CHAPTER 01

General Introduction

1.1Pyrotechnics:

Pyrotechnics are an important class of energetic materials and are called the brain of any weapons system. The name pyrotechnics is derived from Greek word 'pyro' which means fire and 'technics' means art, so pyrotechnic means 'art of fire'. Pyrotechnics are composed of fuels and oxidizers together with certain additives. Pyrotechnic fire can not be suffocated- no air is needed for these mixtures. The pyrotechnic compositions burn but not detonate. Pyrotechnics produce the following effects on burning: [1.2]

- Light
- Heat
- Sound
- Gas or motion etc.

These effects are utilized to design pyrotechnic devices for various civil as well as military applications.

1.2 Pyrotechnic devices:

The devices which are filled with pyrotechnic compositions and produce effects as;

- Igniter
- Heating devices
- Gas generator

- Smoke Generator
- Fire work display
- Illumination
- Delay devices

1.3 Application of pyrotechnic devices:

- Pyrotechnics devices are used in very critical applications such as safety operation for opening of emergency exit; an improper operation could lead to serious accidents.
- Numbers of pyrotechnic devices are used in civilian rocket system e.g. pay load separation, 1st, 2nd and 3rd stage boosters separation etc. [3].
- These devices are used for release of stores form military aircraft
- Like other applications one of the most important applications is that these are used as delay devices in sophisticated missile to provide required delay time before performing certain effects.

Aircraft system:

a. Seat ejection systems
b. Release ejections for tanks, stores and equipments
c. Emergency and Rescue system
d. Drone systems

Spacecraft systems:

a. Launch and control systems
b. Emergency systems
c. Stage separation systems
d. Recovery and landing systems

Missile systems

a. Safety and Arming systems

b. Ignition systems

c. Control systems

d. Stage separation systems

e. Destruct systems

1.4 Types of pyrotechnics devices:

1.4.1 Types of pyrotechnic devices on the basis of mode of initiation:

1.4.1.1 Electrically initiated delay devices (delay detonators):

Electric delay detonator is initiated by an electric pulse provided from a battery or any other source. There are two main categories of electrical delay detonator

a. Short delay detonators which are measured in mille second [4].

b. Long delay detonators which are measured in second

Their main components of electrically initiated delay detonators are

- Electric Primer
- Delay body
- Delay composition
- Secondary charge/Bursting charge

1.4.1.2 Mechanically initiated delay devices such delay cartridges:

Mechanical delay detonator is initiated mechanically. Its main components are

- Mechanical percussion
- Delay body

- Delay composition
- Detonating charge/Bursting charge

1.4.2 General types of pyrotechnics delay devices:

1.4.2.1 Obturated delay devices:

These are designed to contain all of the gases produced by the functioning of the igniter and delay compositions before the functioning of bursting charge [5]. Delays in which the gases produced are internally accumulated in a closed chamber in the delay devices are considered to be obturated. These are completely sealed and are not influenced by the effect of the ambient pressure or humidity. Short time delays are often obturated because obturation tends to decrease the average burning time of delay composition in delay detonators.

1.4.2.2 Vented Delay Devices:

Vented delay devices have opening through which the gases produced by the burning of delay/ignition compositions may escape. Vented delays devices are used when large quantities of gas are produced by the burning of the pyrotechnics delay composition and may even be used for gas less pyrotechnic delay compositions when long delay time is desired. In order to eliminated pressure build up with in the delay devices. Venting exposes the burning delay composition to ambient pressure. As a consequence the burning rate of delay composition is sensitive to change in altitude. These vents are required to be sealed up to the time of functioning in order to protect the delays composition from humidity.

1.4.2.3 Confined delay devices:

These are delay devices in which delay composition is completely confined in delay body and no vent space is provided in delay body. It has been reported that when pressure behind the burning front in vented and confined condition is important in increasing the burning rate. The initial pressure developed in confined system is the gas evolved during ignition of primer and delay compositions [6]

1.5 Pyrotechnic compositions:

Pyrotechnic compositions consist of Fuel, Oxidizers and some types of additives.

1.5.1Fuel:

The fuel reacts with the liberated oxygen to produce an oxide and heat. The heat liberated produces a numbers of effects i.e. color motion, light, smoke and noise. Fuel is either a metal, non-metal, In-organic and Organic compounds.

Commonly used fuels for military pyrotechnics delay compositions are boron, silicon, zirconium, magnesium etc.

1.5.2 Properties of fuel:

- Should possess a reasonable heat of combustion
- Should readily oxidized by oxygen
- Form product which gives the required effects
- Should require minimum quantity of oxygen for burning
- Chemically and physically stable in temperature range -40^{0}C to +70^{0}C
- Should be non hygroscopic
- Not be toxic
- Should be easily available

1.5.3 Oxidizer:

In a pyrotechnic reaction the energy is released by oxidation of fuel. The atmospheric oxygen is not required for this process. The required oxygen is delivered from chemical compound called oxidizer. The oxidizer is oxygen rich solid that decomposes, liberating oxygen gas that then reacts with fuel.

Commonly used oxidizers for military pyrotechnic delay compositions are $KCLO_{4}$, Pb_2O_3, PbO_{2}, etc.

1.5.4 Properties of oxidizer:

- Should contain maximum quantity of oxygen
- Should easily give up oxygen during burning process.
- Should be chemically and physically stable in the temperature range $-40^{0}C$ to $+70^{0}C$
- Should be-non hygroscopic
- Be easily available
- Should by non toxic to human beings.
- The oxidizer should have minimum sensitive to mechanical shocks otherwise these can impart explosive properties to the delay composition.

1.5.5 Additives:

Binders: Binders are compounds which increase cohesion between particles of fuel and oxidizer aiding consolidation. Binders also modify the burning rate and thus the performance and also reduce the sensitivities to stimuli as friction.

1.6 Ignition compositions:

In igniter and first fire composition hot solid of liquid particles are desirable to insure the transfer of sufficient heat to ignite the main delay composition.

Fuels producing mainly gaseous products are not commonly used in igniter and first fire composition.

1.7 Pyrotechnic delay compositions:

A general term for a mixture that burns at a selected, reproducible rate, providing a time delay between activation and production of main effect.

A pyrotechnics delay composition provides a predetermined time delay between ignition and the delivery of main effects.

The modern practice of requiring pyrotechnic delays to function very accurately has let to an intensive research into the mechanism and control of combustion both of gas producing and of gasless pyrotechnic mixtures and at the same time it has narrowed the field of investigation to the relatively few combustible materials that can be considered suitable for this kind of precise applications.

1.7.1 Gassy delay compositions:

- Are used in varied conditions and low attitudes e.g. black Powder.
- The burning rate of delay compositions can be very fast i.e. mm/ m sec or quiet slow mm/sec.
- Those pyrotechnic delay compositions which have a fast burning rate of more then 1 mm/ m sec are used in projectiles and bombs that detonate on impact.
- Pyrotechnics delay compositions which have a slow burn rate between 1 and 6mm/ sec are used in ground chemical munitions such as tear gas and smoke grenades.

- Change in ambient pressure affect burning rate of gassy compositions and they must be freely vented to the surrounding atmosphere in order to get constant burning rate.

1.7.2 Gasless delay compositions:

Gasless delay compositions are used in confined conditions or at high attitude, where it is important that variations from normal, ambient condition do not occur i.e. Boron, Silicon and potassium dichromate, Lead chromate, barium chromate and manganese etc.

These compositions are used for producing a controlled amount of heat and for time delays in a number of military applications. The combustion is characterized by high reaction temperature and formation of mainly solids products. By controlling the properties & proportions of the ingredients both the burning rate and calorific output can be varied over fairly wide range. Gasless pyrotechnics delay compositions are particularly valuable for use in short delays detonators.

Gasless delay compositions are usually a combination of a metal oxide or chromate with an elemental fuel. The fuels are metals or high heat non-metal elements such as silicon or boron.

When organic binder is used (Nitro cellulose) the resulting mixture will be low gas rather than gasless due to the CO, CO_2, and N_2 that will result from upon combustion of the binder used for granulation purpose.

1.7.3 Properties of delay compositions:

- Pyrotechnic delay compositions should not detonate but only burn.
- Some compositions when heavily confined incorrectly initiated by stimulus are capable of detonation.

- Pyrotechnics when accurately initiated provide special effects.
- The pyrotechnics mixture must be stable during preparation and storage.Materais having low hygroscopic must be used.
- The pyrotechnics mixture should be easily ignited from ignition stimulus.
- There must be minimum variation in the burning rate of the composition with changes in external temperature and pressure. The pyrotechnics mixture must be readily ignited and easily burn at low temperature and pressure.
- There should be a minimum change in the burning rate of pyrotechnics composition with small percentage change in the various ingredients ratio.
- The burning rate of pyrotechnic composition must be reproducible, both within a batch and between different batches.
- The oxidizer should be exothermic or slightly endothermic
- For slower delay composition, metals with less heat output per unit mass should be selected and oxidizer with high decomposition temperature and more endothermic heat of decomposition should be selected.
- The ratio of oxidizer to fuel can be changed for a given binary pyrotechnic composition to get substantial changes burning rate.
- The fastest burning rate should correspond to an oxidizer/fuel ratio near the stoichmetric ration, with neither substance present in substantial excess.
- When the density of the pyrotechnics composition increases due to increase applied pressure the components are pressed closer together and better heat transfer occur delay compositions.

- When a fast burning rate is desired, a metallic fuel with high heat output per unit mass should be used together with an oxidizer of low decomposition temperature.

1.8 Delay time:

The delay time of an electrically initiated delay detonator is defined as the time from the input of a signal supplied to primer and appearance of flash when it ruptures or breaking of wire at the end of delay detonator.

1.9 Delay Detonator:

A delay detonator is a device which provides a predetermined time delay between ignition and the provision of main effects. The initiated charge is ignited by an electrically heated bridge wire.

The forward end of the delay column, which is adjacent to the initiator charge is made from an easily ignitable and fast-burning metal oxidant composition device to ignite the main part of the delay column, which is made of slow burning pyrotechnic delay composition.

The bursting charge mainly consists of a pressure generating compositions which provide the required power to perform some desired work.

Short delays detonators are used in bomb and missile to detonate the high explosives until some penetration of the target has taken place [7].

1.9.1 Mechanical delay detonator:

1.9.1.1 Percussion delay detonator:

In this type of delay detonator, a firing pin hits the percussion cap and thus the detonator is initiated mechanically. In this type of detonator impact sensitive composition is used.

1.9.1.2 Stab delay detonator:

Stab delay detonators are those in which a stab sensitive composition is initiated by striking a pin.

1.9.2 Electrical delay detonator:

1.9.2.1 Thin Film delay detonator:

In this type of detonator, a thin film is deposited on primer, current flows through the thin film; it gets heat up and thus provides the required energy for the ignition composition to initiate it.

1.9.2.2 Bridge wire delay detonator:

In this type of detonator a resistive wire/bridge wire is spotwelded on the stainless steel wire. When current flows through the resistive wire it heats up and provides the required energy to priming composition for initiation.

1.10 Components of delay detonator:

1.10.1 Initiating/Priming charge:

The first component of the train is called initiator. It produces flash or flame and imparts igniferious effects on the next component.

1.10.2 First fire:

It is a sensitive composition at the primer end of delay composition either in loose form or pressed. It is sensitive to initiate the delay composition.

1.10.3 Delay composition:

A pyrotechnic delay composition provides a required time delay between ignition and the delivery of desired effects.

1.10.4 Base charge/Secondary charge:

It is a high detonation velocity secondary high explosive pressed at the end of delay composition to provide required shock waves to initiate the booster.

1.11 Parameters responsible affect burning rate in delay detonators:

The burning rate in a pressed column of gasless pyrotechnic delay composition is a combustion reaction. In this type of reaction the fuel and oxidizer react to give mainly solid products. The gases which are formed during this reaction are mainly CO_2, H2, N2, CO and traces of organic materials. Factors affecting burning rate of delay composition are

Particles size:

Particles size of compounds of delay composition is of great influence as far as metal fuels are concerned. The surface area of fuel or diameter of fuel is very important. The grain size of an oxidizer such as potassium per chlorate has not great effect on the burning time, though it may influence ignitibility. An effect of particular importance is that of the practical size of the ingredients, particularly the fuel. The finer is the material is the greater the surface area of material available for reaction, and the faster the compositions will burn. The specific surface of the fuel usually has better effect on the burning rate of the composition then mean diameter. Combustion is a surface phenomenon greater is the surface area greater will be burning rate. Compound is passed through a particular sieve after grinding. The more the ingredient is irregular the faster will be burning rate.

