Structure and properties of Ti-C-B coatings produced by non-vacuum electron beam cladding

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Abstract. Cp-Ti/TiB+TiC wear-resistance coatings produced by non-vacuum electron beam cladding of boron carbide and titanium powders are studied in the paper. The X-ray phase analysis of the composite coatings microstructure showed that titanium carbide and boride reinforcing particles are evolved during the process. The obtained data are in good agreement with results of optical and electron microscopy. Undissolved particles of the initial boron carbide powder are detected in the coatings. The microhardness test as well as wear resistance test of materials under conditions of loose abrasive particles are conducted. It is established that the precipitation of reinforcing particles improves the tribological properties of the composite coatings.

Keywords: Cp-titanium, Titanium carbide, Titanium boride, Electron beam cladding, Microstructure, Microhardness, Wear resistance.

1. Introduction

Titanium and titanium-based alloys are attractive materials for many industries due to its unique physical, mechanical and biological properties. However, titanium alloys are characterized by low tribological properties, high and unstable friction coefficient, heavy adhesive wear and tendency to seizure [1-3]. Using the technology of surface hardening can significantly improve the wear resistance of the titanium. One of the most effective methods is to form a composite coating on the surface of titanium alloys. Carbides, borides, silicides and intermetallic compounds are used as the reinforcing components in the coatings of this type. Production of titanium with distributed above-mentioned particles is possible by introducing these particles into the molten bath or in the synthesis process (in-situ) [3-10].

For the moment one of the most researched technologies for the production of such coatings is the laser treatment. Using the laser cladding techniques can significantly increase the hardness and wear resistance of the titanium alloys as well as thermal stability of the alloys [11, 12], [13].

Disadvantages of laser processing technologies associated with a metal high reflectivity and relatively low lasers efficiency. Special coatings are often applied to increase the absorptive capacity of the materials.

An alternative method of laser cladding technology is electron beam processing of powder materials [8-10]. Contrary to the laser, electron beam is a volume source of energy that penetrates deeply into the processed material. In this research the technology of electron beam cladding of powder mixtures in the air is used to improve the wear resistance of Cp-titanium. This technology is

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characterized by high performance and flexible control of processing parameters. Boron carbide powders are used as cladding components. Under electron beam influence on material, boron carbide particles are dissolved in the molten bath. In primary crystallization from supersaturated solution of carbon and boron precipitation of titanium carbide and boride crystals occurs. Ti-TiB-TiC composites combine high strength of carbides and borides as well as toughness and fracture resistance of the titanium substrate.

2. Materials and methods
Cp-titanium plates were used as substrates for the coating formation. The substrates dimensions were 100 mm x 50 mm x 12 mm. Titanium (30 wt.%) and boron carbide (20 wt.%) powders were used as the cladding materials. To protect the material being treated from the oxidation CaF₂ (40 wt.%) and LiF (10 wt.%) fluxes were added to the powder mixture during cladding. All components of the powder mixture were mixed together and uniformly applied to the surface of the titanium substrate. The density of the powder was 0.2 g/cm³. Electron beam cladding was performed in Budker Institute of Nuclear Physics SB RAS using an electron accelerator ELV-6 type. The scheme of the electron accelerator is presented in [14, 15]. The scheme of electron beam cladding is presented in [16, 17].

Table 1 summarizes the main parameters of electron beam treatment. During the experiment three types of samples were formed: Sample 1 was obtained using 27 mA electron beam current, Sample 2 - 28 mA and Sample 3 - 29 mA.

| The energy of the electron beam | 1.4 MeV |
| Scanning frequency            | 50 Hz   |
| The amplitude of the scanning | 25 mm   |
| The speed of the sample transfer | 25 mm/s |
| The distance from the nozzle to the substrate | 90 mm |
| The diameter of the beam       | 10 mm   |

To investigate the structural characteristics of the obtained materials an optical microscope Carl Zeiss Axio Observer A1m was used. Calculation of the volume fraction of the reinforcing particles was performed using software Image J image analysis. Chemical etching was carried out with a Kroll’s solution. To identify structural features of the cladded layers a scanning electron microscope Carl Zeiss EVO 50 XVP, equipped with microanalyzer Oxford Instruments X-Act EDX, was used. The phase composition of the material was examined using diffractometer ARL XTRA. X-ray exposure was performed in step mode using Cu Kα radiation.

A microhardness distribution in depth of the cladded layer was evaluated by five tracks, using the Vickers Woulpert Group 402 MVD hardness tester. The load on the diamond indenter was 0.98 N.

Tribological test of materials obtained by electron beam cladding was carried out in accordance with GOST 23.208-79 (analogue ASTM G65-04) under conditions of loosely fixed abrasive particles. The test configurations are given in [18]. River sand was used as an abrasive. The sand was loaded into a hopper and fed into the friction zone where an experimental sample was contacted with a rotating rubber roller. The load during the test was 44 N. The worn material mass loss was taken as the magnitude of sample wear.

