The influence of non-vacuum electron-beam facing on the structure of Ti-Ta layers formed on the surface of VT1-0 alloy

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Abstract. The influence of electron-beam facing modes on the structure of Ti-Ta layers formed on the surface of commercially pure titanium VT1-0 has been studied in the paper. The mode of the electron-beam treatment of alloying powder mixture, by which there were no defects in the pad, has been identified. The methods of optical microscopy and scanning electron microscopy have shown that in pads there is dendritic segregation typical for the process of initial crystallisation. At greater magnifications it is possible to observe a structure of the laminar type. The X-ray phase analysis of titanium-tantalum layers justifies the presence of two phases: a hexagonal α'-phase and a cubic β-phase of titanium.

1. Introduction

At present titanium and its alloys are widely used in different branches of industry [1-3]. An excessive interest in this material is conditioned by its enhanced set of mechanical characteristics in combination with a low rate of corrosive attack in most of the corrosive solutions. However titanium alloys are subjected to significant corrosion in boiling solutions of strong acids which does not allow its use in the manufacture of machine components and high-duty structural elements operating in similar conditions [4, 5]. In order to increase the corrosion resistance of titanium to the influence of boiling acid solutions it is alloyed with such elements as tantalum, niobium and molybdenum [5, 7]. Titanium-molybdenum alloys possess high corrosion resistance in boiling solutions of sulphuric and hydrochloric acids [8]. Nevertheless these alloys are subjected to significant corrosion in boiling solutions of nitric acid, the rate of which increases with the rise of molybdenum concentration [5]. The introduction of niobium and tantalum in titanium enables the increase of corrosion resistance of the alloy in boiling nitric acid [7, 9]. At that the greatest effect is achieved if titanium is alloyed with tantalum [5]. It is known that introduction of a small amount of tantalum into the titanic alloy facilitates a significant decrease of its corrosion rate in boiling acids [6]. When introducing tantalum in the amount of 40 % wt and more the corrosion resistance of the alloy in 80 % sulphuric acid, heated up to the temperature of 75 °C, becomes comparable to commercially pure tantalum [10]. An essential drawback of tantalum is its high cost and high density [11]. In order to save costly tantalum and to increase corrosion resistance of titanium, surface alloying of titanium alloys with tantalum is a reasonable. It should be noted that the obtainment of tantalum-containing layers on the surface of titanium workpieces does not lead to a significant increase in the weight in comparison with three-dimensionally alloyed workpieces.

The studies undertaken earlier indicate high efficiency of the method of non-vacuum electron-beam treatment during formation of surface-alloyed layers on titanium workpieces [12-14]. For the realisation of this technology the industrial electron accelerators providing extraction of electron beam into the air atmosphere can be used. Such accelerators include, for example, the electron accelerator 'ELV-6' developed by the Institute of Nuclear Physics after G I Budkera, Siberian Branch of the Russian Academy of Science. In papers [14-17] the facing of tantalum-containing layers was applied to the workpiece 50 mm wide by means of the electron beam. The increase in the amplitude of scanning of the electron beam was achieved due to installation of the electro-magnetic scanner on the exhaust diaphragm of the accelerator. However Ti-Ta facing on the surface of titanic workpieces without using the electro-magnetic scanner (in a track mode) has not been applied earlier. In this connection the purpose of this paper is to study the influence of the modes of electron-beam alloying of commercially pure titanium VT1-0 with tantalum at treatment in a track mode.

2. Materials and research techniques

Tantalum-containing layers were faced on the laminae of commercially pure titanium VT1-0 with dimensions of $100\times25\times12$ mm. The mixture of tantalum, titanium and flux powders (75 % CaF_2 + 25 % LiF) in the amount of 40 % wt of Ta, 24 % wt of Ti, 36 % wt of flux was used as an alloying material. Prior electron-beam treatment the powder mixture was applied to the surface of the titanium workpiece in the amount of 0.45 g/sm^2. Then the workpiece with the formed powder layer was mounted on the movable table, where it moved forward relatively the outlet with the rate of 10 mm/s. The distance from the outlet to the workpiece surface equaled to 123 mm. The initial energy of the electrons in the beam amounted to 1.4 MeV. For the purpose of studying the influence of the facing modes on the structure and properties of the surface layer different values of beam current, which are 7, 8, 9 and 10 mA, were used.

The structural peculiarities of the pads were studied by means of the optical microscope 'Carl Ziess Axio Observer A1m' and the scanning electron microscope 'Carl Zeiss EVO 50 XVP'. The concentration of alloying elements in the surface-alloyed layer was determined by means of the energy-dispersive electron probe microanalyser 'X-ACT' (Oxford Instruments).