Percentage of fuels:

The percentage of fuel in a composition has a greater influence on burning rate of pyrotechnics delay composition burning rate become faster and faster with excess of metal. This causes to increase heat conductivity of the composition rather than of the increased surface area of the active fuel. By increasing percentage of fuel in composition the reaction will be mainly solid/solid nature and vice versa [8].

Confinement effect:

If the confinement of low gassing mixtures provides only a small vent volume, the burning time of the column is considerably speeded up. When a burning pyrotechnics composition is confined, pressure can increase to very high limit. Temperature also increases when a burning composition is confined, because high temperature gases and radiant energy cannot escape. Both temperature and pressure act to increase burning rate of delay compositions. Thus, confinement can considerably increase burn rate.

Diameter effect:

The effect of the diameter of the delay column become critical when the composition burn slowly and the heat output is small, as is often the case. A column diameter of 0.20 inches is usually adequate and if ambient temperature is never expected to be very low, accurate burning is possible at 0.11 in diameter [9].Otherwise a composition may perform adequately at normal and at higher temperature but when temperature very low enough heat may soaked away in the surrounding delay body that the glow front becomes narrower and literally tapers off so that burning may terminate before the terminal fire transfer point is reached. Radial losses of heat from delay composition can stop or extinguished the reaction in a delay column. Such losses become more serous as the column diameter, burning rate and ambient temperature are reduced. These effect combine to cause in a failure diameter associated with a given delay composition for a given ambient condition.

Conductivity of delay body:

High conductivity delay body may cause a premature ignition of whatever bursting charge follows the delay composition.

Effect of ingredients:

The chemical nature of compounds has greater effect on the rate of propagation of a chemical reaction for many reasons. For any chemicals reaction to occur between fuel and oxidizer it is necessary for there to be intimate contacts between them or at least between one of them and decomposition products of other. Intimate contact is produced by compaction of the reactants.

Effect of ingredients ratio:

The rate of reaction is related to the exothermicity of the composition is generally observed that in a binary mixture the compositions of maximum burning rate has more fuel then the mixture of maximum exothermicity.The thermal conductivity of fuel is usually greater then that of oxidizer. For boron potassium nitrate mixture system the composition has maximum burning rate occurs at 50% fuel. The exothermicity of this composition is maximum near 17% boron.

Effect of initial temperature:

At low temperature pyrotechnics burn more slowly then at higher temperature, Burning rate of pyrotechnic composition is given by.

$$R = r_0\, e^{bT/T_0}$$

Where R, r_0 are burning rates at absolute temperature T, T_0 and b is constant for the pyrotechnic composition. The higher the initial temperature of the reactants, the more easily will the delay composition achieves required activation energy. According to quantum mechanics, the relative concentration of the same compound in two different energy levels is given by

$$C/C_0 = e^{-(E-E0)/KT}$$

Effect of ambient Temperature:

The ambient temperature of the surrounding and also the temperature of the unreacted delay composition effect burning rate. As ambient temperature is raised, activation energy is lowered because less energy is required to raise a composition to its ignition temperature [9]. Thus the burning rate increases and less time is required to reach the ignition temperature. The burning rates of delay composition were found to increase when temperature is increased. For 90/10 barium chromate- boron composition burning rate increases when temperature is increased.

Effect of ambient Pressure:

Energy in the combustion zone is that radiated back from the hot products also increase burning rate. If the combustion products are gases then the contribution should be sensitive to the gas pressure. If the products are not gaseous, then the gas pressure will have small effects. Reality lies between the two. The pressure dependence generally follows vielle's law

$$R = BP^{\alpha}$$

^{R} is the burning rate at constant pressure P and α, β is constant for chemical. The more is the gassy composition the greater is the value of α.

Effect of consolidation pressure:

The effects of consolidation pressure are more complex. If combustion reaction is completely solid phase (i.e. no melting in the combustion zone) then increase in loading pressure should allow better contacts between reactants, and the reaction rate should increase if the reaction is not solid phase, then the effects is less predictable. The decrease is porosity prevent hot gases permeating the reactants and so reduce the burning rate. In most cases it is the linear burning (mm/s) of the composition, which is of interest to the designer, but the mass consumption rate (g/sec) is also importance because that is a measure of the power out put from the device.

Effect of water:

The effect of water is very important. Water can act as a volatile heat sink and make the composition less sensitive. Water can increase the pressure in an unvented system.Sensititvity to moisture may cause ignition failure, effect burning rate or chemically destroy the compositions, has become a problem in delay work most of the delays are now properly sealed[10].

Effect of terminal charge:

The burning characteristics of pressed pyrotechnic delay compositions are changed when pressed above a thermally sensitive bursting charge. The overall burning time and the reproducibility are both decreased under these conditions. This anticipatory effect has been observed with a variety of thermally sensitive terminal charges for both gaseous and gasless pyrotechnic delay compositions. The effect has also been observed for typical end item delay composition having a lead styphnate-Lead Azide as a end charge. The extent of the reduction in burning time that occurs with delay elements having thermally sensitive terminal charges, compared with similarly pressed delay columns without a terminal charge, approaches a constant value when the length of the delay column above the terminal charge increases. The reduction in burning rate is small for fast burning compositions.Obturation of the delay column substantially increases the magnitude of this effect. The anticipatory effect is reduced by barriers, between the delay columns and the thermally sensitive terminal charge, which would reduce the flow of gases.

Effect of ignition composition:

Some gasless pyrotechnic delay compositions are difficult to initiate. Normally a small charge of an ignition composition on top of the delay column is pressed. Silicon powder fuel form very sensitive ignitable mixture with oxides of lead e.g. Pb_3O_4 or PbO_2. Pyrotechnics compositions containing Silicon, red lead and titanium are very popular [11]

Effect of delay body material:

The body into which a delay composition is loaded act as a heat sink, as metals in general is better conductors of heat than delay compositions. Delay columns close to their low temperature failure diameters tend to have larger temperature coefficient as the surrounding wall thickness is increased. For materials well above their failure diameters, this effect of wall thickness is of greater importance.

Effect of Storage on burning time:

Normally ammunition are stored for a long time, it is important to know the effect of storage on burning times of relatively long periods of time results in little change in burning rate e.g. Barium chromate-boron compositions loaded and stored over a desiccant show little change in burning rate up to 3 Years. For loose powders stored up to two years in unheated magazines and then loaded a in delay body slight increase in burning time has been observed. Storage under dry conditions prevents further increase in burring time and may reverse the trend.

Charge increment:

To get delay time with high degree of accuracy the charge composition is pressed in small increments.

Effect of additives:

Additive are added to delay composition, they may produce less energy than the principal fuel or oxidizer. The presence of these additives lowers the overall heat of reaction for the delay composition, and therefore, generally increase the burning time as well. An additive is used for the primary purpose of adjusting burn rate up or down. A burn rate modifier can act to reduce burn rate as described for typical additives.

A burn rate modifier can act to increase burn rate. This will occurs when it increases the heat of reaction or if it increases the efficiency of thermal energy feedback.

Effect of physical form of burn rate:

There are two general types of pyrotechnic burning.

1. Parallel: Burning layer by layer.

2. Propagative: In this type of burning individual grain burns layer by layer and then engulf all grains. The rate of burning is much greater in propagative burning then in parallel burning.

1.12 Product specifications:

In view of the various parameters discussed above, it is intended to carry out R&D work on a Pyrotechnics delay detonator having the following specifications.

1.12.1 Specification of hot bridge wire:

Table: 1.1Hot bridge wire

Description	**Diameter of wire**	**Length of wire**
Nicrome(Nickel chromium alloy)	0.025 mm	1.5mm

1.12.2 Electrical Specifications

Min. all Fire current	1.5 amperes
No fire current	0.1 amperes
Normal firing voltage	12 V DC

1.12.4 Performance Parameters

Delay Time To proved consistence delay time

1.13 Importance of delay time in delay detonator:

The time of delay detonator is very critical. The delay time should be with in the range and reproducible. Any deviation from the required specification can cause mission failure. The delay detonator initiates the booster and then the main charge before certain time of delay in order to get the desired objectives.

1.14 Energy of initiation:

Different delay detonators have different initiation energy requirement dependent on the initiating charge and bridge wire used. If very sensitive explosives such as lead azide or lead styphnate are used as initiating charge the energy requirement is very small. The bridge wire also affects the energy supplied to the delay detonator for initiation. When the diameter of the bridge wire is increased the resistance of the wire is decreased and thus more energy is required to initiate the same sensitive explosives.

1.15 Pyrotechnics train:

Some pyrotechnics compositions are difficult to initiate an explosive train similar to that used in other explosively loaded items is used to produce the ignition stimulus required to initiate the main pyrotechnics compositons.Such a train can be divided into three prats.The first part contains a sensitive initiating composition that can be initiated by small electrical stimulus. This initiating composition on burning produces enough heat to initiate intermediate pyrotechnics compositiosn.The output of this intermediate thus initiate the main bursting charge.

1.16 Work already done on pyrotechnics delay detonators.

A lot of work on pyrotechnic delay detonators especially on short delay time detonators for missiles has been done. Data on pyrotechnics delay devices and on their burning time is available in different books.Commony used pyrotechnic mixture are Si/Pb_3O_4 for short delay and $Sb/KMnO_4$ for long delay time. Short pyrotechnic delay detonators are used for most applications because they give best results [12]. Some of the ignition and delay compositions which are being using in pyrotechnics devices are given below.

Table-1.2: Effect of applied pressure on burning rate of Barium Chromate-Boron composition (90:10)

S#	Oxidizer %age	Fuel %age	Pressure applied PSI	Burning rate Sec/inch
1	90	10	36000	0.670
2	90	10	18000	0.653
3	90	10	13000	0.558

Table-1.3 Effect of applied pressure on burning rate of Barium Chromate-Boron composition (95:5)

S#	Oxidizer %age	Fuel %age	Pressure applied PSI	Burning rate Sec/inch
1	95	5	36000	1.69
2	95	5	18000	1.60
3	95	5	13000	1.29

Note: With increasing loading pressure burning rate decreases.

Table-1.4 Lead peroxide-Boron delay compositions

S#	Oxidizer %age	Fuel %age
1	70	30
2	89-96	4-11

Table-1.5 Nickel-Zirconium alloy-Barium chromate and Potassium perchlorate delay compositions.

S#	Nickel-Zirconium alloy	Barium Chromate %age	Potassium perchlorate %age
1	26	60	14

Table-1.6 Nickel-Zirconium mixture- Barium chromate and Potassium perchlorate delay compositions.

S#	Nickel-Zirconium mixture	Barium Chromate %age	Potassium perchlorate %age
1	Nickel = 5% Zirconium = 31%	22%	42%
2	Nickel = 5% Zirconium = 17%	70%	8%

Table-1.7 Silicon-Red lead delay compositions.

S#	Silicon(Si)	Oxidizer(Red lead)
1	20%	80%

Table-1.8 Zirconium- Lead dioxide delay compositions

S#	Zirconium(Zr) Fuel	Oxidizer(Red lead)
1	28%	72%

1.17. Research Objectives.

The basic objectives of this research are to design and develop a pyrotechnic delay detonator to be initiated by electrical energy and produce consistence delay time. Time consistency plays an important role in pyrotechnic delay detonators, since the delay detonator is required to function within required delay time. The results of pyrotechnic delay detonators must be reproducible otherwise it can cause a mission failure. This present work includes a detailed literature survey, study of different ignition and delay compositions, and identification of parameters which affect burning time in pyrotechnic delay compositions in delay detonators. Various parameters responsible to affect burning time have been studied and investigated in detail and effect of confinement, Obturation and Venting of delay body on burning rate of pyrotechnic composition have been determined and compared. However, certain constant parameters can not be changed during experimentation e.g. energy provided from discharge of a capacitor, resistance of bridge wire etc.

CHAPTER 02

Experimental:

2.1 Chemicals used.

The following different types of chemicals have been used in ingnition, delay compositions and bursting charges.

