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[54] **METHOD FOR PREPARING HEAT RESISTANT MILD DETONATING FUSE**

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[62] Division of Ser. No. 443,107, March 26, 1965, abandoned.

[52] **U.S. Cl.** ..... **102/27 R;** 86/1; 86/22

[51] **Int. Cl.²** **F42B 3/10**

[58] **Field of Search** 86/1; 102/27, 70

[56] **References Cited**

UNITED STATES PATENTS

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[57] **ABSTRACT**

A process of preparing a mild detonating fuse from such heat resistant explosives as DIPAM, HNS and NONA and includes filling a tubular sheath made of a metal which can be annealed at temperatures below 568°F with an explosive material, reducing the explosive filled tubular sheath to a lesser diameter and annealing the sheath.

7 Claims, No Drawings

METHOD FOR PREPARING HEAT RESISTANT MILD DETONATING FUSE

The invention described herein may be manufactured and used by or for the Government of the United States of America for governmental purposes without the payment of any royalties thereon or therefor.

This application is a division of my copending application Ser. No. 443,107 filed Mar. 26, 1965, now abandoned.

This invention relates to explosive detonating cord. More particularly the invention relates to novel detonating cord characterized by resistance to high temperatures and low pressures.

Detonating cord consists of an outer tube or sheath of tin, lead, or other suitable metal or alloy encasing a continuous inner core of high explosive such as PETN, RDX, HMX and the like. Detonating cord was originally prepared by filling lead tubing with a molten explosive, permitting the explosive to harden, and then drawing the tubing down to a diameter of about 0.200 inch. The old form of detonating cord could not be bent to conform to the shape of an object or to conduct the cord past impediments without disturbing the continuity of the explosive train. Breaks were produced in the rigid cast explosive material by the bending of the casings, and these breaks were likely to cause a failure of the detonation wave as it proceeded down the length of the column. It therefore became the practice in the art to use explosive materials in particulate form in filling the tubular casings to insure that the continuity of the explosive train would not be disturbed by bending of the casing. In fact, some of the common high explosives, such as HMX, have such high melting points that they cannot be cast and can be used only in a particulate form.

The explosives and sheath materials used in the prior art detonating cords have been satisfactory for ordinary purposes. However, detonating cord in the form of mild detonating fuse and flexible linear shaped charge devices are finding increasing use in many aeronautic and space applications. The separation of missile stages from one another is just one of these applications. Aerodynamic heating can produce skin temperatures in missiles sufficiently high to melt most conventional explosives and thus to render inoperative detonating cord incorporating these explosives. High temperature explosives such as DATB have melting points above the expected temperatures but these explosives will decompose or sublime and thus be lost at the high temperatures and low atmospheric pressures which accompany high altitude operation. For example, DATB exhibits a weight loss of 53.8% when subjected to a temperature of 410°F and a pressure of 2.5 mm of Hg.

Accordingly it is an object of the invention to provide detonating cord characterized by resistance to temperatures above 500°F and to very low pressures. More particularly, an object of the invention is to provide detonating cord incorporating novel combinations of high temperature and low pressure resistant explosives and sheath metals.

A further object of the invention is to provide a method of manufacturing this novel detonating cord.

Briefly stated, the invention comprises fabricating detonating cord with high temperature resistant sheath materials and core loadings of explosive compounds which retain their crystalline structure at all temperatures up to 568°F and which can, in addition, resist low pressures. Suitable sheath materials are silver and aluminum. Suitable explosives are 3,3'-Diamino-2,2',4,4',6,6'-hexanitrobiphenyl (DIPAM), 2,2',2'',4,-4',4'',6,6',6''-Nonanitroterphenyl (NONA), and 2,2',-4,4',6,6'-Hexanitrostilbene (HNS). The method of making the detonating cord of the invention adds to the conventional drawing, rolling and swaging operations a novel annealing step in which the cord is annealed to eliminate the cold work of the drawing and swaging steps with the explosive load in place and without in any way affecting the performance capabilities of the cord.

Other objects, advantages and new features of the invention will become apparent from a reading of the following detailed description including the specific illustrative examples.

