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# Low-temperature wave conversion of energetic compounds in filled condensed systems

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The features of flameless low-temperature wave conversion (LWC) of some energetic materials in filled systems were studied experimentally and analyzed theoretically. The results attest to an relay-race mechanism of process propagation in the initial composition. An important role of the filling material in stabilization of the LWC wave of the energetic component is revealed. In the presence of mono- or oligomeric binders, flameless LWC of the energetic component affords strong highly porous composite materials. The possibility of using this method for preparing new high-performance grinding materials and catalysts containing nano-sized particles of transition metals and their derivatives is demonstrated.

**Key words:** low-temperature wave conversion, filled condensed systems, energetic material, solid filler, polymeric binder, preparation, highly porous composite material, nano-sized metal particles, catalytic material, combustion.

The combustion of energetic condensed compounds and systems has been described quite comprehensively in books and periodicals.[1—4] However, one non-traditional and purposeless mode of organization of their conversion comprising introduction into the system of considerable amounts of filling compounds devoid of explosophore groups has been barely studied. Some features of this process were reported in publications,[5,6] which demonstrated the possibility of stable conversion of the proper energetic components (EC) in this type of systems. The conversion that propagates along the sample substantially differs from combustion of the same EC in an individual state by a number of characteristic features. First, this is the absence of a flame and relatively low temperatures in the reaction zone, low propagation velocity, spot propagation, a considerable amount of condensed combustion products, which preserve the initial sample shape almost without changes. Moreover, unlike the classical self-propagating high-temperature synthesis,[7] the propagation of this process is provided by the heat evolved during the EC conversion.

This work summarizes the results of studies of low-temperature wave conversion of filled energetic systems (LWCFES) considering both new and previously existing experimental data.

## Experimental

The energy-releasing components used included cellulose nitrate (CN) with a nitrogen content of 12.6% as spherical particles with an average size of 20—400 μm,[2] cyclotrimethylenetrinitramine (RDX), cyclotetramethylenetetranitramine (HMX) hexanitrohexaazaisowurtzitane (CL-20), and diethanolnitramine dinitrate (DINA) as 30—60 μm powders.

Powders of silicon carbide, boron nitride, alumina, silica, zinc oxide, sodium chloride, graphite, iron and nickel carbonates, *etc.*, of various nature and degree of dispersion served as ballasting fillers.

Hexamethylene diisocyanurate trimer (OI oligomer) containing 40% NCO groups,[8] which polymerized (cured) to give polyisocyanurate (PICU), and industrial epoxide resins ED-20 and ED-40 with polyethylenepolyamine or maleic anhydride serving as curing agents were mainly used as organic binders, which also had a filling effect. In addition, crystalline diisocyanates, namely, cyclohexyl diisocyanate (CHDI) with a melting point of 333 K and diphenylmethane diisocyanate (DPMDI) with a melting point of 311 K, were used.

The components of the initial mixtures were mixed in specified ratios at room temperature for ~10 min. The resulting blend was dead-pressed at ~30 MPa to form cylindrical samples of a specified diameter (*d*) and a height equal to ~(1.0—1.5)*d*. When organic binders were used, either the test samples were preliminarily cured at 340 K for 4 h or they were cured directly in the combustion wave. For studying armored samples, their lateral surface was coated by a composite based on a silicate glue and magnesium oxide mixture.

The fabricated samples were placed into a special ~310 cm$^3$ nitrogen-purged (some experiments were performed in air) combustion reactor. The combustion was initiated by applying a thermal pulse on the block end with a nichrome glow wire heated to ~750 K.

The linear combustion velocity was determined based on the migration of the combustion front, the moving boundary of condensed combustion products, which were most often almost

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black-colored (unlike the initial light-gray-colored sample), being taken as the combustion front.

In view of the considerable length of the reaction zone, the temperature in the zone was measured by a chromel alumel thermocouple of diameter 180 μm. The heat wave structure during the low-temperature combustion was studied using copper constantan thermocouples of diameter 30 μm mounted in the sample. The signal from the thermocouple was digitized by means of a L-780 digitizer (CJSC L-Card), recorded, and computer-treated by the PowerGraph 3.3.5 software (LLC DISoft). The accuracy of temperature and time measurements was ±5 K and ±0.1 s, respectively.

