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Structure and Properties of Ti-Nb-C Coatings Obtained by Non-vacuum Electron Beam Cladding

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Abstract. In this study the structure and properties of surface-alloyed cp-titanium layers obtained by non-vacuum electron beam cladding of niobium carbide powders were analyzed. A thickness of coatings fabricated by single-layer cladding was 1.3 mm. Cladding of the second layer led to an increase in the thickness by 0.8 mm. It was found that titanium carbide particles of different morphology acted as strengthening structural elements. The X-ray diffraction (XRD) analysis revealed the presence of α-Ti (α' Ti), β-Ti, and TiC in the cladded layer. The results of the energy dispersive X-ray (EDX) analysis indicated the presence of Nb in the titanium matrix as well as in the carbide phase. However, such phases as NbC and (Nb, Ti)C were not identified by the XRD analysis. Transmission electron microscopy (TEM) revealed zones containing an increased amount of Nb. The structure of these zones was represented by the β-Ti and ω-Ti precipitation. An average microhardness value of cladded layers was approximately 330 HV.

1. Introduction

A number of machine components and structural elements, which have critical applications, cannot be produced without using titanium and its alloys. These materials possess unique mechanical properties including a high specific strength, a low density and a high corrosion resistance [1, 2]. Titanium alloys are most widely used in aircraft, shipbuilding, space technology, medicine, chemical engineering and sports equipment manufacturing.

However, these materials have also a number of disadvantages, such as high cost and high activity at enhanced temperatures which lead to gas saturation. Moreover, low tribological properties can be classified as serious drawbacks which limit a wide application of titanium alloys. Titanium has a tendency to frictional seizure [1-3].

One of the efficient solutions of the aforementioned problem is based on the formation of high strength and wear resistant coatings on the titanium workpieces [4-9]. Among the variety of methods of surface hardening developed by now only a limited number can be recommended for treatment of titanium alloys.

Laser cladding is considered to be a one of the most widely applied and advanced methods of surface treatment [7-9]. A limitation of this technology is a high reflectivity of metals and consequently a low efficiency of laser radiation.

Electron beam treatment can be suggested as a method alternative to laser cladding [3-6, 10]. In contrast to a laser beam, an electron jet, being a volume energy source, penetrates into the depth of a treated material.
Industrials accelerators such as ELV-6 were developed in the Budker Institute of Nuclear Physics (Russia, Novosibirsk) to generate relativistic electrons [11]. These accelerators are equipped with a unique device providing the injection of electrons in the air atmosphere. Interaction of a beam with a treated material induces accelerated heating of a surface layer to temperatures higher than the melting point of this material. A molten pool, in which modifying components are dissolved, is formed in these conditions.

The technology based on non-vacuum electron beam treatment allows manufacturing large-scale parts with a high degree of precision. Their dimensions are limited only by the table geometry and a shop floor area. Using an ELV-6 industrial accelerator allows treating almost any metallic materials as well as carbides, borides, nitrides and other compounds [3-6, 10-13]. Operational experience of this equipment showed that the parts made of titanium alloys could be surface-hardened to a high depth [4-5, 10].

In this paper the structure and properties of coatings obtained by non-vacuum electron beam cladding of niobium carbide on titanium substrates were investigated.

2. Materials and methods

In this study VT1-0 cp-titanium plates with a thickness of 10 mm were used as substrates. A powder mixture consisted of 50 wt. % of niobium carbide and 50 wt. % of calcium fluoride was used as a surfacing material. CaF\textsubscript{2} provided protection of a molten pool from atmospheric oxygen. Technological experiments were carried out in the Budker Institute of Nuclear Physics (SB RAS).

The scheme of the cladding process is presented in [4]. A surfacing material in an amount of 10 g / 1 cm\textsuperscript{2} was uniformly distributed over a surface of the titanium workpiece and processed by an electron beam. A workpiece, mounted on a table, moved in the longitudinal direction with a speed of 25 mm /sec. In order to increase a volume fraction of a hardening phase in a coating, double-layer electron beam cladding of powder mixtures was employed. Cladding of the second layer was preceded by removal of a slag layer from the workpiece surface. Then the alloying mixture was applied to a surface and a treatment was repeated. The second layer cladding was accompanied by increasing the current (from 31 mA to 38 mA). Treatment of a wide area equal to the width of the workpiece (50 mm) was carried in a scanning mode with a scanning frequency of 50 Hz. The distance from the outlet to the sample was 90 mm. The values of specific surface energy introduced in the material during cladding of the first and second layers were 3.17 kJ/cm\textsuperscript{2} and 4.26 kJ/cm\textsuperscript{2} respectively.

Investigations of the structure of materials in the magnification range from x 25 to x 1500 were carried out using the Carl Zeiss Axio Observer A1m optical microscope. The structure of cladded layers was revealed by sample etching with Kroll’s solution. Particularities of structural transformations were investigated using the Carl Zeiss EVO50 XVP scanning electron microscope (SEM).

A fine structure of coatings was studied by transmission electron microscopy (TEM) method using a FEI Tecnai G2 20 TWIN microscope at an accelerating voltage of 200 kV. TEM samples were prepared using the Gatan Dimple Grinder 656 equipment and the Gatan PIPS 659 precision ion polishing system.

A phase composition of caddied layers was investigated using the ARL X'TRA θ-θ diffractometer. A copper tube (Cu K\textsubscript{α}) was used as a source of X-ray radiation. The recording of diffraction patterns was carried out in a step mode.

