

Article

Creation of Modified Aluminum Powders with Increased Reactivity for Energy Systems

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Abstract

Aluminium plays a key role in developing modern energy technologies, from electrical systems to high-energy materials, providing a combination of functionality, economy, and reliability, but the oxide film on its particles reduces the effective reactivity. This work aims to increase the reactivity of aluminum powder by mechanochemical treatment using modifiers. The materials used were aluminum powder of the ASD brand and graphite of the GL-1 brand. The experiment subjected aluminum powder to mechanochemical treatment (MCT) with different graphite contents. It was shown that MCT significantly increases active aluminum content in the powder due to partial destruction of the oxide film on its surface. In addition, morphological analyses confirm the destruction of the oxide, the graphite coating, and the appearance of lamellar structures measuring 0–58 μm . Thermal analysis shows that the primary exothermic peak shifts from 662.6 $^{\circ}\text{C}$ to 653.9 $^{\circ}\text{C}$ for Al + 10% graphite, and the heat released increases by 27%, which means lower activation energy and more complete oxidation. However, at 20% graphite, the thermal gain decreases, since carbon shields the metal areas. Thus, the optimal content is 10% graphite: at this ratio, the best thermochemical behavior of the powder is achieved. The data obtained indicate that the MCT of aluminum powder with graphite effectively increases its reactivity. The resulting aluminum powders with modified particle surfaces facilitate the development of new technologies for the creation of various high-energy solid propellant systems. For rocket engines, preference is given to solid rocket propellant (SRP), which is a mixture of substances (components) capable of burning in the absence of air, producing a large amount of gaseous working fluid heated to a high temperature, providing thrust.

Keywords: modifier; aluminum; graphite; mechanochemical treatment; thermogravimetric analysis; differential thermal analysis



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1. Introduction

Aluminium (Al) is widely used as a metallic fuel in composite solid propellants [1] due to its high mass heat of oxidation, significant density, and corrosion resistance [1–4]. Including aluminum powder in solid rocket propellants (SRPs) increases the energy density of the charge and the thermodynamic potential of the system [5–7]. However, the operational implementation of these advantages is limited by several factors: the passivating Al_2O_3 film increases the induction period of ignition, molten particles are prone to coalescence

and the formation of coarse agglomerates, incomplete oxidation of the metal leads to the accumulation of solid residue, and worsening heat and mass transfer in the reaction zone. To minimize the listed phenomena, targeted surface modification methods are required, ensuring high aluminum reactivity while maintaining the powder's technological reliability [8]. Aluminum in various forms—foil, foam structures, composites, fibers, aerogels—is used in thermal insulation due to its combination of lightness, mechanical strength, and specific thermal characteristics. Depending on the task (from radiation reflection to high-temperature insulation), the choice of material form is determined by the temperature, density, and stability requirements. Current research focuses on the composition and structure of materials to improve efficiency and functionality [9].

The current paradigm in developing solid rocket propellants is to create systems where the combustion rate can be adequately scaled to maintain a specified relationship between combustion rate and pressure over a wide range of operating conditions. Achieving these objectives relies primarily on using combustion modifiers, including modified aluminum powders with increased reactivity [10–14], which can specifically enhance or suppress the combustion rate, ensuring the required performance characteristics.

One of the practical approaches is the introduction of graphite as a solid functional modifier. Due to its high thermal conductivity and chemical inertness, graphite intensifies heat exchange between the exothermic reaction zone and aluminum particles, accelerating the heating and destruction of the oxide shell [4]. Additionally, the graphite modifier acts as a mechanical separator, which reduces the average diameter of the formed agglomerates and increases the completeness of metal oxidation.

The authors [15] investigated aluminum in the composition of solid fuel, showing that graphite reduces the agglomeration of its particles due to thermal conductivity and mechanical separation, accelerates the destruction of the oxide film, and increases the completeness of metal oxidation. In experiments [16], it was demonstrated that the introduction of functionalized carbon nanomaterials, including graphite-like structures, promotes the acceleration of energy release by reactive compositions, which is of direct importance for increasing the reactivity of aluminum powders. Chong Chen et al. [17] analyze the influence of various graphite materials on the reactivity of activated aluminum composites, including interactions with water and ice. This work contributes to our understanding of the roles of graphite as an activator and heat-conducting agent.

A study of the interaction of aluminum with graphite in composites and carbide formation processes, which are key to understanding the thermal and reaction dynamics in Al-graphite systems, is presented in [18].

Modified particles are produced through mechanochemical activation, ultrasonic dispersion, chemical precipitation, and their combinations. Mechanochemical treatment reduces the particle size, introduces the modifier into the surface layer, and increases the defectiveness of the crystal lattice, accelerating the diffusion of oxygen through the oxide film [19].