The effect of the external pressure on the combustion process was studied in a 2.5 dm$^3$ constant-pressure bomb under nitrogen at room temperature in the pressure range of 0.1—10 MPa. The rate of sample combustion under these conditions was determined from signals of two thermocouples located in the sample at a definite distance from each other.

The composition of the gaseous combustion products was studied using an MI-1201 mass spectrometer and a LKhM-2000 gas chromatograph. The experimental heats of combustion of energetic materials in the absence of oxygen were determined using an AKS-3M combustion calorimeter.

The condensed products of conversion were identified by powder X-ray diffraction as mineral oil mulls on an upgraded Dron-2M X-ray diffractometer. In some cases where the products contained iron derivatives, Mössbauer spectroscopy was used (Wissel spectrometer, Germany).

The morphology of the solid products formed during combustion was studied using an ISM-U3 scanning electron microscope (Japan) and their elemental composition was determined using an Idax-500 local electron probe X-ray microanalysis attachment.

The size of the formed particles of transition metals and transition metal derivatives was estimated by transmission electron microscopy (EM-304 electron microscope) using the SpectrMet 5.6 program for particle-size distribution analysis (CJSC MNPO Spektr). The formation of nano-sized products was also indicated by peak broadening in the X-ray diffraction patterns determined by the Debye—Scherrer method.[9]

## Results and Discussion

Studies of the effect of inorganic fillers with or without organic binders on the combustion process revealed the following key trends.[5,6,10] The variation of the nature and the content of inert fillers and the EC showed that development of the process depends first of all on their ratio and when the EC content is <10 wt.%, the process degenerates after initiation.

In the samples containing 10—20 wt.% of EC, flameless conversion of the EC accompanied by vigorous smoke formation and by frontal movement of the conversion zone along the sample is observed both under nitrogen and in air. If the content of EC (first of all, CN) exceeds 21—23 wt.%, ignition of the conversion products and carbonization of the polymer binder usually take place in air. Under nitrogen, the flameless conversion of EC occurs up to ~30—40%. The condensed products also preserve the shape of the initial sample.

It was shown that for most of the considered systems (Fig. 1, Table 1), the maximum temperature ($T_{max}$) in the flameless EC conversion front lies typically in a rather narrow range of 550—750 K, which is much lower than $T_{max}$ for individual EC (*cf.* ~1700 K for CN[1,3]).

Studies of the temperature profiles in the low-temperature conversion wave of filled energetic systems demonstrated that the length of zones in which the temperature increases from the initial to the maximum value can reach 10 mm, being much longer than these zones arising in the combustion of unfilled energetic materials (Fig. 2). This specific feature is apparently caused, on the one hand, by low front propagation velocity, which usually does not exceed 1 to 2 mm s$^{-1}$ under atmospheric pressure, and, on the other hand, by the porous framework of condensed products, which is retained in the conversion zone and preserves the heat within the zone.

Therefore, it is obvious that the nature of the filler should affect the conductive heat transfer and the macroscopic parameters of the LWCFES (see Fig. 1, *a*). However, in a more realistic situation, the structure of the tem-

