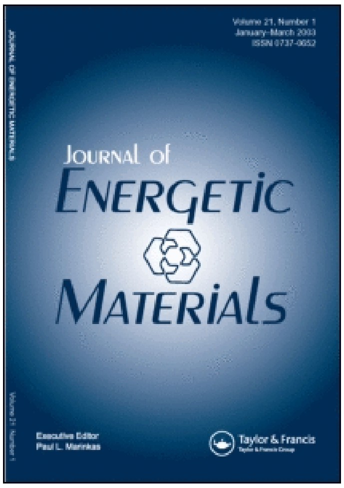

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EUTECTIC AND EMULSIFIED DEAK EXPLOSIVE

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The composite explosive, diethylene triamine trinitrate/ethylene diamine dinitrate/ammonium nitrate/potassium nitrate, DEAK, has been studied in its eutectic emulsified and nonemulsified forms. The purpose of this study was to develop an insensitive explosive for an artillery and bomb fill. Various methods of emulsification, as well as emulsifiers and coemulsifiers, were attempted. Indications are that microemulsions were formed. Sensitivities of these two forms of DEAK, as tested by the drop-weight impact test, showed significant differences, and their explosive performances are compared.

INTRODUCTION

In the ammonium nitrate explosive family, some explosives are simply mixtures off two types of material--oxidizer and fuel. Examples of these are explosives ANFO and Amatol. The oxidizer and fuel do not have a great degree of intimacy; thus, the performance is reduced. Some are eutectic systems with fine-grain particle size.[1,2] Producing a eutectic not only improved the performance but also lowered the melting point of the energetic material for easy casting. Some examples are AN/ANT, AN/ENT, EAK and DEAK, where ANT is the ammonium salt of nitrotetrazole and ENT is the ethylenediamine salt of nitrotetrazole. When DETN is added into EAK to form DEAK, its melting (eutectic) point changes from approximately 104° C to approximately 92° C, and its particle size is also reduced. In striving to develop an insensitive high explosive, two important characteristics must be considered--the performance and the sensitivity. It has been shown that, if fuels and oxidizers can be made more intimate through emulsification, both the performance and the sensitivity are improved.[3] Emulsified particles are small, on the order of a few tenths to ten micrometers. Thus, if all ingredients are present within or on the surface of each particle, there will be a great degree of intimacy between fuel and oxidizer. Also, emulsions are likely to reduce the absorption of moisture from the environment, especially in a W/O-type emulsion. Most emulsion-type explosives also contain various fractions of sensitizers such as gas bubbles or glass microballoons to increase the sensitivity, and metal fuels to increase the explosive power.[3-5]

Emulsions are thermodynamically unstable and have limited shelf lives in the liquid state. A new approach to this is to form microemulsions from the energetic materials. It has been shown experimentally and theoretically that microemulsions are thermodynamically stable for a range of temperatures and compositions, though their microstructures are complicated to probe, at present.[6-8] In this paper, we present some comparisons of eutectic DEAK and emulsified eutectic DEAK with respect to their performance and sensitivity.

EXPERIMENTS

Materials

All chemicals are labeled as reagent grade and are used without purification. Ammonium nitrate (AN) and potassium nitrate (KN) were premixed in 17/3 weight ratio and called AK. The KN was included in this proportion to stabilize the phase of the AN. Ethylene diamine dinitrate (EDDN) and diethylene triamine trinitrate (DETN) were synthesized, respectively, from ethylenediamine and diethylene triamine by reacting them with excess nitric acid, washing them with methanol and drying them. The eutectic DEAK used in this study has the weight composition DETN/EDDN/AK of 35/25/40. Eutectic samples were melted in a steam kettle around 110° C with mechanical stirring and then cast into pipe bombs.

The emulsified DEAK formulation is as follows: DEAK/SDS/mineral oil/1-pentanol of 90/2/5/3 in weight ratio, where SDS is sodium dodecyl sulfate, which was obtained from the Stepan Company; the mineral oil was from Fisher Scientific. To the molten eutectic DEAK, the surfactant (and cosurfactant) and oil were added. This blend was then stirred and cast into final form.

RESULTS AND DISCUSSION

Melting Point - Composition Curve for DEAK

A series of samples was made by varying the DETN to EDDN ratio, while keeping AK constant at 40 wt-%. The melting point of each sample was then determined by DSC, differential scanning calorimetry, and by a Fisher-Johns melting point apparatus. In general, these two methods agreed well with each other; they showed only a few disagreements in melting point temperature readings. Both methods indicate two temperature minima in the range of the experiment. Figure 1 shows the results from the melting point apparatus. The two possible eutectic compositions melted near the same temperature, 92° C; one occurred at the 35/25, and the other at the 20/40 weight ratio of DETN/EDDN. This is likely due to the double salt formation between AN and DETN.[2,15,16] The 35/25 formulation has been used by others and is used here for convenience of comparison. Actually, this formulation, DETN/EDDN/AN/KN, of 35/25/34/6 has about an 11% oxygen deficiency, while the other 20/40/34/6 has only about a 9% oxygen deficiency. This small difference in oxygen deficiency is believed to have no significant effect on either performance or sensitivity. With emulsified DEAK, the oxygen deficiency is expected to be higher and may have some effect on performance.

