Obtaining composite particles of Ti – 25 wt.% TiN powder by mechanical processing in a planetary mill for using in cold and thermal spray

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Abstract. In this work, with a change in the time of mechanical processing in a high-energy planetary mill, composite particles were obtained from a mixture of titanium powders (grade PTS – 1) and titanium nitride powder in the size range from 10 to 100 μm . It was shown that ultrafine refractory particles of titanium nitride are uniformly distributed over the volume of agglomerates. It is assumed that the spraying of the composite powder by the method of cold or thermal spraying will increase the homogeneity of the distribution of refractory inclusions in the coating. The structure and morphology of the composite particle surface were studied, their X-ray diffraction patterns were obtained, and elemental analysis was carried out. It was found that during mechanical processing of the material, titanium nitride particles act as a surfactant and prevent the growth of agglomerates. It was shown that after 3 minutes of mechanical processing of the powder mixture, titanium nitride is uniformly distributed throughout the entire volume of the composite particle.

1 Introduction

The problem of improving the quality of materials and products, increasing the resource of their work and improving the physical and mechanical characteristics is especially relevant for modern development of industry and technology. One of the ways to solve it is to apply multifunctional protective and volume hardening coatings. In addition, the combination of several technological solutions looks promising. Adding to the starting material from 0.01 to 0.5 weight fraction of ultrafine particles based on oxides, borides, carbides and nitrides made it possible to achieve significant results, both in casting and in the welding industry [1–10]. An important point is that the initial ultrafine particles of refractory compounds are poorly wetted by the melt, which is an important problem. To solve it, the particles are covered with an additional layer of metal. Particles are plated with metal in the ratio: three weight fractions of metal and one weight fraction of ultrafine particles proved to be the most effective. Taking into account the peculiarities of the spraying technology, the

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particles should have a size of 20 to 90 microns. There are several methods for obtaining such composite particles: mechanical processing of powders in high-energy mills of the planetary type; obtaining composite compacts by high-temperature sintering from a mechanically mixed powder mixture; using spray drying; synthesis of chemical compounds by self-propagating high-temperature synthesis; vacuum arc cladding of particles with metal films (Ti, Al, Cu, etc.). It is also worth noting the method of treatment in a plasma jet [11, 12]. Specially prepared composite granules are subjected to plasma treatment in a controlled atmosphere of argon or a mixture of argon with helium. As a result, composite particles of a spherical shape are obtained with refractory ultrafine inclusions uniformly distributed over the volume. This method allows not only to increase the wettability of modifiers, but also to protect them from direct interaction with the external environment, thereby increasing the shelf life.

The purpose of the work is to determine the optimal mode of machining a mixture of titanium and titanium nitride powders containing 25 wt. % of ultrafine particles of the solid phase with the size of the resulting composites from 20 to 90 microns.

2 Experimental technique

During the experiment, we used: titanium powder (grade PTS – 1) and titanium nitride powder. A general view of the initial particles obtained with an Evo MA 15 electron microscope (Carl Zeiss) using a backscattered electron detector equipped with an attachment for micro X-ray spectral analysis (Oxford Instruments X – Max 80 mm², UK) is shown in Fig. 1.

Fig. 1. General view of the initial particles of titanium (a) and titanium nitride (b) powders.

Fig. 2. Volumetric distribution of initial particles by size: (1) – titanium nitride powder, (2) – titanium powder.

Using a laser diffraction analyser LS 13 320 (Beckman Coulter), the particle size distribution of the powders was studied and a histogram of the volumetric particle size distribution was obtained (Fig. 2).

Powder mixture with the addition of 25 wt. % of ultrafine particles of titanium nitride was ball milled in a high-energy planetary mill "Activator – 2SL" with water cooling at centrifugal acceleration of grinding bodies – 117 g in air atmosphere. The processing parameters were as follows: the weight of the loaded balls – 160 g for each vial; the weight of the loading of the processed material was 20 g; steel balls with a diameter of 5 mm were used as grinding bodies. Processing was carried out for 1, 3, 6, 9 and 12 minutes. X-ray diffraction patterns of ball milled particles were obtained on a D8 ADVANCE diffractometer (Bruker Corporation, USA) using monochromatic $\text{CuK}\alpha$ radiation.

