AUMENTO DA DURABILIDADE DA LIGA DE TITÂNIO VT6 PELA IMPLANTAÇÃO DE COBRE E IONS DE ALUMÍNIO

VT6 TITANIUM ALLOY WEARABILITY INCREASE VIA IMPLANTATION OF COPPER AND ALUMINUM IONS

ПОВЫШЕНИЕ ИЗНОСОСТОЙКОСТИ ТИТАНОВОГО СПЛАВА VT6 ПУТЕМ ИМПЛАНТАЦИИ ИОНОВ МЕДИ И АЛЮМИНИЯ

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RESUMO

A relevância do artigo se deve ao fato de que a utilização de ligas de titânio dentro de juntas de fricção é restringida por sua baixa resistência ao desgaste, enquanto os métodos tradicionais de aumentar sua resistência ao desgaste são ineficazes. O objetivo deste trabalho foi estudar os processos ocorridos na superfície de amostras da liga de titânio VT6 ao implantar íons cobre e alumínio, bem como no atrito. Estimou-se a composição elementar, estado estrutural, propriedades mecânicas e tribológicas das camadas superficiais da liga de titânio VT6 modificada por íons alumínio e cobre durante o processo de implantação iônica de alta intensidade. Como observado nos estudos realizados, o modo do processo de implantação iônica de alta intensidade torna possível obter camadas superficiais da liga VT6 dopadas com íons contendo fases intermetálicas finamente dispersas com TiAl, Ti₃Al, Ti₃Cu, Ti₂Cu, TiCu e uma solução sólida de alumínio e cobre em titânio de composição variando em profundidade. A espessura da camada dopada com íon, o tamanho médio de grãos das fases intermetálicas (de 18 a 55 nm) e seus conglomerados (de 45 a 280 nm) aumentam com o aumento da dose de implante de 2·10¹⁷ para 1,2·10¹⁹ íons/cm² enquanto a implantação de alumínio (de 0,42 a 2,1 µm) está em andamento. Foi demonstrado que a implantação de íons de alumínio e cobre na liga de VT6 conduz a um aumento considerável na sua microdureza e durabilidade. Com base nos resultados da pesquisa, uma conclusão sobre o efeito positivo de um estado de fase estrutural de camadas de titânio dopado com íon em suas propriedades mecânicas e tribológicas da liga de titânio VT6 foi desenhada.

Palavras-chave: ligas de titânio, liga VT6, implantação iônica, sistema duplo Ti-Al, fator de atrito.

ABSTRACT

The relevance of the article is due to the fact that the use of titanium alloys within friction joints is restrained by their low resistance to wear while traditional methods of increasing their wear resistance are ineffective. The objective of this work was to study the processes occurring on the surface of VT6 titanium alloy samples when implanting with copper and aluminum ions, as well as in friction. Elemental composition, structural-phase state, mechanical and tribological properties of VT6 titanium alloy surface layers modified by aluminum and copper ions during the high-intensity ion-implantation process was being researched. As can be seen from the undertaken studies, the mode of the high-intensity ion-implantation process makes it possible to obtain ion doped surface layers of VT6 alloy containing TiAl, Ti₃Al, Ti₃Cu, Ti₂Cu, TiCu finely dispersed intermetallic phases and a solid solution of aluminum and copper in titanium of composition varying in depth. The thickness of the ion-doped layer, the average grain size of the intermetallic phases (from 18 to 55nm) and their conglomerates (from 45 to 280 nm) increases with the increase in implantation dose from 2·10¹⁷ to 1.2·10¹⁹ ion/cm² while aluminum implantation (from 0.42 to 2.1 µm) is in progress. It has been shown that the implantation of aluminum and copper ions into VT6 alloy leads to a considerable increase in its microhardness and wearability. Based on the research results, a conclusion on the positive effect of a structural-phase state of ion-doped titanium layers on their mechanical and tribological properties of VT6 titanium alloy has been drawn.

Keywords: titanium alloys, VT6 alloy, ion implantation, Ti—Al double system, friction factor.

АННОТАЦИЯ

Актуальность статьи обусловлена тем, что использование титановых сплавов в узлах трения сдерживается их низким сопротивлением изнашиванию при этом традиционные методы повышения их износостойкости малоэффективны. Целью настоящей работы являлось изучение процессов, происходящих на поверхности образцов титанового сплава VT6 при имплантации ионами меди и алюминия, а также при трении. Исследовался элементный состав, структурно-фазовое состояние, механические и трибологические свойства поверхностных слоев титанового сплава VT6 модифицированного ионами алюминия и меди в процессе высокоинтенсивной ионной имплантации. Как видно из проведенных исследований режим высокоинтенсивной ионной имплантации позволяет получать ионно-легированные поверхностные слои сплава VT6, содержащие мелкодисперсные интерметаллидные фазы TiAl, Ti₃Al, Ti₃Cu, Ti₂Cu, TiCu и твердый раствор алюминия и меди в титане, переменного по глубине состава. С увеличением дозы имплантации от 2·10¹⁷ до 1,2·10¹⁹ ион/см² наблюдается рост толщины ионно-легированного слоя при имплантации алюминия (от 0,42 до 2,1 мкм), среднего размера зерен интерметаллидных фаз (от 18 до 55 нм) и их конгломератов (от 45 до 280 нм). Показано, что имплантация ионов алюминия и меди в сплав VT6 приводит к значительному увеличению его микротвердости и износостойкости. На основании результатов исследований сделан вывод о положительном влиянии структурно-фазового состояния ионно-легированных слоев титана на их механические и трибологические свойства титанового сплава VT6.

Ключевые слова: титановые сплавы, сплав VT6, ионная имплантация, двойная система Ti—Al, коэффициент трения.

