New Explosive Materials and Pyrotechnic Formulations with Improved Safety and Sensitivity Properties*

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Abstract

At the Department of Energy (DOE) and Sandia National Laboratories, a major effort has existed to develop new pyrotechnic formulations and explosive materials that have improved safety and sensitivity properties during use and handling. The driving force for these development efforts has been enhanced personnel safety plus improved safety/ sensitivity properties of these materials and their applications in regards to nuclear weapon safety issues. These efforts have produced a series of pyrotechnic and explosive materials that can replace traditional primary explosives such as lead styphnate and lead azide. This development has resulted in new pyrotechnic formulations and explosive materials that are insensitive to initiation by electrostatic discharge from a human body. Other safety properties have also been improved. The electrostatic insensitive pyrotechnics are a family of titanium subhydride (TiH_x, X greater than 0.65)/potassium perchlorate ($KClO_4$) formulations. These titanium subhydride/potassium perchlorate pyrotechnics also have high temperature stability, high impact and friction insensitivity. The new explosive materials are inorganic coordination compounds based upon 5-substituted tetrazolato pentaammine cobalt (III) perchlorates. Substituents in the tetrazole ring that have proven deflagration-to-detonation (DDT) properties include the cyano (-CN), nitro (-NO_2) and chloro (-Cl) groups. Their explosive properties include human body electrostatic insensitivity and high temperature stability along with friction and impact properties similar to RDX and HMX. The properties and uses of these materials has resulted in proven, mature technologies for the Department of Energy, Department of Defense as well as commercial, private sector applications.

Introduction

Nuclear weapon applications use a wide variety of energetic materials for various functional outputs that include actuation, ignition, delay, flame and detonation. The most widely used hot wire initiated energetic materials have been primary explosives, mainly lead styphnate and lead azide. In the early 1970’s, a policy decision was made at Sandia National Laboratories not to use primary explosives because of safety issues associated with such materials. These issues

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included personnel safety in regard to the handling/processing of primaries in bulk and components, plus safety issues during the use of such materials in loaded devices. The main safety issue was the accidental initiation of these materials by electrostatic discharge (ESD) from a human body.

Two parallel paths were taken to eliminate primaries. The first was the increased use and the development of new pyrotechnics for hot wire actuation applications. The second was the development of new deflagration-to-detonation (DDT) explosives that were not classed as true primaries.

ESD Sensitivity

Sandia's efforts in the development of new energetic materials were focused on improvement of ESD safety. At Sandia, two ESD models have been used over the years to measure electrostatic sensitivity of materials. Both models attempt to simulate a human body discharge through the materials and components as a means of characterizing ESD sensitivity. The first model, the Sandia Standard Man Test, was used until recently.[1] In the early 1990's a second model was developed, called the Fisher or Severe Man Model.[2] Table 1 compares the parameters of each ESD model.

Table 1

Comparison of the Sandia Standard Man
and
Fisher or Severe Man ESD Models

Parameter	Standard	Fisher
Voltage	20 kv	25 kv
Resistance	500 ohms	360 ohms
Capacitance	600 pf	410 pf
Inductance	NA	0.6 uhenries

The Fisher or Severe Man Model was developed to simulate a hand/body discharge model for ESD testing. Discharge results in a fast rise time pulse (the hand simulation) that decays and is followed by a second pulse (the body simulation) that closely resembles the Sandia Standard Man Model. As the name implies, the Severe Man or Fisher Model is considered a more demanding test because of the rapid rise time initial pulse. Both bulk materials and components are now tested to the Fisher Model although both tests were typically used during the transition period.

If a response is obtained during as ESD test, at the standard set of conditions (Table 1), the voltage is lowered from the initial set of parameters until a go/no go response voltage is noted. Using standard statistical test formats, such as Bruceton or Langlie, a voltage is then reported as the ESD sensitivity. The voltage can be increased, if desired, during special ESD testing.

Pyrotechnic Development

The use of the pyrotechnic, titanium/potassium perchlorate ($Ti/KClO_4$), for hot wire actuation applications increased during the early 1970's as a response to the decision not to use primary explosives. $Ti/KClO_4$ is static sensitive in the bulk but there is reduced static sensitivity when loaded in a device. Also, its other sensitivity properties such as friction, impact and thermal ignition are significantly better than primary explosives (see later discussions and data).