Table-2.1 Chemicals for Ignition compositions:

S#	Chemical Name	Source
1	Potassium chlorate($KClO_3$)	Cat#12634 Fluka (99%)
2	Lead thiocyanate Pb $(SCN)_2$	Cat#529338(Aldrich 99%)
3	$BaCrO_4$	Cat#00633(Fluka 99%)
4	Bi_2O_3	Cat#10305(RDH 99%)
5	$KClO_4$	Cat#0441(Fluka 99%)
6	Mg powder	Cat#63034(Fluka 99%)
7	Boron	Cat#15580(Fluka 98%)

Table-2.2 Chemicals for delay compositions:

S#	Chemical Name	Source
1	Silicon	Cat#215619(Aldrich 99%)
2	PbO	Cat#15338(Fluka 99%)
3	Pb_3O_4	Cat#241547 (Aldrich 99%)
4	Zirconium	Cat#14602(RDH 98%)

Table-2.3 Bursting charge

S#	Chemical Name	Remarks
1	Service Lead Azide	Primary high explosive
2	Dextrinated Lead Azide	Primary high explosive
3	Tetryl	Secondary High Explosives

2.2 Hardware used.

The following different types of hard ware have been used in pyrotechnic delay detonators.

2.2.1 Bridge wire: Nichrome wire of 25 micrometers diameter was spot welded as a bridge wire on both legs of stainless steel wire.

For a given bridge wire, the higher the applied electric current the shorter the time period for the current flow for explosive vaporization. The material diameter and length of a bridge wire for a specific application are determined according to the desired resistance.

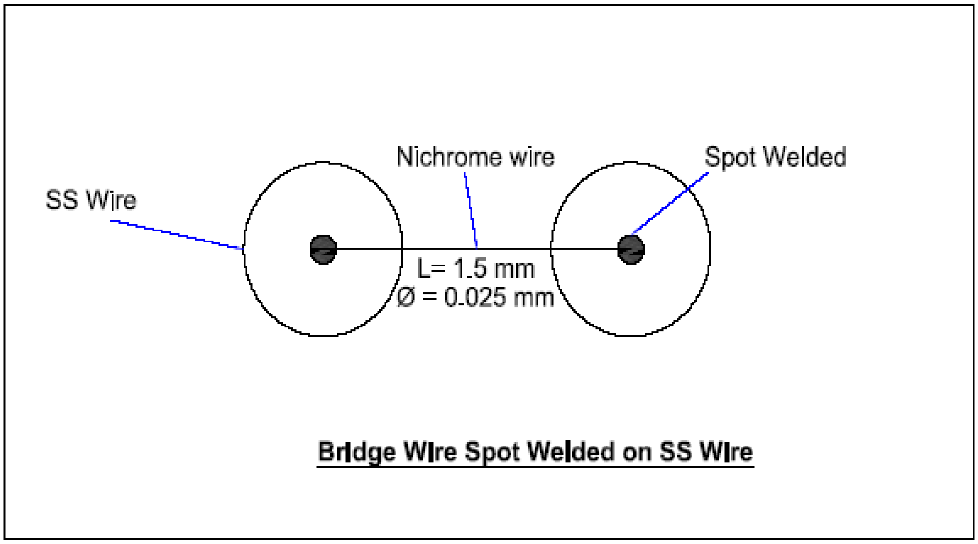

Fig: 2.1 Bridge wire for delay detonator

2.2.2 Primer: Primers were manufactured (molded) from Bakelite inserted with S.S wire as a two legs as per required dimensions.

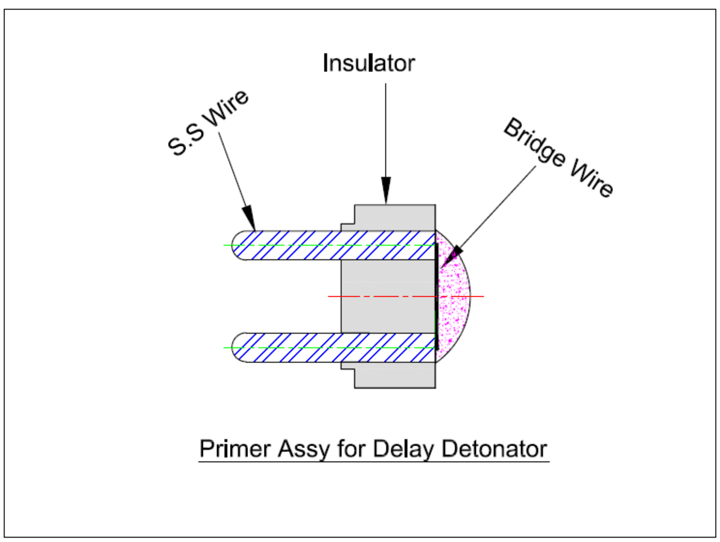

Fig: 2.2 Primer for delay detonator

2.2.3 Delay body: Aluminum AA-2024 as per specifications was used for manufacturing of delay body/cup of delay detonator.

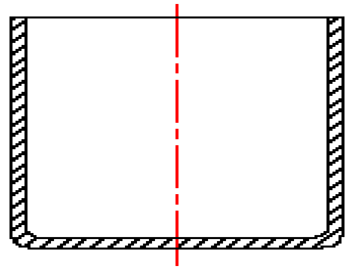

Fig: 2.3 Body for delay detonator initial design

2.3 Ignition and delay composition preparation procedure.

2.3.1 Preparation of individual components:

The individual components i.e. fuels, oxidants and additives were dried and ground in mortar and pestle. After mixing the individual components were then passed through 325 mesh sieve having grain, size of 47 micrometer. After these processes, the individual components were dried at 105 ^{0}C.

2.3.2 Preparation of composition/mixing:

The individual ground components were weighted as required and mixed together in remote automatic mixing machine. After proper mixing, the composition was then passed through 65 mesh sieve having 212 micrometer grain size. The mixing is the key process. It is to done thoroughly to attain homogeneity. Owing to hazardous nature of the process, a special type of remotely controlled machine was used for mixing of delay and ignition compositions.

2.3.3 Granulation:

The mixed compositions were transferred into granules for ease of loading in delay detonators; small quantity of binder i.e. 0.2% Fish Glue using ethyl acetate as solvent was used for the formulation of granules. As it was a small scale experiment, the granulation was done by wetting the composition in solvent. For larger scale process, special granulation machines are normally used. The compositions were converted into granules in order to prevent separation of oxidizers and fuels due to density differences.

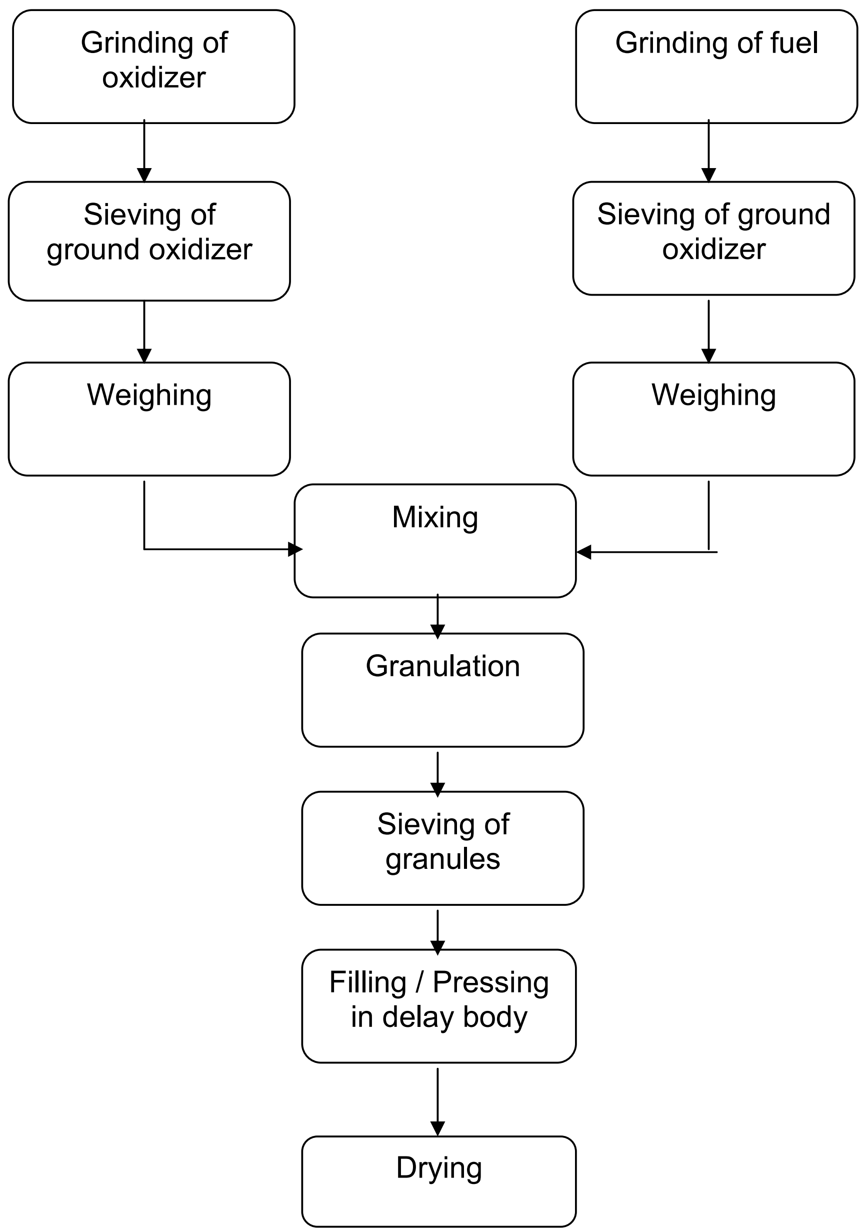

Fig 2.4: Process flow for preparation of ignition/delay compositions

2.4 Pasting of ignition compositions on primer.

Pasting of ignition composition on primer involves following steps:

- Preparation of past of ignition composition in Nitrocellulose lacquer.
- Application of past of ignition composition on primer
- Drying the primer for ½ Hrs in Oven at 105 ^{0}C

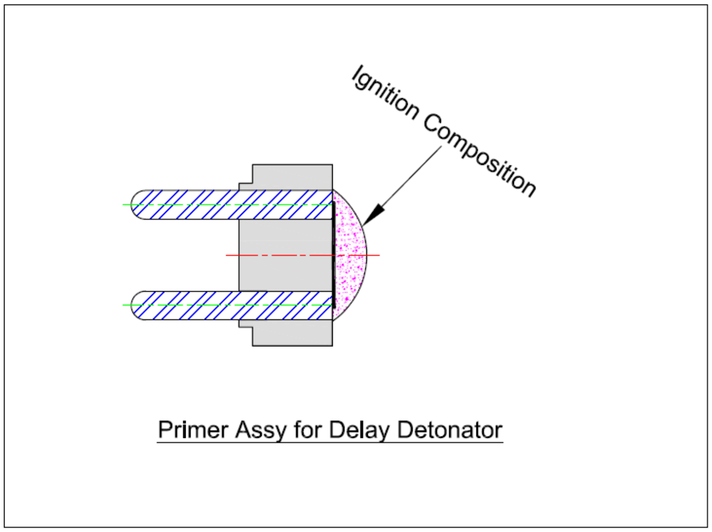

Fig: 2.5 Primer pasted with ignition compositions

2.5 Loading of delay compositions in body delay detonators:

The granulated composition was then loaded into the delay detonator bodies. The compositions were then pressed in body to get the desired consolidation. The loading was done in small increments in order to get consistent delay time. Special designed tools and dies were used during the filling and pressing process.

2.6 Procedure for preparation of primer for delay detonator:

Preparation of primer for delay detonator comprising following steps:

- Spot welding/Soldering of Nirchrome on the legs of primer.
- Preparation of primer composition.
- Pasting of primer composition in wet condition on primer bridge wire.
- Drying of pasted priming composition in the oven.
- Checking of resistance of primer before main assembly.

- All the safety precautions were observed for handling of explosives and pyrotechnics compositions during preparation of primer for delay detonator.

2.7 Assembly procedure of delay detonator:

- Preparation of delay composition as per required percentage of fuel and oxidizer.
- All the safety precautions were observed for handling of explosives and pyrotechnics compositions during preparation of delay compositions and assembly of delay detonators.
- Weighing of empty delay detonator body.
- Filling of delay composition in the delay body as per requirement.
- Weighing of filled delay detonator and record quantity of delay composition.
- Assembly of detonator filled with (Service Lead Azide and Tetryl) as a bursting charge.
- Crimping of detonator in the delay body and applying sealant viz araldite on crimped face and drying it at ambient temperature.
- Assembly of primer assembly in the main delay detonator body.
- Crimping of primer assembly in the delay detonator body and applying sealant araldite and drying it at ambient temperature.
- Checking resistance of delay detonator with multi-meter.
- Checking of physical and dimensional parameters.