Complex modification of aluminum with graphite reduces the induction period of ignition, increases the combustion rate, and reduces the yield of solid condensed products, thereby increasing the thermochemical efficiency of solid fuel. This work aims to systematically study the effect of these additives on the thermal and kinetic parameters of aluminum oxidation under conditions simulating operational loads. It can be used to develop new rocket fuel types, ensuring high engine operation efficiency and reducing process losses.

2. Experimental Part

During the study, aluminum powder ASD containing 95.8% metallic aluminum was used. The average particle size of aluminum ASD is 30 microns.

The modifying additive was GL 1 graphite powder (GOST 17022 81 [20]). Graphite was used to suppress the agglomeration of aluminum particles and reduce heat loss due to high thermal conductivity and thermochemical stability; introducing carbon filler ensures a decrease in the average diameter of agglomerates and a more uniform combustion process. The main physical and chemical characteristics of the substances used are given in Table 1.

Table 1. Physical properties of reagents used for the experiment.

Name of the Connection	Chemical Formula	Density, G/Cm ³	Molecular Weight, G/Mol
Aluminium ASD	Al	2.702	26.982
Graphite GL-1	C	2.26	12.011

The mechanochemical treatment (MCT) process used a planetary ball mill, NXQM-2A (Changsha Tian Powder Technology, Hunan, China). During the grinding process, the amount of modifying additives varied within 10–20%, and the processing time was 20 min according to the results of preliminary studies [21]. The process was carried out at a rotation speed of 460 rpm and a mass ratio of powder to grinding media (Mp/Ms) of 1:4. The samples were passivated with hexane to prevent oxidation of aluminum particles by atmospheric oxygen after MCT.

Microstructural analysis was performed using a JSM 6490 scanning electron microscope (JEOL, Tokyo, Japan). X-ray phase studies were performed using monochromatic radiation on a DRON 4M diffractometer (“Scientific and Production Enterprise “Burevestnik”, Saint Petersburg, Russia). CoK α ($\lambda = 0.17902$ nm) and CuK α ($\lambda = 0.15418$ nm) in the range $2\theta = 10$ – 70° at step 0.02° . The particle size distribution was determined using a laser dispersion analyzer Winner 2005A (Jinan Winner, Jinan, China). Thermal analysis (DTA and TA) was performed on a simultaneous thermal analyzer STA409PC/PG (NETZSCH, Waldkraiburg, Germany). All samples were studied in a nitrogen atmosphere with a purity of 99.99%. Thermal measurements were carried out in the temperature range of 30–700 °C and at a heating rate of 10 K/min.

3. Results and Discussion

The energy-dispersive spectroscopy (EDS) results indicate a high purity of the aluminum powder under study (Figure 1). The spectrum contains almost only aluminum—its mass fraction is 98.27%. This corresponds to the technical characteristics of ASD brand powders, which usually contain at least 99.7% Al and only about 0.3% total impurities [22].

According to the results of volumetric analysis (Table 2), it was established that mechanochemical treatment of aluminum leads to an increase in the content of active metal from 80.52% to 92.90% due to the destruction of the passive oxide film and activation of the surface of aluminum particles. This helps improve aluminum’s flammability and reactivity in the composition of solid rocket fuel.

Table 2. Content of active aluminum in the aluminum-graphite composite (Al/C) after 20 min of MCT.

Composition of Composites	Active Al Content, %
Aluminum (ASD) original	80.52%
Al ASD after 20 min of MCT	92.90%
Al ASD +10%C after 20 min of MCT	76.80%
Al ASD +20%C after 20 min of MCT	74.30%