3. Results and discussion
When implementing non-vacuum electron beam cladding on the surface of titanium substrates, layer with a thickness of 1.2 ... 1.4 mm is formed. The cross section of Sample 2 is shown in Figure 1 a. The cladded layers have a high quality. No pores and cracks were detected. However, undisolved particles of the initial boron carbide powder are found in the coatings. These particles aggregation are clearly identified in the lower coating region and are shown by arrows in Figure 1 a. The presence of individual particles of this type in the coating, obtained by laser treatment has been shown in [7]. High
heating and cooling rates of the material are the features of the laser and electron beam treatments. Thus the boron carbide particles were not completely dissolve. The reaction zone is forming during the diffusion of boron and carbon atoms were observed around the boron carbide particles (Figure 1 b, g). Hence, boron carbide particles have a good contact with the titanium matrix and do not fall out under friction.

**Figure 1.** The structure of the Ti-B-C coatings, analyzed by optical (a) and scanning electron microscopy (b-g) and the surface topography after wear tests (h-j).

The diffraction pattern taken from the middle of the cladded layer of the Sample 2 is shown in Figure 2. The main phases, formed in the surface layer of titanium during the electron beam cladding are titanium carbide, titanium boride and alpha-titanium. No boron carbide phase was detected. This explained by preferential location of these particles close to the heat affected zone (HAZ) (Figure 1 a, g). The volume fraction of the particles in the cladded layer is less than 2 % and 3 % in the Samples 3 and 1, respectively.
The volume fraction of the reinforcing phase in the cladded layers of the Samples 1, 2 and 3 has not been changed with increase of the beam current. This is due to the fact that in the process of cladding the base metal is exposed to a greater fusion penetration during the treatment at higher currents. Increase of the power input leads to more boron carbide particles melting. Thus, the volume fraction of titanium boride and carbide crystals in the deposited layer is about ~32%.

The microstructure of the samples is shown in Figure 1 b-g. There are a large number of initial particles of titanium boride precipitated in the shape of hollow hexagonal prisms (Figure 1 c-d). In [9] it is shown that under similar concentrations of elements in the Ti-B-C system the titanium boride crystals nucleate and grow firstly. These particles are characterized by the preferred crystal growth in one direction. When approaching the eutectic line, the formation of TiC-TiB eutectic takes place. Titanium carbide is evolved mainly in the shape of dendritic crystals. This is accompanied by the growth of the dendrites branches on the edges and inside the particles of titanium boride. The eutectic titanium boride has a fine needle-like morphology, while the needles are hollow as well. Near the heat affected zone due to lack of boron atoms thin defective crystals of titanium boride are formed (Figure 1 e).

Studies have shown that the maximum value of microhardness (~5900 MPa) is recorded in a Sample 1, obtained by cladding of the powder mixture at a beam current of 27 mA. Increase of the beam current leads to decrease of the average microhardness of the cladded layers. In the HAZ in all experimental samples a decrease in microhardness to 1600-1800 MPa is observed. The obtained data have a good correlation to tribological tests. Sample 1 has the lowest wear rate under conditions of loosely fixed abrasive particles (0.65 mg/min). When beam current is raised, a slight increase in the rate of wear is observed (0.74 and 0.66 mg/min for the Samples 2 and 3 respectively). The wear rate of commercially pure titanium is 2.79 mg/min. Figure 1 h - k shows SEM micrographs of the worn-out surface of the samples. On the surface of cp-titanium there is a typical adhesive wear surface characterized by deep grooves in the direction of wear (Figure 1 h). On the surface of the Samples 1 and 2 there are areas of selective material wear. Aggregations of undissolved particles of boron carbide are good barriers to the material intensive wear. According to the EDX analysis data, these particles have the following composition: 85.23 wt. % B, 12.28 wt. % C, 1.65 wt. % O, 0.11 wt. % Si and 0.73 wt. % Ti. In the Figure 1 j it can be seen that particles spalling is not observed in the process of wear. Wear of areas with no particles of boron carbide was more intensive. However, discontinuous grooves indicate on the presence of the reinforcing particles of titanium carbide and boride, preventing wear. Sample 3 has an almost flat wear surface (Figure 1 k). This is due to the absence of boron carbide particles aggregation in the middle zone and uniform wear of the coating.
4. Conclusions

By X-ray phase analysis and structural analysis of the coatings, produced by non-vacuum electron beam cladding, three phases were detected: titanium carbide, α-titanium and titanium boride. Highest wear resistance and microhardness were characterized for samples obtained by cladding with 27 mA beam current. The wear resistance of this sample is in ~ 4.4-times higher than that of commercially pure titanium.

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