PRODUCTION PROCESSES AND PROPERTIES OF POWDERS

Fabrication of Metal Powders for Energy-Intensive Combustible Compositions Using Mechanochemical Treatment: 2. Structure and Reactivity of Mechanically Activated Al−Modifier−SiO₂ Mixtures

N. N. Mofa[a], *, B. S. Sadykov[a], **, A. E. Bakkara[b], ***, and Z. A. Mansurov[a, b], ****

[a]Institute of Combustion Problems, Almaty, 050012 Republic of Kazakhstan
[b]Kazakh National University, Almaty, 050040 Republic of Kazakhstan
*e-mail: mofa46@mail.ru
**e-mail: sadykoff_baha@mail.ru
***e-mail: bakkara_ayagoz@mail.ru
****e-mail: zmansurov@kaznu.kz

Abstract—The results of studying the particles of the aluminum−modifier−quartz composites after mechanochemical treatment (MCT) in a planetary centrifugal mill by various physicochemical methods are presented. Graphite (C), polyvinyl alcohol (PVA), and stearic acid (SA) are used as modifiers. To increase the dispersive ability of plastic metallic powders (metal−modifier), MCT was performed in the presence of quartz, the weight fraction of which in the composite varies from 5 to 20%. The largest grinding of aluminum particles is observed with an increase in the graphite content from 5 to 20% and SiO₂ content from 5 to 10% in the composition of aluminum−modifier−quartz composites. The particle decreases, while that of crystallites increases with the MCT of the Al−SA−SiO₂ system with an increased quartz content in the composite. The maximal imperfection of aluminum after MCT is characteristic of the Al−SA−5%SiO₂ samples. An increase in the particle size and, correspondingly, a decrease in the specific surface with rather small crystallite sizes are observed after MCT for the Al−PVA−SiO₂ composition. It is shown that the imperfection of the crystalline structure of aluminum particles increases with an increase in the quartz content in the system. The synthesized powder material is a composite formation of aluminum and quartz particles bound by polymer formed based on polyvinyl alcohol in this case. The MCT of the Al−modifier−SiO₂ mixtures results in an increase in the powder activity both due to the accumulation and redistribution of defects in aluminum particles and due to the change of the surface structure due to the incorporation of modifying additives into the destructed oxide layer. The conceptual model of transformation of the surface layer and subgrain structure of aluminum particles during MCT is presented.

Keywords: mechanochemical treatment, aluminum, modifier, quartz, structural characteristics, reactive ability

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INTRODUCTION

Aluminum is one of the most efficient combustible additives in various energy systems [1−4]. However, a dense oxide film on the surface of aluminum particles complicates the ignition of compositions and conditions the low combustion rate. Important factors in combustible condensed systems are rapid fuel ignition and high heating capacity of the oxidation reaction, which largely depends on the surface state and structure of Al particles [5−8].

Various methods are applied to remove or decrease the oxide film thickness. One such method is mechanochemical treatment (MCT) in mills of various types—so-called mechanical reactors (friction, cen-trifugal, or planetary) [9−13]. Such treatment leads to an increase in the active metal content depending on grinding parameters (the time, ratio of balls and powder weighs, diameter of milling bodies, and typology and weight fraction of the activating substance).

Simultaneously with the physical effect during mill treatment, the activating change of the surface of aluminum particles can be performed due to the presence of special additives in the treated powder. For example, the authors of [14, 15] showed that powdered aluminum treated in a mixture with finely dispersed graphite in vibration mills manifests the high reaction ability when interacting with water. We previously showed [16] that, in addition to graphite, it is promis-

ing to use other organic compounds during MCT, in particular, polyvinyl alcohol (PVA) and stearic acid (SA). The aluminum activity depends both on the dispersity of powders and on the structural characteristics of powders, which vary depending on the material treatment conditions [17−19].

The introduction of inorganic additives during MCT of aluminum and other metallic powders showed the prospects of forming high-energy compositions. For example, the MCT of Mg + MoO₃ and Al + MoO₃ mixtures was followed by the destruction of metallic particles to a nanodimensional level and a considerable change in the defect structure without reactions between the components, which in totality provides the high-reaction state of compositions [20].

To increase the dispersive ability of plastic metallic powders during MCT, additions of powders of solid inorganic compounds (minerals) with a high abrasiveness can be used. It was shown in previous publications [21−23] that quartz has a high dispersing ability after grinding in a mechanical reactor. As far as it has high hardness, the grinded particles can serve as good abrasive material with the simultaneous treatment of quartz with soft and plastic aluminum.

In addition, quartz is piezoelectric. The lattice strain caused by the mechanical stress leads to the redistribution of electric charges with the presence of polar directions (negative electric charges appear on negative edges of the hexagonal prism zone and positive charges appear on positive edges) [24]. The converse piezoelectric effect, which consists of the fact that internal stresses proportional to the electric field voltage appear in the electric field in crystals, also occurs. In addition, when quartz is destructed, the exoelectron emission appears. It is accompanied by the substance luminescence [25], which clearly demonstrates the change of its energy state.

All these physical processes in totality provide the additional activation of aluminum particles that are present during MCT and promote the formation of reaction centers on the fresh particle surface.

