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Spraying Explosive Formulation

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SUMMARY

A composition of 98/2 weight percent PETN/polybutadiene elastomer was formulated, sprayed on bridgewires and electrically initiated, to determine the feasibility of such deposition of explosives bonded to test surfaces. Test firing resulted in a detonation reaction which could mean that explosive compositions sprayed on test surfaces may be an important tool in testing structural integrity, e.g., as presently in the Vulnerability Testing Program.

A semi-automatic apparatus for spraying explosive formulations on chemically etched electric circuits, support membranes and/or test vehicle surfaces has been designed, and improved application processes are being developed.

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INTRODUCTION

Fabrication of special design explosive charges for Sandia Laboratories' Shock Simulation test organization led to limited funding assistance in investigating the feasibility of deposition of explosives bonded to test surfaces. In collaboration with C. E. Irion, Pantex, a composition of 98/2 weight percent (PETN/polybutadiene elastomer) was formulated, sprayed on bridgewires and electrically initiated. Detonation reaction was indicated, and feasibility of the formulation was thus established. Explosive compositions sprayed on test surfaces might be an important tool in testing structural integrity of material presently under investigation in the vulnerability testing program.

ABSTRACT

A semi-automatic apparatus for spraying explosive formulations on chemically etched electric circuits, support membranes and/or test vehicle surfaces was designed and the application process is being developed.

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DISCUSSION

Prior Work

Shock simulation test parameters may dictate that shock loadings be of short duration, occur simultaneously and uniformly over parts of the surface, or have a non-uniform distribution, or require that initiation of the explosive take place along a ray of the test surface. An explosive system having properties of tenacity, flexibility, sensitivity to exploding wire and/or exploding foil initiation and sustained detonation in thin films would, it seemed, find use. An experimental test series was planned to investigate the feasibility of fabricating explosive geometries with low-pressure spray.

Two compositions were formulated; one was 98 weight percent untreated, Super-fine PETN¹ in terminated, linear polybutadiene; the other was 97/3. Both compositions were mixed in toluene to 40 wt % solids. A novel low-pressure spray system was employed. This system is a simple siphon, pressure jet, trigger valve combination mounted on a 15-ounce can of Freon gas used for recharging automobile air conditioning systems². There was no control of fluid flow and no metered gas flow in the spray system. It was found that a wet film, relatively slow to run or sag, could be built to substantial thickness in 10 to 15 minutes. One such film of 98/2 formulation 4 inches x 6 inches, 14.5 mils thick, was sprayed onto 5-mil thick Mylar in two spray passes. The film was permitted to dry for approximately one hour in ambient operating bay environment, then subjected to severe twists and bends of

¹ Product of Hercules Powder Co., Wilmington, Delaware. (The particle size was reported as follows: Surface area and average particle diameter by FSSS, 4745 cm²/g and 7 μ ; 100% < 40 μ ; 80% < 13 μ ; 13% < 5 μ ; 0 < 2-3 μ .)

² "Spray Tool" No. 8011, complete with power pack ML-5-22805 (WEP), distributed by Crown Industrial Products Co., Hebron, Illinois.

approximately 0.25-inch radius with no visible cracking and only minor releasing from the substrate at the film edges.

A second film, approximately 50 mils thick, was sprayed on 5-mil thick Mylar substrate and sectioned, measured, weighed and calculated for density. Results from four samples are tabulated:

<u>Film Thickness (mils)</u>	<u>Density g/cc</u>
60	.663
48	.647
48	.643
56	<u>.639</u>
Average Density	.648

Initiation and propagation tests were done. Two gap sensitivity test holders were modified by machining down the 0.302-inch diameter by 0.5-inch deep HE cavity portion of the plastic holder to depths of ~ .250 inch, and four were machined to ~ .050-inch depth. Standard detonator bridgewires (1.5-mil diameter gold wire 40 mils long) were seated in the modified holders and both 98/2 and 97/3 PETN slurries of 35% solids were sprayed into the cavity directly onto the bridgewire. The sprayed loadings were allowed to dry to a firm set between spray applications until the cavities were completely filled; average buildup per spray application was about 15 mils. Dross was removed with a draw knife while the explosive was still moist, leaving the loading flush to the cavity face. It appeared that 1-hour exposure to ambient bay conditions was adequate for drying. Aluminum witness plates were fastened over the filled explosives cavities with masking tape.

