Comparison of mechanical and friction properties of composite materials based on AlMg2 containing nanodimensional particles of crystalline graphite and nanofibers of gamma oxide of aluminum

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Abstract. The method of mechanical synthesis in a planetary ball mill was used for production of composite powders based on the AlMg2 alloy containing 1 wt. % of nanosized particles of crystalline graphite or $\gamma$-Al$_2$O$_3$. The resulting powders are consolidated by the sintering under pressure. Using the methods of X-ray diffraction analysis, scanning and transmission electron microscopy, the structural-phase composition of bulk composite materials was studied. Comparative analysis of the microhardness, the conditional yield stress at compression, and the friction coefficient of bulk composite materials is carried out. It has been found out that the mechanical properties of composites reinforced with $\gamma$-Al$_2$O$_3$ nanofibers are higher than when reinforcing with nanoscale particles of crystalline graphite.

1. Introduction

In the world scientific and technical literature there is a rather large number of works showing that dispersed hardening by nanosized reinforcement can significantly improve the structural and functional properties of aluminum and its alloys [1, 2].

The first and one of the most important stages in the production of composite materials by the method of powder metallurgy is the obtaining and preparation of powders. Their properties are largely determined by the physical-chemical conditions of the synthesis. The basis of mechanical synthesis is the mechanical treatment of solid mixtures (for example, in planetary ball mills), which results in grinding and plastic deformation of substances, accelerates mass transfer and mixing of the components down to the atomic level, and chemical interaction of solid reagents is activated [3].

Methods of thermoforming treatment of powder compositions are an effective means of obtaining high-density compacts (bulk materials). Both innovative technologies [4-7] and classical methods of pressure processing can be used for consolidation [8-11].

The aim of this work is investigation and comparison of the structural-phase composition, mechanical and frictional properties of composite materials based on the nanocrystalline matrix of the AlMg2 alloy, reinforced by additions of nanosized particles of crystalline graphite and nano-fibers of gamma-alumina.
2. Experimental methods and equipment

In the first case, the initial charge consisted of globular granules with a diameter of 1-2 mm of an aluminum alloy AlMg2 with addition of 1 wt. % graphite powder (average particle size less than 20 μm). In the second case, the matrix material remained the same, and 1 wt. % γ-Al2O3 nanofibers (diameter 10-40 nm) was used as the reinforcement. The SEM images of the reinforcements used in experiments are shown in Figure 1.

![SEM images](image)

**Figure 1.** SEM images graphite (a) and nanofibers of γ-Al2O3 (b)

Mechanical treatment of the initial charge was carried out in a Fritsch Pulverisette 6 planetary ball mill using surfactants (1 wt.% stearic acid). For grinding, steel balls with a diameter of 8 mm were used at a ratio of the mass of the loaded components to the mass of the grinding bodies equal to 1:15. The treatment was carried out at 600 rpm for 4.5 hours.

The morphology of the resulting powders was studied using an ultra-high resolution field-emission scanning electron microscope Zeiss Ultra Plus based on Ultra 55. The granulometric composition of the obtained powders was determined on a Microsaser-201C analyzer. The powders were previously sieved through a 300 μm sieve.

Consolidation of the obtained powders was carried out by the method of sintering under pressure at temperature of 400 °C, pressure of 600 MPa and a holding under pressure of 30 minutes.

The fine structure of the consolidated samples was studied using the transmission electron microscopy JEM-2010. The microhardness was measured by the Vickers method using a Shimadzu HMV-2 tester at load of 10 N and exposure time of 12 s. The mechanical properties at the compression of the obtained samples were determined using a mechanical testing machine WDW-100E.

The frictional properties were determined on a CSM Instruments tribometer under dry friction conditions in contact with a disc of heat-treated 4Kh5MFS steel. During the tests, the pin-disk scheme was used. The load was 5 N, the friction path length was 200 m.

3. Results

Figure 2 a,b shows typical SEM images of synthesized powders characterizing their morphology. The powder particles have an irregular shape, close in geometry to fragmental. The particles of the reinforcement were not fixed on the surface of the powder particles. This is due to their low concentration and to the regularities of the powder particles formation under conditions of mechanical synthesis [12].

The results of granulometric analysis show that despite the type of reinforcement the average size of the d50 powder particles formed is fairly close and is 59 μm when graphite used as a reinforcement and 62 μm at using γ-Al2O3 nanofibers.
Analysis of the results of X-ray diffraction analysis of the obtained powders (see Figure 3a) shows that regardless of the reinforcement type the diffractograms have a qualitatively similar character.