In 1974, it was found that the pyrotechnic mixture, titanium hydride/potassium perchlorate ($TiH_2/KClO_4$) was insensitive to initiation from an equivalent human body discharge.[3] This ESD insensitivity included bulk, pressed and loaded forms of $TiH_2/KClO_4$. Subsequent ESD tests with $TiH_2/KClO_4$, using discharge voltages in excess of 40 kilovolts and no resistance in the ESD circuit have never initiated this pyrotechnic. Its upper voltage or energy for ESD ignition is not known.

There were numerous development efforts with $TiH_2/KClO_4$ for actuator and igniter designs. It functioned well, but a number of low temperature (less than $-54^oC/219^oK$) ignition failures occurred.[4] This was followed by chemical stability and compatibility test programs that measured an instability of $TiH_2/KClO_4$ at higher test temperatures which might limit its use in long lived applications.[5] The products of this instability, water and chloride ions, also indicated potential compatibility problems with header pins and bridgewires.

Because of these problems, an experimental development program between Sandia National Laboratories and Monsanto Research Corporation, Mound Laboratories, Miamisburg, Ohio was initiated to determine if removing hydrogen from the titanium lattice in titanium hydride would still provide ESD insensitivity and improve ignition reliability. Stability was also expected to improve based upon results of aging/stability studies conducted with thermally pretreated TiH_2 prior to its blending with potassium perchlorate.[5]

From this program, a series of titanium subhydrides (TiH_x) with equivalent hydrogen concentrations from 0.45 to 1.70 ($TiH_{0.45}$ to $TiH_{1.70}$) were produced. When blended with potassium perchlorate to produce pyrotechnic mixtures, it was found that these mixtures were ESD insensitive until the hydrogen concentration dropped to a nominal value of $TiH_{0.60}$.[4] Below X=0.60 for TiH_x, the ESD static sensitivity properties of the pyrotechnic mixtures approached those of $Ti/KClO_4$. Figure 1 is a typical ESD sensitivity curve verses equivalent titanium lattice hydrogen concentration (TiH_x). Note the sharp break from being ESD insensitive to ESD sensitive at a nominal $TiH_{0.60}/KClO_4$ composition.

Results of stability studies with titanium subhydride/potassium perchlorate ($TiH_x/KClO_4$, X=0.45-1.70) blends, found these mixtures to approach the stability found for $Ti/KClO_4$.[5] Figure 2 is stability data measured for various $TiH_x/KClO_4$ mixtures and compared to $Ti/KClO_4$ and $TiH_2/KClO_4$.[5] Stability studies at Sandia measure the rate of formation of reaction products in energetic materials as they are aged. For $TiH_x/KClO_4$ materials the general stability reaction is as follows.

$$2TiH_x + (1+0.25x)\ KClO_4 \text{ ----> } 2\ TiO_2 + (1+0.25x)\ KCL + xH_2O$$

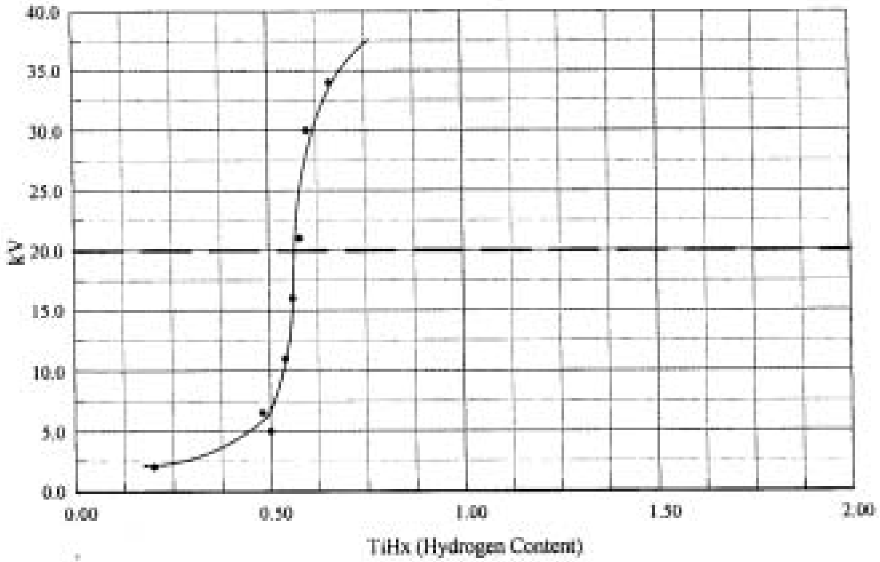

Figure 1
Electrostatic Spark Discharge (ESD) Sensitivity verses Hydrogen Content in Titanium

