Mechanical synthesis of agglomerated aluminum-based hybrid powders containing nano- and microsized filler

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Abstract. This paper reports on the obtained an agglomerated hybrid powder for gas-dynamic spraying based on an aluminum alloy with dispersion-strengthened by nano- and microsized particles by mechanical synthesis in a planetary mill in two stages. Based on the results of scanning electron microscopy, a model for the formation of hybrid powder particles is proposed. The influence of the micro-dimensional filler concentration on the powder particle size distribution is considered. The structural-phase composition of the powder is characterized by X-ray phase analysis.

1. Introduction
Engineering of surface layers is one of the promising methods to improve the functional properties of structural materials. An important place in the framework of this concept is the development and study of new coatings that provide formation of the specified functional properties due to directional formation of the structural-phase composition and architecture of the coating.

Powder coatings based on aluminum, considering their high corrosion resistance, are demand in mechanical engineering for application on the surface of structural elements and machine parts. One of the promising methods that can be effectively used for the application of hybrid coatings based on aluminum matrix is the method of cold gas-dynamic spraying. This method is characterized by a wide area of functional purpose, including both spraying of protective coatings, surface restoration, and prospects in the field of additive production.

For practical application, the most interesting is the use of aluminum alloys as base systems, such as Al–Mg. Alloys of Al–Mg system are widely used in many technical applications, especially for marine environments. Therefore, the synthesis and study of the functional properties of hybrid powder coatings based on this system is justified in practical point.

For the formation of heterogeneous coatings containing nanoscale particles in addition to microsized, it is necessary to use a powder in the form of a mechanical mixture of complex composition agglomerates and ceramic particles. In this case, the agglomerates have to be matrix material particles with nanosized filler distributed in their volume, as well as microsized ceramic particles embedded in the agglomerates and located on the surface [1]. The use of powders with such structure for gas-dynamic spraying allows obtaining hybrid coatings with a uniform structure and a high level of mechanical properties [2]. Since the presence of nanoscale particles increases the mechanical properties of the matrix material, the fixing strength in the matrix of microsized ceramic particles is also enhance. At the same time, the plasticity of the matrix material particles remains sufficient for the effective formation of coating with low porosity [3].
To obtain such powders, a method of mechanical processing in a high-energy ball mill can be successfully used. This method provides a synthesis of multicomponent powders with granulometric composition required for gas-dynamic spraying (average particle size 15-20 μm) [1-3]. The obvious advantages of this method include the possibility of implementing the processes of reinforcing particles distribution, nanostructuring and dispersion of matrix material within one technological cycle [4].

2. Methods and equipment
The process of aluminum-based powders synthesis with hybrid particles containing nano- and microsized filler includes two stages. A schematic representation of the synthesis process is shown in figure 1.

![Figure 1. Schematic representation of the synthesis process of powders based on aluminum with hybrid particles](image)

At the first stage, we carried out the mechanical processing of aluminum alloy AlMg2 in the form of granules with a diameter of 1-2 mm with the addition of 0.1 wt.% multi-walled carbon nanotubes (MWCNTs) decorated with nanoparticles of TiC [5].

Processing was performed in a planetary mill FRITSCH PULVERISETTE 6 with the use of a surfactant (1 wt.% stearic acid). For milling we used a ceramic cup and ceramic balls (ZrO2) with a diameter of 8 mm at a ratio of the loaded components mass to the mass of the milling media 1:15. The processing duration was 4.5 hours at a speed of 600 min⁻¹. At the second stage, we added 10, 30, 50 wt.% of Al₂O₃ (average particle size 18.5 μm) to the obtained powder and continued processing for another 15 minutes.

The surface morphology of the synthesized aluminum-based powders with hybrid particles containing nano- and microsized filler was studied by scanning electron microscopy on QUANTA 200 3D.

Granulometric composition of powders was measured at MICROSIZER 201C. The characterization of the structural-phase composition was carried out at BRUKER D8 ADVANCE.

3. Results and discussion
After the first stage of processing we obtained a composite powder based on aluminum alloy AlMg2, hardened by MWCNT with TiC nanoparticles decorating. Figure 2 shows the SEM-images of the composite powder. The SEM-images show that the powder particles have sizes varying in a wide range of about 5÷100 μm. For particles the irregular shape with a developed surface is characterized.

Nanoscale filler was also fixed on the surface of powder particles (figure 2, indicated by arrow). The length of the MWCNT-hybrid filler particles after mechanical processing was about 600 nm.

After the second stage of mechanical treatment, a powder with hybrid particles containing nano- and microsized filler was obtained. SEM-images characterizing the morphology of the synthesized powders depending on the concentration of microsized Al₂O₃ particles are presented in figure 3.

From the presented SEM-images it can be seen that an increase in the concentration of microparticles leads to a decrease in the particle size of the matrix material. At the same time, the obtained powders are a mechanical mixture consisting of agglomerates of complex composition and microparticles Al₂O₃. Agglomerates are particles of the matrix material AlMg2 containing TiC/MWCNTs embedded in them, as well as located on the surface, nano- and microsized particles Al₂O₃ (figure 4a). For agglomerates characterized by irregular shape with a developed surface. Formation of nanosized Al₂O₃ particles occurs during processing under the destructive action of milling media on the original microsized Al₂O₃ particles. For the depth estimation of ceramic particles entries into the matrix material, SEM-microscopy of cross sections of hybrid agglomerates was performed (figure 4b). The presented data clearly show the presence of ceramic particles not only on the surface of agglomerates but also inside them. The concentration of Al₂O₃ particles inside the agglomerates increases with the mass fraction of ceramic particles in the mixture.
Figure 4. SEM-images of the surface (a) and cross-section (b) of hybrid agglomerates

Figure 5a shows the analysis results of the particle size distribution of the synthesized powders. From the above data it can be seen that the introduction into the powder mixture more than 10 wt.% Al₂O₃ microsized particles has a significant effect on the particle size distribution of the powder being formed.

On X-ray diagrams (figure 5b) of the composite powder obtained at the first stage were recorded only diffraction peaks corresponding to the matrix material. The peaks of the filler, given its small volume fraction, were not recorded on X-ray. For hybrid powders, in addition to the matrix materials peaks, the presence of peaks corresponding to Al₂O₃ was noted.

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