EXPLOSIVES

DIPAM may be prepared by means of a synthesis utilizing dipicric acid (3,3'-dihydroxy-2,2',4,4',6,6'-hexanitrobiphenyl) as a starting material, as has been described in the application of Joseph C. Dacons et al., Ser. No. 334,667, filed Dec. 30, 1963. In general, the synthesis involves conversion of dipicric acid to its dipyridinium salt, the conversion of the salt to dipicryl chloride and finally the conversion of the dipicryl chloride to DIPAM. As summarized in Table I, DIPAM melts at about 568°F and shows substantially no weight loss at a pressure of 1 millimeter of mercury at 410°F. DIPAM crystals are yellow and finally divided, in particle sizes of 10 to 40 microns with some agglomerates of up to 100 microns. DIPAM does not flow well in very humid atmospheres, but can be handled and loaded into detonating cord by normal techniques when the loading room is held at a relative humidity of about 45%. As will be indicated by the test results given in the examples below, the crystal geometry and particle size of DIPAM are adequate to meet the requirements of flexible detonating cord.

NONA is a polynitropolyphenyl compound having satisfactory properties for the present application as summarized in Table I. NONA may be prepared by the reaction of picryl halides and a halotrinitrobenzene by the various processes set out in application Ser. No. 320,579, of Joseph C. Dacons filed Oct. 31, 1963. NONA crystals are bladed or lathlike in form in particle sizes of 10 to 50 microns. Loading is best performed at a controlled relative humidity of about 40–45%.

The preparation of HNS has been described in the application of Kathryn G. Shipp, Ser. No. 365,572, filed May 5, 1964. The method as there described involves a simple one step reaction which comprises the addition of a solution of 2,4,6-trinitrotoluene to an aqueous solution of an alkaline metal hypochlorite, and then recovering the resulting product. The physical properties of HNS are summarized in Table I. The crystals are generally acicular in form and may occur in needles up to 500 microns in length, although much smaller particle sizes are available. HNS, like DIPAM and NONA should be loaded under conditions of controlled humidity.

TABLE I

Physical Properties	DIPAM	HNS	NONA
Melting Point (°C)	298–299	316	440–450
(°F)	568	601	824–842
Theoretical Maximum Density	1.79	1.74	1.78
Volatility at 410°F			
Pressure (mm of Hg)	1.0	0.9	0.8
Weight Loss (%)	Nil	Nil	0.5
Macroscopic Crystal Data			
Shape	Equant	Acicular	Bladed
Size (microns)	10–40μ	500μ	10–50μ

SHEATH MATERIALS

Ordinary lead sheathed detonating cord is made with an antimony-lead alloy containing about 6% antimony. This alloy is not suitable for the present application, however, since it melts at 518°F, well below the anticipated temperatures. For temperatures up to 590°F, a 2 percent antimony-lead alloy would be suitable, but for higher temperature operation, other sheath materials are required. Silver and aluminum have been investigated as possible high temperature sheath materials and have been found suitable. The materials can be easily worked and, as will be indicated in the examples below, produce results which are equal to or superior to those obtainable from prior detonating cord.

FABRICATION

One of the primary advantages of using silver or aluminum as the sheating material is the ability of these materials to be annealed at temperatures well below their melting points. Thus, it is possible to load and fabricate the detonating cord in the standard manner, or with additional working, and then to anneal the cord with the explosive in place without affecting the explosive in any way.

Detonating cord is ordinarily constructed by first pouring a desired quantity of explosive into the tubular sheath and then reducing the diameter and thus compacting the explosive by means of a series of drawing operations. It is also known to increase the apparent density of the explosive by swaging the tube to a smaller diameter. All of these mechanical forming operations introduce cold work into the metal, reducing its ductility and flexibility. This cold work was no problem in the lead sheaths of the prior art.

Silver and aluminum, however, must be annealed at intermediate stages in the forming process and after being worked in order to maintain their flexibility. As stated above, this annealing can be accomplished at temperatures well below their melting points, and well below the melting points of DIPAM, HNS, and NONA.

