Influence of Intense Bombardment of Cu^+ Ions on Microhardness and Structure of VT-23 titanium alloy

Mark P. Kalashnikov[1, 2, a)], Victor P. Sergeev[1, 2, b)], and Vasilii V. Neyfeld[2, c)]

[1] *National Research Tomsk Polytechnic University, Tomsk, 634050, Russia*
[2] *Institute of Strength Physics and Materials Science SB RAS, Tomsk, 634055, Russia*

[a)] Corresponding author: kmp1980@mail.ru
[b)] vserg@mail.tomsknet.ru
[c)] rets@ispms.tsc.ru

Abstract. The structural-phase state of the ion-modified surface layer of the VT-23 titanium samples was investigated by TEM and SEM. The results of the surface modification of the VT-23 titanium alloy by the intense flux of copper ions with energy of 2 keV and the ion current density of 3.5 mA/cm^2 were studied. The dependence of the microhardness and the penetration depth of ions as a function of duration of ion treatment was determined. The microhardness was investigated by the nanoindentation method.

Keywords: intense bombardment, VT-23 titanium alloy, surface layer, high-current ion flux, structural-phase state, microhardness

INTRODUCTION

The problem of new technologies of hardening of constructional materials development is currently important, in particular, in materials on the basis of the ionic-plasma methods of multilayered nanocomposite coatings deposition and high-energy processing of the surface layers, which allow modifying their structurally-phase condition, because the current requirements to the technical characteristics of aerospace engineering of 5th generation have increased [1].

The methods of the ion-beam modification are a promising trend to improve the performance properties of structural materials [2]. The structure-phase state of the surface layer can be effectively modified with the use of a high-current flow of heavy ions of low energy. Tribological, mechanical properties, including the fatigue characteristics of the constructional materials can be improved in this case [3].

In this connection, the high strength VT-23 titanium alloy becomes more and more widely used in designing and manufacturing of modern aerospace engineering devices [4, 5]. For many advanced technical applications it is necessary to improve its mechanical properties. The aim of this work is to investigate the possibility of modifying the mechanical properties of the VT-23 titanium alloy using the surface treatment by an intense flux of copper ions.

EXPERIMENTAL PROCEDURE

The treatment of VT-23 titanium under a continuous copper ion beam with the energy of ~2 keV and the ion current density of ~3.5 mA/cm^2 using the vacuum system KVANT-03MI (Techimplant Ltd., Russia) and a vacuum arc ion source with a copper cathode was carried out [6]. The study was conducted using 5×5×20 mm samples produced from the VT-23 titanium alloy (Ti—88.1%, V—4.5%, Al—5.5%, Cr—1.2%, Fe—0.6%). Two-stage heat treatment was used for sample preparation: 1) hardening-heating up to 1093 ± 10 K for 3 hours, air cooling up to RT; 2) aging-heating in vacuum up to 773 ± 10 K for 8 hours, cooling in a vacuum furnace up to RT.

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TABLE 1. Treatment regimes by a flux of copper ions of the VT-23 titanium alloy

Number of the Sample sets	Processing mode	
	t, min	Fluence, ion/cm^2
1	3	0.9×10^{18}
2	6	1.8×10^{18}
4	7.5	2.25×10^{18}

The sample faces were studied after polishing one of the big faces to the surface treatment class Ra 0.16 μm. The residual vacuum in the chamber during the treatment was better than 1×10^{-3} Pa. The working chamber was evacuated using an oil-free pumping tool—cryogenic and spiral pumps. During the ion treatment the samples were heated. The maximum heating temperature of the samples was 1083 ± 10 K for the ion treatment duration 7.5 min. The treatment regimes are shown in Table 1.

The structural-phase state of the ion-modified layers of the samples was investigated by the TEM method using a JEM-2100 device (Jeol Ltd., Japan). Foils for the TEM studies were prepared by the "cross-section" methods using the Ion Sliser EM-09100IS (Jeol Ltd., Japan) to classify the structures, the grain size and the phase composition. The bright-field images together with the corresponding microdiffraction patterns and dark-field images obtained by TEM were used. The phase composition and the crystalline lattice parameters of the coatings were determined by X-ray. The data base JCPDS was used for diffractograms interpretation.