The microhardness of materials was estimated using the Wolpert Group 402 MVD semi-automatic microharness tester. The load on a diamond pyramid was 0.98 N.

3. Results and discussion

Samples under investigation with one and two cladded layers were identified as "1NbC" and "2NbC" respectively. An X-ray diffraction (XRD) pattern of the niobium carbide powder used for cladding is shown in Figure 1 (red line). The results of the phase analysis of cladded layers are presented in Figure 1 (black line). The main phases formed during cladding of both one and two layers of niobium carbide
were α-Ti (α'-Ti), β-Ti and titanium carbide. The analysis of the XRD patterns revealed the absence of peaks corresponding to niobium carbide or (Nb, Ti)C complex carbide [15].

Figure 2 shows the distribution of chemical elements along the line obtained by the energy dispersive X-ray (EDX) analysis. It is clearly seen in the picture that the concentration of niobium in the carbide phase was lower than in the matrix. Analysis of the elemental composition of the carbide phase and the titanium matrix revealed a 2-fold rise in the niobium concentration in the titanium matrix together with a rise in the niobium quantitative content from about 8 wt. % to 13.5 wt. % as a consequence of a layer amount increase.

Figure 1. XRD patterns obtained from the alloyed titanium layer: red line – niobium carbide powder; black line – a sample fabricated by one-layer cladding of niobium carbide.

Figure 2. Distribution of Ti, Nb and C along the scanning line.

Figure 3. Scheme of the structure of the titanium workpiece obtained by non-vacuum electron beam cladding of niobium carbide: 1 – an area containing fine close-packed TiC particles; 2 – an area containing TiC particles of different morphology; 3 - an area containing eutectic TiC particles; 4 – a heat affected zone; 5 – the initial structure of titanium; C - fine close-packed TiC particles; T – a lamellar structure of the titanium matrix; A – TiC aggregations; E – fine TiC particles found in the TiC-Ti eutectic; D – dendritic TiC particles.
The cladded layer is an area of particular interest. During electron beam treatment niobium carbide particles are dissolved in the melted material and the diffusion of Nb and C occurs. As a result, niobium partially dissolved in the alloy contributes to the stabilization of the titanium $\beta$-phase at room temperature. On the other hand, some of the niobium partially replaces titanium atoms in the carbide phase. As a consequence of a short time of the material being in a melted state, an inhomogeneous distribution of chemical elements across the depth of the cladded layers occurs.

Weakly etched areas were observed in the structure of the cladded layers (Figure 4, a). This phenomenon was generally observed in the areas characterized by the carbide particles accumulation (Figure 4 a, b). Cladding of the second layer resulted in increasing the structural uniformity of the material. Aggregations of carbide particles were formed in the layer with a thickness of 800 – 1000 $\mu$m.

A duplex microstructure of the titanium surface layer was revealed by SEM (Figure 4, d). The doping level determined the structure of local zones of the caddied layer: some areas of the coating were represented by a $\beta$-Ti-based solid solution with nanosized precipitations of $\omega$-Ti (Figure 4, b; Figure 5 a, b); the others were characterized by the presence of a $\alpha$- and $\beta$-Ti mixture (Figure 4, d).

![Figure 4. SEM microphotographs of the structure of titanium surface layers after non-vacuum electron beam cladding of niobium carbide powders.](image)

The presence of martensitic structure attributes was revealed in some zones (Figure 5, c). Taking into account a high Nb concentration in these areas and a high cooling velocity, it can be supposed that martensitic crystals were represented by the $\alpha''$-phase with a composite orthorhombic lattice. The formation of the $\alpha''$-Ti martensitic phase in similar conditions was also observed in [14].

Titanium carbides alloyed with niobium had a different morphology (see Figure 3). In the 1st zone (near the surface of the cladded layer) the carbide phase was represented by round particles Dendritic crystals with long one-fold axes and short secondary branches as well as fine particles distributed
along the grain boundaries and within the grains can be seen in the direction from the surface to the center of the cladded layer (Figure 4).

**Figure 5.** Bright-field microphotographs of the coating structure and the microdiffraction pattern: a – the aggregation zone of carbide particles; b – the electron diffraction pattern obtained from the areas in Figure 4, a and corresponding to β- and ω-Ti; c – the martensitic structure of titanium in the cladded layer.

Analysis of the relationship between microhardness and the depth of layers cladded on "1NbC" and "2NbC" samples (Figure 6) showed that the maximum microhardness level (480 HV) was observed in the double-layer coating. The material microhardness decreased to 330 HV at a depth of 0.4 mm. An increase in the microhardness level occurred at the depth of 0.9 mm due to the accumulation of carbide particles (Figure 4 a, b) and the formation of ω-Ti. An average microhardness level of the modified layer formed on a "1NbC" sample was 330 HV.

**Figure 6.** Distribution of microhardness in the depth of the cladded layer obtained by non-vacuum electron beam cladding of the niobium carbide powder.

4. Conclusions

Non-vacuum electron beam cladding of niobium carbide powders allows efficient hardening of surface layers of titanium workpieces to a depth of approximately 2.1 mm. The main phases formed in the cladded layers were hexagonal titanium, cubic titanium and titanium carbide. The presence of niobium carbide (NbC) and complex carbide ((Nb, Ti)C) was not observed by TEM and XRD. It can therefore be said that niobium is contained in the titanium matrix in the form of a solid solution as well as in the
carbide phase. High velocities of the coating formation resulted in the areas enriched with niobium where the presence of ω-Ti was observed. An average microhardness value of the cladded layer was approximately 330 HV.

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