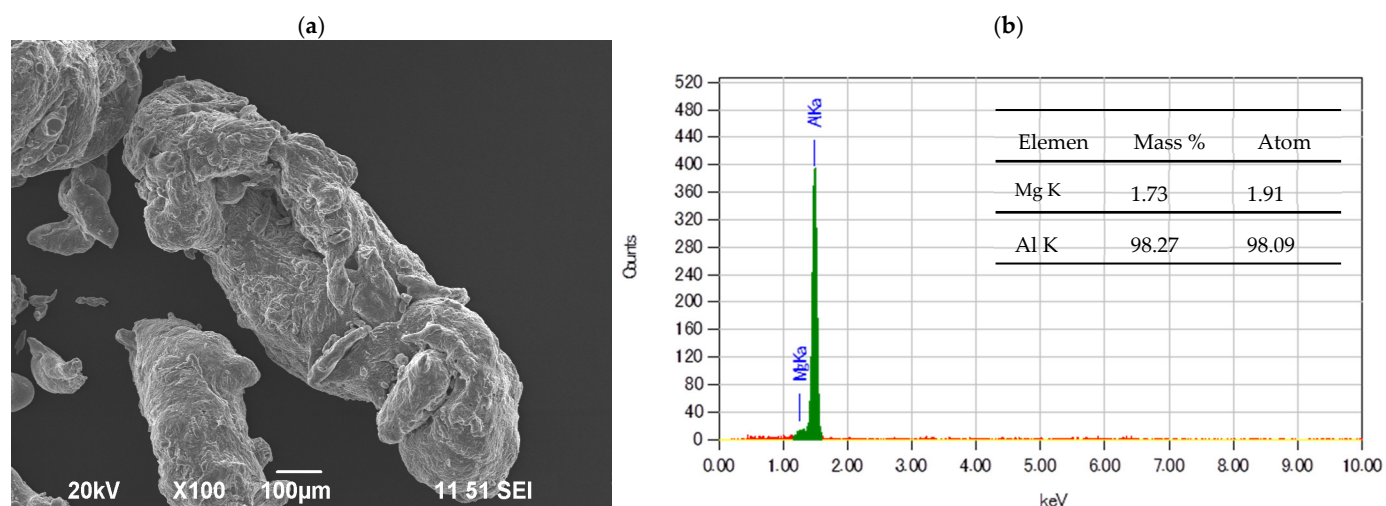


Figure 1. Energy-dispersive spectrum (a) and mass fraction of elements (b) in the composition of the initial sample of Al ASD.

With the introduction of graphite (10–20% C), the activity of aluminum decreases to 76.80% and 74.30%, respectively. This is due to the dilution of the metal phase and partial screening of particles, which limit access to the active surface.

The results of the study of modification of aluminum composites using mechanochemical treatment (MCT) and additives such as graphite showed a significant influence of these factors on the physicochemical properties of the material. MCT destroyed the initial oxide shell on the Al surface (Figure 2a), thereby increasing the availability of the metallic phase for oxidation–reduction processes. The simultaneous increase in oxygen content indicates the formation of a new oxide film, improving the particles' thermal stability and reactivity. Under such conditions, the powder acquires a characteristic plastically deformed, layered plate morphology with a high specific surface area, enhancing the kinetic interaction with the oxidizer. The presence of arsenic (As < 2 wt.%) is associated with technological impurities of the raw materials and does not have a noticeable effect on the functional characteristics of the composite.

The addition of 10% C significantly affects the morphology of aluminum particles. According to the results of EDS analysis, oxygen was not detected, indicating a practical barrier function of the carbon coating, preventing further oxidation of the metal during MCT. The surface of the aluminum particles is covered with a thin carbon film, which reduces their tendency to agglomerate. At the same time, despite partial screening of the surface, introducing 10% graphite (C) increases aluminum's reactivity due to improved heat exchange, dispersion of particles, and destruction of the oxide film (Figure 2b). Thus, carbon performs a dual function—barrier and stabilizing, which critically affects the thermochemical behavior of the resulting composite. Changes in composite morphology are observed, with a further increase in the graphite content of 20%. In Figure 2c, the SEM image demonstrates pronounced heterogeneity of the structure: large aggregates of graphite and dispersed fragments partially embedded in the aluminum matrix are recorded. The absence of oxygen in the spectrum indicates the preservation of the carbon layer's protective function, preventing aluminum's oxidation during mechanochemical processing. The decrease in the proportion of carbon with a simultaneous increase in the aluminum content is probably due to the redistribution of components in the volume of the material and the formation of a more stable composite structure. Such structural features favorably affect the thermal stability and homogeneity of the resulting composite (Figure 2c).