Testing was done with a firing set having fixed circuit values of 2750 volts and 2.5 μ f capacitance, with a moderately fast rise (dI/dt 556 amperes/ μ sec). Firing results are tabulated. All test samples detonated as indicated by the witness plates.

<u>Test No.</u>	<u>PETN/Binder</u>	<u>H.E. Thickness (mils)</u>	<u>Plate Dent Depth (mils) (approx.)</u>
1	98/2	243	14
2	97/3	~ 250	13
3	98/2	47	4
4	97/3	44	2
5	98/2	55	3
6	97/3	52	3

A second test series was designed to find the minimum HE thickness to sustain propagation. Fixturing for these tests consisted of 2-inch square, 0.3-inch thick polymethyl methacrylate (PMMA) plates drilled and tapped to locate a detonator bridgewire (1.5-mil diameter gold wire 40 mils long) in the center of the 2-inch square surface. Bridgewires were installed so that solder connections were flush with the surface. The phenolic bridgewire body was cemented to the plastic plate to prevent change in the bridgewire position in relation to the fixture surface. Slurried PETN of identical composition to the previous test was sprayed directly onto the bridgewire and surrounding fixture surface. After loading and drying, the plastic bridgewire fixtures were attached to 2-inch square, 0.25-inch thick aluminum witness plate with a single thickness of masking tape. A total of 12 test devices were constructed so that the thinnest explosive film was 8 mils, with each successive film increased in increments of approximately 3 mils. Testing was done in a steel

test chamber with the same firing set used in the previous test series.

The 8-mil, 10-mil and 15-mil thick films failed or deflagrated; the 15 mil failures were possibly caused by shorting to ground between the bridgewire solder connection and witness plate (see Figs. 1-7). Of the nine samples showing detonation (as indicated by bends, dents and surface condition), 13 mils was the minimum; all the others from 16-mil to the 41-mil maximum detonated. One additional sample composed of only the bridgewire head, plastic holder plate and 20-mil thick sprayed explosive film was tested to see if detonation would occur without the semi-confinement of the witness plate. That initiation and detonation was achieved and was demonstrated by the shattered plastic holder plate; i.e., it contained a shattered stratum at approximately mid-thickness parallel to the surface loaded with explosive. No characterization studies were done except to ascertain compatibility of the ingredients in the formulation.

Equipment Development

Features designed into the mechanized spray apparatus being developed utilize compressed air powered motion on two axes in either horizontal or vertical planes with finite angular adjustment for spray nozzle travel between 0° and 90°. Capability for rotating a test surface around its axis may be added with special fixturing and/or rotating table(s) where explosive depositions on cylindrical or conical surfaces may be preferred. Provisions were made for controlled stirring and circulation of the heterogenous explosive slurry in a closed distribution system to maintain suspension of solids. The slurry would be carried from the automatic spray nozzle, atomized and deposited with low pressure nitrogen.

Fig. 1

Test Series 2

Test Fixture Components
SE-1 Bridgewire Head
Lucite Holder
Aluminum Witness Plate

Loaded Test Fixture
PETN formulation sprayed
on Lucite holder and
bridgewire head.

Test Fixture Assembled
for Firing
Electrical Circuit Values
Standard Panel
Firing Set
2,500 V
11 mfd Capacitance

Fig. 3

Device No. 982646-07
H.E. Thickness = .013 inch
Result: Detonation
Note: Lack of signature in
lower center of witness
plate may have been
caused by premature
measuring of film
thickness with a dial
indicator.