The presence of peaks corresponding to aluminum was noted [13]. However, there are no peaks corresponding to the reinforcements, which on the one hand can be due to their high dispersion achieved in mechanical synthesis, on the other hand, by the low sensitivity of the X-ray diffraction method to identifying phases with a content of less than 3%. Also, according to the Selyakov-Scherrer formula, the size of the coherent scattering regions was determined, according to which the size of crystallites of the matrix material can be judged with satisfactory accuracy. The volume-averaged sizes of the coherent scattering regions were calculated on the basis of the assumption of the spherical form of the crystallites. The results of the calculations show that for a composite material containing 1 wt.% graphite, the size of the coherent scattering regions is 72 nm, and for 1 wt.% γ-Al₂O₃ is 92 nm. This is due to the influence of graphite on the processes during mechanical synthesis, which consist both in changing the conditions of contact interaction between grinding bodies due to a change in the coefficient of friction, and in the fact that graphite, distributed over the surface of aluminum particles...
(at least at the initial stage of synthesis), reduces the number of juvenile surfaces, making plastic welding difficult and contributing to the dispersion of the powder.

After consolidation of the powders by the sintering under pressure, bulk samples with a diameter of 5 mm and a height of 5 mm were obtained. The results of X-ray analysis of consolidated samples are shown in Figure 3b. We can note a decrease in the width of the diffraction peaks of aluminum. The calculation of the size of the coherent scattering regions shows an increase in their values to 138 and 196 nm when graphite and $\gamma$-Al$_2$O$_3$ nanofibers are used as reinforcement, respectively.

Thus, in the process of aging during sintering under pressure, the regenerative processes take place in the consolidated material and the size of the crystallites passes from nano- to submicron scale.

In addition, the samples containing graphite as the strengthening additive were studied by Raman spectroscopy. This method has a high sensitivity to low concentrations of phases formed in the sample and makes it possible to more accurately identify the presence of Al$_4$C$_3$. The results of Raman spectroscopy show the presence of replicas at 492 cm$^{-1}$ and 857 cm$^{-1}$, corresponding to the crystalline phase of Al$_4$C$_3$. Those the selected temperature-time consolidation conditions lead to the formation of the dispersion carbide phase in the material AlMg$_2$ + 1 wt.% graphite.

Using the transmission electron microscopy, the structure of consolidated samples was studied. Analysis of the structure indicates that the obtained compact materials correspond to a crystalline structure with a crystallite size from tens to hundreds of nanometers. It can be noted that the calculated values of the average sizes of the coherent scattering regions are consistent with the crystallite size from the transmission microscopy data.

![HRTEM images of consolidated composite material](image)

**Figure 4.** HRTEM images of consolidated composite material: a – AlMg$_2$+1 wt.% graphite; b – AlMg$_2$+1 wt.% $\gamma$-Al$_2$O$_3$

Figure 4 shows HRTEM images characterizing the fine structure of the resulting composite materials. An analysis of the obtained HRTEM images shows that during the mechanical grinding process a mechanical splitting of the microdimensional graphite particles occurs to form particles smaller than 50 nm. Also, the agglomerated $\gamma$-Al$_2$O$_3$ nanofibers are dispersed.

In the course of measuring the microhardness, it was established that the composite materials obtained have microhardness exceeding the matrix material in the initial state by an average of 3.4 times. For AlMg$_2$ + 1 wt.% graphite and AlMg$_2$ + 1 wt.% $\gamma$-Al$_2$O$_3$ microhardness was 1.9 and 2 GPa, respectively.

Figure 5 shows the results of the compression and friction tests of the obtained composite samples. Analysis of the results of compression tests shows a significant increase in the mechanical properties of the composites obtained as compared to the matrix material. Thus, for example, the conditional yield strength increases in comparison with the matrix by 5.6 and 6.4 times for AlMg$_2$ + 1 wt% graphite and AlMg$_2$ + 1 wt% $\gamma$-Al$_2$O$_3$, respectively. A comparison of the mechanical properties of the obtained composites (see Figure 5b) shows that the use of $\gamma$-Al$_2$O$_3$ nanofibers as a reinforcement
makes it possible to obtain a material having not only high strength properties, but also a relatively high plasticity. While for the material reinforced with dispersed nanosized particles of graphite and dispersion particles $\text{Al}_4\text{C}_3$, despite the increase in strength properties, plasticity is significantly reduced, since the presence of the carbide phase promotes embrittlement of the material.

![Figure 5](image)

**Figure 5.** The results of compression tests (a) and friction (b) of the resulting composite samples. The numbers are: 1 – AlMg2+1 wt.% graphite; 2 – AlMg2+1 wt.% $\gamma\text{-Al}_2\text{O}_3$

The summary analysis of the data on the structural-phase composition and mechanical properties of the composite materials obtained allows us to conclude that hardening occurs with the additive contribution of the nanostructuring of matrix material and the dispersive hardening by the reinforcing particles. Moreover, in the case of composite AlMg2 + 1 wt.% graphite, additional dispersion hardening takes place due to the precipitation of the $\text{Al}_4\text{C}_3$ phase.

Friction tests show that the composite material reinforced with $\gamma\text{-Al}_2\text{O}_3$ nanofibers have a lower coefficient of friction 0.38. The use of nanosized particles of graphite as a reinforcement led to an increase in the coefficient of friction to 0.44.

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