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## DELAY FUSE COMPOSITIONS

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1

This invention relates to delay fuse compositions such as are used for the timing of the discharge of rockets, artillery shells, grenades, and other ordnance devices, as well as with explosives and pyrotechnical devices.

A considerable variety of such delay compositions have been proposed and used, but all of them of which we have knowledge are subject to various disadvantages, and none embodies to a satisfactory degree all of the various properties that are requisite in such compositions when stored or used under widely different conditions. For example, the burning rate of some compositions can not be varied within substantial limits, as is desirable for certain purposes, while others produce gaseous products of combustion, which is undesirable, particularly in the case of ordnance devices. Again, it is generally characteristic of the previously known delay compositions that they are not consistently reliable at low temperatures. The action of still others is adversely affected by elevated temperatures or by exposure to high humidity.

The primary object of the present invention is to provide delay fuse compositions that are highly reliable, whose burning rate may be varied accurately over wide ranges, which are useful at relatively low temperatures and whose efficiency is not adversely affected by exposure to high humidity or to elevated temperatures, or both, such as are encountered in warehouses, ships, and the like, in the tropics, which can be produced readily, and which produce substantially no gaseous products of combustion.

We have discovered, and it is upon this that the invention is largely predicated, that compositions conforming to the objects stated above are provided by intimate mixtures of powdered zirconium metal, powdered metallic nickel, an oxidizing agent that oxidizes the zirconium and nickel at a relatively high rate of speed, and another oxidizing agent that oxidizes the said metals at a relatively low rate of speed.

Actual experience with these compositions has shown that they are admirably adapted for use in delay elements. Thus, they may be ignited by any type of primer, their rate of burning may be varied within wide limits, and for any given burning rate the time of burning is accurately reproducible, e. g., the consistency of burning rate meets the rigid limits that are set by ordnance requirements. Experience has shown also that the preferred embodiment of these compositions stands up satisfactorily under surveillance, e. g.,

2

upon exposure to a temperature of 140° F. and 70 per cent to 100 per cent humidity. If the preferred compositions are dried after protracted exposure to such conditions it is found that their

performance remains unaffected, while if they are not dried they fire consistently although the speed of burning may be lowered slightly. Again, compositions according to the invention will satisfactorily ignite relay compositions of the type used to produce high order explosions.

A particular advantage is that these compositions may be balanced so that no gaseous products of combustion are formed. This permits the construction of various ordnance devices without the vents that have been necessary with prior types of such fuse compositions, whereby accuracy of high altitude firing is increased and there is nothing to indicate the course of, for example, a grenade such as may be the case where incandescent matter is expelled from the vents used heretofore. In addition to reproducing only solid products of combustion, the products of combustion that are formed tend to progressively seal the delay element, which is of advantage for some purposes.

In these compositions the zirconium functions to supply large amounts of heat and rapid combustion, and it is particularly of advantage because it ignites at very low temperatures, so that the compositions are perfectly reliable at temperatures as low, for instance, as minus 70° C.

The nickel is important because it supplies the main source of heat but because its heat of combustion is low, compared with that of zirconium, there is no tendency to burning or melting of bouchons or other metallic containers that carry the fuse compositions. Experience has shown that where surveillance is necessary, i. e., where the compositions are stored under high temperature or humidity conditions, the nickel must be supplied in the form of the extraordinarily fine nickel powder that results from evaporation of mercury of a nickel amalgam. Nickel in this form is made, for example, by the procedure described in Patent No. 1,893,879, granted January 10, 1933, on an application filed by one of us and Joseph C. W. Frazer. Briefly, a nickel amalgam is subjected to distillation under high vacuum to volatilize the mercury. The nickel remains as a fine powder that is so exceedingly fine as to have outstandingly high surface activity, and it has a very strong tendency to react with atmospheric oxygen. The powder may be stabilized, so that it can be handled freely in air, and without affect-

3

ing its activity, by passing carbon dioxide into contact with it subsequent to the distillation operation and prior to contact with oxygen. The use of nickel in this form appears to be critical where the desirable surveillance characteristics of the compositions provided by this invention are needed although other forms of finely divided nickel may be used where resistance to heat and/or humidity is not needed.

