The effect of ball milling in a planetary mill on aluminium particles microstructure and properties of cold sprayed coatings

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Abstract. Cold spraying allows one to obtain coatings without significant powder particles overheating. Hence, cold sprayed coatings have properties similar to that of sprayed particles. This paper shows results of the aluminium powder preparation for cold spraying. It is demonstrated that preliminary ball milling in the high-energy mill «Activator-2SL» has a significant impact on the particles shape and its structure. The ball milled particles have layered structure containing of a variety of the ultrafine aluminium particles.

1. Introduction
At present, coating deposition on machine parts by the thermal spraying and cold spraying becomes more significant part of technological processes in automobile, aircraft, heavy, medical and biological industries. The main function of coatings is to increase the durability of details and functional units, which work at high mechanical loads, enhanced temperatures, chemically aggressive media, etc. The coating is formed as a result of consistently collision of individual particles with the substrate. In process of spraying the basic physical parameters influence internal coating structure. These parameters include velocity, temperature, size, internal structure of particles, as well as temperature, material and condition of substrate surface [1-3]. For the methods of thermal spraying, heating above a material melting point is a distinctive feature. In contrast, the main feature of the cold spraying is ability of deposition without significant heating, which allows one to obtain coatings with properties similar to that of initial particles material [4].

As a rule, preliminary high-energy effect on initial material in a planetary mill leads to particle structure change. For example ball milling of powders in planetary mill leads to deformation energy storage throughout the volume of particles, appearing of point defects and dislocations in a crystal structure; surface cleaning as a result of destruction of oxide films and adsorbed layers that are barriers for chemical transformations, chemical reactions, etc [5]. Bearing this in mind, one could suggest that properties of coatings deposited from ball milled powder particles will be differ from physical, chemical and mechanical parameters of coatings obtained during the initial powder deposition.

The goal of this study is experimental research of the effect of aluminium particle ball milling in high-energy mill in dependence on milling time on its structure and structure of obtained cold sprayed coatings.
The following tasks have been accomplished to achieve the goal:
1. Ball-milling of powders during 15, 30, 45, 60 and 90 seconds at the constant ratio of balls weight to processed material weight and acceleration of grinding bodies;
2. Particle size composition and specific surface was determined; surface morphology and internal structure of ball milled and initial particles were investigated.
3. Cold sprayed coatings were obtained using initial and ball milled powder particles; its physical and mechanical properties were determined; its internal structure was investigated.

2. Materials and Methods
In this study the ASD-1 grade aluminium powder was used. Initial aluminium powder particles have spherical shape with a mean size of 18 µm. Figure 1 shows microphotographs of initial aluminium particles morphology and its cross section.

![Microphotographs of initial aluminium particles](image)

Figure 1. Microphotographs of main view (a) and cross section (b) of initial aluminium particles.

Ball milling of the initial powder was carried out in the high energy mill «Activator-2SL». Its main technical details: two cylinders and each of them has volume of 250 ml and inside diameter of 42.5 mm; weight of processed balls – 160 g for each cylinder, grinding bodies acceleration -117 g, processed material shot weight – 30 g; peak velocity of centroid axis is 1045 rpm, that of cylinders – 1550 rpm. The steel balls with diameter of 5 mm were used as grinding bodies. Classification of particles by sizes was conducted with vibration table using a set of the analytical sieves. Determination of histograms of volume and counting distributions was conducted using particle size analyzer (0.04 – 2000 µm) LS 13 320 (Beckman Coulter). Powder particle morphology was investigated using scanning electron microscope Evo MA15 (Carl Zeiss). The microhardness of the coatings was measured using an automatic microhardness meter DuraScan-50. Porosity of coatings was determined using Image Analysis Software and metallographic microscope OLYMPUS GX-51. Deposition was carried out at experimental cold spray setup developed at the ITAM SB RAS, using Laval nozzle of length 145 mm and critical and exit diameters 2.8 and 6.5 mm respectively. Air was used as working and carrying gas. Pressure in the stagnation chamber was constant and it was equal to 3 MPa. Temperature in the stagnation chamber was equal to 570 K. The substrates for cold spraying were aluminium plates of 3 mm in thickness. Standoff distance was equal to 30 mm, nozzle travers speed was 25 mm/s.