Measuring the rate of formation of free chloride ions and water, as $TiH_x/KClO_4$ mixtures age, provides confirming stability data. It was observed that both the free chloride ion and water formation stability data for $TiH_x/KClO_4$ materials measured the same trends.

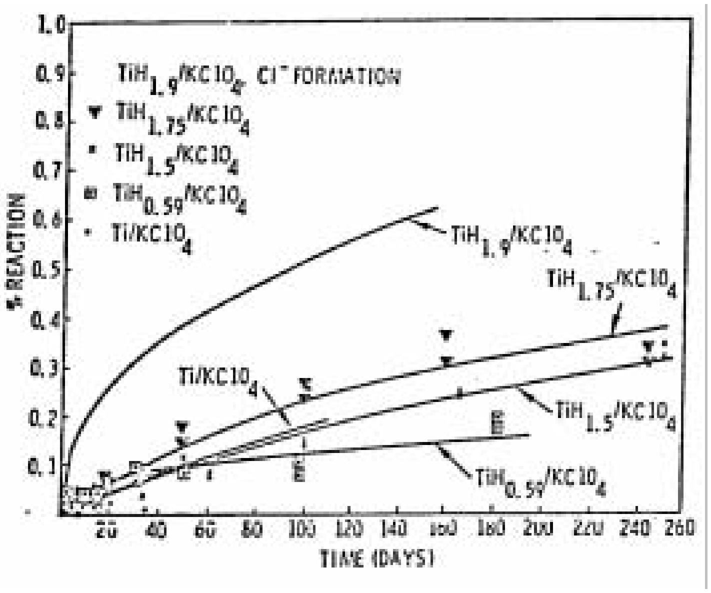

Figure 2
Stability of $TiH_X/KClO_4$ (Chloride Formation at 100°C)

Ignition reliability data at low temperatures also improved to meet requirements as the hydrogen content in TiH_x was lowered. Functional output for $TiH_x/KClO_4$ compositions remained the same over the entire TiH_x hydrogen composition range and met requirements. Later problems with ignition reliability found that the problem was related to powder/bridgewire decoupling and not materials problems. The proper density at this interface was not maintained and was primarily due to major mismatches in coefficients of thermal expansion between the header materials of construction and the $TiH_x/KClO_4$ pyrotechnic. Mechanical methods are now employed to maintain the density at the powder/bridgewire interface and have eliminated low temperature ignition problems.

From this development work with various $TiH_x/KClO_4$ compositions, weapon component designs using $TiH_{0.65}/KClO_4$ have been produced through the present. These designs have included valve actuators, igniters, special igniters for high temperature materials such as thermites and high pressure outputs. These designs meet both personnel and application safety needs for a static insensitive pyrotechnic mixture.

In the early 1980's, ESD requirements were imposed on Sandia actuator designs which required a reliability of 0.9995 to not function from a human body electrostatic pulse. Because $TiH_{0.65}/KClO_4$ was near the transition in the ESD curve (Figure 1) from insensitive to sensitive, it was decided that this formulation may not be able to meet this requirement. The equivalent hydrogen concentration was increased to $TiH_{1.65}$. An understanding of the material properties that lead to static insensitivity in TiH_x were also known.[6] This has produced a pyrotechnic, $TiH_{1.65}/KClO_4$, that can meet the 0.9995 ESD requirement plus meet all ignition reliability, functional output and long term stability/compatibility requirements necessary for Sandia components. All recent actuator designs at Sandia presently use $TiH_{1.65}/KClO_4$.