The phase composition of the pads was analysed by means of the X-ray diffractometer 'ARL X'TRA'. The diffraction patterns were recorded using $CuK\alpha_1/\alpha_2$ radiation. The accumulation time for one point amounted to t = 3 s, a scanning pitch was $\Delta2\theta = 0.05$ °.

3. The results and discussions

By means of the methods of optical metallography it has been established that the minimum thickness of the pad corresponds to the sample obtained at 7 mA current, which amounts to 0.6 mm (Figure 1a). The increase of current of the electron-beam treatment enables thickness growth of the surface-alloyed layer. Thus for samples faced at current of 8 and 9 mA the layer thickness equals 1.2 and 2.0 mm correspondingly (Figure 1b, c).The maximum thickness of the pad is fixed for samples obtained at the electron beam current of 10 mA and is equal to 2.5 mm (Figure 1d).

In the structure of the surface-alloyed layer faced at 7 mA current there are pores as well as unfluxed tantalum particles. The increase in the current of electron-beam treatment up to 8 mA leads to formation of a structure without any pores. However there is a small amount of unfluxed tantalum particles. The structure of the layers formed by the electron beam at currents of 9 and 10 mA has a homogeneous and defect-free structure. The difference between the layers obtained by facing at these points consists in the chemical composition. The titanium-tantalum alloy formed on the titanium surface at 10 mA current is less alloyed with tantalum in comparison with the sample obtained at 9 mA current as a result of a greater depth of melting of the base material and dilution of the pad with titanium. The results obtained by the method of the electron probe microanalysis justify this fact (Table 1).
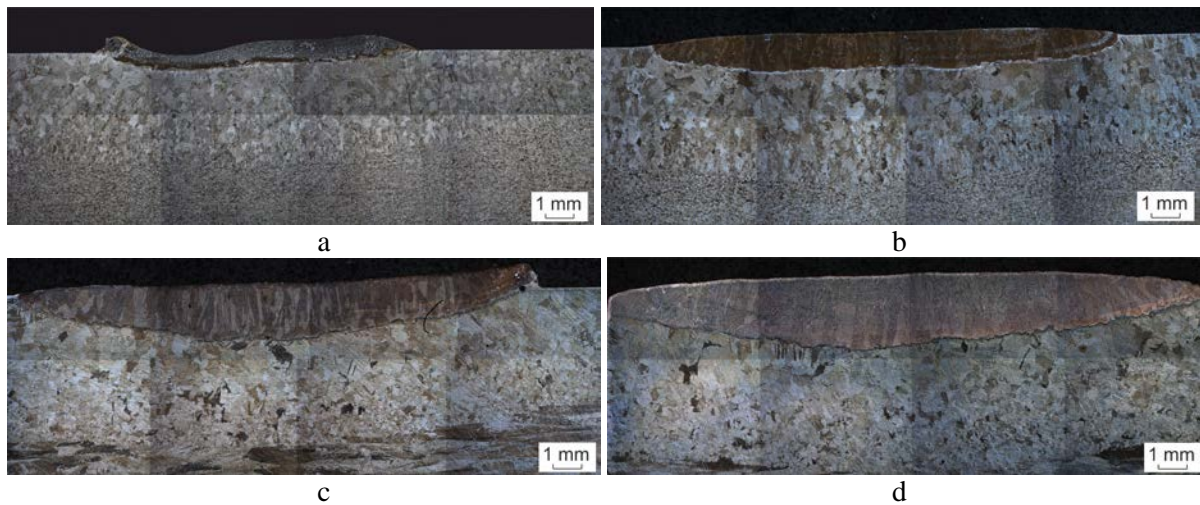
Figure 1. Micrographs of the layers obtained by means of non-vacuum electron-beam facing of Ti-Ta powder mixture using currents of 7 (a), 8 (b), 9 (c) and 10 mA (d).

The tantalum concentration in the layer formed at 9 mA current amounts to 23.2 % wt. The increase of the current of the electron beam up to 10 mA contributes to reduction of tantalum content in the pad up to 15.5 % wt. Thus in order to obtain defect-free coatings with maximum tantalum concentration we should use treatment with the beam current of 9 mA.

