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[54] **METHOD OF PRODUCING FINE-CRYSTALLINE CAST CHARGES WITH UNORIENTED CRYSTALLINE STRUCTURE OF 2,4,6-TRINITROTOLUENE OR EXPLOSIVE COMPOSITIONS CONTAINING 2,4,6-TRINITROTOLUENE**

5 Claims, No Drawings

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[50] **Field of Search** 149/18, 107, 88, 105

[56] **References Cited**

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ABSTRACT: Fine-crystalline cast charges with an unoriented crystal structure of TNT or explosive charges containing TNT are obtained by adding 2,2',4,4', 6, 6'-hexanitrostilbene to the melt from which the cast charge is made.

METHOD OF PRODUCING FINE-CRYSTALLINE CAST CHARGES WITH UNORIENTED CRYSTALLINE STRUCTURE OF 2,4,6-TRINITROTOLUENE OR EXPLOSIVE COMPOSITIONS CONTAINING 2,4,6-TRINITROTOLUENE

The present invention relates to a method of producing fine-crystalline cast charges of 2,4,6-trinitrotoluene of explosive compositions containing 2,4,6-trinitrotoluene with an unoriented crystalline structure which, consequently, has an increased resistance to cracking.

At present, and for many year's time, 2,4,6-trinitrotoluene (TNT) has been the most widely used explosive in cast charges for shells, bombs etc., owing to its suitable properties for this purpose, e.g., low impact sensitivity, good stability, appropriate melting point and good fluidity in a melted condition. 2,4,6-trinitrotoluene can be used alone, but also with admixtures of e.g., a pulverized more violent explosive with a high melting point, whereby charges with an increased explosive effect are obtained. When charges are cast, the trinitrotoluene explosive is melted, and is cast into e.g., a shell, where it is allowed to solidify into a solid charge. Owing to the solidification contraction for trinitrotoluene and the difference in the coefficient of expansion between the trinitroluene explosive and steel, and the brittleness of the explosive etc. particularly complicated casting-technical procedure must often be used, as otherwise the cast charges of the trinitrotoluene explosive will have unsatisfactory properties, e.g., poor homogeneousness, cavities and cracks, low density and insufficient initiation sensitivity etc. Through the present invention it has now quite surprisingly become possible to achieve a cast charge in which the above-mentioned disadvantages have been eliminated at the same time as it has been possible to simplify the casting procedure.

When casting trinitrotoluene or explosive containing trinitrotoluene, as a consequence of a too small number of crystal nuclei and a slow crystallization, a strongly oriented, coarse crystal structure is usually obtained. In castings, the trinitrotoluene normally crystallizes in the form of markedly needle-shaped crystals, with the longitudinal axis at right angles to the cooling surface. Nearest to this cooling surface there normally first arises a layer of fine needle-formed crystals, but at the further crystallization very coarse crystal needles are obtained. This type of crystallization results in a characteristic pattern of needle-formed straight lines from the periphery in towards the center, which can easily be noticed if a casting is sawed into two pieces. In explosive compositions in which, at the casting, crystals of a more violent explosive have been suspended in the trinitrotoluene, the crystals obtained are, indeed, not as coarse as those obtained in pure trinitrotoluene, but the oriented structure is nevertheless substantial also in this case. This can be observed by the presence of the characteristic pattern also in this case.

The oriented structure of the trinitrotoluene involves considerable disadvantages in the cast charge; it will thus be less homogeneous, has lower density and less uniform sensitivity to initiation by detonators. However, the most inconvenient disadvantage is that since the crystals of the trinitrotoluene have an approx. four times higher coefficient of longitudinal expansion along the shortest axis than along the longest one, cracks arise between the crystals, parallel with the long axis, when the cast charge is cooling. This cracking is particularly inconvenient when making shell castings. The requirements for a cast shell charge are very stringent, particularly owing to the great acceleration that arises when the shell is fired through a gun barrel. This can involve that cracks in the cast charge and spaces between the charge and the shell body, particularly then in the rear part of the shell, can lead to bursts in the bore, as a consequence of unintentional initiation of the explosive, through impact or by adiabatic compression.

When using explosive compositions consisting of a high explosive and more sensitive explosive than trinitrotoluene (e.g., cyclotrimethylenetrinitramine) suspended in trinitrotoluene, the requirements for the east charge are still greater, and it has

therefore been considered advisable to cast the explosive so that it is fixed to the shell body, in order to entirely avoid a space between the explosive and the shell body. At such attempts to cast the explosive to that it adheres to the shell, the risk for cracking increases, however, owing to the difference in thermal coefficient of expansion between the steel in the shell body and the explosive.

