Influence of grinding on service properties of VT-22 powder applied in additive technologies

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Abstract. Powder of titanium alloy (VT-22) produced by plasma-spraying was subjected to grinding to obtain powder with size less 100 microns. These powders were sprayed by plasma unit using two types of gases, namely, air and air with methane (spraying in water and sputtering of coating on steel support). Influence of grinding time on yield of powder of required fraction was studied. Morphology and phase composition of the grinded powder and plasma sprayed one were under investigation. In the result of experiments, it appears that the grinding time genuinely influences the chemical and phase compositions, but there is no effect on physical-processing properties. For powders after plasma spraying some changes of non-metal elements content were detected by chemical analysis. Using gaseous mixture of air and methane in plasma spraying unit leads to formation of a new phase in the powder according X-ray diffraction data.

1. Introduction
Additive technologies e.g. selective laser melting and sintering, plasma spraying, and other are the production methods of 3D objects with predetermined mechanical properties using metal powders. Such technologies provide a possibility to manufacture different components having complex geometry in accord with technical requirements for product and mechanical properties of material.

The key requirements for powders applied in additive technologies are particles sphericity and dimensions. In references [1,2] the required particles dimensions are within the range from 20 to 100 microns.

In the present work the powder of VT-22 titanium alloy (Ti-5Al-5V-5Mo-Fe-Cr) were chosen as study subject. This titanium alloy is the widely used structural material. The powder was produced by plasma spraying of rotating electrode.

The dimensions of obtained particles were larger than required 100 µm. This raises the question of possibility to grind the particles down to target dimensions with further influence study of grinding duration on powder chemical and physical-processing properties.

Aim of the work was to study the influence of mechanical grinding process and plasma-forming gas composition on chemical and phase compositions of titanium-containing powder applied in additive technologies.
2. Research methods

The powder obtained by plasma spraying was subjected to grinding in IV-micro vibro-grinder during 15, 30, 45, and 60 minutes. Depending on grinding time the yield of powder with required fraction (from 20 to -100 microns) was determined by sieve analysis in accord with 18318-94 State Standard. After grinding, a degree of sphericity and symmetry of powder particles was estimated using Camsizer XT (Retsch Technology Gmbh, Germany). Carl Zeiss EVO40 XVP scanning electron microscope was used for morphology studying of VT-22 powder before and after grinding.

Physical-processing properties of initial powder and after grinding were measured according to the following State Standards: 19440-94 - the tap density, 25279-93 - the density after shake down, 20899-98 - the fluidity.

Phase composition was determined using X-ray diffractometer D8 ADVANCE (Bruker AXS, Germany).

The obtained powders were treated by Corvette 4 (Russia) plasma spraying unit using two types of plasma-forming gases: air and air+methane.

3. Results and discussion

For investigation the powders of two fractions (+100) and (-100) were used. Powder samples (+100) of 100 g were subjected to grinding in vibro-grinder. After grinding during different intervals of time, the amount of powder of required fraction was determined by sieve analysis, than the average values were calculated. The results are shown in Fig. 1.

![Fig. 1. Yield of powder of required fraction versus grinding time.](image)

The longer time of grinding is the higher powder yield of required powder is.

Assessment of sphericity and symmetry of powder particles after grinding was carried out using Camsizer XT, besides the comparison with the initial powder of (-100) fraction.

The longer time of grinding is, the slightly higher the degree of powder particles sphericity is, but the degree of symmetry is almost independent on grinding time. The SEM images prove the results concerning the sphericity of particles before and after grinding (Fig. 2).

The values of physical-processing properties of initial sieved powder (-100) and after grinding (-100) are similar (Table 1).
Figure 2. Surface morphology of VT-22 powder particles. 
(a) - before grinding, (b) - after grinding. Magnification − 1000.

Table 1. Physical-processing properties of VT-22 powder

| Initial state after sowing, (-100) | Apparent density, g/cm³ | Fluidity, g/s | Tap density, g/cm³ |
|-----------------------------------|--------------------------|----------------|-------------------|
| After grinding, granulometric composition (-100) | 2.72                      | 2.94           | 3.02              |
|                                    | 2.74                      | 2.91           | 3.04              |

It was defined by chemical analysis of initially sieved (-100) and grinded powders that content of metal constituents in alloy is independent on grinding time. While the longer grinding time is, the higher content of non-metal components is. This can be attributed to the fact that the longer grinding time is, the higher temperature of powder is. Hence, the content of non-metal constituents is increased due to ability of titanium alloys to adsorb gases at elevated temperatures.