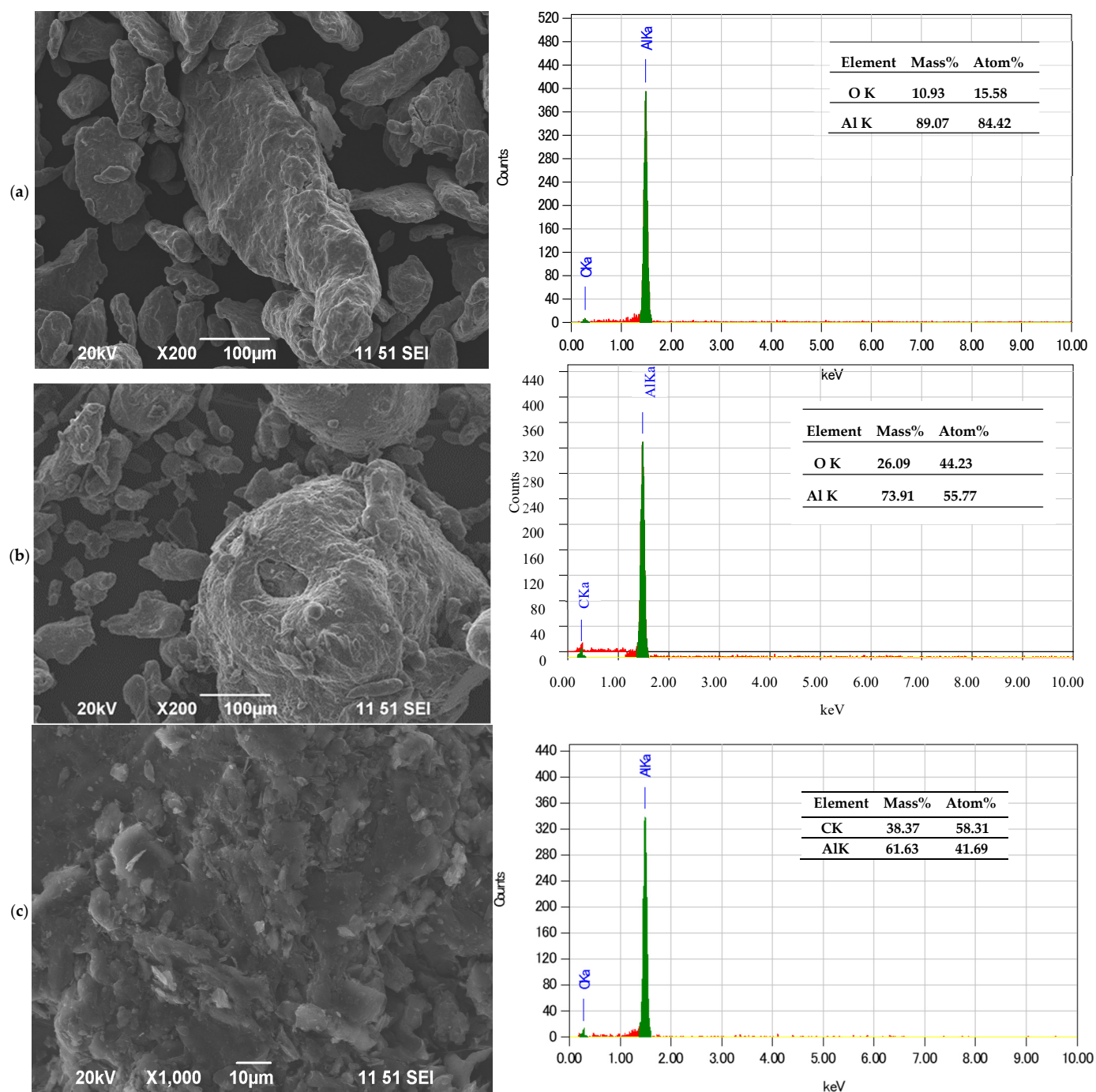


Figure 2. Energy-dispersive spectrum and mass fraction of elements in the sample. (a) Al ASD after MCT; (b) Al ASD +10% after MCT; (c) Al ASD + 20% C after MCT. MCT time 20 min.

In the initial Al ASD sample (Figure 3a), sharp intense diffraction peaks are observed, indicating a high degree of crystallinity. After MCT (Figure 3b), a broadening of the diffraction peaks of aluminum and a decrease in their intensity are observed, indicating a reduction in the size of crystallites and an increase in micro-deformations of the crystal lattice (a decrease in crystallinity). In the sample with the addition of 10% graphite (Figure 3c), the diffraction pattern shows only aluminum reflections, while the diffraction peaks remain broadened, and their relative intensity decreases. The decrease is most noticeable at a content of 20% graphite (Figure 3d). This indicates that the introduction of graphite during MCT leads to further refinement of aluminum crystallites and an increase in the defectiveness of the crystalline structure but does not cause the appearance of new

crystalline phases. Diffraction lines of aluminum carbide, Al_4C_3 , were not detected. This means that, in these samples, aluminum remains the only crystalline phase, and carbon is in an amorphous or finely dispersed state (Figure 3c,d).