Table 2 is a listing of the various energetic material properties for $TiH_x/KClO_4$ compositions and shows the improved safety properties when compared to lead azide and lead styphnate.[6,7,8]

$TiH_x/KClO_4$ pyrotechnic compositions do not detonate or DDT. Various applications have 25 year minimum lifetime requirements which includes functional reliability, stability and compatibility. Compatibility has been proven for typical hot wire design materials and includes epoxy type materials as well as all cleaning agents. Cleaning agents include past systems such as chlorinated materials, CFC freons, normal organic solvents and water based detergents. Modern cleaning agents to replace chlorinated solvents and CFC's have also been found to be compatible with $TiH_x/KClO_4$ mixtures and include d-limonene type liquids and specially formulated water based alkaline cleaners.

$TiH_x/KClO_4$ blends, if not properly dried before loading into a device, can initiate corrosion reactions of metallic header materials due to the presence and release of moisture. These materials must be properly dried prior to loading. An indirect safety property is environmental issues associated with energetic materials. $TiH_x/KClO_4$ materials are considered to be as close to being "green", i.e. environmentally benign, as any that exist. Decomposition and function result in the inert reaction products-TiO_2, KCl, and water.

Table 2

Energetic Material Properties

Material Property	Lead Azide	Lead Styphnate	Ti/$KClO_4$	$TiH_{0.65}/KClO_4$	$TiH_{1.65}/KClO_4$
Friction	<0.1 kg	<1 kg	6.3 kg	7.2 kg	15.8 kg
Impact	<15 kg-cm	<20 kg-cm	225 kg-cm	>250 kg-cm	>225 kg-cm
ESD	<3 kv	<5kv	<5 kv	>40 kv	>40 kv
Autoignition	320°C	280°C	500°C	500°C	500°C

Notes:

1. Friction sensitivity tests were performed using a BAM friction tester manufactured by Julius Peters K. G., Berlin, Germany.
2. Impact sensitivity tests were performed using various impact testers and the data reduced to drop heights expressed in kg-cm.
3. ESD measurements were performed using the Sandia Standard Man Model and Sandia Severe Man Model and testers.
4. Autoignition temperatures were measured using thermal analysis differential scanning calorimetry (DSC) procedures at heating rates of 10°C/min in sealed hermetic cups and lids.

Over 150 publications have been written on $TiH_x/KClO_4$ materials and their properties. Summaries on the overall processing, properties and use of $TiH_x/KClO_4$ materials can be found in References 4 and 6.

DDT Explosive Development

Based upon the policy decision not to use primary explosives in component designs, Sandia has, since the early 1970's, been directing development work on new DDT explosives. This development work has been done at Unidynamics/Phoenix, Inc., now Pacific Scientific Co., Energy Dynamics Division, Chandler, Arizona under partial support from Sandia National Laboratories. Development of DDT explosives continues to the present day at Pacific Scientific.

It was determined at the start of this development program that a series of cobalt based, energetic, coordination compounds would act as DDT explosives under the right set of conditions.[9] These coordination compounds based upon 5-substituted tetrazolato pentaammine cobalt (III) perchlorate (Figure 3) , under proper confinement, undergo DDT. These compounds were also found to have much improved safety properties over traditionally used initiating primaries such as lead azide.[10] Figure 3 is the general structure for 5-substituted tetrazolato pentaammine cobalt (III) perchlorate explosives. The "X" can be -Cl, $-CH_3$, -CN, -H, $-NO_2$, -NH_3ClO_4 or other functional groups.

Characterization studies found that the X=-CN material performed best as a DDT explosive for DOE applications. In the late 1970's, this explosive, pentaammine (5-cyano-2H-tetrazolato-N^2) cobalt (III) perchlorate, given the acronym CN or CNCP has been used in production of various DDT explosive detonators (see Figure 4 for the structure of CP). In addition to DOE applications, CP has found use in commercial high temperature DDT detonator applications for down hole completion work for the oil industry. In the past few years, CP has found additional uses as an exploding bridgewire (EBW) detonator material in DOE, DOD and oil completion work applications, where special hermetically sealed detonators are required.

