Investigation of Ti/TiB₂ System Composite Coatings Sprayed by Microplasma Method

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Abstract. The present work studies Ti/TiB₂ system of metal matrix composite powders and coatings obtained on their basis by microplasma spraying. Ti/TiB₂ is the system of potentially perspective materials being operated in aggressive environments. Morphology of the microgrinding of the composite powder and coating was investigated by a scanning electron microscope, with the distribution assessment of the reinforcing component. The phase composition and its evolution in the composite powder and coating were also studied.

1. Introduction
In the modern metallurgy much attention is paid to the creation of new composite powders based on a combination of various metallic and non-metallic components for spraying functional coatings with an advanced set of properties. In some cases, such coatings have higher physical and mechanical properties than coatings applied from traditional materials. Novel materials development for structural and functional purposes with an advanced level of properties is a topical challenge for a number of branches of modern industries, such as the branches of mechanical engineering in particular. Creation of metal matrix composites (MMC) of new classes allows us to achieve the necessary level of physical, mechanical and operational properties of coatings for parts and assemblies for products that meet modern requirements. When developing MMC, researchers’ interest is currently focused on oxygen-free refractory compounds, with borides of transition metals and boride based alloys as an an important part [1-4].

Promising materials are titanium borides with high hardness, heat resistance, wear resistance, molten metals resistance, electrical and thermal conductivity combined with low density. However, despite a number of useful properties, pure titanium borides are not widely used in the technology due to the high fragility and complexity of the process of direct application to products. Therefore, it is advisable to use titanium borides with a metal bond and adapt the main deposition methods to the developed materials [5-10].

Titanium powder is actively used as a metal base, due to its high physical and mechanical properties, low specific gravity, and high corrosion resistance [11-14].

Previously, a method for creating a composite powder of the Ti/TiB₂ system was investigated, as well as the properties of the resulting powder and its coating had been studied. The morphology and fractional composition of synthesized powders with different mass content of titanium diboride were studied and their deposition was carried out by the microplasma method. Morphology, hardness and porosity were studied in the obtained coatings.
The purpose of this study is to investigate the evolution of the phase composition of the composite powder of the Ti/TiB$_2$ system at microplasma sputtering of the composite coating, including the morphology of composite particles, distribution of the reinforcing component.

2. Materials and equipment

The following was selected as the objects of study:

- Composite powder of the Ti/TiB$_2$ system with different mass content of titanium diboride (10%, 20% and 30%), that correspond composite powder Nos. 1, 2 and 3. The size of the composite powder is in the range from 10 to 40 µm.

- Coatings formed by the microplasma method from composite powders of the Ti/TiB$_2$ system with different mass contents of titanium diboride (10%, 20% and 30%) [15-16].

For the mecanosynthesis of composite powder of the Ti/TiB$_2$ system, a vibrating bowl attritor IVCH-3 was used, with a rotation mode of the internal elements of the bowls at a speed of 1200 rpm, for 3 minutes. Sputtering was carried out by the microplasma method using UGNP-7/2250 and Kawasaki FS003N robot manipulator. The developed powders were sprayed using microplasma. The essence of the method is that the powder used falls into the plasma jet, is partially melted and transferred to the substrate [17-19].

When studying the distribution of titanium diboride in composite powders of the Ti/TiB$_2$ system with different percentages of titanium diboride (10%, 20%, 30%), as well as the microstructures of coatings based on them, microsections were made in a current-carrying resin. The studies were carried out by LYRA 3 XMH scanning electron microscope (SEM), TESCAN. The principle of operation of the SEM is based on the use of effects that occur when the surface of objects is irradiated by a finely focused electron beam – a probe. Reflected and absorbed electrons are used to obtain an image of the sample surface. In the conducted study the survey was carried out in the mode of registration of backscattered electrons (BSE).

The phase composition was studied by Bruker D8 Advance X-ray diffractometer. The voltage on the X-ray tube was 40 kV, the current was 40 mA. The images were taken in the scanning mode are: the scanning interval of 2 theta was 15-153 degrees, the scanning step was 0.02 degrees, the shutter speed at the scanning point was 1 second, the sample rotated at a speed of 15 rpm.

3. Research results

For the conducted research, microsection with a composite powder and a planar coating sample with a transverse arrangement in the section were developed.

The images of microsection obtained on the SEM allow us to characterize the distribution pattern of titanium diboride particles in the matrix powder, Figure 1.