Listed factors were taken into account when performing this work on mechanochemical treatment (activation and modification) of aluminum in the presence of quartz and organic modifiers. The target of our work was to study the regularities of changing the state and structure of the surface layer of aluminum particles during MCT in the presence of organic additives and silicon dioxide powder. It was required to establish the optimal ratios of the aluminum powder and organic and inorganic additives in the Al−modifier−SiO₂ composite that promote both the formation of the defect structure and transformation of the surface layer of the particles, which will increase the reaction ability of aluminum powder.

Previously, the authors of [26] used IR spectroscopy to evaluate the charge of the state and structure of aluminum powders. We performed thermogravimetric analysis, which allows us to show the variation in the activity of modified powders immediately during heating, which is important to evaluate the prospects of their use in combustible compositions.

EXPERIMENTAL

Mechanochemical treatment of the aluminum powder of the PA-4 brand with an average grain size of 50 μ was performed using a Pulverisette 5 centrifugal planetary mill (CPM) (FRITSCH, Germany): the volume of the working chamber is 500 cm³, the revolution rate of steel drums is 400 rpm, and acceleration is 40 g. Treatment was performed by steel balls in air with a powder-to-ball weight ratio $M_p : M_b = 1 : 4$ for 20 min. Graphite (C), polyvinyl alcohol (PVA), and stearic acid (SA) in optimal amounts were used as modifiers, which showed previously [26] the best results on varying the structural characteristics (the crystallite size; specific surface of the aluminum powder; and, consequently, the activity increment resulted from MCT). Quartz in an amount from 5 to 20 wt % was introduced into these compositions[1]. To prevent the oxidation of Al particles by air oxygen after MCT and evaluate the changes associated with the mechanical effect, the samples of the dispersed mixture were passivated by hexane (C_6H_{14}).

The morphology of aluminum particles after MCT with various additives and elemental analysis of composites were performed using a Quanta 3D 200 I Dual system scanning electron microscope (FEI Company, United States). To determine the particle size after MCT, we used the dry measurement method using a Scirocco-Malvern Mastersizer 2000 device (Malvern Instruments Ltd., Great Britain). The specific surface (S_{sp}, m²/g) of composites was determined by the chromatographic method of the thermal adsorbate desorption (BET) using a Sorbtometr-M device (Russia, Novosibirsk). The carrier-gas (helium) flow rate was 48−50 mL/min at an Ar concentration in the mixture of 3−6%. To analyze the crystalline structure, we evaluated the crystallite sizes (coherent scattering regions—CSRs) of the substance by the Scherrer method and relative lattice strain by the WinFit software using a DRON-4M diffractometer when recording X-ray diffraction patterns with a small rate of 1−2°/min in range θ = 40°−80° accurate to ±(8−10%).

To evaluate the state of the powders of treated aluminum, we performed the thermal analysis, notably, thermogravimetry (TG), differential scanning analysis (DSC), and differential thermal analysis (DTA) using a Netzsch 449F3A-0372-M device (Netzsch, Germany). All samples were studied in nitrogen with purity of 99.99%. Thermal measurements were performed in a temperature range of 30−1000°C with a heating rate of 10 K/min. The activity of the grinded

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[1] Here and below, we keep in mind wt % if not noted otherwise.

Table 1. Changes in the specific surface, average particle size, crystallite size, and microdeformation of the crystal structure of aluminum after MCT of the Al−modifier−SiO$_2$ composites

Formulas of composites, wt %	$S_{уд}$, м^2/г	d_{sp}, μm	L, Å	$H_μ$, %
Al−5C−5SiO$_2$	2.447	23	451	0.152
Al−5C−10SiO$_2$	3.678	18	383	0.165
Al−5C−20SiO$_2$	4.981	15	365	0.203
Al−20C−5SiO$_2$	3.583	8.7	368	0.213
Al−20C−10SiO$_2$	5.991	7.6	354	0.237
Al−20C−20SiO$_2$	6.756	10	343	0.230
Al−3SA −5SiO$_2$	6.493	11.2	374	0.232
Al−3SA−10SiO$_2$	5.116	9.5	408	0.187
Al−3SA−20SiO$_2$	3.988	8.2	415	0.165
Al−20PVA−5SiO$_2$	9.937	29	455	0.196
Al−20PVA−10SiO$_2$	2.191	36	360	0.205
Al−20PVA−20SiO$_2$	2.975	41	343	0.245

aluminum powder modified by organic additives in the mechanical reactor was evaluated using the volumetric method by the liberation of hydrogen during the interaction of aluminum with a 20% aqueous sodium hydroxide solution.

RESULTS AND DISCUSSION

The analysis of the Al powder after MCT with organic modifying additives and quartz showed that the amounts of graphite and quartz largely depend on the degree of grinding and size distribution of Al particles. The most considerable grinding of Al particles was observed with an increase in the graphite content to 20% and SiO$_2$ content to 10% (Table 1). The average particle size in Al−5% C−5% SiO$_2$ and Al−20% C−10% SiO$_2$ compositions is 23 and 7.6 μm. The minimal crystallite size (L) occurs at its smallest value and, consequently, microdistortions of the crystal lattice enhance.

When performing MCT of the system with stearic acid, the particle size decreases with an increase in the quartz content in the composite, but the crystallite size decreases. The decrease in the specific surface is associated with the agglomeration of modified composite particles. The average particle size in Al−3% SA−5% SiO$_2$ and Al−3% SA−20% SiO$_2$ samples is 11.2 and 8.2 μm, respectively. The maximal imperfection of aluminum was inherent to the Al composite with 5% SiO$_2$. It seems likely that the aggregates of modified Al particles with quartz particles introduced into the metallic surface are also formed in this case.