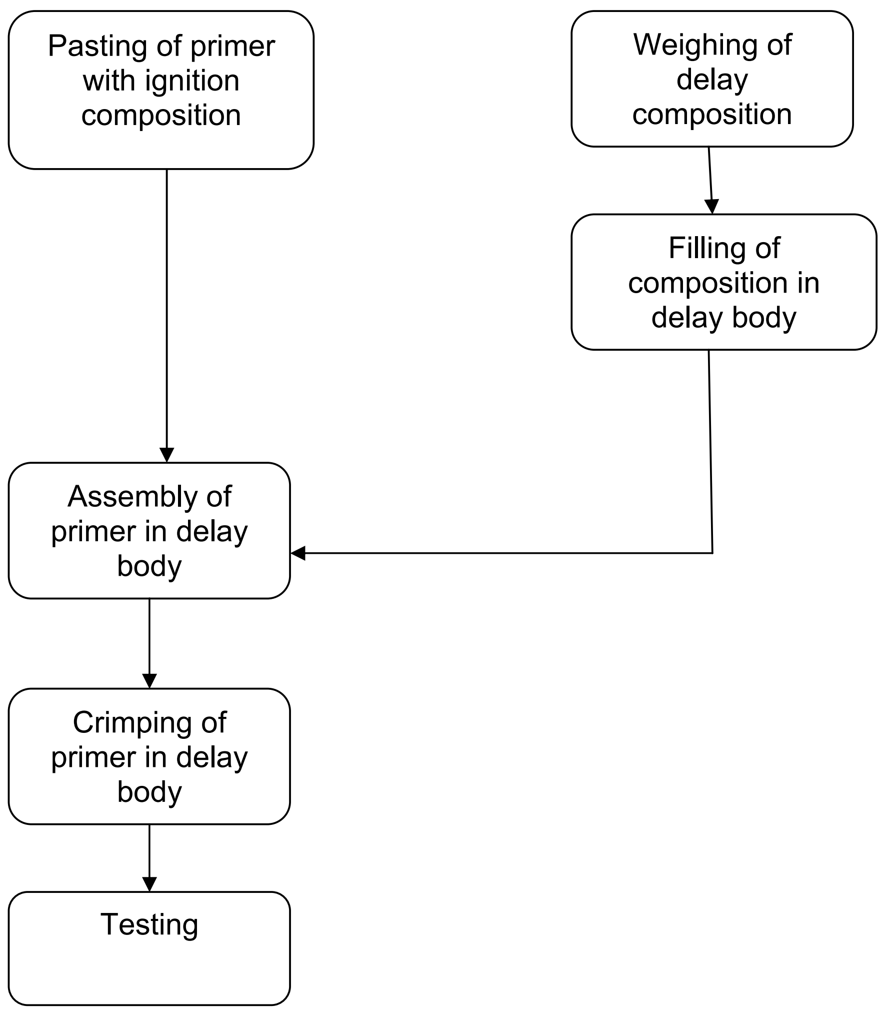

Fig 2.6: Process flow for manufacturing of delay detonators

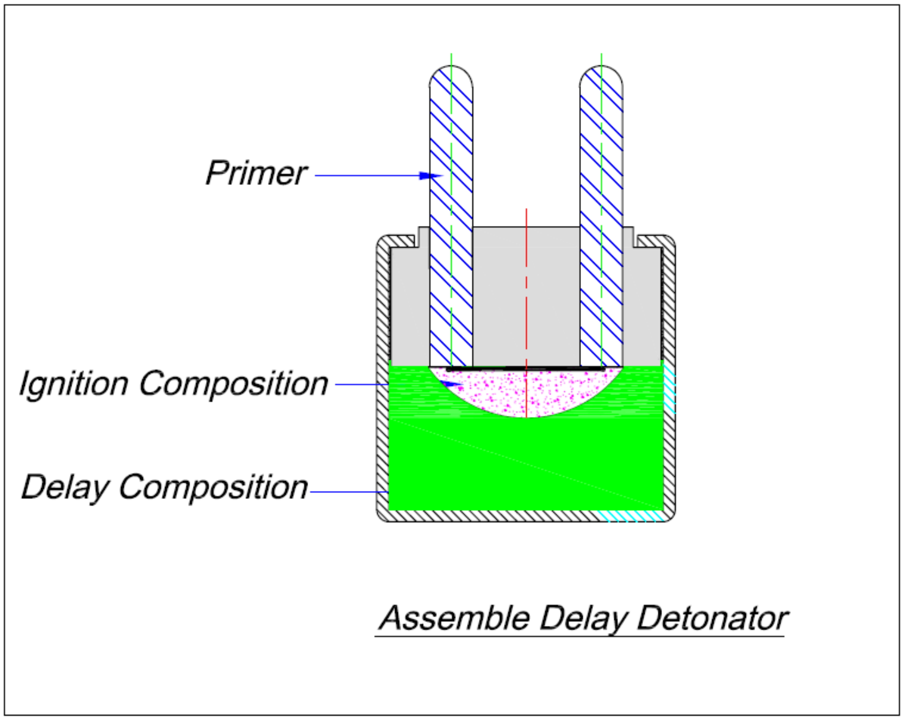

Fig: 2.7 Assembled delay detonator initial design.

2.8 Testing of delay detonators:

The finished/assembled products were tested by using approved method/equipments by following all safety Standard Operating Procedures to ensure safety and performance. For performance testing, different batches with same quantity of delay detonators were prepared and tested.

Procedure:

All the safety precautions were observed for handling of explosives and pyrotechnics compositions before and during testing of delay detonators.

The procedure comprises:

- Place test fixture at the firing site at normal environmental conditions.
- Fit test sample (Delay detonator) in the fixture with its adapter and hand tight it.

- Connect firing lead with delay detonator and check electrical continuity at firing lead end.
- Connect Oscilloscope with firing module and test fixture. Set time recording system in recording mode.
- Connect firing lead with firing module and charge it to 70±2 Volt DC.
- Press firing button to fire the delay detonator and record the delay time.

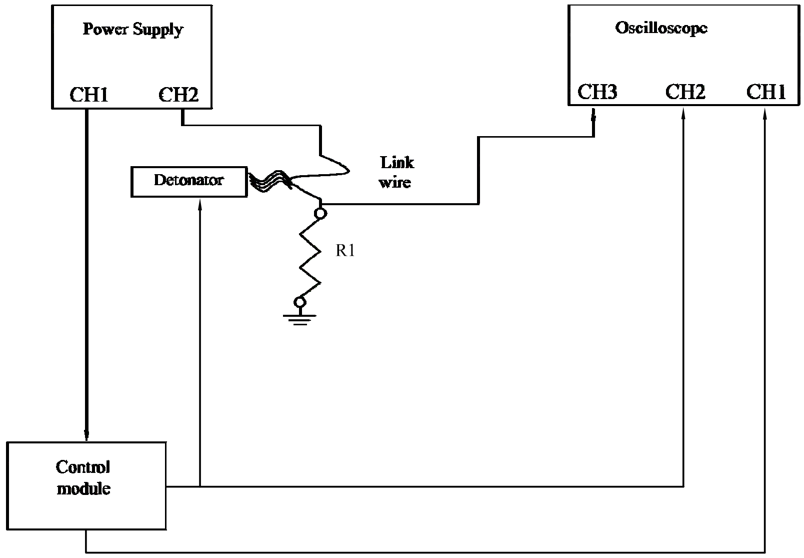

Fig: 2.8 Schematic Diagrams for Testing of Delay Detonator

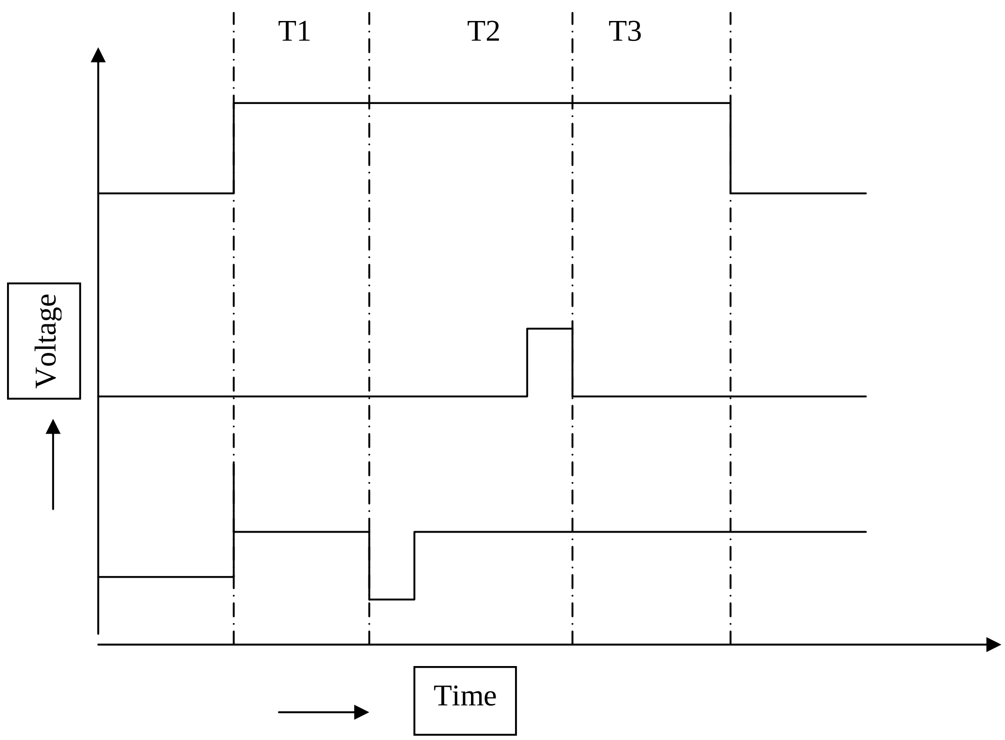

Fig: 2.9 Delay time versus volts

In fig: 2.9

T1= Time elapsed between power on and trigger signal.

T2= Delay between the trigger and fire pulse.

T3= Delay time between delay detonator firing and terminal wire break.

2.9 Equipments/Apparatus used in testing of delay detonators.

2.9.1 Resistance measuring tester:

It is an instrument which is used to measure the resistance of different types of electrical detonators/squibs etc.It has accuracy of ± 0.1% .The resistance of delay detonators which is in the range between 2.8 and 3.8 Ohm is measured using this Ohm meter. It contains a 9 volt battery.

Fig: 2.10 Resistance measuring tester:

2.9.2 Oscilloscope:

Oscilloscope of following specification was used for measurement of delay time of delay detonator.

Time scale = from nanosecond to Second (n.sec to sec)

Maximum frequency = 200MHz

The primary function is to provide a graph of signal voltage against time. This is useful for measuring propagation delay of signal rise and fall time. This method has proven to be very reliable, simple and accurate one [13]. The delay time of delay detonator based on the ability of oscilloscope to respond to initial current supplied from power supply/capacitor discharge and its response to breaking of wire installed at the end of delay detonator. The initial input of power supply/capacitor discharge and breaking of wire installed at the end of detonator transmit into electric signal that is recorded by oscilloscope.

Oscilloscope setting: Channel 01 of oscilloscope was connected with the trigger input which receives trigger command. Channel 01 was set on 5v/div and 01 sec of time scale.

Channel 02 of oscilloscope was used for checking of fire pulse. It was also set on 5v/div and 01 sec time scale.

Channel 03 was used to check the breakage of wire (installed at the end of delay detonator) and was set on 5v/div and 01 sec of time scale.

Fig: 2.11 Oscilloscopes

2.9.3 Power supply:

Power supply is used to provide required energy for initiation of delay detonator.

Channel 1 of power supply is set on 10 V and 01 A, this channel provides the power to control module.

Channel 2 of power supply is set on +5V and 100mA.

Fig: 2.12 Power supply

2.9.4 Delay testing holder

This is used for holding of delay detonators during testing. The two pins of delay detonators are inserted in the holder for proper holding detonators.