1. INTRODUCTION

High specific strength and corrosion resistance of titanium and its alloys have caused their widespread application in engineering and medicine. However, the use of titanium alloys in friction joints is restrained by their low resistance to wear (Molinari *et al.*, 1997; Long and Rack, 1998; Gritsenko *et al.*, 2011). A fair amount of attention has been paid to the research of titanium alloys wearability (Yashkova and Bobkov, 2016; Muratov and Morozova, 2007; Khorev, 2012).

Analysis of the previous studies results brings us to the conclusion that traditional methods of wear resistance increasing (nitriding, cementing, boriding) of the articles made of titanium and its alloys are ineffective. A substantial increase in the friction pair parts wearability is achieved if the conditions for the formation of secondary structures implementing the load damping are established and if the abovementioned ones represent a solid lubricant as well as are being restored by themselves (Garkunov, 2001; Kuksenova *et al.*, 1999; Berkovich and Gromakovsky, 2000).

Ion-implantation process is one of the promising methods for controlling the strength properties of metals and alloys surface layers. Ion-implantation process is an effective method of modifying the microstructure, and elemental

composition of structural and tool materials surface layers due to the formation of high-density solid solutions, interstitial phases, or intermetallic compounds (Uchevatkina *et al.*, 2014; Ovchinnikov *et al.*, 2015a, 2015b; Kurzina *et al.*, 2005; Bozhko *et al.*, 2005).

In this regard, the objective of this work was to study the processes occurring on the surface of VT6 titanium alloy samples when implanted with copper and aluminum ions, as well as during friction.

2. MATERIALS AND METHODS

VT6 alloy samples having the form of a parallelepiped with dimensions of 5×5×30 mm were used as a study material. VT6 titanium alloy samples were originally in two states: coarse-grained (average grain size $d \sim 11 \mu\text{m}$) and ultra-fine-grained (UFG) obtained via ABC deformation method (Sharkeev *et al.*, 2012; Smyslov and Selivanov, 2007).

The average crosswise size of UFG VT6 titanium alloy grains and subgrains was $0.250 \pm 0.020 \mu\text{m}$, the size of coarse grains reached 3 μm , and the volume fraction of grains with dimensions of 1-3 μm was 23%.

Structural modification of the friction surface of VT6 titanium alloy samples was fulfilled via irradiation with a high-intensity ion beam of submillisecond exposure time. Irradiation

was carried out via copper and aluminum ions. The ion implantation mode of samples: duration, number and repetition frequency of the ion beam action pulses were 30 μ s, 3 pulses, 0.3 s⁻¹; the implantation dose was 2 $\cdot$ 10¹⁷, 7.2 $\cdot$ 10¹⁷, 5.2 $\cdot$ 10¹⁸, 1.2 $\cdot$ 10¹⁹. The treatment was accomplished in a residual argon atmosphere under a pressure of $\sim$ 10⁻³ Pa.

Irradiation of VT6 alloy samples was carried out at an accelerating voltage of 30 kV. The distance from the samples to an ion source was 0.35 m. The treatment was being begun at room temperature.

The structural study of samples was performed via scanning (Carl Zeiss EVO 50 instrument) and transmission diffraction (EM-125K) electron microscopy.

Ion-doped surface layers of VT6 titanium alloy were researched for elemental composition via Rutherford backscattering, secondary ion mass spectrometry, and Auger electron spectroscopy methods. Studies of samples surface morphology before and after the ion-implantation process were performed by optical microscopy method. The research of the structural-phase state of implanted samples was conducted via X-ray phase analysis.

Research of tribological characteristics of ion-doped titanium layers was performed in the air according to the 'disk-finger' scheme at a sliding speed of 3m/s and at a load of 25 N (Vinokurov *et al.*, 2010; Gritsenko *et al.*, 2005; Belyy *et al.*, 1998). The counter body was made of ShH15 hardened steel. Measurements of samples mass losses during the wear test were carried out by means of weighing them on the analytical scales.

3. RESULTS AND DISCUSSION

The mass loss dependence of VT6 titanium alloy coarse-grained samples on the test period (Figure 1, curve 1) demonstrates the stage with a high wear rate when the friction proceeds by an adhesive mechanism.

Dependences of VT6 alloy samples weight loss on the test period are linear ones, which is typical for the adhesive wear mechanism.

The dependence of VT6 titanium alloy ultra fine-grained samples weight loss on the test period (Figure 1, curve 2) has a linear

relationship; the wear intensity is higher than in the event of VT6 coarse-grained alloy. Apparently, this is due to high non-equilibrium of the UFG structure, which, in case of titanium, leads to an increase in the adhesion interaction, which leads to an increase in wear as a result.

VT6 alloy friction surface has a grooved structure, which is formed as a result of plowing the surface of the test sample via the counter body (Figure 2).

The availability of microcracks, which indicates the material breakage fracture pattern, has not been established on the friction surface via metallographic analysis. The traces of setting in the form of material areas displaced in friction direction are clearly visible on the friction surface (Sienz *et al.*, 2002).

The availability of carbon and iron in the setting areas has been found out via X-ray microanalysis of VT6 alloy samples friction surface. This indirectly indicates the manifestation of the adhesive interaction of a sample under test with a counter body. Intense formation of wear fragments occurs during the friction of VT6 alloy (Figure 3). The wear fragments have a multilayer structure specific to brittle rupture of material regarding morphology.

The obtained results make us possible to assume that the sample material forms a thin transferred layer on the counter body at the spots of contact with the counter body due to adhesive interaction and the material from the samples can then be transferred again on the abovementioned thin transferred layer. In this case, a multilayer structure is being formed in the form of extended areas.

A high degree of deformation in a specimen and a counter body contact area, as well as high-temperature gradients, lead to embrittlement of a transferred material, and as a consequence of this, it is being split off from the counter body surface in the form of friable wear fragments.