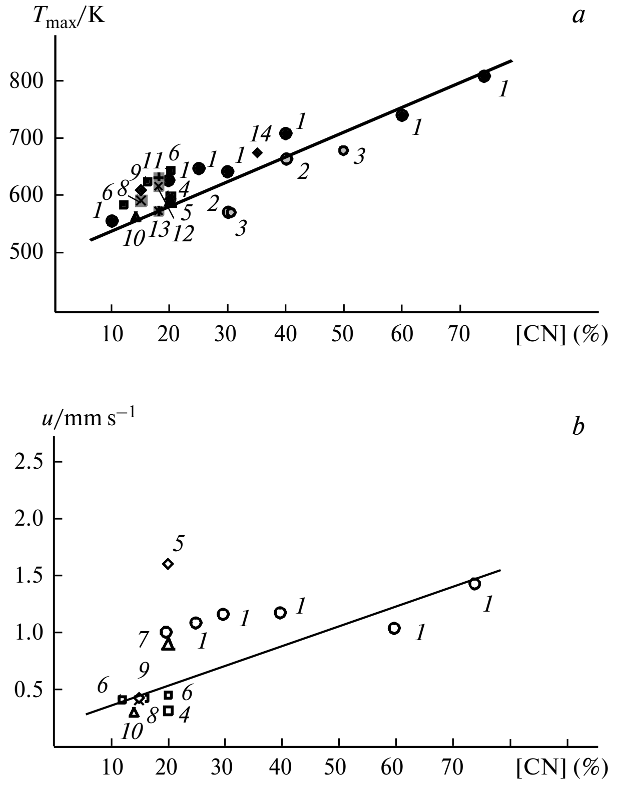

**Fig. 1.** Maximum temperature ($T_{max}$) (*a*) and propagation rate ($u$) (*b*) in the LWCFES zone *vs.* CN content in a OI mixture with SiC (*1*), MgO with armoring (*2*) and without armoring (*3*), with $Al_2O_3$ (*4*), with graphite (*5*), with NaCl (*6*), with BN (*7*), DINA mixed with NaCl (*8*), CN plasticized with 8% nitroglycerine (*9*), plasticized with 16% dibutyl phthalate (*10*), Cl-20 mixed with SiC (*11*), HMX with SiC (*12*), RDX with SiC (*13*), RDX with ZnO (*14*).

**Table 1.** Effect of the heat of combustion of the energetic component ($Q_c$) and specific heat of its conversion ($Q$/J (g of the mixture)$^{-1}$ in the filled energetic systems at a 14% content of the energetic component and 70% content of NaCl (the rest is OI) on the maximum temperature ($T_{max}$) and velocity of movement ($u$) of the LWCFES front

| Energetic component* | $Q_c$ | $Q$ | $u$ /mm s$^{-1}$ | $T_{max}$/K |
|---|---|---|---|---|
| | J g$^{-1}$ | | | |
| CN + 8% NG | 3700 | 518 | 0.42 | 608 |
| CN | 3198 | 447 | 0.42 | 593 |
| DINA | 2775 | 391 | 0.40 | 593 |
| CN + 11% DBP | 2416 | 339 | 0.38 | 588 |
| CN + 16% DBP | 1601 | 226 | 0.30 | 573 |

* NG is nitroglycerine, DBP is dibutyl phthalate.

perature wave is largely determined by secondary processes. For example, the conversion products of both EC and (in certain cases) fillers react with each other or chemical processes caused by the presence of reactive binder components develop in the wave front.

In some cases (mainly in CN-based filled systems), the temperature profiles had flat sections (plateaus) with slight change in the temperature, both in the presence and in the absence of reactive binder components of various nature and also in the case of preliminary curing of the binders. The main flat section occurs usually at temperatures somewhat below 200 °C. Its presence and length depend on the properties and the content of the inert and energetic fillers: the lower the fraction of the latter, the higher the probability that this section appears. The appearance of this sections was attributed[11] to the specific features of the thermal decomposition of the CN, which was reliably demonstrated[12,13] to be stepwise due to successive destruction of nitrate groups in the polymer elementary unit.

This is supported to some extent by a study of heat evolution kinetics during the decomposition of CN-based filled systems containing silicon carbide, zinc oxide, and polyisocyanurate on a differential automated calorimeter, which demonstrated[14] that the onset temperature of spontaneous decomposition does not depend on the component ratio and corresponds to the self-ignition temperature range of CN.[1] An estimation of the composition of the gaseous products of the LWCFES based on CN indicates[10] that it coincides with the products of CN thermolysis at elevated temperature,[12,13,15] which thus suggests that the physicochemical processes are the same in the two cases.

Without dwelling in more detail on the causes for the formation of the specific temperature profile in filler-diluted systems, note that they may be related,[11,14] in particular, to stepwise chemical conversion of EC (in particular, CN) under these specific conditions. Note also that in the case where reactive components of the binder that occur in the crystalline state before LCFES undergo a phase transition, the profiles have flat sections corresponding to the melting points.

