Influence of Electron Beam Treatment Regimes on the Structure and Properties of Intermetallic Clads Obtained on Titanium Substrates

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Abstract. Intermetallic clads were obtained on the basis of the titanium workpieces. The cladding was carried out using a non-vacuum electron beam treatment technique. A powder mixture which contained 16.97 Al–28.27 Ti–41.07 CaF$_2$–13.69 LiF (wt. %) was used for cladding. Two regimes with the beam current of 16 mA and 18 mA were used. The beam current influenced significantly the structure and phase composition of clads. In case of the electron beam treatment with a beam current of 16 mA a lamellar structure consisted of a mixture of AlTi$_3$ and AlTi was fabricated. An increase of the beam current to 18 mA led to the formation of an AlTi$_3$ acicular structure. Microhardness and wear resistance of the intermetallic coating was significantly higher in comparison with cp-Ti. The maximum microhardness of clads was 480 HV. A wear rate of clads was 10 times lower than that of cp-Ti.

1. Introduction
Currently titanium and its alloys are widely used in many areas of industry such as airspace engineering, automotive industry and chemical engineering due to its enhanced corrosion resistance, high specific strength and plasticity [1, 2]. However, low wear resistance and high-temperature oxidation resistance of titanium alloys restrict significantly the areas of their application. They are almost unsuitable for fabrication of the elements of friction pairs and details, which operate at high temperatures (higher than 500 °C). Surface modification of titanium alloys by elements, entering into the reaction with titanium which results in the formation of intermetallic compounds, can be the reliable solution of the aforementioned problems. Particularly, coatings consisted of titanium aluminides are considered to be the most efficient due to a combination of such properties as high-temperature strength, oxidation resistance, low density and high hardness [3, 4]. The above-mentioned statement suggests that titanium aluminides can possess the enhanced wear resistance level in comparison with titanium alloys.

Nowadays many technologies can be applied to produce Ti-Al intermetallic coating on the surface of the titanium workpiece. Among them there are mechanical alloying [5], aluminizing [6, 7], different methods of deposition [8-10] and others [11-13]. However, technologies based on the application of highly concentrated energy flows such as laser [14-16] or electron beam cladding are supposed to be the most prospective. Application of these technologies allows fabricating thick layers (up to 3 mm per one pass) with a good adhesion to metallic substrates [17]. However, application of non-vacuum electron beam cladding for formation of Ti-Al intermetallic coatings has not been yet described in the
literature. Thus, the aim of this study was the investigation of the possibility of using the electron beam treatment for producing the titanium aluminide coatings. The influence of electron beam cladding regimes on the structure and properties of the cladded samples is discussed.

2. Materials and methods

Ti (98.69 wt. %) and Al (97.28 wt. %) powders and cp-Ti substrates were chosen as starting materials for cladding. Intermetallic coatings were fabricated by non-vacuum electron beam cladding using an ELV-6 industrial electron accelerator produced by Budker Institute of Nuclear Physics. The powder mixtures consisted of Al, Ti, CaF$_2$ and LiF were uniformly poured on cp-Ti substrates with a thickness of 12 mm. After that surfaces of the workpieces were heated by an electron beam. A velocity of a sample movement with respect to the electron beam was 10 mm/s. Transversal scanning with a frequency of 50 Hz and an amplitude of 25 mm was used to increase the area of the treated surface. A distance from the sample to the outlet was 90 mm. Electron beam cladding regimes are shown in Table 1.

| Powder composition, wt. % | Flux composition, wt. % | Beam current, mA | Initial energy of the electrons, MeV | Velocity of a sample movement, mm/s | Distance to the outlet, mm | Scanning frequency, Hz |
|---------------------------|-------------------------|------------------|-------------------------------------|-----------------------------------|------------------------|-----------------------|
| 16.97 Al – 28.27 Ti       | 41.07 CaF$_2$ – 13.69 LiF | 16               | 1.4                                 | 10                                | 90                     | 50                    |

Structural investigations of obtained materials were carried out by means of scanning electron microscopy (SEM) using a Carl Zeiss EVO 50 XVP microscope in a back-scattered electron regime. Preparation of samples was carried out using the following approach. Materials molded into the resin were grinded by abrasion papers of different grit (from 100 to 2500) using a Struers LaboPol-5 machine and polished by alumina with a grain size of about 3 µm. Final polishing was carried out using a colloidal silica solution.