When treating aluminum with polyvinyl alcohol in the presence of quartz, on the contrary, an increase in the particle size and, correspondingly, a decrease in the specific surface at rather low crystallite sizes were observed. The imperfection of the crystal structure of Al particles after MCT increases with an increase in the quartz content in the system. It seems likely that the analyzed particles comprise the composite formation of aluminum and quartz particles bound by the PVA-based polymer in this case. Our data evidence complex structural changes after MCT of aluminum with graphite, polyvinyl alcohol, and stearic acid both in the particle bulk and on its surface.

The change of the surface morphology of the particles during MCT is clearly shown by scanning electron microscopy images (Fig. 1). It is seen from Fig. 1a that the aluminum particles in the post-MCT Al−20% C−10% SiO$_2$ sample have the platelet form and clearly pronounced surface inhomogeneity. It follows from the results of the energy dispersive analysis (the EDS procedure allows us to find the elements from carbon to uranium in amounts up to 1%) that the aluminum content in the composite is almost 65%, carbon—25.57%, oxygen—7.45%, and silicon—2.16% (Fig. 1b). Oxygen enters the composition of both silicon dioxide (quartz) and the oxidized surface layer of aluminum particles. The grinded graphite particles are incorporated into the surface of deformed Al particles, promoting their fracture. Quartz particles also abrade Al particles during treatment, which, finally, causes the distortion of the surface layer and their fracture.

It is seen in the image of the Al−20% PVA−10% SiO$_2$ composite (Fig. 1c) that it consists of the
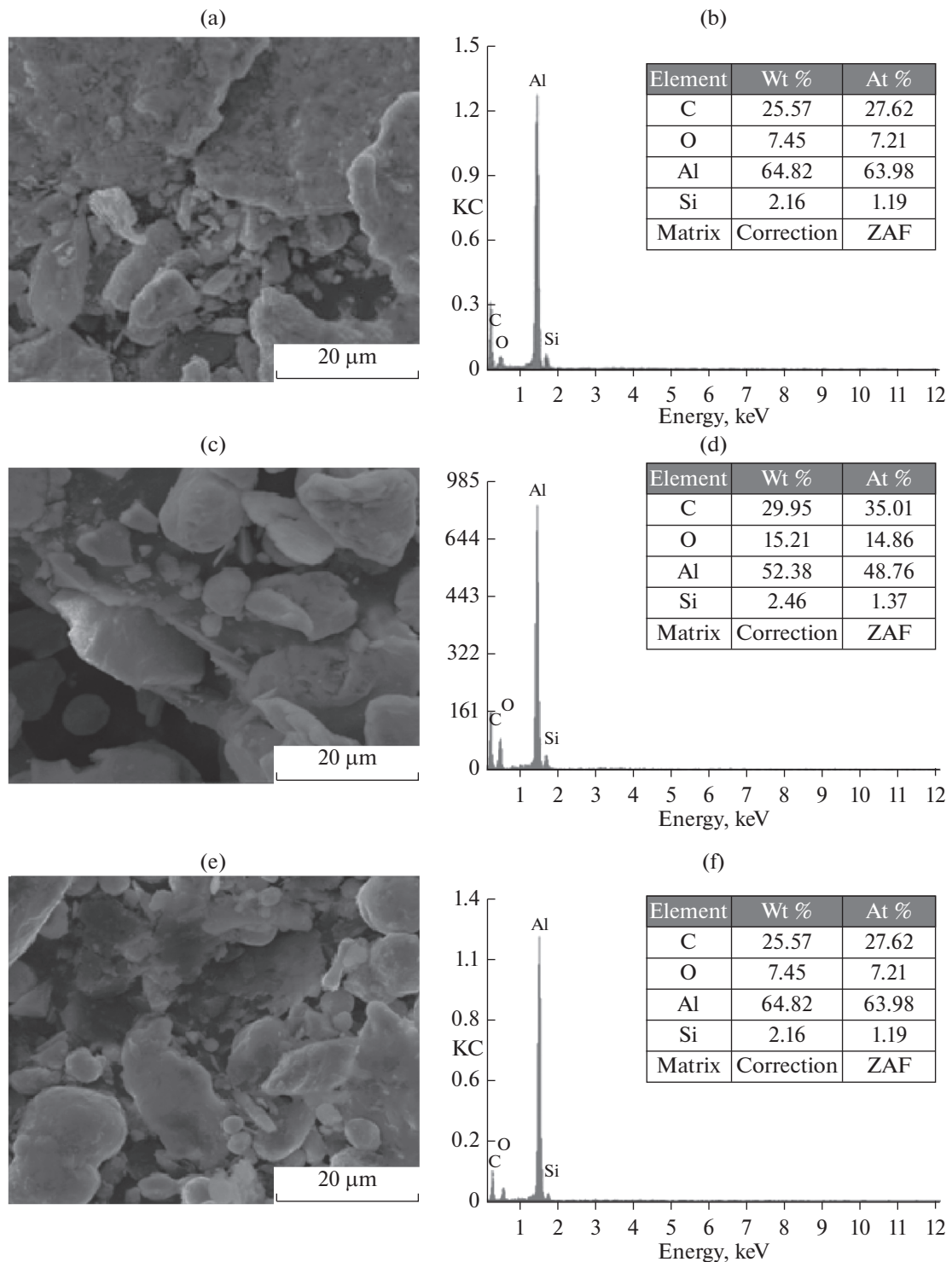
(a)

