



Study of Submicron Structured Energetic Coordination Metal Complexes for Laser Initiation Systems^{*)}

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Abstract: Development of condensed energetic systems, especially photosensitive primary explosives for the systems of laser initiation is one of the foremost fields of application of energetic materials. There have been synthesized and investigated a coordination complex of mercury(II) chlorate(VII) with 5-hydrazinotetrazole as ligand (compound **I**) as a potential photosensitive primary explosive. This complex consisting of particles of 1.1 ± 0.8 μm size demonstrated the highest sensitivity to laser irradiation combined with an extremely low initiation threshold. On the basis of compound **I** a photosensitive formulation EC-2 containing ~90% of the complex mercury(II) chlorate(VII) and ~10% of an optically transparent polymer as an inert matrix has been proposed. Formulation EC-2 has extremely high sensitivity to Q-switch IR-laser irradiation. Formulation EC-2 with added ultra disperse diamonds (UDD) has shown lower initiation thresholds in comparison with neat EC-2 or EC-2 with added submicron black carbon, fullerenes or nanotubes instead of UDD.

Keywords: chlorate(VII) complex, threshold of initiation, IR-laser, pulse, UDD

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Introduction

Nearly all of the energetic compounds and formulations used in basic and research, mining works, civil and airspace industry are initiated by electrical means. Because of this, such devices are inherently susceptible to accidental initiation by external stimuli such as electrostatic discharge (ESD), electromagnetic interference (EMI) and radio irradiation (RI). At the same time pulse lasers provide a practical means of accomplishing electrical isolation of the energetic material, thereby eliminating hazards associated with ESD, EMI and RI [1-3].

A photosensitive energetic compound is one of the basic elements of laser initiation circuits. In comparison with the other means of shock initiation, laser radiation has a number of features. Irradiation of a photosensitive explosive by the laser mono pulse results in a sequence of complicated non-linear non-equilibrium thermochemical processes that can be divided into several stages, the main ones being: 1) light absorption by the substance, 2) the energy and matter transfer, 3) chemical reactions of thermal decomposition [4, 5].

Laser IR radiation provides high rates of local changes in temperature of the irradiated material approaching to $10^{10} Ks^{-1}$ and forming the gradients of temperature up to $10^7 Kcm^{-1}$ that results in strong interactions of energy, substance, charge and other streams in the sample. Such parameters can be achieved with no other ways of action. The modern theory of initiation of high explosives (HE) under the conditions of pulsed stimuli is based on the concept of "hot spots" – the centers of intense local heating that are responsible for fast reactions of thermal decomposition, including an explosion. Local heating centers are formed owing to the structural heterogeneity of the substance (dot defects, congestions of vacancies, dislocations, interfaces, etc.), chemical impurities, microinclusions occurring due to non-stoichiometric composition of the initial products, presence of various additives and so on. The defects can also arise in the field of an intense light wave. The main hypothesis of warming up the optical dissimilarities resulting in HE ignition has been taken up to rationalize the laser initiation considering a short duration and therefore the high power of the laser pulse. Carbon inclusions embedded in organic high explosives, or metal microinclusions present in heavy metal azides can play the role of the nuclei. The smallest size of experimentally found "hot spots" under the action of laser monopulse (Q-switch) ($\lambda = 1064 \text{ nm}$, $\tau_q = 8 \text{ ns}$) depends upon the nature of an explosive. In a covalent-bonded explosive (RDX, hexogen) it equals to $\sim 20 \text{ nm}$. For ionic compounds (*viz.*, ammonium chlorate(VII)) the size of "hot spots" is larger and amounts to 200-300 nm or even to 1000 nm [6]. The center of decom-

position is most likely located at depth of the substance rather than on its surface. Such location is more favorable for the development of thermal decomposition processes due to the minimal scattering of hot products of explosion from the initiation zone. For the visible and near infrared regions of electromagnetic spectrum HE behaves as a diffuse-dispersing medium with a closely packed diffuser. On irradiation of a sample a repeated light scattering takes place in its volume that results in the growth of illumination inside the sample and leads to the production of "hot spots" in the most illuminated zone located in the volume of the irradiated sample [7].