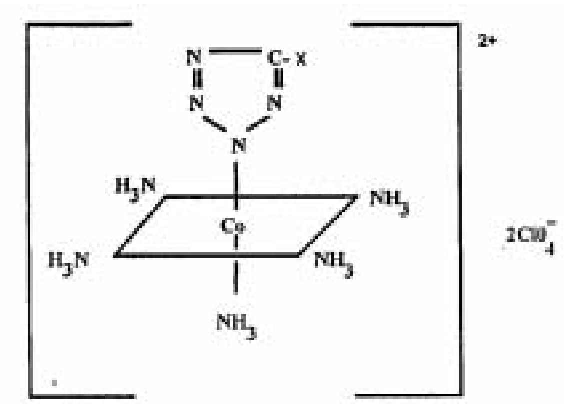

Figure 3
Chemical Structure for
5-Substituted Tetrazolato Pentaammine Cobalt (III) Perchlorates

Unfortunately, one of the starting materials used in the synthesis of CP, cyanogen, is no longer available in the United States due to severe EPA restrictions. Sufficient stocks of CP were produced to meet immediate needs in various applications. Because of the non-availability of cyanogen, CP will probably no longer be manufactured in the United States.

This has led to further development efforts to find additional DDT explosives for various applications. From these efforts have developed two additional 5-substituted tetrazolato cobalt (III) perchlorate explosives. The first, uses -Cl instead of -CN and has been given the acronym ClCP. The second, uses $-NO_2$ instead of -CN and is di-substituted in the cobalt coordination sphere. It has been designated the acronym BNCP. The proposed structures for CP, ClCP and BNCP are shown in Figure 4.

