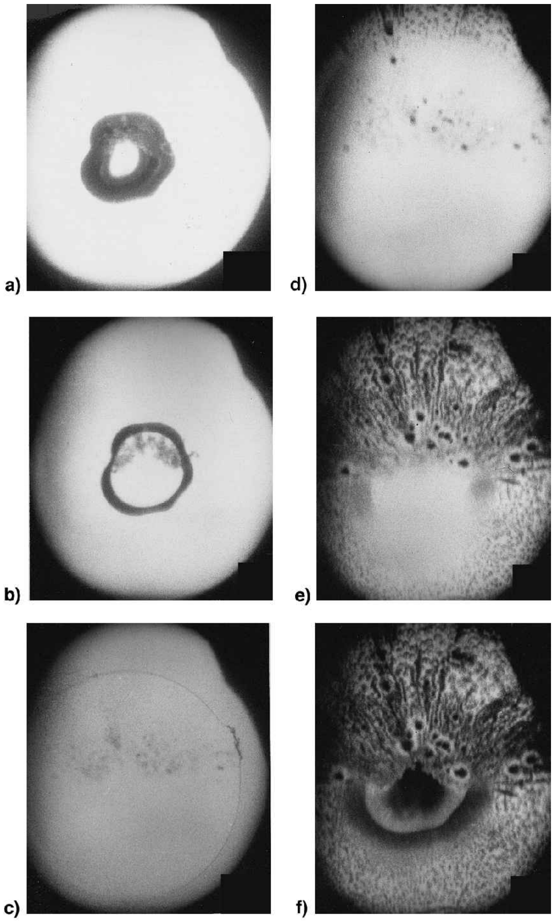

Fig. 8 Selected frames from a high-speed photographic sequence of the rapid deformation of a drop of GAP in the presence of 60-μm Pyrex glass powder (seen in the top half of the drop in frame b). The drop height was 1.3 m. Time from the instant of impact: a) 0, b) 80, c) 256, d) 656, e) 696, and f) 704 μs. Note that on rebound only the shape of that part of the drop not containing glass powder reappears (frame f).

nitrate, 17% triethanol ammonium nitrate, 20% water) both show collisional jetting in this apparatus and geometry (see Ref. 81 and Fig. 11 of this paper; jetting is labeled J). The drops of the water-based propellant also showed microjetting (labeled M in Fig. 11b) before collision, and developed a boundary between them when they collided (labeled B in Fig. 11d).

Discussion

Earlier high-speed photographic studies have shown that there are a wide variety of responses of explosive layers to impact. In the case of powdered layers, the material is initially compacted and compressed, and during this stage the radial expansion speed is slow, typically a few $\mathrm{m\ s^{-1}}$. If there are no additives it is rare to have ignition at this stage. As the impact progresses, the stresses in the layer increase so that the layer is no longer able to take the loads elastically. Plastic deformation and much more rapid flow then results with expansion speeds of typically a few tens (but with some layers a few hundreds) of $\mathrm{m\ s^{-1}}$. It is after rapid deformation starts that hot spot ignition is most likely. PETN and RDX sinter and become transparent before ignition. HMX does not. However, the key event in all of these cases is the onset of rapid flow. For these explosives, hot spot ignition by localized adiabatic shear deformation is the usual ignition mechanism. With hard additives such as glass, frictional hot spots are important. With polymeric additives it is hot spot production in shear bands and at crack tips in the polymer additive that is the key factor.[72,73,75,85]