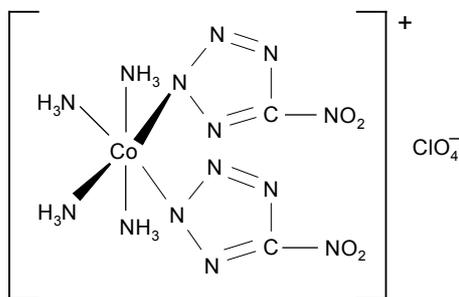
It was possible to explain regularities of HE initiation with laser monopulse using the hypothesis of "hot spots" as ignition centers. Supersonic cracking in a charge during irradiation explains the high sensitivity to laser monopulse of confined HE with partly blocked ways for scattering of the gaseous reaction products. The process of HE cracking may also result in the failure of ignition. The above assumption allowed one to explain the significant increase in sensitivity of pressed HE charges having a glass plate near their free surface [8].

As opposed to HE, the ignition of primary explosives (PE) by laser monopulse does not depend on the presence of a barrier at the surface of explosive. It means that the rate of chemical reaction in PE is so high that the scattering of gaseous products into free space does not significantly influence the process of ignition under the action of laser IR radiation. Among various energetic compounds inorganic azides show the greatest susceptibility to laser irradiation. Monocrystals of α -lead- and silver azides have a threshold of initiation equal to 4-6 mJ cm⁻². As the size of HE monocrystals is reduced, the critical density of initiation energy grows. There exists the limiting size of monocrystals capable of detonation. The samples of smaller dimensions decompose without explosion at any capacity of the initiating laser monopulse. Great variability is observed in the sensitivity of pressed charges of lead azide to the monopulse of the Nd-laser (from ~10 mJ cm² up to ~1 J cm⁻²), according to the data of different authors. Such deviations are apparently connected with dissimilar conditions of the experiments, first of all with respect to the variations in the area of the irradiated zone. It has been found that the energy necessary for initiation decreases and approaches its lower limit with the diminution of the area of a light stain. Thus the threshold of the energy density grows indefinitely. As the size of the influence zone grows, the threshold of energy density gradually decreases, reaching the limiting value [7, 8].

Minding the results of the investigation on the influence of crystal size on the susceptibility of photosensitive PE to the laser pulse, it is possible to suppose that each photosensitive compound has its optimal size of particles possessing

the lowest threshold of initiation. Their diameter should be close to the size of "hot spots". Apparently, the size of nanostructures for energetic ionic compounds should be less than 100 nanometers, according to the latest results in the investigation of centers of intense local heating emerging under the laser initiation of PE.

Depending on particular tasks, various energetic materials with differing values of initiation thresholds to laser Q-switch pulse (pulse-time $\sim 10^{-8}$ s) or a non-Q-switch pulse (pulse-time $\sim 10^{-3}$ s) have been suggested [9-14]. So, in the system of pyroautomatics of rocket complexes in USA are successfully used blasting caps using light-sensitive charges of energetic metal complex tetraamine-*cis*-bis(5-nitro-2*H*-tetrazolate- N^2)cobalt(III) chlorate(VII) (BNCP) [15-18].



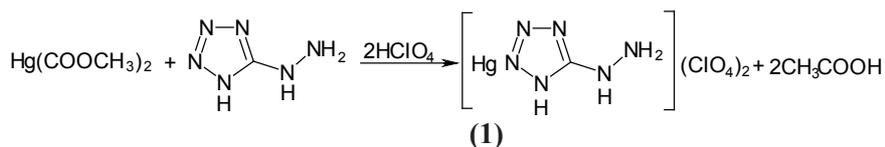
Tetraamine-*cis*-bis(5-nitro-2*H*-tetrazolate- N^2)cobalt(III) chlorate(VII)

Discussion

There have been synthesized and investigated as potential photosensitive energetic primary explosives a number of complex chlorates(VII) of mercury(II) with hydrazinoazoles as ligands. The choice was based on the high positive enthalpy of formation of hydrazinoazoles, high oxidizing ability of the chlorate(VII) ion and high ionization potential of the mercury(II) cation. This suggestion has been experimentally tried. There have been investigated the following hydrazinoazoles as ligands of coordinated mercury(II) chlorates(VII) [19]:

