ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)

ҚАЗАҚСТАН РЕСПУБЛИКАСЫ
ҰЛТТЫҚ ҒЫЛЫМ АКАДЕМИЯСЫНЫҢ

Д.В.СОКОЛЬСКИЙ АТЫНДАҒЫ «ЖАНАРМАЙ,
КАТАЛИЗ ЖӘНЕ ЭЛЕКТРОХИМИЯ ИНСТИТУТЫ» АҚ

Х А Б А Р Л А Р Ы

ИЗВЕСТИЯ

НАЦИОНАЛЬНОЙ АКАДЕМИИ НАУК
РЕСПУБЛИКИ КАЗАХСТАН

АО «ИНСТИТУТ ТОПЛИВА, КАТАЛИЗА И
ЭЛЕКТРОХИМИИ ИМ. Д.В. СОКОЛЬСКОГО»

N E W S

OF THE ACADEMY OF SCIENCES
OF THE REPUBLIC OF KAZAKHSTAN

JSC «D.V. SOKOLSKY INSTITUTE OF FUEL,
CATALYSIS AND ELECTROCHEMISTRY»

ХИМИЯ ЖӘНЕ ТЕХНОЛОГИЯ СЕРИЯСЫ

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СЕРИЯ ХИМИИ И ТЕХНОЛОГИИ

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SERIES CHEMISTRY AND TECHNOLOGY

4 (430)

ШІЛДЕ – ТАМЫЗ 2018 ж.
ИЮЛЬ – АВГУСТ 2018 г.
JULY-AUGUST 2018

1947 ЖЫЛДЫҢ ҚАҢТАР АЙЫНАН ШЫҒА БАСТАҒАН
ИЗДАЕТСЯ С ЯНВАРЯ 1947 ГОДА
PUBLISHED SINCE JANUARY 1947

ЖЫЛЫНА 6 РЕТ ШЫҒАДЫ
ВЫХОДИТ 6 РАЗ В ГОД
PUBLISHED 6 TIMES A YEAR

АЛМАТЫ, ҚР ҰҒА АЛМАТЫ, НАН РК ALMATY, NAS RK

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«ҚР ҰҒА Хабарлары. Химия және технология сериясы».
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)
Меншіктенуші: «Қазақстан Республикасының Ұлттық ғылым академиясы» Республикалық қоғамдық бірлестігі (Алматы қ.)
Қазақстан республикасының Мәдениет пен ақпарат министрлігінің Ақпарат және мұрағат комитетінде 30.04.2010 ж. берілген **№1089-Ж** мерзімдік басылым тіркеуіне қойылу туралы куәлік

Мерзімділігі: жылына 6 рет.
Тиражы: 300 дана.

Редакцияның мекенжайы: 050010, Алматы қ., Шевченко көш., 28, 219 бөл., 220, тел.: 272-13-19, 272-13-18, www:nauka-nanrk.kz / chemistry-technology.kz

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Типографияның мекенжайы: «Аруна» ЖК, Алматы қ., Муратбаева көш., 75.

«Известия НАН РК. Серия химии и технологии».
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)

Собственник: Республиканское общественное объединение «Национальная академия наук Республики Казахстан» (г. Алматы)

Свидетельство о постановке на учет периодического печатного издания в Комитете информации и архивов Министерства культуры и информации Республики Казахстан **№10893-Ж,** выданное 30.04.2010 г.

Периодичность: 6 раз в год
Тираж: 300 экземпляров

Адрес редакции: 050010, г. Алматы, ул. Шевченко, 28, ком. 219, 220, тел. 272-13-19, 272-13-18,
http://nauka-nanrk.kz / chemistry-technology.kz

Адрес редакции: 050100, г. Алматы, ул. Кунаева, 142,
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Адрес типографии: ИП «Аруна», г. Алматы, ул. Муратбаева, 75

News of the National Academy of Sciences of the Republic of Kazakhstan. Series of chemistry and technology.
ISSN 2518-1491 (Online),
ISSN 2224-5286 (Print)
Owner: RPA "National Academy of Sciences of the Republic of Kazakhstan" (Almaty)
The certificate of registration of a periodic printed publication in the Committee of Information and Archives of the Ministry of Culture and Information of the Republic of Kazakhstan N 10893-Ж, issued 30.04.2010

Periodicity: 6 times a year
Circulation: 300 copies

Editorial address: 28, Shevchenko str., of. 219, 220, Almaty, 050010, tel. 272-13-19, 272-13-18,
http://nauka-nanrk.kz / chemistry-technology.kz

Editorial address: Institute of Organic Catalysis and Electrochemistry named after D. V. Sokolsky
142, Kunayev str., of. 310, Almaty, 050100, tel. 291-62-80, fax 291-57-22,
e-mail: orgcat@nursat.kz

Address of printing house: ST "Aruna", 75, Muratbayev str, Almaty

N E W S
OF THE NATIONAL ACADEMY OF SCIENCES OF THE REPUBLIC OF KAZAKHSTAN
SERIES CHEMISTRY AND TECHNOLOGY
ISSN 2224-5286
Volume 4, Number 430 (2018), 71 – 79

UDC: 541.124.16

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MODIFICATION OF THE SURFACE OF ALUMINUM AND MAGNESIUM PARTICLES UNDER THE CONDITIONS OF MECHANOCHEMICAL TREATMENT AS A METHOD OF OBTAINING ENERGY-INTENSIVE COMPOSITIONS

