Formation of a Microcomposite Structure in the Surface Layer of Yttrium-Doped Titanium

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Abstract—Surface layers containing oxides and carbides of titanium and yttrium are prepared by the electroexplosive doping of titanium with yttrium. The subsequent electron-beam irradiation leads to dispersion of the structure to a nano- and submicron state. The formation of two types of eutectics is revealed using scanning electron microscopy. The eutectics enriched in titanium and yttrium have a globular and plate-like shape, respectively. The formation of a modified surface layer (enriched in yttrium, carbides and oxides of titanium and yttrium) leads to a threefold increase in the microhardness of the titanium, a more than twofold decrease in the friction coefficient of the doped layer, and a more than 2.8-fold decrease in the wear rate.

Keywords: titanium, electroexplosive doping with yttrium, electron irradiation, modified layer

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INTRODUCTION

According to the state diagram, yttrium and titanium are completely miscible in the liquid state; upon solidification, they form a eutectic mixture of two limited solid solutions [1]. In the solid state (below a temperature of 875°C), the material is a mixture of two (α -Ti and α -Y)phases. The α -Y phase is formed through a reverse peritectic reaction at $T = 1440^{\circ}$ C; the eutectics are formed at ~20 at % Ti and a temperature of 1355°C; and the α -Ti phase is formed through a eutectoid reaction at $T = 875^{\circ}$ C. Since yttrium and titanium exhibit low solubility in the solid state, the constants of the hexagonal crystal lattices of α -Ti and α -Y vary only slightly upon doping of one metal with the other [1]. Thus, the Ti-Y system belongs to partially miscible binary systems that do not have intermetallic compounds and is of considerable interest for the development of materials with improved properties [2, 3]. Alloys based on these systems with a high content of one of the components which deliberately exceeds the limit of its solubility in the other component make it possible to prepare materials with fairly high physical and mechanical characteristics via a relatively simple method [3, 4].

The aim of this study is to analyze the structure of the surface layer of the titanium–yttrium system formed during high-speed processing of the film/substrate system.

EXPERIMENTAL

Commercially pure titanium VT1-0 was used as the base material [5]. The surface layer of the Ti-Y system was formed according to a two-stage scheme. At the first stage, the electroexplosive doping method was used (EVU-60/10M setup of the Siberian State Industrial University, Novokuznetsk) [6]. A foil of commercially pure titanium VT1-0 with a weight of 100 mg was used as the explosive electroconductive material. A 400-mg weighed portion of yttrium nanopowder was placed on the foil in the region of the explosion. The time of plasma treatment of the sample surface was $\sim 100 \,\mu s$; the absorbed power density along the axis of the jet was $\sim 5.5 \text{ GW/m}^2$; the pressure in the shockcompressed surface layer was ~12.5 MPa; the residual gas pressure in the chamber was ~ 100 Pa; the plasma temperature at the nozzle output was $\sim 10^4$ K; the thickness of the surface alloy was $\sim 30 \mu m$; and the thickness of the heat-affected zone was ~50 µm. Subsequent high-speed heat treatment of the alloy was conducted with a high-intensity pulsed electron beam using a SOLO system (Institute of High-Current Electronics, Siberian Branch, Russian Academy of Sciences, Tomsk). Estimates show that, at an electronbeam exposure time for the metal surface of 50-200 µs, the heating and cooling rates for the modified layer are $\sim 10^6$ K/s [7]. In the exposure mode the electron energy was 18 keV; the electron-beam energy



Fig. 1. SEM image of the surface layer of commercially pure titanium after electroexplosive doping with titanium and yttrium.

density was 20 J/cm²; the pulse duration was 150 μ s; the number of pulses was 3; and the pulse-repetition frequency was 0.3 s⁻¹. Examination of the surface morphology and determination of the elemental and phase composition were conducted by optical microscopy, scanning electron microscopy (SEM), and X-ray diffraction analysis. The tribological properties of the surface alloy were studied under dry-friction conditions in the disc-ball geometry (a counterbody of a WC–Co hard alloy ball with a diameter of 3 mm, a load of 1 N). The hardness of the doped layer as a function of distance to the treatment surface was determined using a PMT-3 hardness tester.

RESULTS AND DISCUSSION

The electroexplosive doping of the commercially pure titanium surface with titanium and yttrium leads to the formation of a multilayer structure. A coating with a highly developed relief composed of microdroplets, floating metal, micropores, and microcracks is formed on the surface (Fig. 1). Electron-probe microanalysis of this coating revealed the presence of

Data of electron-probe microanalysis of the surface regions marked in Fig. 4a

Region number	Element concentration, wt %			
	Ti	Y	0	С
1	18.0	67.3	12.5	2.2
2	85.3	7.2	5.7	1.8
3	37.6	45.4	13.5	3.5

oxygen and carbon atoms in addition to titanium and yttrium (Fig. 2); this fact can be attributed to doping of the material under low-vacuum conditions.

The distribution of the detected elements over the surface layer is fairly nonuniform, as evidenced by the analysis results shown in the table. The most nonuniform distribution in the surface volume of the material is found for yttrium; its concentration can differ from region to region by 5-8 times.