Apparently, the wear fragments have higher hardness, so they work like abrasive particles being within the friction zone. Thus, in the case of VT6 alloy, an abrasive wear mechanism is added to an adhesive wear mechanism. This is evidenced by 'plowing' traces on the samples friction surface.

The research results analysis of VT6 alloy surface layer structure is the fact that the intense progress of intermetallic compound phase-formation processes occurs under the conditions

of high-intensity implantation of aluminum ions. This is due to the high concentration of alloying element being introduced while ion implantation is in progress, as well as with a sufficiently high temperature to be achieved in the target during ion irradiation. An admixture of oxygen, which forms Al_2O_3 aluminum oxide, is observed in VT6 titanium alloy surface layer after aluminum ion-implantation process.

Comparison of the embedded aluminum concentration profiles and the results of phase composition layer-by-layer research by transmission electron microscopy and X-ray phase analysis methods with Ti-Al system state diagram made it possible to establish the locations of phases being formed under conditions of high-intensity ion implantation of aluminum and copper into VT6 titanium alloy (Table 1).

The research of structural-phase state, elemental composition and mechanical properties of VT6 titanium alloy surface layers modified by ion-implantation method with aluminum ions in a high-intensity mode showed that the modified layers with a thickness from 420 nm to 2,100 nm were obtained (Table 1). The maximum concentration of embedded aluminum reached 50-55 at. %.

The research of VT6 titanium alloy ion-doped layers structural-phase state by means of transmission electron microscopy (Table 1) showed that intermetallic phases of Ti_3Al and TiAl composition, which particles average size is 18-55 nm, are formed in its surface layers during implantation with a dose of $7.2 \cdot 10^{17}$ ion/cm².

A solid solution of aluminum is formed in titanium along with intermetallic phases. It should be pointed out that with the growth of irradiation dose, the evolution of microdiffraction patterns type, implanted samples of VT6 titanium alloy is observed from ring to point-like ones, which is primarily due to the fact of an increase in the average particle size of the phases being formed. The detected phases are nanocrystalline ones. The formation of the aforesaid phases is sufficiently in line with the equilibrium phase diagram of the Ti-Al system.

Formation of titanium and aluminum oxides of the various stoichiometric compound, as well as titanium carbide, is also observed in addition to intermetallic phases (Table 1). The formation of aluminum and titanium oxides modifications, as well as their ratio, depends on the implantation conditions.

The proportion of aluminum oxide mainly localized in the oxide-carbide film area increases with the increase of implantation time in case of a relevant increase in the irradiation dose via aluminum ions.

It has been experimentally established that titanium oxides and titanium carbide are formed in trace amounts throughout the implanted layer, excluding the surface oxide-carbide film.

The evolution of microdiffraction patterns of VT6 titanium alloy implanted layers caused by an increase in the average grain size of the phases being formed is observed with an increase of irradiation dose. In this case, it is possible to distinguish three types of microdiffraction patterns, specifically:

- consisting of a set of continuous Debye rings;
- on which the Debye rings lose their continuity and split into separate reflexes;
- consisting of a set of stand-alone reflexes.

The microdiffraction pattern of VT6 titanium alloy ion-doped surface layer implanted with a dose of $2 \cdot 10^{17}$ ion/cm² (Figure 4, b) represents a set of separate Debye rings, where the most intense rings correspond to $\alpha_2\text{-Ti}_3\text{Al}$ phase (222). The intermetallic phases (Ti_3Al , TiAl) being formed based on the first mode are nanoscale ones, which can be clearly seen from the dark-field images (Figure 4, b, c). The average grain size of intermetallic phases was 18 nm (Table 1). Conglomerates of intermetallic particles have not been established at this mode of aluminum ions implantation.

A marked increase in the average grain size of crystalline phases and their unification into conglomerates is observed, which in turn leads to appearance of a plurality of point reflexes on the Debye rings of microdiffraction patterns (Figure 5, b) in VT6 titanium alloy surface layer implanted with an irradiation dose of $7.2 \cdot 10^{17}$ ion/cm².

Analysis of the dark-field images (Figure 6 c, d) implanted based on the mode with an irradiation dose of $7.2 \cdot 10^{17}$ ion/cm² of titanium layer showed that the intermetallic phases have an average grain size equal to 26 nm and their conglomerates have an average grain size equal to 45 nm. A further increase in the implantation dose leads to an increase in both the average grain size of intermetallic phases and their conglomerates (Lukyanenko *et al.*, 2015).

A further increase in the implantation dose

leads to an increase in both the average grain size of intermetallic phases and their conglomerates. When implanted with a dose of $5.2 \cdot 10^{18}$ ion/cm² (Table 1), the average grain size of the phases being formed is 42 nm already (Table 1), and the average grain size of the conglomerates is 130 nm (Figure 7, c, d).

The microdiffraction pattern of VT6 titanium alloy ion-doped layer with an implantation dose of $1.2 \cdot 10^{19}$ ion/cm² (Table 1) is a set of separate point reflexes (Figure 7, b). As can be seen from the dark-field image (Figure 7, c), the conglomerates of intermetallic phases formed during implantation with an implantation dose of $1.2 \cdot 10^{19}$ ion/cm² have a complex geometrical shape. Their average size was 280 nm.

According to the data obtained with the secondary ion mass spectrometry (SIMS) method, the maximum concentration of aluminum in VT6 surface alloy implanted with a dose of $2 \cdot 10^{17}$ ion/cm² is 35% at. and falls on a depth of 70 nm from the surface and does not exceed 5% at. at a distance of more than 200 nm. The peak concentration of aluminum also increases to 55% at. and its localization shifts to an area of deep depths with the increase in the irradiation dose. Thus, the maximum concentration of aluminum falls on a depth of 150 nm at an irradiation dose of $7.2 \cdot 10^{17}$ ion/cm², the maximum concentration of aluminum falls on a depth of 210 nm at an irradiation dose of $5.2 \cdot 10^{18}$ ion/cm² and the maximum concentration of aluminum falls on a depth of 410 nm at an irradiation dose of $1.2 \cdot 10^{19}$ ion/cm².