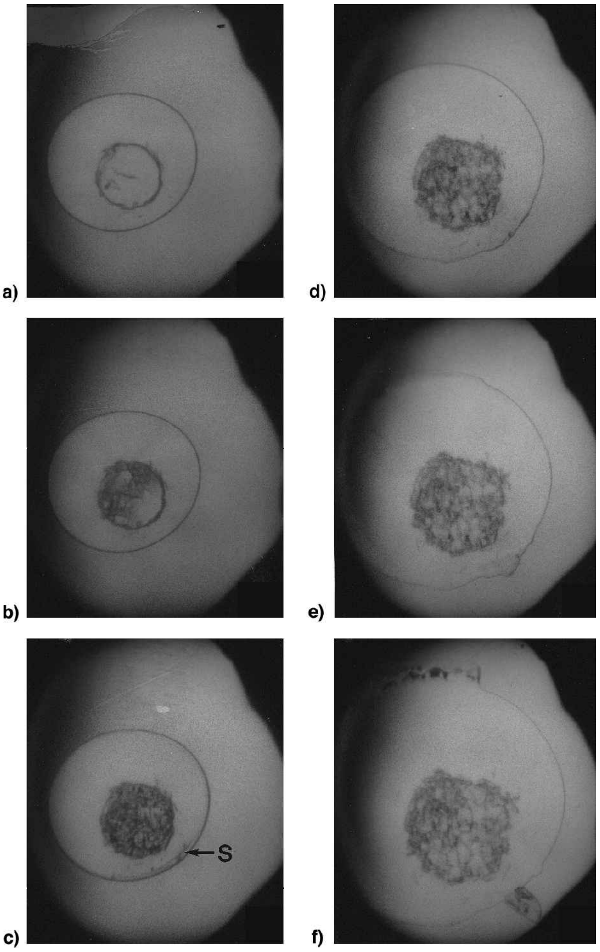

Fig. 9 Selected frames from a high-speed photographic sequence of the rapid deformation of a drop of GAP in the presence of a 2-mm-diam disc of PC. The drop height was 1.3 m. Time from the instant of impact: a) 352, b) 360, c) 368, d) 408, e) 424, and f) 464 μs. Note the jet that forms (frame f) at the periphery in line with the direction of the fracture wave in the PC disc (frames a–c).

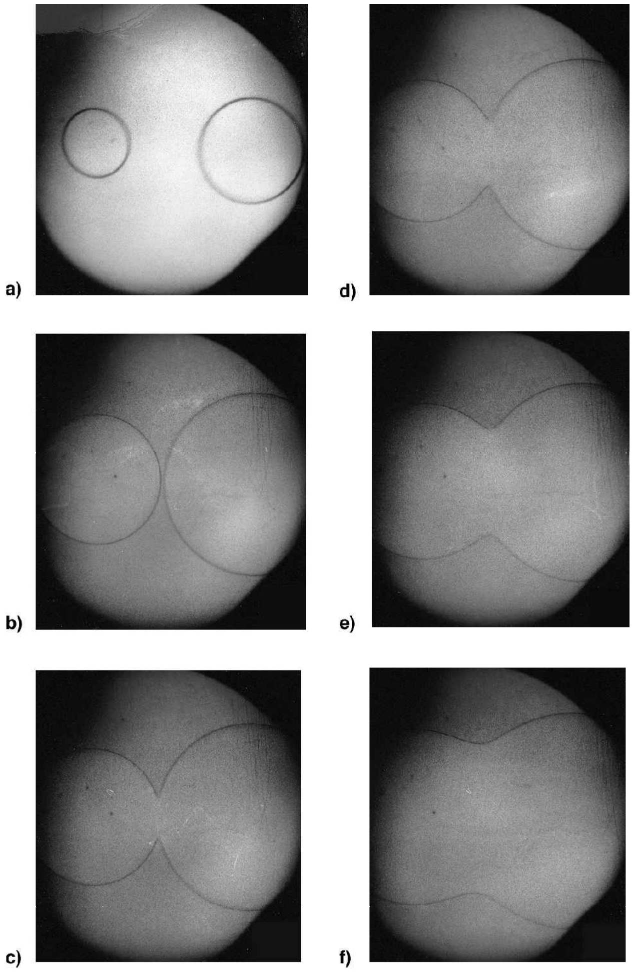

Fig. 10 Selected frames from a high-speed photographic sequence of the rapid deformation and collision of two drops of GAP. The drop height was 1.3 m. Time from the instant of impact: a) 240, b) 352, c) 360, d) 368, e) 392, and f) 440 μs. Note that the drops coalesce smoothly without the formation of microjets (compare with Fig. 11).