A significantly heterogeneous multilayer structure is revealed in the analysis of the cross sections of the doped material. Typically, three layers that differ in morphology and size of structural elements are detected (Fig. 3). The surface layer (designated by I in Fig. 3) has the roughest structure; layer III adjacent to the heat-affected layer of the substrate (designated by IV in Fig. 3) is the most dispersed.

Exposure of the sample after electroexplosive doping to a high-intensity electron beam leads to melting of the surface layer. As a consequence, the surface topography becomes smooth and the micropores almost completely disappear; however, the microcracks remain. An island structure is formed on the exposed surface (Fig. 4). Electron-probe microanalysis showed that the majority of the surface layer is enriched in yttrium (Fig. 4, spectrum *I*) and islands with a size of 10–40 μ m are enriched in titanium (Fig. 4, spectrum *2*). The quantitative results of the electron-probe microanalysis of the portion of the material shown in Fig. 4a are listed in the table.

The islands and regions between them contain globular inclusions with sizes varying in the range 100–300 nm (Fig. 5). The former and the latter contain yttrium (Fig. 5) and titanium inclusions (Fig. 4a), respectively. Analysis of the data in Fig. 5 shows that the yttrium inclusions are arranged in an order form-



Fig. 2. Morphology of the surface layer of commercially pure titanium after electroexplosive doping with titanium and yttrium. The surface regions for electron-probe microanalysis are marked with frames.

ing a contour around the titanium region (possibly a grain) with a size of $0.5-1.5 \ \mu m$.

Comparison of the results shown in Figs. 4 and 5 and in the table suggests that the titanium-enriched islands (Fig. 5) are the product of eutectic transformation of the titanium-enriched melt. The possibility of the formation of this eutectic at an yttrium concentration of 18 at % has been indicated in [8]. According to the table, the yttrium concentration in the islands shown in Fig. 5 is slightly lower, 12 at %.

Quite frequently, the interface between the titanium-enriched islands is the place of origin of a platelike titanium/yttrium eutectic. A characteristic image of this structure is shown in Fig. 6. The transverse sizes of the plate-like eutectic elements vary in the range of 200–300 nm.



Fig. 3. SEM images of the cross section of commercially pure titanium after electroexplosive doping with titanium and yttrium under varying magnification. The numbers indicate the sublayers of the modified surface layer.



Fig. 4. (a) Surface morphology after electroexplosive doping of titanium and electron-beam irradiation; (b, c) energy spectra of the analyzed regions.

Electron-probe microanalysis revealed that the yttrium and titanium concentration in the eutectics is 85 and 15 at %, respectively. It should be noted that in the case of equilibrium crystallization of the titanium—yttrium system, the eutectic is formed at 80 at % of yttrium [1].

The high-intensity electron-beam melting of the surface layer of the titanium/yttrium system formed by electroexplosive doping is accompanied by dispersion of the structure not only of the exposed surface, but also the total volume of doping with a thickness of 30–

40 μ m (Fig. 7). The most significant modification is undergone by the structure of the surface layer with a thickness of 10–15 μ m (Figs. 7b, 7c), which is apparently formed during crystallization of the layer melted by the electron beam.

The surface modification of titanium by a combined method (exposure to plasma formed during the electrical explosion of a conductive material and subsequent treatment with a high-intensity electron beam) is accompanied by significant improvement in the mechanical and tribological properties of the



Fig. 5. SEM image of the titanium surface after (a) electroexplosive doping and (b) high-intensity electron-beam irradiation.



Fig. 6. SEM images of the plate-like eutectic formed on the surface after the electroexplosive doping of titanium and high-intensity electron-beam treatment under varying magnification.

material. That is, the microhardness of the 20-µmthick surface layer increases approximately threefold (compared to the substrate), the friction coefficient of the doped layer decreases more than twofold, and the wear rate decreases more than 2.8-fold.

CONCLUSIONS

A surface alloy of the titanium/yttrium system has been formed by electroexplosive doping and electronbeam treatment. The structure, elemental and phase composition, and mechanical and tribological properties of the doped layer have been studied. It has been shown that the electroexplosive doping of titanium with yttrium is accompanied by saturation of the surface layer with oxygen and carbon atoms, which leads to the formation of oxides and carbides of titanium and yttrium. Subsequent electron-beam irradiation is accompanied by dispersion of the structure to a nanoand submicron state and a decrease in the oxygen and carbon concentrations in the surface layer. The formation of two types of eutectics has been revealed. It has been shown that the eutectics enriched in titanium and yttrium have a globular and plate-like shape, respectively. It has been found that the formation of a surface layer enriched in yttrium and oxides and car-



Fig. 7. (a-c) SEM images of the cross section of the sample after the electroexplosive doping of titanium and high-intensity electron-beam treatment under varying magnification; (d) structure of the transition layer separating III and IV.

bides of titanium and yttrium leads to a significant (about threefold) increase in the microhardness of the titanium, a more than twofold decrease in the friction coefficient of the doped layer, and a more than 2.8fold decrease in the wear rate.

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