(b)

Element	Wt %	At %
C	25.57	27.62
O	7.45	7.21
Al	64.82	63.98
Si	2.16	1.19
Matrix	Correction	ZAF

(c)

(d)

Element	Wt %	At %
C	29.95	35.01
O	15.21	14.86
Al	52.38	48.76
Si	2.46	1.37
Matrix	Correction	ZAF

(e)

(f)

Element	Wt %	At %
C	25.57	27.62
O	7.45	7.21
Al	64.82	63.98
Si	2.16	1.19
Matrix	Correction	ZAF

Fig. 1. Structural characteristics of composites after MCT. (a, c, e) Electron microscopy images, (b, d, f) energy-dispersive spectra and weight fraction of elements, (a, b) Al−20% C−10% SiO_2 sample, (c, d) Al−20% PVA−10% SiO_2 sample, and (e, f) Al−3% SA−10% SiO_2 sample.

particles of various degrees of dispersity. They are scaly shaped and have a rather smooth surface. The latter reflects the composite formation of the polymer layer with dissolved carbon-containing inclusions on the surface of composite formations. The specific surface of the particles is 2.191 m^2/g. The aluminum content in the system from the results of the energy dispersive analysis (Fig. 1d) is 52.38%. An increase in the
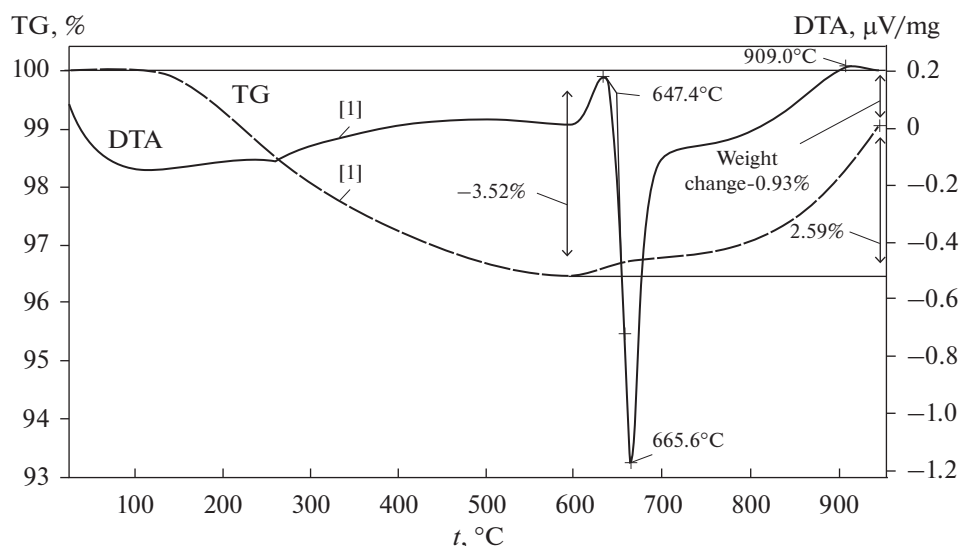
Fig. 2. Thermometric and gravimetric curves of initial aluminum.

oxygen concentration to 15.21% is observed due to the fact that it is contained not only in silicon and aluminum oxides but is also present in a modifier (polyvinyl alcohol) in a large amount.

Figures 1e and 1f show electron microscopy images, energy dispersive spectrum, and weight fraction of elements of the Al–3% SA–10% SiO_2 composite. It follows from its data that most particles are spherically shaped, while the aluminum content is 58.07%; i.e., it is lower than in the case with graphite (65%), but higher than for the treatment with PVA. When introducing 3% SA, the carbon content in the composite exceeds 31%, which can be associated, first, with its higher concentration in the composition of stearic acid ($C_{17}H_{35}COOH$) and second, with the composite saturation by the passivating substance (hexane), which is used at the final MCT stage to prevent the ignition of the system. The specific surface of the particles of this composite is 5.116 m^2/g. The difference in the amount of carbon in this system is determined by the different ability of the polymeric film formed on the particle surface during MCT with various modifiers to absorb hexane.

Thus, MCT of aluminum with additions of quartz and organic modifiers results both in the change in the powder dispersity and transformation of morphology, composition, and structure of the surface layer of particles. It follows from our data and the results of [26] that the capsulation of conglomerates into polymer films occurs when using polyvinyl alcohol and stearic acid as modifiers. All observed structural changes in a complex determine the quantitative change of the state of the Al powder resulted from MCT.

To analyze the state of modified Al particles, we used the thermogravimetric analysis. Investigations were performed in the nitrogen medium. Figure 2 shows the thermometric and gravimetric curves of the initial aluminum powder. The weight loss in this sample upon heating to 600°C was 3.52%, which can be associated with the separation of hydroxides form the particle surface. The presence of OH groups on the surface of Al particles in the initial state is established by IR spectra [26]. Melting of aluminum and complete transformation into the liquid phase occurs at 665.6°C. The exotherm is fixed before the melting onset at $t = 647.4$°C, which is caused by the oxidation of aluminum. The sample weight increases by 2.59% with an increase in temperature to 909°C, which can be associated with the saturation of liquid aluminum by nitrogen with the formation of aluminum nitride.