Abstract. The paper presents the results of a mechanical treatment of metal powders (aluminum brand PA-4 and magnesium brand MPF-3) in a dynamic action mill using graphite as a surfactant additive in order to improve the dispersion of powders and modify the surface layer of particles. The mechanical treatment of metals, with graphite, contributes to the change in the structure, the composition of the surface of metal particles, an increase in the proportion of the active metal, and the formation of an organic coating of dispersible particles. The obtained metal particles with graphite were studied by physicochemical analysis methods, a granulometric method for estimating the particle size distribution carried out on the instrument Malvern 3600E. The effect of mechanochemical treatment of metal powders on the process of technological combustion of thermite mixtures is investigated. The results of the study showed that after the machining, the particle size of the metal powders decreases and, as a consequence, the specific surface area of the metal particles increases with the accumulation of defects in the crystal lattice. In the process of mechanochemical treatment, the size of the crystallites depending on the mass of the fraction of graphite used in the composition of the Me/C composite. When using aluminum and magnesium as a fuel component after mechanochemical treatment in the presence of graphite, the thermal kinetic characteristics of the combustion process increase.

Key words: mechanochemical treatment, aluminum, magnesium, modification, technological combustion.

Introduction. Metal powders are one of the most important components of fuels of various compositions and purpose. Their use is primarily due to the high thermal effect of oxidation of the metal, as well as the decrease in the average molecular weight of the gaseous combustion products as a result of deoxidation of H_2O and CO_2 during their interaction with the metal [1]. This is especially important for hydro-reacting fuel systems, in which the metal contains up to 80% and it is the main fuel [2-4]. The most common and quite energy-intensive metal fuel for fuel systems for various purposes is aluminum. In some fuels, especially ballistite, aluminum particles, because of the low oxidative activity of oxygen-containing combustion products, ignite with a large delay in time. In such cases, magnesium or its alloys with aluminum are used, the particles of which ignite faster than aluminum and burn completely [1, 3]. The most important characteristic of metallic powders, when used in combustible mixtures, is the content of the active (non-oxidized) metal, as well as the size and shape of the particles. In most cases, ultrafine powders with a particle size of less than 1 μm are used. And in recent years, more attention has been paid to nanodispersed powders, since they are characterized by increased chemical activity, this allowing to increase the burning rate of fuel [5-7].

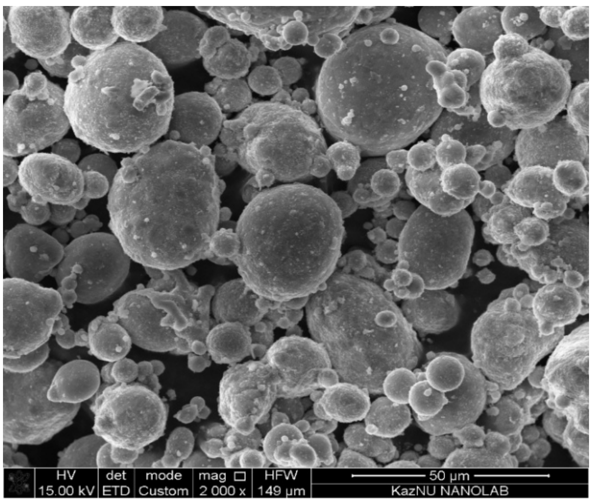
To ensure the stability of the properties of metallic powders and to maintain the active metal content, they are passivated and hydrophobized [8]. In the first case, a solid and strong oxide-hydroxide film is formed on the surface of the particles preventing interaction of the metal with the oxidizing medium. And in the second, the surface of particles is covered with a layer of a fatty acid salt, in particular, sodium stearate. However, the presence of an oxide-hydroxide film on the surface of particles firstly reduces the proportion of the active metal, and secondly, the ignition begins only from the moment of contact of the fuel with the oxidizer as a result of the cracking of the oxide film by the volumetric expansion of the molten metal inside the oxide capsule.

To a large extent, the state of metal particles, in particular aluminum and magnesium, is primarily modified in terms of increasing the proportion of the active metal and provides resistance to the external oxidizing medium, as well as to increase the activity of combustion in the composition of combustible mixtures, using mechanochemical treatment (MCT) of the powder in planetary centrifugal mills. In the mechanochemical treatment with various organic modifiers, the fraction of the oxide film of the particles can be reduced to a considerable extent in the process of grinding the powder, replacing it with an organic one. As was shown in [9, 10], as a result of MCT of aluminum with graphite in an inert atmosphere, the reactivity of aluminum increases substantially, and in the first stages of processing a homogeneous composite product Al/C is formed, in which fine-dispersed aluminum particles are stabilized in highly dispersed graphite. With prolonged mechanical treatment, there takes place chemical interaction of aluminum with carbon with the formation of the crystal phase of Al_4C_3 [10]. Much attention is also paid to the possibility of mechanical activation of magnesium [11].

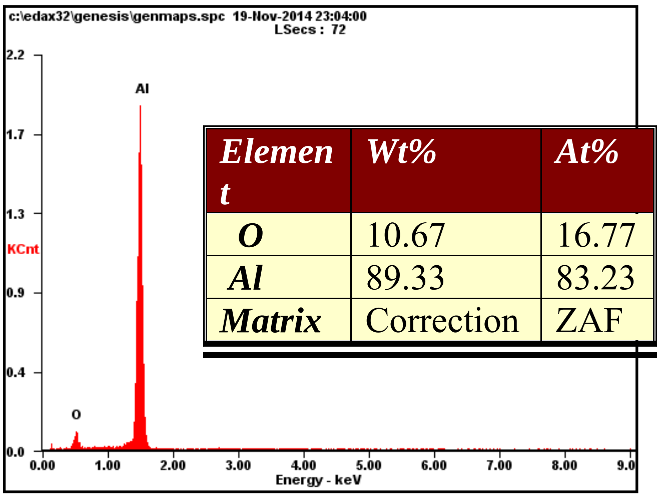
To obtain highly dispersed metal particles of aluminum and magnesium with a modified particle surface, it is important to select the optimum MCT conditions for a particular modifying additive. In this paper, we present the results and a comparative analysis of the MCT studies of aluminum and magnesium in the presence of graphite.