1 <i>H</i> -3-Hydrazino-5-aminopyrazole	1 <i>H</i> -3-Hydrazinopyrazole-5-one	3(5)-Hydrazino-4-amino-1,2,4-triazole
3-Hydrazino-4-amino-5-methyl-1,2,4-triazole	3-Hydrazino-4-amino-5-mercapto-1,2,4-triazole	1 <i>H</i> -5-Hydrazinotetrazole

All the salts turned to be light-sensitive explosives and can be initiated by laser pulse (pulse time $\sim 1 \mu\text{s}$, $E \sim 0.2 \text{ J}$, $\lambda \sim 1.06 \mu\text{m}$, ray diameter $\sim 0.5 \text{ mm}$). The chlorate(VII) complex of mercury(II) with 5-hydrazinotetrazole as ligand showed the greatest sensitivity to the laser monopulse. A laboratory synthesis of the complex may be carried out by mixing of solutions of a mercury(II) salt (for example, acetate) and the ligand (1*H*-5-hydrazinotetrazole) in HClO_4 according to the general Scheme [20]:



Scheme

The residue was filtered, washed successively with water and alcohol and then dried.

The chlorate(VII) complex of mercury(II) (1) is a white crystalline solid with the following characteristics (Table 1):

Table 1. Characteristics of complex **1**

Property	Value
Molecular weight	499.5
Density of monocrystals, g cm ⁻³ (calc.)	3.45
Oxygen balance, %	+12.8
Oxygen coefficient, %	+180
Ignition temperature, K (5 s delay)	459
Onset of thermal decomposition, K	448
Impact sensitivity, mm (Veller drop hammer)	60 /125
Flame sensitivity, mm (100% ignition / 100% refusal)	60 /150
Detonation velocity at density 3.4 g cm ⁻³ , km s ⁻¹ (calc.)	6
Minimum charge of 1 for RDX in blasting cap No 8, g	~0.015

Coordination compound **1** is a non-hygroscopic solid, insoluble in alcohol, acetone, aliphatic, chlorinated and aromatic hydrocarbons and soluble in dimethylsulphoxide. It is decomposed by an alkaline solution of KMnO₄ to produce unexplosive compounds.

As was shown by the microscopic dispersion analysis, the synthesized complex contains both fine and coarse crystals (Table 2, Figure 1):

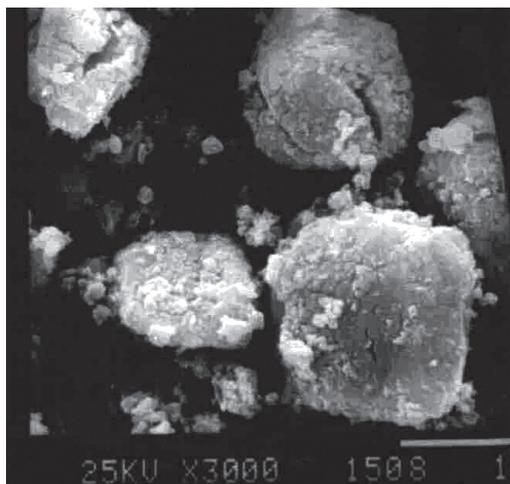
**Figure 1.** SEM photograph of polydispersion crystals of **1**.

Table 2. Distribution of particle size of complex **1**

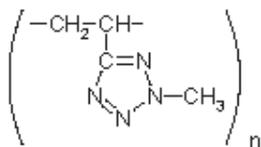
N	d, μm	d _{min} - d _{max} , μm
935	1.1 \pm 0.8	0.5-8.1

N - data file, d - average size of crystals

The microscopic dispersion analysis of complex **1** was carried out on an electronic microscope JSM-35CF (JEOL company, Japan).

Using of complex **1** as photosensitive PE is problematic because of its high sensitivity to mechanical stimuli and propensity to spontaneous aggregation of nanostructures resulting in the increase of the threshold of laser monopulse initiation. The problem of stabilization of ultradispersed powders of energetic compounds is usually solved by preparing composite materials containing submicron particles of solids embedded into a chemically inert polymer matrix. Such procedure allows to avoid aggregation of submicron particles, protects them from external influences but considerably hinders practical application of such materials. Optically transparent polymers are traditionally used as an inert matrix. Polymer-bound samples of complex mercury(II) chlorate(VII) **1** have an increased sensitivity to laser pulse in comparison with the pressed charge of the metal complex. The initiation threshold reduces to 2.3 mJ cm⁻² ($\lambda = 1.06 \mu\text{m}$, $\tau_q = 30 \text{ ns}$, $d_{\text{beam}} = 4 \text{ mm}$), which is smaller than that for the pressed charges of heavy metal azides. The sensitivity to mechanical stimuli for such formulation is reduced to the level of HE making them safe enough for transportation, storage and application.