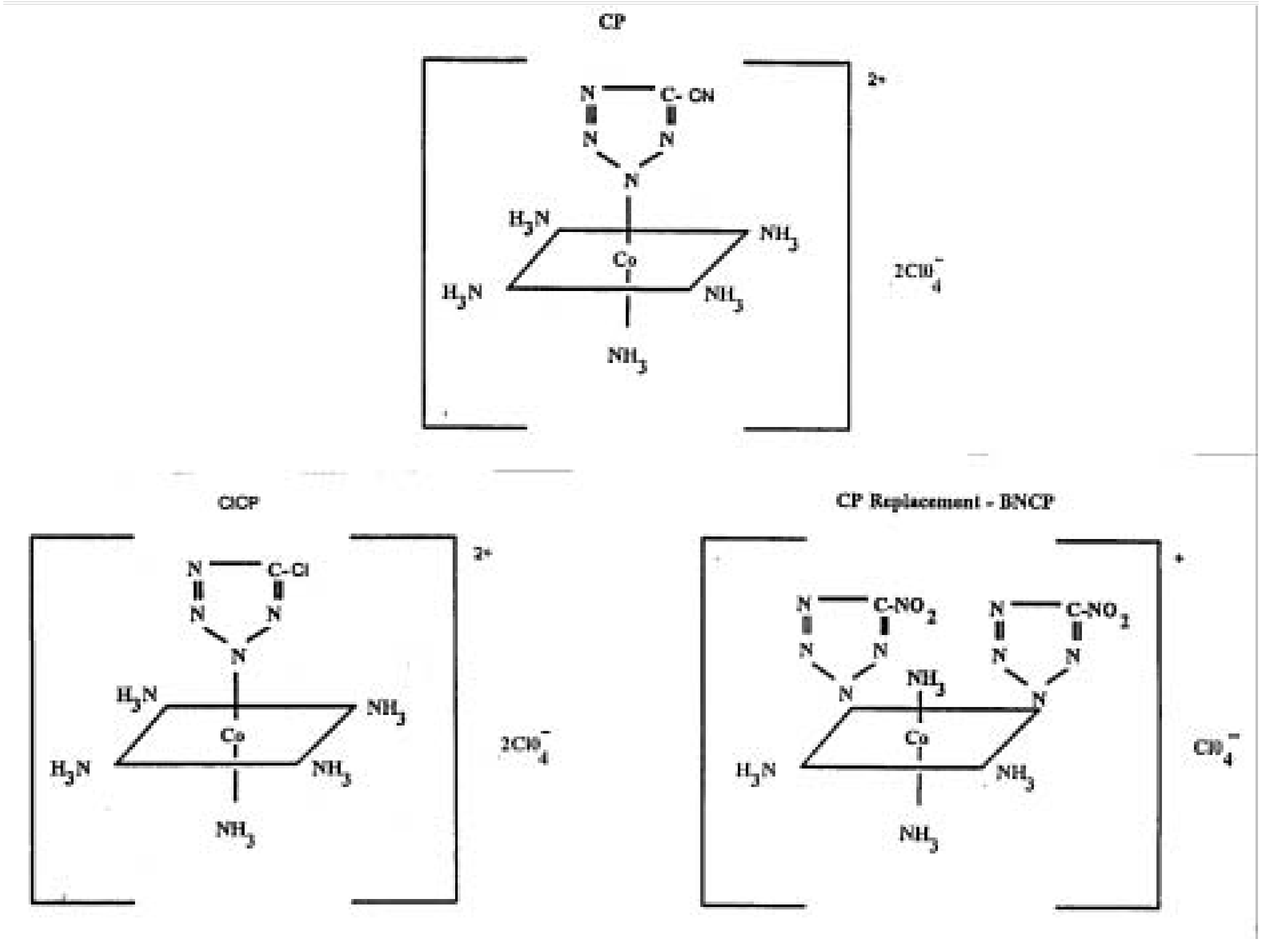

Figure 4
Chemical Structures for CP, ClCP and BNCP

ClCP is a candidate to substitute for CP in commercial down hole completion work for the oil industry because it will meet the high temperature requirements. BNCP may not meet all high temperature requirements, but has application in various DOE and DOD hot wire DDT designs. It is being promoted as a substitute for lead azide.

All 5-substituted tetrazolato pentaammine cobalt (III) perchlorate explosives have superior energetic material safety properties compared to primary explosives. Table 3 lists the safety properties for CP, ClCP and BNCP compared to lead azide.[7,8,10,11]

Table 3

Energetic Material Safety Properties of CP, ClCP and BNCP

Material Property	Lead Azide	CP	ClCP	BNCP
Friction	<0.1 kg	1-1.5 kg	1-1.5 kg	0.6-1 kg
Impact	<15 kg-cm	100-140 kg-cm	>100 kg-cm	100 kg-cm
ESD	<3 kv	>25 kv	>25 kv	>25
Autoignition	320^{o}C	290^{o}C	290^{o}C	270^{o}C

Notes:

1. Friction sensitivity tests were performed using a BAM friction tester and procedure and is manufactured by the Julius Peters K. G., Berlin, Germany.
2. Impact sensitivity tests were performed using various impact testers and the data reduced to drop heights expressed in kg-cm.
3. ESD measurements were performed using the Sandia Standard Man Model and Sandia Severe Man Model and testers.
4. Autoignition temperatures were measured using thermal analysis DSC procedures at heating rates of 10^{o}C/min with the samples inside sealed hermetic pans and cups.

With the exception of thermal autoignition properties, CP, ClCP and BNCP have superior energetic material safety properties compared to lead azide, especially ESD properties. These materials have a friction sensitivity comparable or superior to primaries, especially lead azide. Each of the 5-substituted tetrazolato pentaammine cobalt (III) perchlorate materials passes both the Sandia Standard Man and the Fisher (or Severe Man) ESD models when tested in the bulk.

Because of their enhanced energetic materials safety properties, especially ESD safety, these of DDT explosives have found application in DOE, DOD and commercial detonator designs. These DDT detonator designs, using CP, have been in production for weapon applications since 1980. Recent needs for a hermetically sealed EBW detonator have led to the use of CP in these designs for both the DOE and DOD. Commercial versions of high temperature DDT detonators to replace lead azide for the oil industry have been in production since the early 1990's.

CP, ClCP and BNCP are considered moderately high temperature explosives with thermal properties similar to HMX. Their thermal properties have been tested for potential application in down hole completion work for the oil industry.[11] BNCP may not meet a one hour/260^{o}C not to initiate/deflagrate requirement for down hole completion work, whereas CP and ClCP will meet this requirement. BNCP is about 20^{o}C less stable than CP or ClCP. The oil industry uses CP for down hole oil completion applications while ClCp is a candidate material for these applications..

CP and ClCP will DDT, but both require fairly long column lengths (4-6 mm) in steel confinement to achieve steady state detonation. High confinement within steel charge holder assemblies is necessary for CP to DDT. BNCP requires lesser confinement than CP or ClCP and has been found to achieve steady state detonation pressed in materials such as aluminum and plastics such as Lexan. BNCP will also DDT in much shorter column lengths (2-3 mm) than CP or ClCP when confined in steel.

The output of BNCP is also greater than CP achieving typical steel witness plate dents of 0.66 mm verses 0.45 mm for CP. Typical flying plate velocities of 3.2 mm/us verses 2.7 mm/us for BNCP and CP respectively have been measured, again showing greater output for BNCP.