Figure 3 shows the results of the thermogravimetric analysis of the Al–20% C composite after MCT. No oxidation of aluminum was observed while this sample heated, but its carbide can form after melting at 660°C:

$$4Al + 3C \rightarrow Al_4C_3.$$
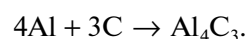
This process is exothermic; it results in an increase in sample temperature and weight in range $t = 705–770$°C. The weight increases by 5.87% during further heating from 800 to 1000°C, which is possibly associated with the formation of aluminum nitride:

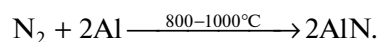$$N_2 + 2Al \xrightarrow{\quad 800–1000°C \quad} 2AlN.$$

Figure 3b shows the results of the influence of the presence of silicon dioxide during MCT on the state of the Al composite. The increment of the temperature and sample weight from 20% Cu and 10% SiO_2 to melting can be associated with the oxidation (combustion) of carbon. The exotherm from the redox reaction between the composite components occurs after melting:
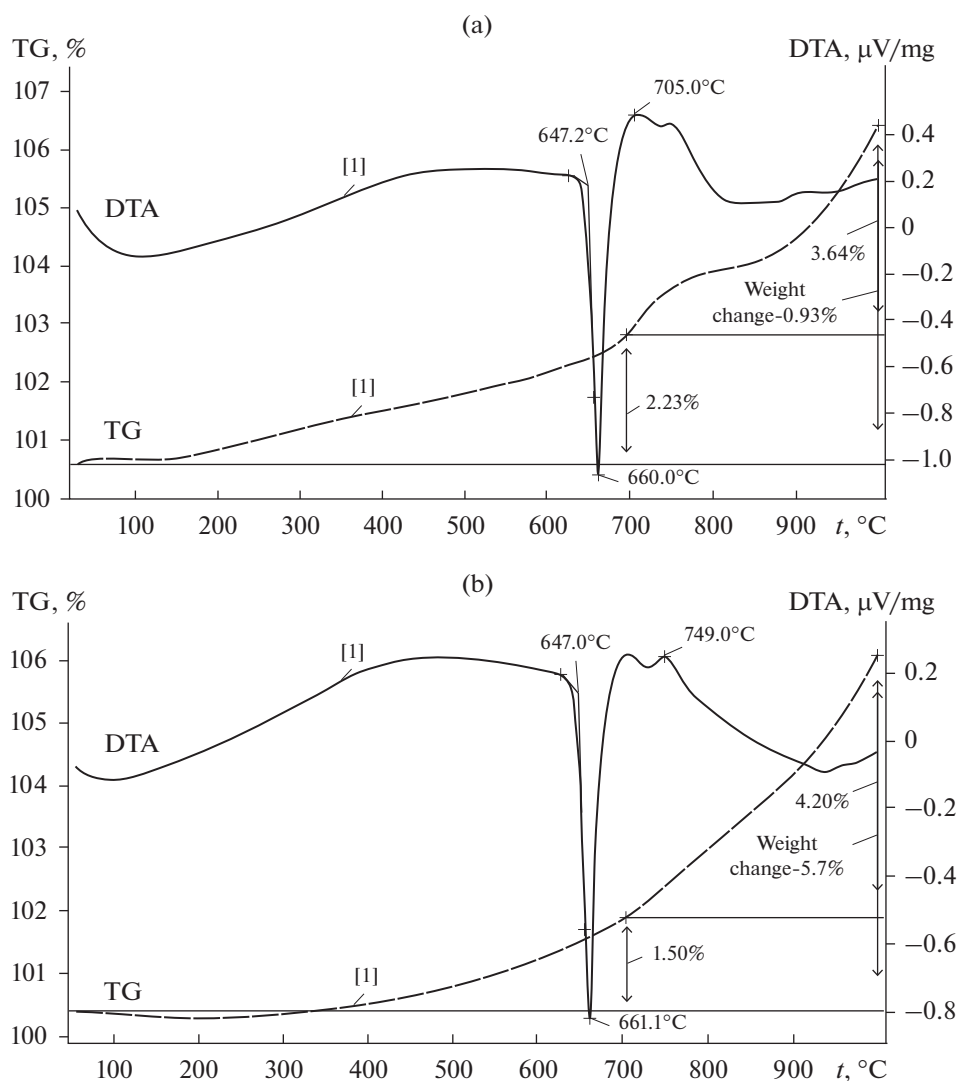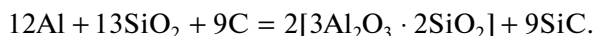
$$4Al + 3SiO_2 = 2Al_2O_3 + 3Si.$$
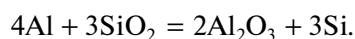
Fig. 3. Thermo- and gravimetric curves of composites (a) Al−20% C and (b) Al−20% C−10% SiO_2 after MCT.

Aluminum carbide forms in the Al−20% C−10% SiO_2 composite at 749°C and above. In addition, the endothermic reaction with the formation of mullite and silicon carbide is possible [27]:

$$12Al + 13SiO_2 + 9C = 2[3Al_2O_3 \cdot 2SiO_2] + 9SiC.$$

The total change of the weight of this sample was 5.7%.