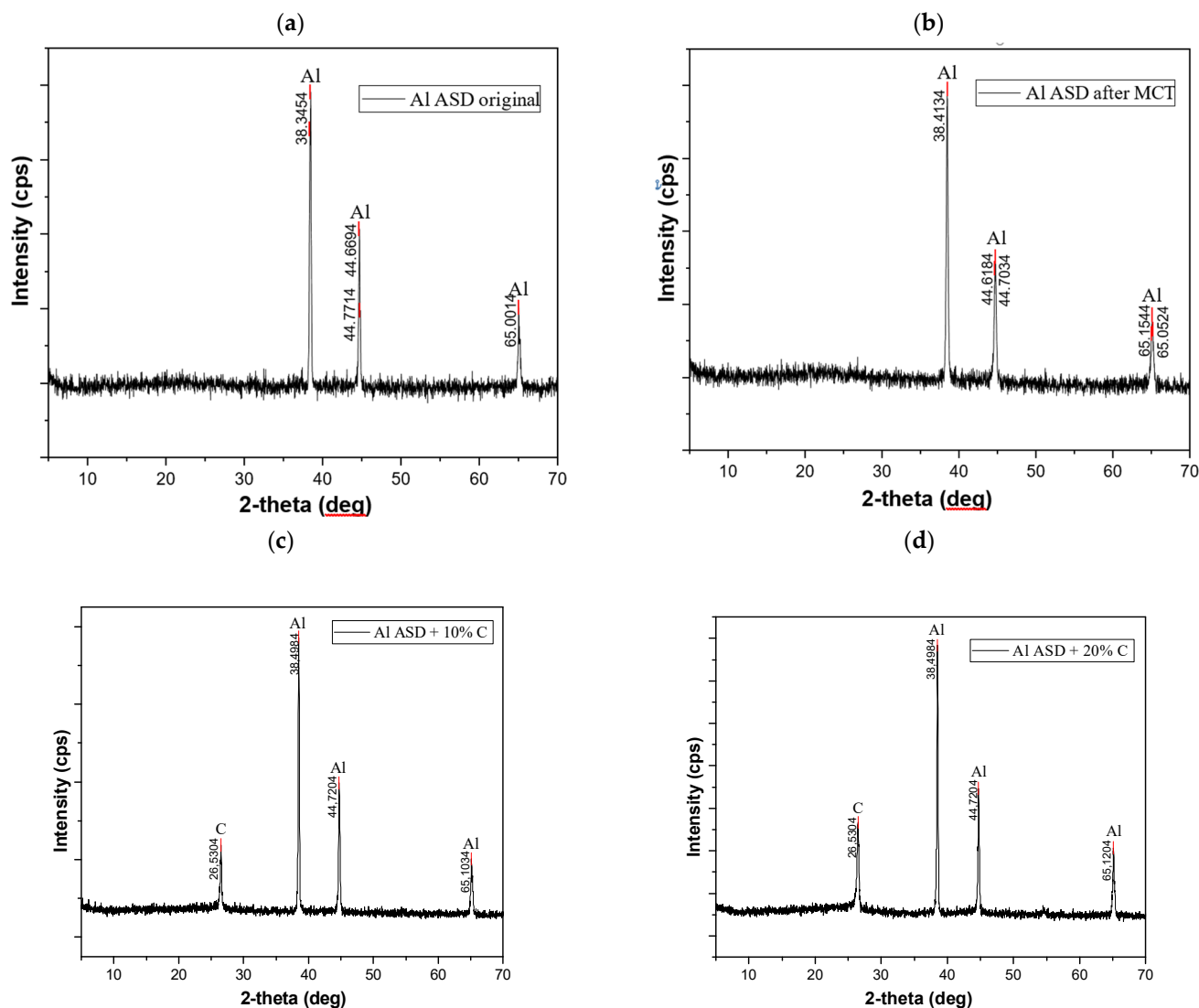


Figure 3. Diffractogram of samples: (a) Al ASD original; (b) Al ASD after MCT; (c) Al ASD/10% C; (d) Al ASD/20% C after 20 min MCT.

Figure 4a shows the particle size distribution of the initial aluminum powders. A predominant content of the medium fraction characterizes ASD. According to the granulometric analysis data, the leading share of particles (57.1%) is in the range of 100–153 μm , indicating a narrow fractional region formation. A significant part is also made up of particles sized 153–205 μm (28.0%). Fine fractions with sizes of 0–58 μm and 58–100 μm are found in significantly smaller quantities—3.5 and 6.2%, respectively. The share of the largest particles (205–273 μm) is 5.3%. Granulometric analysis of aluminum powder particles after 20 min of MCT (Figure 4b) is characterized by a noticeable change in the granulometric composition compared to the original powder. Most particles are concentrated in the 100–153 μm (43.3%), indicating a shift in the maximum distribution toward smaller fractions. Mechanochemical treatment reduces the particle size via impact/attrition, hence the observed shift toward finer fractions. A significant proportion of particles is also observed in the ranges of 58–100 μm (25.1%) and 153–205 μm (21.1%). The share of the

smallest particles sized 0–58 μm is 6.5%, and that of large particles in the 205–273 μm range is only 4%.