In order to avoid cracking to the greatest possible extent, it has proved to be desirable to have an unoriented crystal structure in the cast charge, i.e., all crystal needles of trinitrotoluene are to lie unarranged, contrary to the oriented structure, in which the crystal needles are substantially parallel. The most simple method of obtaining such an unoriented structure seems to be to stir in fine-grained crystals of trinitrotoluene into the melt just before the casting takes place. These fine crystals would then be entirely unarranged in the cast charges, and would serve as crystallization nuclei. In practice, however, considerable difficulties are involved in carrying out this procedure, among other things owing to the fact that very accurate temperature control is required at the casting in order to obtain the effect desired. Nor can the method be applied if the trinitrotoluene contains another, suspended explosive, as the suspension will then as a rule have too high a viscosity to permit casting.

At another method, relatively large quantities of another explosive are added. During the cooling, the added substance precipitates if it is sufficiently sparingly soluble, and when the eutectic temperature is reached, a fine-crystalline eutectic mixture is obtained. However, this method has many disadvantages. For one thing, many explosives do not have the properties required. This is the case, for instance, of the cyclotrimethylenetrinitramine. Further, troublesome supercooling tendencies often occur, and a still greater disadvantage is that the mixture often obtains an altogether too low stiffening point, as a relatively large quantity of the substance added is required. At storage in a hot climate, the explosive in the shell can then exude an eutectic mixture that can run out in, for instance, the threads of the shell, causing risks in handling. Moreover, the explosives that have the properties required are expensive to manufacture.

Quite generally, it is not desirable to have any additives in military explosives which to any major extent change the composition and performances of the explosives.

Through the present invention it has proved possible to achieve an unoriented fine-crystalline structure of the trinitrotoluene and explosive compositions containing trinitrotoluene without having the disadvantage mentioned of the previously known methods. Through the invention it has moreover become possible to cast the explosive so that it adheres to shells without any critical cracking, which is remarkable with consideration to the heavy shrinking stresses that arise in connection with the casting of the explosive.

The method of producing fineH-crystalline cast charges with an unoriented crystal structure of the trinitrotoluene of explosive compositions containing trinitrotoluene is characterized according to the present invention in that 2,2', 4,4', 6,6'-hexanitrostilbene is added to the melt from which the cast charge is made. The quantity of hexanitrostilbene added should amount to not more than 2 percent by weight counted on the quantity of trinitrotoluene used, and can appropriately be limited to 0.5 percent by weight. The composition containing trinitrotoluene and hexanitrostilbene should appropriately be heated to 95°–100° C. and stirred so that as homogeneous a melt as possible is obtained, which melt is thereafter allowed to stiffen, after which it is heated again, but this time to a temperature immediately below 85° C., so that a new melt is obtained, which can thereafter be cast. In addition to hexanitrostilbene, also aromatic nitro compounds with a high solubility in trinitrotoluene can be added, which gives the trinitrotoluene greater plasticity.

As 2,2', 4,4', 6,6'-hexanitrostilbene has proved capable of serving as forming nuclei for trinitrotoluene, thereby leading to an unoriented fine-crystalline structure, it could possibly

have been expected that the same good results would also have been obtained with the addition of other polynitro compounds, if these only had a high melting point and low solubility in molten trinitrotoluene, and a molecular configuration similar to that of trinitrotoluene, but this did not prove to be the case. Trials have been made with, for instance, dipicrylsulfon (melting point 320° C.), hexanitroazobenzene (melting point 216° C.), dinitroantrakinon (melting point approx. 500° C.), trinitromesithylene (melting point 235° C.), dipicrylsulfide (melting point 234° C.), hexanitrobibenzyl (melting point 220° C.). Although up to 10 percent of these additives have been used, none of these substances had any nucleus-forming effect on trinitrotoluene.

In order to function, hexanitrostilbene must be present in the form of fixed particles in the molten trinitrotoluene. This is very easy to achieve, as the hexanitrostilbene dissolves only to approx. 0.2 percent by weight in molten trinitrotoluene at temperatures just above 80 ° C. Molten trinitrotoluene to which hexanitrostilbene has been added causes super cooling before the crystallization to a much lower degree than trinitrotoluene without such an additive. In a motionless melt, for instance in a shell where the cooling takes place from the wall and bottom of the shell, crystals begin to grow simultaneously from a large number of nuclei. A relatively thick moving zone is obtained within which the crystallization takes place (endogenous crystallization and thus not only a growing on of crystals along a layer parallel with the cooling surface (exogenous crystallization), and the unoriented crystal structure desired is obtained and, accordingly, also the higher degree of freedom from cracking.

Hexanitrostilbene differs in the present respect radically from the closely related compounds such as 2,4,6-trinitrostilbene and substituted 2,4,6-trinitrostilbenes, which cannot form nuclei for the trinitrotoluene. On the contrary, they have a great capability of preventing the formation of nuclei in trinitrotoluene, and also retard the crystallization speed of the trinitrotoluene and causes supercooling phenomena.