Thus, LCFES is accompanied by a sharp (~300 K) temperature change in the reaction zone, which propagates along the sample at a velocity of 0.1 to 1.0 mm s$^{-1}$. Under these conditions, it is possible to implement the frontal polymerization of reactive binder components.[6] If the combustion wave is followed by the polymerization wave, the shape of the temperature profile depends also on the nature of reactive components of the binder. It was found that for polymerization in the combustion wave, the length of the isothermal plateau considerably decreases as compared with the case of preliminary curing of the composite and the plateau shifts to higher temperature. This is apparently due to the action of an additional source of heat evolution coming from exothermic polycyclotrimerization of diisocyanates, which develops most actively at elevated temperature. If diisocyanates with different reactivity are used, it can be seen (Fig. 3) that for CHDI, the plateau is retained (unlike DPMDI). This is attributable to the considerably higher reactivity of the aromatic DPMDI.[16]

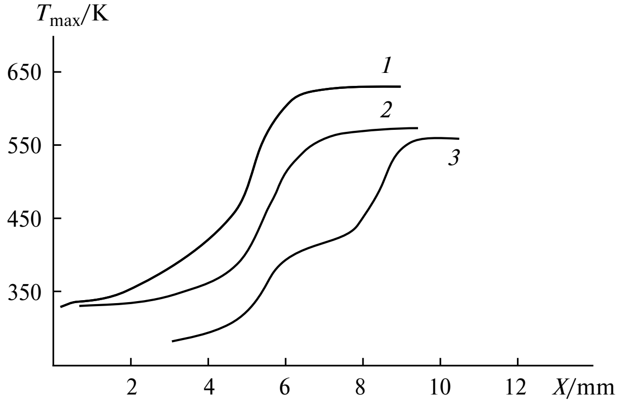

**Fig. 2.** Temperature profiles of LWCFES containing 20% Cl-20 (*1*), RDX (*2*) or CN (*3*), 16% PICU and silicon carbide.

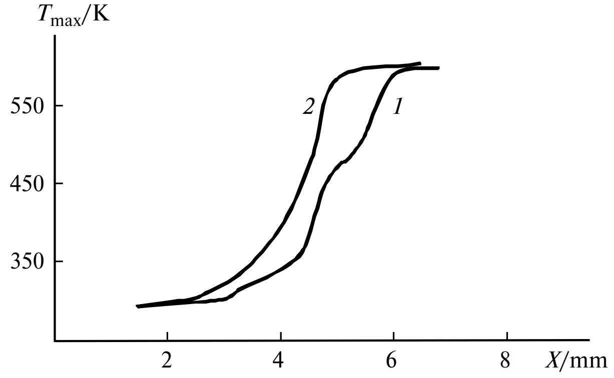

**Fig. 3.** Temperature profiles of LWCFES containing 16% CN, 16% uncured DPMDI (*1*) and CHDI (*2*) and silicon carbide.

Attention is drawn by the weak dependence of $T_{max}$ and the process rate on the heat of combustion of the energetic filler, which was changed by introducing nitroglycerine and dibutyl phthalate into the CN grains and by replacing CN by DINA (see Table 1).

Presumably, due to the ballasting action of the inert filler, under atmospheric pressure in the flameless mode, the gas phase reactions occur at low rates and are not completed. Hence, the major part of the evolved heat comes from the exothermic reactions that occur in the condensed and vapor-smoke-gas phases. Considering the views developed in the literature,[7] we estimated the parameter determining the possibility of degeneration of the steady-state autowave: the source energy should be ~250—290 J $g^{-1}$. This approximately corresponds to the experimentally determined lower limit of CN content at which combustion wave propagation still takes place.[6]

Apart from the effects of pressure and temperature, the effect of the geometric size (diameter) of the samples on the combustion rate and maximum temperature under normal conditions was considered.[10] A change in the diameter in the range of 10—25 mm almost did not affect the combustion parameters (Fig. 4). However, when the diameter of the block is <10 mm, the combustion of the mix becomes unsteady. In this case, after ignition, some samples burn completely and some other samples do not. As the sample diameter decreases, the deviations of the maximum temperature and the combustion rate (toward lower values) become more pronounced and the probability of the complete sample combustion decreases. A similar effect was observed in the combustion of ballistite powder near the critical diameter.[17] A possible explanation to this fact is the non-one-dimensional propagation of the lateral combustion waves in these systems or the spot, relay-race mechanism of combustion in heterogeneous mixtures.[4,18]