The elemental composition of coatings was estimated by an INCA X-Act (Oxford Instruments) energy dispersive X-ray (EDX) analyzer. The investigation of the phase composition was carried out using an ARL X’TRA X-ray diffractometer (XRD). Diffraction patterns were obtained in a step mode with a step size of 0.05° and a dwell time of 6 s using Cu K$_{α1,2}$ radiation.

A WolpertGroup 402 MVD semiautomatic tester measured the microhardness level. A load on a diamond indenter was 100 g. Tribological tests in the conditions of dry sliding friction were carried out by an II 5018 friction machine according to GOST 23.204-78 using a “disc-on-plane” scheme. Dimensions of samples were equal to 20x10x10 mm. A load on a sample was 100 N and a disc rotation speed was 100 rpm. Preliminary preparation of samples was carried out by a surface grinder. Surface roughness of samples before tests was equal to 0.32 µm. The quenched carbon steel disk (HRC 55) was used as a counterbody. An outer disk diameter was 50 mm; its width was 10 mm. The dimensions of the wear crater were measured to estimate the wear rate.

3. Results and discussion

Structural investigations showed that thicknesses of the layers obtained by cladding at a beam current of 16 mA and 18 mA was 900 and 1200 µm, respectively. The increase of the layer thickness was obviously caused by the increase of the energy input in the material. The beam current can play a critical role in formation of a structure and a phase composition of the clads. The increase of the beam current causes the increase of the depth of the molten substrate. This in turn leads to a significant decrease of the concentration of alloying elements due to mixing with the substrate material. This statement could be easily confirmed using an EDX analysis. An average Al and Ti content in the coating obtained at 16 and 18 mA was: 48 Al – 52 Ti and 35 Al – 65 Ti (at. %), respectively.
The analysis of the Ti-Al phase diagram indicates that the aforementioned concentrations corresponded to different phases. In case of 48 at. % Al an AlTi compound with a wide homogeneity range (48...63 at. % Al) forms. In the range between 23 and 35 at. % Al the formation of an AlTi intermetallic compound should appear. In case of 35...48 at. % Al a mechanical mixture of two aforementioned compounds can be observed. Thereunder, we can suppose that the coating obtained at 16 mA contained \(\gamma\)-AlTi, while the electron beam treatment at 18 mA led to formation of the coating consisted of \(\alpha_2\)-AlTi\(_3\). For a more sophisticated and nuanced consideration of the structural and phase compositions of the coatings, the SEM and XRD analyses were applied.

The coating fabricated by the electron beam with a current of 16 mA possessed mainly a dendritic structure (Figure 2a). A difference in the contrast between dendritic arms and the interdendritic space can indicate the variation of the composition in these areas. It was mentioned in [19] that in the alloys containing 45...48 at. % Al the bright dendritic arms were enriched with Ti, while the interdendritic space consisted mainly of \(\gamma\)-grains. Microphotographs obtained at higher magnifications give the evidence that the coating had a lamellar structure. According to [19], such structure can be represented by the mixture of \(\alpha_2\)- and \(\gamma\)-aluminides, as confirmed by the XRD analysis data (Figure 1). This coating contained 48 at. % Al, which corresponded to the line between a \(\gamma\)-phase and the mechanical mixture of \(\gamma\)- and \(\alpha_2\)-phases on the phase diagram. Since the XRD analysis revealed the presence of two phases, then it can be concluded that formation of AlTi close to equiatomic composition occurred and excessive Ti was used to form AlTi\(_3\).