A smoother decrease is observed in the aluminum concentration profile with the increase in time and thus the implantation dose while the distance from the irradiated surface is increasing, which is due to the progress of diffusion processes.

The thickness of the entire implanted layer with consideration for the oxide-carbide film is to be determined by implantation conditions. A titanium sample made of VT6 alloy irradiated with aluminum ions with a dose of $2 \cdot 10^{17}$ ion/cm² has an ion-doped layer thickness equal to 420 nm (Table 1). The increase in the thickness of ion-doped layers is observed with the increase in the irradiation dose, and it is 2,100 nm in case of a mode with an aluminum ion irradiation dose of $1.2 \cdot 10^{19}$ ion/cm².

Comparison of concentration profiles with the results of electron microscopic studies (with consideration for Ti-Al system phase diagram)

made it possible to find out the localization spots of intermetallic phases being formed.

The aluminum concentration according to Ti—Al diagram corresponds to TiAl phase homogeneity range for an implantation dose of $2 \cdot 10^{17}$ ion/cm² starting from the surface oxide-carbide film boundary and to a depth of 110 nm. The lower boundary of TiAl phase existence shifts to deep depths with an increase of irradiation dose and reaches 800 nm in case of an implantation dose of $1.2 \cdot 10^{19}$ ion/cm².

According to the Ti-Al system state diagram, an intermetallic of Ti₃Al composition can be formed at lower aluminum concentrations. Accordingly, the region of its existence extends to deeper depths in comparison with the Ti-Al phase.

Ti₃Al exists at the depths from 45 to 195 nm for an implantation dose of $2 \cdot 10^{17}$ ion/cm², and Ti₃Al already exists at the depths from 300 to 1,700 nm at implantation with a dose of $1.2 \cdot 10^{19}$ ion/cm² (Table 1). A solid solution of aluminum in titanium is formed over the entire thickness of the ion-doped layer, excluding the oxide-carbide film.

Certain regularity is observed during the formation of surface layers on the alloys based on titanium when implanting VT6 titanium alloy with aluminum ions. The layout of phase layers being formed during the ion-implantation process is shown in Figure 8.

Two regions smoothly passing into each other can be distinguished from the irradiated surface of the sample deep into the implanted layer. The first region is an oxide-carbide film containing aluminum, oxygen, and carbon (zone IV). A region consisting of intermetallic phases and a solid solution of aluminum in titanium is formed from the oxide-carbide film existence boundary.

Three zones can also be distinguished in this region: the first zone contains three phases: α_2 -Ti₃Al, γ -TiAl and α -solid solution; the second zone consists of two phases: α_2 -Ti₃Al and α -solid solution; there is only a solid solution of aluminum in titanium in the third zone.

A regular increase in the thickness of the implanted layer and the oxide-carbide film is observed with the increase in the dose of VT6 titanium alloy specimen irradiation with aluminum ions (Figure 9, a).

The increase in the average grain size of intermetallic phases and their conglomerates is observed at the same time (Figure 9, b). Formation of conglomerates has been detected

at irradiation doses of $7.2 \cdot 10^{17}$ ion/cm² and more. Grains of intermetallic phases being formed on all four implantation modes of aluminum ions are nanoscale ones according to the classification (Smolyakova and Vershinin, 2012). Conglomerates can reach the sizes of up to 280 nm at the same time.

Structural changes in the depth of VT6 alloy sample after implantation with copper ions are shown in Figure 10. The white stripe from above is a platinum coating associated with the procedure for preparation of a sample for electron microscopy.

A two-phase layer of α and β phases, the grain sizes of which are 1–5 μm , follows further along with the depth. The dislocation density in the grains of α and β phases is insignificant.

To confirm the dimensional features of the element concentrations depth distribution obtained by the SIMS method, scanning was performed along the line in the transverse direction to the implanted layer. The scanning procedure is shown in accordance with Figure 10, from which it follows that there is a carbon adsorbed from the vacuum system residual atmosphere which penetrates to a depth of 70 nm in the surface ion-implanted layer in addition to doping atoms Al and V.

An electron microscopical technique of crystal lattice direct resolution was employed to study a fine structure of VT6 alloy implanted layer. Atomically disordered regions of VT6 alloy surface layer implanted with Cu ions are presented in accordance with Figure 11. It has been possible to identify atomically disordered regions in the surface layer, possibly being a transition structure during the phase transformation of a crystalline state into an amorphous state using this technique.

The nature of diffraction patterns shown in accordance with Figure 12 is indicative of a strongly pronounced fragmentation of structure; rings consisting of a large number of reflexes are clearly visible. The azimuthal blurring of certain reflections also indicates the presence of continuous misorientation of the microstructure fragments.

The study of a structural-phase state, elemental composition and mechanical properties of VT6 titanium alloy surface layers modified by the ion-implantation method with copper ions showed that modified layers with a thickness of 320 nm to 860 nm were obtained (Table 1). At the same time, the maximum concentration of

embedded copper reached 20-25 at. %.

There are reflections from TiCu_3 , TiCu , Ti_2Cu , Ti_3Cu intermetallics on the X-ray pattern taken from the surface of the sample exposed to ion implantation with copper. It has been determined that Ti_2Cu and Ti_3Cu intermetallics are present at a depth of $\sim 10 \mu\text{m}$ in large quantities.