Table 1 – Chemical composition of the surface-alloyed layers faced by the electron beam at currents of 9 and 10 mA

| Current intensity	Content, % wt.	
	Ta	Ti
9 mA	23.2	76.8
10 mA	15.5	84.5

The structure of the pad formed by the electron beam at 9 mA current is represented in figure 2. In the pad there is a significant dendritic segregation typical for the materials obtained in the process of the primary crystallisation (Figure 2a). Dendritic crystals grow from the boundary containing unfluxed base material in the direction of the surface. During etching the former grain boundaries of a β-phase emerge against the dendritic background. The grains have an elongated shape and are comparable in size to dendritic crystals. The study of the material at greater magnifications has shown that the material has a structure of a lamellar type (Figure 2b). A similar structure results at cooling from β-area of titanium alloys alloyed with beta-isomorphic stabilizers. At this the cooling rate must be insufficiently high for fixing a pure α' or α" phase.

<center>a   b</center>
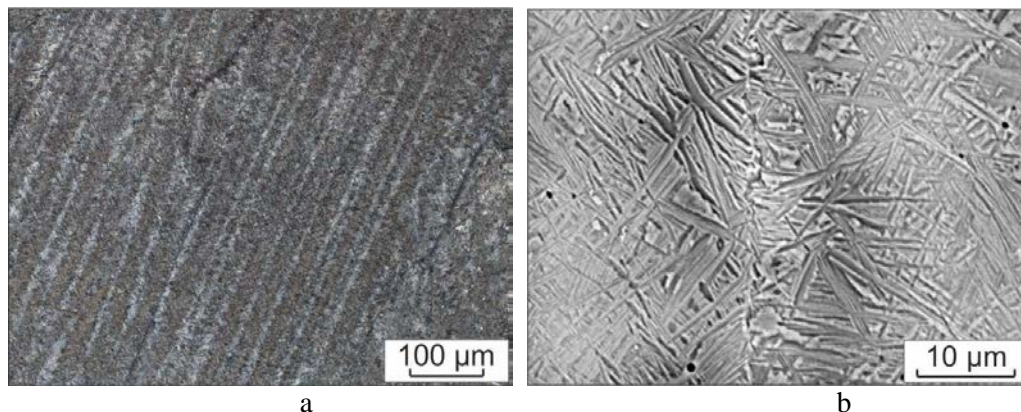
Figure 2. The structure of a Ti-Ta layer obtained by the electron-beam facing of the alloyed powder mixture at 9 mA current.

In order to determine the phase composition of the surface-alloyed layer obtained at the beam current of 9 mA, an X-ray diffraction studies have been conducted. The X-ray phase analysis has shown the presence of peaks of a hexagonal α (or α')-phase and a cubic β-phase of titanium (Figure 3). The analysis of the X-ray picture of the sample is evidence of the difference in intensity of experimental peaks from intensity of the peaks typical for the materials with a random distribution of orientation. Redistribution of peaks intensity is probably connected with texture formation typical for the process of initial crystallisation.
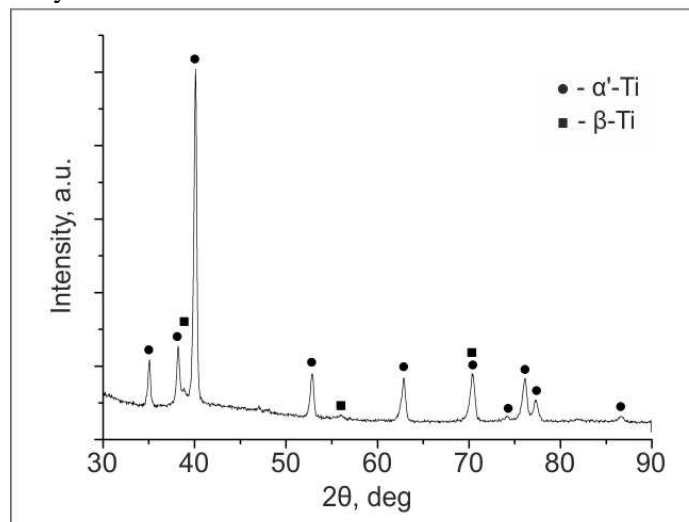
Figure 3. The X-ray picture of Ti-Ta alloy formed by the electron-beam facing at the beam current of 9 mA.

Conclusions

In the course of the work the mode of the electron-beam alloying of titanium with tantalum, providing the formation of the defect-free layer with high concentration of tantalum, has been selected. The surface-alloyed layers obtained by means of this mode are characterised by the absence of such defects as pores and undissolved tantalum particles. During the study of the structure of titanium-tantalum layers a significant dendritic segregation has been identified. It has been stated that a faced alloy has a structure of a lamellar type. The analysis of the x-ray pictures justifies the presence of two phases: a hexagonal α (or α')-phase and a cubic β-phase of titanium.

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