The research reported in this paper shows that the ADN powder we tested was slightly more sensitive than ammonium perchlorate in our drop-weight apparatus (though relative sensitivity can be affected by particle size and morphology, e.g., Ref. 86). Ignition occurred in the absence of additives after an initial compaction and compression stage. Once ignition occurred, the products moved outward at high velocity. ADN was sensitized both by a high melting point grit (60-μm Pyrex powder) and by brittle polymers. The ignition mechanisms are most likely hot spots caused by frictional rubbing between the particles and the anvils (in the case of the glass additive), and hot spots at crack tips and in shear bands (in the case of polymer additives). This statement is based on earlier studies.[44,73,75]

GAP was found to be ignition-insensitive in our drop-weight apparatus. It did, however, exhibit interesting deformation behavior, particularly a memory effect that could be disrupted by glass powder. Unlike lower-molecular-weight liquids, the collision of two drops did not produce microjetting or a visible boundary between them. High-speed jets can be important in ignition events, either by causing hot spots when they impact a neighboring surface or because they give a spray of droplets that can aid ignition in a heated gas space. The absence of jetting, therefore, is likely to help in the safe handling of GAP.

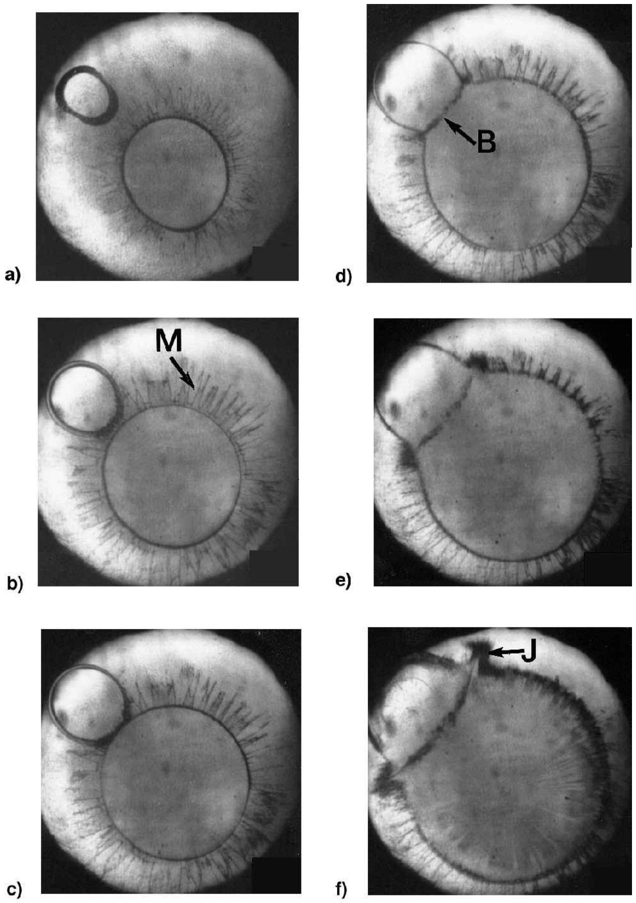

Fig. 11 Selected frames from a high-speed photographic sequence of the rapid deformation and collision of two drops of a solution of 63% hydroxylammonium nitrate, 17% triethanol ammonium nitrate in water. Time from the instant of impact: a) 70, b) 105, c) 112, d) 119, e) 126, and f) 133 μs. Note the collisional jetting that forms during frames d–f after the speed of the point of contact falls below the speed of sound in the liquid.

Acknowledgments

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