Reflections from copper mainly disappear from the depth of 12-15 μm and the last reflection from copper and Ti_3Cu titanium compound has been recorded at a depth of 30-35 μm . No signs of intermetallic compounds have been detected at a depth of more than 35 μm , but reflections from α_{Ti} (010), (002), (011) and (012) lines are only observed.

It is difficult to say anything definitively for this system about the formation of TiCu intermetallic since its three most intense lines coincide with the intense lines of titanium, copper and TiCu_3 compound α -phase by the Woolf-Bragg angle. In all likelihood, TiCu intermetallic compound is present in the alloying zone. The intermetallic compounds are found in TiCu_3 , TiCu , Ti_2Cu , Ti_3Cu sequence, i.e., intermetallics increasingly enriched in titanium and copper depleted are observed with the distance from the sample surface.

Layer-By-Layer phase analysis showed that the intermetallics determining the surface layers hardening propagate to deep depths, which is a qualitative characteristic for improving the mechanical-and-physical properties of the surface layer when alloying with copper.

Oxygen and carbon impurities coming from the vacuum system residual atmosphere and absorbed by the target surface during ion-beam processing are observed in modified titanium layers along with copper ions. An 'oxide-carbide film' containing up to 18% at of copper and adsorbed impurities is formed on the surface during implantation of titanium samples. The titanium concentration is close to zero (no more than $\sim 0.2\%$ at.) in this film.

The concentration maxima of oxygen and carbon (up to 35% at.) fall within this region. The oxide-carbide film thickness increases with the increase of the ion-implantation process, and the amount of impurities increases accordingly but does not have a great impact on the dynamics of copper accumulation in titanium.

According to Auger electron spectroscopy profiles, the maximum copper concentration in VT6 alloy surface layer implanted with a dose of

$2 \cdot 10^{17}$ ion/cm² is 11% at and falls on a depth of 70 nm from the surface, as well as does not exceed 3% at a distance of more than 250 nm.

The peak concentration of copper also increases to 25% at with the increase in the irradiation dose, and its localization shifts to an area of great depths. Thus, the maximum concentration of copper falls on a depth of 100 nm at an irradiation dose of $7.2 \cdot 10^{17}$ ion/cm², the maximum concentration of copper falls on a depth of 170 nm for an irradiation dose of $5.2 \cdot 10^{18}$ ion/cm² and the maximum concentration of copper falls on a depth of 380 nm for an irradiation dose of $1.2 \cdot 10^{19}$ ion/cm².

A smoother decrease in the copper concentration profile, which is due to the progress of diffusion processes, is observed with the increase in time and thus the implantation dose while the distance from the irradiated surface is being increased.

The thickness of the entire implanted layer during copper implantation with consideration for the oxide-carbide film is determined in the same way as in the case of aluminum implantation, i.e., by the implantation conditions. A titanium sample made of VT6 alloy irradiated with copper ions with a dose of $2 \cdot 10^{17}$ ion/cm² has an ion-doped layer thickness of 320 nm (Table 1). The increase in the thickness of ion-doped layers is observed with the increase in the irradiation dose and is 860 nm in case of a mode with aluminum ions irradiation dose of $1.2 \cdot 10^{19}$ ion/cm².

The research of concentration profiles made it possible to find out the localization spots of formed intermetallic phases. The aluminum concentration according to Ti—Cu diagram corresponds to Ti₃Cu phase homogeneity range for an implantation dose of $2 \cdot 10^{17}$ ion/cm² starting from the boundary of the surface oxide-carbide film and down to a depth of 70 nm. The displacement of Ti₃Cu phase existence lower boundary to deep depths is observed with the increase in the irradiation dose and reaches 280 nm in case of implantation dose of $1.2 \cdot 10^{19}$ ion/cm².

An intermetallic of Ti₃Cu compound can be formed at lesser aluminum concentrations according to Ti—Cu system state diagram. The region of its existence extends to deep depths in comparison with TiCu phase accordingly.

Ti₃Cu exists at the depths from 30 to 70 nm for an implantation dose of $2 \cdot 10^{17}$ ion/cm² and it exists at the depths from 120 to 280 nm even when implanted with a dose of $1.2 \cdot 10^{19}$

ion/cm² (Table 1). A solid solution of copper in titanium is formed over the entire thickness of the ion-doped layer, excluding the oxide-carbide film.

A certain regular pattern is observed in the formation of surface layers on titanium-base alloys when implanting VT6 titanium alloy with aluminum ions. The layout of the phase layers formed during the ion-implantation process is shown in Figure 13.

Two areas smoothly passing into each other are located from the sample irradiated surface deep into the layer implanted via copper atoms. The first area is an oxide-carbide film containing copper, oxygen, and carbon in its composition. An area consisting of intermetallic phases and a solid solution of copper in titanium is formed from the boundary of the oxide-carbide film position.

The following zones can also be distinguished in this area: the outer zone contains the following phases: TiCu₃ and α -solid solution; the second zone consists of three phases: TiCu + Ti₂Cu and α -solid solution; there is only a solid solution of copper in titanium in the third zone.

The increase of the implanted layer and oxide-carbide film thickness is observed with the increase in the irradiation dose of VT6 titanium alloy samples via copper ions as in the case of implantation with aluminum ions.

The increase in the average grain size of intermetallic phases is observed at the same time. Formation of conglomerates has not been established during implantation with copper ions. Grains of intermetallic phases were formed based on the entire modes of copper ions implantation are nano-scale ones regarding sizes.

Mechanical and tribological properties of VT6 titanium alloy ion-doped samples. Changes in microhardness are characterized by relevant curves shown in Figure 14, depending on the penetration depth of initial titanium and ion-doped samples indenter.

The microhardness at the depths of up to 1.8-2 microns exceeds the microhardness at the relevant depth for VT6 initial titanium alloy for the entire implanted samples.