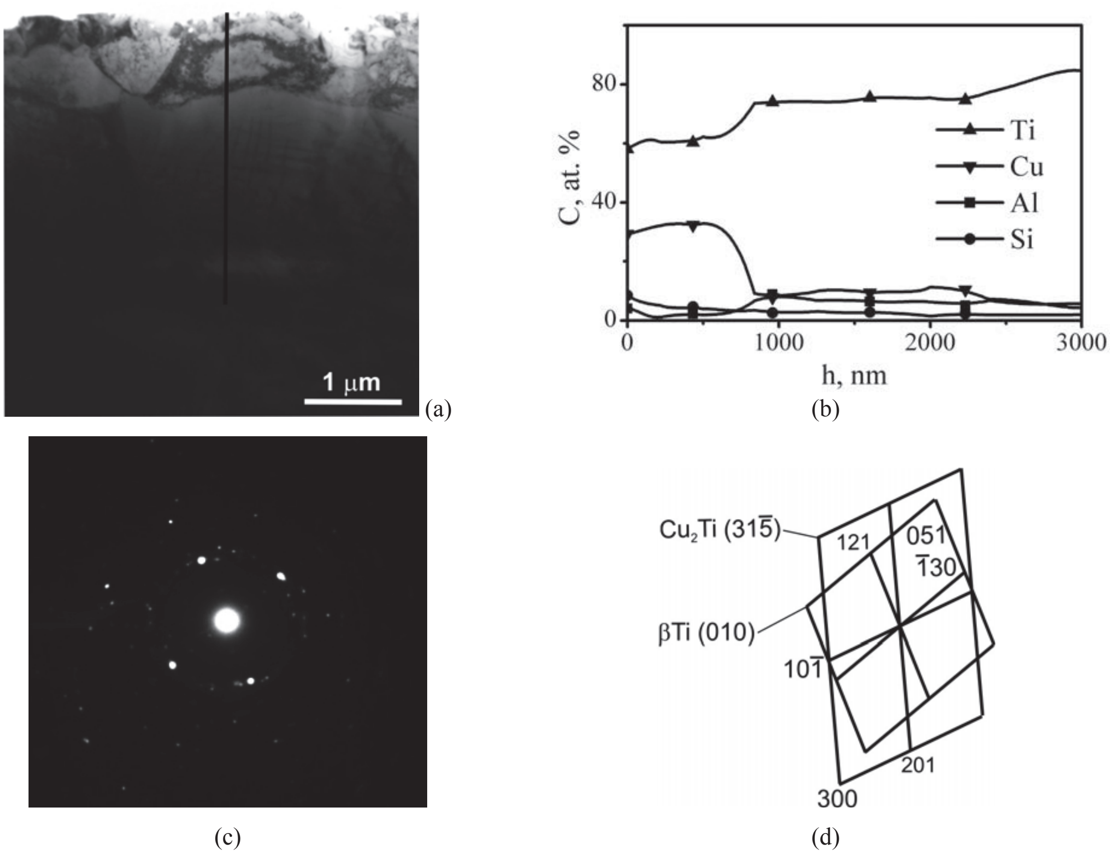
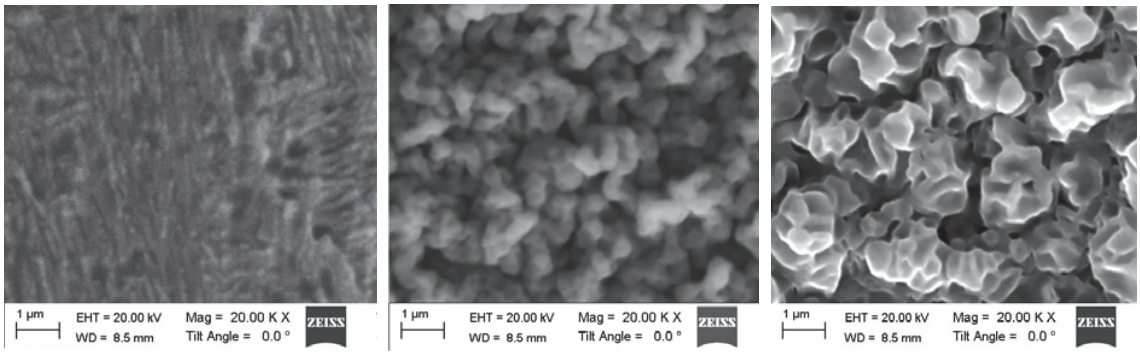


FIGURE 1. The TEM images of the surface layer of VT-23 titanium alloys treated by copper ions for 6 min. (cross-section) (a); the distribution of the elements obtained by MRSA along the black line (b); the diffraction pattern of the surface layer and its indexing (c, d)



(a) (b) (c)

FIGURE 2. The surface morphology of the VT-23 alloy treated by a copper ion beam of durations: 3 min (a), 6 min (b), 7.5 min (c)

The chemical composition of the coatings was determined by the energy dispersive X-ray (MRSA) using a microanalyzer (EDX) INCA-Energy (Oxford Instruments), a built-in TEM JEM-2100 and SEM LEO EVO-50XVP. The microhardness, the modulus of elasticity and the elastic recovery coefficient of the coatings and the glass substrates were measured using the NanoHardnessTester (CSM Instruments, Switzerland) under the load 20mN on the indenter.