Device No. 982646-09
H.E. Thickness = .016 inch
Result: Detonation

Fig. 5

Device No. 982646-12
H.E. Thickness = .025 inch
Result: Detonation

Device No. 982646-13
H.E. Thickness = .028 inch
Result: Detonation

Fig. 6

Device No. 982646-14
H.E. Thickness = .029 inch
Result: Detonation

Device No. 982646-15
H.E. Thickness = .032 inch
Result: Detonation

Fig. 7

Device No. 982646-16
H.E. Thickness = .041
Result: Detonation

Device No. 982646-17
H.E. Thickness = .020 inch
Result: Detonation
Note: This device was tested
without a witness plate,
hence no confinement.

Processes and controls peculiar to this explosive technology had to be developed to operate safely. Therefore initial spraying operations were planned and executed with a mock explosive system of like character (substitution of pentarythritol for PETN). The sensing probe of an electrostatic field meter³ was attached proximate to the spray nozzle to monitor possible electrostatic buildup during spray operations. The peak reading observed during ~ 10 cycles depositing 30% solids slurry on a grounded Mylar target was 200 volts/centimeter for approximately 5 seconds. This peak occurred when an observer made a valve adjustment on the automatic spray gun. Nominal field voltages of 50 to 100 volts/centimeter were observed; they dissipated to ground immediately. It was concluded that the spray apparatus was adequately grounded against the possible hazard of electrostatic spark discharge.

Solvent vapor concentration and airborne particulate samples were collected by an industrial hygienist during these initial spraying operations, to assess the effectiveness of "hog hair pad Kimwipe sandwiches" as filters in the collection and air filtration system. Sample analysis showed vapor concentrations and aerosol particulates exceeded acceptable physiological tolerance levels. Examination of the "filter sandwiches" showed that solids collected on the Kimwipes had sealed approximately 50% of the exposed area thereby restricting air flow to substantially less than capture velocity. The collection and filtration problem was solved by placing two thickness of Dycon filter media⁴ in the existing collector. Subsequent filtration tests

³ Model 146-2 Isoprobe Electrostatic Millivolt/Fieldmeter, Monroe Electronics Inc., Middleport, New York

⁴ Bonded modacrylic textile fiber mat manufactured by Continental Air Filter Co. (a Division of WEHR Corp.) Louisville, Kentucky.

indicated only a slight drop in air velocity with no detectable passage of solid particulates and with vapor concentrations lowered to acceptable tolerance levels. Installation of a one-inch thick clear PMMA shield, for operator protection against flash fire, and extension of the deluge fire protection system in the operating bay qualified the spray apparatus for non-remote operation.

Process Development

Development of processes for controlling deposition of consistently uniform, high quality explosive film proved more difficult than anticipated. Predictably, a low viscosity fluid delivered to the spray nozzle at an adequate rate of flow could be atomized and deposited in a controlled pattern at controlled speed and produce a well defined, quality product. Of the predictable variables, the speed at which the spray nozzle moved would be the most difficult to change due to rather sensitive air pressure differentials demanded for overall apparatus operation. It was therefore decided that viscosity of the explosive slurry (i.e., solids to solvent ratio) was of prime importance. Viscosity and flow rate investigations commenced with 1,500 milliliters of 98/2 PETN formulation mixed to 40% solids in toluene. Attempts to process 500 milliliters of the 40% solids mixture were stopped because of clogging in the fluid shut-off valve, spray orifice and nozzle passages with the larger PETN particles. The remaining 1000 ml of mixture was reduced to 25% solids with toluene and spraying resumed. Atomized spray was obtained with 9 psi nitrogen on the fluid supply reservoir and 13 psi nitrogen pressure at the spray nozzle for about 3 minutes of programmed cycles until fluid stoppage recurred. After thorough cleaning of fluid lines, orifice and nozzle, spraying was resumed and continued for approximately 10 minutes before clogging again. Formulation deposited on the target was excessively wet and when