All DDT materials have good stability characteristics for long lived applications. CP has been aged for over 15 years at temperatures up to 80^{o}C with less than 0.05% reaction occurring at that temperature. Down hole oil completion applications require functionality after exposure to temperatures of 160^{o}C for 100 hours and these DDT materials including BNCP meet this requirement. Chemical analytical stability procedures for these materials, measures the rate of formation of free chloride ions, ammonium ions, and cobalt II ions versus time and temperature. These are the expected reaction products during the decomposition of 5-substituted tetrazolato pentaammine cobalt (III) perchlorate materials.

These materials are compatible with all typical hot wire and EBW materials of construction.[12] The only exception has been CP which was found to be incompatible with copper and zinc; thus these combinations should be avoided.[12] This includes alloys, such as Nicusil, which contain copper. CP is compatible with silver and gold. These DDT explosives are compatible with adhesives such as epoxies, plus have been found compatible with past and current header cleaning materials such as those listed for $TiH_x/KClO_4$.

As with $TiH_x/KClO_4$ materials there have been numerous publications and presentations on the synthesis, processing, properties and use of 5-substituted tetrazolato pentaammine cobalt (III) perchlorate materials. The number of publications exceeds 100.

Uses

The uses of $TiH_x/KClO_4$ pyrotechnics and 5-substituted tetrazolato pentaammine cobalt (III) perchlorate DDT explosives are quite extensive and varied. The primary use of TiHx/KClO4 pyrotechnics has been in actuator designs to function valves and open gas bottles. Their reliability to function has been 0.9995 along with a 0.9995 ESD reliability requirement not to function from an electrostatic pulse. Because of their insensitivity, especially to ESD, personnel safety is also enhanced during handling.

Other applications have included hot particulate ignitors for thermal batteries, ignition and high pressure outputs in reefing line cutters, next assembly igniters for special high temperature materials such as thermites, and percussion actuated igniters. Commercial uses include igniters for air bag deployment modules, with enhanced ESD safety over traditionally used zirconium/potassium perchlorate pyrotechnics.

Ignition of $TiH_x/KClO_4$ materials is achieved by a variety of methods. Included are traditional hot wire methods, through bulkhead initiation from thermal and detonation sources, percussion primer ignition, semiconductor bridgewire (SCB) ignition, and laser initiation. Future designs at

Sandia will probably incorporate laser ignition methods. This technology has been proven in development and will be the method of choice in the future. These pyrotechnics do not require doping with materials such as carbon to enhance their laser ignitability.

The 5-substituted tetrazolato pentaammine cobalt (III) perchlorate explosives can achieve steady state detonation from hot wire/exploding bridgewire methods, percussion primer ignition, SCB ignition, and laser methods. As with $TiH_x/KClO_4$ materials, future designs will probably incorporate laser methods of ignition for achieving DDT. Laser technology for initiation has been proven and provides additional safety features during use. These DDT materials, though typically yellow in color, still require doping with such materials as carbon black to enhance their absorbtivity for ignition reliability from a laser ignition source.

Current applications of these materials includes next assembly initiation of an explosive, such as HMX, destruct detonators, EBW explosives, explosive actuators, and delay detonators.

Conclusions

New pyrotechnic and DDT explosive materials have been developed for safety related issues during their handling and use. $TiH_x/KClO_4$ pyrotechnics were developed to meet high reliability requirements not to function from an ESD human body electrostatic pulse. This has been achieved which enhances both personnel and application safety issues during their use. These materials have proven methods of initiation and provide functional outputs for many kinds of applications.

DDT explosives based upon 5-substituted tetrazolato pentaammine cobalt (III) perchlorate structures have also been developed to replace traditionally used primary explosives such as lead azide. Their enhanced energetic material safety properties, especially ESD insensitivity, are proven, and they have sufficient output, especially BNCP, to substitute for lead azide. Their applications have been proven. Hot wire, EBW, SCB and laser ignition methods for a steady state detonation output have also been demonstrated.

Both kinds of materials have been developed to an extent that they are considered mature technologies. Because of their enhanced safety properties, especially ESD insensitivity, both kinds of materials are finding new application outside of the DOE within the DOD and private, commercial interests. With the advent of laser initiation methods, these materials will see additional uses in the future.

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