2.9.5 Delay testing fixture:

A fixture was design to hold the delay detonator and delay holder. This helped to properly hold the delay detonator and to prevent the igniter to eject from the delay detonator.

CHAPTER 03 RESULTS AND DISCUSSIONS:

3.1 Introduction:

These experiments can be divided into the following phases:

- Finalization of ignition compositions for igniter.
- Finalization of delay compositions for delay time
- Development of delay detonator within required range.
- Effect of different parameters on burning time.

The first two phases were investigated and finalized with different experiments. After finalization of ignition and delay composition, the delay detonator was developed within range of delay time. In fourth phase, the following parameters that affect burning rate were investigated with detailed experiments:

- Confinement
- Obturation
- Venting

The following sections will give the results of such experiments including discussions.

3.2 Finalization of ignition composition:

Step-1: Preparation of ignition composition:

The following fuels and oxidizers were used in preparation of ignition composition according to procedure given in section 2.3.

Table 3.1 Ignition composition#1

S #	Materials	Percentage
1	**$BaCrO_4$**	50±3%
2	Bi_2O_3	15±0.5%
3	KClO4	20±0.5%
4	Mg powder	5±0.1%
5	Boron	10±0.2%

Step-2: Testing of ignition composition:

The ignition composition was pasted and tested by 70VDC through discharged capacitor and having 01 Ampere current.

Table 3.2 Test results of ignition composition#1

S#	Resistance(Ohm) (2.8 to 3.8)	Wt of primer composition	Results	Remarks
1	2.80	13.0 mg	Not fired	Failed
2	3.15	15.0mg	Not fired	Failed
3	3.59	14.5mg	Not fired	Failed
4	3.25	14.4mg	Not fired	Failed
5	3.35	15.0mg	Not fired	Failed

3.3 Discussion of result:

These results show that primers pasted with ignition composition is not sensitive to be initiated by 70VDC through discharged capacitor and 01 Ampere current.

In order to overcome this problem, 50% of lead styphnate was added with the ignition composition mentioned in table 3.1.

Step-3: Testing of ignition composition:

The ignition composition containing 50% Lead styphanate was pasted and tested by 70VDC through discharged capacitor and having 01 Ampere current.

Table 3.3 Test results of ignition composition#2

S#	Resistance(Ohm) (2.8 to 3.8	Wt of primer composition	Results	Remarks
1	2.90	15.0 mg	Fired	Passed
2	3.20	12.0mg	Fired	Passed
3	2.95	14.5mg	Fired	Passed
4	3.00	14.4mg	Fired	Passed
5	3.25	13.0mg	Fired	Passed

The above composition when subjected to functional tests to initiate the delay composition to get required delay time gave undesirable results as mentioned in section 3.7; therefore, electrically sensitive composition is required to be developed to overcome the problems.

3.4: Preparation of electrically sensitive primer composition:

After experimenting with different ratios of Potassium chlorate ($KClO_3$, Lead thiocyanate Pb $(SCN)_2$, the following ratio of these fuel and oxidizer was finalized as an ignition composition.

Table 3.4 Ignition composition#03

S #	Description	Percentage
1	Potassium chlorate(KClO3)	70%
2	Lead thiosinate Pb $(SCN)_2$	30%

Specifications/Characteristics of chemicals used in ignition composition:

Potassium chlorate ($KClO_3$):

Melting point = 356^0C

Ignition: Easily ignitable

Application: Commonly used in ignition compositions

Availability: Easily available

Hygroscophicity: Low hygroscopic

Heat of reaction = -10.6 K cal/mole (Exothermic)

Lead thiocyanate Pb (SCN) 2

Melting point = 190^0C (Decomposes)

Boiling Point = 190^0C (Decomposes)

Solubility in water = < 0.05%

Specific gravity 3.82

Step-4Testing of igniter/primer composition:

Table 3.5 Test results of ignition composition#03

S#	Resistance(Ohm) (2.8 to 3.8	Wt of primer composition	Results	Remarks
1	2.90	15.0 mg	Fired	Passed
2	3.10	14.0mg	Fired	Passed
3	3.50	14.5mg	Fired	Passed
4	3.20	14.0mg	Fired	Passed
5	3.00	15.0mg	Fired	Passed

3.5 Finalization of delay compositions:

Step-1: Preparation of delay composition:

Different ratios of Silicon, PbO(Red) and Pb_3O_4 were used to finalize delay composition (Table 3.6)which can be easily initiated by the ignition composition as mentioned in Table 3.3.The following ratio of Fuels and oxidizers was used in the preparation of delay composition and was prepared according to procedure in Para 2.3. Detonator containing Lead Azide and Tetryl was used as a bursting charge.

Table 3.6 Delay composition#01 used in this research work

S#	Description	Percentage
1	Silicon	16%
2	PbO	10%
3	Pb_3O_4	74%

Specifications/Characteristics of chemicals used in delay composition:

Silicon (Si):

Melting point = 1410^0C

Availability: Easily available and Cheap as compared to boron (B)

Applciation: Used both as in ignition and delay compositions:

Lead monoxide (PbO):

Melting point = 888^0C

Availability = commonly used oxidizer for delay composition and also cheap

Water solubility = 0.0017 gram/100ml

Pb_3O_4:

Melting point = 500^0C

Water solubility = Insoluble in water

Availability = commonly used oxidizer for delay composition and also cheap

Heat of formation = -171.7 Kcal/mol

Step-2Testing of delay detonator:

The delay detonators were tested using procedure mentioned in section 2.8 by using delay body Aluminum cup of specification AA-2024 Fig: 03(a) 70VDC through discharged capacitor and 01 Ampere current, by using ignition composition mentioned in Table 3.3 and delay composition mentioned in Table 3.6.

Table 3.7: Test result of delay detonator using delay body (Fig: 3)

S#	Resistance(Ohm) (2.8 to 3.8	Wt of delay composition in mg	Pressure applied	Results	Delay time in m.sec
1	2.95	500	75 Mpa	Fired	2.00
2	3.00	495	75 Mpa	Fired	0.5
3	3.15	500	75 Mpa	Fired	3.0
4	2.90	490	75 Mpa	Fired	1.5
5	3.60	504	75 Mpa	Fired	0.2

3.6 Discussion of results:

Although these compositions have been fired successfully but the delay time provided is very very short.

There are three possible reasons for failure. The failure is either due to one, two or all of the following;

1. The first reason is that it may be due to the formation of shock waves due to Lead Styphnate which pushed through the delay composition and detonated the secondary charge before the burning of delay composition.
2. The second reason is that it may due to the closed aluminum body with no obturation/vent; the gasses accumulated inside the body which developed high pressure and temperature and caused deflagration to detonation.
3. The third reason for the failure is due to the conduction of heat through the aluminum body from the initiator which caused premature detonation before burning of delay composition.
4. Fourth reason could be due to rupture of the aluminum body for not withstanding the pressure development.

3.7 Remedial actions taken on these problems:

3.7.1 Taking into consideration problem No 01

3.7.1.1 Preparation of electrically sensitive ignition composition containing no primary explosive:

The electrically initiated ignition composition containing the fuel and oxidizer (Table: 3.5) was prepared and tested for functionality. Both fuel and oxidizer have low melting point and are electrically sensitive.

3.7.2 Discussion of results: This ignition composition was pasted on igniters and tested by 70VDC through discharged capacitor and 01 Ampere current. The ignition composition successfully initiated because both fuel and oxidizer have very low melting point and are electrically sensitive. These results show that this composition is sensitive and can be easily initiated by 70VDC through discharged capacitor and having 01 Ampere current, the primer successfully fired. The potassium chlorate has very low melting point and can easily be initiated with fuel.

Table: 3.8 Test results of delay detonator (initial design) by using delay composition (Table: 3.6) and ignition composition (Table: 3.4)

S#	Resistance (Ohm) (2.8 to 3.8	Wt of delay compositio n in mg	Pressure applied	Results	Delay time in m.sec
1	3.20	600	75 Mpa	Fired	38
2	3.12	595	75 Mpa	Fired	62
3	3.17	550	75 Mpa	Fired	40
4	2.98	560	75 Mpa	Fired	47
5	3.20	550	75 Mpa	Fired	35

3.8 Discussion of results: All the delay detonators successfully fired and gave delay time, although the delay time is not in required range but the delay composition burned properly. It means that the problem which occurred in the initial testing in which Lead styphnate was used in ignition composition, the shock waves of lead styphnate which penetrated in the delay composition and caused detonation of end charge before burning of delay composition. After the tests, it was observed that body of delay detonator was found in small pieces from which it was not clear whether that the link wire which was installed at the end of delay body for measuring of the delay time broke at end of burning of delay composition or during the burning of delay composition when the body broke into pieces from side because the thickness of the delay body was only 0.3 mm(soft aluminum).Therefore, it was decided to redesign the delay body to ensure that the link wire to break when the bursting charge is exploded.

3.9 Redesigning of delay body and development of delay detonator: The body of delay detonator was redesigned to withstand the pressure and heat of delay composition as shown in Fig 02, 03 and 04: using material to specification Aluminum AA-2024.

3.10 Testing of delay detonators using the redesigned delay body (effect of confinement) on burning time of delay composition.

The internal cavity of delay body was completely filled with out leaving any internal space(confinement)[14,15,16]

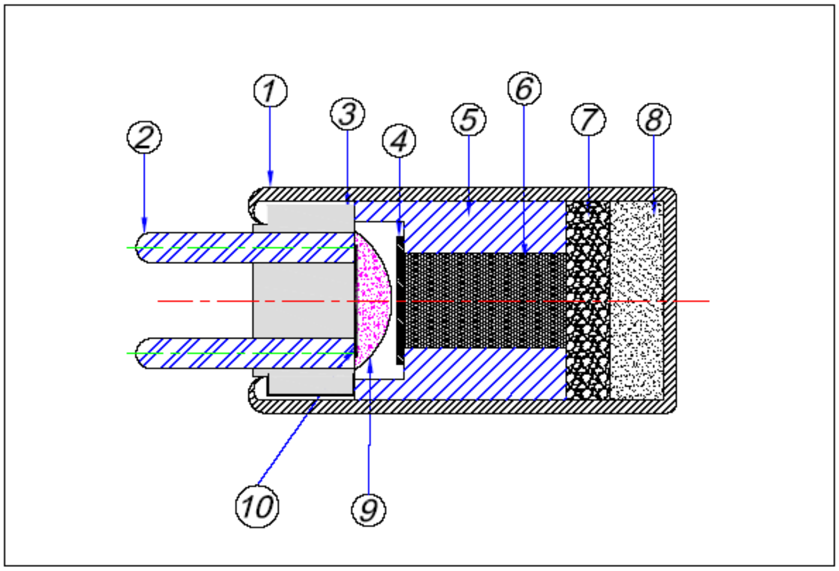

Fig: 3.1 Redesigned confined delay detonator

Description of delay detonator: This delay detonator consists of

1. Delay cup
2. Contact wires for current flow
3. Insulating material
4. Paper disc
5. Delay element
6. Delay composition
7. Lead Azide
8. Tetryl
9. Ignition composition
10. Hot bridge wire

After filling and assembly, the delay detonators were subjected to function testing for delay time by using finalized ignition composition. The results shown in Table: 3.9 and Table: 3.10.

Table: 3.9 Test results of redesigned delay detonator (Confined effect)

S#	**Resistance(Ohm) (2.8 to 3.8**	**Wt of delay composition in mg**	**Pressure applied**	**Results**	**Delay time in m.sec**
1	2.8	100	75	Fired	5.0
2	3.2	100	75	Fired	26
3	3.8	100	75	Fired	6.0
4	3.3	100	75	Fired	19
5	3.0	100	75	Fired	16

Table: 3.10 Test results of redesigned delay detonator (Confined effect)

S#	**Resistance(Ohm) (2.8 to 3.8**	**Wt of delay composition in mg**	**Pressure applied in MPa**	**Results**	**Delay time in m.sec**
1	2.95	100	75	Fired	17
2	3.25	100	75	Fired	6.0
3	3.44	100	75	Fired	14
4	3.21	100	75	Fired	10
5	3.80	100	75	Fired	12

3.10.1 Discussion of results: The results shown in table: 3.9 and table: 3.10 are not consistent. A detailed study was carried out in order to identify the reasons of inconsistent results. During study it was found that the primer did not withstand the high pressure of burning delay compositions and was ejected when primer is fired. When the primers eject form the delay body, the gases escape from that space and increase the burning rate. Those primers which are not properly crimped easily eject and increase burning rate as compared to those primers which are properly crimped (decrease burning rate) due to confined effect. After testing it was also found that the terminal wire (which was used to measure the time when the bursting charge detonated) broke from side instead of place at which it makes contact with terminal charge. So these are the possible reasons for inconsistency in burning time.