Aluminum sheathing can be annealed at 500°F, with an annealing period of about 5 hours. Higher temperatures can be used if they can be tolerated by the particular explosive involved.

The annealing time and temperature for silver sheathing is dependent on the size of the tube and the amount of cold work. For thin walled tubes, a temperature of 350°F held for one-half hour, followed by cooling in air, is satisfactory. For thick walled tubes, 460°F for at least one hour is required.

The annealing process has no effect on the detonation velocity or on the ability of the explosive to be detonated. No change in the performance of detonating cord in the form of flexible linear shaped charge was observed after the annealing operation.

To further describe this invention, attention is now directed to specific examples of detonating cord according to the invention. The examples are illustrative only, the invention not being limited thereby.

EXAMPLE I

Several lengths of detonating cord were fabricated with core loadings of DIPAM in the amounts of 5, 10 and 15 grains per foot in a 6½ percent antimony-lead alloy as the sheath material. The explosive was found to support detonation in the loading of 5 grains per foot, which represents a column diameter of approximately 0.030 inch. The detonation velocity was uniform in all the loadings at approximately 5,440 meters per second.

Each of the detonating cords of this example was able to propagate the detonation around a bend. The cornering tests were divided into two groups, spirals and sharp bends. In the spiral test the detonating cord was wrapped around aluminum rods of 1⅛ inch and ⅝ inch diametes. The spiral arrangement did not affect the performance of the explosive. In the sharp bend test, a one foot length of detonating cord was bent into a shape containing seven or eight 90° bends. The detonating cord so bent was placed on an aluminum witness plate and detonated. The bends did not affect the performance of the DIPAM detonating cord.

A high altitude simulating chamber was used to determine the suitability of the DIPAM explosive for the high altitude application. Tests were conducted by detonating the samples on aluminum witness plates, at room temperature and atmopsheric pressure, as well as at temperatures above 500°F and pressures of 0.13 millimeters of mercurcy. The results of the tests are summarized in Table II. Note that one sample initiated and functioned successfully at 567°F which is very close to the melting point of DIPAM. No variation in the propagation properties was observed as a result of the variation in temperature or the variation in pressure in this test.

TABLE II

Functioning of DIPAM Detonating Cord				
Functioning Temp ±5°F		Pressure	Simulated Altitude	
(°F)	(°C)	(mm of Hg)	(feet)	Result
75	24	760	250	
528	275	760	250	Complete
552	289	760	250	Propa-
567	297	760	250	gation
530 (Several runs)	277	0.13	205,000	

EXAMPLE II

Several lengths of HNS loaded detonating cord were fabricated in a 2 percent antimony-lead alloy sheath, with core loadings ranging from 10 grains to 22 grains per foot. No variation in performance was observed in the spiral and sharp bend tests conducted with this detonating cord.

Simulated high altitude tests were performed on this detonating cord at 5×10^{-3} mm of Hg, approximately representative of an altitude of 271,000. Some of the results are given in Table III. The cords were exposed to the "Soak Temperature" for the lengths of time indi-

cated and were detonated at the end of those times. The heating rate was about 32°F per minute.

TABLE III

High Temperature Performance of HNS Detonating Cord

Loading	Soak Time (minutes)	Soak Temperature (°C)	(°F)	Results
11.4 grains/ft.	8	280	536	
11.4 grains/ft.	13	312	594	Complete
11.4 grains/ft.	28	310	590	Detonation

Some sublimation of the HNS occurred during the high temperature, high altitude testing. It was not sufficient, however, to degrade the propagation characteristics of the detonating cord.

EXAMPLE III

Linear shaped charge has been fabricated with DIPAM as the explosive and silver as the sheath material. A number of charges, differing in cross-sectional shape and dimensions were prepared and fired to determine detonation velocity and performance as measured by the penetration distance of the shaped charge into an aluminum alloy target. The effect of stand-off distance was investigated and it was found that an optimum stand-off distance lies between 0.035 inch and 0.125 inch. The best performance was obtained with a 17.1 grain per foot charge, which gave a penetration of 0.106 inch at 0.125 inch stand-off. A 15.5 grain per foot sample gave a mean penetration of 0.072 inch and a maximum of 0.099 inch. This is approximately equal to the performance of standard 15 grains per foot RDX in lead.