It was established by X-ray diffraction analysis that the main phase of initial sieved (-100) and grinded powders is β-Ti solid solution. Whereas the longer grinding time is, the higher percentage of α-Ti solid solution is. Likewise the content of titanium nitride and carbide (Ti3N1.29 and TiC) is gradually increased, however most likely that after 60 min grinding the nitride and carbide change structure and Ti4N2.33 and TiC0.3N0.7 are formed.

Table 2. Phase composition of VT-22 powder in initial state and after grinding of (-100) fraction

| Time of grinding, min | Phase content | β-Ti, % | α-Ti, % | TiC, % | Ti3N1.29, % | Ti4N2.33, % | Ti2CN, % |
|-----------------------|---------------|---------|---------|--------|-------------|-------------|----------|
| Initial powder after sieving (-100) | the balance | 4.0     | 1.5     | 2.0    | -           | -           | -        |
| 15                    | the balance   | 6.0     | 1.0     | 4.0    | -           | -           | -        |
| 30                    | the balance   | 8.0     | 2.5     | 5.0    | -           | -           | -        |
| 45                    | the balance   | 9.0     | 2.0     | 6.0    | -           | -           | -        |
| 60                    | the balance   | 10.0    | -       | -      | 5.0         | 2.5         |          |
The obtained powders were treated by Corvette 4 plasma spraying unit using two types of plasma-forming gases: air and air with methane. The powders were quenched in water. In the first case an insignificant increase of content of non-metal elements (carbon, nitrogen, oxygen) in powders treated in plasma spraying unit using air was detected by chemical analysis. In the second case there was detected some growth of carbon and nitrogen content, whereas the oxygen content was increased significantly. According data collected from X-ray diffraction there was no change in phase composition of powder (in comparison with initial one) after application in air formed plasma. While there was detected a new phase, mostly comprised of titanium oxide mixture, when gaseous mixture of air and methane was applied.

Figure 3. Microstructure of VT-22 powder particles after plasma spraying using (a) - air, (b) - air + methane. Magnification – 200.

Coatings of the VT-22 powder sprayed by plasma on steel substrates are going to be investigated thoroughly. Now they are subjected to hardness, micro hardness, adhesion (three-point bending), corrosion-resistance (in chamber of salt fog) tests.

4. Conclusions
1. The longer time of grinding is, the higher powder yield of required powder is.
2. The longer time of grinding is, the slightly higher the degree of powder particles sphericity is, but the degree of symmetry is almost independent on grinding time.
3. There are differences in chemical and phase compositions of initial and grinded powders. The increase of oxygen and nitrogen occurs depending on the grinding time. Hence, it is recommended to grind the powder in a protective atmosphere.
4. Using air in plasma spraying unit the phase composition of powder is stable, but using gaseous plasma-forming mixture of air and methane leads to significant oxidizing of powder, so a new oxide phase is formed.

Acknowledgement
The authors acknowledge Ural Branch of Russian Academy of Sciences for financial support (research grant № 15-17-3-41). The study was carried out using equipment of the Ural-M Shared Research Center.

References
[1] Krashaninin V A, Il’inykh S A, Chusov S A and Gelchinskiy B R 2015 Sub- and supersonic plasma spraying of metal powders and production of combined protective coatings //
Proceedings of 11th International Scientific-Technical Conference devoted to State-of-the-art metal materials and technologies (SMMT’15) (Saint-Petersburg: Polytechnic university) P.1103-1114.

[2] Zlenko M A, Nagaycev M V and Dovbysh V M 2015 Additive technologies in machine-building (Moscow: NAMI), 220p. (in Russian)

[3] Leontiev L I and Gelchinsky B R 2014 Proc. VI Int. Conf. on New perspective materials and technologies for production (Volgograd: State technical University) p 278 (in Russian)

[4] Illarionov A G 2014 Technological and service properties of titanium alloys A G Illarionov and A A Popov (Ekaterinburg: Ural State University) 137(in Russian)

[5] Froes F H 2015 Titanium: Physical Metallurgy, Processing and Applications (Ohio: ASM International®Materials Park) 416

[6] Chumakov D M 2014 Electronic journal Trudy MAI 78 1 (in Russian)

[7] INTRODUCTION TO ADDITIVE MANUFACTURING TECHNOLOGY/A guide for Designers and Engineers/ European Powder Metallurgy Association/ www.epma.com/am

[8] Exploring Chemical and Thermal Non-equilibrium in Nitrogen Arcs. S Ghorui and A.K. Das, Journal of Physics: Conference Series 406 (2012) 012012

[9] M N Zakharov O F Rybalko S A Il’inykh S A Chusov and A V Dolmatov 2016 XX Mendeleev congress on general and applied chemistry The XX Mendeleev Congress is held under the auspices of the International Union of Pure and Applied Chemistry (Volgograd: Volgograd State Technical University) Vol 2 pp 214-215