If these formulations show different performance and sensitivity, it will likely be due to the smaller grain size resulting from the additional DETN. This could cause greater intimacy between the fuel and oxidizer and, therefore, better performance. It has also been shown that small particle size produces decreased sensitivity to low-order shocks,[3] probably due to small void size.

<u>Small Scale Sensitivity and Performance Test</u>

Drop-weight tests with a National Bureau of Mines Type 12 machine, with a 2.5-kg mass, were performed according to standard procedures, and the results are shown in Table 1, along with other data for comparison purposes. Detonation speeds were measured using both piezoelectric pins and continuous-resistance probes. Pins were placed 2 in apart along a 12-in steel pipe with a 2-in ID and a 3-in OD. The continuous-resistance probe was attached to the inside wall of the pipe, parallel to its axis. The pipe was placed upright on a 2-in-thick rolled homogeneous armor, RHA, witness plate, shown in Figure 2. The results are summarized in Table 1. In general, we observed a speed increase along the pipe as calculated from piezoelectric pins. This verified the result obtained by Mathieu, et al.[17] Because of the spacing of the pins, we failed to find a dip in speed around 3 cm from the starting point, which had been observed by Mathieu, et al. In their 60-mm diameter test, the speed reached a stable value around 7.3 km/sec. A typical continuous-resistance probe result is shown in Figure 3. It displays voltage drop as a function of time. From the slope of this line, $\Delta V/\Delta t$, resistance per unit length of the probe, μ, and the value of the electrical current, I, the detonation speed, D, can be calculated by the formula $D = (\frac{1}{\mu I}) (\frac{\Delta V}{\Delta t})$. The two methods of measuring detonation speed are in good agreement.

TABLE 1
DEAK Sensitivity and Performance

Sample	Drop-weight Type 12, cm	Plate dent, mm	Detonation speed, km/sec	at	Diameter, mm	Density, g/cc
DEAK (Lee, et al)	109	-	7.16		41.3	1.60
DEAK (Mathieu, et al)[a]	>82	-	7.3		60	1.628
			6.96		40	1.61
			6.82		30	1.59
DEAK (present)	114	12.1	7.14		48	1.61
DEAK (emulsion)	>180	11.1	6.9		48	1.647
COMP B	49-85[b]	12.8	7.9		48	-
TNT	148[b]	11.1	6.8		48	-

Note: [a]Drop-weight height calculated from published energy value for 50% initiation probability.

[b]LLNL Explosives Handbook by B.M. Dobratz, 1981.

CONCLUSION

With eutectic DEAK, the drop-weight impact sensitivity and detonation speed of the present work are in close agreement with Mathieu's and Lee's. For emulsified DEAK, the drop-weight impact sensitivity is reduced more than 50 %, with a slightly lower detonation speed, in comparison with unemulsified, eutectic DEAK. Thus, it appears that DEAK itself is very close to an ideal explosive. Emulsification reduced the sensitivity but also lowered the detonation speed. This is not surprising since 10 wt-% of this emulsified DEAK was inert (surfactant, cosurfactant, and mineral oil) and may contribute to the oxygen deficiency. The density data indicate that it also contains less void volume. Emulsified DEAK is at least comparable with TNT in performance and better than TNT in drop-weight sensitivity; it is low in cost and less hazardous to health. Emulsification will likely yield improvements in performance only for highly nonideal explosives. Improvements in sensitivity will be achievable through emulsification. In order to avoid significant performance loss, adding nonenergetic materials should be kept to a minimum. Therefore, we plan to experiment with the formulation, 98 % DEAK / 1 % AOT / 1 % Dodecane.