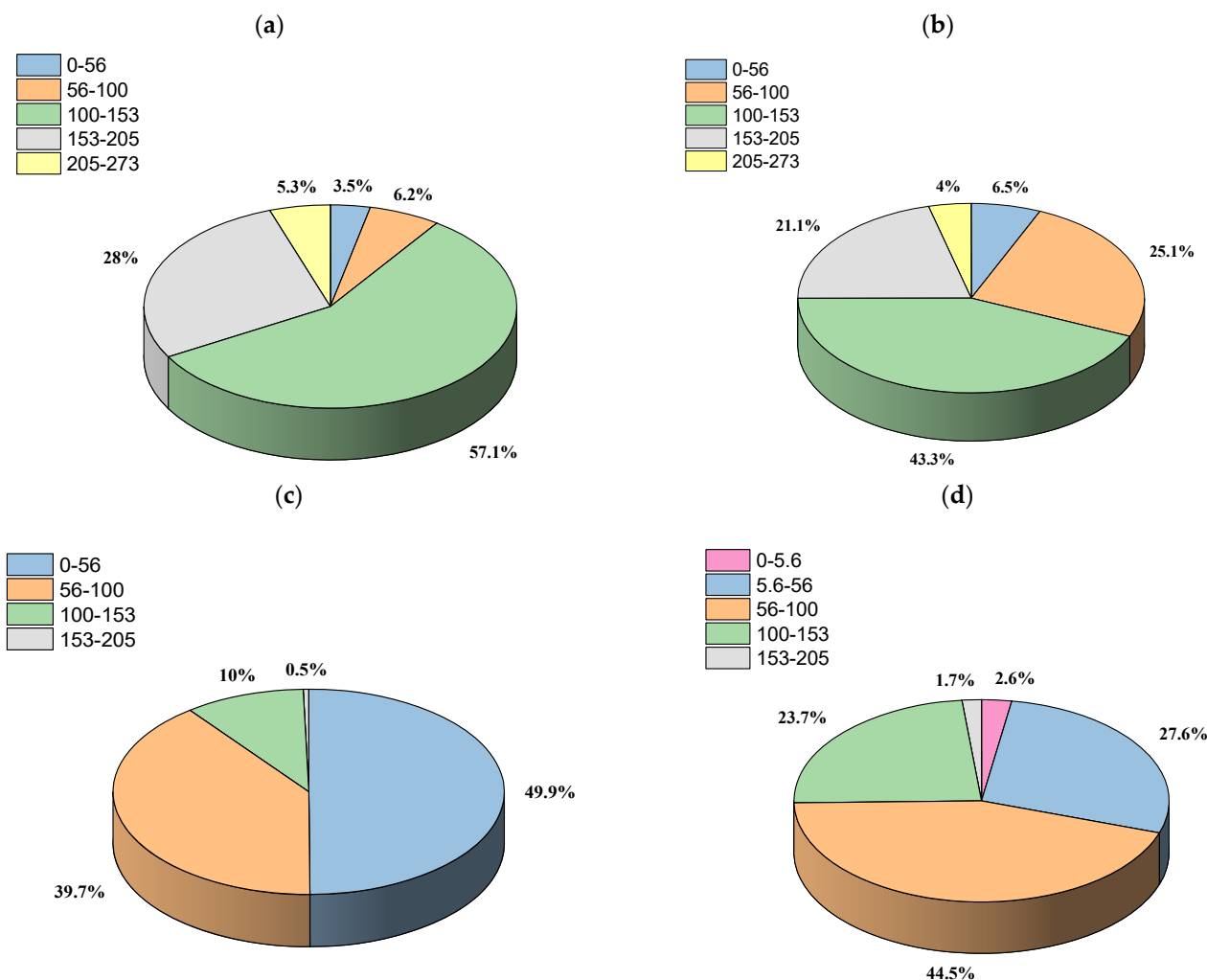


Figure 4. Particle distribution: (a) Al ASD original; (b) Al ASD after 20 min of MCT; (c) Al ASD/10% C after 20 min of MCT; (d) Al ASD/20% C after 20 min of MCT.

Figure 4c shows the particle distribution of the mechanical mixture of aluminum powders (ASD 90%—C 10%) after 20 min of MCT. It demonstrates a significant shift in the granulometric composition towards fine fractions. The largest share of particles falls in the range of 0–58 μm , 49.9%, indicating intensive powder system grinding. A significant portion of the particles is also concentrated in the 58–100 μm (39.7%). The average-sized fractions of 100–153 μm and 153–205 μm are present in smaller quantities—10.0% and 0.5%, respectively. During mechanochemical treatment (MCT), the oxide layer on aluminum particles is destroyed, defects accumulate in the subgrain structure, and an encapsulating layer of modifying additives is formed on the particle surface. The decomposition products of organic compounds penetrate along subgrain boundaries, increasing particle reactivity and forming a defective structure with excess energy that stabilizes the active state. Mechanical action distorts the crystal lattice, leading to the formation of defects or amorphization, which enhances chemical activity at dislocation sites. These processes generate short-lived active centers (SLCs), whose relaxation releases excess energy, often observed as exothermic effects or luminescence during MCT. The addition of graphite further increases aluminum reactivity by enlarging the specific surface area and altering the crystal structure parameters [19].

