ANNOTATION

PhD dissertation work

Specialty 6D073400 – "Chemical technology of explosives and pyrotechnics"

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"Synthesis of solid compounds with high energy characteristics"

The dissertation is devoted to the development and research of new approaches to the synthesis of solid compounds that form the basis of rocket fuel using Kazakhstani raw materials. The high demand of the aviation and space industry, as well as the Ministry of Defense of the Republic of Kazakhstan in the development of its own rocket fuel, determines the scientific, economic and industrial interest in implementing the proposed project and obtaining its results.

A thermodynamic analysis of the studied compositions was carried out. The adiabatic combustion temperatures of NA T_{AD} are calculated to be 1305^0C; with a specific impulse J_{UD} - 99-100 s and an oxidizer excess coefficient of 0.5. Tad for NA-SKDM80-A1 systems, depending on the excess of the oxidizing agent – 2537-2544 K; for a system using a mixed oxidizer –3398-3425 K; for a system with an active fuel-binder - 3297-3334 K, specific impulse, respectively - 240-265 s; 269-270 s; 257-260 p.

Methods for regulating the combustion rate of metallized solid fuel systems using a recipe method have been studied. It has been established that the addition of a silicon dioxide (SiO_2) catalyst increases the combustion rate of the systems under study when using ASD-6 aluminum.

An aluminum diboride alloy was obtained. Phase and X-ray spectral analysis of the samples showed that the main phase - 95.78% - is AlB_2, the impurities present are within the acceptable limits. The influence of the synthesized diboride on the combustion rate and completeness of combustion of high-energy compositions when used as a metal fuel was studied.

The influence of MCM on the modification of aluminum and on the combustion parameters of high-energy compositions based on ammonium nitrate was studied. The dependence of the combustion rate efficiency coefficient on the amount of modifier in the composite and the time of mechanochemical treatment has been established.

The first chapter is devoted to a literature review, which examines the properties of solid compounds with high energy characteristics, the most promising solid compounds, conditions for the synthesis of dibasic and mixed fuels, the role of combustible binders in fuels, the use of plasticizers in condensed energy systems, metal powders and their role in processes synthesis by SHS method.

The formulation of problems and research methods are determined by the need to obtain solid compounds with high energy characteristics capable of releasing

large quantities of a gaseous working fluid with a high temperature due to the heat of exothermic reactions.

The second chapter of the work provides an analysis of the properties and composition of the selected raw materials, methodologies for determining the combustion temperature of compositions with a pyrometer and research in the combustion chamber of energy-intensive materials, modes of preparation of raw materials and determination of the deformation-strength and rheological characteristics of compositions, methodology for X-ray phase analysis

The third chapter provides a thermodynamic analysis of the selection of components for high-energy compositions that are environmentally friendly, affordable and provide a high combustion rate in a self-propagating mode. Optimal compositions and density of fuel charges were selected to ensure maximum combustion rate. The excess coefficients of the oxidizing agent from the selected ones – ammonium nitrate and potassium nitrate – were calculated. It has been established that the reaction of aluminum nitride formation in the endothermic regime is possible at a temperature of about 3000 K.

In the fourth chapter, the equivalent formulas of all components of the mixture are calculated, the equivalent formulas of high-energy mixtures and the composition of the compositions are calculated. The influence of the amount of excess oxidizer on the combustion rate was studied. It has been established that as α increases, the combustion rate of the system increases. The influence of the amount of metal fuel on the combustion rate was studied. The optimal density of fuel samples was chosen to be 1.75-1.8 g/cm^3. The optimal amount of aluminum in the high-energy mixture was 15%.

In the fifth chapter, the efficiency coefficient of the additive for the combustion rate with ASD-6 is determined to be 1.59. For mixed metal fuel K – 1.5-2.38. For aluminum powder in the fuel mixture, the addition of an inhibitor in the form of tin chloride ($SnCl_2$) is effective, blocking the formation of aluminum nitrides at high process temperatures, which reduce the combustion temperature and speed. The influence of ASD grade aluminum modified with metals on the combustion rate of a solid fuel system based on ammonium nitrate and the completeness of fuel combustion was studied. It has been established that the use of modified aluminum makes it possible to increase the combustion rate of the system compared to the base one by 1.7-2.1 times and reduce the slag content by 60 wt. % for AlSn, 42 wt. % – AlPb and 2 wt. % – AlGa and reduce the aluminum content in the cinder to 8.1-1.29 wt. % compared to the base composition of 18.9%.