The thermogravimetric analysis of aluminum with polyvinyl alcohol after MCT showed that alcohol decomposes at $t > 329°C$, while the weight loss up to 500°C is 16.93%; i.e., the almost complete decomposition of the organic additive takes place (Fig. 4a). However, a 7.51% increase in the sample weight is observed after melting the Al powder, while its decrease is observed above 900°C, possibly due to the reaction

$$2Al_2O_3 + 9C \rightarrow Al_4C_3 + 6CO.$$
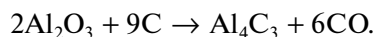
Polyvinyl alcohol also destructs in the Al−20% PVA−10% SiO_2 composition to 400°C, and weight loss is 13.65% (Fig. 4b). The exothermic reaction proceeds at $t \geq 900°C$ with large heat liberation. An increase in the sample weight is 17.36%. In this case, the reaction products can be mullite and aluminum carbide [28].

Figure 5 shows the results of the thermal analysis of the Al−3% SA and Al−3% SA + 10% SiO_2 samples. Similarly to previous cases, weight loss is observed due to the decomposition of the organic additive in the sample. The exotherm before melting and subsequent weight increment can be associated with the oxidation of powders, and a 5.35% increase in the weight can be associated with the dissolution of the gas phase, particularly nitrogen, in liquid aluminum.

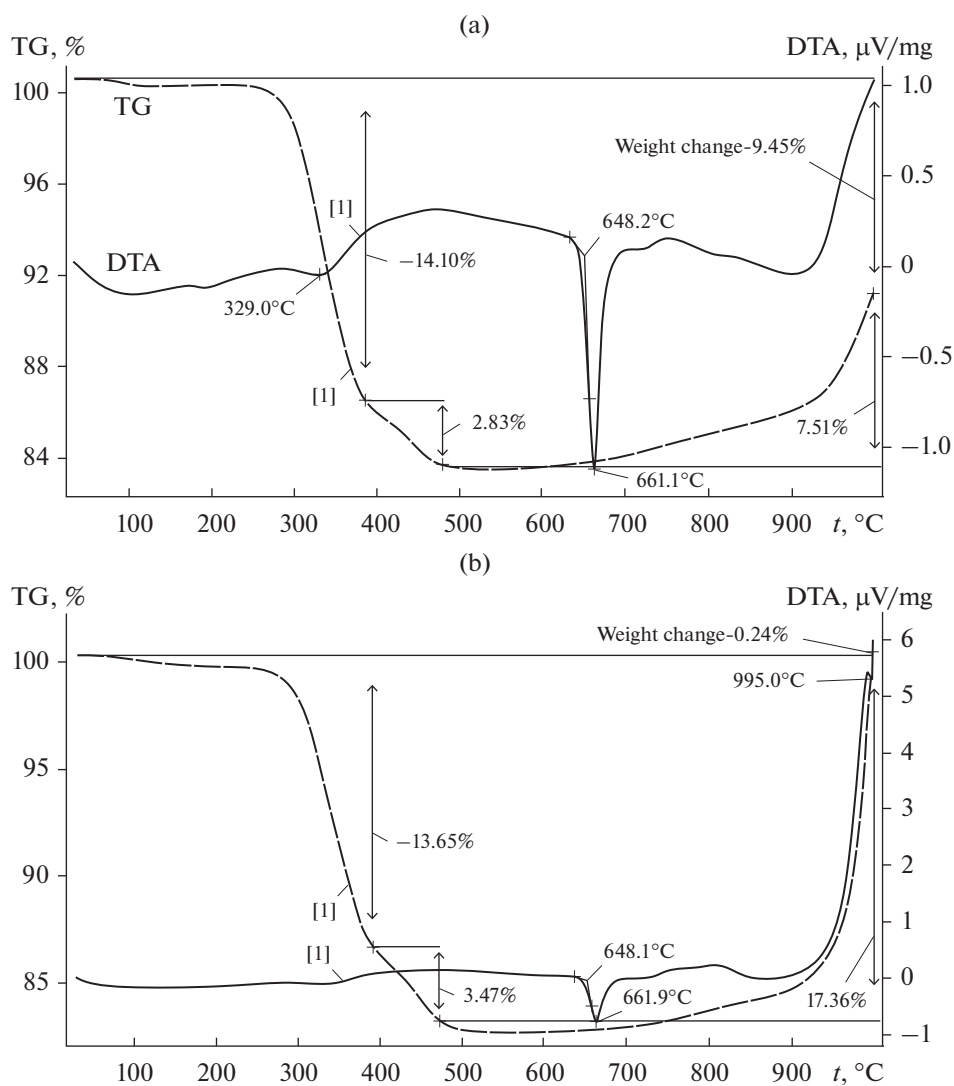The intense heat liberation and a 8.63% sample weight increment are observed for the Al−3% SA−

Fig. 4. Thermo- and gravimetric curves of composites (a) Al−20% PVA and (b) Al−20% PVA−10% SiO$_2$ after MCT.

10% SiO$_2$ sample above 900°C due to the intense interaction reaction of silicon oxide and aluminum.

Thus, the results of the thermogravimetric analysis of aluminum powders showed that mechanochemical treatment leads to a variation in their activity during the interaction both with gases and with solid components of the system. This can be associated with the presence of radicals of the destructed modified and the presence of dispersed quartz, which in totality should be reflected in the activity of the treated powder.

Subsequently, we evaluated the variation in the active aluminum content after MCT in the composition with the organic modifier by the volumetric method and calculated the relative increment in the aluminum activity [26] in the Al−modifier−SiO$_2$ systems with various quartz contents. It follows from Fig. 6 that the highest activity increment up to 24.5% is observed for the Al−3% SA−5% SiO$_2$ composite.

This is associated with the fact that similarly to aluminum, stearic acid also enters the reaction with alkali with the formation of stearates [29].