The photosensitive formulation EC-2 consists of ~90 % of complex mercury(II) chlorate(VII) **1** and ~10% of an optically transparent polymer. For example, poly-2-methyl-5-vinyltetrazole **2** can be used as a matrix. Properties of polymer **2** are listed in Table 3.



Poly-2-methyl-5-vinyltetrazole (**2**)

Table 3. Physical and chemical characteristics of polymer 2

No.	Property	Value
1	Intrinsic viscosity in DMF, $10^{-3} \text{ m}^3 \text{ kg}^{-1}$	0.64
2	Mass fraction of the residual monomer, %	0.09
3	Mass fraction of ashes, %	0.16
4	Mass fraction of moisture, %	1.9
5	Weight-average molecular weight	68400
6	Number-average molecular weight	27700
7	Polydispersity coefficient	2.5
8	Enthalpy of formation, kJ kg^{-1} (calc.)	1850

A suspension of EC-2 formulation in a volatile solvent was deposited on the substrate surface. Photosensitive film charges were manufactured according to this procedure. These films were used in laboratory experiments for the generation of shaped shock waves, hardening of steel by explosion, development of test models of optical detonators.

Table 4. Results of derivatographic analysis (rate of heating 5 K min^{-1})

Sample	Decomp. stage	ΔT of the effect, [K]	T_{max} of the effect, [K]	Mass loss [%]	Character of the effect
Complex 1	1	443-468	459	30	Exo
	2	468-533	473	40	Exo
Polymer 2	1	493-583	548	40	Exo
Complex 1 - 90% + Polymer 2 - 10% (Formulation EC -2)	1	443-468	459	30	Exo
	2	468-533	473	70	Exo

The influence of polymer 2 on salt 1 decomposition was of great interest. The results of derivatographic analysis are given in Table 4.

From Table 4 it is obvious that salt 1 decomposition takes place in two steps. The first step is the ligand oxidation, the second one is the total decomposition of the complex. Table 4 also shows that polymer 2 decomposes in a single step that involves tetrazole ring destruction. It is also seen that formulation EC-2 decomposes in two steps, like salt 1. The oxygen balance of EC-2 is near zero so that its destruction produces only gaseous products of decomposition.

The results also show that polymer 2 and complex 1 is compatible.

Since explosives are convenient models for investigating fast reactions in chemically active media, so it seems rather important to test the sensitivity of

lightsensitive explosives to laser initiation, though the mechanisms of their low threshold initiation are not yet worked out.

Sheets of lightsensitive EC-2 explosive (thickness ~ 1.5 mm, diameter ~ 5 mm) were also prepared and investigated in this respect. Two lasers, viz, Nd-glass laser for the investigation of ignition in a monopulse mode ($\tau_q = 30$ ns), and a laser with an active element comprising Nd-containing potassium-gadolinium tungstate were used to generate initiation in a free generation mode ($\tau_q = 30 \mu\text{s}$). Both lasers operate at wavelength 1060 nm. The laser beam illuminates the central area of charges. The thresholds of initiation of the charges were calculated with 50% probability of ignition. The accuracy of calculation of the thresholds of initiation was near 20%. The thresholds of initiation were calculated in terms of the minimum energy, E_o (mJ), or minimum of energy density H_o (mJ cm^{-2}). The results of experiments are listed in Tables 5-7.

Table 5. The sensitivity of EC-2 explosive as a function of the diameter of the illuminated zone ($\tau_q = 30$ ns)

d, diameter of the illuminated zone [mm]	E_o [mJ]	H_o [mJ cm^{-2}]
0.48	1.8×10^{-2}	10.14
3.18	0.6	7.48
9.52	1.8	2.57

Table 6. The thresholds of initiation of EC-2 explosive as a function of exposure

τ_q , pulse duration [ns]	d, diameter of the illuminated zone [mm]	E_o [mJ]	H_o [mJ cm^{-2}]
30	1.02	6.8×10^{-2}	8.32
30×10^3	1.02	2.52	308.4