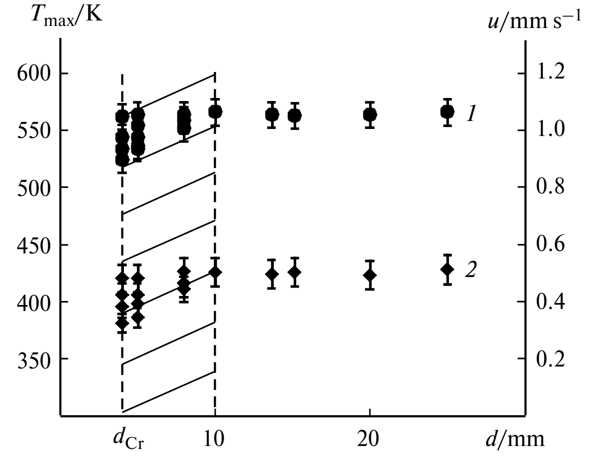

**Fig. 4.** Maximum temperature ($T_{max}$, *1*) and linear velocity ($u$, *2*) of the LWCFES for a mixture comprising CN (16%), oligomer (14%), and silicon carbide (70%) *vs.* sample diameter ($d$). The unsteady combustion area is marked.

Under these conditions, the percolation propagation of the chemical transformation wave occurs through chains (clusters) of the neighboring particles of the energetic material.[19]

Armoring of the sample lateral surface does not change considerably the sample combustion parameters. The maximum temperature and linear velocity of the combustion do not depend on the sample diameter. Only a slight (20—30 K) increase in the maximum temperature is observed. Typically, the critical combustion diameter is ~8 mm in this case, and no unsteady combustion modes are observed. This may be due to more stable heat exchange conditions on the armored sample surface.

The effect of the external conditions on the wave conversion of CN as a part of filled system was studied by measuring the influence of the initial temperature ($T_0$) in the range of 293—353 K and the external pressure ($P$) in the range from atmospheric pressure to 10 MPa. The dependences of the combustion parameters ($T_{max}$ and the front velocity)[10] on the initial temperature indicate a regular increase. The combustion rate increases ~1.7-fold in this temperature range, which corresponds to a temperature coefficient of the combustion rate, which characterizes the change in the combustion rate upon a temperature change by 1 K, $\beta = 13 \cdot 10^{-3}$ $K^{-1}$. Note that nitro-cellulose powders, which are composed almost exclusively of pure cellulose nitrate, have somewhat lower temperature coefficient of ~$5 \cdot 10^{-3}$ $K^{-1}$.[2] Thus, the maximum temperature in the combustion front also grows. This trend can be interpreted by considering the fact that the heat lost for heating the filler in the composite is essentially counterbalanced by external pre-heating of the system.

It was also found that the power law of combustion[1—4]

$$u = u_1 P^{\nu} \qquad (1)$$

($u_1$ is the combustion rate at a pressure of 1 MPa) adequately describes the combustion of compositions with various CN and inert filler (silicon carbide) contents at ambient temperature (Table 2).

It follows from the obtained data that the exponent in the combustion law remains invariable as the CN content varies. Its value ($\nu = 0.23$) is several times lower than this parameter in the combustion law for pure CN, which is close to unity.[3] As the CN content in the initial composition increases, only the coefficient of the initial combustion rate $u_1$ somewhat increases.

The effect of pressure on the LWCFES, which was studied for porous CN-containing composites, deserves special mention. It was found that at a particular external pressure, the process propagation velocity increases jumpwise. The higher the sample porosity, the lower the critical pressure where the high-rate conversion mode starts (Figs 5 and 6) The appearance of the low-temperature high-rate mode is apparently due to the convective heat transfer over the porous matrix of the initial material. Note that

**Table 2.** Front propagation velocity ($u$) and parameters of combustion law (1)* in the pressure range of 0.1—10 MPa for the LCFES based on CN, silicon carbide and polyisocyanurate at a polyisocyanurate content of 16%

| [CN] (wt.%) | $u_1$ | $P$ /MPa | $u$ /mm s$^{-1}$ |
|---|---|---|---|
| 18 | 1.44 | 9.7 | 2.51 |
| | | 4.2 | 2.07 |
| | | 1.7 | 1.51 |
| | | 0.7 | 1.32 |
| | | 0.1 | 0.87 |
| 20 | 1.55 | 10.1 | 2.68 |
| | | 4.6 | 2.23 |
| | | 1.6 | 1.63 |
| | | 1.0 | 1.50 |
| | | 0.1 | 0.95 |
| 22 | 1.72 | 9.9 | 2.92 |
| | | 5.1 | 2.45 |
| | | 3.1 | 2.35 |
| | | 1.6 | 1.82 |
| | | 0.1 | 1.03 |