The stable tendency of increasing the activity increment from 12.3 to 20.93% is observed with an increase in an amount of silicon dioxide in the composition of the Al composite with 20% polyvinyl alcohol. Mechanochemical treatment of aluminum with graphite as a modifying additive in the presence of SiO$_2$ leads to an increase in the relative aluminum activity in limits of 7%. It is established that the use of organic modifiers increases the aluminum activity in limits of 12%, while the presence of quartz in some cases ensures an increase in the characteristic to 20−25%.

Thus, our investigations with the help of the thermogravimetric analysis, X-ray structural analysis, electron microscopy, and energy dispersive analysis—as well as using EPR and IR spectroscopy [26]—
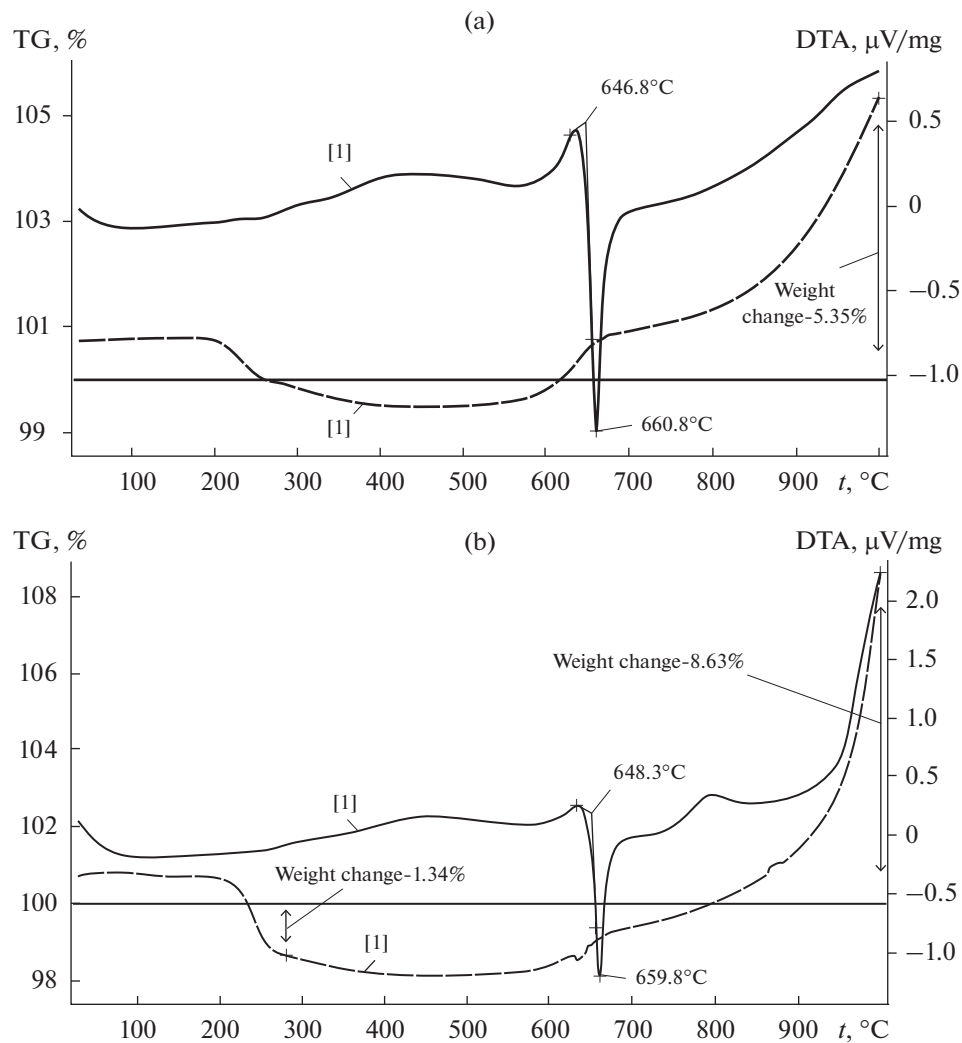
Fig. 5. Thermo- and gravimetric curves of composites (a) Al−3% SA and (b) Al−3% SA−10% SiO$_2$ after MCT.

showed that MCT of aluminum with various modifiers and quartz additive in a mechanical reactor lead to a change of the surface structure, morphology, and activity of Al particles. The optimal MCT conditions were selected to attain the maximal aluminum activity. Such treatment makes it possible to fabricate the material in the highly active state due to the high degree of dispersity and large specific surface, as well as the structural imperfection in the particle volume, which is reflected in the change of subgrain characteristics of aluminum.