Results and discussion. For the experiments we used aluminum PA-4 and magnesium powder of the brand MPF-3. The microstructure of the initial particles of powdered aluminum and magnesium was investigated. According to the results of microstructural analysis, PA-4 aluminum particles have a spherical shape with a size of 20 to 63 microns (figure 1 a, b).The specific surface of such samples, according to the results of the BET analysis, is 3.692 m^2/g. The energy dispersive spectrum showed that in the composition of the initial aluminum grade PA-4, the mass fraction of oxygen is more than 10%. The presence of oxygen atoms indicates the presence of a sufficiently dense layer of oxide film on the surface of particles.

The results of the microstructural analysis of the original MPF-3 magnesium powder showed (figure 1c, d) that magnesium particles have a scaly form and the average particle size of the sample exceeds 200 µm, with a flake thickness of about 20 µm. The specific surface of such samples, according to the results of the BET method, is 0.181 m^2/g. The results of EDX analysis show the presence of 2.26% oxygen in magnesium, i.e. the presence of oxide film on the surface of particles. However, the X-ray phase analysis of initial magnesium of MPF-3 brand showed that it contains 9.6% Mg $(OH)_2$, i.e. the surface of the particles is covered with a hydroxide film.

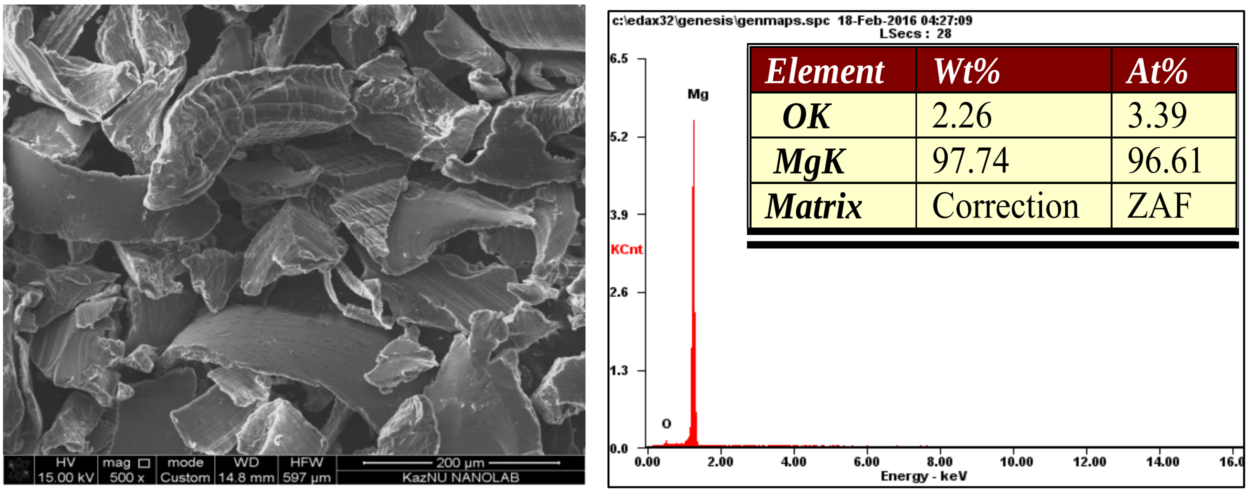


a



Element	Wt%	At%
O	10.67	16.77
Al	89.33	83.23
Matrix	Correction	ZAF

b



c d

Figure1 - Electron-microscopic images (a, c), the energy-dispersive spectrum and the mass fraction of the elements (b, d) of the original aluminum powder PA-4 (a, b) and magnesium MPF-3 (c, d)

Mechanical grinding of Al and Mg particles is difficult due to their plasticity. To facilitate the dispersion process, surfactants, for example, stearic acid, graphite and other organic compounds, are added. Thus, when processing aluminum with graphite additives, the dispersing process is facilitated, and the presence of graphite in a mixture with the metal is a positive factor for subsequent targeted use, for example, in the composition of energy condensed systems [12-17].Thus, modification of the surface of metallic nanoparticles by graphite during MCT is carried out not only to protect the metal from oxidation, but also to increase the energy content of the obtained composite mixture.