* $\nu = 0.23$.

the initial rate present in the combustion law increases more than 40-fold above the critical pressure, while the exponent becomes considerably lower than that for the convective combustion.[20]

The fundamental results of the studies of LCFES, the established process regularities for the condensed phase, and the currently known useful properties of the condensed products of the conversion that may be of practical value provided the formulation of new approaches to the preparation of composite materials with specified functional properties. In particular, the products of LCFES containing widely known superhard fillers (silicon carbide, alumina, industrial grade diamonds) were tested as polishing materials for the finishing treatment of various metals and alloys ranging from tempered steel to brass. One version of the porous material structure (Fig. 7) that can be controlled, in particular, by using polydisperse EC ensures high-performance high-quality surface treatment to a microroughness level of 0.02—0.04 μm and more.

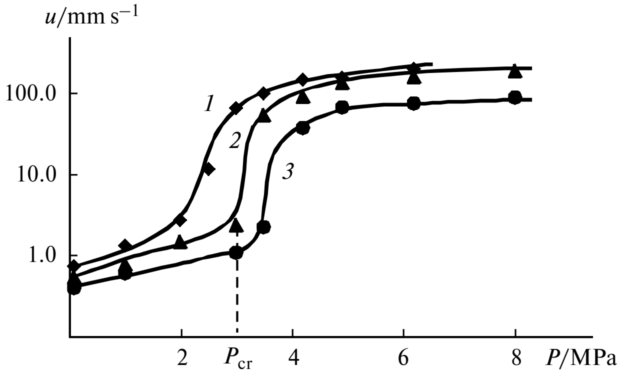

**Fig. 5.** Linear velocity of LWCFES ($u$) for a mixture comprising 40% CN, 22% PICU, and nickel carbonate *vs.* external pressure ($P$) for sample porosity of 39 (*1*), 29 (*2*), 12% (*3*); $P_{cr}$ is the critical value.

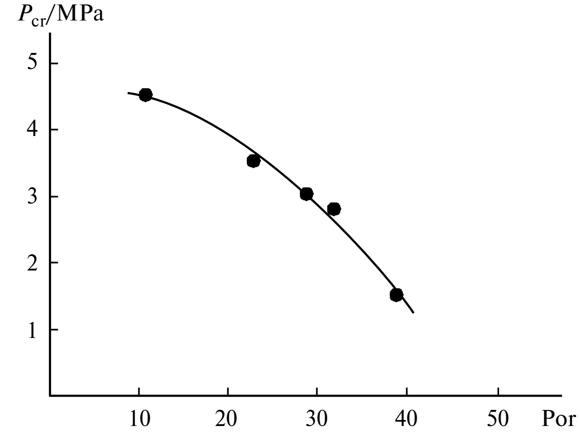

**Fig. 6.** Critical external pressure ($P_{cr}$) for the switching of the LWCFES for a mixture comprising 40% CN, 22% PICU and nickel carbonate to high-rate mode *vs.* sample porosity (Por).

It became possible to design new catalytic materials by targeted action on the structure, composition, and properties of the condensed products of LWCFES. The catalytic systems manufactured in this way are composite materials filled by nano-sized particles of various (depending on the nature of precursors) transition d metals and their derivatives. This is exemplified by the scanning electron microscopy data demonstrating the macrostructure of the