The increase in microhardness of 1.4—2.9 times is observed in the near-surface region in the thickness of ~ 0.9 -1 μ m for a sample of VT6 alloy implanted with aluminum ions with a dose of $1.2 \cdot 10^{19}$ ion/cm².

The measurement results of microhardness correlate quite well with the

distribution data of embedded elements (aluminum and copper) in the depth of VT6 titanium alloy.

It has been established according to SIMS and transmission submicroscopical research that the titanium sample implanted at the maximum irradiation dose is characterized by the maximum thickness of the ion-doped layer. Therefore, it was of interest to research the tribological properties of a given sample in comparison with the original VT6 alloy.

The diagram of VT6 alloy samples wear rate change (Figure 15) as a function of temperature has shown that there is no significant difference in the wear rate for ion-doped and initial alloy at room temperature.

However, there is a marked increase in the wear of VT6 initial alloy with the temperature increase in contrast to the doped alloy, which wears does not change in the researched temperature range. Thus, as the temperature rises up to 200°C, the wear level of VT6 initial alloy has increased by a factor of 2.3, while the wear level of the implanted alloy has remained almost unchanged.

The average friction factor of test samples has been monitored during the performance of tribological tests. The friction factor of VT6 alloy implanted layer is much lower at the elevated temperatures than for the initial state. The average friction factor of VT6 alloy being in the initial state was increasing with the rise of sample temperature while it almost was not changing for the ion doped alloy.

It should be noted that the implantation of aluminum ions into VT6 titanium alloy is more effective as compared with implantation of copper ions from the standpoint of increasing its wearability at an elevated temperature.

Thus, the formation of multiphase ion-doped layers containing intermetallic phases during implantation of VT6 alloy with aluminum and copper makes it possible to increase the alloy mechanical properties significantly. The formation of ion-doped layers consisting of several regions different from each other in phase composition and base of an alloy is important for practical purposes.

The three-phase field containing Ti_3Al , $TiAl$ intermetallide phases and solid solution (in case of aluminum implantation) and $TiCu + Ti_2Cu$ as well as α -solid solution (in case of copper implantation) is the closest to the surface. The increase in the thickness of this three-phase field

contributes to significant improvement in mechanical properties.

Stable corrosion and oxidation resistant material is obtained as a result of the formation of intermetallic phases mixture. It should be highlighted that the surface oxide-carbide film having high adhesion also contributes to the improvement of the mechanical-and-physical properties of the implanted samples.

The estimation of the residual stresses value in VT6 alloy samples surface layer after implantation with metal ions with a high implantation dose has been performed within the framework of this work.

Flat sheet specimens of 1 mm in thickness and 10 mm width were used to research the ion implantation effect on residual stresses in VT6 titanium alloy surface layer. Samples were exposed to annealing in a vacuum furnace before implantation in order to relieve the stresses that arose during rolling of the sheets. The annealing temperature was 750 °C.

The implantation dose was being varied within the range of $5 \cdot 10^{16} - 5 \cdot 10^{19}$ ion/cm² in addition to varying the implanter cathode material during experiments.

An experimental plant was being used to measure the level of residual stresses in VT6 alloy samples surface layer after implantation (Gusev and Rempel, 2001; Lotsko and Milman, 1993). The plant operation principle is based on measuring the sample surface displacement during metal stripping via the electrochemical method and automatic recalculation of sample surface displacements into residual stresses by means of a special program to be measured via a laser displacement controller.

The measurement results of residual stresses are shown in Figure 16. These data were obtained at an implantation dose of $5 \cdot 10^{17}$ ion/cm². It follows from the obtained results that the residual stresses at a level of 85 MPa are present in the initial sample without implantation (Uchevatkina *et al.*, 2016).

Implantation with aluminum ions contributes to the appearance of compressive stresses of the order of 540–550 MPa in the surface layer, which relaxes within a layer of 56–60 μm in thickness.

Further growth of residual stresses of up to 670–685 MPa is observed during implantation with copper in case of the stress distribution area of approximately 100 μm in depth from the sample surface.

The highest residual stresses were being observed with successive implantation with aluminum and copper. They have amounted to approximately 850 MPa at a depth of the compressive stress distribution range of 160–180 μm .

Thus, it can be said that the complication of implantation (successive implantation) contributes to an increase in the level of residual compressive stresses in the irradiated sample and in the depth of their propagation.

It may be concluded while analyzing the presented graphical dependences of changing residual compressive stresses in the surface layer depth on the processing mode within the indicated process range that the absolute value of residual stresses increases (Figure 17) and then drastically decreases while increasing an implantation dose both with aluminum and copper.

The effect of reducing residual stresses in the implantation dose range $(1-5) \cdot 10^{18}$ ion/cm² may be related to the formation of new phases in the implanted layer.

It is important to note that the presence of residual stresses of the second kind, i.e., micro stresses were identified during measurement of residual stresses, which indicates the availability of a high dislocation density in them.

It may be concluded based on the experimental studies that the residual compressive stresses of the surface layer increase multiple times in comparison with the initial state after VT6 titanium alloy implantation. The regularity of residual compressive stress increase was established with the increase in the implantation dose of up to $5 \cdot 10^{17}$ ion/cm², following which the decrease of residual stresses is observed with a further increase of an implantation dose. Generally, the occurrence depth of residual stresses slightly exceeds the depth of the implanted layer.

It may be concluded in studying electron microscopic images of VT6 titanium alloy surface layer obtained via X-raying the thinned films in the thickness of 100 nm at a scale of 500...50 nm ($\times 80000 \dots 1060000$) via an electron beam that ion-implantation process makes it possible to obtain a finely divided nanostructure (Domkus and Pranavičius, 1990) on the irradiated sample surface (Figure 18).

