Effect of type of ceramic particles on efficiency of gas dynamic spraying and hardness of hybrid coatings AlMg6/C60

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Abstract. The hybrid powders AlMg6/C60 + 10-70 wt.% Al₂O₃ and AlMg6/C60 + 10-70 wt.% AlN were obtained by the method of mechanical synthesis in a planetary ball mill. These powders were used to create coatings using cold gas-dynamic spraying. The influence of the type and concentration of ceramic reinforcement on the growth efficiency and microhardness of the formed hybrid coatings was studied. It has been found out that the use of ceramic particles of AlN (up to 50 wt.%) as a reinforcement contributes to a more efficient growth of the coating compared to Al₂O₃. At the same time, the microhardness of coatings reinforced with AlN is lower than that containing Al₂O₃.

1. Introduction
In the last decade, gas-thermal methods of forming protective coatings on the surfaces of machine structures and parts have been widely developed. These methods have played a decisive role in the development of fundamentally new high-tech methods for the synthesis of coatings that have significantly competed with traditional methods of hardening and coating.

One of the promising methods underlying the creation of such technologies and coatings should be considered the method of cold gas-dynamic spraying, developed at the Institute of Theoretical and Applied Mechanics of the Siberian Branch of Russian Academy of Sciences [1-3].

At present, both in the Russian Federation and abroad, intensive investigations are being conducted, which are aimed at the development of gas-dynamic spraying technologies, the study of the structure-phase composition and functional properties of coatings. The diversity of the research conducted and the results obtained can be judged by at least detailed reviews [4–7], a considerable part of which is the development of new powder materials for the synthesis of coatings with an increased complex of properties.

Nevertheless, an analysis of the current state of research shows that works on the preparation and study of heterogeneous coatings strengthened simultaneously by nano- and micro-sized particles are of a single character. At the same time, taking into account the experience of creating bulk hybrid composites based on aluminum and its alloys [8–12, etc.], the creation of heterogeneous coatings
obtained by gas-dynamic spraying seems to be promising. However, the gas-dynamic spraying of powders with a particle size of less than 2 microns is difficult due to their inhibition in the compressed layer, which occurs when an ultrasonic gas stream flows onto an obstacle. In addition, when accelerating particles of a powder in a gas stream, there is a strong variation in velocity for particles of different sizes, while the nanoscale fraction can scatter without falling into the coating. Therefore, the use of polydisperse powder, which is a mechanical mixture of nano- and micro-sized particles, for gas-dynamic spraying seems to be unpromising. For the formation of heterogeneous coatings, in addition to micro-sized reinforcement containing nano-sized particles, it is necessary to use powder in the form of a mechanical mixture consisting of agglomerates of complex composition and ceramic particles. In this case, the agglomerates should be particles of a matrix material with nanosized reinforcement distributed in their volume, as well as micro-sized ceramic particles embedded in the agglomerates and located on the surface [13]. Use for gas-dynamic spraying of powders having such a structure, allows to obtain heterogeneous coatings with a uniform structure and a high level of mechanical properties [14]. Since the presence of nanoscale particles increases the mechanical properties of the matrix material [15], increasing the strength of fixing micro-size ceramic particles in the matrix. At the same time, the plasticity of the particles of the matrix material remains sufficient for the effective formation of a coating with a low porosity [16].

To obtain such powders, a method of mechanical processing in a high-energy ball mill can be successfully used, providing the synthesis of multicomponent powders with a particle size distribution necessary for gas-dynamic spraying (average particle size 15-20 μm) [13-16]. The obvious advantages of this method include the possibility of implementing the distribution of reinforcing particles, nanostructuring and dispersing of a matrix material within one technological cycle [17].

The aim of this work is to study the effect of the type of ceramic particles on the efficiency of gas-dynamic spraying and the hardness of hybrid coatings AlMg6 + 0.3wt.% C60.

2. Materials and Methods
Powder for cold gas-dynamic spraying was obtained by mechanical synthesis in a planetary ball mill for two technological stages. At the first stage, nanocomposite powder AlMg6 + 0.3 wt.% C60 was obtained. At the second stage, 10, 25, 30, 35, 50, and 70 wt.% α-Al2O3 or AlN were added to the obtained nanocomposite powder (hereinafter the content of ceramic particles in the powder is indicated) and the treatment was continued. The average particle size of α-Al2O3 and AlN was 18.5 and 5.9 μm, respectively.