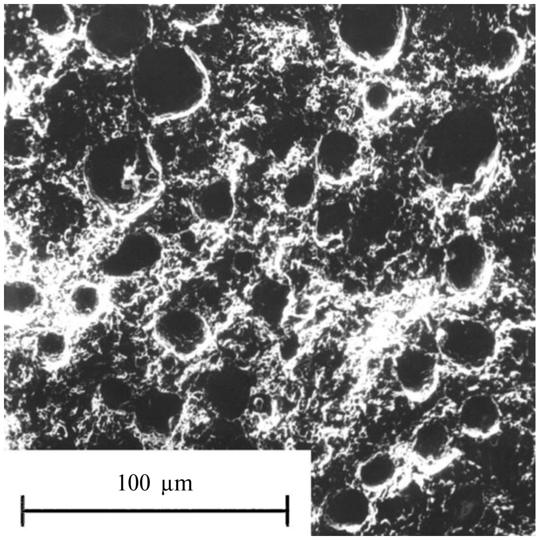

**Fig. 7.** Macroporous structure of the condensed products of the LCFES based on a mixture of CN, PIO, and corundum spherical particles.

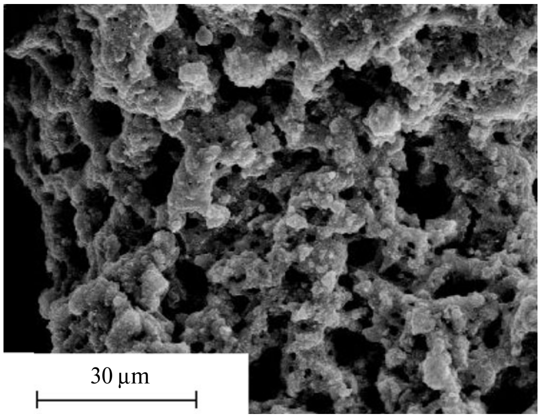

**Fig. 8.** Microporous structure of the condensed products of the LCFES based on RDX.

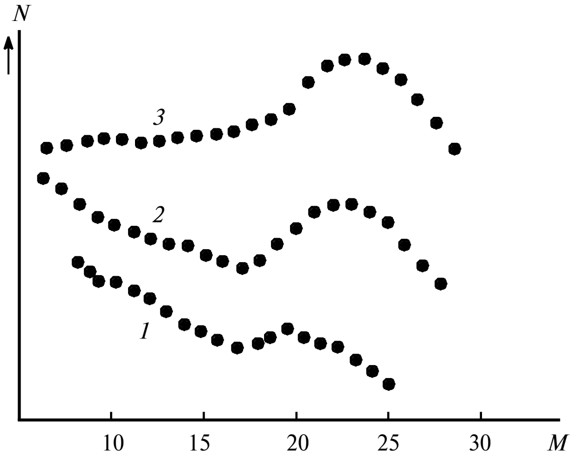

**Fig. 10.** Molecular-weight distribution of saturated hydrocarbons obtained in the presence of the composite material containing nano-sized iron and cobalt particles: (*1*) 19.6% Fe, (*2*) 32.5% Fe, (*3*) 44.0% Fe.

iron-containing condensed products of LWCFES and the nano-sized iron particles isolated therefrom (Figs 8 and 9).

A distinctive feature of the obtained materials filled by nano-sized metal particles is the absence of pyrophoric properties, which substantially simplifies their use, as this eliminates the stage of passivation of the manufactured catalysts and their recovery prior to use.

The unique combination of the highly porous structure of the new catalytic materials and the possibility of formation of nano-sized particles of transition metals or their compounds opens up prospects for the use of these compounds in various organic and petrochemical synthetic processes. In particular, the estimation of the catalytic activity of the products containing nano-sized iron and cobalt particles in the Fischer—Tropsch synthesis showed that depending on the conditions, the reaction may give hydrocarbons with different molecular weights in relatively high yields (Fig. 10). It was also found that unlike their analogs, the novel catalysts provide higher content of alcohols in the products along with hydrocarbons.

Testing of the LCFES products of deliberately formed compositions in the hydrogenation of several types of compounds also showed their high efficiency. For example, in the selective hydrogenation of vinylacetylene, the catalytic system containing nano-sized nickel particles (Fig. 11) showed activity comparable with the activity of commercial palladium catalyst (Fig. 12).

Thus, it can be stated that new composite materials containing nano-sized particles of transition metals and their compounds have considerable advantages over the existing catalysts. The use of energetic compounds to be disposed markedly reduces the prime cost of the production of new catalysts.