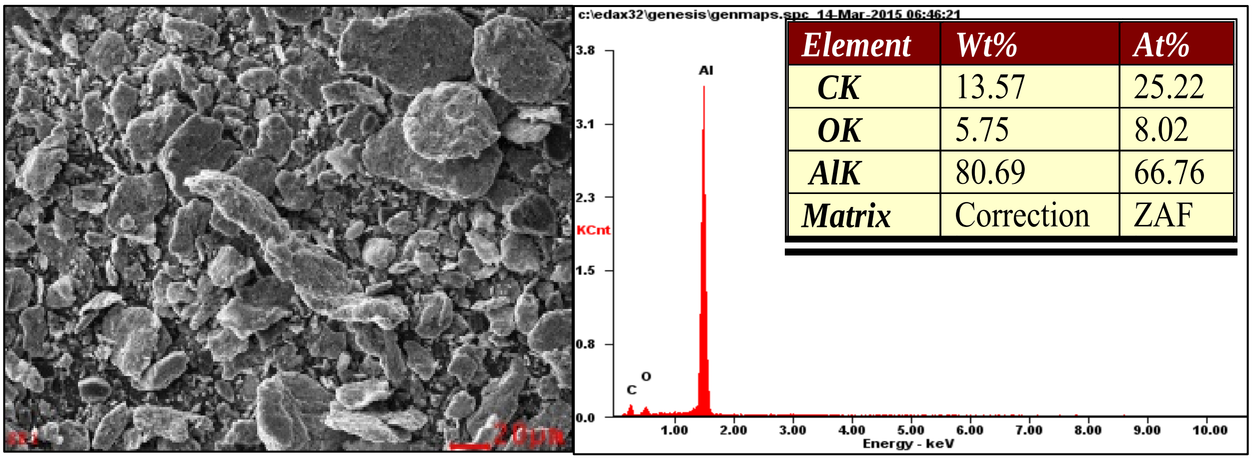
Mechanochemical treatment of powders was carried out in the centrifugal planetary mill Pulverzette 5 (manufactured by FRITSCH) with the volume of each working chamber of 500 mm^3, the rotation speed of the platform is 400 rpm, the acceleration of the movement of grinding balls 40 g, the energy consumption 1.5 kW/h. Mechanochemical treatment was carried out in an air atmosphere at a powder/ball ratio (M_P/M_B) = 1/4. When grinding, the amount of the modifying additive varied (5-20%). The processing time was not more than 20 minutes to exclude self-ignition. The choice of the optimal time for the MCT was due to the results of previous studies [18,19]. To prevent oxidation of aluminum particles by air oxygen after MCT and to assess the changes actually associated with mechanical action, samples of the dispersed mixture were passivated with hexane (C_6H_{14}).

After MCT of aluminum with graphite, the particles have a plate (scaly) shape of different thickness, i.e. in the process of grinding, the shape of the particles changes and the formation of the layer structure of the Al/C composite occurs (figure 2 a).

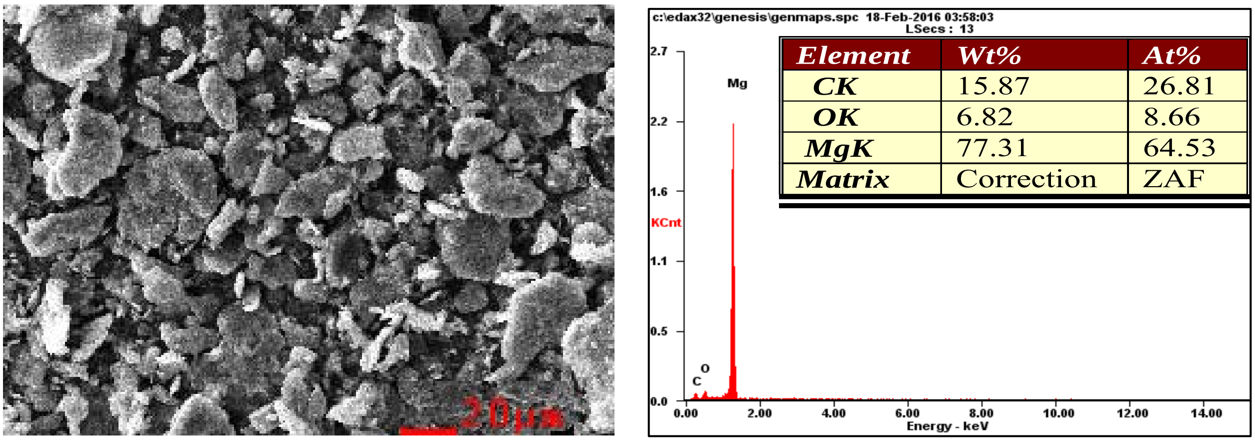
The specific surface area of the powders, which was determined by the BET method, increases substantially after the MCT. Thus, the specific surface area of the treated mixture (Al 80% + C 20%) increases to 9.554 m^2/g according to BET analysis. The state of the surface layer also changes. Elemental analysis of the composite (Al 80% + C 20%) after MCT showed that the mass fraction of aluminum in the composite is 80.69%, that of carbon 13.57% of the total mass of the sample, and the amount of oxygen is 5.75% (figure 2 b). Consequently, in the aluminum-graphite MCT process, aluminum is partially reduced in the surface oxide layer of the particles and the oxygen content in the composite decreases.

As a result, MCT of magnesium with graphite, the particles retain a plate-like shape (figure 2 c). The specific surface for the composite particles (Mg 80% + C 20%) increases to 16.383 m^2/g. The EDX analysis of the elemental composition of the Mg/C composites showed that the mass fraction of oxygen atoms increases after MCT, so for Mg 80% + C 20% it is more than 6% (figure 2d).

Consequently, on the surface of magnesium particles after MCT, the thickness of the oxide layer increases.However, based on the results of X-ray phase analysis, neither oxides nor hydroxides are formed on the surface of the particles, the amount of which can reach 15% (figure 3).