The surface modification will make it possible to exclude the escape of defects from the particle bulk on its surface, i.e., fix the active state of metallic particles and power intensity of the combustible system in general. The following processes occur when mechanically grinding aluminum with various modifiers. The oxide film coating aluminum is crushed in increased stress zones at the first stage, and fragments of crushed particles are accumulated in the gaps between the particles (along the subgrain boundaries). Here, oxide
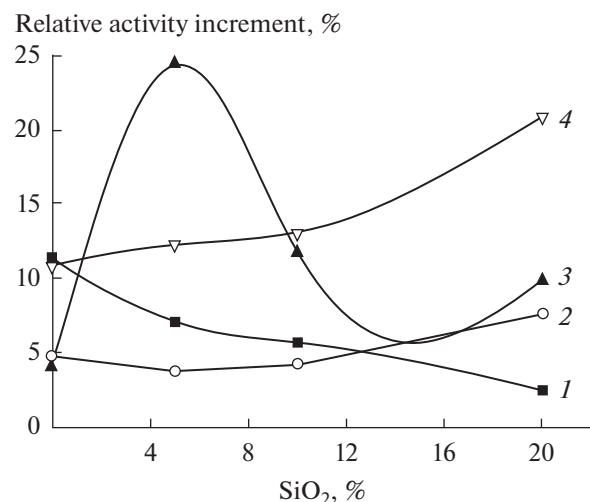

Fig. 6. Relative increment of aluminum activity in the Al composites with various modifiers after MCT depending on the SiO$_2$ content. (*1*) 5% C, (*2*) 20% C, (*3*) 3% SA, and (*4*) 20% PVA.
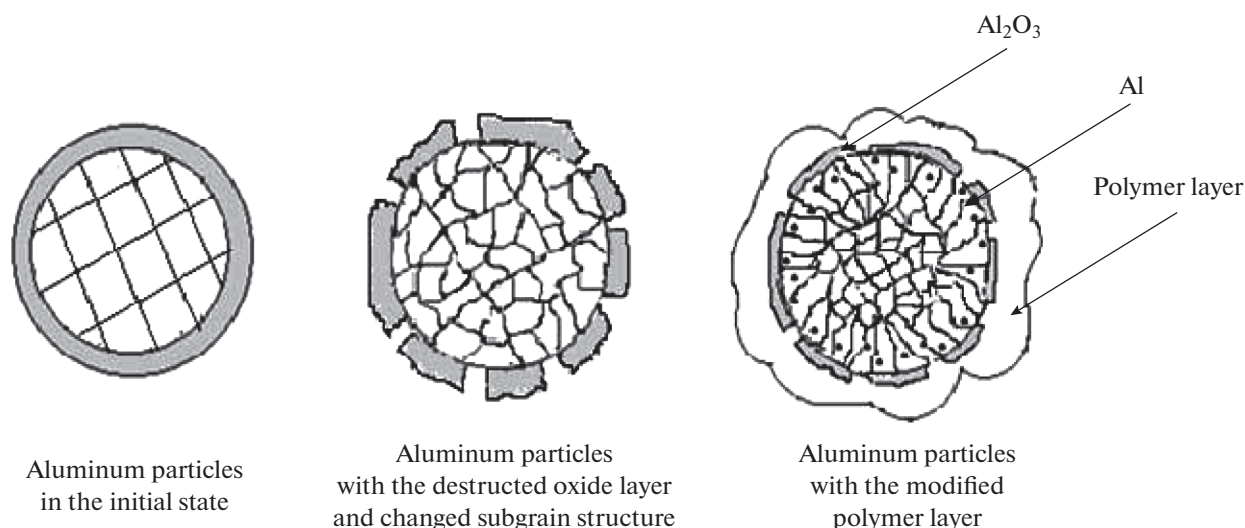
Fig. 7. Transformation model of the surface layer and subgrain structure of the Al particles during MCT.

prevents the motion of boundaries and holds formed subgrains in the boundaries of initial powder particles. Agglomerations of fragments of particles are dispersed at the second stage by the distribution of oxide plates in the plastic deformation direction. Crushing of subgrains is a consequence of this phenomenon. The presence of quartz during composite MCT promotes the attrition of the surface oxide layer of the Al particle and its saturation by organic compounds of a modifier.

All transformation stages of the surface layer and subgrain structure of aluminum particles during MCT are presented in the model (Fig. 7) reflecting the concept of modifying metallic particles. The following processes occur stage-by-stage during MCT:

(i) the destruction of the oxide layer on the surface of aluminum particles;

(ii) the change of the subgrain structure due to the accumulation and redistribution of defects in the particle bulk;

(iii) the formation of the capsulation layer consisting of modifying organic additions on the particle surface.

The destruction products of organic compounds during MCT, when propagating into the near-surface layer along the subgrain boundaries, also promote an increase in activity of aluminum particles. Thus, the developed defect structure is formed, as is, consequently, the "excess" energy of the system that provide its stable active state.

CONCLUSIONS

Thus, the results of our investigations of aluminum powders after mechanochemical treatment in a planetary centrifugal mill in the presence of modifiers (graphite, polyvinyl alcohol, and stearic acid) and quartz showed that the aluminum activity substantially increases after MCT. The changes in both the defect structure and in the surface layer occur in Al particles due to the introduction of modifying additives into the destructed oxide layer. The presence of quartz in the treated mixture promotes an increase in the dispersive ability of metallic particles and a change of their surface structure.

Depending on the type of the modifying addition and amount of introduced quartz powder into the treated mixture, qualitative changes are observed in the defect structure of aluminum particles and degree of their activity. The maximal activity was showed by aluminum modified by stearin in the presence of 5% quartz. When modifying aluminum with polyvinyl alcohol, an increase in the quartz content in the treated mixture promotes an increase in its activity. Such treatment results, on the one hand, in the protection of the aluminum particle against the oxidation in air and, on the other hand, its more active interaction with the oxidizer during heating. The transformation of structural changes of the surface layer and block structure is reflected in a model of modifying aluminum particles during MCT (Fig. 7).

The use of modified aluminum powders is promising for various combustible mixtures with the controlled power intensity, particularly for SHS systems [30] and various solid propellants for rocket engines [5, 6].

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