3.10.2 Remedial action taken: A testing tool was designed which properly held the delay detonator and did not allow the primer to eject form the delay body after burning of pyrotechnic composition. The terminal wire was also changed and a comparatively strong wire was used which broke after bursting of terminal charge.

3.10.3Testing of delay detonator in a special designed testing tool: The delay detonators were tested in a special design testing tool. These testing tools properly hold the delay detonator and did not allow the primer to eject from the rear due to improper crimping.

Table: 3.11 Test results of redesigned delay detonator (Confined effect) with new testing tool

S#	Resistance(Ohm) (2.8 to 3.8	Wt of delay composition in mg	Pressure applied in MPa	Results	Delay time in m.sec
1	2.9	100	75	Fired	8
2	3.0	100	75	Fired	11
3	2.8	100	75	Fired	12
4	3.2	100	75	Fired	11
5	3.5	100	75	Fired	8

3.11.1 Discussion of results: The results show that the delay composition was properly initiated and burnt smoothly. The quantity of delay composition is small because the thickness of redesigned delay body has been increased to withstand the pressure and heat. After the test it was found that the delay bodies withstood the pressure and heat and that link wire broke when the bursting charge exploded.

3.12 Determination the effect of obturation of delay body on burning time of delay composition:

The internal cavity of the delay detonator was not completely filled and a small space was left open (Obturation) for accumulation of gases [17]

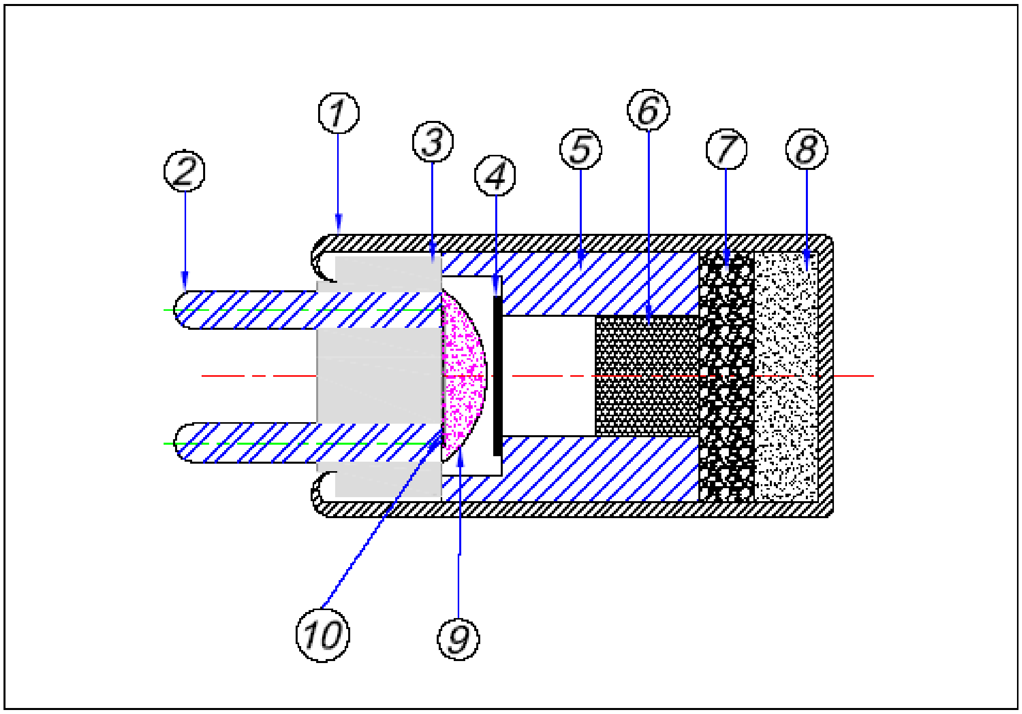

Fig: 3.2 Redesigned obturated delay detonator

After filling and assembly, the delay detonators were subjected to function testing for delay time with finalized ignition composition. The results shown in Table: 3.12

Table: 3.12.Test results of redesigned delay detonator (Obturation effect)

S#	Resistance(Ohm) (2.8 to 3.8	Wt of delay composition in mg	Pressure applied	Results	Delay time in m.sec
1	3.83	100	75	Fired	25
2	3.46	100	75	Fired	20
3	3.71	100	75	Fired	16
4	3.01	100	75	Fired	18
5	2.98	100	75	Fired	17

3.13 Determination the effect of venting of delay body on burning time of delay composition:

A hole of one mm diameter was drilled in the delay body in order to provide escape for the gasses produces during combustion.

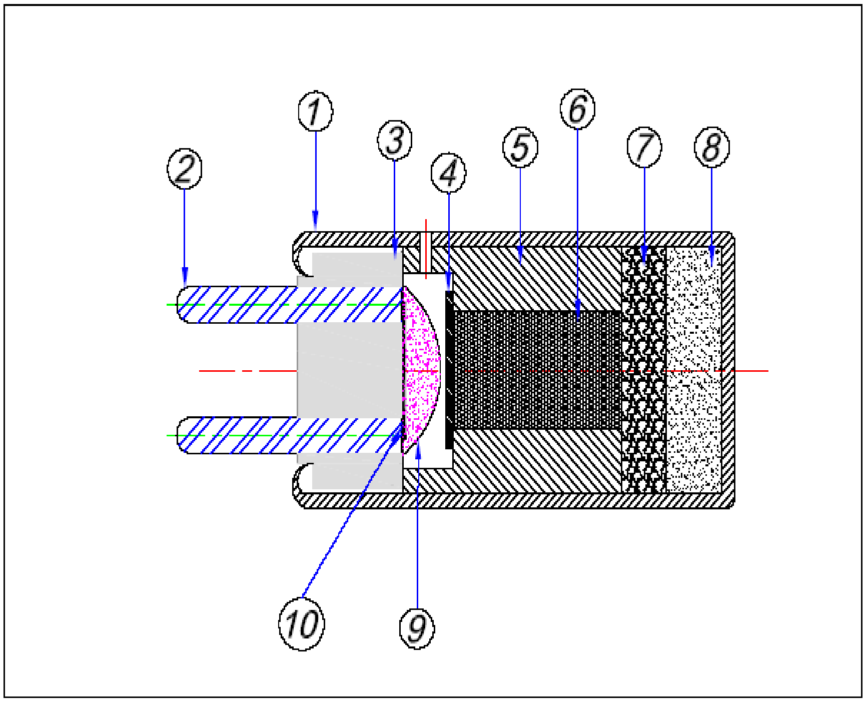

Fig 3.3 Redesigned venting delay detonator

After filling and assembly the delay detonators were subjected to function testing for delay time with finalized ignition composition and delay compositions. The results shown in Table: 3.13

Table: 3.13 Test results of redesigned delay detonator (Venting effect)

S#	Resistance(Ohm) (2.8 to 3.8)	Wt of delay composition in mg	Pressure applied in MPa	Results	Delay time in m.sec
1	3.67	100	75	Fired	78
2	3.71	100	75	Fired	78
3	2.81	100	75	Fired	84
4	3.24	100	75	Fired	80
5	3.06	100	75	Fired	82

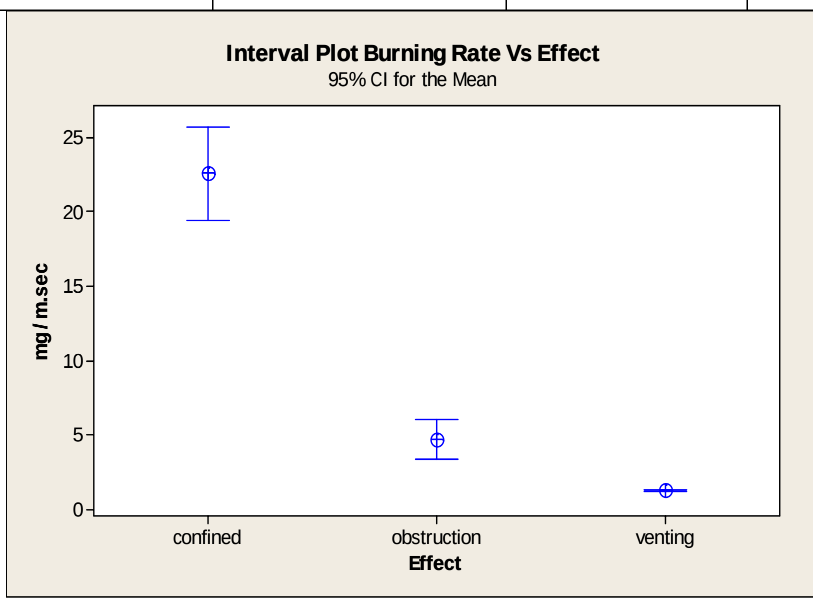

Fig: 3.4 burning rate vs. different affects

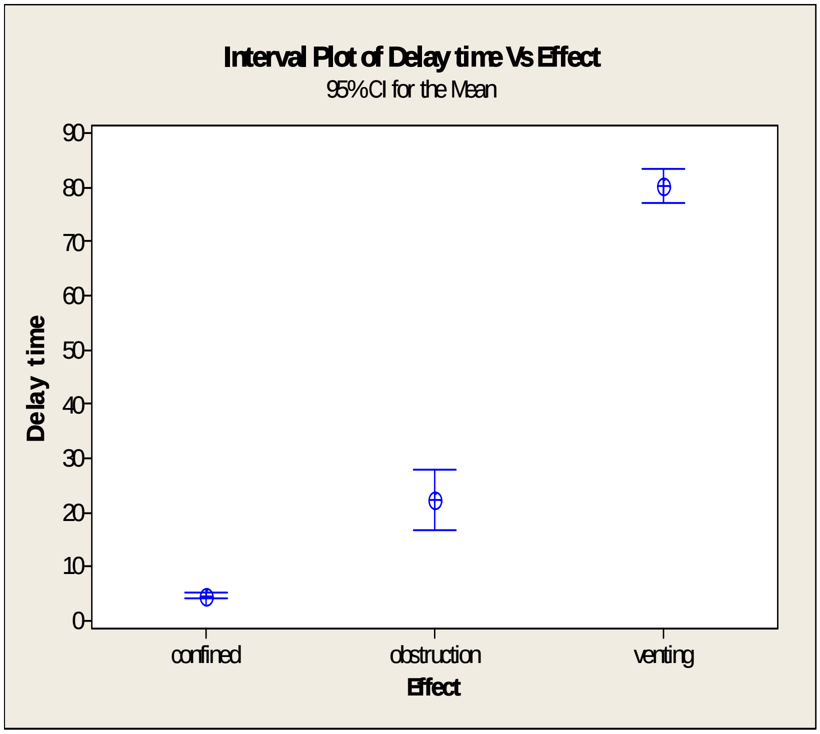

Fig: 3.5 Delay time vs. different affects

3.14 Discussion of results: During first experiment, the delay body was completely filled with delay composition and delay body was completely confined and no internal space was left for accumulation of gasses. After firing the pressured inside the delay body increased and thus helped to increase the burning rate [19] (Avg time 10 m.sec) as all the components are theoretically gasless, that is to say, at the temperature of reaction all products of the reaction have theoretically low or negligible gas pressure. However, traces of adsorbed moisture, chemically-bound water, hydrogen or carbon dioxide, produce gas the pyrotechnic delay composition composition. Impurity in the delay composition is also responsible for the production of gas during combustion.

During second experiment an internal empty space (obturation) was left for accumulation of gasses inside the delay body. This obturation increased the burning time (9.2 m.sec Avg).The obturation provided a space for some gases while excess gases developed a pressure inside the delay body and thus slightly increased the burning time.

During third experiment ,a small hole of 01mm was drilled in the delay body and the delay body was tested; all gasses produced during burning emitted from the 01mm diameter hole and the maximum burning time was measured i.e. (80.4m.secAvg) while keeping all the other parameters constant.

Quantity of delay composition (100 mg) was kept constant during (Confinment, Obturation and Venting) and length of delay body was varied to adjust the delay composition.