(Al 80%+C20%) a b



(Al 80%+C20%) c d

Figure 2 - Electron-microscopic images (a, c), the energy-dispersive spectrum and the mass fraction of elements (b, d) in the composite (Al 80% + C 20%) and (Mg 80% + C 20%) after 20 minutes of MCT

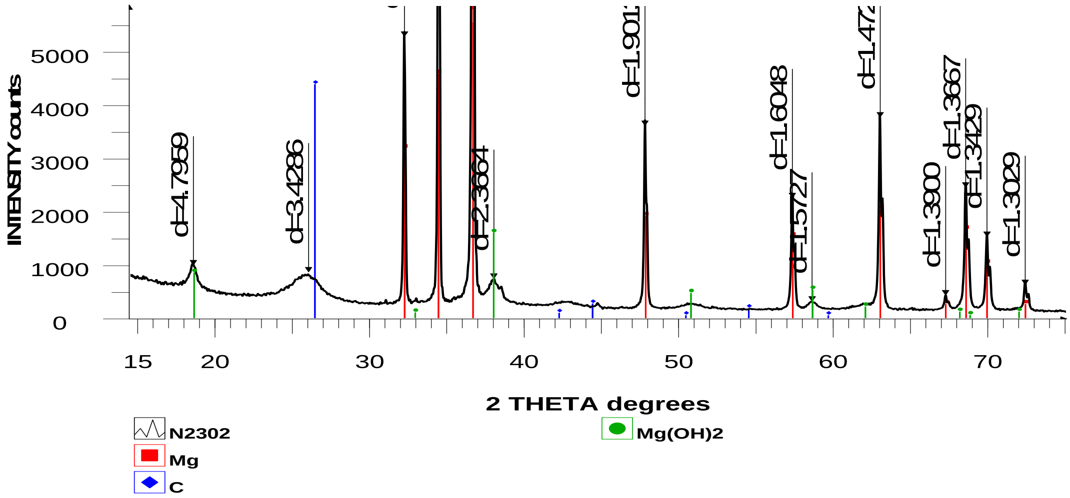


Figure 3- Diffractogram of the sample (Mg 80% + C 20%), after 20 minutes of MCT

Evaluation of the particle size distribution carried out on the Malvern 3600E showed that when the graphite content in the aluminum system increases to 15-20% after grinding, the bulk of the powder has a particle size of less than 5 μm. Almost half of them have a size of less than 2 μm (figure 4 a) resulting in the increase in the specific surface area of the aluminum particles of grade PA-4 from 3.7 to 9.5 m^2/g.

After grinding magnesium in a mixture with graphite, the bulk of the powder of the Mg/C mixture has a particle size of less than 5 μm, which are practically agglomerates of nanosized particles (figure4 b).

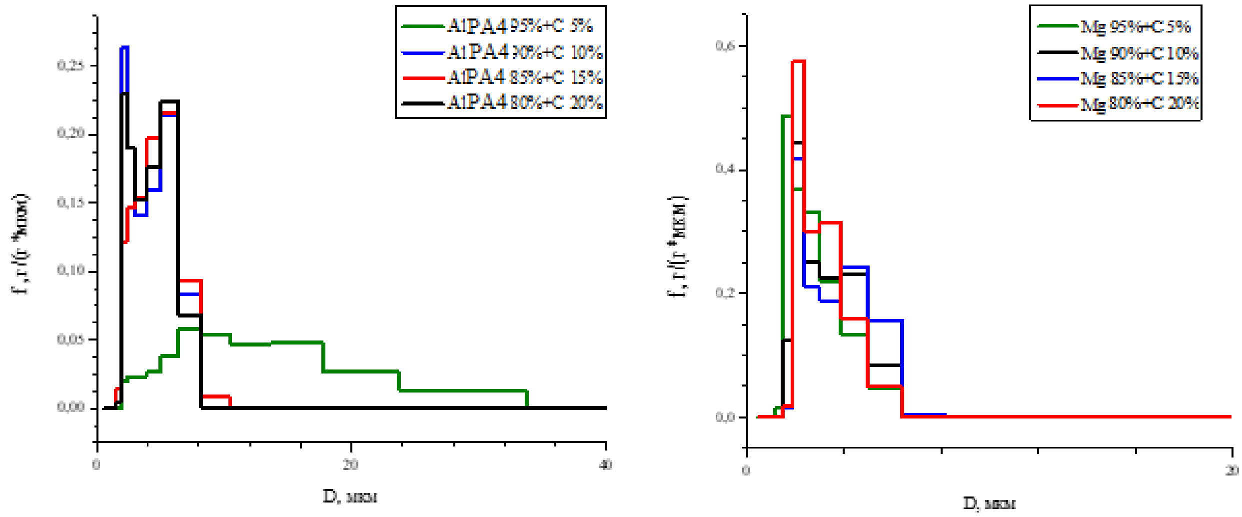


Figure 4 - The mass distribution of the Al/C (a) composite particle and the Mg/C (b) composite after 20 minutes of MCT

To evaluate the substructure features of aluminum particles after MCT, crystallite sizes were measured by the XRD method in the obtained Al/C, Mg/C composites. According to the results of the analysis, in the process of mechanochemical treatment, the size of the crystallites varies from the amount of the modifier used (table 1).

Table 1 - The size of aluminum and magnesium crystallites after 20 minutes of MCT with graphite

The content of graphite in composites	The size of crystallitesL, Å	
	Al	Mg
-	690	580
5 % C	560	600
10 % C	490	770
15%C	440	590
20 % C	410	520

With mechanical action, both accumulation and redistribution of defects over the volume of the particle takes place. As a result of MCT aluminum with graphite, the size of crystallites decreases and the content of carbon increases in the Al/C composite. During MCT of magnesium with graphite, at first there proceeds growth of crystallites and at a carbon content of 15-20%, the size of the crystallites, decrease i.e. there takes place more intensive accumulation of defects in the volume of grains. This may be due to the fact that during MCT carbon atoms penetrate into the grain of the aluminum particle and together with the defects diffuse by its volume under the action of mechanical stresses. In some cases, this process is likely to contribute to stabilization of defects, in other cases, it transfers them to the particle grain boundary and, as a consequence, the size of crystallites grows[20]. The surface film of particles of both aluminum and magnesium is destroyed (loosened) and saturated with highly disperse carbon particles (figure 5).