Detonation velocity for all the DIPAM in silver linear shaped charge devices was approximately 6900 meters per second.

EXAMPLE IV

Linear shaped charges of HNS in silver showed performances approximately equal to those exhibited by DIPAM in silver. The best performance was exhibited by a sample having a core loading of 15.42 grains per foot which gave a 0.090 inch mean penetration at a stand-off of 0.125 inch.

EXAMPLE V

Several samples of NONA-silver linear shaped charge were constructed and were found to give satisfactory results. Detonation velocities for NONA in silver are slightly higher than for the other explosives, being approximately 7150 meters per second. The best performance for this combination of materials was 0.089 inch mean penetration at a stand-off of 0.125 inch and a core leading of 14.93 grains per foot.

EXAMPLE VI

Several samples of detonating cord were constructed with DIPAM, HNS and NONA as the explosive core materials in fine silver as the sheath material. These cords were constructed in various loadings and column diameters ranging from 0.174 inch in outside diameter to 0.0395 inch in outside diameter. The range of explosive loadings for these diameters was from 42 grains per foot to 1.9 grains per foot in each of the explosives. The average detonation velocity for DIPAM loaded MDF was approximately 6900 meters per second with no diameter effect being noted. The detonation velocity for HNS was 6700 meters per second with no diameter effect, and the detonation velocity of NONA was 7000 meters per second, no obvious diameter effect being observed.

EXAMPLE VII

A sample of detonating cord was made with an aluminum sheath and NONA as the explosive, with a core loading of 10 grains per foot. This sample has been subjected to the high temperature, low pressure test and has been found to function satisfactorily, that is with complete detonation, after being held at a temperature of 772°F for a period of 30 minutes.

As to safety, each of the explosives under consideration is relatively insensitive to impact and may therefore be easily handled. The explosives are safe from inadvertent initiation by electrostatic discharges.

The detonating cord and the process of making it, including the annealing step, are subject to wide modification as will be apparent to those skilled in the art after considering the above detailed description. Accordingly within the scope of the appended claims, the invention may be practiced other than as herein specifically described.

What is claimed is:

1. A method of making detonating cord comprising the steps of

filling a tubular sheath made of a metal which can be annealed at temperatures below 568°F with an explosive material in particulate form selected from the group consisting of 3,3′-diamino-2,2′,4,4′,6,6′-hexanitrobiphenyl; 2,2′,2″,4,4′,4″,6,6′,6″-Nonanitroterphenyl; and 2,2′,4,4′,6,6′-Hexanitrostilbene,

reducing said explosive filled tubular sheath to a lesser diameter to thus compact said explosive,

and annealing by application of heat said sheath at a temperature below 568°F.

2. A method as recited in claim **1** wherein said metal is silver and said annealing step is carried out at temperatures in the range of 350°F to 460°F for periods of at least one-half hour.

3. A method as recited in claim **1** wherein said metal is aluminum and said annealing step is carried out at a temperature of 500°F for a period of at least approximately 5 hours.

4. A method according to claim **1**, wherein said filling step is carried out in a regulated atmosphere at a relative humidity of less than 45%.

5. A method of fabricating detonating cord comprising

filling a tubular sheath made of a metal selected from the group consisting of silver and aluminum with an explosive material in particulate form selected from the group consisting of 3,3′-diamino-2,2′,4,4′,6,6′-hexanitrobiphenyl; 2,2′,2″,4,4′,4″,-6,6′,6″-Nonanitroterphenyl; and 2,2′,4,4′,6,6′-Hexanitrostilbene, said filling step being carried out in an atmosphere in which the relative humidity is maintained below 45%,

compressing said sheath to a lesser diameter by a series of drawing and swaging steps to thus compact said explosive, and

annealing said sheath both intermediate of said drawing and swaging steps and after the last of said steps at a temperature less than 568°F.

6. A method of making detonating cord according to claim 1 wherein the metal is taken from the group consisting of aluminum and silver.

7. A flexible detonating cord made by the process of claim 1.

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