The results of granulometric analysis of particles of a mechanical mixture of aluminum powders (Al ASD/20% C) after 20 min of MCT (Figure 4d) are characterized by the dominance of finely dispersed fractions. The main share of particles is concentrated in the 0–58 μm range, which accounts for almost half of the volume (about 50%). A significant part of the powder is represented by the fraction of 58–100 μm (approximately 40%). Fractions of larger sizes are much less common: 100–153 μm make up about 9–10%, and the share of particles sized 153–205 μm is minimal and does not exceed 1%. In total, the obtained data demonstrate that mechanochemical activation in the presence of 20% graphite is an effective method for regulating the granulometric composition and activating aluminum powders for subsequent use in synthesizing composite materials.

A comparative analysis of the granulometric composition of the studied powder systems showed a pronounced effect of MCT and the introduction of graphite on the distribution of particles. The initial ASD powder is characterized by the predominance of particles of average size 100–153 μm (57.1%) with a relatively low content of fine fractions (<100 μm in total, about 10%). After 20 min of MCT, a redistribution of the granulometric composition is observed with a decrease in the proportion of large particles and an increase in the content of fractions less than 100 μm (up to 31.6%), which indicates partial grinding of the powder and an increase in its specific surface. The introduction of graphite significantly enhances the disaggregation effect: with the addition of 10% graphite, the proportion of particles < 100 μm increases to 89.6%, and, with an increase in the graphite content to 20%, fine fractions make up about 90%. At the same time, the content of particles greater than 153 μm becomes minimal (<1%). Thus, mechanochemical activation in the presence of graphite ensures the formation of a finely dispersed structure with a high specific surface area and uniform distribution of the carbon phase, which creates favorable conditions for increasing the reactivity, compressibility, and quality of the formed composite materials.

The results of thermogravimetric (TG) and differential thermal analysis (DTA), presented in Figure 5, demonstrate significant differences in the thermal behavior of the original and modified aluminum powders. The initial Al ASD sample (Figure 5a) is characterized by a broad exothermic region with peaks at 662.6 $^{\circ}\text{C}$ and 681.1 $^{\circ}\text{C}$, accompanied by relatively low heat generation and a low degree of oxidation, which is due to the presence of a dense oxide shell on the surface of the particles. After MCT (Figure 5b), the exothermic peaks shift to the region of lower temperatures (655.0 $^{\circ}\text{C}$ and 672.6 $^{\circ}\text{C}$), which indicates an increase in the reactivity of Al due to the destruction of the passivation layer and an increase in the defectiveness of the structure. The most pronounced thermal response is observed in the sample with the addition of 10% graphite (C) (Figure 5c), the central oxidation peak is recorded at 653.9 $^{\circ}\text{C}$. This indicates a complex effect of graphite, which is manifested in improving heat exchange, preventing particle agglomeration, and stabilizing the reaction surface. The thermal signal's intensity and mass increase indicate a complete and more uniform oxidation of Al in the Al/C composite. With an increase in the graphite content to 20% (Figure 5d), a decrease in the intensity of the thermal effect and some narrowing of the reaction temperature range is observed. This is due to the dilution of the metal phase and the screening effect of excess graphite plates, limiting oxygen access to the aluminum areas.

Thus, the reduction in particle size increases the specific surface area and reduces thermal inertia, accelerating the heating and destruction of the oxide film. This leads to a shift in the exothermic peaks of TG/DTA to the region of 653.9–655.0 $^{\circ}\text{C}$ (Figure 5) and an increase in the proportion of active Al (Table 2). With the addition of 10% C, the necessary balance is ensured between heat transfer, surface shielding, and reactive availability of Al, which is confirmed by maximum heat release and more complete oxidation of the composite.