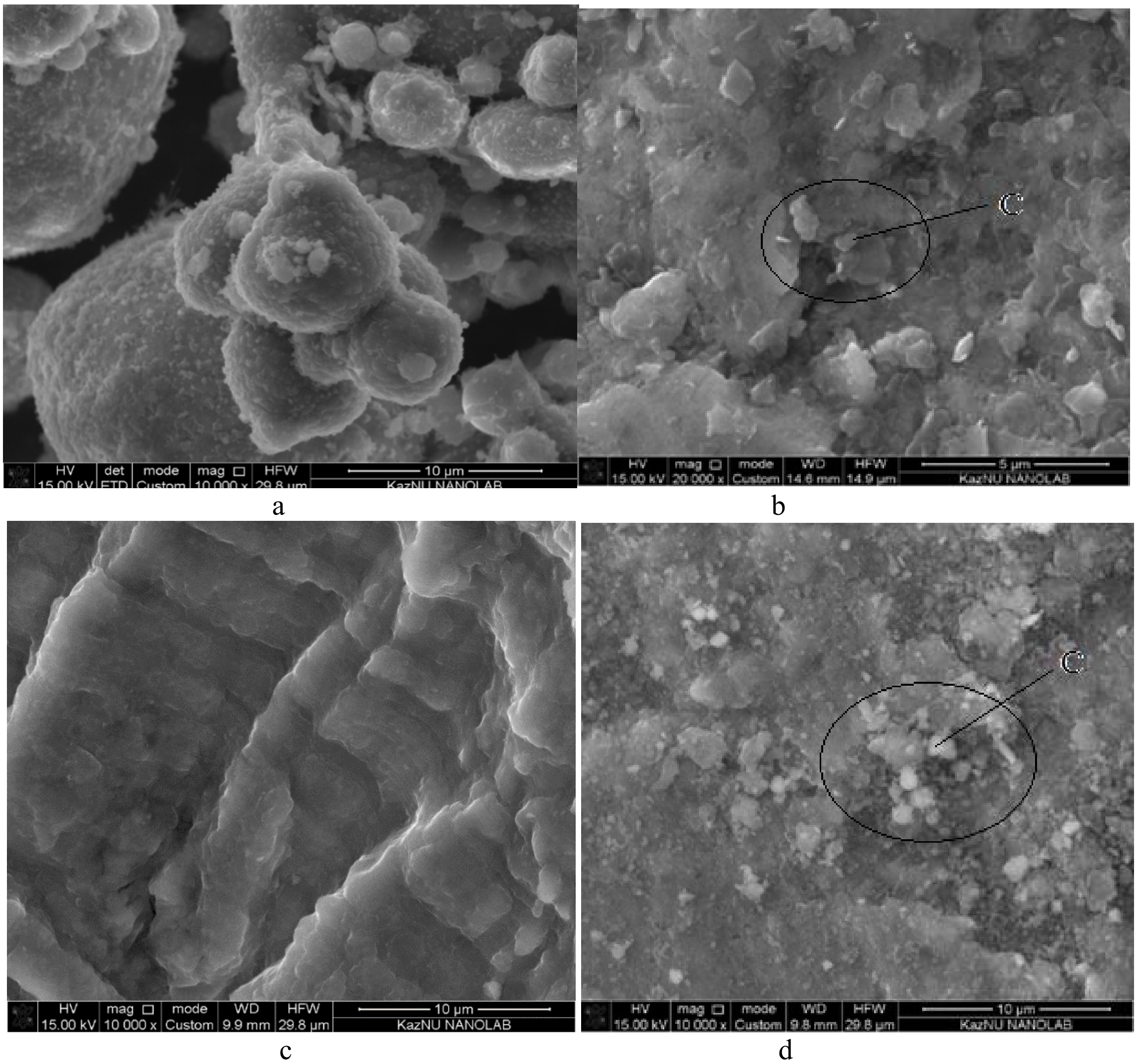


Figure 5 - Electron-microscopic images of aluminum and magnesium particles in the initial state (a, c) and in the composite (Al 80% + C 20%) and (Mg 80% + C 20%) after 20 minutes of MCT (b, d)

Thus, the use of graphite during MCT of aluminum and magnesium, according to all the characteristics analyzed, contributes to a change in the morphology and structure of the particles of the formedmetal/carbon composites (Me/C). The observed changes in the size of aluminum and magnesium particles modified by the organic additive (graphite) during MCT is a consequence of the fact that in the formation of the surface layer of particles in all the considered cases, an important role is played by carbon, also dispersed in the MCT process.

Structural changes during MCT of the investigated Me/C composites lead to a change in their chemical activity, which is clearly manifested in the solid-phase combustion (ie self-propagating high-temperature synthesis - SHS) of the mixture of aluminum or magnesium powder, as a fuel, with silicon dioxide, used as an oxidizing agent. Silicon dioxide, in this case, is used in an unactivated state. Mixtures were prepared at the stoichiometric ratio of the components: (Al 37.5% + SiO_2 62.5%) and(Mg 44% + SiO_2 56%). After MCT of aluminum with graphite and introduction of the resulting powder, respectively, in an amount of 37.5% and 44% into the charge with quartz, a considerable reduction in the induction period of ignition is observed as well as an increase in the rate and temperature at all stages of the combustion process as compared to a non-activated fuel (figure 6 a).For a mixture of quartz with a composite (Mg/C) after MCT, the induction period of ignition also decreases and the temperature and burning time of mixtures with SiO_2 increase, but this is less expressed than in the case with aluminum(figure 6 b).