Chapter 4: Conclusions and suggestion for future work

4.1 Conclusion:

During the present work, a detailed study was carried out on pyrotechnic delay compositions together with identification of different parameters affecting burning rate of pyrotechnic compositions in electrically initiated pyrotechnic delay detonators. During this study it was found that ignition composition containing lead styphnate (50%) caused premature detonation of bursting charge so firstly a detailed investigation was carried out on ignition composition which was required to be initiated through 10 VDC and 01A in 10 microseconds by discharge capacitor and required to initiate pyrotechnic delay composition. Electrically sensitive ignition composition was developed which was easily initiated by available energy. After finalization of ignition composition, a pyrotechnics delay composition was then developed and tested by ignition composition. The delay composition was initiated successfully through ignition composition. A study was also carried out to check the effect of confinement, obturation and venting of delay body on delay time of pyrotechnics delay composition. During this study it was found that confinement, Obturation and Venting of delay body plays an important role in delay time/burning time of pyrotechnic compositions. Due to confinement, pressure inside delay body developed and thus helps in increasing burning rate of the delay compositions (Quantity 100 mg).The average burning time in case of confined condition obtained was 10 m.sec.When the delay composition Qty: 100 mg) was tested in Obturated condition the burning time slightly increased due to small empty volume where the product gases of ignition and delay compositions expanded. The average time obtained in obturation condition was 19.2 m.sec.During last experiments the delay composition(Qty:100 mg) was tested in vented condition where the product gases of ignition and delay compositions vented through 0ne mm hole and thus increased burning time. The average burning time obtained during venting condition was 80.4 m.sec.

During this study, it was also found that delay body thickness also plays an important role in burning time. Use of thin body/cup of aluminum (Thickness 0.3 mm) caused rupturing of delay body and it was difficult to measure the delay time accurately. When the delay body was redesigned and thickness of delay body was increased it withstood the pressure and heat of ignition and delay compositions and thus provided better results. These parameters(Confinment,Obturation,Venting and thickness of delay body play an important while designing and developing electric pyrotechnic delay detonator for special application.

4.2 Suggestion for future work

The present project involves the study pertaining to address time inconsistency in electric pyrotechnic delay detonators in consonance with requisite delay time. A detailed study and practical work was conducted for finalization of ignition and delay compostions.The effect of (Confinment, Obturation and Venting) of delay body on burning rate were also studied in detail while developing pyrotechnic delay detonators.

It is suggested that effects of other parameters such as load pressure, particle size, ambient temperature and shelf-life on electrical pyrotechnics delay detonators may be determined. As these parameters affect delay time and cause inconsistent results in pyrotechnic delay detonators especially in short delay detonators used in sophisticated missiles, therefore, it is important to undertake such studies.

It is also suggested that kinetic study of the pyrotechnic delay composition may be carried out in order to determine the behavior of delay composition with change in temperature.

The primers of the delay detonator eject some time which create space for product gases to vent and thus some time produce inconstant results. So some study is required to be undertaken to redesign the primer to withstand the pressure of the gases of delay and ignition compositions on order to produce consistent delay time.

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6. Accomplishment: A research paper by the author related the last part of this research work having Tilte**" A study of effect of confinement, Obtuaration and venting on burning rate of modified pyrotechnics delay composition in delay detonator"** has been accepted in 2011 International Autumn Seminar on Propellents, Explosives and Pyrotechnic to be held in Nanjing, Jiangsu province China from 20-23rd September, 2011.Full paper is attached herewith.

A study of effect of confinement, Obtuaration and venting on burning rate of modified pyrotechnics delay composition in delay detonator

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Abstract: A study was carried out to determine effect of confinement, obturation and venting on burning rate of modified pyrotechnics delay composition (Silicon 14%, PbO 11% and Pb_2O_3 75%) in electrically initiated delay detonator. A cylindrical aluminum body of specification AA-2024 having inner bore to contain delay composition was used as a delay body and Nichrome wire 80/20 was used as hot bridge wire. This delay detonator consists of electrical primer, detonator body, delay composition and bursting charge. In this study effect of Confinement, Obturation and Venting of delay body on burning rate of modified delay composition was determined and compared. All other parameters such as consolidation pressure and quantity of delay composition were kept constant.

Key words: Delay time, Obturation, Venting, Confinement, Delay detonator.

1. Introduction: Delay detonator is an explosive device used in various warheads. Delay detonators provide required delay before certain effect is produced.Deley detonators are classified according to means of initiation like bridge wire or hot wire delay detonators. In bridge wire delay detonator required current is passed through the resistive wire which heats up and provides required energy for explosives/ pyrotechnic composition to initiate. Delay time is one of the most important performance parameter of warheads and weapons. It is critical when accurate delay time is required for some affect to occur in weapons i.e. to perform some chemical/ mechanical operation after a certain delay time.Confinment of delay body means when the primer/igniter is completely sealed and the gasses are accumulated inside the delay body.Obturation of delay body means when an internal volume is left empty for accumulation of products gases produced by burning of ignition and delay compositions. Venting of delay body means when a delay body has opening through which the gases produced during burning of delay composition. Venting is provided in order to avoid pressure build up inside the delay body [1].Like other parameters the delay time is greatly effected by conferment, obturation and venting of delay body, when the delay composition is initiated in confirmed body the time considerably reduced as compared to unconfirmed delay composition. The maintenance of pressure behind the burning front in condition of confinement and vented is important to increase the burning rate. The initial pressure which developed in confined condition is due to the gas evolved due the burning of ignition composition of igniter [2, 3].By increasing the percentage of silicon in the delay composition the reaction is mainly a solid/solid nature. By decreasing the percentage of silicon in delay composition the solid/solid reaction will not predominate [4].The more is the gassy composition the more is the composition sensitive to pressure [5].In condition of confinement the temperature of reactants can rise when heat is not lose to the surrounding then a sharp rise the reaction rate occurs[6].In case of confined condition of lose gassing pyrotechnic composition when provided with a minimal vent volume then the burning time of delay composition speed up[7].The primer or igniter of pyrotechnic device is also very important it is used to initiate the main delay composition, the composition of primer or igniter used to enhance the probability of successful ignition of pyrotechnic composition [8].Pyrotechnic delay compositions should be readily ignitable and required to be safe from explosive and health view point and should also be non hygroscopic and stable on storage[9].

In this research work, results of burning of modified pyrotechnic delay composition Si/PbO/Pb3O4 in confined condition in electrically initiated delay detonators have been determined and compared with results of same composition in condition of obturation and venting.

2. Experimental work:

2.1 Material used: Silicon (Si) of Aldrich was used as a fuel and Lead oxide (PbO) and lead trioxide (Pb_2O_3) of Fluka and Aldrich respectfully were used as oxidizers for delay composition. Both the fuel and oxidizers used were of Analytical reagent grade. Potassium chlorate $KClO_3$ of Fluka and Lead thiocyanate Pb $(SCN)_2$ Aldrich was used as oxidizer and fuel respectively for ignition composition. Like delay composition the fuel and oxidizer used for ignition composition were also of Analytical reagent Grade The particle size of fuel and oxidizers was 212 micro meter.

2.2 Preparation of individual chemicals for ignition and delay composition: The individual chemical i.e. fuels; oxidizers and additives were dried at 105^0C and grinded in mortar and pestol manually. Letter the individual chemicals were passed through 325 mesh sieve machine having particle size of 47 micro meters.

2.3 Preparation of composition/mixing: The individual grinded chemicals were dried and weighted as desired and mixed to gather in three dimensional automatic mixing machines for three hours in order to get homogenous delay composition. The mixing is the key process; It was done thoroughly to attain homogeneity.

2.4 Granulation: The mixed compositions were transferred into granules. The main purpose of granulation was to coat the composition with binder and avoid the separation of fuels and oxidizers due difference in density. The granulation was done by adding of 0.20% Nitrocellulose the binder helped in formulation of granules. Ethyl acetate was used as solvent. . As it was a small scale experiment the granulation was done by wetting the composition in NC lacquer. The composition was then passed through sieve (65 meshes) and grains of 212 micro meter size were obtained. The grains were then dried at 105^0C for one hour.

2.5 Loading: The granulated composition was then loaded into the delay detonator bodies. The compositions were then pressed in body at 75MPa to get the desired consolidation. The loading was done in two small increments in order to get consistence delay time. Special designed tools and dies were used during the filling and pressing process.

2.6 Preparation of electrically sensitive primer composition:

An electrically sensitive ignition composition consisting of Potassium chlorate $KClO_3$ (45%) and Lead thiocyanate $Pb(SCN)_2$(55%).The individuals chemicals were grinded and dried at 105^0C for one hour. The composition was then mixed in three dimensional automatic mixing machines for three hours in order to get homogenous composition. A past of ignition composition was then prepared in Nitrocellose by using ethyl acetate as solvent. The ignition composition was then pasted on igniters and dried for two hour at 105^0C.

3. Measurement:

3.1 Resistance measuring tester:

It is an instrument which is used to measure the resistance of different types of electrical detonators/squibs etc.The resistance of delay detonators which is in the range between 2.8 and 3.8 Ohm is measured using this Ohm meter. It contains a 9 volt battery.

3.2 Oscilloscope:

Oscilloscope was use for measurement of delay time of delay detonator. This method has proven to be very reliable, simple and accurate one. The delay time of delay detonator based on the ability of oscilloscope to respond to initial current supplied from power supply/capacitor discharge and its response to breaking of wire installed at the end of delay detonator. The initial input of power supply/capacitor discharge and breaking of wire installed at the end of detonator transmit into electric signal that is recorded by oscilloscope.

3.3 Power supply:

Power supply is used to provide required energy for initiation of delay detonator.

Channel 1 of power supply is set on 12 V and 01 A, this channel provides the power to control module.

Channel 2 of power supply is set on +5V and 100mA.

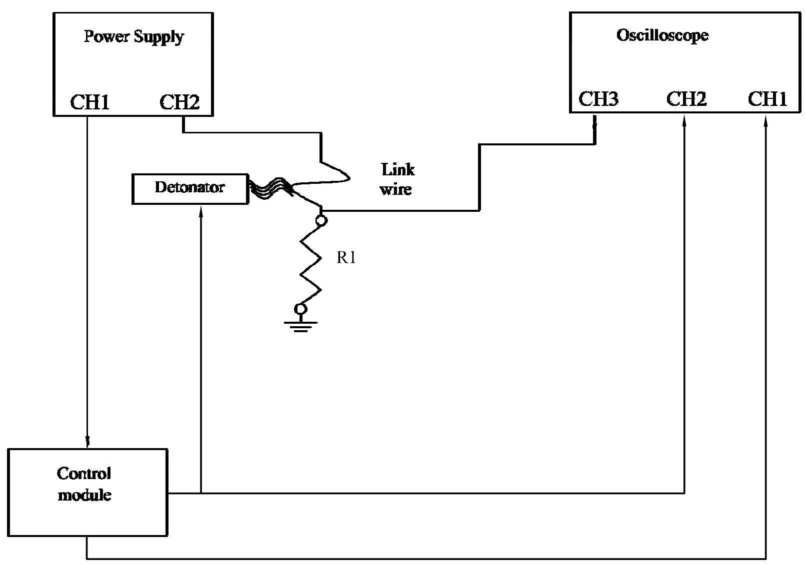

Fig: 1 Schematic Diagram for Testing of Delay Detonator

Experiment # 01: During first experiment a small hole of one mile meter was drilled from on side of delay body above delay composition and below igniter in order to vent the gases produced due burning of delay and ignition composition as shown in fig:01.The one mm hole was sealed by a tin disc.