3. Results and Discussion
Initial aluminium powder was ball milled during 15, 30, 45, 60, 90 and 120 seconds. After the each ball milling powder was divided into two fractions of sizes smaller and greater than 90 µm. Weight content of each fraction was determined. It was established that the increase of ball milling time leads
to enhance of weight content of particles of size greater than 90 µm. Figure 2 shows the change of weight content of particles of size greater than 90 µm depending on ball milling time. The separation of the particles into two fractions is caused by the application of these powders. The particles of the main fraction with the sizes smaller than 90 µm are widely used in thermal spraying, detonation spraying and cold spraying. The particles of sizes larger than 90 µm in the shape of deformed disk can be processed in the plasma to obtain the powder consisting of micron sized spherical particles. At present, these particles are desirable for additive technologies, laser cladding and other areas of powder metallurgy.

![Figure 2](image)

**Figure 2.** The change of weight content of particles of size greater than 90 µm depending on the ball milling time.

When a grinding body interacts with a particle, the kinetic energy is consumed on the plastic deformation of the particle (up to 95% [6]) with the release of a large amount of heat. As the milling time increases, the amount of energy introduced into the material increases. This leads to an increase in the heat released during particle deformation. Heat does not have time to distribute, as a result of which there is a rapid increase in the temperature of the entire system. The material is heated to a temperature close to the melting point or to its melting [7]. The particles having a temperature close to the material melting point can be plastically deformed and easily interact with each other. Increase of their size is occurred as a result of granulation process by the method of rolling. Melting of aluminium particles is observed with a ball-milling during the 120 seconds.

Based on the research of particle size distribution for initial and ball milled aluminium particles, volume bar chart and counting bar chart of particle size distribution were plotted. Figure 3 shows the dependences of mean \(d_{\text{mean}}\), \(d_{10}\) and \(d_{90}\) sizes on the ball milling time of initial particles, where \(d_{10}\) – the diameter a which 10% of the sample mass is comprised of particles with a diameter less than this value; \(d_{\text{mean}}\) – the diameter of the particle that 50% of a sample mass is smaller than and 50% of a sample mass is larger than; \(d_{90}\) - the diameter a which 90% of the sample mass is comprised of particles with a diameter less than this value. It can be seen from figures 3 and 4 that the increase of mean particle size during the ball milling characterizes the formation of agglomerates, consisting of ultrafine aluminium particles. Morphology and cross section of ball milled particles is presented in figure 4.
Figure 3. Size distribution of the initial and ball milled aluminium powders: (a) volume and (b) counting distribution of particles.

Figure 4. Microphotographs of main view (a) and cross section (b) of ball milled aluminium particles.

To study the internal structure of coatings, cross sections of samples were prepared. Internal structure of coatings is presented in figure 5. The feature of the coatings is a layered structure with clearly defined boundaries formed by deformed particles, which indicates a high particle velocity before collision. It is clear that microstructure of individual particles in the coating obtained from agglomerated particles is similar to that of sprayed particles (figure 4).

Figure 5. Cross-section of coatings obtained by cold spraying of initial particles (a) and particles ball milled for 30 s (b).
Microhardness tests of coatings sprayed from both the initial powder and ball milled powder are shown in Table 1 (results were averaged over 10 measurements). Table 1 shows also porosity of cold sprayed coatings. One can see that porosity and microhardness of coatings produced from ball milled and initial powders are almost the same.

### Table 1. Parameters of cold sprayed metal coatings.

| Coating                                      | Porosity, % | Microhardness, HV<sub>0.1</sub> |
|----------------------------------------------|-------------|----------------------------------|
| Initial powder                               | 4.2         | 59±3.5                           |
| Ball milled powder during 30 s; sizes of particles is smaller than 90 µm | 4.14        | 61.45±9.15                       |

### 4. Conclusions

In this study experimental research of the ball milling time effect on the particle size distribution and internal microstructure of particles was conducted. It is shown that increase of ball milling time leads to enhance of system temperature (ball - particle), resulting in granulation process by the rolling method. The weight fraction of particles, which is greater than 90 µm in size, increase. When ball milling for 120 seconds the processed material reaches the melting point. It is established that microstructure of individual splats of cold sprayed coatings inherits features of initial particles microstructure.

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