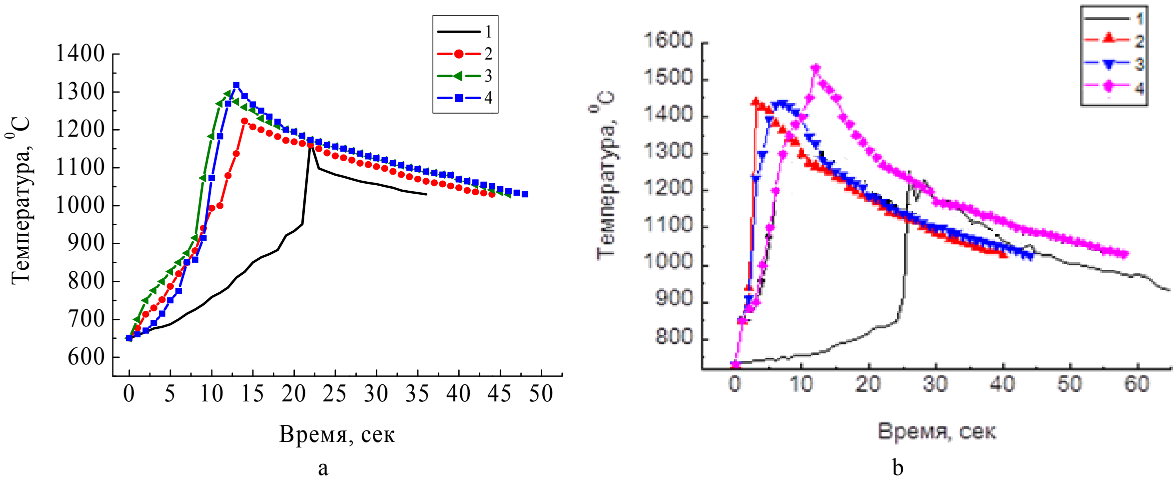
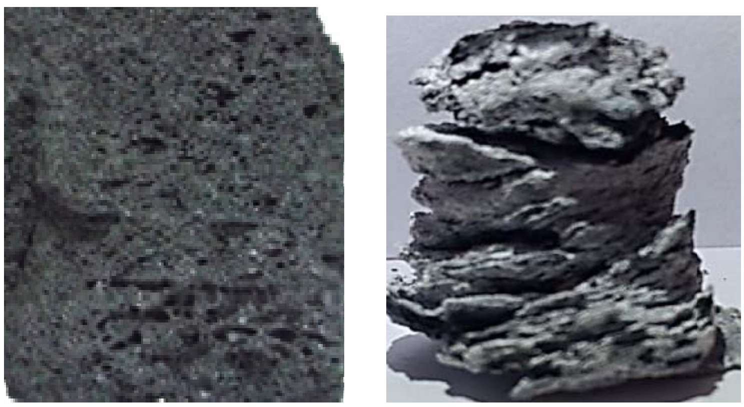


Figure 6 - Thermograms of combustion of the system (SiO_2 + Me) with aluminum and magnesium in the initial state and after 20 minutes of MCT with different amounts of graphite: a-SiO_2 + (Al/C), b-SiO_2 + (Mg/C); 1 - Me initial; 2 - 5%; 3 - 10%; 4 - 20% C

Table 2 shows the parameters of the main characteristics of the combustion process and the strength of the synthesized samples. It is seen in Table 2 that composition [(Al + C 20%)$_{MCT}$ 37.5% + SiO_2]has the maximum combustion temperature, but its strength is significantly reduced compared to the sample without carbon. This is due to the release of gaseous products, the amount of which increases with theincrease in carbon content in the mixture, thus leading to formation of the porous structure of the sample (figure 7 a). The maximum burning rate (118.2 deg/sec) during SH-synthesis was stated for the system [(Al + C 5%)$_{MCT}$+ SiO_2]. This is possibly related to the optimum ratio of the particle size of the constituent components of the mixture, and correspondingly to the increase in the packing density, which provides a close contact between the oxidant and the fuel.

Table 2 - The indices of the maximum temperature, the burning rate of mixtures of SiO_2 with modified aluminum and magnesium, and the strength characteristics of the synthesized samples

Composition of modified fuel based on aluminum	T_{max}, ^{0}C	Burning rate, deg/sec	σ, MPa
Al initial + (SiO_2)	1319	19.16	37.6
(Al + 5 % C)$_{MCT}$ + (SiO_2)	1441	118.2	8.36
(Al + 10 % C)$_{MCT}$ + (SiO_2)	1436	83.7	12.54
(Al + 20 % C)$_{MCT}$ + (SiO_2)	1532	56.8	2.11
Mginitial + (SiO_2)	1170	23.6	50
(Mg+5%C)$_{MCT}$+ (SiO_2)	1295	40.9	5.8
(Mg+10%C)$_{MCT}$+ (SiO_2)	1318	58.6	1
(Mg+20%C)$_{MCT}$+ (SiO_2)	1223	51.4	1



a b

Figure 7 - The break and appearance of SHS samples obtained with aluminum (a) and magnesium (b) modified during MCTwith the content of graphite equal to 20%

In the samples obtained with a fuel in the form of carbon-modified aluminum, a fine-porous structure with dense partitions is formed. This fact testifies to the prospects of using such materials for obtaining heat-insulating systems. Products of technological combustion of samples, the combustible component of which is the composite (Mg/C), have a low index of strength characteristics due to the porous, loose structure of the samples (figure 7 b). This is due to the fact that combustion proceeds layer-by-layer and a large amount of gaseous synthesis products are formed.