The particle size was about 30 nm in the initial state in the near-surface layer. The particle

size has increased to 40...55 nm as a result of ion implantation using a copper cathode with a dose of $5 \cdot 10^{17}$ ion/cm², and the particle size has increased to 70...85 nm at an implantation dose of $5 \cdot 10^{19}$ ion/cm².

The increase in dislocation density of approximately 8.9–9.4 times was observed at initial dislocation density $\rho_{\text{Dinit}} \approx 5.4 \cdot 10^{11}$ (cm⁻²) as a result of ion-implantation process with a dose of $5 \cdot 10^{17}$ ion/cm² and the increase in the dislocation density was approximately 19.6 times in case of implantation with a dose of $5 \cdot 10^{18}$ ion/cm², (Table 2).

It may be concluded while analyzing the submitted data that the dislocation density increases, and the particle sizes increase while increasing an implantation dose.

4. CONCLUSION

The high-intensity ion implantation mode makes it possible to obtain the implanted layers which thickness exceeds the projective range amount of aluminum and copper ions by times. The increase in the thickness of implanted layers was found out with the increase in the implantation dose and reaching 2.1 μm in case of aluminum implantation as well as achieving 0.86 μm in case of copper implantation with a maximum irradiation dose ($1.2 \cdot 10^{19}$ ion/cm²).

Nanocrystalline phases of $\alpha_2\text{-Ti}_3\text{Al}$, $\gamma\text{-TiAl}$ intermetallics and a solid solution of aluminum in variable composition titanium are being formed in the implanted layer during ion implantation of aluminum into VT6 titanium alloy.

The growth of formed phase grains is observed in the surface ion-doped layers with the implantation dose increase. Finely divided grains of intermetallic phases are united into conglomerates of irregular geometrical shape at an irradiation dose of $7.2 \cdot 10^{17}$ ion/cm² and more. Formation of oxides and carbides (TiO_2 , TiO , Al_2O_3 , TiC) in various modifications in traces is also possible during aluminum implantation.

It has been shown that several polyphase regions are distinguished in the implanted layers of VT6 alloy. A three-phase field containing Ti_3Al , TiAl , and a solid aluminum solution in titanium is most close to the irradiated surface and has the greatest influence on the physical and mechanical properties of implanted samples.

A three-phase field containing Ti_3Al , TiAl intermetallic phases and a solid solution (in case of aluminum implantation) both $\text{TiCu} + \text{Ti}_2\text{Cu}$ and

α -solid solution (in case of copper implantation) is closest to the surface when implanting VT6 alloy with copper ions. The increase in the thickness of this three-phase field contributes to a significant improvement in mechanical properties.

The surface layers of VT6 alloy ion-doped with aluminum and copper have substantially better macroscopic features in comparison with the original target material. A significant increase in microhardness is observed in the surface layer in the thickness of up to 2.1 μm .

The wearability and friction factor of VT6 alloy implanted samples almost do not change during testing at room temperature (20–25°C) in comparison with the initial state. The wear level of VT6 initial alloy increased by 2.3 times, while the wear level of the implanted alloy changed little, if at all with the increase in the temperature of samples of up to 200°C.

VT6 alloy sample implanted with aluminum ions at an irradiation dose of $1.2 \cdot 10^{19} \text{ion/cm}^2$ is characterized by a maximum thickness of a three-phase intermetallic area of the entire implanted layer (2.1 μm) as well as enhanced mechanical and tribological properties.

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TABLES

Table 1. Basic characteristics of VT6 titanium alloy surface layers implanted with aluminum and copper ions

System	Implantation dose, ion/cm ²	Ion-doped layer thickness, nm	Average size, nm		Phase composition	
			Phases	Conglomerates	Intermetallics (phase localization areas)	Oxides and carbides
Ti-Al	2•10 ¹⁷	420	18	-	γ-TiAl (95–110 nm); α ₂ -Ti ₃ Al (45–195 nm); α-solid solution (50–420 nm)	TiO (monocl.), TiO ₂ (hex.), TiC (cub.), Al ₂ O ₃ (hex.)
	7.2•10 ¹⁷	680	26	45	γ-TiAl (75–310 nm); α ₂ -Ti ₃ Al (90–395 nm); α-solid solution (90–680 nm)	TiO ₂ (hex.), TiC (cub.), γ-Al ₂ O ₃
	5.2•10 ¹⁸	1550	42	130	γ-TiAl (125–610 nm); α ₂ -Ti ₃ Al (125–700 nm); α-solid solution (125–1550 nm)	TiO ₂ (hex.), γ-Al ₂ O ₃
	1.2•10 ¹⁹	2100	55	280	γ-TiAl (225–800 nm); α ₂ -Ti ₃ Al (300–1700 nm); α-solid solution (300–2100 nm)	TiO (monocl.), TiO ₂ (hex.), γ-Al ₂ O ₃ ; γ'-Al ₂ O ₃
	2•10 ¹⁷	320	41	-	Ti ₃ Cu (30-70 nm); α-solid solution (70–320 nm)	TiO (monocl.), TiO ₂ (hex.)
Ti-Cu	7.2•10 ¹⁷	460	52	-	Ti ₂ Cu (10-30 nm); Ti ₃ Cu (30-110 nm); α-solid solution (110–460 nm)	TiO ₂ (hex.)
	5.2•10 ¹⁸	680	61	-	TiCu + Ti ₂ Cu (10-70 nm); Ti ₃ Cu (70-180 nm); α-solid solution (180–680 nm)	TiO ₂ (hex.)
	1.2•10 ¹⁹	860	70	-	TiCu ₃ (10-30 nm); TiCu + Ti ₂ Cu (40-120 nm); Ti ₃ Cu (120-280 nm); α-solid solution (280–860 nm)	TiO ₂ (hex.)