The substances which in earlier literature have been reported as 2,2', 4,4', 6,6'-hexanitrostilbene have subsequently proved to be 2,2', 4,4', 6,6'-hexanitrobibenzyl. The reported melting point is 211° C., while 2,2', 4,4', 6,6'-hexanitrostilbene melts at approx. 320° C. 2,2', 4,4', 6,6'-hexanitrostilbene was produced the first time in 1964 by K. G. Shipp. Hexanitrostilbene has good explosive properties, and has a stability which is higher than for most other now known explosives.

As examples of the aromatic nitro compounds which are easily soluble in trinitrotoluene which gave such eutectic mixtures with trinitrotoluene that an increased plasticity is obtained can be mentioned mono-, di- and trinitrotoluene, 2,3,5-trinitrobenzene, 2,4,6-trinitrophenyl methylnitramine (tetryl), nitronaphthalenes, nitrophenols, 2,4,6-trinitrophenyl ethanol, hexanitrobibenzyl and dipicryl sulfide.

In the following, the invention will be illustrated with the aid of some examples, which, however, do not limit the concept of the invention within the scope of the present patent claims.

EXAMPLE 1

Ninety-nine parts by weight of 2,4,6-trinitrotoluene and one part by weight of fine-grained 2,2', 4,4', 6,6'-hexanitrostilbene were mixed and melted at approx. 100° C. This melt was cast in a shell body and a corresponding casting was made with a melt containing only 2,4,6-trinitrotoluene but no hexanitrostilbene. After examinations of the two cast charges it could be established that the melt containing hexanitrostilbene gave an essentially more fine-grained crystalline structure than the cast charge made of 2,4,6-trinitrotoluene without the addition of hexanitrostilbene.

EXAMPLE 2

A mixture of 99.7 parts by weight of 2,4,6-trinitrotoluene and 0.3 arts by weight of pulverized but not particularly fine-grained hexanitrostilbene was heated to 100° C. while stirring. From the melt obtained, through an entirely conventional procedure, 2,4,6-trinitrotoluene flakes were thereafter produced. These 2,4,6trinitrotoluene flakes were thereafter melted at a temperature of approx. 84° C. and cast in a shell body, and for this melting at 84° C. and the subsequent casting a time of approx. 2 hours was used. The corresponding heating to 100° C. while stirring, transferring into flakes and renewed melting at 84° C. and casting in a shell body was carried out with only 2,4,6-trinitrotoluene. At the examination it was established that the crystal structure was considerably more fine-grained in the cast charges that contained hexanitrostilbene than the corresponding cast charge made of 2,4,6-trinitrotoluene without additive.

EXAMPLE 3

A mixture of 99.4 parts by weight of 2,4,6-trinitrotoluene, 0.3 parts by weight of hexanitrostilbene and 0.3 by weight of trinitrobenzene was heated while stirring to 100° C. and from this melt there was subsequently made, through a previously known granulating procedure (see e.g. Swedish Pat. No. 158,663) adding cyclotrimethylene trinitramine (hexogen) and wax, a granulated hexotol with the composition 60 percent cyclotrimethylene trinitramine, 39 percent 2,4,6-trinitrotoluene (containing hexanitrostilbene and trinitrobenzene) and 1 percent wax. This granulated hexotol was thereafter melted at approx. 84° C. and the melt was cast in a shell body. The corresponding procedure with heating to 100° C., granulation, renewed heating to 84° C. and casting was also carried out with hexotol to which hexanitrostilbene had not been added. At an examination of the two cast charges it was established in the cast charge made of hexotol to which hexanitrostilbene had not been added that an oriented crystal pattern had been formed, and that heavy cracks had formed in the cast charge. In the charge made of hexotol with hexanitrostilbene added, the crystal structure was extremely fine grained, and no formation of either oriented patterns of large cracks could be found in it.

We claim:

1. A method for producing cast charges containing 2,4,6-trinitrotoluene, said 2,4,6-trinitrotoluene having an unoriented crystal structure, which comprises adding 2,2', 4,4', 6,6'-hexanitorstilbene to the melt from which the cast charge is made.

2. A method according to claim 1 wherein the amount of 2,2', 4,4', 6,6'-hexanitrostilbene is not greater than 2 percent of the 2,4,6-trinitrotoluene.

3. A method according to claim 2 wherein the amount of 2,2', 4,4', 6,6'-hexanitrostilbene is not greater than 0.5 percent by weight of the 2,4,6-trinitrotoluene.

4. A method according to claim 3 and comprising the steps of:

heating the melt containing 2,4,6-trinitrotoluene and 2,2', 4,4', 6,6'-hexanitrostilbene at about 100° C. with stirring until a substantially homogeneous mass is obtained,

permitting the melt to cool until it stiffens,

reheating the melt to a temperature immediately below 85° C., and

permitting the melt to cool.

5. A method according to claim 1 wherein an aromatic nitro compound soluble in 2,4,6-trinitrotoluene is added to the melt.

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