Conclusion. Thus, MCT of aluminum and magnesium with graphite contributes to a change in the morphology and structure of the particles during formation of composites (Me/C), the change in the size of aluminum and magnesium particles, and the surface modification with an organic additive (graphite). The use of mechanical treatment leads to a decrease in the particle size of metal powders and, as a consequence, an increase in the specific surface area of metal particles with an accumulation of defects in the crystal lattice. In the grinding process, the particle surface is constantly in an excited highly active state, and the presence of organic additives in the course of MCT provides the formation of an organic coating on the surface of the particles.

The combustion results of mixtures in which aluminum and magnesium were used as a fuel component after MCT in the presence of graphite showed the efficiency of this method in increasing the thermo-kinetic characteristics of the combustion process, as well as the conditions for the preparation of the combustible material and procedure the combustion process due to which formation of a large volume of gaseous synthesis products. The latter fact is important when using the obtained nanostructured Me/C composites in the composition of combustible systems intended, for example, for gas generators or for swelling and production of porous systems of a certain purpose. Such compositions are generally heterogeneous condensed systems.

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АЛЮМИНИЙ ЖӘНЕ МАГНИЙ БӨЛШЕКТЕРІНІҢ БЕТТЕРІН МЕХАНОХИМИЯЛЫҚ ӨҢДЕУ РЕЖИМІНДЕ МОДИФИЦИРЛЕУ – ЖЫЛУСЫЙЫМДЫ КОМПОЗИТТЕР АЛУ ТӘСІЛІ

Аннотация. Мақалада металл ұнтақтарын (алюминий РА-4 маркасыжәне магний MPF-3 маркасы) беттік белсенді зат ретінде графит көмегімен, ұнтақ дисперстілігін арттыру және бөлшек беттік қабатын модифицирлеу мақсатында динамикалық диірменде механохимиялық өңдеу жұмыстарының нәтижелері келтірілген. Металдарды графитпен механохимиялық өңдеу металл бөлшектерінің құрылымы және қасиеттерінің өзгеруіне, белсенді металл мөлшерінің жоғарылауына және дисперстелінетін бөлшектер бетінде органикалық жабындылардың пайда болуына акеледі. Алынған металл және графит бөлшектері физика-химиялық талдау әдістері, «Малверн 3600Е» құрылғысы көмегімен жүргізілетін, бөлшек өлшемдерінің таралуын гранулометриялық әдіс көмегімен зеттеулер жүргізілді. Термитті жүйелердің технологиялық жану үдерісіне металл ұнтақтарын механохимиялық өңдеудің әсері зерттелінді. Зерттеу нәтижелері механохимиялық өңдеуден кейін металл ұнтақтарының бөлшектерінің өлшемдері төмендеп, сәйкесінше кристаллитті торда ақаулар жиналып, меншікті беттік көлемі жоғарылайтындығын көрсетті. Механохимиялық өңдеу үдерістері кезінде Ме/С композит құрамында графиттің массалық үлесіне байланысты кристаллиттер өлшемі өзгеретіндігі анықталды. Алюминий және магний бөлшектерін графитпен механохимиялық өңдеуден кейін жанғыш зат ретінде қолдану жану үдерістерінің термо-кинетикалық сипаттамаларының жоғарылауына алып келетіндігі көрсетілді.

Түйін сөздер: механохимиялық өңдеу, алюминий, магний, модифицирлеу, қаттыфазалы жану

УДК: 541.124.16

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МОДИФИЦИРОВАНИЕ ПОВЕРХНОСТИ ЧАСТИЦ АЛЮМИНИЯ И МАГНИЯ В РЕЖИМЕ МЕХАНОХИМИЧЕСКОЙ ОБРАБОТКИ – СПОСОБ ПОЛУЧЕНИЯ ЭНЕРГОЕМКИХ КОМПОЗИЦИЙ

Аннотация. В работе представлены результаты механохимической обработки порошков металлов (алюминия марки ПА-4 и магния марки MPF-3) в мельнице динамического действия с использованием графита в качестве поверхностно активной добавки с целью повышения дисперсности порошков и модифицирования поверхностного слоя частиц.Механическая обработка металлов с графитом способствует изменению структуры и состава поверхности металлических частиц, повышению доли активного металла и формированию органического покрытия диспергируемых частиц.Полученные частицы металлов с графитом были исследованы физико-химическими методами анализа, гранулометрическим методом для оценки распределения частиц по размерам, проводимая на приборе «Малверн 3600Е».Исследовано влияние механохимической обработки порошков металлов на процесс технологического горения термитных смесей. Результаты исследования показали, что после механической обработки размеры частиц порошков металлов уменьшается и как следствие увеличивается удельная поверхность частиц металлов с накоплением дефектов в кристаллической решетке.В процессе механохимической обработки, размер кристаллитов изменяется от массовой доли используемого графита в составе композита Ме/С. Приипользовании в качестве горючего компонента алюминия и магния после механохимической обработки в присутствии графита повышаются термо-кинетические характеристики процесса горения.

Ключевые слова: механохимическая обработка, алюминий, магний, модифицирование, твердофазное горение.

МАЗМҰНЫ

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ISSN 2518-1491 (Online), ISSN 2224-5286 (Print)

Редакторы: *М. С. Ахметова, Т. А. Апендиев, Аленов Д.С.*
Верстка на компьютере *А.М. Кульгинбаевой*

Подписано в печать 04.08.2018.
Формат 60х881/8. Бумага офсетная. Печать – ризограф.
11,5 п.л. Тираж 300. Заказ 4.

Национальная академия наук РК
050010, Алматы, ул. Шевченко, 28, т. 272-13-18, 272-13-19