dried, crusted to a smooth, dense surface. A specimen of the dried formulation was carefully removed and trimmed to 1.470 inches wide, 2.0 inches long and 0.0658 inch thick (average of 9 measurements). The specimen was measured, weighed and calculated to have a density of 0.83 g/cc. The accumulation of PETN restricting flow through the fluid shut-off valve passages, automatic spray orifice and nozzle was evidently resulting from some settling and compaction of solid particulates each time the fluid shut-off valve was closed. It was concluded that control of slurry viscosity could not be imposed until residual slurry (in excess of the flow rate through the orifice and nozzle for optimized spray deposit) in the fluid supply system could be returned to the slurry supply reservoir. The air operated, diaphragm type, fluid shut-off valve was modified by drilling a return passage in the valve body and connecting a peristaltic pump between the fluid return connection and the fluid supply reservoir. Attempts to spray explosive mixture at 30 weight percent solids on brass discs for gap sensitivity testing were abandoned after processing about 150 ml when a fluid stoppage again occurred and investigation revealed large particles of PETN (30 to 40 μ) accumulated on the valve seat. During the mechanical check that followed, it was determined that the diaphragm operated actuator rod was mechanically limiting valve opening to 6 mils off the seat. This condition was corrected by removing ~ 15 mils from the machine screw head holding the air operated diaphragm assembly to the upper end of the actuator rod and by shimming the entire air actuator an additional 10 mils above the valve body seat. Functional checks showed the fluid valve, when fully opened, had 23 mils clearance off the valve seat allowing an adequate flow of slurry; however, the 30 weight percent solids mixture was still too thin to produce sprayed film without crusting. An explosive mixture (300 mls) was prepared at 35 weight percent solids to spray load brass discs for gap sensitivity testing. During

preliminary or "set-up" operations, nitrogen pressure on the fluid reservoir was increased to 9.5 psi and pressure at the spray nozzle was regulated at 8 psi (gage reading). Six brass discs were coated with ~ 25-mil thick sprayed PETN on one side only. During this operation, it was noted that: (1) the sprayed explosive was deposited in 7-8 mil thicknesses each pass; (2) the deposited explosive films appeared excessively rich in solvent, and (3) intermittent sputtering occurred at the nozzle where the slurry was partially atomized into small droplets. The condition was diagnosed as erratic regulator control of nitrogen pressure at the spray nozzle and/or fluctuations in fluid flow. During a minute check of the fluid system, a single stainless steel chip had not broken clear when the recirculating passage was drilled in the valve body and had, instead, been turned into the fluid inlet passage and under pressure from the circulating slurry, was forced against the upper side of the inlet passage causing about a 50% restriction. The chip was removed and the valve reassembled. Analysis of the nitrogen system supplying pressure to the spray nozzle and fluid reservoir indicated that a 1/4-inch diameter plastic tube connected between the nitrogen supply, the spray nozzle, and fluid reservoir was undersized, thus creating a pressure drop through the nozzle regulator completely nullifying pressure control. Relocating the pressure regulator from the nozzle to the supply manifold and replacing the 1/4-inch diameter plastic tube with 3/8-inch diameter tubing corrected the atomizing pressure problem. A separate 1/4-inch diameter plastic tube was connected from the nitrogen supply manifold through the apparatus control panel to the fluid reservoir thereby allowing the fluid reservoir to be pressurized before actuating the spraying cycle.

The foregoing changes and modifications permitted spraying 98/2 PETN formulation

mixed to 40 weight percent solids in toluene. Nitrogen pressures were adjusted to ⁷13.5 psi at the pressurized fluid reservoir and ¹⁸15 psi at the spray nozzle with the atomizing pressure adjustment on the spray gun set full open (approximately 2-1/2 turns). The effective spray pattern is 4.7 inches wide with the spray nozzle positioned ⁵²⁻⁵⁴ 8 inches above the target surface. Explosive film deposited on plastic substrates were measured in open set-up with a dial indicator and thicknesses ranged from 5 to 6.5-mil thickness over a distance of 8.5 inches for a single spray pass. type nozzle

$$TAP = \frac{dy}{sec/cm^2}$$

800-1000 TAP/mil thick.