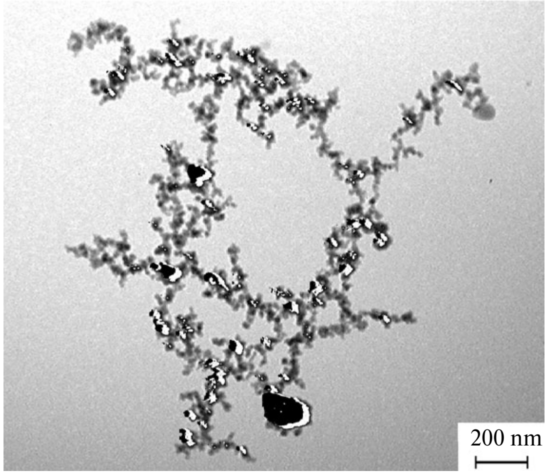

**Fig. 9.** Iron nanoparticles isolated from the products of LCFES based on RDX.

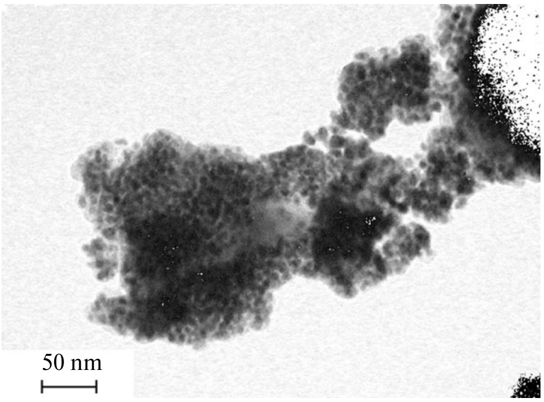

**Fig. 11.** Nickel nanoparticles isolated from the products of LCFES based on RDX.

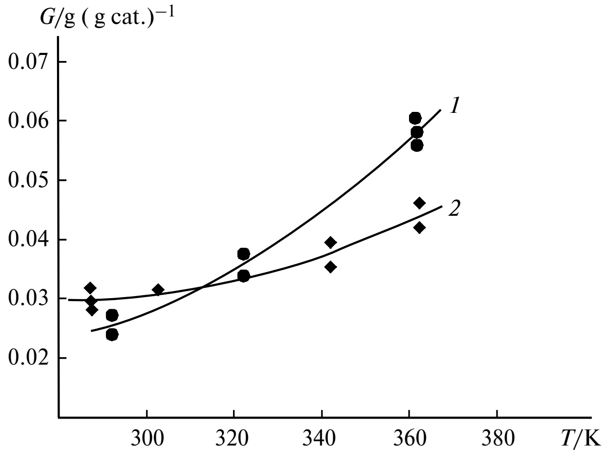

**Fig. 12.** Temperature dependence on the degree of conversion of vinylacetylene to 1,4-butadiene ($G$) for the nickel-containing catalytic material based on the condensed products of LCFES (1.6% Ni, $H_2$ : vinylacetylene molar ratio of 10 : 1) (*1*) in comparison with the commercial palladium catalyst (0.25% Pd, $H_2$ : vinylacetylene molar ratio 5 : 1) (*2*).

It was shown experimentally that the flameless propagation of the LWCFES wave is largely due to the heat that is evolved in the processes taking place mainly in the condensed and vapor-smoke-gas phases of the energetic compounds during decomposition.

The thermal insulation of the reaction zone by the filler matrix, which is retained after the conversion front has passed stabilizes the process. The heat content of the EC, the initial temperature of the nonporous filled energetic systems, and the external pressure affect only slightly the maximum temperature in the front and the wave propagation velocity.

It was found that under certain conditions, depending on the chemical nature of the components that constitute the filled energetic condensed systems, it is possible to deliberately obtain highly porous composite materials of various composition in their low-temperature transformation wave, in particular materials containing nano-sized particles of transition metals and their compounds. For a number of systems containing monomeric and oligomeric reactive components (diisocyanates and the derivatives, epoxy resins, and various curing agents.), it was also shown that a polymer matrix may form in the LCFES thermal wave in the frontal mode.

The results are presented, indicating that LWCFES can be used as the foundation of new technology that implies the use of condensed products of conversion as new functional components and materials for various purposes, for example, highly efficient catalysts, abrasive materials, magnetic liquids, *etc*.

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