![Figure 1. Results of the XRD analysis of coatings fabricated on cp-Ti substrates by the electron beam cladding.](image)

The EDX analysis revealed segregation of P, Cu and Si (Figure 2a, Table 2) in the interdendritic areas. Their appearance was probably caused by the presence of the aforementioned elements in the initial powders.

The increase of the beam current led to a deeper melting of the material and consequently to stronger dilution of the alloying material in a substrate material. Thereby, a single phase coating consisted only of the \(\alpha_2\)-phase was formed (Figure 1). A quantitative analysis showed that AlTi\(_3\), which composed the coating, was enriched with aluminum. An acicular structure of the coating is shown in Figure 2b.
Figure 2. A structure of coatings cladded at \(a - 16\) mA, \(b - 18\) mA. Transition layers between the substrate and a coating obtained at \(c - 16\) mA, \(d - 18\) mA.

Table 2. The elemental composition of the local areas of intermetallic layers.

| Spectrum | Al, at. % | Ti, at. % | P, at. % | Si, at. % | Cu, at. % |
|---------|-----------|-----------|---------|---------|---------|
| 1       | 46.15     | 53.85     | –       | –       | –       |
| 2       | 47.56     | 45.45     | 4.97    | 0.35    | 1.68    |
| 3       | 47.58     | 52.42     | –       | –       | –       |
| 4       | 33.58     | 66.42     | –       | –       | –       |
| 5       | 23.72     | 76.28     | –       | –       | –       |
| 6       | 3.48      | 96.52     | –       | –       | –       |
| 7       | 36.42     | 63.58     | –       | –       | –       |
| 8       | 26.97     | 73.03     | –       | –       | –       |
| 9       | –         | 100       | –       | –       | –       |

The contrast of transition zones of both samples in SEM microphotographs was different from the substrate and a cladded material (Figure 2c, d). The transition layer between the base material and the coating obtained at 16 mA consisted of two interlayers which corresponded to AlTi3 enriched and depleted with aluminum (Figure 2c, Table 2). The transition area between titanium and intermetallic obtained at 18 mA corresponded to AlTi3 with almost a stoichiometric composition (Figure 2d, Table 2).

The microhardness and the wear resistance of the clads were measured to estimate the properties of the sample (Figure 3). It was revealed that the microhardness of the intermetallic layers cladded
according to the different regimes was 3.7...3.9 times higher compared to cp-Ti (Figure 3a). Besides, the microhardness of the coating obtained at 18 mA was slightly higher than the microhardness of the other coating. However, the wear resistance of the coating obtained at 18 mA was significantly higher, than that of the coating, obtained at 16 mA.

![Figure 3](image)

**Figure 3.** Microhardness of materials with cladded layers (a) and a volume of the material worn during the sliding friction tests (b).

Wear resistance of cp-Ti in the conditions of sliding friction was significantly lower compared to that of both coatings. The volume of the worn material equaled to 3 mm$^3$ was reached already after one minute of testing. The similar volume of the coating material (2.4...4.2 mm$^3$) was worn only after 10 minutes of friction.

**4. Conclusions**

Structural investigations of coatings obtained on cp-Ti substrates by non-vacuum electron beam cladding of a 16.97 Al – 28.27 Ti (wt. %) powder mixture according to different regimes revealed that a beam current value influenced significantly structure and phase compositions of cladded layers. Treatment by the electron beam with a current of 16 mA led to the formation of a two-phase lamellar coating consisted of AlTi and AlTi$_3$. An increase of a beam current to 18 mA led to the formation of a single-phase layer, which contained only AlTi$_3$. Formation of the intermetallic layers on the surface of cp-Ti led to a significant increase of its hardness (up to 480 HV) and wear resistance. The volume of the worn material in samples obtained at the beam current of 16 and 18 mA after 10 minutes of testing was 2.4 and 4.2 mm$^3$, respectively. Similar values of the worn material were reached after 1 minute of titanium testing.

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