CHARACTERIZATION STUDIES

Explosive system characteristics studies made during the feasibility investigation were limited to testing for compatibility by Infrared Spectral Analysis and Differential Thermal Analysis of polybutadiene elastomers with solvents (toluene and hexane) and PETN. Data indicate no detectable chemical reaction occurring in either the slurry or dry film, and the composition is therefore considered stable. One 300-hour weight-loss test was performed on a similar composition, yielding like results. Other reported data relating to performance characteristics and/or physical properties generated during the original test series must be considered as approximate and not typical values. Data gathered in subsequent tests of explosive film made under controlled processes with the spray apparatus follow.

Gap Sensitivity Tests

Gap sensitivity tests (LASL 1/2 inch, modified) showed the 50% probability gap to be 0.300 inch which indicated 100-kilobar pressure at the brass/explosive interface. A calculation of transmitted pressure is not presently possible. The low density (50% of TMD for PETN or less) of sprayed film would indicate

very low pressure in the explosive system and this, coupled with the short run distance, indicates high sensitivity. Gap sensitivity testing is expected to measure relative shock sensitivity, but confinement of a thin layer of explosive between a massive witness plate and a driven brass shim may confound these data.

Impact Sensitivity Tests

Impact sensitivity tests using a 2.5-kg drop hammer in Type 12A (sandpaper) test gives a 50% probability that detonation would occur at a height of 20.1 cm. Type 12B (smooth steel surface) tests were inconclusive due to the limited number of samples available for testing; however, a hammer drop height of about 100 cm was tried with negative results.

Film Density

Density measurements of PETN film deposited in sequential spray passes onto glass plates were calculated to have densities ranging from 0.707 to 0.759 g/cc of the eight specimens prepared; excluding the minimum and maximum values, five were grouped around 0.74 to 0.75 g/cc with the sixth specimen showing 0.72 g/cc. It was noted during these density calculations that the incremental buildup was relatively uniform from about 7 mils to about 25 mils thickness and from 48 to 62 mils. An abrupt change found between 25- and 48-mil thicknesses was probably caused by improper programming of spray nozzle travel relative to target position which caused overspray buildup to occur.

*May + thickness?
-120*

SUMMARY

The semi-automatic apparatus designed and built to spray explosive formulations on supporting surfaces has been made operable. Safety requirements for non-

remote spray operations were met and standard operating procedures established. Processes and process controls have been defined; however, additional work is needed to obtain optimum repeatability and generate data useful in the design of explosive test loads. Explosive formulations have been sprayed on flat surfaces held on a horizontal plane and on a cylindrical surface rotated around its horizontal axis. No attempts have been made yet to spray test surfaces in other positions, although these capabilities were designed into the apparatus. 7
1
Tape

Characterization tests on the 98/2 PETN formulation spray explosive system show type 12A impact sensitivity results of 20 cm (cap grade PETN, 11-15 cm and RDX, 20 to 23 cm); gap sensitivity (modified test) of .300 inch and density of 0.74 g/cm³ (average of 8 samples). All ingredients have been tested for chemical reaction(s) with each other and were determined to be compatible. No accurate measurements of detonation velocity have been made.

FUTURE WORK

Areas of interest needing early investigation to implement test applications are as follows:

1. Development of test techniques to accurately measure simultaneity, velocity, and pressure(s) of detonation in thin explosive film as related to methods of initiation. These data are urgently needed to properly design explosive charges for simulation testing.
2. Characterization studies to optimize particle size and distribution for precise matching of the 98/2 PETN formulation for initiation from bursting wires or foils. These studies may ultimately define the maximum area that

could be simultaneously detonated.

3. Further equipment and process development to establish high order repeatability in the fabrication of explosive film on test surfaces not yet attempted; e.g., conical and spherical shapes.
4. Additional chemical research to improve physical properties of the terminated polybutadiene elastomer used in the 98/2 PETN formulation. A group of very active (non-terminated) butadiene elastomers has recently been announced which, if added to the terminated elastomers in current use, may induce some cross linking; thereby, enhancing the system's flexibility and bonding properties. Further investigation of aromatic hydrocarbon chemicals as solvents and carriers for explosive formulations (i.e., heptanes through decanes) could prove very beneficial.

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