3 Results and Discussions

Figure 3 shows the experimental dependences characterizing the change in the volumetric size of particles of a powder mixture of titanium and titanium nitride, depending on the time of their mechanical processing. When the material of the particles is deformed, due to collision with the grinding bodies, it heats up, the particles of titanium become more plastic and begin to form agglomerates, which leads to an increase in their size. Ultrafine particles of titanium nitride work as a surfactant: after initial mixing, they prevent further growth of agglomerates and allow, with an increase of the ball milling time (from 3 to 9 minutes), to achieve a decrease in the volumetric composite particle size [13].

Fig. 3. Changes in the volumetric particle size depending on the ball milling time.

The general view of the powder particles after processing for 1, 3, 6 and 9 minutes is shown in Figure 4. In the ball milling time interval from one to three minutes, the components of the mixture are mixed with a change in the shape of the titanium particles. After three minutes of mechanical processing, agglomerates are formed, in which ultrafine refractory particles of titanium nitride are distributed both over the surface and in their volume (Fig. 5). It should be noted that at long times of machining (9 minutes or more), titanium particles reach the melting point, as in [14], which leads to a significant increase in material loss (from 5–10% at 1–6 minutes to 40 % at 9 minutes) due to sticking and adhesion of the material to the grinding bodies and the walls of the vials. Thus, the composite particles obtained after 3 minutes of mechanical processing have an average size of 38 microns, and the mass fraction of particles of the fraction 20–90 microns is more than 60 %.

Fig. 4. General view of the mechanically processed powder mixture for 1 minute (a), 3 – (b), 6 – (c) and 9 minutes – (d).

Fig. 5. Elemental analysis of a cross section (a, b, c) of mechanically processed particles of a powder mixture of the composition Ti - 25 wt. % TiN for 3 minutes, obtained using scanning electron microscopy: a) - the analyzed area; b - titanium; c - nitrogen.

The X-ray diffraction patterns (Fig. 6) show that the obtained peaks of titanium and titanium nitride almost coincide with the tabulated values. During ball milling there is a constant deformation of the particles, which changes their structure and surface shape. In the agglomerated particles, non-equilibrium defects and strains of the crystal lattice are formed. This is reflected in the change in the X-ray diffraction patterns: a shift and broadening of the main titanium peak to the region of smaller angles occurs, which is explained by the compression of the unit cell and, accordingly, a change in the grain size (Fig. 6b).

Fig. 6. X-ray diffraction patterns of mechanically processed powder mixtures (a), values of the region of coherent scattering of titanium (b) depending on the ball milling time: 1 – 1 min, 2 – 3 min, 3 – 6 min, 4 – 9 min, 5 – 12 min.

Figure 7 shows the volumetric particle size distribution for a powder mixture containing 1, 15, 25 and 40 wt. % when processed for three minutes. It is seen that an increase in the weight concentration of titanium nitride in the processed powder mixture significantly reduces the size of agglomerates. Consequently, it is necessary to carry out additional research to establish the dependence of the influence of the weight concentration of titanium nitride in the powder mixture on the time of its mechanical processing, all other things being equal.

Fig. 7. Changes in the volumetric particle size, depending on the concentration of titanium nitride powder in the processed powder mixture for three minutes.

4 Conclusion

Effect of the time of mechanical processing on characteristics of the particles of a mixture containing titanium powder and 25 weight percent of titanium nitride powder has been studied. It was shown that after three minutes of processing the powder mixture, ultrafine particles of titanium nitride were uniformly distributed throughout the entire volume of agglomerates, indicating effective cladding and an increase in wettability, due to which they can be used as modifiers in casting, as well as in the cold gas-dynamic and gas-thermal spraying method. An increase in the weight concentration of titanium nitride in the processed powder mixture significantly reduced the size of resulting agglomerates.

Consequently, it is necessary to conduct an additional study on the effect of the weight concentration of titanium nitride in the powder mixture depending on the time of its mechanical processing, all other things being equal.

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