In the sixth chapter, an aluminum diboride alloy was obtained. Phase and X-ray spectral analysis of the samples showed that the main phase - 95.78% - is AlB_2, the impurities present are within the acceptable limits.

The influence of the synthesized diboride on the combustion rate and completeness of combustion of high-energy compositions when used as a metal fuel was studied.

It has been established that the combustion rate of HEAs using aluminum diboride is 3.47 times higher than the combustion rate of the base composition based on ammonium nitrate. The mass of cinders after combustion of the wind turbine is: base composition 2.58 g or 58.6%, composition with AlB_2 0.85 g or 17.9%. Content of residual aluminum in cinders, %: base composition 18.9, composition with AlB_2 0.0-0.76.

Relevance of the research topic.

Condensed energy systems are rocket, artillery, plasma, laser and rifle powders, mixed solid rocket propellants, all types of explosives, pyrotechnics and hydro-reacting solid propellant compositions. Energy-saturated condensed systems are the basis of the state's defense capability and influence the economy and the development of science and technology.

Systems with high energy characteristics – ECS (energy condensed systems) – are an effective source of energy for equipment and new technologies. Special ECS made it possible to create unique and highly relevant technologies.

With the help of EKS, welding of materials that cannot be welded by the classical method is carried out, stamping and cutting of metals, strengthening of steel structures, synthesis of diamonds from carbon, and much more.

Fuel in the broad sense of the word is a substance capable of releasing energy during certain processes, which can be used for technical purposes. Chemical fuels release energy through exothermic chemical reactions when burned.

The development of the physicochemical foundations for the synthesis of solid substances or a mixture of individual substances capable of burning, releasing a large amount of gaseous working fluid heated to a high temperature, to improve jet propulsion remains a relevant and important problem, the solution of which is aimed at the research dissertation.

Purpose of the study: Obtaining solid compounds with high energy characteristics capable of releasing large amounts of gaseous working fluid at high temperatures due to the heat of exothermic reactions

Research objectives:

1. Determination of the thermodynamic characteristics of the VES formulation containing oxidizing agents: KNO_3, $NaNO_3$, NH_4NO_3, $(CH_2)_nN_n(NO_2)_n$, binder butyl rubber, plasticizer dibutyl phthalate, catalyst SiO_2 and metal combustible grades ASD-6 and aluminum PAP-1.

2. Optimization of the composition of HEAs and technological modes of SHS, taking into account the excess of oxidant α=0.5 and calculation of equivalent formulas of the initial compositions. Carrying out synthesis, testing and control of physico-chemical parameters of compaction of the resulting product.

3. Modification of Al powders of the ASD-6 and PAP-1 grades with Sn, Ga and Pb compounds in the presence of the SiO_2 catalyst and the $SnCl_2$ inhibitor with the determination of the optimal technological parameters of the kinetics of the synthesis process and regulation of the preparation of the feedstock from the theoretically calculated ones.

4. Determination of the optimal ratios of the components of mechanically activated aluminum PAP-1 and graphite, optimization of the SHS modes of aluminum boride AlB_2 with the establishment of the speed and thermometric parameters of the completeness of combustion of the wind turbine.

Research methods

To determine the possibility of the combustion process occurring in the SHS mode of the fuel composition used, thermodynamic calculations were carried out for the reactions taking place during the combustion process of the system, and factors influencing the efficiency indicators of solid fuel were studied. Research methods: photo recording and video recording, thermometry using thermoelectric sensors or pyrometers, modern analytical physical methods, etc.

The main provisions of the dissertation submitted for defense:

1. The thermodynamic calculation determined the adiabatic combustion temperature of T_{AD} 2537-3398 K, specific impulse J_{UD} 240-260 s and oxidizer excess coefficient 0.5 for the synthesis of high-energy solid combustible materials with a basic composition of NH_4NO_3 - 64 wt. %, SKDM-80 – 20 wt. %, Al-15 wt.% as a result of the reaction of Al nitride formation in endothermic mode at 3000 K.

2. In the SHS reaction, the mass composition of the high-energy composition and the optimal empirical formulas of its components were established $C_4H_8N_8O_8$ ($C_{13,513}H_{27,027}O_{27,027}N_{27,027}$) KNO_3 ($K_{9,89}N_{9,88}O_{29,67}$) and Al, with sample densities from 1.69 to 1 .8 g/cm3s, the combustion rate is observed in the range of 0.6 to 2.8±0.61 mm/taking into account the oxidizer excess coefficients α=0.5-0.9.