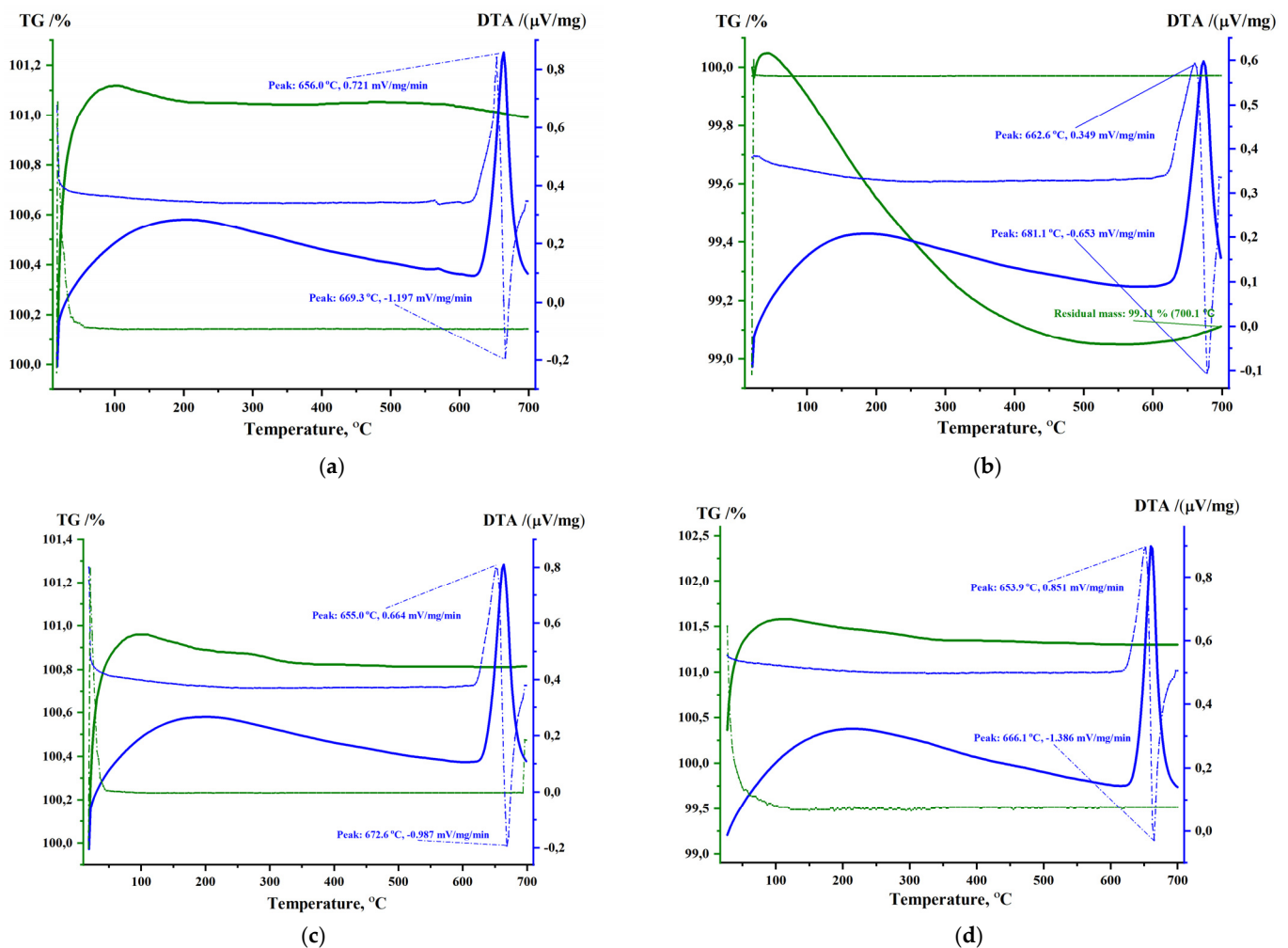


Figure 5. Thermal analysis of composites: (a) Al ASD original; (b) Al ASD; (c) Al ASD/10% C; (d) Al ASD/20% C after 20 min of MCT.

4. Conclusions

1. As a result of the research conducted, optimal conditions for the mechanochemical processing of aluminum with the addition of graphite were determined, allowing for a significant improvement in its thermoreaction characteristics for use in energy systems.
2. Mechanochemical treatment (MCT) of Al ASD substantially modifies particle morphology and reactivity: MCT increases the fraction of active aluminum (80.52% → 92.90% for Al after MCT) and shifts exothermic oxidation peaks to lower temperatures.
3. It was found that adding 10% C ensures optimal dispersion of particles, increases thermal conductivity, and suppresses agglomeration. At the same time, the average size of Al crystallites decreases, and a uniform carbon layer is formed, which, according to TG/DTA data, leads to the complete combustion of aluminum and increased energy efficiency of materials.
4. At a content of 20% C, the proportion of active Al decreases to 74.30%, the carbon protective film is preserved, but structural heterogeneity occurs, and the enthalpy of the reaction decreases, which is associated with dilution of the active phase and shielding of aluminum areas.

Thus, an approach has been developed in which the introduction of graphite in an optimal content allows for the simultaneous stabilization of the structure and activation of aluminum, ensuring more complete combustion and increased efficiency of energy systems.

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