Table 7. Delay of ignition of EC-2 explosive as a function of energy density of the laser beam $\tau_q = 30 \mu\text{s}$)

H_0 [J cm ⁻²]	Delay, τ [μs]
0.26	15
0.35	14.5
1.10	11.5
3.06	1

It was found that the sensitivity of EC-2 explosive depended both on the diameters of the illuminated zone and on the duration of illumination with the laser beam (Tables 5, 6). The thresholds of initiation of EC-2 explosive are smaller than those published for lead azide. For example, they are $H_0 \sim 10.1 \text{ mJ cm}^{-2}$ for EC-2 explosive and $H_0 \sim 23.4 \text{ mJ cm}^{-2}$ for a pressed charge of lead azide (1500 MPa) of $\sim 0.48 \text{ mm}$ in diameter [21].

The times of ignition delay for EC-2 explosive depended on the energy density of the laser beam, H_0 , and were always smaller than the duration of free generation (see Table 7).

Nanoparticles possessing the size in the range 1-100 nanometers often show unusual properties in comparison with those of the micron size. Grade of the particles substantially changes macroscopic properties of materials. From among the numerous applications of nanostructured materials it is expedient to single out the design of energetic condensed systems, for example, photosensitive PE for the systems of laser initiation.

It is known that black carbon particles of micron size decrease the thresholds of initiation of explosive charges by non-Q-switch IR-laser pulses [22, 23].

But there also exist some unknown effects of other forms of carbon on the sensitivity of explosive charges to laser pulses.

The results of investigation of the influence of black carbon (particle size $\sim 1 \mu\text{m}$) and nano-diamonds (ultra disperse diamonds of detonation synthesis) (particle size $< 100 \text{ nm}$) fullerenes ($C_{60}/C_{70} - 80/20$) (particle size $< 1 \mu\text{m}$), and nanotubes (particle size $< 100 \text{ nm}$) on the sensitivity of charges of EC-2 to Q-switch IR-laser pulses are presented in Table 8 ($\lambda = 1.06 \mu\text{m}$, $\tau_q = 8 \text{ ns}$, $d_{\text{diaphragm}} = 0.86 \text{ mm}$). The diameter of charges of EC-2 equals to 5 mm and its thickness is 2 mm [24].

Table 8. Initiation of EC-2 by Q-switch laser pulse

Additive	Charge	E_0 [μ J]	Result of initiation	
-	EC-2	310	Explosion	
Black carbon	EC-2 + 0.5%	2450	Explosion	
	EC-2 + 1%	2550	Explosion	
	EC-2 + 2%	3050	Explosion	
	EC-2 + 3%	3250	Explosion	
	UDD	EC-2 + 0.5%	260	Explosion
UDD	EC-2 + 1%	200	Explosion	
	EC-2 + 3%	190	Explosion	
	EC-2 + 5%	340	Explosion	
	Fullerens	EC-2 + 1%	630	Explosion
		EC-2 + 3%	830	Explosion
EC-2 + 5%		730	Explosion	
Nanotubes	EC-2 + 0.5%	640	Explosion	
	EC-2 + 1%	590	Explosion	
	EC-2 + 2%	640	Explosion	
	EC-2 + 3%	850	Explosion	

The accuracy of results was ~10%.

Black carbon, fullerenes and nanotubes have been found to be phlegmatizers. They increase the threshold of initiation of EC-2. The additives increase the surface of absorption of irradiation that results in increase of the dissipation of laser energy from the surface of the charge and in increase in threshold of initiation.

The influence of UDD on EC-2 differs from that of other forms of carbon. UDD has a high enthalpy of formation. The experimental value of ΔH_f^0 of UDD is about 2.56-2.95 MJ kg⁻¹ and the calculated ΔH_f^0 equals to 3.5 MJ kg⁻¹. [25]. UDD in up to ~3% concentration increases the sensitivity of EC-2 to Q-switch laser pulse. This effect may be the result of increase in energy absorption inside the EC-2 charge because of an extremely high refractive index of UDD. An increased quantity of UDD decreases the sensitivity of EC-2 to the monopulse of IR-laser, playing the role of an inert substance lowering the sensitivity of EC-2 to laser pulse.

So UDD is the first example of a substance which increases the sensitivity of a light sensitive energetic material to Q-switch laser pulse.

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