![Figure 1. Images of cross-sections of composite powder particles containing titanium diboride: 1) 10%; 2) 20%; 3) 30.](image)

The figures show that after synthesis using IVCH-3 titanium diboride particles were embedded in the matrix component. Titanium diboride particles are represented by dark rounded inclusions ranging in size from 0.5 to 4 µm, without interface with the matrix. According to the data presented, it can be
argued that the matrix and reinforcing powders are consolidated in all batches into a single mechanical system, the composite powder consists of particles with a fraction of 20-40 µm and is suitable for microplasma spraying.

The Figures 2, 3, 4 show images of transverse sections of coatings sprayed by the microplasma method from the studied powders.

![Figure 2](image1.png)

**Figure 2.** Microsections of the coating made of composite powder (10%) № 1, a) the transverse section of the coating (20 µm), b) the transverse sections of the coating (2 µm).

When spraying composite powders by the microplasma method, a partial penetration of the sprayed material occurs and the coating inherits the particles of the reinforcing component without degradation of the microstructure.

![Figure 3](image2.png)

**Figure 3.** Microsections of the coating made of composite powder (20%) № 2, a) the transverse section of the coating (20 µm), b) the transverse sections of the coating (2 µm).

In Figure 3 (a) the peeling of the adhesive layer occurred during cutting and overheating of the sample.

The presence of hardening particles in the coatings is determined by rounded inclusions with a characteristic size and a dark contrast. With an increase of titanium diboride in the composite powder, the number of corresponding structural components in the sprayed coatings increases too.
Figure 4. Microsections of the coating made of composite powder (30%) № 3, a) the transverse section of the coating (20 µm), b) the transverse sections of the coating (2 µm).

The preservation of titanium diboride particles during the deposition process and their presence in the coating leads to an effective increase in operational properties compared to traditional titanium coating [20].

The phase composition was studied from the surfaces of composite powders pressed into the cuvette and planar samples of sprayed coatings. A characteristic diffractogram of a composite powder of the Ti/TiB₂ system is shown in Figure 5 as the example of a composite powder with 20% titanium diboride.

Figure 5. Diffractogram of synthesized titanium powder and titanium diboride (20%).

The interpretation of the diffractograms shows the distinct presence of the reflexes Ti and TiB₂ (the crystal lattice of titanium and titanium diboride is hexagonal), as well as the presence of titanium oxide TiO₂ (rutile). It is known that up to a temperature of 800 °C, the oxide layer consists exclusively of TiO₂, with a rutile structure [21, 22].

The results of the study of the coating formed by spraying a composite powder are presented in the diffractogram, Figure 6.
The interpretation of these diffractograms shows the presence of Ti and TiB reflexes, as well as the presence of titanium oxide TiO$_2$ (rutile). It can be concluded that as a result of sputtering by the microplasma method, due to temperature exposure, TiB$_2$ turns into TiB. Titanium diboride has a cubic face-centered grid. The physical and mechanical properties do not undergo significant changes, the hardness index of titanium diboride powder reaches approximately 35 GPa, with titanium boride of 32 GPa.

4. Conclusions

1. The study of microsections of composite powders of the Ti/TiB$_2$ system showed that titanium diboride is embedded in the matrix component and is evenly distributed in the volume of the composite particle. The reinforcing component is characterized by such parameters as a darker contrast compared to the matrix in the BSE images, the size and shape of the particles, with no explicit phase boundary between matrix and reinforcing component in the composite particle.

2. X-ray phase analysis revealed that titanium oxide TiO$_2$ in the rutile modification was present in the synthesized powders. Titanium diboride did not undergo a phase transition into TiB, during mechanical and chemical synthesis.

3. During microplasma sputtering of a composite powder of the Ti/TiB$_2$ system, the particles of the reinforcing component are transferred to the coatings without fusing with the matrix. The presence of reinforcing particles in the coating is identified by spherical areas with characteristic dimensions and color contrast.

4. X-ray phase analysis of the coatings showed the presence of TiO$_2$ in the rutile modification. Also, due to the use of the microplasma sputtering method, the TiB$_2$ phase is converted into TiB. This is caused by the temperature effect of the plasma jet during spraying, but does not entail a significant decrease in the physical and mechanical properties of the coating.

References

[1] Lepakova O 2000 Abstract of the dissertation of the Candidate of Technical Sciences Tomsk 26
[2] Antsiferova I 2004 Powdered titanium materials (Bulletin of the Orenburg State University) 198-202
Acknowledgments

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