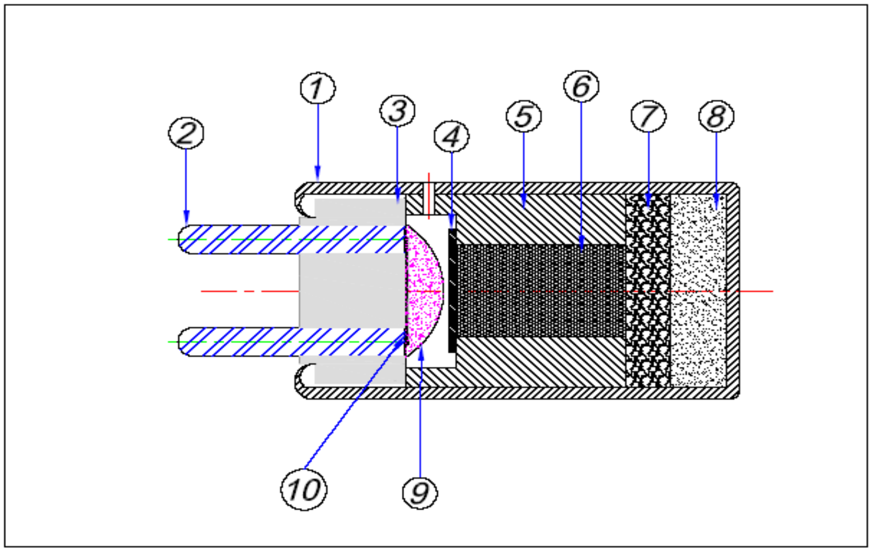

Fig: 02 vented delay detonator

Experiment # 02: During 2nd experiment a small volume was left empty for accumulation of gases produced during burning of delay and ignition compositions as shown in fig: 02

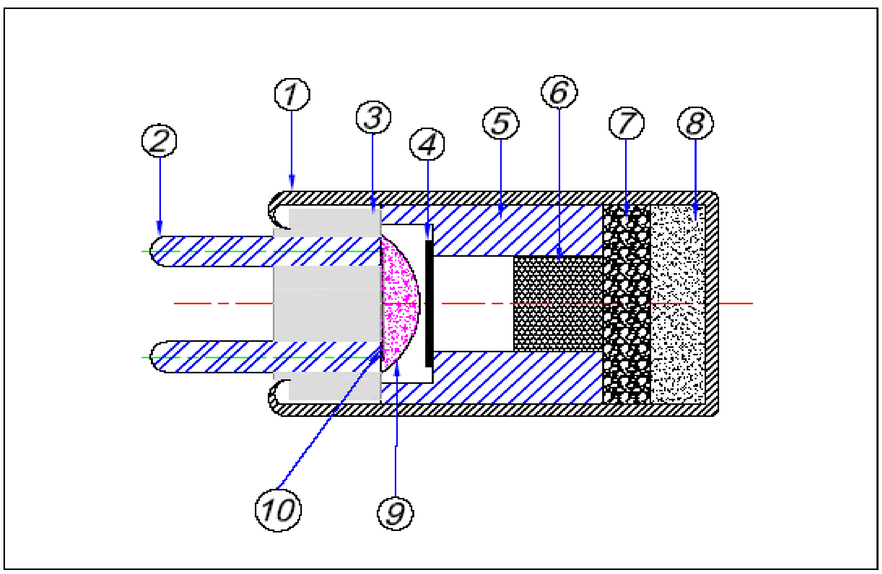

Fig: 03 Obturated delay detonator

Experiment # 03: In third the delay body was completely confined and no space was left for gases to escape from the delay body as shown in fig: 04

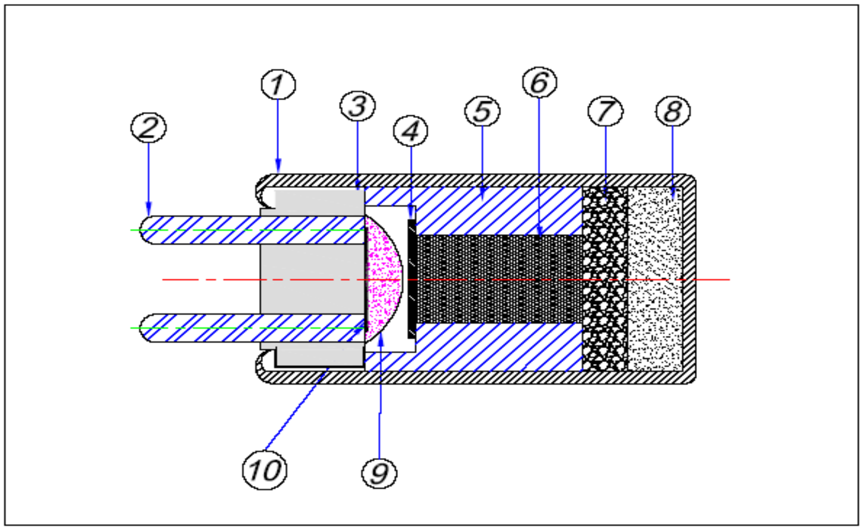

Fig: 04 Confined delay detonator

4.2 Results and discussion:

4.1 Different effect of on burning time of delay composition:

Table: 01 Venting effect on delay time of pyrotechnic composition

S#	Resistance(Ohm) (2.8 to 3.8)	Wt of delay composition in mg	Pressure applied in MPa	Results	Delay time in m.sec
1	3.67	100	75	Fired	78
2	3.71	100	75	Fired	78
3	2.81	100	75	Fired	84
4	3.24	100	75	Fired	80
5	3.06	100	75	Fired	82

Table: 02 Obturation effect on delay time of pyrotechnic composition

S#	Resistance(Ohm) (2.8 to 3.8	Wt of delay composition in mg	Pressure applied in Mpa	Results	Delay time in m.sec
1	3.83	100	75	Fired	25
2	3.46	100	75	Fired	20
3	3.71	100	75	Fired	16
4	3.01	100	75	Fired	18
5	2.98	100	75	Fired	17

Table: 03 Confined effect on delay time of pyrotechnic composition

S#	Resistance(Ohm) (2.8 to 3.8	Wt of delay composition in mg	Pressure applied in Mpa	Results	Delay time in m.sec
1	2.9	100	75	Fired	8
2	3.0	100	75	Fired	11
3	2.8	100	75	Fired	12
4	3.2	100	75	Fired	11
5	3.5	100	75	Fired	8

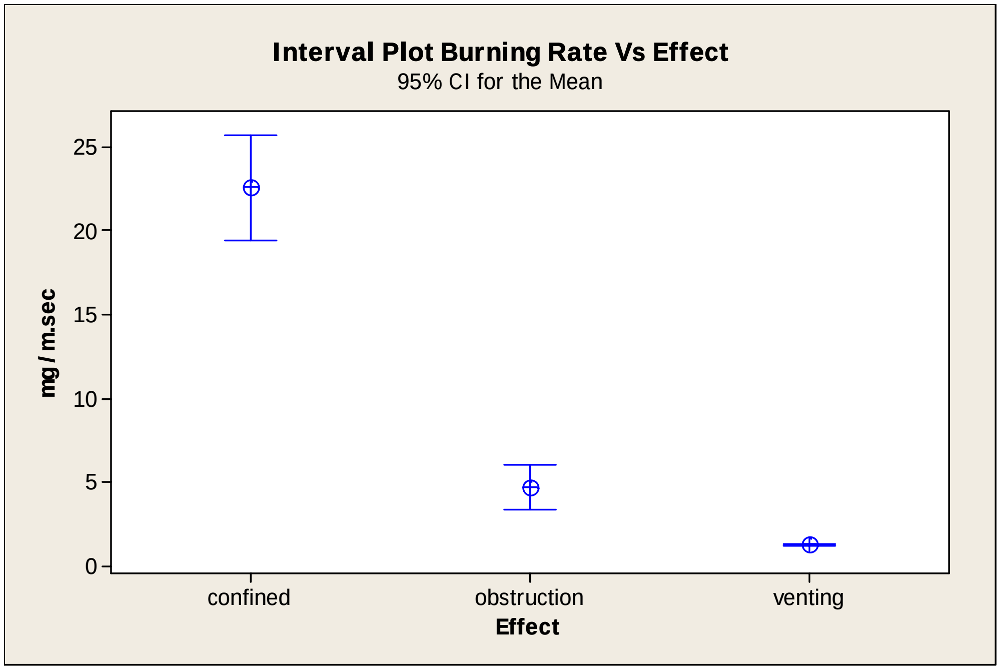

Fig: 04 Delay time vs different effects

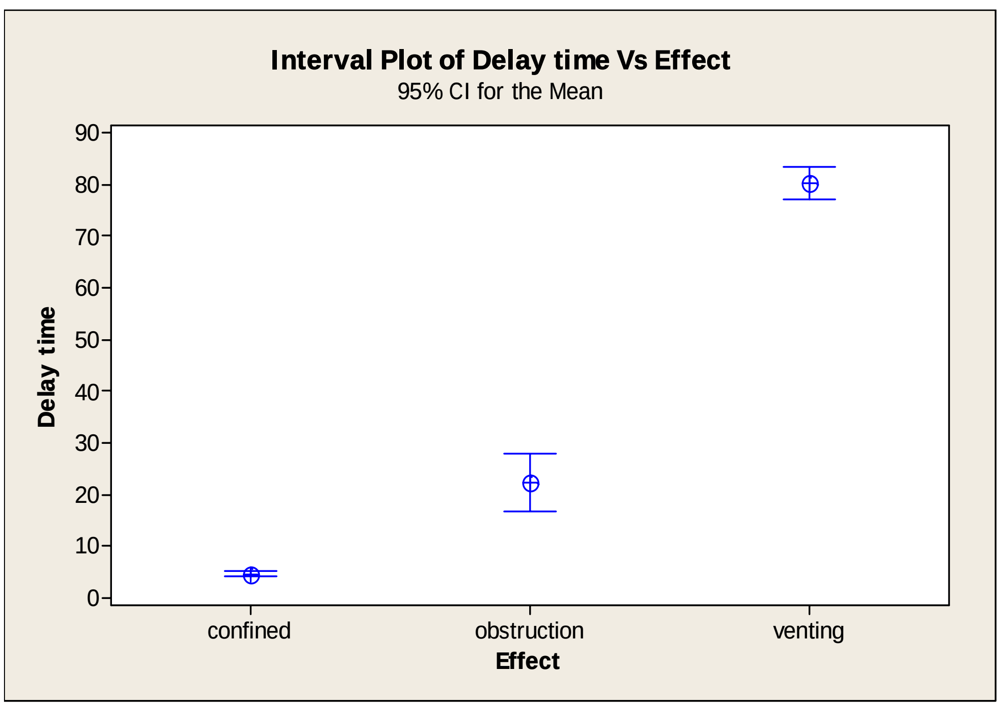

Fig: 06 Delay time vs. different effects

First of all an electrically sensitive ignition composition was developed, both potassium chlorate and lead thiocyanate has very low melting point and easily decompose at very low temperature and therefore very sensitive to electrical pulse: During first experiment the main purpose of drilling a small hole of 01mm in the delay body to vent all the gasses produced during combustion, the gasses produced during burning of ignition and delay composition vented through one mile meter hole and the maximum burning time was measured i.e. (80.4m.secAvg)as shown in Table:01.The purpose of sealing 01 mm hole in the delay body. The purpose of using the tin disc was to protect the delay composition from moisture because moisture can cause ignition failure effect burning rate and destroy the composition**.** The tin disc was weak enough and easily broke when pressure inside delay body develops.

During second experiment an internal empty space (obturation) was left for accumulation of gasses inside the delay body. This obturation slightly decreased the burning time (9.2 m.sec Avg) as shown in Table: 02.The obturation provided a space for some gases to be accumulated while excess gases developed a pressure inside the delay body and thus slightly decreased the burning time.

During third experiment the delay composition was completely confined inside the delay body no internal space was left for accumulation of gasses. The pressured inside the delay body increased and thus helped to increase the burning rate and thus further decreased the delay time (10 m.sec Avg) as shown in Table: 03 because one source of energy in the combustion zone in that radiated back from the hot products. If the combustion products are gas then the contribution is sensitive to the gas pressure, but in these experiments the delay composition used was assumed to be gasless but still gaseous products developed and thus increased the burning rate because all the gasless delay composition cannot be completely gasless reality of course lies between the two. Pyrotechnics delay composition Si = 20% and PbO = 80% have been reported to produce sufficient pressure of lead vapor to double its burning rate when confined [5]

5. Conclusion: During present work a detailed study was carried out to determine the effect of confinement, obturation and venting of delay body on burning rate of delay composition (Si, PbO and Pb2O3) in delay detonator. During this study we were abled to show that confinement, Obturation and venting of delay body greatly effect burning of pyrotechnic delay composition. Due to confinement pressure inside delay body increased and thus helped in increasing burning rate or reduces burning time for same compositions. It means pyrotechnic compositions are not totally gasless; gases are produced during combustion and may be due to impurity in the chemicals used for delay compositions.

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