The granulometric composition of the obtained powders was determined on a Microsizer-201C instrument. The morphology of the obtained hybrid powders and the formed coatings, as well as their microstructures, was studied using an ultra-high resolution auto-emission scanning electron microscope Zeiss Ultra Plus based on Ultra 55.

The resulting powders were sprayed onto sheet substrates from the steel of 1008 grade. For spraying was used installation of cold gas-dynamic spraying DYMET-404. The sputtering mode is stationary (sprayed to a point) at an air flow temperature of 450 °C, the distance from the nozzle exit to the substrate surface is 10 mm, and the exposure time is 15 seconds. The effectiveness of the deposition of the coating was determined by the magnitude of the growth of the coating, which was calculated as the difference in the mass of the substrates before and after the cold gas-dynamic spraying.

The microhardness of the coatings was measured on transverse thin sections by the Vickers method using a Shimadzu HMV-2 microhardness tester with a load of 245 mN and a holding duration of 10 s.

3. Results
As a result of processing in a planetary ball mill, hybrid powders were obtained. Figure 1 a, b shows typical SEM images of synthesized powders characterizing their morphology. The synthesized hybrid powders were a mechanical mixture consisting of agglomerates of complex composition and micro-sized ceramic particles. Agglomerates of complex composition were particles of a nanocrystalline
matrix material containing fullerenes embedded in them, as well as micro-sized ceramic particles located on the surface. It is noted that with an increase in the content of ceramic particles in the mixture, their concentration on the surface of the particles of the matrix material increases markedly.

![SEM images](image)

**Figure 1.** SEM images of AlMg6/C60 + Al2O3 and AlMg6/C60 + AlN powders: a, d – n = 10 wt.%; b, e – n = 30 wt.%; c, f – n = 50 wt.%;

Numbers indicate: 1 - agglomerates; 2 - ceramic particles

The study of the granulometric composition of the hybrid powder shows that it is characterized by the content of particles described by a bimodal distribution, with the first maximum with a particle size of about 5 microns corresponding to a ceramic reinforcement. It should be noted that with the adding of 10–70 wt.% of ceramic particles into the powder mixture, mechanical processing even for 15 minutes has a significant effect on the average size of the formed agglomerates, which decreases by more than 3 times and is ~17 µm. Depending on the content of ceramic particles, the average particle size of the synthesized powders is 12.3–17.8 µm and 8.2–16.4 µm for Al2O3 and AlN, respectively. It should be noted that 26.4% of AlMg6/C60 powder particles + 70 wt.% AlN have a size less than 5 microns. This circumstance allows us to predict that the deposition of this composition may be ineffective.
Figure 2. The effect of the content of ceramic particles on the efficiency of gas-dynamic spraying of powders (a) and the microhardness of the formed coatings (b)

Figure 2a presents the data characterizing the efficiency of gas-dynamic spraying of mechanically synthesized powder mixtures. The presence in the powder mixture of AlN particles contributes by 7–10% more effective growth of the coating compared to Al₂O₃. However, it should be noted that the use of Al₂O₃ particles as a ceramic additive allows to achieve good sputtering efficiency of powder mixtures with a high content of ceramic particles (70%). The highest sputtering efficiency, in the experiments performed, regardless of the type of ceramic particles, is achieved at 30-35 wt.% of ceramic particles in the powder.
mixture. A further increase in the content of ceramic particles leads to a decrease in the efficiency of cold gas-dynamic spraying.

Figure 2b shows the change in microhardness (obtained on transverse polished sections) of hybrid coatings depending on the content of ceramic particles in the powder. A comparative analysis of the data shows that addition of 10–70 wt.% Al$_2$O$_3$ into the powder leads to an increase in the hardness of the coatings from 262 to 387 HV. The hardness of coatings obtained by gas-dynamic spraying of powder containing AlN is slightly lower and varies in the range of 260-306 HV at 10-50 wt.% AlN.

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