RESULTS AND DISCUSSION

An effective nanostructuring of the surface layer by intense titanium ion beams is advisable if we can use ions of the elements of substitution with an atomic radius less than the radius of the elements of the surface, and the atomic matrix mass more than that of titanium. These ions can penetrate deeply and produce a significant elastic deformation in the crystalline lattice at implantation. For this purpose, the copper ions are selected so that they have the most favorable characteristics. Also according to the state diagram of the copper-titanium, they can form a large number of intermetallic compounds [7], which are of additional interest. The structural-phase state of the high strength VT-23 titanium alloy, which is widely used for the manufacture of the critical parts of an aircraft, after quenching and aging is doped ($\alpha + \beta$) martensite [4, 5].

It was established by MRSA that there was copper in the concentration profile of the ion-modified surface layer (Fig. 1(a, b)). It is seen that when the penetration depth increases the concentration of the implanted copper ions increases too from some initial value C_0 to the maximum value $C_m \sim 33$ at.%, then the maximum value is maintained to a certain depth h_m, and then it decreases almost to zero. The thickness h_m of the copper-doped layer increases from 100 to ~1400 nm when the ion treatment duration increases from 3 to 8 min accordingly.

The phase composition of the implanted layers was investigated using TEM cross-section images. There are some intermetallic phases on the basis the Cu–Ti system in the samples with the treatment time about 3–4 min. These phases have, as is known, a higher hardness than α-Ti и β-Ti. When the duration of the ion treatment by copper ions increases, the volume fraction of the intermetallic phases increases. Thus, the number of different types of the compounds on the basis of the Cu–Ti system decreases so that at $t \geq 6$ min in the surface layer we can observe only two phases (Fig. 2(c, d))—β-Ti и Cu_2Ti with the orthorhombic lattice Amm_2 and the crystalline lattice parameters $a = 4.363$, $b = 7.977$, $c = 4.478$ Å. Further increase in the duration of the surface layer processing has, predominantly, a two-phase structure and only at $t \geq 7$ min there is the Cu_4Ti phase with the orthorhombic lattice Pnma and the lattice parameters $a = 4.530$ Å, $b = 4.342$ Å, $a = 12.930$ Å.

Figure 2 shows the surface morphology of the VT-23 alloy modified by copper ions with a different duration of treatment. It is evident that the surface morphology changes during the ion processing. This is due to different velocities of erosion of the adjacent surface segments differing in the crystallographic orientation and the chemical composition, the density of defects and impurities, the initial geometry [8].

When the duration of the ion treatment is more than 6 minutes, the surface relief increases and a whisker nucleation appears on it (Fig. 2(c)). When the sputtering time is 7.5 min. (Fig. 2(b)) the surface morphology evolves so that the etch pits deepen and the elements that are poorly etched in the form of short whiskers partly connected by horizontal jumpers appear on the substrate surface.

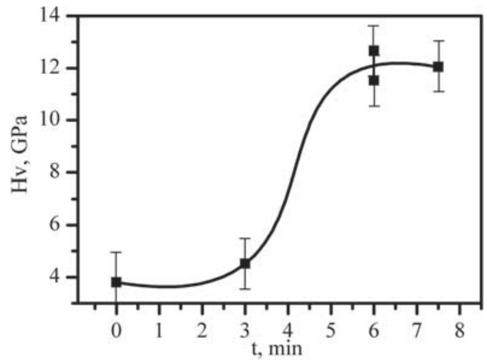


FIGURE 3. The microhardnes of VT-23 alloy as a function of the treatment time by copper ions

Further surface treatment of the substrate by the Ti ions leads, on the one hand, to a considerable reinforcement of the etch pits, and on the other hand, to the growth and coarsening of the horizontal bridges between the whiskers of the crystals due to the formation of the intermetallics of the Cu–Ti system as a result of the dynamic mixing of the titanium atoms from the substrate with the copper atoms of the ion flux. As a result, a microporous structure consisting of the Cu_xTi—intermetallic skeleton, partially filled with the microcrystal β-Ti in the modified surface layer is formed.

Figure 3 shows the experimental dependence of the microhardness of the VT-23 surface layer on the duration of their treatment by copper ions. It is seen that a sharp increase in the microhardness H_v takes place (3 times) within a time interval of 3 to 6 min. An increase in H_v slows down when the duration of the treatment increases and remains constant when the microhardness reaches $H_v = 12.1 \pm 0.9$ GПa.

Thus, an increase in the microhardness of the surface layer can be associated with the formation of the solid intermetallic phases on the basis of Cu_xTi in the ion-modified surface layer, and its increase when the duration of treatment by ions increases is associated with increasing thickness of the ion-doped layer.

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