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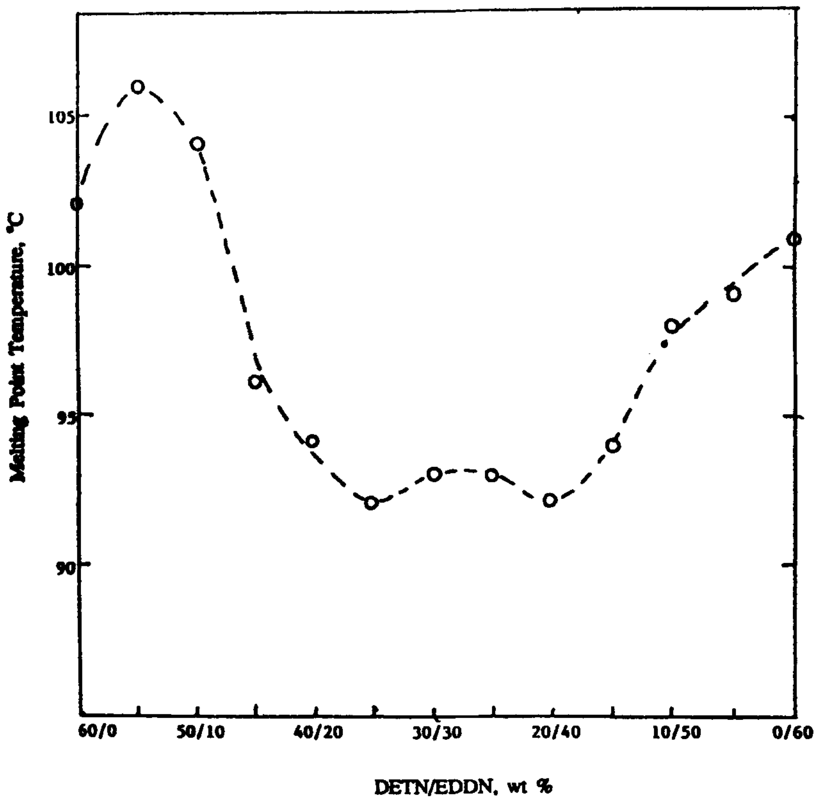

FIGURE 1.

Variation of Melting Point With DETN/EDDN Ratio at Constant 40 Weight-Percent AK.

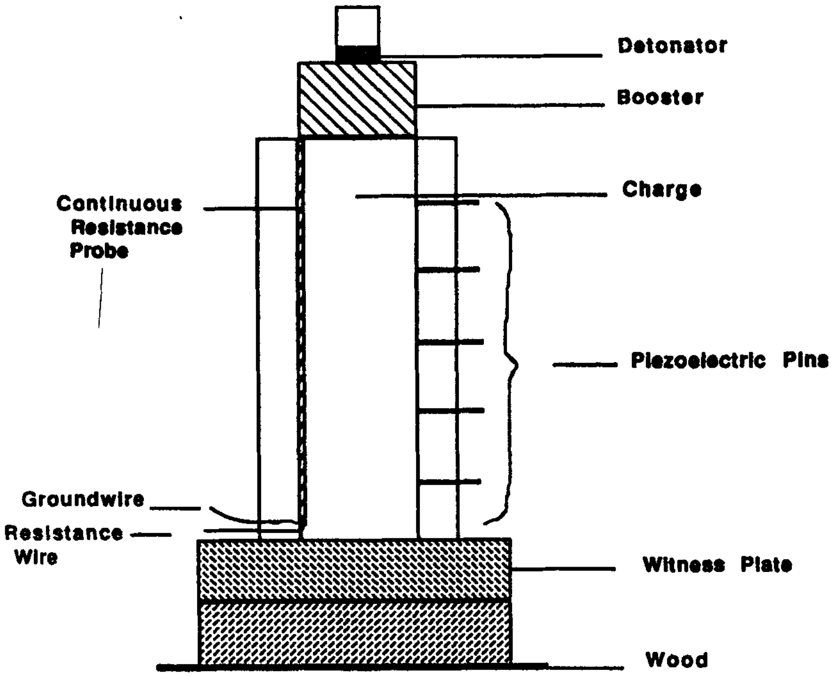

FIGURE 2.

Explosive Charge: Top, Primary Explosive; Middle, Booster; Bottom, Main Charge of Secondary High Explosive.

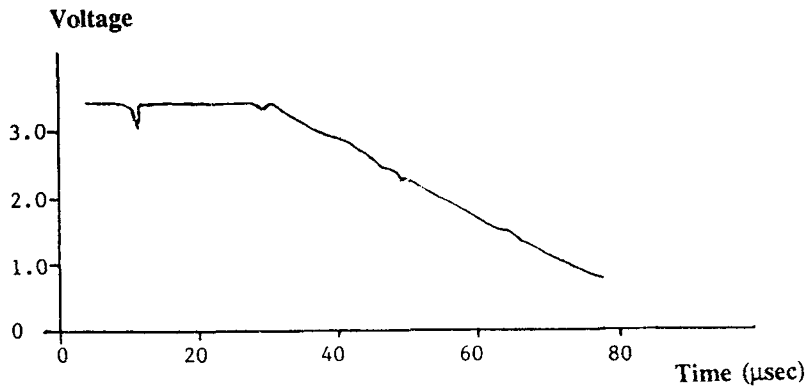

FIGURE 3.

Continuous Resistance Probe Output. Voltage Versus Time.