The metals and the oxidizing agents are used in proportions to give desired, i. e., predetermined, burning rates. That is, by increasing or decreasing the amount of nickel, for example, the burning rate can be increased or decreased accordingly. Likewise, by increasing the proportion of the slow oxidizing agent relative to that of the fast oxidizing agent the burning speed can be retarded, and vice versa.

As the slow oxidizing agent we now prefer to use oxidizing agents that are water insoluble. A variety of these are known, all of which act as relatively slow oxidizers of the metals used in these compositions, examples being the insoluble chromates, such as barium chromate ( $\text{BaCrO}_4$ ) and lead chromate ( $\text{PbCrO}_4$ ), manganese dioxide ( $\text{MnO}_2$ ), lead monoxide (litharge,  $\text{PbO}$ ), and red lead ( $\text{Pb}_3\text{O}_4$ ). A considerable variety of oxidizing agents are likewise known in the art that act to oxidize these two metals at relatively high rates of speed. Generally, the oxidizing agents of this class are salts of oxygen containing acids, such as the chlorates and nitrates, particularly those of the alkali metals. Of this latter class we prefer those that are water insoluble, or of low water solubility, most suitably potassium perchlorate ( $\text{KClO}_4$ ).

For most purposes we now prefer to use barium chromate as the slow oxidizing agent and potassium perchlorate as the rapid oxidizing agent. As exemplifying the manner in which the burning rate may be varied, reference may be made to the following typical compositions:

Zr	Ni	$\text{KClO}_4$	$\text{BaCrO}_4$	Sec./in.
Percent	Percent	Percent	Percent	
4.5	31	22	42	4.5
4.5	20	12	63	9.5
3.0	15	7	75	15

These typical compositions show how the burning rate may be varied by varying the amount of nickel and of oxidizing agent. Barium chromate being a slow oxidizing agent serves, in effect, as a diluent to retard the rapid reaction between the the potassium chlorate and the metals. For this purpose it is better than inert diluents, such as diatomaceous earth, because it provides a locus of nascent oxygen in contact with the metallic constituents throughout the composition and thus renders the action of the compositions much more uniform and reliable than where an inert diluent is used which separates the particles of active matter and may retard, or even prevent, reaction locally.

The compositions should be packed in delay

4

elements under a pressure sufficient to prevent segregation and layering of the constituents during transportation and handling. Generally speaking, a packing pressure of about 6000 p. s. i. is sufficient. A strong advantage of the compositions is that the use of greater packing pressures has little influence upon the performance of the compositions.

According to the provisions of the patent statutes, we have explained the principle and mode of practicing our invention and have described what we now consider to represent its best embodiment. However, we desire to have it understood that, within the scope of the appended claims, the invention may be practiced otherwise than as specifically described.

We claim:

1. A delay element composition comprising powdered zirconium metal, powdered nickel metal produced by evaporation of mercury from a nickel amalgam, a water-insoluble chromate, and an oxidizing agent selected from the group consisting of chlorates, perchlorates, and nitrates, said metals, chromate and oxidizing agent being in proportions adapted to give a predetermined burning rate with production substantially wholly of solid products of combustion.

2. A composition according to claim 1, said oxidizing agent being an alkali metal perchlorate.

3. A delay element composition comprising powdered zirconium metal, powdered nickel metal produced by evaporation of mercury from a nickel amalgam, barium chromate, and potassium perchlorate, said materials being present in proportions adapted to give a predetermined burning rate with production substantially wholly of solid products of combustion.

4. A delay element composition comprising powdered zirconium metal, powdered nickel metal produced by evaporation of mercury from a nickel amalgam, an oxidizing agent selected from the group consisting of chlorates, perchlorates and nitrates, and an oxidizing agent selected from the group consisting of water-insoluble chromates, manganese dioxide, lead monoxide, and red lead, and said metals and oxidizing agents being in proportions to give a predetermined burning rate with production substantially wholly of solid products of combustion.

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