Table 2. Change in the dislocation density depending on the implantation dose amount of aluminum and copper

Implantation dose, ion/cm ²	Dislocation density, cm ⁻²	
	Aluminum implantation	Copper implantation
Initial state	$5 \cdot 10^{11}$	$5 \cdot 10^{11}$
$5 \cdot 10^{16}$	$6.6 \cdot 10^{11}$	$9.6 \cdot 10^{11}$
10^{17}	$9.7 \cdot 10^{11}$	$2.7 \cdot 10^{12}$
$5 \cdot 10^{17}$	$1.7 \cdot 10^{12}$	$4.7 \cdot 10^{12}$
10^{18}	$3.8 \cdot 10^{12}$	$9.8 \cdot 10^{12}$
$5 \cdot 10^{18}$	$4.3 \cdot 10^{12}$	$8.8 \cdot 10^{12}$

FIGURES

Figure 1. Kinetic dependences of VT6 alloy samples mass loss on the test time: 1 – VT6 coarse-grained (CG) titanium alloy; 2 – VT6 ultra-fine-grained (UFG) titanium alloy.

Figure 2. VT6 coarse-grained alloy sample friction surface (x100).

Figure 3. VT6 titanium alloy wear fragments (a, x200; b, x500). The obtained results make us possible to assume that the sample material

Figure 4. Light-field electron microscopic image (a), microdiffraction pattern (b) and dark-field images in Ti_3Al (222), $TiAl$ (311) (c) reflexes of VT6 titanium alloy surface layer implanted with aluminum ions implanted with aluminum ions with an implantation dose of $2 \cdot 10^{17}$ ion/cm².

Figure 5. Light-field electron microscopic image (a), microdiffraction pattern (b) and dark-field images in Ti_3Al (004), $TiAl$ (222) reflexes (left arrow at b) (c) and Ti_3Al (201), $TiAl$ (111) (right arrow at b) (d) of VT6 titanium alloy surface layer implanted with aluminum ions with an implantation dose of $7.2 \cdot 10^{17}$ ion/cm².

Figure 6. Light-field electron microscopic image (a), microdiffraction pattern (b) and dark-field images in Ti_3Al (302), $TiAl$ (003) (c) and Ti_3Al (004), $TiAl$ (222) (d) reflexes of VT6 titanium alloy surface layer implanted with aluminum ions with an implantation dose of $5.2 \cdot 10^{18}$ ion/cm².

Figure 7. Light-field electron microscope image (a), microdiffraction pattern (b), and a dark-field image in TiAl (200) reflex (c) of VT6 titanium alloy surface layer implanted with aluminum ions with an implantation dose of $1.2 \cdot 10^{19}$ ion/cm².

Figure 8. Schematic structure of titanium surface ion-doped layers implanted with aluminum ions: I – single-phase zone (solid solution of Al in Ti), II – two-phase zone (solid solution of Al in Ti, Ti₃Al), III – three-phase zone (solid solution of Al in Ti, Ti₃Al and TiAl); IV – oxide-carbide film.

a

b

Figure 9. Influence of aluminum implantation conditions on the thickness of VT6 titanium alloy oxide-carbide and ion-doped layers (a). The average grain size of formed intermetallic phases and their conglomerates (b). Implantation doses: 1 - $2 \cdot 10^{17}$ ion/cm², 2 - $7.2 \cdot 10^{17}$ ion/cm², 3 - $5.2 \cdot 10^{18}$ ion/cm², 4 - $1.2 \cdot 10^{19}$ ion/cm².

Figure 10. The layer implanted with copper ions and VT6 alloy grained structure at various magnifications (scale rule = 0.2 μm)

Figure 11. Atomically disordered regions of VT6 alloy surface layer implanted with Cu ions (scale rule = 5 nm)

Figure 12. Diffraction patterns

Figure 13. Influence of copper implantation conditions on the thickness of VT6 titanium alloy oxide-carbide and ion-doped layers

a

b

Figure 14. Change in microhardness of VT6 titanium alloy samples implanted with aluminum (a) and copper ions (b). Implantation dose: 1 – VT6 initial alloy without implantation; 2 - $7.2 \cdot 10^{17}$ ion/cm²; 3 - $5.2 \cdot 10^{18}$ ion/cm²; 4 - $1.2 \cdot 10^{19}$ ion/cm²

Figure 15. Wear of VT6 titanium alloy samples at various test temperatures: 1 – without implantation; 2 – implantation of aluminum ions; 3 – implantation of copper ions. The implantation dose is $1.2 \cdot 10^{19}$ ion/cm²

Figure 16. Distribution curve of the residual macrostresses magnitude σ'_{res} (MPa) of VT6 titanium alloy surface layer over the depth h (μm) as a result of layer-by-layer analysis: 1 – initial state; 2 – implantation with aluminum; 3 – implantation with copper; 4 – successive implantation with aluminum and copper

Figure 17. Dependence of the residual compressive macrostresses σ'_{res} (MPa) variation of VT6 titanium alloy surface layer on the implantation dose (Cu cathode): 1 – implantation dose $5 \cdot 10^{16}$ ion/cm²; 2 – implantation dose 10^{17} ion/cm²; 3 – implantation dose $5 \cdot 10^{17}$ ion/cm²; 4 – implantation dose 10^{18} ion/cm²; 5 – implantation dose $5 \cdot 10^{18}$ ion/cm²;

Figure 18. Nanostructure of the near-surface layer (thickness 100 nm) of VT6 titanium alloy before and after ion-implantation process using a copper cathode (x235000 on the surface): a – dose of $5 \cdot 10^{17}$ ion/cm²; b – dose of $5 \cdot 10^{18}$ ion/cm²; c – initial state