3. Modification of metallic Al grades ASD-6 and PAP-1 with Sn, Ga and Pb compounds in a mixture with high-energy compositions NH_4NO_3, SKDM in the presence of catalyst SiO_2 and an inhibitor $SnCl_2$ allows you to regulate combustion rates using a recipe method.

4. The burning rate of condensed systems based on NH_4NO_3 - 65, KNO_3 -7, SKDM80 - 13, AlB_2 - 15 is 3.47 times higher than the burning rate of the base composition of ammonium nitrate with the participation of powdered Al, while the completeness of combustion of high-energy systems by weight of cinders is AlB_2 0.85 g or 17.9% of the original in the reaction products.

Scientific novelty of the results obtained

1. For the first time, the indicators of the thermodynamics of the formation of hydroelectric compounds containing oxidizing agents: $NaNO_3$, KNO_3, NH_4NO_3, binder butyl rubber, plasticizer dibutyl phthalate, SiO_2, and Al grades ASD-6 and PAP-1 were determined.

2. With an excess of oxidant α = 0.5, the composition of the wind turbine and the combustion mode were optimized for the first time with the calculation of equivalent formulas of the initial compositions, synthesis, testing and control of the physicochemical parameters of compaction of the resulting product.

3. For the first time, Al powder of the ASD-6 and PAP-1 grades was modified with Sn, Ga and Pb compounds in the presence of the SiO_2 catalyst and the $SnCl_2$ inhibitor with the determination of the optimal technological parameters of the kinetics of the synthesis process and regulation of the preparation of the feedstock from the theoretically calculated ones.

4. For the first time, the optimal ratios of the components of mechanically activated aluminum PAP-1 and graphite, the modes of high-temperature combustion of combustible mixture compositions were determined with the establishment of the speed and thermometric parameters of the completeness of combustion of wind turbines.

Practical significance of the results obtained

High-energy systems are widely used in metallurgy in the energy sector, in particular, they are used to weld materials that cannot be welded by the classical method, stamping and cutting metals, strengthening steel structures, synthesizing diamonds from carbon, and much more.

The resulting systems based on aluminum diboride with high energy characteristics are an effective source of energy for equipment and new technologies.

The complexity of research work is ensured by the creation of waste-free domestic technology for the processing of mineral and technogenic raw materials and the synthesis of solid energy fuel.

Approbation of work

The materials of the dissertation work were reported and discussed at various international symposia and foreign conferences:

Collection of proceedings of the v international scientific conference “Modern problems of condensed matter physics, nanotechnology and nanomaterials” (Sarsembino Readings); Sat. IV International Scientific Conference of Students, Postgraduate Students and Young Scientists “Chemical Problems of Our Time”. – Donetsk, May 19-21. – 2020; Abstracts of the report. X International Symposium “Physics and Chemistry of Carbon and Nanoenergy Materials” Almaty, -2018; Republican conference "Science And Business", Almaty. – May 22, 2019; Collection of XI International symposium "Combustion and plasmochemistry". – 2019; Collection of proceedings of the international conference of students and young scientists "FARABI ALEMI", Almaty. – April 10, 2019.

Publications. As a result of research on the topic of the dissertation work, 21 scientific works were published, including: 2 articles included in the Scopus database, 2 articles in publications recommended by the Committee for Control in the Sphere of Education and Science of the Republic of Kazakhstan, 7 articles in collections of international symposiums and foreign conferences, 2 utility model

patents, 1 monograph, 1 copyright, 6 certificates of entering information into the state register of rights to objects protected by copyright.

Connection with research activities and government programs

This dissertation work was carried out within the framework of a grant funding program for scientific and scientific-technical projects for 2018-2020 on the topic: AR 05130784 “Synthesis of solid compounds with high energy characteristics.”

Scope and structure of work. The dissertation is presented in 104 pages and includes 42 figures and 42 tables. The work consists of an introduction, main part, conclusion and a list of sources used from 103 bibliographic references to domestic and foreign authors.

The author's personal contribution lies in setting up and conducting experiments, determining methods of analysis and ways to solve posed practical and theoretical problems, summarizing and interpreting the results obtained, writing articles and reports.